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FEDERAL STANDARD

TESTING METHOD OF LUBRICANTS, LIQUID FUELS, AND RELATED PRODUCTS

AMSC N/A FSC 91GP

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August 24, 2006

Foreword

This Federal Test Method Standard is issued in loose leaf form to permit the insertion or removal of new or revised sections and test methods.

All users of Federal Test Method Standards should keep them up to date by inserting revised or new sections and test methods as issued and removing superseded and cancelled pages.

New and revised material and cancellations will be issued under Change Notices which will be numbered consecutively and will bear the date of issuance. Change Notices should be retained and filed in front of the Alphabetical Index of the Standard until such time as they are superseded by a reissue of the entire standard.

This federal standard is approved by the Commissioner, Federal Supply Services, General Service Administration for use of all Federal agencies.

CONTENTS

Introduction

TESTING METHOD OF LUBRICANTS, LIQUID FUELS, AND RELATED PRODUCTS

1. SCOPE

1.1 This test method standard is composed of standard test methods for sampling, inspecting and testing lubricants, liquid fuels and related products. It is issued pursuant to the Federal Property and Administration Services Act of 1949, as amended and its application to the purchase of commodities referred to herein is mandatory on all Federal agencies.

1.2 The test methods of this standard, when cited in a specification form an integral part of that specification. Such a citation ,although referenced to the basic number of a method is intended to apply to the latest version of the method as revised (see paragraph 2)and as amended by any subsequent Change Notices to this standard

CAUTION

Change Notices are not cumulative.
Therefore all notices must be consulted to insure that the latest requirements are observed

1.3 Some methods are identical to those published by the ASTM International. These methods are listed in the indexes together with their corresponding ASTM method numbers and the details of such test methods are omitted from this standard.

2. NUMBERING SYSTEM

2.1 The test methods are identified by numerical designations, each consisting of a basic number and in some instances a revision number (decimal)

a. Basic number. The basic number of a method is its official designation and should be used without revision number whenever the method is referenced. The number always refers to the same method of determination and the same accuracy, and the same end results regardless of revision status.

b. Revision Number. Revision numbers appear as decimal addition to the basic numbers. These are assigned to basic numbers when changes are made in the method to clarify it or to give additional details that will increase the reproducibility of the test results.

3. NOTES

3.1 Purchase of specification and Standards. Federal and military specifications and standards may be obtained under general provisions in the index of specifications and standards. They can be found in the websites also. Federal Government activities may obtain the index from established distribution points within their agencies. All others may purchase the index with cumulative monthly supplements as issued from the Superintendent of documents U.S. Government Printing office, Washington D.C 20402

3.2 Purchase of ASTM test methods. ASTM test methods may be purchased from the ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428

3.3 Patent notice. When Government drawings, specifications or other data are used for any purpose other than in connection with a definitely related government operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the government may have furnished, formulated or in any way supplied the said drawings, specifications or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation or conveying any rights or permission to manufacturer, use or sell any patented invention that may in any way be related thereto.

Table I. Numerical index of test method

Method No	Method Title	Replacement test	Test cancelled by
103.6	Color of gasoline		
202.1	Cloud intensity at low temperature		
203.1	Pour stability at low temperature		
204.1	Diluted pour point	None	Navy
313.3	Penetration of lubricating greases after prolonged working		
321.3	Oil separation from lubricating greases (static technique)	ASTM D 6184	Navy
335.3	Gear wear		
339.6	Performance of diesel engine lubricating oils under severe	None	Navy
347.3	Performance of diesel engine lubricating oils under severe operating condition	None	Navy
350.2	Evaporation loss of lubricating greases and oils (high Temperature)		
352.1	Effect of evaporation on flammability		
353.1	Evaporation		
354.1	Performance of lubricating oils in a two cycle diesel engine under cyclic,turbo-supercharged conditions	None	Army
355	Performance of lubricating oils in a two cycle diesel engine under cyclic,turbo-supercharged conditions	ASTM D 5862	Army
361.4	Stroking properties of hydraulic brake fluids		
500.1	Induction system deposit (ISD) tendencies of motor gasoline	ASTM D 6241	Army
550.1	Emulsification tendencies of petroleum fuels by multiple contact extractions		
1110.2	Quenching speed		
1151.2	Explosive vapors in boiler fuel oil		
1303.2	Flock point (refrigerant compressor oil)	None	Navy
2508.1	Thermal stability of lubricating and hydraulic fluids		
3005.4	Dirt content of grease		
3006.3	Contamination in engine oil	None	Navy
3007.2	Water displacement and water stability		
3009.3	Solid particle contamination in hydraulic fluids		
3010.1	Solid particle contamination in aircraft turbine engine Lubricants (gravimetric procedure)		
3011.1	Particulate contamination in aerospace hydraulic fluid using particle counter	None	AF

3012	Determination of particulate matter in aerospace hydraulic fluids		
3013	Determination of particulate contamination in synthetic turbine engine lubricants		
3110.1	Fatty oil in cutting oils by infrared spectrophotometry		
3180.2	Sulfur in cutting fluids (active and total)		
3201.7	Emulsion(petroleum and petroleum –like products)		
3205.3	Emulsion (soluble cutting oils)		
3213.1	Foaming characteristics of air craft turbine lubricating oils (static foam test)	ASTM D 892	Navy
3214.1	Foaming characteristics of aircraft turbine lubricating oils (dynamic foam test	None	AF
3289.1	Total solids in water for aircraft injection system	None	AF
3403.2	Compatibility of turbine lubricating oils		
3407.2	Oxidation and thermal stability of aircraft engine lubricating oils (CLR engine)	None	Navy
3410.1	High temperature deposits and oils degradation characteristics Of aviation turbine oil		
3411.1	Thermal stability and corrosivity of aircraft turbine engine lubricants		
3430.2	Compatibility characteristics of universal gear lubricants		
3432.1	Compatibility of elastomers with aircraft turbine lubricants		
3433	Compatibility of synthetic aircraft turbine lubricants with silicone rubber		
3440.1	Storage volubility characteristics of universal gear lubricants		
3450	Bearing deposition of aviation turbine engine lubricants		
3451.4	Stability of lubricating oils (work factor)	None	Navy
3452.2	Bearing compatibility of turbine oils		
3456.2	Channeling characteristics of lubricants		
3457.2	Hydrolytic stability		
3458.1	Low temperature stability test for oil		
3459.1	Low temperature stability		
3463.2	Stability of grease in hot water (water immersion)	ASTM D 2603	Navy
3465.1	Storage stability test or fluids and lubricants		
3467.1	Storage stability of lubricating grease		
3470.1	Homogenity and miscibility of oils		
3472	Sonic shear stability of polymer-containing oils	ASTM D	Navy

		2603	
3480.1	Volatility of blended oils		
3500.1	Monobasic acid components of synthetic ester lubricants by gas chromatography		
3603.5	Swelling of synthetic rubbers	ASTM D 4289	Army
3604.2	Swelling of synthetic rubber by aircraft turbine lubricants		
3710.1	Molybdenum disulfide purity		
3720.2	Molybdenum disulfide content of lubricating grease		
3722.2	Molybdenum disulfide content of non-soap thickened lubricating greases		
3816.1	Film thickness of dry solid film lubricants		
4001.3	Corrosion protection by coating: salt-spray (fog) test		
5003.2	Deposit- foaming tendencies of aircraft turbine lubricants		
5101.7	Neutrality (qualitative)	ASTM D 1093	Navy
5102.1	Acid and base number by extraction (color-indicator titration)	None	Navy
5304.5	Corrosiveness of greases or semi-solid products at 25°C		
5305.1	Corrosiveness of lubricants at 232° C (450° F)		
5306.5	Corrosiveness of emulsifyable cutting fluids		
5307.2	Corrosiveness and oxidation stability of aircraft turbine	ASTM D 4636	AF
5308.7	Corrosiveness and Oxidation Stability of Light Oils	ASTM D 4636	Army
5309.5	Corrosiveness of grease (copper strip, 100 °C)		
5321.2	Corrosion of lead by lubricating oils		
5322.2	Corrosiveness of oil on bimetallic couple	ASTM D 6547	ARMY
5327	Fuel system icing inhibitors in hydrocarbon fuels (iodometric method)	None	AF
5329.2	Corrosion protection (humidity cabinet)		
5330	Fuel system icing inhibitor in hydrocarbon fuel (calorimetric method)	None	AF
5331.1	Corrosion protection of steel against sulfurous acid-salt spray by solid film lubricants		
5333.1	Fuel system icing inhibitor by freezing point depressing method	None	AF
5340.2	Fuel system icing inhibitor in hydrocarbon fuels (refractometer method)	ASTM D 5006	AF

5341	Fuel system icing inhibitor in hydrocarbon fuels (redox method)	None	AF
5342	Fuel system icing inhibitor in hydrocarbon fuels (hand refractometer method)	ASTM D 5006	Navy
5350.1	Silting index of hydrocarbon fuels	None	Navy
5414.4	Resistance of grease to fuel		
5415.1	Resistance of grease to water and a 1:1 water ethanol solution		
6050.1	Nitrate- type ignition improvers in diesel fuel		
6052.1	High temperature , High pressure spray ignition		
6053.1	Manifold ignition test		
6505.2	Load carrying capacity of universal gear lubricants by the Timken machine	ASTM D 2782	Navy
6508.2	Load carrying capacity of lubricating oils (Ryder gear machine)		
6509.2	Gear-fatigue characteristics of aircraft gas turbine lubricants at 204°C	None	Navy
6511.2	Load carrying capacity of lubricating oils at 204° C	None	Navy
6516.2	Oscillation test of grease in helicopter bearings	None	Navy
6517	High temperature gear load –carrying capacity of lubricating oils		
6520.1	Extreme pressure properties of lubricants (four ball testers)	ASTM D 2783	Navy
7501	Low Temperature Torque Test Method for Lubricating Greases		
7502	Solvent Cleaning power by a Soil Test Method		
10000	Material Handling Safety Precaution		

Table II - Alphabetical key word index of test methods

Method Title	Method No	Replacement test	Test Cancelled by
Acid and base number by extraction (color indicator titration)	5102.1	None	NAVY
Bearing deposition of aviation turbine engine lubricants	3450		
Bearing compatibility of turbine oils	3452.2		
Channeling characteristics of lubricants	3456.2		
Cloud intensity at low temperature	202.1		
Color of gasoline	103.6		
Compatibility characteristics of universal gear lubricants	3430.2		
Compatibility of elastomers with air craft turbine lubricants	3432.1		

Compatibility of synthetic air craft turbine lubricants with silicone rubber	3433		
Compatibility of turbine lubricating oils	3403.2		
Contamination in engine oil	3006.3	None	Navy
Corrosiveness of emulsifyable cutting fluids	5306.5		
Corrosiveness of grease(copper strip, 100° C)	5309.5		
Corrosiveness of greases or semi-solid products at 25°C	5304.5		
Corrosiveness of lubricants at 232°C (450°F	5305.1		
Corrosiveness of oil on a bimetallic couple	5322.2	ASTM D 6547	ARMY
Corrosion of lead by lubricating oils	5321.2		
Corrosion protection by coating : salt-spray (fog) test	4001.3		
Corrosion protection (humidity cabinet)	5329.2		
Corrosion protection of steel against sulfurous acid- salt spray by solid film lubricants	5331.1		
Corrosiveness and oxidation stability of aircraft turbine engine lubricants	5307.2	D 4636	AF
Diluted pour point	204.1	None	Navy
Dirt content of grease	3005.4		
Determination of particulate matter in aerospace hydraulic fluids	3012		
Determination of particulate contamination in synthetic turbine engine lubricants	3013		
Deposit- foaming tendencies of aircraft turbine lubricants	5303		
Evaporation loss of lubricating greases and oils (high temperature)	350.2		
Extreme pressure properties of lubricants (four ball tester)	6520.1	D 2783	NAVY
Effect of evaporation of flammability	352.1		
Evaporation	353.1		
Emulsification tendencies of petroleum fuels by multiple contact extractions	550.1		
Explosive vapors in boiler fuel oil	1151.2		
Emulsion (petroleum and petroleum like products)	3201.7		
Emulsion(soluble cutting oils)	3205.3		
Fatty oil in cutting oils by infrared spectrophotometry	3110.1		
Film thickness of dry solid film lubricants	3816.1		
Flock point (refrigerant compressor oil)	1303.2	None	NAVY
Foaming characteristics of air craft turbine lubricating oil (dynamic foam test)	3214.1	NONE	AF

Foaming characteristics of air craft turbine lubricating oils (static foam test)	3213.1		
Fuel system icing inhibitor in hydrocarbon fuels (calorimetric method)	5330	ASTM D 5006	AF
Fuel system icing inhibitors by freezing point depression method	5333.1	NONE	AF
Fuel system icing inhibitor in hydrocarbon fuel (iodometric method)	5327	None	AF
Fuel system icing inhibitor in hydrocarbon fuel (redox method)	5341	NONE	AF
Fuel system icing inhibitor in hydrocarbon fuel (refractometer method)	5340.2	ASTM D 5006	AF
Fuel system icing inhibitor in hydrocarbon fuel (hand refractometer method)	5342	ASTM D 5006	NAVY
Gear-fatigue characteristics of aircraft gas turbine lubricants at 204°C	6509.2	None	NAVY
High temperature , High pressure spray ignition	6052.1		
High temperature deposits and oil degradation characteristics of aviation turbine oils	3410.1		
High temperature gear load –carrying capacity of lubricating oils	6517		
Homogeneity and miscibility of oils	3470.1		
Hydrolytic stability	3457.2		
Induction system deposit (ISD) tendencies of motor gasoline	500.1	ASTM D 6241	ARMY
Low temperature stability test for oil	3458.1		
Low temperature stability	3459.1		
Low Temperature Torque Test Method For Lubricating Greases	7501		
Load carrying capacity of lubricating oils (Ryder gear machine)	6508.2		
Load carrying capacity of lubricating oils at 204° C	6511.2	None	NAVY
Load carrying capacity of universal gear lubricants by the Timken machine	6505.2	ASTM D 2782	NAVY
Molybdenum disulfide purity	3710.1		
Molybdenum disulfide content of lubricating grease	3720.2		
Molybdenum disulfide content of non-soap thickened lubricating greases	3722.2		
Monobasic acid components of synthetic ester lubricants	3500.1		
Manifold ignition test	6053.1		
Materiel handling safety precaution	10000		
Nitrite type ignition improvers in diesel fuel	6050.1		

Neutrality (qualitative)	5101.7	D 1093	NAVY
Oil separation from lubricating greases (static technique)	321.3	D 6184	NAVY
Oxidation and thermal stability of aircraft engine lubricating oils (CLR engine)	3289.1	None	AF
Oscillation test of grease in helicopter bearings	6516.2	None	NAVY
Oxidation and thermal stability of aircraft engine lubricating oils(CLR engine)	3407.2	None	NAVY
Particulate contamination in aerospace hydraulic fluid	3011.1	None	AF
Penetration of lubricating greases after prolonged working	313.3		
Performance of arctic lubricating oils in a two cycle diesel engine under cyclic, turbo supercharged conditions	354.1	None	ARMY
Performance of aviation piston engine oils in CLR engine (low temperature dispersancy and detergency)	347.3	None	NAVY
Performance of diesel engine lubricating oils under severe operating conditions	339.6	None	NAVY
Performance of lubricating oils in a two cycle diesel engine under cyclic turbo supercharged conditions	355	ASTM D 5862	ARMY
Pour stability at low temperature	203.1		
Quenching speed	1110.2		
Resistance of grease to fuel	5414.4		
Resistance of grease to water and a 1:1 water-ethanol solution	5415.1		
Silting index of hydrocarbon fuels	5350.1	None	NAVY
Solid particle contamination in hydraulic fluids	3009.3		
Solid particle contamination in aircraft turbine engine lubricants (gravimetric procedure)	3010.1		
Solvent cleaning power by a soil test method	7502		
Sonic shear stability of polymer-containing oils	3472		
Stability of lubricating oils (work factor)	3451.4	None	NAVY
Storage volubility characteristics of universal gear lubricants	3440.1		
Storage stability test or fluids and lubricants	3465.1		
Storage stability of lubricating grease	3467.1		
Stroking properties of hydraulic brake fluids	361.4		
Thermal Stability of Lubricating and hydraulic Fluid	2508.1		
Thermal stability and corrosivity of aircraft turbine engine lubricants	3411.1		
Total solids in water for aircraft injection system	3289.1	None	AF

Volatility of blended oils	3480.1		
Water displacement and water stability	3007.2		
Sulfur in cutting fluids (active and total)	3180.2		
Swelling of synthetic rubber by aircraft turbine lubricants	3604.2		

Table III. Numerical Index of Cancelled and Superseded test Method

Title of cancelled Test Method	Cancelled method Number	Substitute Method
Diluted Pour Point	204.1	None
Oil Separation from Lubricating Greaser (Static Technique)	321.3	ASTM D 6184
Performance of Diesel Engine Lubricating Oil Under severe operating condition	339.6	None
Performance of Aviation Piston Engine Oil In CLR Engine(low temperature dispersancy and detergency)	347.3	None
Performance of Arctic Lubricating Oil in a two cycle diesel Engine under cyclic, turbo supercharge condition	354.1	None
Performance of Engine Lubricating Oils in a Two Cycle Diesel Engine under cyclic Turbo supercharge condition	355	ASTM D 5862
Induction System Deposit (ISD) Tendencies of Motor Gasoline	500.1	ASTM D 6241
Flock Point (refrigerant compressor oil)	1303.2	None
Contamination of Engine oil	3006.3	None
Particulate Contamination in Aerospace Hydraulic Fluids Using the HIAC Particulate Counter	3011	None
Foaming Characteristics of air craft turbine lubricating oils(Static foam test)	3213.1	ASTM D 892
Foaming characteristics of air craft turbine lubricating oils(dynamic foam test)	3214.1	NONE
Total solids in water for aircraft injection system	3289.1	NONE
Oxidation and Thermal stability of air craft turbine engine lubricating oils(CLR Engine)	3407.2	NONE
Stability of Grease in Hot Water (Water Immersion)	3463.2	ASTM D 2603
Stability of lubricating oils (work factor)	3451.4	None
Sonic shear stability of polymer-containing oil	3472	ASTM D 2603
Swelling of Synthetic Rubbers	3603.5	ASTM D 4289
Neutrality (Qualitative)	5101.7	ASTM D 1093

Acid and base number by extraction (color indicator)	5102.1	None
Corrosiveness and oxidation stability of aircraft turbine lubricants	5307.2	ASTM D 4636
Corrosiveness and Oxidation Stability of Light Oils	5308.7	ASTM D 4636
Corrosiveness of Oil on a Bimetallic Couple	5322.2	ASTM D 6547
Fuel system icing inhibitor in hydrocarbon fuels (iodometric method)	5327	None
Fuel system icing inhibitor in hydrocarbon fuels (calorimetric method	5330	None
Fuel system icing inhibitor by freezing point depression method	5333.1	None
Fuel system icing inhibitor in hydrocarbon fuels (Refractometer method)	5340.2	ASTM D 5006
Fuel system icing inhibitor in hydrocarbon fuels(Redox method)	5341	None
Fuel system icing inhibitor in hydrocarbon fuels (hand refractometer method)	5342	ASTM D 5006
Silting index of hydrocarbon fuels	5350.1	None
Load carrying capacity of universal gear lubricants by the Timken machine	6505.2	ASTM D 2782
Gear fatigue characteristics of air craft gas turbine	6509.2	None
Load carrying capacity of lubricating oils at 204°	6511.2	None
Oscillating test of grease in Helicopter bearings	6516.2	None
Extreme pressure properties of lubricants (four ball tester)	6520.1	ASTM D 2783

Table IV. Numerical index of ASTM Equivalents for Federal standard test methods.

ASTM test method	FED-STD-791 Test Method
D892	3213.1
D1093	5101.7
D2603	3472
D2782	6505.2
D2783	6520.1
D4636	5307.2 and 5308.7
D5006	5342
D5862	355.3
D6184	321.3

Table V. Identifying symbols and addresses of activities responsible for preparation and maintenance of test methods.

Symbol	Service	Activity
NAPC	Navy	Department of the Navy Naval Air Systems Command Fuels and Lubricants Team Air-4.4.122229 Elmer Road, Bldg 2360 Patuxent River, MD 20670-1534
AFTL	Air Force	Department of the Air Force HQ Air Force Petroleum Agency Wright-Peterson AFB, OH 45433-7632
AR	Army	Department of the Army US Army Armament Research and Development Center Dover, New Jersey 07801-5001
SH	Navy	Department of the Navy Navel Sea Systems Command Washington D.C. 20362
AFWAL/POSF	Air Force	Department of the Air Force Air Force Wright Aeronautical Laboratories POSF
AFWAL/POSL	Air Force	USAF/ Aero Propulsion Laboratory AFWAL/POSL Wright- Patterson AFB, OH 45433-6563
AT	Army	US Army TARDEC/RDECOM Fuel and Lubricants Technology Team Warren, Michigan 48092

Method 103.6
September 30, 1986

COLOR OF GASOLINE

1. SCOPE

1.1 This method is used for determining the acceptability of color of dyed gasolines (aviation or motor).

2. SUMMARY

2.1 The method consists of comparing the hue and color intensity of the gasoline with those of permanent-color (glass) standards.

3. SAMPLE SIZE

3.1 Approximately 480 mL of gasoline to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Color comparator (Hellige Inc., Garden City, N.Y., Aqua-Testers No. 611A, or equal), split-field, for viewing simultaneously (by transmitted light) the sample and the combination of the reference liquid and the permanent-color standard.

4.2 Fluid tubes (2), glass, 20.0-cm viewing depth, with a fused-on plane bottom plate (Hellige Nessler tube No. 611-T, or equal).

4.3 Plungers, fluid-tube (2), polished optical glass (Hellige Nessler plunger No. 611-PL, or equal).

4.4 Gasoline color standards, permanent-color glass disc (as required):

a. Aviation gasoline standard (Hellige 611-95 and 611-96, or equal).
b. Motor gasoline standard (Hellige 611-97 or equal).

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Reference liquid; either a sample of the gasoline under test (prior to addition of dye or tetraethyl lead), or distilled [1,3,0].

6. PROCEDURE

6.1 Clean the optical surfaces of the color comparator.

6.2 Insert empty tubes in the color comparator, and adjust the position of the light source until the intensities of the light from both halves of the comparator split-field are equal.

6.3 Rinse and fill one of the tubes with reference liquid, and insert the plunger.

6.4 Wipe all excess reference liquid from the outside of the tube and from the top of the plunger, and insert the tube in the one side of the comparator.

6.5 Rinse and fill the remaining tube with the sample and insert the plunger.

6.6 Wipe all excess sample from the outside of the tube and from the top of the plunger, and insert the tube in the other side of the comparator.

6.7 Insert the color standard(s) into the comparator, and rotate the standard until the color selection can be viewed by transmitted light.

6.8 Turn on the light source, and visually compare the two fields for hue and intensity. (When testing aviation gasoline, compare its hue and intensity with the minimum and maximum color sections of the glass color standards.)

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the color as acceptable if the hue is approximately the same as the standard and if the intensity of the color is as follows:

a. Aviation gasoline intensity: between minimum and maximum standards.
b. Motor gasoline intensity: equal to or greater than standards.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - ME - 1986
September 30, 1986

Method 202.1
September 30, 1986

CLOUD INTENSITY AT LOW TEMPERATURE

1. SCOPE.

1.1 This method is used for determining the stability (at low temperature) of hydraulic fluids or highly refined light lubricating oils.

2. SUMMARY

2.1 The method consists of storing a sample at -54°C (-65°F) or lower for a period required by the petroleum product specification, agitating it, and comparing its turbidity with a standard. The sample is poured in a sealed, cleaned, dried sample bottle and stored for a specified number of hours at a maximum of -54°C (-65°F). Within 5 min of the comparison time, the turbidity standard is shaken vigorously for 10 s and poured into a sample bottle. At the end of the storage time, the sample is removed from cold storage and shaken vigorously for 10 s. The frost is eliminated from the sample bottle and it is compared with the turbidity standard. The turbidity of the sample relative to the standard is reported as less than, more than, or equal to the standard.

3. SAMPLE SIZE.

3.1 Approximately 120 mL of test oil (or fluid).

4. REFERENCES, STANDARDS, AND APPARATUS.

4.1 Cooling apparatus, -54°C (-65°F) or lower (ASTM cloud and pour point apparatus, or equal).

4.2 Sample bottles (2), clear-glass 120 mL, with cork stoppers.

5. MATERIALS.

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Methanol-glycerol (technical grade), 1:1 solution (by volume) [1,3,0].

5.3 Turbidity standard, prepared within 30 min of use, as follows:

a. Pour 25 mL of 0.00322 M barium chloride into a 250-mL volumetric flask [0,0,2].
b. Add 125 mL of 0.0050 N sulfuric acid, and shake well to insure complete precipitation [3,0,2].
c. Add approximately 25 mL of 1 N sodium hydroxide to make solution alkaline [3,0,1].
d. Add distilled water to bring total volume to 250 mL.
e. Pour solution into a sample bottle, and seal it tightly with stopper.

NOTE: For red hydraulic liquids, in place of plain distilled water, use distilled water containing 200 ppm red dye (National Erie Bordeaux B, manufactured by National Aniline Division of Allied Chemical and Dye Corp., or equivalent).

6. PROCEDURE.

6.1 Clean sample bottles, and dry at approximately 100°C for a minimum of 24 h.

6.2 Pour sample into bottle, and seal bottle tightly with stopper.

6.3 Store sample for the specified number of hours at a temperature no warmer than -54°C (-65°F).

6.4 Before end of storage period, cool the methanol-glycerol solution to -54°C (-65°F).

6.5 Within 5 min of making comparison, shake the turbidity standard vigorously for 10 s, and pour into the other sample bottle.

6.6 At the end of the storage period, remove sample from cold storage, and shake vigorously for approximately 10 s.

6.7 Dip sample into methanol-glycerol solution to eliminate frosting (if necessary), and within one minute of removal from cold storage, compare sample visually with the turbidity standard.

6.8 Note the turbidity of the sample.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING.

8.1 Report turbidity of sample relative to standard as less than, more than, or equal to the standard.

9. PRECISION.

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986

Method 203.1
September 30, 1986

POUR STABILITY OF LUBRICATING OILS

1. SCOPE

1.1 The test for pour stability is primarily intended for use with engine lubrication oils. However, by establishing proper final cooling temperatures, this method can be used for other lubricating oils such as power transmission fluids and hydraulic oils.

1.2 Good correlation with field test results has been obtained. However, since only two temperature/time cycles are reproduced, this method cannot duplicate all field ambient cycle conditions.

2. SUMMARY

2.1 After preliminary warming, the sample is subjected to a controlled temperature/time cycle over 5-1/2 to 7 days. Two cycles have been established to reproduce pour instability or reversion which has occurred during storage of oils in moderately cold cyclic ambient conditions. Oils exhibiting pour reversion are essentially "solid", resulting from wax gel formation, at temperatures significantly above their ASTM D 97 pour points.

3. SAMPLE SIZE

3.1 Approximately 100 mL of test lubricant.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Test Jar - Identical to ASTM D 97 and D 2500 pour point/cloud point test jar.

4.2 Thermometer - ASTM E-1 6C with temperature range of +20° to -80°C.

4.3 Cork - to fit test jar.

4.4 Heating bath - hot water to precondition test samples (80°C).

4.5 Cooling bath - low temperature with controller to follow temperature/time cycles from +15°C to -45°C. Spacing between test jars is to be about 1.5 cm with jars suspended so that cooling medium circulates around bottom and sides of jars.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Engine crankcase oil.

6. PROCEDURES

6.1 DEFINITIONS

6.1.1 Pour Stability Temperature - That specified temperature at which an oil remains fluid on completion of an established temperature/time cycle. See 6.2.8.

6.1.2 Stable Pour Point - The lowest temperature at which an oil remains fluid when subjected to an established temperature/time cycle. See 6.2.9.

6.2 Test procedures.

6.2.1 Adjust cooling bath temperature to +15°C with one temperature sensing bulb in the cooling medium.

6.2.2 Prepare two temperature measurement samples as follows:

6.2.2.1 Select a sample oil which is known to be fluid below the lowest temperature SAE Grade to be tested.

6.2.2.2 Fill each of two test jars with approximately 40 mL of selected oil sample. Identify these bottles as "Temperature Measurement Sample".

6.2.2.3 Prepare cork stopper to accommodate the standardized calibrated ASTM thermometer.

6.2.2.4 Insert stopper and thermometer into one jar so that thermometer immersion line is visible but not more than 0.3 cm above top of stopper. Place jar in center of cooling bath.

6.2.2.5 Prepare cork stopper to accommodate recorded temperature sensing bulb.

6.2.2.6 Insert stopper and one temperature sensing bulb in the second jar and position the bulb approximately 0.5 cm into the control oil sample. Place jar in center of cooling bath next to jar with thermometer.

6.2.2.7 Place the other temperature sensing bulb in cooling bath medium adjacent to the two control sample bottles.

6.2.3 Determine cloud point of test oil(s) by ASTM D 2500 procedure.

6.2.4 Prepare duplicate samples of test oil(s) using test jars filled with approximately 40 mL of test oil(s). Identify these with their SAE grade.

NOTE 1: Clean, dust-free jars and stoppers are required to eliminate possible contamination which may contribute to erroneous results.

6.2.5 Pretreat the duplicate test oil samples.

6.2.5.1 Place test sample jars in a hot water bath at +80°C for 2 h. Shake each test jar with sample several times.

6.2.5.2 Loosen stopper and allow sample to cool to room temperature. Then reinsert stopper.

6.2.5.3 Place test sample jars in cooling bath adjacent to control sample jars. All samples must be at same level if liquid bath is used.

6.2.6 Prepare bath for cyclic temperature test.

6.2.6.1 Temperature of bath should be at +15°C. Check thermometer and recorded temperature of temperature sensing bulbs.

6.2.6.2 If liquid bath is used, adjust level in bath to slightly above sample level in test jars.

6.2.6.3 Select temperature cycle A or cycle C (Figure 1 or Figure 2): use cycle A followed by cycle C for oil with cloud point of -5° to +45° C and cycle C for oils with cloud point below -5°C. Figure 2 includes temperature/time tables for use with programmable controllers.

6.2.6.4 Initiate the temperature cycle selected in 6.2.6.3.

6.2.7 During the final cool down, check proper temperature control each day as follows:

6.2.7.1 Read the "Temperature Measurement Sample" thermometer. Return this sample to the center of the bath.

6.2.7.2 Compare this temperature with the recorded temperature.

6.2.7.3 Determine whether a correction is required in the reading of recorded temperature. Estimate the correct time to make the first pour stability determination at the correct thermometer temperature ("1°C).

6.2.8 Pour stability determinations are made at the temperatures specified for the SAE grades of the test samples. The determination whether a test sample is "fluid" or "solid" at the specified temperature is conducted as follows:

6.2.8.1 Select the SAE grade test sample to be checked.

6.2.8.2 Carefully remove the test jar vertically from the bath and slowly tilt 90° to horizontal position. For 5 s observe the surface of the sample for movement.

<u>NOTE 2:</u> Handle jars at stopper end only to prevent heating the sample. If frosting occurs, wipe jars with cloth or glove; do not use unprotected finger.

6.2.8.3 If the surface moves, the sample is "fluid". If no movement is detected, the sample is "solid".

6.2.8.4 Select the duplicate test sample and repeat pour stability determination (see 6.2.8.2 and 6.2.8.3).

6.2.8.5 If additional SAE grades are included in the test bath, repeat all steps in 6.2.8 for the remaining duplicate test samples at specified temperatures.

6.2.9 Alternate method for determination of stable pour point. In this determination, the viscosity grade of the oil is not relevant. The stable pour point is determined during the final cool down in the temperature/time cycle as follows:

6.2.9.1 At the sample temperature of -5°C, carefully remove the test jar vertically from the bath and carefully tilt only enough to ascertain whether the oil surface moves and is "fluid". If movement is detected while tilting, return the bottle to vertical and carefully replace in bath. Total time for this operation shall be less than 3 s.

<u>NOTE 3</u>: Handle jar at stopper end only to prevent heating the sample. If frosting occurs, wipe jar with a cloth or glove; do not use unprotected finger.

6.2.9.2 If no movement of the oil is detected when the jar is tilted to 90° (horizontal) for 5 s the sample is "solid".

6.2.9.3 For oils which remain fluid, repeat step 6.2.9.1 at successively lower temperatures, in 3°C increments, until no movement of the oil is detected and the oil is "solid" by 6.2.9.2. Record the reading of the temperature measurement sample thermometer.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report pour stability as follows: If both (duplicate) samples show movement, report as "fluid at -XX°C". If one sample is fluid and the duplicate is solid or if both samples are solid, report as "solid at -XX°C".

8.2 Report stable pour point as 3°C higher than the temperature recorded in 6.2.9.3.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - ME - 1986

Method 313.3
September 30, 1986

PENETRATION OF LUBRICATING GREASES AFTER PROLONGED WORKING

1. SCOPE

1.1 This method is used for determining the consistency of lubricating greases that have been subjected to severe mechanical working.

2. SUMMARY

2.1 The method consists of working the grease mechanically under specified conditions for 100,000 double strokes (approximately 28 h), then checking the worked penetration in accordance with ASTM D 217.

3. SAMPLE SIZE

3.1 Sufficient grease to be tested to pack grease worker.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Grease worker, as specified in the "Cone Penetration of Lubricating Grease" ASTM D 217, with the following differences:

a. Machine grease-working, 60 double strokes per minute.
b Plunger plate, 270 holes 0.16-mm (1/16-in) diameter, arranged as in Figure 1.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Grease to be tested.

6. PROCEDURE

6.1 Pack sample in grease worker, and assemble and vent grease worker as described in "Procedure for Worked Penetration" of ASTM D 217.

6.2 Mount grease worker in machine and work grease for 100,000 double strokes while maintaining the ambient temperature at 25° " 1°C (77° " 2°F).

6.3 Remove the worker from the machine, making certain that plunger is pushed all the way down.

6.4 Open the vent cock and insert the thermometer as in "Procedure for Worked Penetration" of ASTM D 217.

6.5 Place grease worker in the water bath, bring temperature of sample to 25° " 1°C (77° " 2°F) and determine worked penetration in accordance with ASTM D 217.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report penetration after mechanical working as determined in paragraph 6.5.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Navy - NADC - 1986

Method 335.3
September 30, 1986

GEAR WEAR

1. SCOPE

1.1 This method is used for determining the relative lubricity of greases.

2. SUMMARY

2.1 This method consists of lubricating test gears (2 sets) of known wearing quality with the grease to be tested, running the gears under specified loads, and reporting the wear as the average loss in weight of the gears.

3. SAMPLE SIZE

3.1 Approximately 1 g of grease to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Gear wear tester (Figure 1), consisting essentially of:

a. Drive shaft, including 2.54-cm (1-in) O.D. drive pulley and provision for mounting a brass test gear (see Figure 2).
b. Driven shaft, including 2.54-cm (1-in) O.D. loading weight pulley and provision for mounting a steel gear (see Figure 2).

4.2 Drive mechanism, reciprocating (sinusoidal linear motion), 7.98 cm (3.14 in) amplitude, 60 CPM, with a cycle counter (6000 cycles minimum).

4.3 Weight, 2.27 kg (5 lb) (see Figure 1).

4.4 Weight, 4.54 kg (10 lb) (see Figure 1).

4.5 Brush, stiff-bristle (toothbrush or equal).

4.6 Oven, 71°C (160°F).

4.7 Balance, sensitivity 0.1 mg.

4.8 Container (for holding di-2-ethylhexylsebacate in contact with steel gear).

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Dry Cleaning Solvent (A-A-59601D).

5.3 Petroleum Ether (O-E-751). [1,4,0].

5.4 Di-2-ethylhexylsebacate, commercial grade. [0,0,0].

5.5 Test gear sets (2), composed of one brass and one steel gear (see Table 1).

TABLE I. Gear specifications.

	Brass gear	Steel gear
Pitch diameter	1.107 cm (0.4359 in)	1.211 cm (0.9769 in)
Helix angle	55 LH	35 LH
Number teeth	16	25
Root diameter	0.000 cm (0.000 in)	0.000 cm (0.000 in)
Composition	ASTM B 121/B 121/M, ASTM B 36/B 36M	SAE AMS-S-7720
Brinell hardness	100-140	210-250
Tooth form	Involute 14-1/2 pressure angle	Involute 14-1/2 pressure angle
Normal pitch	64	64
Shaft angle	90°	90°

6. PROCEDURE

6.1 Mount the brass gear on the drive shaft and the steel gear on the driven shaft. Scribe alignment marks on the gears to insure that the gears are placed in the same position relative to each other on each assembly at the test setup.

6.1.1 Preparation and cleaning of gears.

6.1.1.1 Clean, dry, and weigh test gears as follows:

a. Scrub test gears, using stiff-bristle brush and dry cleaning solvent (see Note 1).
b. Rinse gears in petroleum ether, and dry in oven at 71°C (160°F) (see Note 1).

NOTE 1: CAUTION. Dry-cleaning solvent and petroleum ether are both toxic and flammable. Use only in a well ventilated area. Do not breathe their fumes or allow them to come in contact with the skin. Keep all flames from the solvent, especially the petroleum ether.

c. Remove gears from oven, cool, and record the weight (to the nearest 0.1 mg) at the brass gear.

6.2 Assemble the test setup as follows:

a. Mount brass gear on drive shaft and steel gear on driven shaft of the gear wear tester (see Figure 1). Insure proper positioning of the test gears by lining up the scribe marks (see 6.1).
b. Using flexible cord wound around the pulleys, couple the drive shaft to the reciprocating drive mechanisms and apply a 2.27-kg (5-lb) weight to the driven shaft.

6.3 Perform gear break-in run as follows:

a. Place container (see 4.8) under the gears, and fill it with di-2 ethylhexylsebacate until the lower teeth of the steel gear are immersed.
b. Start the reciprocating drive mechanism, and allow it to operate for 1500 complete cycles.
c. After the test period, remove, clean, and dry the gears, and reweigh the brass gear (see paragraph 6.1.1.1).
d. Retain the gear set for use in testing if loss in weight of brass gear does not exceed 2.0 mg; discard (as defective) both gears of the set if loss exceeds 2.0 mg.

NOTE 2: A drop feed lubricator has been found effective to insure removal of wear debris during break-in run for the tests.

6.4 Assemble the test setup (paragraph 6.2) and throughly coat the gear teeth with the grease to be tested. Then start the reciprocating drive mechanism, and allow it to operate for 6000 cycles.

6.5 At the end of the test run, remove the gears, clean and dry them, and reweigh the brass gear as described in paragraph 6.1.1.1.

6.6 Repeat the procedures described in paragraph 6.2, substituting a 4.54 kg (10-lb) weight and 3000 cycle operation.

6.7 At the end of the 3000-cycle run, remove and clean gears, and reweigh the brass gear as described in paragraph 6.1.1.1.

6.8 Conduct two complete tests, using new gear sets each time, and calculate the average weight loss (per 1000 cycles) of the brass gears for the 6000- and 3000-cycle runs.

7. CALCULATIONS

7.1 Calculation for average weight " loss, to the nearest mg.

$$WL = A-B$$

where:
WL = weight loss in brass area.
A = original weight of brass gear, in mg.
B = final weight of brass gear in mg.

8. REPORTING

8.1 Report the average weight loss (per 1000 cycles) of the brass gears for the 6000- and 3000-cycle runs.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Navy - NADC - 1986

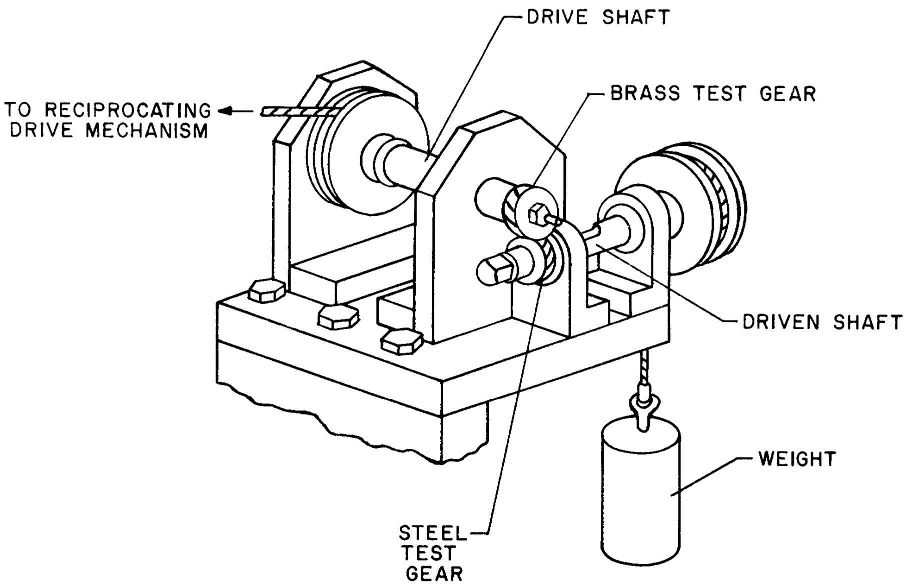
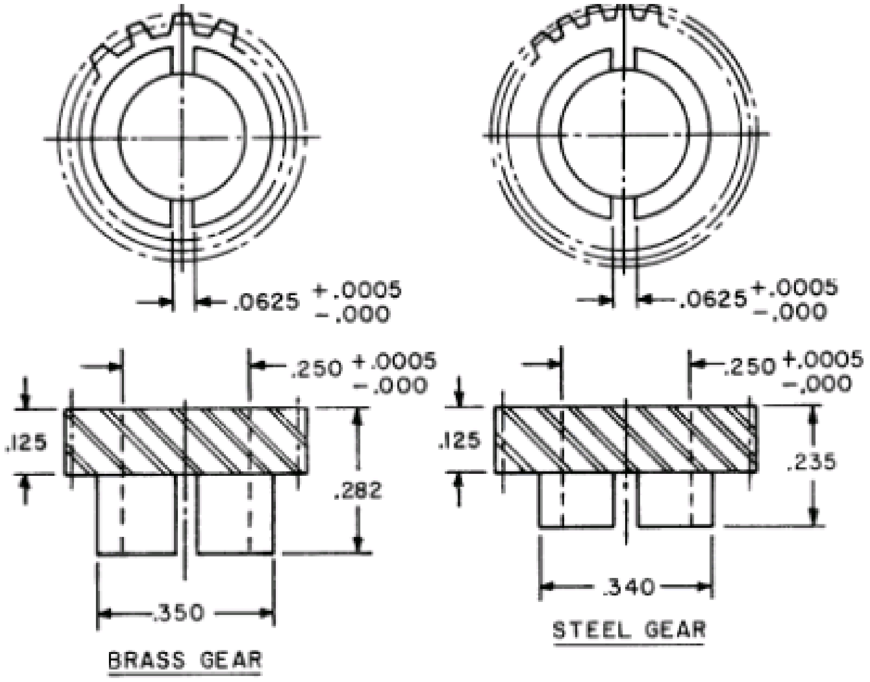


FIGURE 1. Gear wear tester.



SPECIFICATIONS		
	BRASS GEAR	STEEL GEAR
MATERIAL	ASTM B 121/B 121M, ASTM B 36, B 36M	SAE-AMS-S-7720
BRINELL HARDNESS	100-140	210-250
OUTSIDE DIAMETER	.467+.000,-.002	.508+.000,-.002
PITCH DIAMETER	.4359	.4769
TOOTH FORM	INVOLUTE 14-1/2° PRESSURE ANGLE	
HELIX PITCH	55° L.H.	35° L.H.
NORMAL PITCH	64	
ROOT DIAMETER	.400 MAX.	.441 MAX.
SHAFT ANGLE	90°	
NO. OF TEETH	16	25
TOOTH THICKNESS TOLERANCE	-.001 -.003	

NOTES:
1. DIMENSIONS ARE IN INCHES.
2. TOLERANCES: UNLESS OTHERWISE SPECIFIED, DECIMALS 3 PLACES ±.001.

FIGURE 2. Test gears for gear wear test apparatus.

Method 350.2
September 30, 1986

EVAPORATION LOSS OF LUBRICATING GREASES AND OILS (HIGH TEMPERATURE)

1. SCOPE

1.1 This method describes the test procedure and apparatus for the determination of evaporation loss of lubricating greases and oils for applications where evaporation loss is a factor. Evaporation loss data can be obtained at any temperature in the range 99° to 538°C (210° to 1,000°F).

2. SUMMARY

2.1 The method consists of heating the oils or greases in containers vented with 2 L/min of air in order to determine the percentage evaporation loss of the lubricant.

3. SAMPLE SIZE

3.1 Approximately 20 g of the grease or oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Evaporation cell and attachments conforming with the dimensional tolerances indicated in Figures 6 and 7. Other structural details are given below.

4.1.1 The body and cover of the cell shall be constructed of stainless steel and air-heating coil also of stainless steel.

4.1.2 The sample cups (recommended maximum weight 200 g each), hood, eduction tube, and orifice shall be constructed of stainless steel. A suitable material is an alloy steel conforming to grade S, type 304, of ASTM A204. To facilitate removal and separation of the cup and hood for inserting the sample and weighing, The sample cup shall be threaded to the hood and this in turn to the eduction tube of the cover. The cover of the cell shall be made air-tight.

4.2. Air supply system, capable of supplying to the cell the required flow of air free of mechanically entrained particles. A 61.0-cm (24-in) length of 12.7-cm (5-in) diameter pipe, reduced to 2.54-cm (1-in) diameter at each end, packed with glass wool at each end, and filled with a suitable drying agent has been found satisfactory for filtering and drying. Means must be provided for filling the drying and filtering tube.

4.3 Heating blocks of cast aluminum with evaporation cells and air coils cast within. The heating block shall be heavily insulated and equipped with sufficient electric heating elements to maintain the test temperature. There shall be one thermocouple provided for each heating element. Figures 1, 2, and 3 show a satisfactory heating block.

4.4 Temperature controller. A suitable temperature controller capable of maintaining temperatures between 99° to 538°C " 0.5°C. A suitable temperature recorder shall also be provided.

4.5 Flowmeter. A rotameter calibrated to deliver air at a rate of 2.58 " 0.02 g/min between 15.6° to 29.4°C (60° and 85°F) (2 L/min at standard temperature and pressure). It shall be furnished with a needle valve and may be mounted as shown in Figure 7.

4.6 Evaporation tester stand. A suitable stand for supporting the test apparatus is shown in Figure 5.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 1,1,1-Trichloroethane (ASTM D 4126), [2,1,0].

5.3 Tissue, Facial.

6. PROCEDURE

6.1 Greases

6.1.1 Clean the grease-sample cup and hood thoroughly, using warm 1,1,1-trichloroethane (38°-54°C [100°-130°F]) and facial tissues. Dry thoroughly in warm air.

NOTE 1: Caution. 1,1,1-trichloroethane is toxic. Use it only in a well ventilated area. Avoid contact with skin.

6.1.2 Weigh the clean grease-sample cup and hood to the nearest mg. Remove the hood and fill the cup with sample, taking care to avoid occlusion of air. Smooth the surface level with the rim of the cup with a straight edged spatula. Remove with a clean cloth any grease which may remain on the rim or threads of the cup. Thread the hood tightly onto the cup without disturbing the smoothed grease surface. Weigh the assembly and record the net weight of the sample to the nearest mg.

6.1.3 With cover in place, but without the hood and sample cup attached, allow the evaporation cell to acquire the temperature of the block (controlled to 2°C ["1°F]), at which the test is to be made. Allow the block to maintain the temperature of the test at least 2 h before beginning the test. During this period, allow clean air to flow through the cell at the prescribed rate, 2.58 " 0.02 g/min (2 L/min at standard temperature and pressure), as indicated by the rotometer. Then remove the cover, thread the weighed hood and sample cup into place, and replace the cover. Tighten the three knurled cover- tightening screws securely to prevent air leakage under the cover. Pass clean air through cell for 22 h " 5 min.

6.1.4 At the end of the 22-hour period, remove the assembled sample cup and hood from the cell, and allow to cool to room temperature. Determine the net weight of the sample to the nearest mg.

6.2 Oils.

6.2.1 Clean the oil-sample cup and hood as described in 6.1.1.

6.2.2 Weigh the clean oil-sample cup and hood to the nearest mg. Transfer, by means of a pipet, 10.00 " 0.05 g of sample to the cup. Assemble the cup and hood, being careful not to splash oil on the underside of the hood. Weigh the assembly and record the net sample weight to the nearest mg.

6.2.3 Evaporate the sample as described in 6.1.3 and 6.1.4.

7. CALCULATION

7.1 Calculate the evaporation loss of the sample as follows:

$$\text{Evaporation loss, percent by weight} = \frac{S - W}{S} * 100$$

where:
S = initial weight of sample, in grams.
W = weight of sample, in grams, after the test.

8. REPORTING

8.1 Report the evaporation loss percentage.

9. PRECISION

9.1 Results should not differ from the mean by more than the following amounts:

Repeatability: 2.5% of mean
Reproducibility: Not established

Method prepared by:

Navy - NADC – 1986

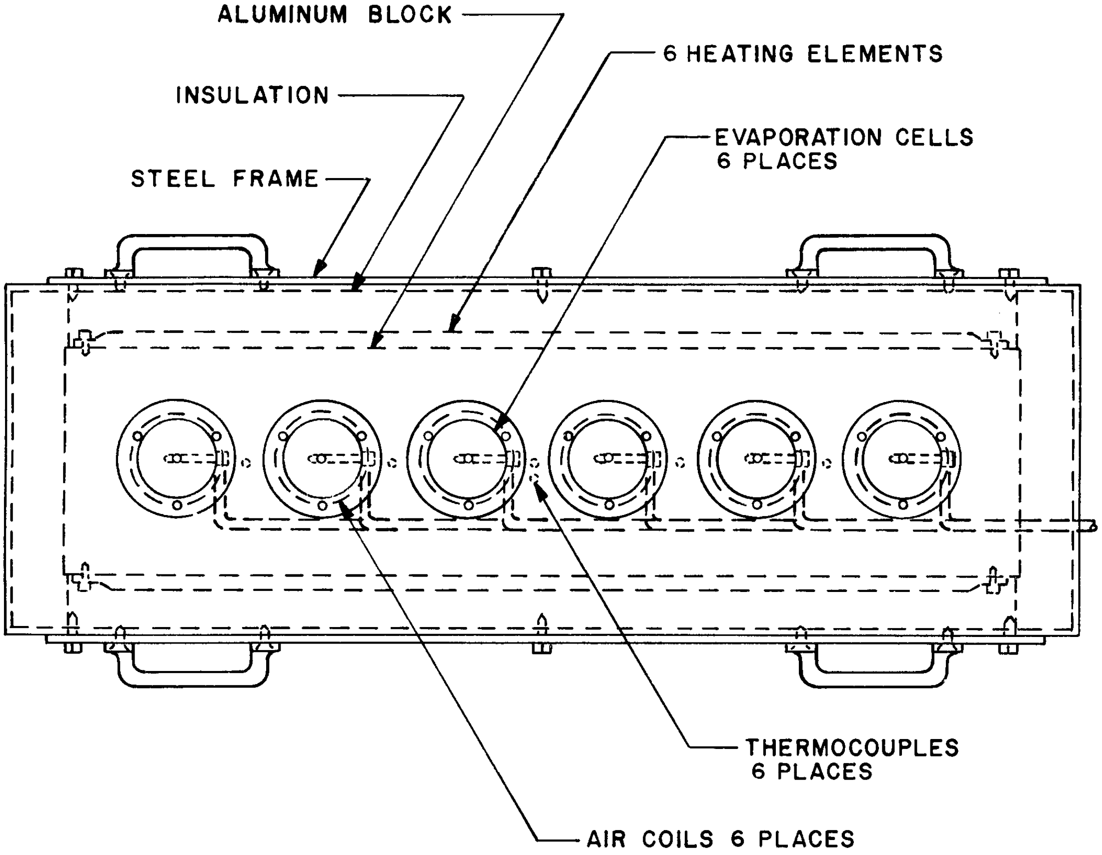


FIGURE I. Top view high temperature evaporation tester.

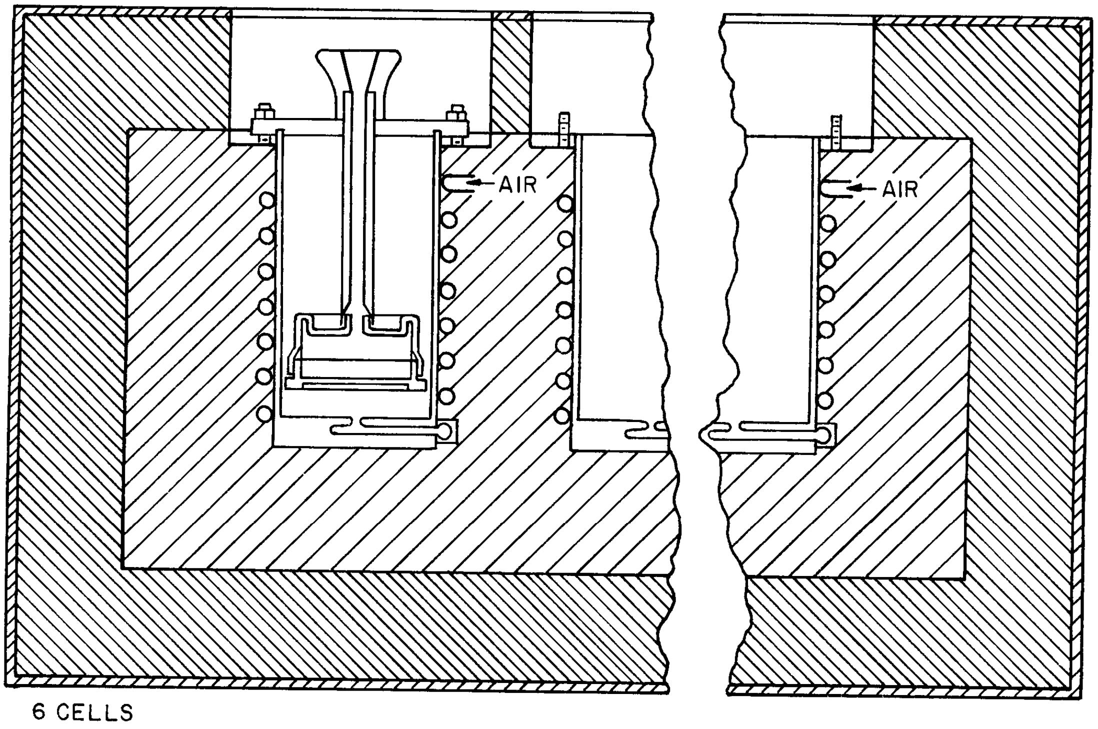


FIGURE 2. Side view high temperature evaporation tester.

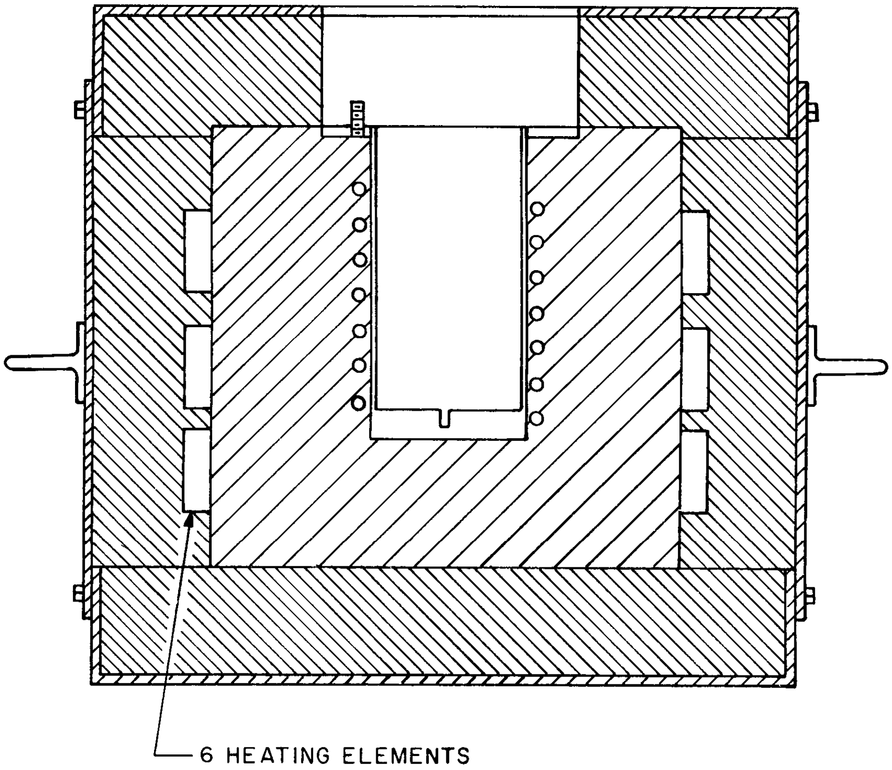


FIGURE 3. Cut end view high temperature evaporation tester.

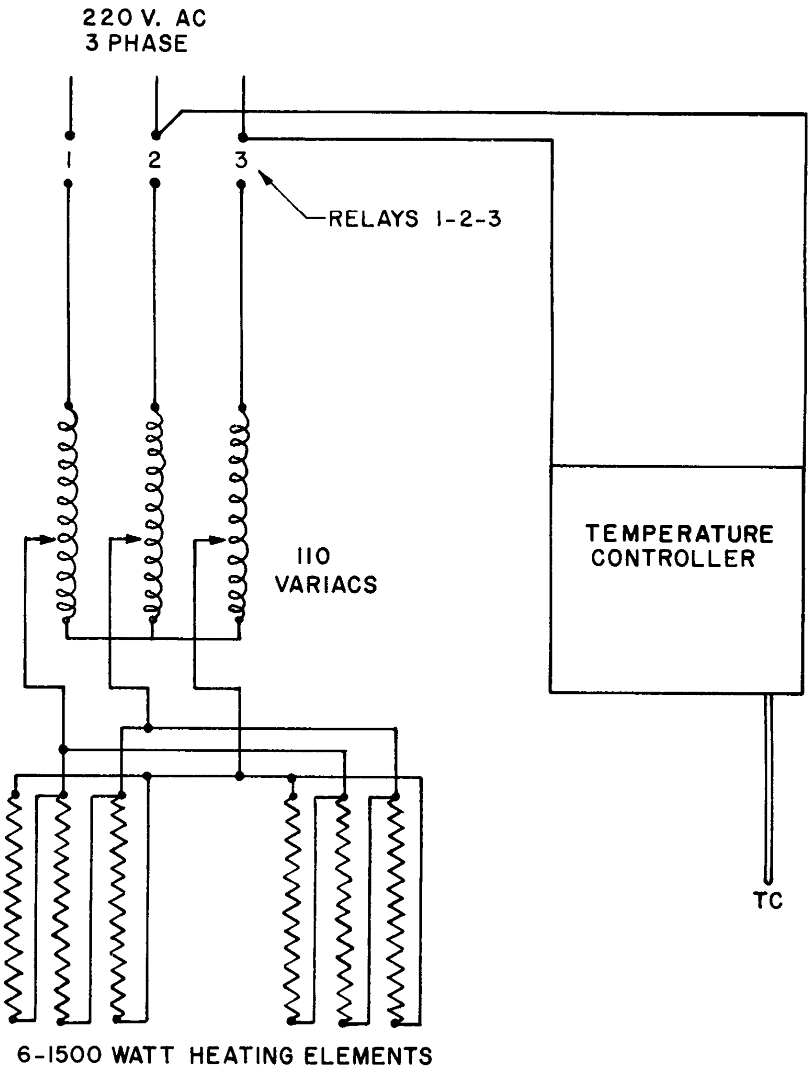
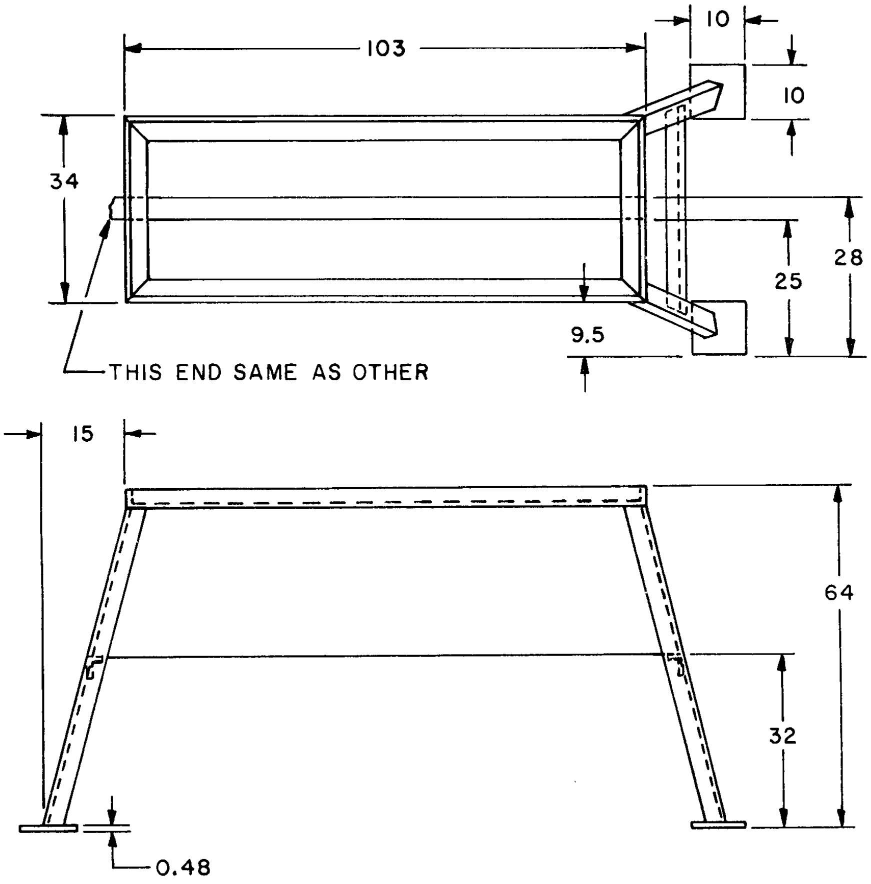
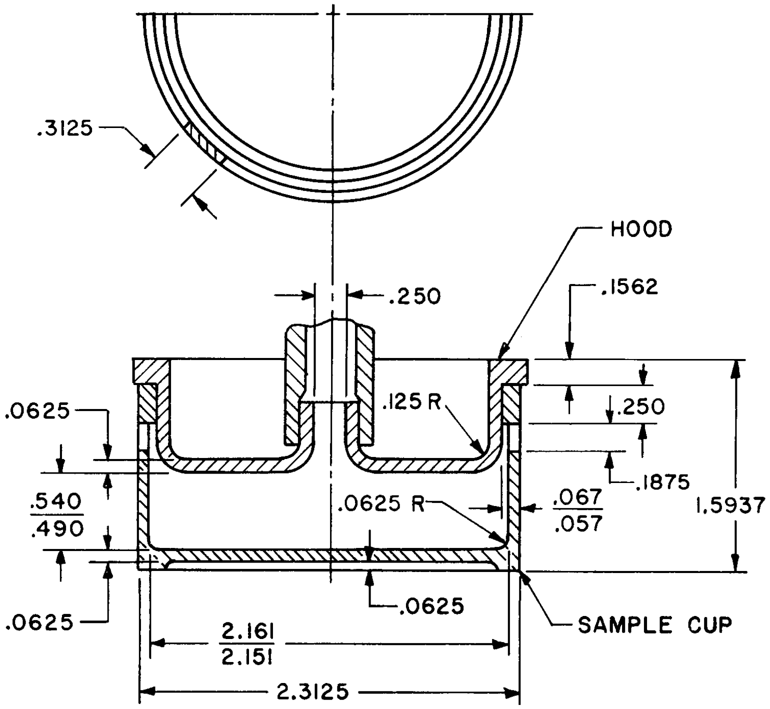


FIGURE 4. High temperature evaporation tester.



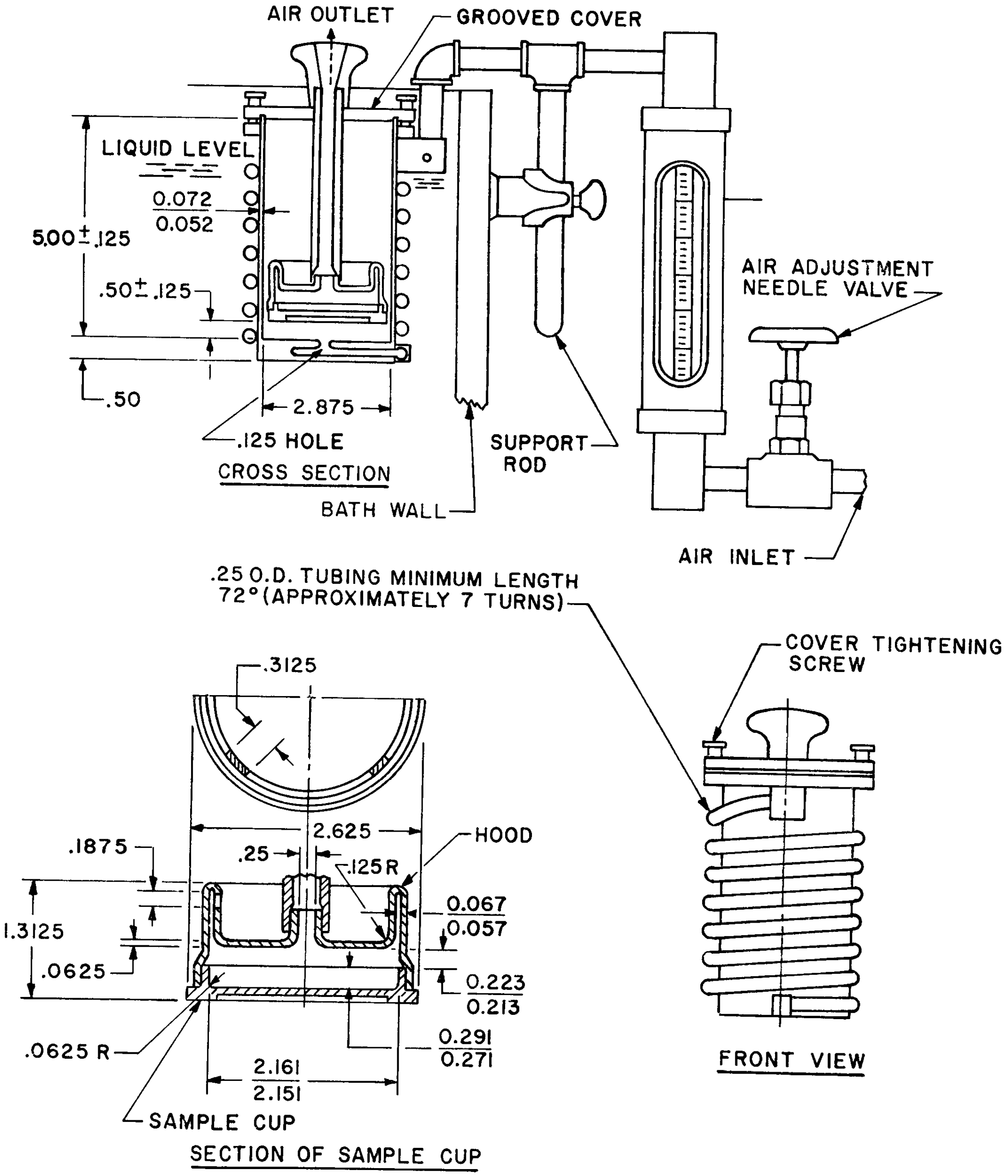
NOTE:
DIMENSIONS IN CENTIMETERS.

FIGURE 5. Evaporation tester stand.



NOTE:
1. DIMENSIONS IN INCHES.
2. ALL DIMENSIONS ± .0156 UNLESS OTHERWISE SPECIFIED.

FIGURE 6. Oil sample cup.



NOTES:
1. DIMENSIONS IN INCHES.
2. ALL DIMENSIONS ± .0156 UNLESS OTHERWISE SPECIFIED.

FIGURE 7. Evaporation test cell.

Method 352.1
September 30, 1986

EFFECT OF EVAPORATION ON FLAMMABILITY

1. SCOPE

1.1 This method is used for determining the effect of evaporation on the flammability of a liquid petroleum product.

2. SUMMARY

2.1 It consists of checking the flammability of the liquid (by passing a sample-soaked pipe cleaner repeatedly through a flame and noting the number of passes required for ignition), storing the sample in an oven for the time and at the temperature required by the specification, and rechecking flammability.

3. SAMPLE SIZE

3.1 Approximately 30 mL of liquid to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Oscillating device (such as a windshield wiper), approximately 120° arc at 25 . 2 cpm.

4.2 Petri dish, approximately 9.0-cm diameter by 1.6-cm deep.

4.3 Oven, gravity-convection, capable of maintaining the specified temperature within 1°C (2°F).

4.4 Bunsen burner.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Pipe Cleaners (U.S. Tobacco Co., Dill's or equal).

6. PROCEDURE

6.1 Cut at least ten 5.1-cm (2-in) lengths of pipe cleaner.

6.2 Assemble a test setup (Figure 1) as follows:

a. Mount a 5.1-cm (2-in) piece of pipe cleaner in the arm of the oscillating device so that the end of the pipe cleaner describes an arc of approximately 120° at a radius of 10.2 . 0.31 cm (4 . 1/8 in).
b. Adjust the Bunsen burner to provide a completely blue flame approximately 10 cm (4 in) high, and without a sharp inner cone.
c. Position the Bunsen burner so that the center of the 5.1-cm (2-in) piece of pipe cleaner will pass through the center of the flame at the reversing point of the cycle.

6.3 Pour approximately 30 mL of the sample into the petri dish, and soak five 5.1-cm (2-in) pieces of pipe cleaner in the sample for two min.

6.4 Drain off excess sample from one of the pieces of pipe cleaner, and mount cleaner in test apparatus as described in paragraph 6.2. Start the oscillating device, and record the number of cycles necessary for a self-sustained flame to appear on the pipe cleaner. Repeat this procedure with four other pieces of pipe cleaner.

6.5 Store the uncovered petri dish containing the sample in the oven, for the specified time.

6.6 At the end of this time, remove the dish from the oven. Place five 5.1-cm (2-in) pieces of pipe cleaner in the dish.

6.7 Recheck flammability as specified in 6.4.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

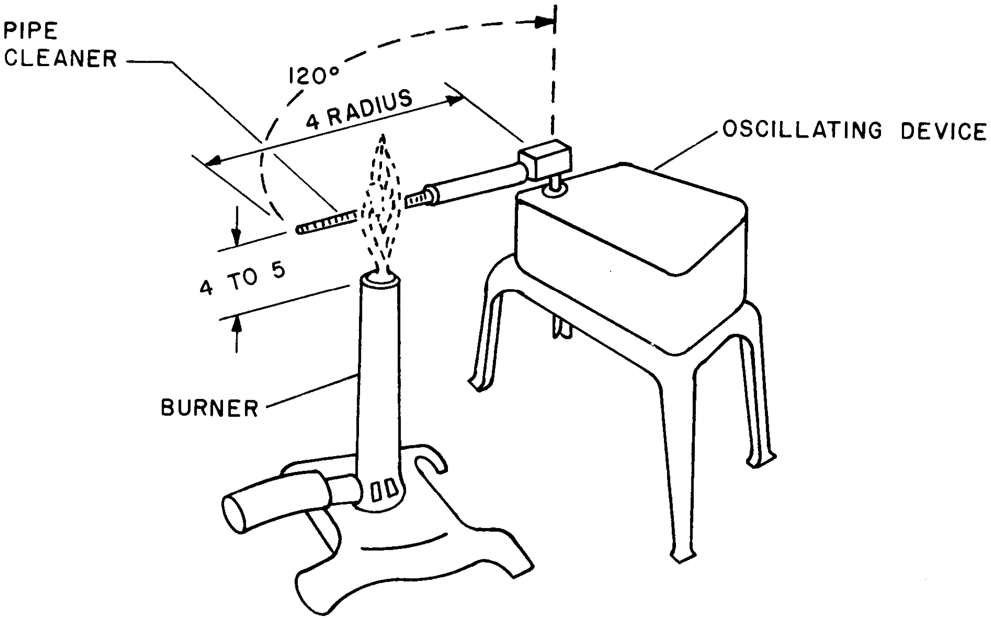
8. REPORTING

8.1 Report the average number of cycles necessary for a self-sustaining flame to be achieved on the pipe cleaner both before and after partial evaporation.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:
Navy - NADC – 1986



NOTE:
DIMENSIONS IN CENTIMETERS.

FIGURE 1. Test setup.

Method 353.1
September 30, 1986

EVAPORATION

1. SCOPE

1.1 This method is used for determining the tendency of oil to form a hard or tacky residue upon evaporation.

2. SUMMARY

2.1 It consists of heating an oil-coated glass slide in an oven, at the temperature and for the time required by the specification, and reporting the condition of the residue.

3. SAMPLE SIZE

3.1 As needed, usually less than 100 mL.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Slide, microscope, glass.

4.2 Oven, gravity-convection, specified temperature within 0.5°C ("1° F).

4.3 Thread or wire, heat-resistant (for suspending slide in oven).

4.4 Beaker, 250 mL (for immersing the slide in the sample).

5. MATERIALS

5.1 This section is not applicable to the test procedure.

6. PROCEDURE

6.1 Heat oven to specified temperature "0.5°C ("1°F).

6.2 Cut notches near one end of slide so that it can conveniently be suspended by a thread or wire.

6.3 Immerse slide in oil at room temperature.

6.4 Remove slide from oil, and suspend it in oven for the specified time.

6.5 After the specified heating period, remove slide from oven, and cool it to room temperature. 6.5 After the specified heating period, remove slide from oven, and cool it to room temperature.

6.6 Observe the condition of oil residue on slide, paying particular attention to note the presence of any hard or tacky film.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the condition of the oil residue on the slides, paying particular attention to the presence of any hard or tacky film.

9. PRECISION

9.1 Precision data has not been developed for this method.

Method prepared by:

Navy - NADC - 1985

Method 361.4
September 30, 1986

STROKING PROPERTIES OF HYDRAULIC BRAKE FLUIDS

1. SCOPE

1.1 This method describes a procedure for evaluating the stroking properties of brake fluids used in the hydraulic brake systems of automotive vehicles. This method, with slight modifications, may be used to evaluate hydraulic brake rubber cups against a standard brake fluid.

2. SUMMARY

2.1 The method utilizes standard Society of Automotive Engineers (SAE) hydraulic brake parts mounted in a manner similar to that of an automobile brake system. The test brake fluid is subjected to various stroking rates, pressures, and temperatures designed to evaluate the lubrication properties of the brake fluid. This method complements the chemical and physical tests required by the brake fluid specifications.

2.2 It is essential to have meaningful reference data before a significant interpretation of test results can be made. Reference data must include comparable test information on a brake fluid of known service performance characteristics.

3. SAMPLE SIZE

3.1 Approximately 3.8 L (1 gal) of the brake fluid to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 SAE Standard.

J 1703 - Motor Vehicle Brake Fluid

4.2 ASTM Standard.

D 2240 - Rubber Property-Durometer Hardness

4.3 Apparatus.

4.3.1 Wheel assemblies.[1] Four left front wheel brake assemblies.

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[1] An alternative to the wheel assemblies is described in section 4.12 of SAE J 1703.

4.3.1.1 Bearing assemblies. Four front outer bearing assemblies, and four front inner bearing assemblies, with bearing races and grease seals.

4.3.1.2 Hub and brake drum assemblies. Four left front wheel hub and brake drum assemblies.

4.3.1.3 Helper springs. One helper spring per wheel assembly.

4.3.2 Brake pressure actuating mechanism. A suitable actuating mechanism for applying pressure to the master cylinder push rod to simulate, as closely as possible, the braking operation in a vehicle.

4.3.2.1 Type of actuating mechanism. The actuating mechanism shall be operated by suitable air pressure or hydraulic means. Mechanical actuation is not recommended.

4.3.2.2 Pressure application. The amount of pressure applied by the actuating mechanism shall be adjustable. The mechanism shall be capable of applying sufficient thrust to the master cylinder to create a minimum pressure of 6.9 gage MPa (1000 psig) in the simulated brake system.

4.3.3 Stroking rate and rate-of-load application. The actuating mechanism shall be designed to permit adjustable stroking rates ranging from 500 to 1,000 strokes per hour as desired. The ratio of percent time-of-load application to non-load application shall also be adjustable, from a minimum ratio 25/75 percent to a maximum of 60/40 percent per stroke cycle. (A "60/40 percent" ratio per stroke cycle indicates that pressure is applied during 60% of the duration of the stroke cycle, while the pressure is released, or not applied, during the remaining 40% of the cycle.)

4.3.4 Pressure gages. Two recording pressure gages of 0 to 6.9 gage MPa (0 to 1000 psig) capacity with connecting tubing. One of the gages shall have a 24-hour cycle, while the other gage shall have a maximum cycle of 6 min. Each gage shall be provided with a shut off valve for removing air from the connecting tubing.

4.3.5 Revolution counter. A mechanical or electrical revolution counter with recording capacity for 500,000 strokes.

4.3.6 Tubing. Approximately 4.6 m (15 ft) of 0.63 cm (1/4 in) O.D. copper or steel tubing with inverted flare nuts, connectors, adaptors, and fittings.

4.3.7 Stand. A stand or frame for mounting the four left front wheel assemblies as shown in Figure 1.

4.3.8 Cabinet or oven. A large cabinet or oven, of sufficient capacity to house the stand with

the four mounted wheel assemblies, together with the master cylinder and necessary connections.

4.3.9 Temperature control. The cabinet or oven shall be insulated, and shall be equipped with suitable means of heating and cooling to provide the specified test temperatures. The temperatures shall be thermostatically controlled within "2.8°C ("5°F) of the specified test temperature.

4.3.10 Durometer, Type A-shore durometer. The description of the durometer, calibration, and application in testing rubber hardness may be found in ASTM D 2240.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Ethyl alcohol, (ACS) [0, 3, 0].

5.3 Crocus cloth. Crocus cloth shall conform to the requirements of ANSI B74.18.

5.4 Wheel cylinder assemblies. Four cast iron housing straight bore hydraulic brake wheel cylinders having a diameter of approximately 2.8 cm (1-1/8 in). SAE part number RM-14a.

5.5 Master cylinder assembly. One cast iron housing hydraulic brake master cylinder having a diameter of approximately 2.8 cm (1-1/8 in.) and fitted with an uncoated steel standpipe. SAE part number RM-15a.

5.6 Rubber cups. All wheel cylinder and master cylinder cups used in the test brake system shall be made of SBR type elastomer. SAE part numbers for wheel cylinders and master cylinders are RM-3, RM-4 and RM-5.

5.7 Wheel cylinder pistons. Unanodized aluminum pistons (SAE RM-12) made from SAE AA 2024 aluminum alloy are to be used.

5.8 Master cylinder piston. Use a SAE RM-13 piston made from SAE CA 360 copper-base alloy (half hard).

NOTE: All standard SAE parts are available from the Society of Automotive Engineers, Inc, 400 Commonwealth Drive, Warrendale, PA 15096.

6. PROCEDURE.

6.1 Preparation of the test apparatus.

6.1.1 Inspection and cleaning of parts.

6.1.1.1 Wheel cylinder assemblies. The wheel cylinder assemblies shall be completely disassembled. All rubber parts, with the exception of the cylinder boots, shall be discarded. The expanders in the assembly shall also be discarded. All metal parts shall be cleaned with 95% ethyl alcohol to remove preservative oils, grease, and dirt, then dried in a stream of dry air, and examined for cuts, scores, galling, or pitting. Any part having any of these flaws shall be replaced. Light stains on the cylinder walls shall be removed by rubbing with crocus cloth and 95% ethyl alcohol. Any cylinder which cannot be freed of stains by this procedure shall be discarded.

6.1.1.2 Master cylinder assemblies. The master cylinder assemblies shall be completely disassembled. All rubber parts, including the piston washer if it forms an integral part of the primary cup, shall be discarded. The metal parts shall be cleaned, air-dried, examined for flaws, and light stains removed from the cylinder walls in the manner specified for the wheel cylinder assemblies (see 6.1.1.1). Any deficient parts shall be replaced. The cylinder's relief and supply parts shall be inspected for burrs and wire-edge. If present, they may be removed with a burring tool or a jeweler's file.

6.1.1.3 Rubber parts. The rubber parts to be used in the test shall be cleaned with a lint-free cloth and 95% ethyl alcohol to remove all dirt and grease, then dried in a stream of dry air and examined for cuts, molding flaws, or blisters. Any deficient part shall be discarded.

6.1.1.4 Tubing. The tubing used as brake fluid lines shall be flushed with 95% ethyl alcohol, and dried by blowing dry air through it, prior to connecting to the test apparatus.

6.2 Initial measurements and tolerance.

6.2.1 Cylinders. The internal diameter of the master cylinder and of each of the wheel cylinders shall be measured with a micrometer, to the nearest 0.0025 cm (0.001 in). Four readings, approximately 45° apart, shall be taken on each cylinder, and their average considered as the diameter of the cylinder. If the largest and smallest diametrical readings differ from each other by more than 0.0075 cm (0.003 in), the cylinder in question shall be discarded.

6.2.2 Pistons. The external diameter of the master cylinder piston and of each of the wheel cylinder pistons shall be measured with a micrometer, to the nearest 0.0025 cm (0.001 in). Two readings, at right angles to each other, shall be taken on each piston, and their average considered as the diameter of the piston. If the two readings differ from each other by more than 0.0025 cm (0.001 in) the piston in question shall be discarded.

6.2.2.1 Clearance of piston in cylinder. Each piston shall be checked for clearance within the cylinder for which it is intended. If the clearance is less than 0.0075 cm (0.003 in) or more than 0.0125 cm (0.005 in), the cylinder or the piston shall be replaced by another similar part.

6.2.3 Rubber cups.

6.2.3.1 Base and lip diameters of the cups. The internal base and lip diameters of each rubber cup shall be determined with a micrometer, to the nearest 0.0025 cm (0.001 in). The cups shall be placed on a flat surface for 24 h before measuring. In measuring the diameters of the cups, care should be taken that the micrometer does not extend more than 0.08 cm (1/32 in) beyond the edges of the cup. If difficulty is encountered in measuring the diameter of the master cylinder secondary cup, the cup may be placed on a mandrel having a slight taper. Two readings at right angles to each other, shall be taken on the base and lip of each cup, and their average considered as the base and lip diameters of the cup. If the two readings on the base or the two readings on the lip differ by more than 0.0025 cm (0.001 in) respectively, the cup in question shall be discarded.

6.2.3.2 Hardness of rubber. The durometer hardness of each cup shall be measured at four different points near the center of each cup. The average of the four readings shall be considered as the hardness of the rubber cup. For the sake of uniformity the durometer readings should be made as follows: The proper rubber anvil is inserted in the fixture, and the rubber cup placed on the anvil. The back edge of the durometer is placed on the shelf of the fixture, with the bottom surface of the durometer kept parallel to the surface of the base of the cup. The durometer is grasped between the thumb and the third and fourth fingers of one hand, with the index finger placed on the top of the depressor. The indentor point is lowered gently until it rests on the surface of the cup. With a minimum amount of rocking, pressure is then applied so that the pressure plate makes firm contact with the surface of the cup. The durometer reading is taken as soon as the dial needle comes to rest. If the needle does not come to rest, then the reading is taken at that moment when a definite change in the speed of the needle is observed, with the reading estimated to the nearest whole point of the dial scale.

6.2.3.2.1 Wheel cylinder cups. The average durometer hardness of the wheel cylinder cups shall be 60 " 3 points. Nonconforming cups shall be discarded.

6.2.3.2.2 Master cylinder cups. The average durometer hardness of the master cylinder cups shall be 63 " 3 points. Nonconforming cups shall be discarded.

6.3 Assembling, adjusting, and checking the apparatus.

6.3.1 Assembly of brake parts. After the required measurements have been taken and recorded, the wheel cylinders and the master cylinder shall be assembled for test. All parts with the exception of the cylinder housings and the rubber boots, shall be dipped in the hydraulic fluid under test when they are assembled. The assembled cylinders shall be checked for ease of operation, and then installed in their respective places in the simulated brake systems. Helper springs shall be installed in holes beneath the shoe flanges, pushed up and hooked over the flanges (see Figure 1).

6.3.2 Shoe clearance adjustment. The brake shall be adjusted to have a clearance of 0.102 " 0.0075 cm (0.040 " 0.003 in) between the shoe and the drum, at the toe.

6.3.3 Filling and bleeding the brake system. The assembled simulated brake system shall be filled with the fluid under test by means of a pressure pot, or by hand. If filled by hand the master cylinder shall be stroked several times to completely fill the system. All the wheel cylinders and the tubing connected to the pressure gages shall be bled to remove the entrapped air from the system.

6.3.4 Checking the system for leaks. After the system has been bled, a pressure of 6.9 gage MPa (1,000 psig) shall be applied to the system. The system shall be slowly stroked for 10 min, after which an inspection shall be made for leaks. The pressure shall be released, and the brake shoe clearance rechecked (6.3.2).

6.3.5 Fluid level. A constant level of test fluid shall be maintained at the master cylinder throughout the test. Accurate record shall be kept of the number of mL of the fluid added to the system during the test. It may be necessary to install special antisplash plugs on the master cylinder reservoir to avoid loss of the fluid due to splashing.

6.4 Test procedure.

6.4.1 Procedure A. Stroking properties at high temperatures.

6.4.1.1 Conditions of test.

6.4.1.1.1 Test temperature. Unless otherwise specified the test temperature shall be 120° " 3°C (248° " 5°F). The test shall be started at room temperature (23° " 5°C). The rise in temperature from room temperature to the test temperature shall take place in not more than 8 h from the start of the test.

6.4.1.1.2 Pressure. The initial stroking pressure in the simulated brake system shall be adjusted at room temperature (23° " 5°C), to 3.4 gage MPa (500 psig) and recorded. The pressure stroke pattern shall be adjusted at 60/40 percent stroke ratio 17 strokes/min, with the aid of the short-cycle pressure recorder, so as to produce a relatively smooth curve (see Figure 2). Accurate record shall be kept of any pressure variations during the test. The pressure stroke pattern shall be compared with the initial pattern at intervals of 50,000 strokes during the test.

6.4.1.2 Brake-in or burnishing run. The stroking rate shall be adjusted at 1,000 " 50 strokes per hour, using the 60/40 percent stroke ratio and the stroking pressure of 3.4 MPa gage (500 psig) applied at room temperature (23° " 5°C). While at room temperature, the system shall be stroked 16,000 " 500 strokes. At the end of this stroking period the system shall be checked for fluid level. The cylinder shall be filled to the given level, if necessary, and the test resumed.

6.4.1.3 Duration of test. The test shall be run until 94,000 strokes have been completed. The number of strokes given in the burnishing run (see 6.4.1.2) and in coming to test temperature (see 6.4.1.1.1) shall be included as part of total 94,000 strokes. The number of strokes required, if other then 94,000 shall be specified in the material specification.

6.4.2 Procedure B. Stroking properties at sub-zero temperatures.

6.4.2.1 Conditions of test.

6.4.2.1.1 Test temperature. The test temperature shall be -40° " 3°C (-40° " 5°F) or -55° " 3°C (-67° " 5°F), as specified. The test shall be started at room temperature (23° " 5°C). The drop in temperature from room temperature to the specified test temperature shall take place in from 8 to 24 h, following the start of the test.

6.4.2.1.2 Pressure. The initial stroking pressure in the simulated brake system shall be adjusted at room temperature (23° " 5°C) to 3.4 gage MPa (500 psig), and recorded. The pressure stroke pattern shall be adjusted at 60/40 percent stroke ratio, with the air of the short cycle pressure recorder so as to produce a relatively smooth curve (see Figure 2). Accurate record shall be kept of any pressure variations during the test.

6.4.2.2 Break-in or burnishing run. The stroking rate shall be adjusted at 1,000 " 50 strokes/h, using the stroking pressure of 3.4 gage MPa (500 " 10 psig) applied at room temperature (23° " 5°C). While at room temperature the system shall be stroked 16,000 " 500 strokes, at the end of which the system shall be checked for leaks, and proper brake shoe clearance and the master cylinder checked for fluid level. The cylinder shall be filled to the given level, if necessary. The stroking rate shall then be adjusted to 720 " 50 strokes/h, using a 40/60 percent stroke ratio. The pressure-stroke pattern shall be adjusted, with the aid of the short-cycle pressure recorder, to give a relatively smooth curve (see Figure 3). Accurate record of observed pressure variations shall be compared with the initial pattern at approximately every 20,000 strokes. The stroking cycle, if other than that listed in this paragraph, shall be specified in the product specification.

6.4.2.3 Duration of test. The test shall be run until 94,000 strokes have been completed. The number of strokes given in the burnishing run (see 6.4.2.2) and in coming to the specified test temperature shall be included as part of the total 94,000 strokes. The number of strokes required, if other than 94,000, shall be specified in the product specification.

6.5 Observations.

6.5.1 Observations during test.

6.5.1.1 Pressure change. Observations at 12-hour intervals shall be made of the stroking pressure in the simulated system, to determine changes from the initial pressure.

6.5.1.2 Functioning of wheel cylinder pistons. The functioning of the wheel cylinder pistons shall be observed. Improper functioning of the pistons, such as freezing of the piston in the cylinder, or failure of the brake shoe to be released from the brake drum, shall be recorded. However, very slight drag of the brake shoe on the drum, when the drum is rotated by hand, shall not be considered as improper functioning of the cylinder pistons. Mechanical defects of the brake mechanism may cause excessive pressure rise or improper functioning of the wheel cylinder pistons. If inspection of the malfunction shows this to be the case, the test shall be repeated using new parts.

6.5.1.3 Leakage (or evaporation). The leakage (or evaporation) loss of the hydraulic brake fluid at 24,000-stroke intervals shall be determined by the mL of the fluid required to maintain the constant fluid level in the master cylinder reservoir. The level of the fluid should be checked daily.

6.6 Observations and measurements at end of test.

6.6.1 Leakage. The equipment shall be allowed to come to room temperature within 16 h following completion of the required number of strokes. The brake wheel cylinder shall be examined for evidence of excessive leakage. The assembly shall then be stroked an additional 100 strokes at 3.4 gage MPa (500 psig), and the cylinders again examined for evidence of excessive leakage.

6.6.2 Disassembly of cylinders. The wheel cylinders and the master cylinder shall be removed from the simulated system. The inlet ports of the cylinders shall be plugged or capped immediately upon removal to retain the fluid remaining therein. Each cylinder shall then be carefully disassembled. The fluid remaining in the cylinder shall be drained into a small screwcap jar, and saved for examination for evidence of sludging, jelling, sedimentation, and grittiness.

6.6.3 Inspection. A visual inspection of the disassembled cylinder shall be made, and a record made of the type and quantity of dirt; gum deposition; rusty, scored, and galled areas on the surface of the pistons and on the cylinder walls; chipping, scoring, and general condition of the rubber cups. All parts shall then be cleaned with 95% ethyl alcohol, and dried in a stream of air. The metal parts shall then be re-examined for evidence of corrosive action, such as pitting, etching, and severe stain.

6.6.4 Measurements.

6.6.4.1 Cylinders. The internal diameter of each wheel cylinder and of the master cylinder shall be measured as specified in 6.2.1.

6.6.4.2 Pistons. The external diameter of the master cylinder piston and of each wheel cylinder piston shall be measured as specified in 6.2.2.

6.6.4.3 Rubber cups. Measurements of the base and lip diameters of the rubber cups shall be made as specified in 6.2.3.1. Durometer hardness of the cups shall be determined as specified in 6.2.3.2. The diameter measurements and the hardness determinations shall be made within 1 h following removal from the cylinder assembly.

7. CALCULATIONS

7.1 Change in cylinder and piston diameter:

$$C = C_1 - C_2$$

where:
C = change in cylinder diameter.
C_1 = original cylinder bore diameter.
C_2 = final cylinder bore diameter.

$$P = P_1 - P_2$$

where:
P = change in piston diameter.
P_1 = original piston diameter.
P_2 = final piston diameter.

7.2 Change in base and lip diameter of rubber cups.

$$B = B_2 - B_1$$

where:
B = change in rubber cup base diameter.
B_1 = original base diameter.
B_2 = final base diameter.

$$L = L_2 - L_1$$

where:
L = change in rubber cup lip diameter.
L_1 = original lip diameter.
L_2 = final lip diameter.

7.3 Lip diameter interference set.

$$LDIS = \frac{L_1 - L_2}{L_1 - C_1} * 100$$

where:
LDIS = lip diameter interference set percentage.
L_1 = original lip diameter of rubber cup.
L_2 = final lip diameter of rubber cup.
C_1 = original cylinder bore diameter.

7.4 Change hardness of rubber cup.

$$H = H_2 - H_1$$

where:
H = change in durometer hardness.
H_1 = original hardness value.
H_2 = final hardness value.

7.5 Change in fluid pressure.

$$P = P_2 - P_1$$

where:
P = change in system pressure.
P_1 = original pressure value.
P_2 = final pressure value.

7.6 Change in fluid volume

$$V = V_2 - V_1$$

where:
V = change in fluid volume.
V_1 = original volume.
V_2 = final volume.

8. REPORTING

8.1 Summary. A summary of the test conditions and the inspection results shall be made at the completion of the stroking test. Reports shall be made on the following:

a. Degree (slight, moderate, or excessive) of corrosion, scoring, and galling of metal parts.
b. Change in the initial diameter of the cylinders and pistons (see 7.1).
c. Change in the base and lip diameters of the rubber cups (see 7.2).
d. Percentage of lip diameter interference set (see 7.3).
e. Change in the durometer hardness of rubber cups (see 7.4).
f. Degree (slight, moderate, or excessive) of swelling, tackiness, scoring, scuffing, or blistering of the rubber cups.
g. Pressure change in the system at end of each successive 12-hour interval (see 7.5).
h. Volume loss of fluid due to leakage or evaporation, at the end of each successive 24,000-stroke interval during the test (see 7.6).
i. Volume loss of the fluid at the end of the test (see 6.2.1 and 7.6).

j. Degree (slight, moderate, or excessive) of sludging, jelling, sedimentation, or grittiness of the fluid after testing (see 6.2.2).
k. Observed improper functioning of the wheel cylinder pistons.

9. PRECISION

9.1 Duplicate test runs should show the same general trend for a given hydraulic brake fluid, particularly in the matter of leakage during and after test, excessive increase in the stroking pressure, and improper functioning of the wheel cylinder pistons.

9.2 The test shall be repeated using new parts if mechanical difficulties, which cannot be attributed to the fluid proper, are encountered during the test.

Method Prepared By:

Army - ME - 1986

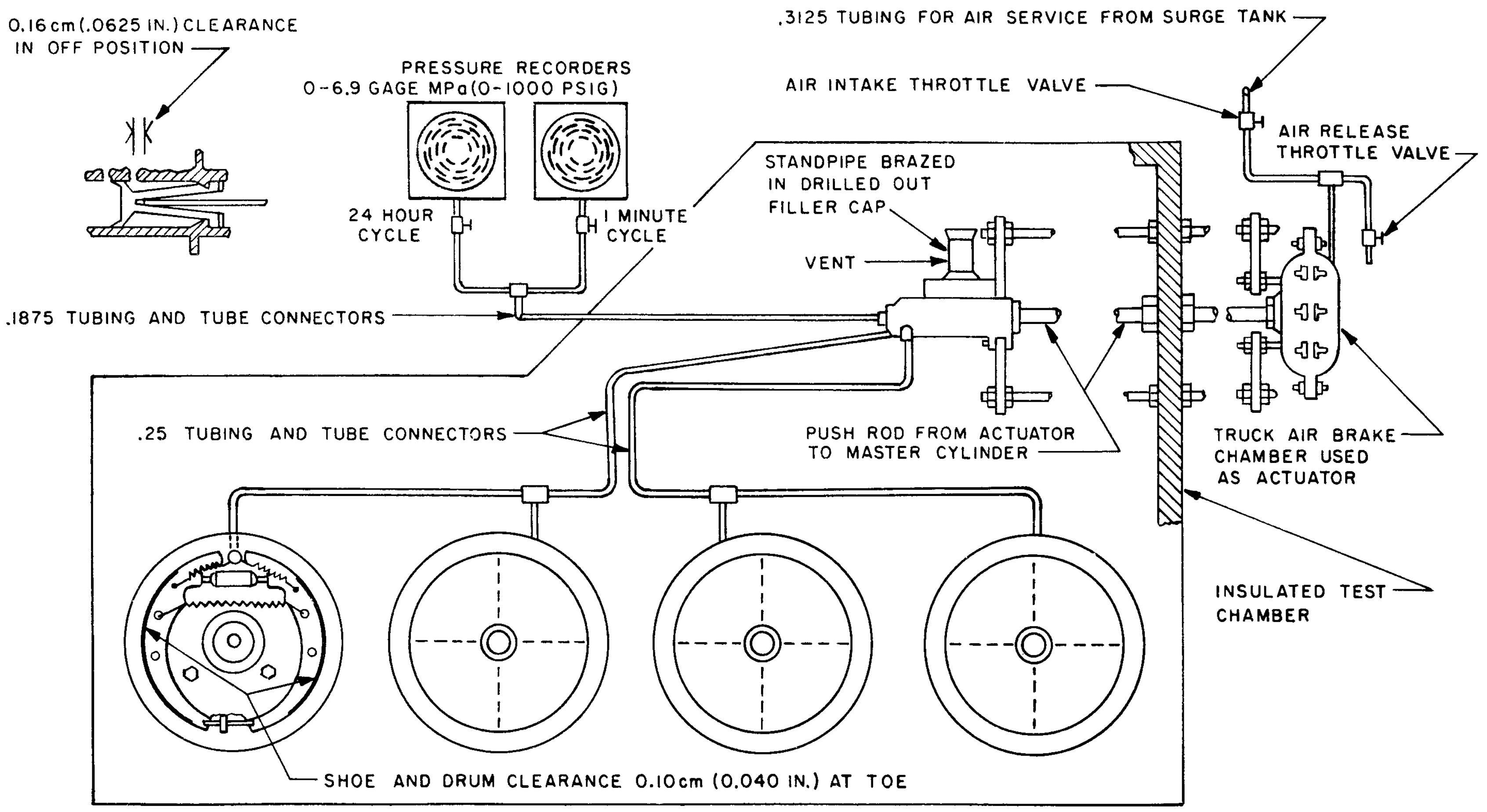


FIGURE I. Diagrammatic sketch of suitable apparatus.

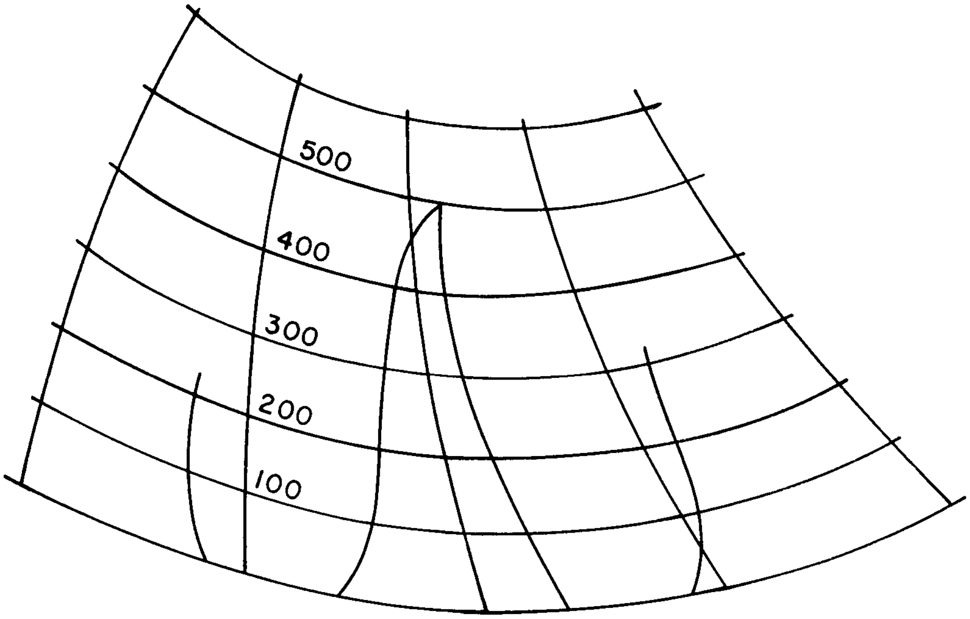


FIGURE 2. Pressure-stroke pattern 60/40 cam, 17 strokes per minute, chart speed - 1 r.p.m.

Method 550.1
September 30, 1986

EMULSIFICATION TENDENCIES OF PETROLEUM FUELS BY MULTIPLE CONTACT EXTRACTIONS

1. SCOPE

1.1 This method is used for determining the emulsification tendencies of petroleum fuels resulting from contact with the water bottoms usually found in fuel storage environments.

2. SUMMARY

2.1 A sample composed of water and test fuel is placed in a sealed bottle and subjected to a cycle consisting of shaking, settling, rating, siphoning, and addition of fresh fuel. The appearance of the fuel-water interface is reported after the settling period. A total of ten complete cycles is required.

3. SAMPLE SIZE

3.1 Approximately 1.1 L of the fuel to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Bottle, oil sample, 125 mL capacity (Kimble No. 60520L or equal), with No. 24 plastic screw cap.

4.2 Gasket material, fluorocarbon vulcanizate (Viton or equal), 0.16-cm thick sheet stock, cut to the I.D. of the No. 24 screw cap (approximately 2.1 cm).

4.3 Syringe, B.D., Luer-Lok, 100-mL capacity.

4.4 Syringe needle, Teflon (or equal), with Kel-F (or equal) hub, 16-gauge, 30.5-cm (12-in.) length (Hamilton No. KF16TF or equal).

4.5 Mechanical reciprocating shaker device (Equipoise Heavy-Duty or equal).

4.6 Graduated cylinder, 100-mL capacity.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Cleaning solution glass. Concentrated sulfuric acid [3,0,2] saturated with technical grade potassium dichromate [1,0,1].

5.3 Distilled or deionized water.

6. PROCEDURE

6.1 Immerse the oil sample bottle in the acid-dichromate cleaning solution, rinse it with tap water followed by distilled or deionized water, and allow it to drain until dry. If the bottle is still not clean, soak it in hot cleaning solution for 30 min. Rinse it with tap water followed by distilled or deionized water, and allow it to drain until dry.

6.2 Pour 10 mL of distilled or deionized water into the sample bottle. Carefully add 100 mL of the fuel to be tested and seal the bottle with the screw cap lined with a Viton gasket.

6.3 Place the sample bottle in the mechanical shaker in an upright position and shake it for 15 min. Remove the bottle and allow it to remain at 25° ± 3° C, undisturbed in darkness for 24 h (or 72 h during a weekend period).

6.4 After positioning the sample bottle on a flat surface with adequate lighting, rate and report the condition of the fuel-water interface in accordance with Table 1.

TABLE 1. Appearance of interface.

Rating	Appearance
0	Clean break at fuel-water interface
1	Very slight "skin" at fuel-water interface
2	Skin at fuel-water interface, heavier than #1
3	First sign of white emulsion forming at fuel-water interface
4	Emulsion occurring at interface and starting into water layer
5	Slightly more emulsion than #4 and with thin film on sides of bottle
6	Emulsion on bottom layer, water still visible
7	Semi-solid emulsion with bubbles (assuming a sponge-like configuration
8	Same emulsion as #7, but smaller bubbles
9	Almost a solid emulsion
10	Solid emulsion resembling mayonnaise

6.5 After rating the condition of the fuel-water interface, carefully remove approximately 95 mL of the fuel using the 100-mL syringe fitted Teflon needle.

NOTE: In removing the fuel, be careful not to disturb the fuel-water interface or the water bottom.

6.6 Carefully add a fresh 95-mL sample of the fuel to be tested and seal the sample bottle with the plastic screw cap as before.

6.7 Repeat the shaking, settling, rating, and siphoning steps as described in 6.3 through 6.6 until ten cycles (or extractions) have been performed.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report interface ratings as measured above. A rating of 3 or greater, denoting the presence of water-reactive impurities is considered unsatisfactory. The initial formation of these stable interfacial emulsions are indicative of potential filter-plugging and storage problems.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - ME - 1986

Method 1110.2
September 30, 1986

QUENCHING SPEED

1. SCOPE

1.1 This method is used to determine the quenching speed or rate of heat removal provided by quenching oils.

2. SUMMARY.

2.1 The test consists of immersing heated steel test bars in samples of the test quench oil. From temperatures recorded during the test procedure the quenching speed of the oil is calculated.

3. SAMPLE SIZE.

3.1 Approximately 4.5 L (1.2 gal) of quenching oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 One electric timer, graduated in 1/10 seconds.

4.2 Two containers, insulated with 5.1 cm (2 in) of insulation, approximately 30.5 cm (12 in) deep and 11.4 cm (4-1/2 in) wide (inside dimensions).

4.3 One stirrer, electric, of proper size to stir contents of containers.

4.4 One thermometer, 0°-100°C, with 1/10° graduations.

4.5 One furnace, controlled atmosphere, capable of being controlled at 816° ± 14°C (1500° ± 25°F).

4.6 One hook, steel, approximately 0.64 cm (1/4 in) diameter by approximately 1.2 m (4 ft) long (to lift and carry test bars).

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Five steel test bars, made from steel meeting the requirements of ASTM A 576, A675A, 675M, Steel No. 1050, approximately 2.5 cm (1 in) diameter by 6.4 cm (2-1/2 in) long, with loop on one end, scale free, weighing 250 ± 5 g.

5.3 Abrasive paper, 280 grit (ANSI B74.18).

5.4 1,1,1-Trichloroethane (ASTM D 4126).

5.5 Facial tissue.

6. PROCEDURE

6.1 Polish all five steel test bars with 280 grit abrasive paper to a uniform finish. Place the five test bars in the furnace and permit them to remain for at least one hour. The atmosphere in the furnace must be so controlled as to prevent the formation of carbon smut or scale. Clean the inside of the two insulated containers with 1,1,1-trichloroethane and facial tissue. Place 2000 ± 10 mL of the quenching oil under test in each of the containers. The oil must be at the temperature of the furnace room. Number the containers 1 and 2. Place the stirrer in container 1 and stir until the oil has reached a constant temperature. Record the temperature of the oil in this container. Turn off the stirrer. Remove one of the test bars from the furnace using the hook and vertically immerse the test bar beneath the surface of the oil. Start the stirrer and record the highest temperature obtained.

6.2 Place the stirrer in position in the second container and stir until the oil has reached a constant temperature. Record the temperature of the oil in this container and turn off the stirrer. Remove one test bar from the furnace and plunge it vertically beneath the surface of the unagitated oil. Hold the test bar in the oil for 5 ± 0.10 s. Remove the bar from the oil and start the stirrer. Operate the stirrer for approximately 15 s. Stop the stirrer and plunge the third test bar into the oil. Repeat this sequence until all of the test bars have been quenched. Record the total rise in temperature for the 4 test bars. The total rise in temperature produced by the four test bars quenched in container 2, divided by 4 gives the average temperature rise for each 5-second test.

NOTE: Speed and timing are essential in order to avoid heat losses. Stirring should not be violent, only sufficient to produce a uniform temperature.

7. CALCULATIONS.

$$Quenching\ speed = \frac{a}{b} * 100$$

7.1 Calculate the quenching speed of the oil by the following formula:

where:

a = the average rise in temperature produced by the four test bars quenched in container 2.

b = temperature rise produced by the fully quenched test bar (quenched in container 1).

8. REPORTING

8.1 Report the calculated quenching speed of the test oil.

9. Precision

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - AR - 1986

Method 1151.2
September 30, 1986

EXPLOSIVE VAPORS IN BOILER FUEL OIL

1. SCOPE

1.1 This method is used for determining the approximate percent explosiveness of gas and vapor mixture given off by fuel oil.

2. SUMMARY

2.1 The test consists of measuring the explosiveness of fuel-oil vapors produced under specified conditions, using a hot-wire type combustible-gas indicator that has been calibrated against a hydrocarbon gas of known explosiveness.

3. SAMPLE SIZE

3.1 Approximately 1 L (1 qt) of oil representative of the lot to be tested.

CAUTION: The one sample is to have been immediately stoppered (allowing a minimum of air space) upon removal from the lot.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Indicator, combustible-gas, hot-wire (Explosimeter Model No. 2, Mine Safety Appliances Co., Pittsburgh, Pa., or equivalent).

4.2 Flask, volumetric, 2 L, with 2-hole rubber stopper.

4.3 Graduated cylinder, 100 mL.

4.4 Tank, water (deep enough to accommodate 2-L volumetric flask and 100-mL graduated cylinder.).

4.5 Tube, drying, containing anhydrous calcium chloride.

4.6 Bulb, aspirator.

4.7 Vapor generating apparatus (Figure 1), consisting of:

a. Bottles (2), glass, 1 qt, wide mouth.
b. Stopper, rubber, 2-hole (for 1-qt bottle).
c. Stopper, rubber, 3-hole (for 1-qt bottle).
d. Thermometer, 40° to 82°C (100° to 180°F) (ASTM El).

e. Tube, outlet, copper, approximately 3/16-in O.D., sealed at one end with approximately 1/8-in hole, 1/2 in from sealed end.
f. Tubing, glass and rubber, as required.
g. Clamp, screw (for rubber tubing). Bath heating, water, 66° to 69°C (150° to 155°F).

4.9 Compressed-air supply.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Gas, hydrocarbon, with known lower limit of explosiveness (in volume percentage of gas concentration).

6. PROCEDURE

6.1 Prepare the 2-hole rubber stopper (for the volumetric flask) by inserting a short length of glass tubing in one hole and the sealed end of the copper outlet tube, used in figure l, in the other hole. Position the outlet tube so that the stopper seals the side opening of the tube; seal the outside end of the glass tube with a short length of rubber tubing and a screw clamp.

6.2 Prepare an air-gas mixture of 50% explosiveness (for use in calibrating the combustible-gas indicator), as follows:

a. Compute the volume of hydrocarbon gas (see. 5.2) required to produce a 2-L air-gas mixture of 50-percent explosiveness.
b. Place approximately 500 mL of water in the 2-L volumetric flask, and invert in the water tank.
c. Measure the required amount of hydrocarbon gas, using an inverted water-filled 100-mL graduated cylinder supported in the water tank so it will contain the computed volume of gas when the water levels in the graduate and the tank are the same.
d. Transfer the gas to the volumetric flask by carefully raising the lip of the graduate beneath the neck of the submerged flask.
e. Add air to the volumetric flask until the water levels (inside and outside) are at the graduation mark.
f. Insert the prepared 2-hole stopper (see 6.1) into the volumetric flask taking care to maintain the water seal of the gas mixture. Remove the flask from the tank and invert several times.

6.3 Calibrate the combustible-gas indicator by passing the air-gas mixture through the indicator and noting the reading as follows:

a. Fill one of the quart bottles about half full of water, and insert its 2-hole stopper (and tubing) as indicated for bottle A of Figure 1.
b. Connect the water-filled bottle to the rubber tubing on the volumetric flask.
c. Connect the copper outlet tube to the drying tube, and the drying tube to the combustible-gas indicator. Adjust the indicator needle to zero.
d. Reposition the copper outlet tube so that its side opening is exposed to the air-gas mixture, and apply low-pressure air to the water-filled bottle.
e. Adjust the screw clamp to produce a uniform flow of water of approximately 500 mL in 20 s, and record reading at which the indicator becomes stable.

6.4 Prepare the sample as follows:

a. Fill one of the quart bottles (bottle B) half full of test sample.
b. Insert the 3-hole stopper with its inlet tube, outlet tube, and thermometer. Position the outlet tube so that its side opening is covered by the stopper. Seal the inlet tube with a short length of rubber tubing and a screw clamp. Position the thermometer so that its bulb is approximately centered in the test sample.
c. Immerse the bottle (containing the sample) in the heating bath so that the level of the sample is below the level of the bath, and allow the sample to reach approximately 52°C (125°F). During the immersion (approximately 5 min) shake occasionally to insure even heating.

6.5 Test the explosiveness of the gas and vapor mixture of the sample as follows:

a. When the temperature of the sample reaches 52°C (125°F), remove the bottle and shake vigorously for a total of 5 min, with periodic immersions of the bottle in the water bath to maintain the fuel oil temperature at 52°C (125°F).
b. Fill bottle A approximately one-half full of water at about 52°C (125°F) and as soon as bottle B has been shaken for 5 min, connect the two bottles as shown in Figure 1.
c. Connect the outlet tube to the combustible-gas indicator, zero the indicator needle, and push the copper outlet tube down to expose its opening to the vapors in the bottle.
d. Apply low-pressure air to bottle A. Then while observing the indicator needle, slowly open and adjust the screw clamp to permit the water to flow into the sample-filled bottle at a uniform rate over a period of approximately 20 s. When the reading becomes constant, stop the flow, and record the reading.

CAUTION: Do not allow fuel oil to flow into the combustible-gas indicator.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the sample as having passed if its combustible-gas indicator reading does not exceed the reading for the hydrocarbon gas.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Navy - SH - 1986

Method 2508.1
September 30, 1986

THERMAL STABILITY OF LUBRICATING AND HYDRAULIC FLUIDS

1. SCOPE

1.1 This method describes a procedure for determining the thermal stability of fluids.

2. SUMMARY

2.1 In this method, the volatile decomposition products are in continuous contact with the fluid during test. To eliminate possible catalytic metal effects, the fluid is confined in a glass apparatus. To remove the variables of oxidation and hydrolysis, air and moisture are removed by heating and shaking under vacuum prior to the initiation of the test. This method will not measure the thermal stability threshold (the temperature at which volatile oil fragments begin to form), but will indicate bulk fragmentation occurring at a specified temperature and testing period.

3. SAMPLE

3.1 Approximately 20 mL of the lubricant to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Test cell. A borosilicate glass test cell having the dimensions given in Figure 1 is recommended as the sample container. Any heavy walled borosilicate glass closed end tube of the proper O.D. having sufficient capacity to hold 20 mL of fluid and of sufficient length to permit heating and drawing the tube to a sealed end reasonably above the fluid level should be satisfactory.

4.2 Heater. Aluminum block with at least two openings of the proper diameter (approximately 2 cm) and depth (approximately 21 cm) so that the sealed test cell is completely immersed in the heater. A suitable heating bath may be used in place of the aluminum block heater if desired. The temperature of either the aluminum block or the bath shall be capable of being controlled within ±1°C (±2°F) at the test temperature. The temperature gradient between fluid sample and the aluminum block or bath shall be kept at a minimum (less than 1°C [2°F] is recommended).

4.3 Test cell holder. When a heating bath is used a heavy walled steel sample holder of the proper size to contain the test cell shall be provided for safety reasons.

4.4 Becton, Dickinson and Co. Yale Luer-Lok (20 YL) or equal 20 mL hypodermic syringe equipped with a No. 14 square tip needle approximately 21 cm long.

4.5 Vacuum pump, capable of reducing the pressure to at least 1.0 mm of mercury.

4.6 Water bath, capable of maintaining a temperature of 80° ± 2°C (175° ± 5°F) and sufficient size to contain the test cell.

4.7 Laboratory safety shield, constructed of either safety glass or wire reinforced plastic.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Cleaning solution glass (concentrated sulfuric acid [3,0,2] saturated with potassium dichromate, technical grade).

5.3 Detergent for cleaning glassware [u,u,u].

6. PROCEDURE

6.1 Cleaning test cell. Clean the test cell by scrubbing with a suitable detergent and rinse with distilled water. Prior to the introduction of the sample into the test cell, the cell shall be rinsed in hot potassium dichromate-sulfuric acid cleaning solution, thoroughly rinsed in distilled water, and dried in an oven at 107° ± 2.5°C (225° ± 5°F) for 1 h.

6.2 Preheat and adjust heater temperature to 260° ± 1°C (500° ± 2°F).

6.3 Perform duplicate test procedures of 6.4 through 6.13.

6.4 Filter the sample through a nominal 10-µm filter paper to remove particle contaminants.

6.5 Introduce 20 mL of the filtered sample below the constricted area of the test cell with the aid of the hypodermic syringe.

NOTE: This syringe is capable of delivering 20.0 ± 0.5 mL of sample which is within the accuracy required by this test procedure.

6.6 Connect the filled test cell to the vacuum pump and degas the cell and its contents, while shaking gently for at least 5 min or until foam no longer persists.

6.7 Place the test cell, while still connected to the operating vacuum pump, in a water bath maintained at 80° ± 2°C (175° ± 5°F) for 5 min. Remove the cell from the bath, shake gently for a minute or two, and return the cell to the water bath. Continue this heating and shaking sequence for at least 4 cycles or until there is no longer evidence of foaming.

NOTE 1: Ester base as well as certain other type synthetic lubricants are hydrolytically unstable at 260°C (500°F). Therefore, it is necessary that dissolved as well as occluded water be removed before sealing the cell. Each 0.01% by weight moisture consumed by hydrolysis produces sufficient acid to raise the neutralization number by 0.31.

6.8 Seal off the constricted area of the test cell while the cell is under vacuum. Weigh the test cell to the nearest mg.

6.9 Insert the test cell in the heater (preheated to 260° ± 1°C [500° ± 2°F]) for a period of 24 h. If a heating bath is used insert the test cell in the steel holder and place the assembly in the bath.

NOTE 2: A safety shield should be used as a safety precaution when placing the test cells in the heater, removing the cells from the heater, and opening the cells.

6.10 At the conclusion of the heating period, remove the test cell from the heater and permit it to cool to room temperature. Visually observe the sample for evidence of insolubles, phase separation, or other change. A difference in color between the duplicate tests will necessitate retesting since color differences indicate improper preparation of one or both combustion tubes.

6.11 Reweigh the test cell and calculate the percent evaporation loss (see 7.1). If the evaporation loss is more than 0.05% by weight, repeat the test since a weight loss in excess of this amount indicates leakage of the test cell.

6.12 Remove the sample from the test cell. This may be accomplished by file scratching the cell near the top and applying a hot glass rod to the scratch mark.

6.13 Determine the following characteristics on a portion of both the original untreated sample and the heated sample in accordance with the test methods listed in Table I:

TABLE I. Characteristics of test methods.

Characteristic	ASTM Method
Viscosity (Kinematic) at 37.8°C (100°F)	D 445
Acid and base numbers	D 664

7. CALCULATIONS

7.1 To calculate evaporation loss, use the following formula:

$$\text{percentage loss} = \frac{W_b - W_a}{W_b} * 100$$

where:
W_b = weight of sample before heating.
W_a = weight of sample after heating.

8. REPORTING

8.1 Report the following:

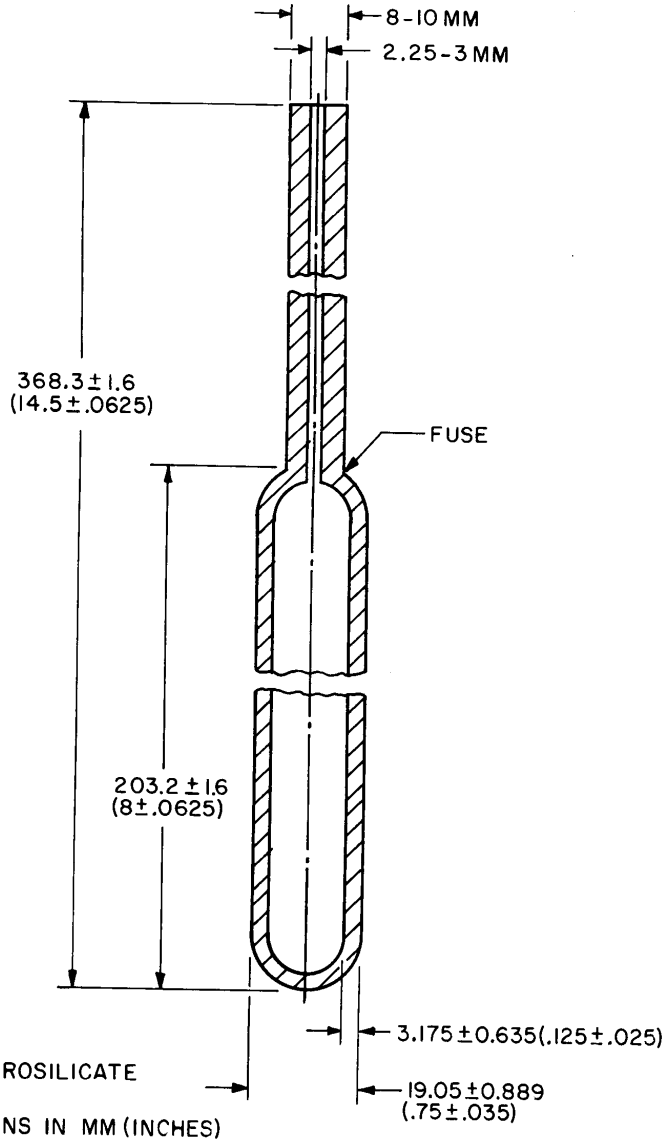
a. Visual appearance of fluid and test cell.
b. Average change of acid or base numbers of the duplicate tests.
c. Average change in kinematic viscosity at 37.8°C (100°F) of the duplicate tests.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Navy - NAPC - 1986



NOTES:

1. MATERIAL - BOROSILICATE GLASS.
2. ALL DIMENSIONS IN MM (INCHES) EXCEPT WHERE NOTED.

FIGURE 1. Test cell.

Method 3005.4
September 30, 1986

DIRT CONTENT OF GREASE

1. SCOPE

1.1 This method is used for determining the size and concentration of foreign particles in lubricating greases.

2. SUMMARY

2.1 The test consists of applying a known quantity of grease to a microscope slide, and examining the slide with a microscope to determine the size and number of particles present.

3. SAMPLE SIZE

3.1 Approximately 1 mL of grease to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Microscope, mechanical stage, 60 diameter magnification, with eyepiece micrometer (not filar) calibrated against suitability ruled, stage micrometer.

4.2 Slide, microscope, approximately 2.5 × 5.0 cm with cover glass.

4.3 Template (Fig. 1), steel (shim stock), 0.01 cm thick, approximately 2.5 × 5.0 cm, slotted from one end for a distance of approximately 2.0 cm with a slot width of approximately 1.0 mm. The template thickness and slot width are accurately measured so that the end area of the slot can be calculated. (A hemacytometer may be used in place of the template). See Note 1.

NOTE 1: Calculation of test results will be simplified if the template is ground to such thickness, or the slot width is adjusted so that the end area of the slot is 0.01 cm^2.

4.4 Vacuum chamber, 1.0 cm of mercury (absolute), or lower (to accommodate microscope slide).

4.5 Spatulas (2)

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

6. PROCEDURE

6.1 Compute the cross sectional area of the end of the slot (see 7.1).

6.2 Clean template, microscope slide and cover glass, and spatulas.

6.3 Prepare the sample for examination as follows:

a. Open a fresh container of grease to be tested, and with a clean spatula, scrape off and discard the surface layer.
b. Hold the template by the unslotted end, and place it against the clean microscope slide. Using another clean spatula fill the slot with the test grease. Make sure the corners are filled and the surface of the grease is level with, or slightly above the sides of the slot. Then press the side of the slot firmly against the slide.
c. Place the slide and template in the vacuum chamber for 10 to 15 min to remove bubbles or voids from the grease in the slot. (See Note 2).

NOTE 2: Vacuum exposure may be omitted if the grease is free from entrained air.

d. Slide the clean cover glass onto the template to shear off surplus grease. Then press the cover glass firmly against the template until it is parallel to the microscope slide.

6.4 Mount the assembled slide on the stage of the microscope. Adjust the illumination so that light passes straight up through the grease in the slot, as well as straight across the surface of the grease.

6.5 Determine the size and number of opaque particles.

a. Adjust the stage and micrometer eyepiece so that the micrometer scale coincides with one side of the slot, and so that one end of the scale coincides with one corner of the slot.
b. Move the stage slowly, so that the slot passes across the field of view. Count each opaque inclusion having a size of 25 µm or more. Particle size is determined by measuring along the largest dimension of the particle.
c. Record the count in three columns headed: "25 to 75 µm", "75 to 125 µm", and "over 125 µm".
d. When the micrometer scale has reached the opposite side of the slot, move the stage along the axis of the slot for a distance equal to the length of the scale. (Use a minor irregularity on the wall of the slot, or a specific particle in the grease, as a point of reference.) Scan this section completely across the slot.
e. Continue scanning successive sections in the slot until approximately three-quarters of the area (1 □ 1.5 cm) of the slot has been inspected.

f. Record the number of sections scanned, and the total number of particles according to size.

7 CALCULATIONS

7.1 Calculation of slot cross sectional area.

$$T = D - W$$

where:
T = end area of template slot.
D = depth or thickness of template (cm).
W = width of slot (cm).

7.2 Compute the number of particles per cubic centimeter of grease, using the following formulas:

25 µm or larger:

$$\frac{(A + B + C)}{TSN}$$

75 µm or larger:

$$\frac{(B + C)}{TSN}$$

Over 125µm:

$$\frac{C}{TSN}$$

where:
A = total number of particles 25 to 75 µm.
B = total number of particles 75 to 125 µm.
C = total number of particles 125 µm or larger.
N = number of sections of grease scanned.
S = length of micrometer scale, cm.
T = end area of template slot, sq cm.

NOTE 3: If dirt in excess of specification limits is not found in the first run, the grease shall meet the requirements of the test.

If dirt in excess of the specification limits is found in first run, two more runs shall be conducted. If dirt in excess of specification limits is found in one or both of these runs, the grease shall fail the test.

8. REPORTING

8.1 Report the number of particles in the following ranges:

25-75 µm
75-125 µm
125 or larger µm

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Navy - NADC - 1986

Method 3007.2
September 30, 1986

WATER DISPLACEMENT AND WATER STABILITY

1. SCOPE

1.1 This method is used to determine the water displacing ability of corrosion preventive oils and their stability in the presence of water.

2. SUMMARY

2.1 Steel test panels are immersed in distilled water and then either the test oil or a water emulsion of the test oil. After exposure in a static humidity chamber the panels are examined for the presence of rust.

3. SAMPLE SIZE

3.1 Approximately 60 mL of the test oil.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Standards

4.1.1 ASTM D 1748, Rust Protection by Metal Preservative In The Humidity Cabinet.

4.2 Static humidity chamber (a desiccator containing some distilled water).

4.3 Oven, forced draft, 54° ± 1°C (130° ± 2°F).

4.4 Desiccator, containing an indicating desiccant.

4.5 Panel hook, stainless steel (for handling panels during processing).

4.6 Erlenmeyer flasks, 125 mL.

4.7 Beakers, tall-form, 500 mL.

4.8 Petri dishes, 10 cm in diameter and 2 cm in height.

4.9 Controlled atmosphere (to provide 25° ± 3°C [77° ± 5°F]) and a maximum of 50 percent relative humidity.

4.10 Shaded fluorescent light. (A 15W balance illuminator type that will permit the panel to be viewed from all angles at a distance of 3 in).

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Test panels, FS 1009 steel, 5.1 □ 7.6 □ 0.16 cm (2 □ 3 □ l/16 in), fabricated from open hearth "killed" type low carbon, No. 4 soft temper cold rolled sheet or strip conforming to QQ-S-698. Cold drawn bar stock is not satisfactory.

5.3 Naphtha, aliphatic (TT-N-95) [1,4,0].

5.4 Methyl alcohol (O-M-232) [1,3,0].

5.5 Distilled water.

5.6 Silica sand, white dry, sharp. The size shall be such that it meets the following sieve requirements ASTM E11:

a. 100% must pass through a No. 10 sieve.
b. Minimum of 90% must pass through a No. 20 sieve.
c. Maximum of 10% permitted to pass through a No. 50 sieve.

5.7 Surgical gauze.

5.8 Absorbent tissue.

5.9 Emulsion, water and oil prepared as follows:

a. Place 50 mL of the test sample and 5 mL of distilled water in a 125-mL Erlenmeyer flask.
b. Shake mixture thoroughly and store overnight in an oven a 54° . 1°C (130° . 2°F).
c. Remove from oven and cool at 25° . 3°C (77° . 5°F) for testing.
d. Shake the mixture immediately prior to placing in the petri dish.

6. PROCEDURE

6.1 Clean the steel panels as follows:

a. Round the edges and ream out the suspension holes in accordance with ASTM D 1748.
b. Wipe the surfaces with solvent soaked rags and scrub with surgical gauze swabs in a beaker of hot naphtha.
c. Rinse in a beaker of hot methyl alcohol.

d. Cool the panels in a desiccator until further processing.
e. Test panels must be handled with a panel hook at all times, avoiding contact with any type of contaminated surface.
f. Maintain the solvents at a temperature high enough to keep the temperature of the panels above the dew point during the cleaning operation.

6.2 Sandblast the panels as follows:

a. Sandblast the edges and lightly sandblast the back of the panels with silica sand.
b. Sandblast the unnumbered side, or test surface, of the panels to a fresh, uniformly abraded surface.
c. Immediately after sandblasting, place the panels in a beaker of anhydrous methyl alcohol.
d. Heat the methyl alcohol so that the solvent will evaporate from the panels immediately upon withdrawal from the solvent.
e. Remove remaining residue by holding the panels in a rack at 20° from the vertical and spraying downward with naphtha.
f. Spray the test surface, then the back of the panel and the test surface again.
g. Rinse the panels in hot naphtha and hot methyl alcohol and store in a desiccator until cool.
h. Panels are to be used the same day as prepared.

<u>NOTE 1</u>: Caution. Methyl alcohol and naphtha are both toxic and flammable. Do not permit them to come in contact with the skin or breathe their fumes. Use only in a well ventilated area. Keep all flames away from these materials.

6.3 Three test panels are processed with the original oil as follows:

a. Dip the panels into distilled water, momentarily, and drain in a vertical position for not more than 5 s, with the bottom edge in contact with absorbent tissue.
b. Immerse the panels horizontally, and without agitation, for 15 s in a petri dish containing 50 mL of the test sample maintained at 25° ± 3°C (7.70° ± 5°F) and 50% maximum relative humidity.
c. Drain the panels momentarily and place in the static humidity chamber for 1 h at 25° ± 3°C (77° ± 5°F).
d. At the completion of the static humidity exposure, remove the panels and rinse in methanol.

e. Examine the test surfaces for the presence of rust, mottling, or other abnormal surface stains. The outer 0.32 cm (1/8 in) edge of the panels and 0.32 cm (1/8 in) around the holes is excluded from the evaluation.

6.4 Three test panels are processed with the oil-water emulsion in accordance with the procedure outlined in paragraph 6.3 and similarly examined.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Follow the specification requirements.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - AR – 1986

Method 3009.3
September 30, 1986

SOLID-PARTICLE CONTAMINATION IN HYDRAULIC FLUIDS

1. SCOPE

1.1 This method is used for determining the amount of solid particle contamination in hydraulic fluids.

2. SUMMARY

2.2 The method consists of determining the length of time required to filter the hydraulic fluid through a single membrane filter, and then examining the filter under a microscope to determine the number and sizes of solid particles.

3. SAMPLE SIZE

3.1 Approximately 100 mL of hydraulic fluid to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Microscope, mechanical stage, 100X and 45X magnifications, with micrometer eyepiece calibrated (for both magnifications) against a suitably ruled, stage micrometer.

4.2 Filter holder, Pyrex (Millipore Corporation, Bedford, Mass. 01730, No. XX10-047-00; also Gelman Instrument Co., 600 S. Wagner Road, Ann Arbor, MI, No. 1200WP).

a. Fritted-glass base and rubber stopper (No. XX10-047-02).
b. Holding clamp, spring action (No. XX10-047-03).
c. Funnel, graduated, 250 mL (No. XX10-047-04).

4.3 Cover, plastic (for graduated funnel) equipped with membrane filter to minimize contamination of test filter by air.

4.4 Flask, filter, side-tube (to accommodate filter holder, par. 4.2.2), 1000 mL.

4.5 Vacuum source, 36 to 43 cm (14 to 17 in) of mercury.

4.6 Slides (4), glass 5.0 × 7.5 cm (2 × 3 in).

4.7 Bottles (3), sample, with ground-glass stoppers, 1000 mL.
4.8 Forceps, stainless-steel, plastic tipped.

4.9 Petri dish, with cover (for four 5.0 × 7.5 cm glass slides).

4.10 Tongs, stainless-steel.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Distilled water.

5.3 Alcohol, isopropyl (TT-I-735) [1,3,0].

5.4 Petroleum ether (O-E-751) [1,4,0].

5.5 Detergent, water-soluble, commercial.

5.6 Cleaning solution, glass (concentrated sulfuric acid [3,0,2] saturated with potassium or sodium dichromate [1,0,1], technical grade).

5.7 Tape, transparent, pressure-sensitive.

5.8 Filters, cellulose membrane, 0.45 . 0.02 µm pores, 4.7-cm diameter, white, 0.308-cm grid, capable of filtering 200 mL of distilled water per square cm per minute at 69 kPa (10 psi) differential pressure and at 25°C. (Available from Millipore Corp., Gelman Instrument Co., or Decontamination, Inc.)

6. PROCEDURE

6.1 Clean the filter flask, filter holder, graduated funnel cover, and three sample bottles and stoppers as follows:

6.1.1 Wash in hot tap water and detergent.

6.1.2 Rinse with tap water.

6.1.3 Soak in hot glass-cleaning solution until clean.

6.1.4 Rinse with tap water.

6.1.5 Rinse with distilled water.

6.2 Prepare filter solvents as follows:

6.2.1 Insert a new membrane filter into the filter holder, and filter three 100-mL portions of distilled water to rinse the filter flask. Discard the rinsings.

6.2.2 Continue filtering enough distilled water to rinse each of the three prepared sample bottles (see 6.1 through 6.1.5) and stoppers with three 100-mL portions, and to fill one of them with filtered distilled water; stopper them immediately.

6.2.3 Replace the used filter with a new one, and using the cleaned filter flask, filter enough isopropyl alcohol to rinse the two clean sample bottles and stoppers with three 100-mL portions and to subsequently fill one with filtered alcohol; stopper them immediately.

6.2.4 Using a new filter, filter enough petroleum ether to rinse (with three 100-mL portions) and subsequently fill the last sample bottle; stopper immediately.

6.2.5 Remove the filter holder from the filter flask, and discard the used filter.

6.3 Using tongs to handle them, prepare two glass slides as follows:

6.3.1 Soak in hot glass-cleaning solution until clean.

6.3.2 Rinse with filtered, distilled water.

<u>CAUTION:</u> Immediately re-stopper the sample bottles after each use to avoid contamination.

6.3.3 Rinse with filtered isopropyl alcohol.

6.3.4 Rinse with filtered petroleum ether and allow to air dry.

<u>NOTE 1:</u> Caution. Isopropyl alcohol and petroleum ether are flammable. Use only in a well ventilated area. Keep all flames away from these materials.

6.3.5 When dry, immediately hinge the two slides together, using transparent tape, and store them in a clean covered petri dish.

6.4 Using tongs, prepare the filter holder for the filtration of the sample as described in 6.4.1 through 6.4.3.

6.4.1 Rinse the filter holder and the funnel cover, first with filtered distilled water, then with filtered, distilled isopropyl alcohol, and finally with filtered petroleum ether.

6.4.2 Using clean forceps to grip the edge, rinse a new membrane filter with filtered petroleum ether, and install it (grid side up) on the fritted glass surface of the filter holder. Also, install a filter in the cover for the graduated funnel.

6.4.3 Install and clamp the funnel to the filter holder. Ground the clamp (to prevent buildup of static electricity while filtering).

6.5 Filter the sample as follows:

6.5.1 Measure 100 mL of the sample into the funnel and cover the funnel.

6.5.2 If filtration time only is being determined, proceed to 6.5.2.1 and stop. If particle count is to be measured, proceed to 6.5.2.2 and continue.

6.5.2.1 Within one minute after pouring the sample into the funnel, apply suction of 36 to 43 cm (14 to 17 in) of mercury to the filtration apparatus and start the timer. When the first dry spot appears on the membrane filter, stop the timer, and disconnect the suction source.

6.5.2.2 Within one minute after pouring the sample into the funnel, start timer and apply suction of 36 to 43 cm (14 to 17 in) of mercury to the filtration apparatus. Disconnect the suction source and stop timer when the fluid level is 0.5 cm above the filter membrane, then add the rinse from 6.5.3 before the last of the hydraulic fluid has passed through the filter.

NOTE 2: If the time of filtration is more than 8% of the filtration time requirement, repeat the test using the filtration procedure in 6.5.2.1.

6.5.3 Wash the graduated cylinder with 50 mL of filtered petroleum ether. Remove the cover from the funnel, pour the washings into the funnel, replace the cover, and apply suction. Perform this washing operation once again, disconnecting the suction source between washings.

6.5.4 Allow suction to continue until the filter is dry.

6.6 Prepare the test filter for microscopic counting as follows:

6.6.1 Using clean forceps, remove the filter from the filter holder, and sandwich it between the cleaned glass slides (see 6.3).

6.6.2 Using transparent tape, seal the remaining unsealed edges of the slides.

6.7 Prepare a blank for analysis, using a fresh test filter and substituting filtered petroleum ether for the sample, as specified in 6.4, 6.5, and 6.6.

6.8 Place the slide under the microscope (45X), and scan the entire filter to ascertain that the particle distribution on the filter paper is uniform. If the distribution is not uniform, discard the filter and repeat the test.

6.9 Estimate the number of particles present in the filter grid area (100 squares), listing the numbers of particles by sizes according to the six size ranges given in Table I.

TABLE I. Size ranges and counting magnifications.

Particle size range (largest dimension), µm	Counting magnifications, diameters
5 to 15	100
15 to 25	100
25 to 50	100
50 to 100	100
Over 100 [1] (length-wide ratio under 10:1)	45
Over 100 [1] (length-wide ratio over 10:1)	45

[1] Fibers.

6.10 For each size range, determine sampling area and calibration factor on the basis of the estimate. If the estimated number of particles of that size was 5000 or less, use sampling area and factor shown in Table II. If the estimated number was more than 5000, determine area and factor as follows:

6.10.1 Select a sampling area consisting of at least 10 sections (chosen at random), each of which is one grid length long (0.308 cm), and narrow enough to contain no more than 50 particles (of that size range under consideration). (See Figure 1.)

6.10.2 Using the micrometer eyepiece, measure the width of each unit area, and determine the sum or the widths. Compute the calibration factor (see 7.1).

6.11 For each size range, count the particles within the sampling area, using the counting magnification specified in Table I. (See Figure 2). Include in the tally those particles lying on the upper and left boundary lines of the area as well as those within the areas selected. (Particles on the lower and right boundary lines are tallied with those in the square below or to the right.)

TABLE II. Calibration factors

Estimated number of particles	Sampling area	Calibration factor
1 to 50	100 squares (total area)	1
51 to 1000	20 squares (at random)	5
1001 to 5000	10 squares (at random)	10
More than 5000	10 sections [1]	

[1] See paragraph 6.10.

7. CALCULATIONS

7.1 Compute calibration factor:

$$Calibration\ factor = \frac{960}{3.08\ A} = \frac{317}{A}$$

where:
A = sum of widths of unit areas, cm.

8. REPORTING

8.1 For each size range, multiply the results by the calibration factor (see 6.10), subtract the values obtained for each size range for the blank from those obtained on the specimen filter and report the results as the total number of particles (of the size range) present (see Figure 2). Also, report the total filtration time and subparagraph from which it was obtained.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - ME - 1986

Method 3010.1
September 30, 1986

SOLID PARTICLE CONTAMINATION IN AIRCRAFT TURBINE ENGINE LUBRICANTS (GRAVIMETRIC PROCEDURE)

1. SCOPE

1.1 This method describes a gravimetric procedure for the determination of solid particle contamination in packaged aircraft turbine lubricants and related fluids.

2. SUMMARY

2.1 A known volume of fluid is filtered through a pre-weighed membrane filter. The increase in filter weight is determined after washing and drying and reported as total contaminant. The test filter is ashed and the inorganic material remaining is reported as ash.

3. SAMPLE SIZE

3.1 Approximately 1 L (1 qt) of turbine engine lubricant.

3.2 The sample must be thoroughly representative of the material to be tested. This requires vigorous agitation of the fluid before sampling. The importance of sampling cannot be too strongly emphasized. All suspected sediment must be homogeneously suspended in the oil. When packaged liter (quart) samples are available the entire liter (quart) should be used.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Membrane filters, white, plain, 4.7-cm diameter, pore sizes 1.2 and 0.22 µm.

4.2 Filter holder, 4.7 cm.

4.3 Analytical balance, sensitivity . 0.1 mg.

4.4 Petri dishes, 5.0 cm in diameter.

4.5 Oven, non-circulatory air, capable of maintaining a temperature of 90°C (200°F)

4.6 Forceps, flat-bladed with unserrated tips.

4.7 Vacuum system.

Muffle furnace capable of maintaining a temperature of 810°C (1500°F). 4.9 Crucibles, 30-mL capacity, squat form.

4.10 Desiccator, with desiccant.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Isopropyl alcohol, reagent grade [1,3,0].

5.3 Petroleum ether, reagent grade, boiling point range 30° to 60°C [1,4,0].

6. PROCEDURE

6.1 Preparation of apparatus and containers.

6.1.1 All components of the filtration apparatus and containers must be washed with hot tap water containing detergents.

6.1.2 Rinse with hot tap water.

6.1.3 Rinse with distilled water.

6.1.4 Rinse with filtered isopropyl alcohol.

6.1.5 Rinse with filtered petroleum ether.

6.1.6 Place clean plastic film or clean aluminum foil over containers and filtration apparatus.

NOTE 1: Rinse a sufficient number of times to completely remove the previous solvent.

6.2 Preparation of reagents.

6.2.1 Filter the petroleum ether, and isopropyl alcohol, through a 0.22-µm membrane filter. Discard the first 50 mL of each and filter enough solvents to complete testing of samples to be filtered. Containers used for filtered reagents must be cleaned initially as described in Section 6.1.

NOTE 2: Ground the filter mechanism when filtering flammable solvents to prevent buildup of static electricity.

6.3 All equipment used for handling samples must be cleaned as described in Section 6.1. Precaution must be taken to minimize apparatus contamination from all sources including airborne particles.

6.3.1 Duplicate tests should be performed whenever more than 1 L (1 qt) of lubricant from the same lot or batch is available.

6.4 Preparation of test filters.

6.4.1 Identify each filter by marking the rim with a ballpoint pen, or by marking the petri dishes that will contain them. Place each filter separately in petri dish and dry it for 1/2 h in an oven regulated to 90°C (194°F). The petri dishes are then placed in a desiccator and moved to the balance room in the vicinity of a balance on which weighings are to be made. Allow 1 h for the closed desiccator and contents to come to equilibrium with ambient temperature. Use forceps to place the test membrane on the balance pan by handling the edge only.

6.4.2 An air ionizer must be kept permanently on the base of the balance adjacent to the pan to eliminate weighing errors due to electrostatic charge effects. Make all weighings to the nearest 0.1 mg.

6.5 Laboratory filtration and contamination determination.

6.5.1 Thoroughly clean outside of sample container in the region to be opened by washing with detergent and water and rinsing with tap water and filtered isopropyl alcohol. Shake the 1 L (1 qt) sample of lubricant for 15 min. Cautiously open can so as to prevent the introduction of metal chips into the sample.

6.5.2 To keep airborne contaminants on the membrane surface to a minimum, pour some of the sample into the filter funnel before applying vacuum. Filter the entire contents of the 1 L (1 qt) sample and thoroughly wash down the inside of the sample can with filtered petroleum ether, passing the washings through the filter.

6.5.3 Wash down the inside of the filter funnel. This will complete the transfer of the contaminant to the filter membrane. With the vacuum applied, carefully remove the funnel. Wash the periphery of the membrane by directing a gentle stream of petroleum ether from the edge toward the center, taking care not to wash any contaminant from the surface of the membrane.

6.5.4 Carefully remove the test membrane from the filter base, using clean flat forceps, and place it in a covered petri dish. Dry and reweigh as described in Section 6.4, taking care not to wash any contaminant from the surface of the membrane.

NOTE 3: For greater accuracy, control membranes may be used as described in ASTM D 2276

(Particulate Contaminant in Aviation Turbine Fuels).

6.6 Determination of non-combustible contamination or ash.

6.6.1 Clean crucibles with hot detergent, wash and rinse with tap and distilled waters and dry. Place crucibles in a muffle furnace maintained at 810°C (1500°F) for 1/2 h. Remove and place crucibles in a desiccator. Allow closed desiccator and contents to cool and come to equilibrium with the atmosphere in the vicinity of the balance for 2 h. Handle crucibles with clean tongs. Weigh crucibles to the nearest 0.1 mg. Repeat this procedure until constant weight is obtained.

6.6.2 Handling the membrane filter only with clean forceps, lay it, contaminant side upwards, in the center of the crucible. Wet it with filtered isopropyl alcohol and push it gently into the crucible.

6.6.3 Cover the crucible containing the filter membrane with a porcelain lid and place it in a cold muffle furnace. Start the furnace and allow to come to 810°C (1500°F). Hold at this temperature for 1/2 hour. Remove and place crucibles in a desiccator. Allow closed desiccator and contents to cool and come to equilibrium with the atmosphere in the vicinity of the balance for 2 h. Handle crucibles with clean tongs. Weigh crucibles to the nearest 0.1 mg. Repeat this procedure until constant weight is obtained. Increase in weight is inorganic material or ash.

NOTE 4: An alternate method is to place the crucible in a triangle and put a porcelain cover over it. Place a small flame under the crucible to one side. When dry, char the filter membrane, being careful not to ignite. Increase the burner flame until all organic matter is distilled from the filter. Place the crucible in a preheated muffle furnace 810°C (1500°F) for 1/2 h. Weigh as in 6.6.3.

7. CALCULATIONS

$$Contaminant, mg\ per\ L\ of\ oil = \frac{A - B}{C}$$

where:
A = final weight of the membrane filter with the material collected in filtration, mg.
B = initial weight of the test membrane, mg.
C = volume of sample in L filtered through the test membrane.

$$Ash, mg\ per\ L\ of\ oil = \frac{D - B}{C}$$

where:
D = final weight of the crucible and noncombustible material, ash, mg.
B = initial weight of the crucible, mg.
C = volume of sample in L filtered through the test membrane.

8. REPORTING

8.1 Contamination, mg per L of oil.

8.2 Ash, mg per L of oil.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by

Navy - NAPC - 1986

Method 3012
September 7, 2007

DETERMINATION OF PARTICULATE MATTER IN AEROSPACE HYDRAULIC FLUIDS

1. SCOPE

1.1 This method describes the determination of particulate matter in aerospace hydraulic fluids with an automatic particle counter analyzer working on the light interrupt principle. This document when cited in product specifications becomes a mandatory test requirement.

2. SUMMARY

2.1 This method utilizes an automatic particle counter with a minimum of five channels, fitted with photoelectric level detectors, a liquid sensor suitable for counting particles in the 2-400 µm range size, and an automatic bottle sampler with a built-in pressure/vacuum chamber that can subject samples to pressures up to 60 psi. The particle counter must be able to deliver flow rates of 10 to 200 mL/min, handle a minimum sample volume of 100 mL and have a minimum concentration limit of 10,000 particles/mL.

3. SAMPLE SIZES

3.1 Minimum sample size for analysis is 120 mL (4 oz).

4. STANDARDS AND APPARATUS

4.1 Automatic particle counter with a minimum of five channels, fitted with photoelectric level detectors, a liquid sensor suitable for counting particles in the 2-400 µm range size, and an automatic bottle sampler with a built-in pressure/vacuum chamber that can subject samples to pressures up to 60 psi.

4.2 Sample bottles - Round flat-bottomed glass bottles fitted with a polypropylene threaded cap forming a seal with the bottle without the use of any insert, approximately 250 mL capacity or manufacturer's recommended size.

4.3 Solvent dispenser – Suitable dispensers fitted with a ≤0.45 µm membrane filter (Millipore HAWP 047 or equivalent) at the outlet. If solvent has been pre-filtered using a 0.45 µm or smaller size filter, then an inline filter is not required.

4.4 Timer - Timer that is accurate to the second, ±0.5.

5. MATERIALS

5.1 Solvent – Hexanes (certified) filtered through a ≤ 0.45 µm (Millipore HAWP 047 or equivalent) filter prior to use.

5.2 Detergent – Micro-90 Cleaning solution (available through Fisher Scientific and Cole-Parmer) or equivalent.

6. PROCEDURE

6.1 CALIBRATION

6.1.1 Particle counters will be calibrated every 6 months, and after any repairs that could affect calibration or whenever there is a reason to suspect inaccuracy in test results.

6.1.2 Instrument calibration will be performed using a certified calibration kit consisting of a known suspension of Arizona Certified Fine Test Dust (ACFTD) in MIL-PRF-5606 (NATO Code H-515) hydraulic fluid by qualified personnel and adhering to the manufacturer's operations manual.

6.2 SAMPLER BOTTLE PREPARATION

6.2.1 Wash sample bottle thoroughly with warm tap water containing detergent.

6.2.2 Rinse with warm tap water.

6.2.3 Rinse with either distilled or deionized water.

6.2.4 Oven dry at 90 ± 5°C.

6.2.5 Allow to cool at room temperature in a suitable contamination-free environment.

6.2.6 Rinse with filtered solvent.

6.2.7 Drip dry.

6.2.8 Store bottles with caps in place at room temperature, in a suitable contamination-free environment.

6.3 SAMPLE PREPARATION

6.3.1 Bottle sample.

6.3.1.1 Fluid samples shall be rejected if visibly contaminated with water or excessive solids.

6.3.1.2 Using filtered solvent, thoroughly clean the outside of the bottle, particularly in the region of the cap.

6.3.2 Hermetically sealed containers.

6.3.2.1 Thoroughly clean the outside of the container, particularly the top where it will be opened, with filtered solvent.

6.3.2.2 Shake sample vigorously by hand for one minute.

6.3.2.3 Puncture the cleaned surface with a cleaned and rinsed tool, making a hole that is no larger than necessary.

6.3.2.4 Transfer a mid-stream sample of at least 125 mL (or manufacturer's recommended volume) to a sampler bottle and cap it.

6.3.3 Other containers.

6.3.3.1 Thoroughly clean the outside with filtered hexanes paying particular attention to the cap region.

6.3.3.2 Agitate container to ensure adequate distribution of particles in the fluid.

6.3.3.3 Transfer a mid-section sample of at least 125 mL (or manufacturer's recommended volume) to a sampler bottle and cap it.

6.4 SET-UP PARAMETERS

6.4.1 The particle counters will have the following set-up:

6.4.1.1 Flow rate – 60 ± 2 mL/min (or that recommended by manufacturer).

6.4.1.2 Vacuum rate – a minimum of 53.5 kPa (15.8 in Hg).

6.4.1.3 Sample volume – The sample shall be counted in at least two equal volumes of at least 20 mL each, run consecutively.

6.4.1.4 Set particle counter to differential mode.

6.4.2 Ensure that the particle counter has been switched on at least 30 min prior to start of analysis or follow manufacturer's recommendation.

6.4.3 Prior to daily analysis, run a sample of filtered solvent to verify particle cleanliness level. The particle counter must meet SAE AS 4059 (NAS 1638) Class 00 (Table 1) limits.

Table 1 – SAE AS 4059 (NAS 1638) Class 00

Particle Size Range (µm)	Number of Particles / 100 mL (max)
5 - 15	125
16 - 25	22
26 - 50	4
51 - 100	1
>100	0

6.5 SAMPLE ANALYSIS

6.5.1 Shake the bottle vigorously by hand for one minute. Particle counting shall commence within two minutes after shaking.

6.5.2 Place sampler bottle in sample assembly and apply a vacuum of at least 53.5 kPa (15.8 in Hg) for 40 seconds to remove air bubbles.

6.5.3 Turn-off vacuum source and immediately start particle count.

6.5.4 Flush the sensor with a small volume of sample.

6.5.5 Record count result per calibrated channel.

6.5.6 Repeat sample counting as appropriate.

6.5.7 After each sample run, flush the sensor with filtered solvent.

7. CALCULATIONS

7.1 Average the particle count of the runs (discarding any statistically suspect data) per volume used per channel.

8 REPORTING

8.1 Report to no more than three significant figures, the number of particles per 100 mL in the following size ranges: (DELTA mode).
5 to 15 µm
16 to 25 µm
26 to 50 µm
51 to 100 µm

>100 µm

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

AFTL - 2007

Method 3013
September 30, 1986

DETERMINATION OF PARTICULATE CONTAMINATION IN SYNTHETIC TURBINE ENGINE LUBRICANTS

1. SCOPE

1.1 This method describes a gravimetric procedure for the determination of particulate contamination in synthetic turbine engine lubricating oils.

2. SUMMARY

2.1 A known volume of oil is filtered through a pre-weighed membrane filter. The increase in filter weight is determined after washing and drying and is considered particulate contaminant. Particulate contaminant per liter of oil is calculated from the volume of oil filtered and the weight of particulate contaminant.

3. SAMPLE

3.1 Approximately 1 L of the oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Membrane filters, 4.7 cm diameter, absolute pore size of 3 µm and 0.45 µm. Filters must be compatible with the required reagents and with MIL-PRF-7808 lubricants at elevated temperatures. Millipore MF filters or equivalent shall be used.

4.2 Filter holder, 4.7 cm diameter.

4.3 Analytical balance, sensitivity . 0.1 mg.

4.4 Petri dishes, 10.0 cm diameter with covers.

4.5 Oven, non-circulatory air, capable of maintaining temperature of 90°C.

4.6 Forceps, flat-bladed without serrated tips.

4.7 Vacuum system, 62.5 cm Hg or better.

4.8 Laboratory wash bottle.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Isopropyl alcohol, reagent grade [1,3,0].

5.3 Petroleum ether, 30° to 60°C boiling range, reagent grade [1,4,0].

6. PROCEDURE

6.1 Sampling

6.1.1 The sample must be representative of the material to be tested. When samples represent packaged quart containers, the entire contents must be filtered. When samples represent packaged 8-oz containers, the entire contents of at least two, but no more than four, of the 8-oz containers must be filtered. When the material is packaged in containers larger than 1 qt, a representative 1 L (1 qt) sample must be taken in a clean container for the filtration test. The entire contents of these samples must be filtered.

6.2 Preparation of apparatus and containers.

6.2.1 Wash all components of the filtering apparatus with hot water and detergent.

6.2.2 Rinse with hot tap water.

6.2.3 Rinse with distilled water.

6.2.4 Rinse with filtered isopropyl alcohol.

6.2.5 Rinse with filtered petroleum ether.

6.2.6 Place clean plastic wrap or aluminum foil over the filtering apparatus when not in use.

6.3 Preparation of reagents.

6.3.1 Filter the isopropyl alcohol and petroleum ether through a 0.45-µm membrane filter. Discard the first 50 mL of each solvent filtered. Filtering equipment and storage containers for the solvents must be cleaned as described in 6.2.

NOTE: Ground the filter mechanism to prevent build-up of static electricity.

6.4 Preparation of test filters.

6.4.1 Place a 3-µm membrane filter in a petri dish and dry for 30 min in an oven regulated to 90°C with petri dish lid ajar. Remove petri dish and with lid closed, place in vicinity of balance on which weighings are to be made. Allow filters to cool for 30 min and weigh to the nearest 0.1 mg. Use forceps to handle the test membranes. An air ionizer must be utilized during all weighings to eliminate errors due to electrostatic charge effects.

6.5 Filtering of test sample.

6.5.1 Inspect visually the top of the sample container for dust and dirt. If present, wash top of container using laboratory wash bottle containing filtered petroleum ether.

6.5.2 Shake sample mechanically for 5 min prior to test. Puncture small opening in top of container (vent in case of screw cap containers) and heat to a maximum of 60°C to facilitate filtering. Lubricant formulations which filter readily (30 min or less per L [qt]) need not be heated.

6.5.3 Cautiously open containers so as to prevent the introduction of metal particles into the sample. Pour approximately 50 mL of sample into the assembled filtering apparatus containing the pre-weighed filter and apply vacuum. Filter entire contents of the sample container and record filtering time in min. All of the sample must be filtered through one filter. Thoroughly wash down the inside of the sample container with filtered petroleum ether, passing the washings through the filter.

6.5.4 Wash down the inside of the filter funnel using laboratory wash bottle containing filtered petroleum ether. With vacuum still applied, carefully remove the funnel. Wash the periphery of the membrane by directing a gentle stream of filtered petroleum ether from the edge toward the center, taking care not to wash any contaminant from the surface of the membrane.

6.5.5 Carefully remove the test membrane from the filter base and place it in a petri dish. Dry and reweigh as described in 6.4.1.

7. CALCULATIONS

7.1 Calculate particulate contaminant per liter of oil from the initial and final weight of the test filter and the volume of sample filtered.

$$Particulate\ contaminant, mg/liter\ of\ test = \frac{A - B}{C}$$

where:
A = final weight of test filter in mg.
B = initial weight of test membrane in mg.
C = volume of sample measure in liters passed through the filter.

8. REPORTING

8.1 Report the contaminant in mg/L and filtering time in min.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Air Force - AFWAL/POSL - 1986

Method 3110.1
September 30, 1986

FATTY OIL IN CUTTING FLUIDS BY INFRARED SPECTROPHOTOMETRY

1. SCOPE

1.1 This method of analysis is intended for the determination of fatty oil in sulfurized fatty mineral and sulfurized, chlorinated fatty mineral cutting fluids. The method is based on the assumption that the fatty oil will consist entirely of lard oil. The method is not applicable to the fatty oil analysis of cutting oils which contain oxidized oils or compounds containing carbonyl groups which also absorb at 1743 cm^{-1}, the point in the spectrum upon which this analysis is based. Sulfur- and chlorine-containing compounds which often interfere with the conventional fatty oil analysis do not interfere with this method.

2. SUMMARY

2.1 The total concentration of fatty oil in a cutting fluid is determined by measuring the absorbance at the carbonyl absorption band at 1743 cm^{-1} by means of infrared spectrophotometry. A curve showing the relationship between fatty oil content and absorbance at 1743 cm^{-1} is used to determine the percent of fatty oil in the cutting fluid.

3. SAMPLE SIZE

3.1 Approximately 100 mL of the oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 An infrared spectrophotometer capable of producing a distinct carbonyl absorption band at 1743 cm^{-1}. The Perkin-Elmer Infrared Spectrophotometer, Model 621, has been found satisfactory. A sealed liquid cell with sodium chloride or potassium bromide windows may be used. It is recommended that the path length used be approximately 0.025 mm.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Spectrophotometric standards of A-A-59197 Fatty Oil in 0.0-15% Mineral Oil.

6. PROCEDURE

6.1 Calibrate the spectrophotometer in accordance with the manufacturer's instructions. Prepare a series of at least five standards consisting of mixtures of fatty oil meeting the requirements of A-A-59197 and mineral oil meeting the requirements of 0.0 – 15%. If the sample being analyzed contains more than 15% fatty oil, dilute it with sufficient mineral oil which meets the requirements of fatty oil in 0.0 – 15% mineral oil, to reduce the fatty oil content below 15% so that the standards prepared above can be utilized. Determine the absorbance values of the standards in accordance with 5.2 and plot the percent fatty oil against the absorbance values at 1743 cm^{-1}.

6.2 Thoroughly mix the sample but avoid producing air bubbles. Fill the cavity in the sealed cell, avoiding any air inclusion. Samples suspected of containing any moisture should not be used as the moisture will fog the sodium chloride or potassium bromide windows or change the effective path length of the cell. Place the filled cell in the beam of the infrared spectrophotometer and scan between 2000 and 1500 cm^{-1}. Determine absorbance of the band at 1743 cm^{-1}. Determine absorbance of the band at 1743 cm^{-1} using the Base Line Method. This method consists of drawing the base line as nearly parallel as possible to the radiant energy background as shown in Figure 1. The absorbance is then calculated from the equation in Section 7.1. Determine the percent fatty oil in the sample from the plot prepared from the standards (see 5.2). It is suggested that standards bracketing the expected fatty oil concentration be run concurrently with the sample undergoing test to check the previously prepared plot prepared from the standards and to compensate for any variability in equipment operation. If the absorbance of these standards do not agree with the previously prepared plot, prepare a new working curve prior to analyzing the sample.

7. CALCULATIONS

7.1 Calculation of absorbance of 1743 cm^{-1} band:

$$A = \log_{10} \frac{T_B}{T_P}$$

where:
A = absorbance
T_B = the distance from the zero line to the base line
T_P = the distance from the zero line to the selected absorption peak.

8. REPORTING

8.1 Report the percent of fatty oil found in the cutting fluid.

9. PRECISION

9.1 Use the following criteria as a basis for judging the acceptability of results (95% confidence):

a. Repeatability. Duplicate results by the same operator should be considered suspect if they differ by more than 0.2%.
b. Reproducibility. The results submitted by each of two laboratories should be considered suspect if they differ by more than 0.3%.

Method Prepared By:

Army - AR - 1986

Method 3180.2
September 30, 1986

SULFUR IN CUTTING FLUIDS (ACTIVE AND TOTAL)

1. SCOPE

1.1 This method is used for the determination of active and total sulfur in cutting fluids. It is used in conjunction with ASTM D 129.

2. SUMMARY

2.1 Total sulfur content of the fluid is determined in accordance with ASTM D 129 with the noted exceptions. Active sulfur content is determined by first reacting the fluid with some copper powder, determining the sulfur content of the reacted fluid and then subtracting this sulfur content from the total sulfur content.

3. SAMPLE SIZE

3.1 Approximately 45 mL of the cutting fluid to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Standards

4.1.1 ASTM Method D 129, Sulfur in Petroleum Products by the Bomb Method.

4.2 Beaker, lipless, tall-form, 250 mL capacity.

4.3 Auger stirrer blade, 1.0 cm wide and 7.5 cm high.

4.4 Thermometer, 177°C (350°F), range of 150° to 200°C.

4.5 Bomb, minimum capacity 300 mL, electrically fired (see ASTM D 129).

4.6 Sample cup, platinum or nickel (see ASTM D 129).

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment.

5.2 Copper powder, ACS. Baker Chemical Company Catalog No. 1728, Copper, Purified Powder, has been found satisfactory.

5.3 Nitric acid, 5% [2,0,0].

5.4 Acetone, ACS [1,3,0].

5.5 Isooctane, knock test grade [0,3,0].

5.6 Filter paper, Whatman No. 4 and No. 42 or equivalent.

5.7 Firing wire, iron, approximately 26 B&S gage.

5.8 Blue litmus paper.

5.9 Materials required for ASTM D 129.

6. PROCEDURE

6.1 Preparation of copper powder. Place approximately 20 g of copper powder in a 250-mL beaker and add 5% nitric acid until all the powder is covered. Heat the beaker and contents gently until the surface of the copper powder begins to react. Allow the reaction to continue for about one minute or until the copper powder is free of oxides. Add sufficient distilled water to stop the reaction. Decant and discard supernatant liquid. Wash the copper powder on a No. 4 Whatman or equivalent filter paper with distilled water until the wash water shows no trace of nitric acid when tested with blue litmus paper. When all the nitric acid has been removed, wash the copper powder with sufficient acetone to remove all traces of water. If not ready to proceed with the test, the dried copper powder may be stored under knock test grade isooctane.

NOTE 1: Caution. Isooctane is flammable. Do not use isooctane near an open flame.

6.2 Sulfur determinations.

6.2.1 Active sulfur. Place 20 ± 1 g of the sample fluid in a 250-mL tall-form lipless beaker and add approximately 2.0 ± 0.1 g of the dried copper powder prepared in 6.1. Place the beaker and contents on a hot plate and heat the mixture to 149° ± 3°C (300°± 5°F) while stirring at 1750 rpm for at least 1 hour. At the end of the heating and stirring period, cool the mixture to room temperature and filter through a Whatman No. 4 or equivalent filter paper. Determine the sulfur content of the filtrate (the reacted fluid) in accordance with ASTM D 129 with the following exceptions:

a. Paragraphs 3.2, 3.3: A nickel cup and iron wire may be used

b. Paragraph 5.1: Place 15 mL of sodium carbonate solution in the bomb in place of the prescribed 5 mL

c. Paragraph 5.5: After removing the beaker containing the barium sulfate from the hot plate, permit the barium sulfate to settle for a minimum of 4 hours, preferably overnight.

d. Paragraph 5.5: After the barium sulfate has settled for a minimum of 4 hours, filter the supernatant liquid through a Whatman No. 24 or equivalent filter paper and wash with hot water until all traces of barium chloride are removed. The active sulfur content of the cutting fluid is equivalent to the difference between the total sulfur content of the cutting fluid (as determined in 6.2.2) and the sulfur content of the reacted cutting fluid.

6.2.2 Total sulfur. Determine the total sulfur content of the cutting fluid in accordance with ASTM D 129 with the exceptions listed in paragraph 6.2.1.

7. CALCULATIONS

7.1 Weight percent of sulfur as calculated in 6.1 of ASTM D 129.

7.2

$$Active\ sulfur\ content = Total\ sulfur\ content - sulfur\ content\ of\ reacted\ fluid$$

8. REPORTING

8.1 Report active and total sulfur contents to the nearest 0.01%.

9. PRECISION

9.1 Reference paragraph 8.1 of ASTM D 129 for percent sulfur contents.

9.2 No precision data available for sulfur content of reacted fluids.

Method Prepared By:

Army - AR – 1986

Method 3201.7
September 30, 1986

EMULSION (PETROLEUM AND PETROLEUM-LIKE PRODUCTS)

1. SCOPE

1.1 This method is used for determining the emulsifying tendency of petroleum and petroleum-like products.

2. SUMMARY

2.1 The test consists of stirring the sample and its emulsifying agent at a specified temperature, allowing the emulsion to remain undisturbed at the test temperature for the time required by the specification, and examining the emulsion for any separation.

3. SAMPLE SIZE

3.1 Approximately 40 mL of the oil or fluid to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Graduated cylinder, 100 mL (approximately 28.6 mm [1-1/8 in] I.D.).

4.2 Heating bath (oil or water), 54° " 0.5°C (130° " 1°F) (unless otherwise specified) deep enough to cover 85 mL mark of the graduated cylinder.

4.3 Stirring mechanism (Figure 1) consisting of:

a. Paddle, copper strip, 12.1 H 1.9 H 0.16 cm (4-3/4 H 3/4 H 1/16 in).
b. Shaft, stirring, with means for attaching paddle long enough to immerse paddle to approximately 0.64 cm (1/4 in) from bottom of graduated cylinder.
c. Motor, stirring, approximately 1500 rpm, for rotating shaft and paddle.

4.4 Thermometer (range to suit requirements of specification).

4.5 Wooden stick or rubber policeman.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Emulsifying agent (distilled water, sodium chloride solution, or synthetic seawater) as required by the specification.

6. PROCEDURE

6.1 Secure paddle to shaft and connect shaft to stirring motor.

6.2 Preheat and regulate bath to maintain test temperature.

6.3 Pour approximately 40 mL each of sample and emulsifying agent into graduated cylinder. Immerse cylinder in bath to 85 mL mark, and allow contents of cylinder to reach test temperature.

6.4 Lower paddle into cylinder so that bottom of paddle is approximately 0.64 cm (1/4 in) from bottom of cylinder. Start stirring motor and allow it to rotate paddle at approximately 1500 rpm for 300 " 15 sec.

6.5 Stop motor, raise paddle from cylinder, and remove emulsion from paddle with stick or rubber policeman. Return as much of the emulsion as possible to cylinder.

6.6 Allow emulsion to remain undisturbed at the test temperature for the time required by the specification.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Examine emulsion for any separation, and report whether end product conforms to specification.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - AR - 1986

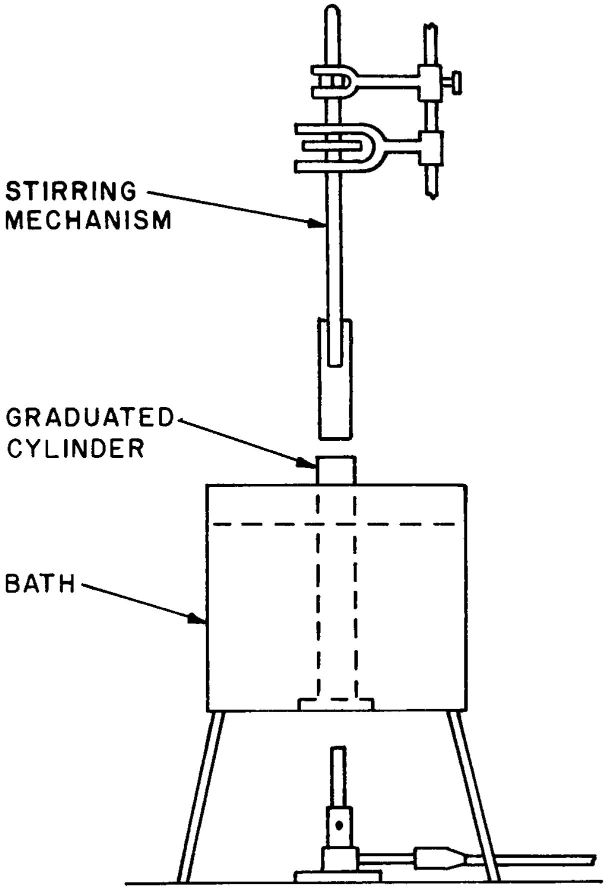


FIGURE I. Stirring mechanism.

Method 3205.3
September 30,1986

EMULSION (SOLUBLE CUTTING OILS)

1. SCOPE

1.1 This method is used for determining the emulsifying tendency of soluble cutting oils.

2. SUMMARY

2.1 The test consists of shaking a mixture of sample oil and water, allowing the mixture to stand, and examining for evidence of separation and for air bubbles.

3. SAMPLE SIZE

3.1 Approximately 5 mL of oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Sample bottle, 120 mL (4 oz), with stopper.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Distilled water.

6. PROCEDURE

6.1 Pour 95 mL of distilled water into sample bottle and add 5 mL of test oil. Shake vigorously for 1 min.

6.2 Let bottle stand for 1 h at 21° to 32°C (70° to 90°F). At end of this period, examine for evidence of separation.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the sample as acceptable if no separation is apparent and as unacceptable if separation occurs.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - AR - 1986

Method 3403.2
September 30, 1986

COMPATIBILITY OF TURBINE LUBRICATING OILS

1. SCOPE

1.1 This method is used for determining the compatibility of aircraft turbine lubricants with specific referee lubricants.

2. SUMMARY

2.1 This method consists essentially of preparing mixtures of the sample lubricant and referee lubricant in three concentrations, maintaining the mixtures at a fixed temperature for 7 days, and then centrifuging them for ten-minute intervals until the volumes of sediment becomes constant.

3. SAMPLE SIZE

3.1 Approximately 300 mL of the aircraft turbine lubricant to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Oven, gravity-convection, 105° . 1°C (221° . 2°F) (to accommodate three 250-mL flasks).

4.2 Erlenmeyer flasks, (3), Pyrex, 250-mL with glass stoppers.

4.3 Centrifuge tubes, cone-shaped, 100 mL (see ASTM D 2273).

4.4 Centrifuge, capable of spinning two or more centrifuge tubes, and applying a relative centrifugal force of 600 to 700 at the tips of the tubes.

4.5 Bath, hot water, 93° . 3°C (200° . 5°F) (for simultaneous heating of centrifuge tubes to be spun).

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Referee lubricant (in accordance with lubricant specification), 300 mL.

5.3 1,1,1-Trichloroethane (ASTM D 4126).

5.4 Cleaning solution, glass (concentrated sulfuric acid [2,0,2] saturated with potassium or sodium dichromate [1,0,1], technical grade).

6. PROCEDURE

6.1 Clean all flasks and centrifuge tubes as follows:

a. Thoroughly rinse with 1,1,1-trichloroethane in hood, and allow to air dry in hood.

NOTE 1: Caution. 1,1,1-Trichloroethane is toxic. Do not breathe its fumes nor allow it to come in contact with the skin.

b. Immerse in glass cleaning solution for at least 2 h.
c. Rinse first with tap water, then with distilled water, and dry in an oven 105°C (220°F).

6.2 Prepare three 200-mL mixtures, in 250-mL Erlenmeyer flasks, using the following proportions:

a. Sample, 180 mL; referee lubricant, 20 mL.
b. Sample, 100 mL; referee lubricant, 100 mL.
c. Sample, 20 mL; referee lubricant, 180 mL.

6.3 Stopper with a cork stopper, and identify each flask.

6.4 Manually shake each flask vigorously for 1 min.

6.5 Loosen the stoppers, and heat the flasks in an oven for 168 h at 105° ± 3°C (221° ± 5°F).

6.6 After the 168-hour period, remove the flasks, and allow them to cool to room temperature.

6.7 Manually shake each flask (one at a time) vigorously 1 min, and immediately fill two clean centrifuge tubes to the 100 mL mark with its contents. Identify the contents of each tube.

NOTE 2: Do not fill more pairs of tubes than can be spun by the centrifuge at one time. Allow the remaining filled flasks to stand until the first set of tubes have been completely centrifuged.

6.8 Process the centrifuge tubes as follows:

a. Heat the tubes for 5 min in a hot-water bath at 93° ± 3°C (200° ± 5°F).

b. Mount the tubes opposite each other in the centrifuge, being careful to locate the tubes to achieve proper balance of the centrifuge.
c. Turn on the centrifuge, and increase the speed until the relative centrifugal force at the tips of the tubes is 600 to 700. Run the centrifuge for 10 min.
d. After the ten-minute period, record the amount of sediment in each tube to the nearest 0.001 mL.
e. Repeat the 10 min centrifuging and sediment readings until the amount of sediment remains constant for three consecutive readings.

7. CALCULATIONS

7.1 Compute the average sediment volume to the nearest 0.001 mL of each tube pair.

8. REPORTING

8.1 Report the average volume of sediment in each pair of centrifuge tubes.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Air Force - AFWAL/POSL - 1986

Method 3410.1
September 30, 1986

HIGH TEMPERATURE DEPOSIT AND OIL DEGRADATION CHARACTERISTICS OF AVIATION TURBINE OILS

1. SCOPE

1.1 This method describes procedures used to evaluate the thermal stability characteristics of turbojet lubricants under varying levels of severity.

2. SUMMARY

2.1 Sample of the lubricant is subjected to one of the severity levels (type 1, 1-1/2, or 2) for a controlled number of hours. At periodic intervals during the test and at the end of test, samples of the oil are examined for both physical and chemical changes when compared to the sample of new oil. A visual inspection is made at the end of test of the accumulated deposits on the bearing and bearing housing. A weighted numerical rating system is used for rating individual areas as well as for obtaining an overall rating. Additional data on relative sludge forming tendencies on oils are obtained by weighing a 100-mesh filter element and a 40-mesh filter element.

3. SAMPLE SIZE

4. REFERENCES, STANDARDS AND APPARATUS

4.1 The apparatus used in this method is the Erdco high temperature bearing head mounted on a suitable drive test stand, similar to the Erdco universal test stand. It is not mandatory to use the "universal drive" to power the bearing. The support oil system and the test oil system are separate units and of special design for the Erdco high temperature bearing head.

4.1.1 Erdco high temperature bearing head. The bearing head is divided inter-nally into two main sections. The front or test section with its separate test oil system houses the unshielded 100-mm straight roller test bearing. The temperature of the test bearing is controlled by supplying heat to the outer race of the bearing. The outer race is secured in a bearing mount assembly containing wound Calrod heating elements packed in aluminum oxide for even heat distribution. The rear or support section of the bearing head houses the externally loaded, main shaft support bearing, together with a separate oil system. The test and support sections of the modified bearing head are separated by a screw thread seal to prevent mixing of the test and support oil. Details covering the bearing head and screw thread seal assembly are shown in Figure 1.

4.1.1.1 Test oil jet. A standard No. 60 drill size jet located at 12 o'clock on a 0.13335-m (5.250-in) diameter should be used. The size of the jet should be checked with a No. 60 drill and a visual check of axial straightness made with the drill inserted in the jet. Corrections for orifice size and axial straightness should be made by silver soldering the orifice end of the jet tube and redrilling.

4.1.2 Test bearing. An unshielded 0.10-m straight roller bearing is mounted on the free end of the shaft in the test oil compartment of the bearing head. The test bearing is loaded radially by the slight downward displacement of the free end of the shaft caused by a straight downward force from the loading piston acting on the outer race of the main support ball bearing shown in Figure 1. The test bearing is lubricated by a single jet located at 12 o'clock which supplies essentially a solid stream of oil midway between the I.D. of the outer race and the O.D. of the retainer on the top or unloaded position of the test bearing.

4.1.3 Test bearing mount. The bearing mount is made with an outer race removal feature which permits the removal of the bearing outer race for cleaning and inspection without disturbing the aluminum oxide packing. Three spot-type thermocouples are located 120° apart (radially around the I.D. of the bearing mount and indicate the skin temperature on the periphery of the outer race, which is pressed into contact with these thermocouple junctions when it is installed in the bearing mount. One of these three thermocouple leads is connected to a differentiating type temperature controller (proportional controller) or to a suitable autotransformer. "ON-OFF" type control is not recommended.

4.1.3.1 Three thermocouple junctions are silver-soldered to the outer-race housing. The soldering operation should leave enough excess metal to provide good contact with the outer race when it is pressed into the mount. Each of the three thermocouple leads are routed out directly through the bearing housing or end cover at 120° intervals.

4.1.3.2 Thermocouple packing. Teflon or an equivalent should be used for all thermocouple leads where they pass through the housing or end cover fitting. This also applies to the two Calrods where they pass out through the end cover.

4.1.4 Test oil system. A schematic of the test oil system is shown in Figure 2. The test oil cycle starts at the tank where three 1500-W, series connected heater elements (refer to 4.1.7) maintain the desired temperature by means of a differentiating type temperature controller or a suitable autotransformer. "ON-OFF" type control is not recommended. The test oil is then pumped from the tank to the bearing head where it passes through a No. 60 drill size orifice and lubricates the test bearing. The test oil is drained from the bearing head at three locations. The front drain handles splash oil which does not pass through the test bearing, whereas the rear drain collects only oil that has gone through the bearing. The middle drain is looped into a 360° configuration to slow oil flow and lessen air entrainment in the oil. The three drains go into a sump from which the test oil is pumped back into the top of the tank.

4.1.4.1 The test oil system provides for the installation of a fine 100-mesh filter screen (stainless steel, Erdco P/N 1RB 2006 S.S. for Type 1-1/2 test conditions) in the pressure line only just ahead of the jet to collect the sludge particles circulated in the test oil system. Pressure taps are located before and after the fine strainer in order to monitor the degree of screen plugging.

4.1.4.2 Heat loss from the test oil system has been minimized by placing a 0.6-cm (1/4-in) insulating box around the test oil tank. For screening tests at 149°C (300°F) oil-in temperature, no other insulation is usually used. However, at 176°C (350°F) and 204°C (400°F) oil in temperature, the test oil lines are wrapped, if necessary, with 2.5-cm (1-in) wide insulating tape. In addition, insulating covers are placed over the test oil pressure pump housing. The amount of insulation required is fixed for any individual system by the oil-in temperature requirements for each test. For all types of operation the test oil tank is vented to atmosphere, although plant exhaust suction systems are permissible as long as suction pressures do not exceed 12.7 cm (5.0 in) of water. Normally, the test tank vapor space is slightly pressurized (0 to 0.76 cm [0 to 0.3 in] Hg) due to back pressure from vapor condensers in the vent line.

4.1.4.2.1 Only stainless steel tubing or pipe lines and fittings (including sump below head) should be used in the test oil system. No insulation is required on scavenge lines but is required on pressure lines and pump housings, as necessary to maintain the test oil temperature conditions; however, the test oil in the thermocouple tee should be well insulated at all times. The tee should be located not more than 7.6 cm (3 in) from end cover and should be large enough to provide an oil annulus around the thermocouple. The tip of the thermocouple should be located in the center of the tee. A 40-mesh filter screen (stainless steel, Erdco P/N 1RB 2007 S.S. for type 1-1/2 test conditions) should be used in the test oil scavenge filter housing which should be located between the bearing head and scavenge pump.

4.1.4.3 Test oil tank.

4.1.4.3.1 Overall configuration. The overall configuration and general construction details of the test oil tank should conform to the detailed Erdco Part No. 01RB2160. The tank and baffle is constructed from AMS 5504C material; (AISI 410) 12.5% chromium steel sheet and welding rod. Baffle plate height should be checked and adjusted if necessary to insure that the top of the plate is 12.1 cm (4-3/4 in) from the botom of the tank. The top of the stirrer propeller is to be even with the top of the baffle plate as determined by a straight edge.

4.1.4.3.2 Cover gasket material. A Teflon sheet gasket or equivalent cut to match the outside flange dimensions should be used between the cover and mounting flange around the top of the tank.

4.1.4.3.3 Stirrer and stirrer mounting. A Lightning Model L 1/30 hp motor having a no-load (in air) shaft speed of 1800 rpm should be used in conjunction with a 5.1 cm (2.0 in) diameter, three-blade propeller.

4.1.5 Air system. Saturated air is metered to the test oil compartment in the bearing head. The air supply for the test bearing compartment should be shop air connected in the following sequence; air supply to pressure regulator to flow control valve to rotameter to water saturator to water separator tank to end cover fitting on bearing head. The air directed through the end cover (at about 11 o'clock or 1 o'clock on the same diameter as the test oil jet) into the bearing compartment is used primarily to control the pressure drop across the seal so as to insure against oil transfer from the support section into the test oil section. A 1/4- H 1/8-in tubing to pipe fitting should be used for the metered air supply to the rig.

4.1.5.1 Air to the test head is metered through a rotameter. Prior to each run, the rotameter is calibrated against a wet test gas meter for a flow of 9.90 L/min (0.35 CFM). The procedure is to disconnect the air supply line at the fitting where the air enters the head and then connect the supply line to a wet test gas meter which in turn discharges to atmosphere. A mercury manometer is located immediately upstream from the rotameter. The settings of the manometer and rotameter are observed and recorded for a wet test gas meter reading of 9.90 L/min (0.35 CFM). The air line is then reconnected to the bearing head and the previously observed manometer and rotameter settings are maintained throughout the test.

4.1.5.2 Seal (test support). The screw-type seal (P/N IRBB-6204) should be vented to the test cell at both top and the botom of the seal housing connections.

4.1.5.3 Connections for seal differential pressure measurement. The lines to the manometer used to measure the differential pressure across the seal should be connected to top, front of the bearing head, and the rear vertical surface of the bearing housing between 11 and 1 o'clock 2.5 cm (1 in) above the junction of the rear case with the main housing. After startup there should be no venting of the manometer lines. In this connection, the pipe plug in the top of the gear box, if used, should be removed and replaced with a small valve to insure atmospheric pressure in the gear box and hence in the support system.

4.1.6 Fenwall thermostatic control switch (Cat. No. 17802-510 or equivalent) (-18° to 315°C [0° to 600° F] normally closed). This switch should have a stainless steel sheath and be located in end of tank opposite stirrer motor 10.8 cm (4-1/4 in) from bottom and on the center line of the tank.

4.1.7 Heater unit. A Chromalox tubular heater unit (MT0345) having a rating of 4500 W, rated at 115-120 V should be series connected to a 220 V supply and controlled by means of a differentiating type temperature controller or a suitable auto-transformer. Three thermocouples installed 31.8 cm (12-1/2 in) from terminal end of tank should be used. All thermocouple lead wire should be of the Ceramo, a metal-sheathed type. Test oil heater thermocouples are to be attached to the tubular heater by clamps, welded shoes, or spot weld.

4.2 Graduated cylinder, borosilicate, 1000 mL.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials refer-ences and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Lubricating oil, qualified under MIL-L-23699.

5.3 Dry cleaning solvent, (A-A-59601D).

5.4 No. 400 emery paper.

5.5 No. 30 grit aluminum oxide.

5.6 Bi-solvent; isopropyl alcohol:acetone - 1:1 [1,3,0].

6. PROCEDURE

6.1 Cleaning procedure.

6.1.1 Test oil tank. The test oil tank should be removed from the stand at the end of each test. After the deposits have been rated and photographed the tank assembly should be completely disassembled. The interior of the tank and the immersed part of the heater should each be thoroughly cleaned by solvents, hand scrubbing, and or mild abrasive blast to remove all traces of deposits from previous test. An electrical continuity check should be made on the immersion heaters before the tank is reinstalled on the stand. Instances of apparent electrical "shorts" in the immersion heater circuit can often be cured by additional air or oven drying.

6.1.2 Test oil pressure and scavenge pumps. The test oil pressure and scavenge pumps are to be completely disassembled and cleaned to remove all deposits from the previous test.

6.1.3 Oil-out sump. The oil sump located directly below the bearing head should be disassembled and cleaned with bisolvent followed by ultrasonic cleaning in an oakite/water mixture, if necessary. Rinse in bisolvent and air dry.

6.1.4 Test oil lines. All of the straight run test oil lines and fittings (elbows, tees) should be removed from the test oil system and hand reamed with wire brushes having bristles of steel or other metal except copper in a bath of cleaning solvent. Oil lines with bends greater than 10° should be new at the start of each test.

6.1.5 Oil filter assemblies. All oil filter housings in the test and support oil systems should be removed and wire brushed in cleaning solvent. A soft fiber brush is usually adequate in scrubbing the filter elements after a short soaking in a solvent bath.

6.1.6 Gage lines. The oil lines connected to gages on the instrument console require cleaning with air after each test.

6.1.7 Bearing head. The bearing head should be disassembled as far back as the seal and all of the removable parts, except the test bearing, are to be thoroughly cleaned by scouring with No. 400 emery paper, or glass beads (vacu-blasting), and solvent to remove all traces of deposits from the previous test. In cases of extreme dirtiness, the entire seal assembly should be removed and the parts solvent cleaned using No. 400 emery paper if necessary.

6.1.8 Test bearing. For qualification tests a new 10.0-cm test bearing should be used for each 100-hour endurance run. The new bearing should be cleaned in solvent and dipped in test oil prior to installing.

6.2 Bearing head assembly.

6.2.1 Screw thread seal. In installing the seal (P/N IRBB 6204) the right hand threads on the O.D. of the seal must be toward the front (test compartment) end of the rig. Reversing the seal position will cause the test and support oil to be whirled toward the center of the seal by the rotating action of the shaft and enhance the possibility of mixing the two oils.

6.2.2 Test bearing heater mount. The bearing outer race should be lightly pressed into the bearing heater mount with the serial number facing the front of the rig. The recommended fit on the bearing outer race is -0.000508 to +0.00178 cm (-0.0002 to +0.0007 in). On repeated usage, the I.D. of the bearing mount may become worn and result in a loose fit which might tend to cock the outer race. It may also result in erroneous temperature readings of the bearing outer race. When this happens, the I.D. of the mount should be chrome plated and brought back to proper dimension or that section of the mount replaced.

<u>NOTE:</u> The heater mount must be disassembled and repacked with No. 30 grit aluminum oxide after each test.

6.2.2.1 Reassembly of heater mount. Prior to assembly of the heater mount, the heater coils should be centered and checked to insure that no point of the coils comes within 0.318 cm (1/8 in) of the inside surfaces of the front and rear mount plates. A method of accomplishing this is to assemble the unit without retainer ring; this allows visual inspection of the location of the heater- coils within the assembly. Areas where the coils come within 0.318 cm (1/8 in) of either front or rear inner faces are marked. During final assembly, transite spacers 0.32 H 0.64 H 1.3 cm (1/8 H 1/4 x 1/2 in) are placed at these marked points. In reassembling the heater mount, care should be exercised to insure that the front and rear vertical faces are parallel to each ther and square with the I.D. of the mount. If these conditions are not maintained, the concentricity of the bearing outer race in relation to the axis of the shaft may be affected. Before installing the heater mount in the rig, the bearing outer race thermocouples should be checked for continuity.

6.2.3 Test bearing. Before installing the test bearing on the hub, it should be examined visually for any obvious mechanical defects. If the bearing appears satisfactory, the inner race and cage assembly should be pressed onto the hub with the serial number facing the front of the rig. The recommended fit of the bearing inner race on the hub is 0.0020 to 0.0036 cm (0.0008 to 0.0014 in) tight. After repeated usage the O.D. of the bearing hub may become worn and result in a loose bearing fit. When this happens, the hub should be chrome plated back to proper dimension or replaced. After the bearing has been installed in the bearing head, turn the shaft by hand to make sure it turns freely. Bearing roughness can usually be detected in this manner. When properly installed the rollers should be evenly centered in the outer race. If the rollers are more than 0.32 cm (1/8 in) off center on the outer race, check for missing shaft assembly parts or an improperly installed bearing outer race. It is important that the lock nut on the test bearing retainer be tight since it imparts the axial pinch on all of the shaft assembly parts back to the load bearing and in addition helps to prevent the bearing inner race from turning on the hub during operation.

6.2.4 End cover. Before installing the end cover on the rig, check the test oil jet tube alignment to make sure it is aimed midway between the test bearing outer race and the cage. A gasket, made from material to withstand high temperatures, should be installed under the end cover.

6.2.5 Bench check of seal operation. After assembly of the bearing head, the screw thread seal operation should be checked under a 2240-N (500-lb) radial load to the test bearing by applying 352 gage kPa (51 psig) shop air pressure to the loading cylinder and hand rotating the shaft to detect rubbing. If any rubbing occurs, the bearing head should be disassembled and a slight amount of material removed from either the screw threads or the seal plate, depending on the location and severity of the rubbing. The material is removed from the seal plate if the rubbing is localized, and the screw thread is ground if the rubbing is severe and occurs over a considerable area. On a new build, the seal plate may be coated with Prussian blue to determine the area of contact. On later rebuilds, the rubbing can usually be detected without the use of the dye. The seal should be checked for rubbing after every build.

6.2.6 Miscellaneous joint compounds. No pipe dopes, thread compounds, or gasket sealants should be used in the assembly of the rig or test oil system. Teflon tape (no adhesive) is an effective sealant when applied to pipe threads.

6.3 Rig installation and stand checks. After assembly, the bearing head is installed on the stand. The test oil tank is filled with 7.6 L (2 gal) of the oil to be tested. This oil is heated to 121°C (250°F) in the tank and then circulated for 20 min through the test oil system, including the bearing head. The oil is then drained from the tank and the bottom sections of the two oil filter housings. Fresh test oil is then added to the tank until the fluid level is 1.3 cm (1/2 in) above the baffle plate with the test oil pressure and scavenge lines filled in preparation for the test.

6.3.1 A qualified MIL-L-23699 or MIL-PRF-7808 lubricating oil is used in the support system and is not normally changed after each run. Drain periods on the support oil are governed by the amount of deterioration as determined by laboratory tests and the degree of the filter plugging in the support oil system.

6.4 Critical test items. Before starting a test, care should be taken that critical items such as dimensional tolerances and heater electrical connections conform to those listed in Table II.

TABLE I. Operating conditions for different severity levelsof Erdco high temperature bearing test.

Condition	Type		
	1	1-1/2	2
Test oil-in temperature, °C	149° " 2.5°	177° " 2.5°	204° " 2.5°
Test tank bulk oil temperature, °C	171° " 2.5°	199° " 2.5°	227° " 2.5°
Test bearing temperature -max., °C	260° " 2.5°	260° " 2.5°	260° " 2.5°
Air flow to end cover, L/min	9.9 " 1.4	9.9 " 1.4	9.9 " 1.4
Test oil-in temperature, °F	300° " 5°	350° " 5°	400° " 5°
Test tank bulk oil temperature, °F	340° " 5°	390° " 5°	440° " 5°
Test bearing temperature -max., °F	500° " 5°	500° " 5°	500° " 5°
Air flow to end cover, CFM	0.35 " 0.05	0.35 " 0.05	0.35 " 0.05

TABLE II. Critical test items.

	Minimum, cm	Maximum, cm	Minimum, in	Maximum, in
1. Dimensional tolerances				
a. Test bearing outer race fit in heater mount	-0.00051	0	-0.0002	0
b. Test bearing inner race fit in heater mount	-0.0020		-0.0008	
c. Axial run-out of front vertical face of testbearing outer raceassembled in rig	0.000		0.000	
d. Screw type seal	0.046		0.018	
2. Electrical connections				
a. Test bearing heater. This is a single wrapped 4700-W unit which should be connected to 220-V power source.				
b. Test oil tank immersion heater. This unit is made up of three 1,500-W heaters which should be series-connected to a 220-V power source.				

6.5 Operating conditions.

6.5.1 Throughout the entire test the variables for different test severities (types 1, 1-1/2, and 2) as shown in Table I should be controlled within the limits specified.

6.5.2 Initial test oil jet calibration. To calibrate the test oil jet, the test tank should be heated until bulk oil temperature is about 149°C (300°F). Turn on air to the end cover. Then the test oil and support oil pumps are turned on, and the rig speed increased to 10,000 rpm. The rig speed should be brought up immediately after oil circulation is started as the screw thread seal depends on relative motion for its sealing action. The previous setting on the test oil pressure pump or a minimum of 69 gage kPa (10 psig) oil pressure at the jet should be used to insure against possible oil starvation during this initial startup. The pressure drop across the seal is maintained at 0.8 to 1.5 cm (0.3 to 0.6 in) of test oil by making fine adjustments to the gear box vent valve. Under these conditions of air flow, the pressure in the test compartment should be 0.25-0.51 cm (0.1-0.2 in) of test oil above rig room pressure conditions. As soon as a 121°C (250°F) oil temperature at the jet has stabilized, a minimum of two separate flow checks of one-minute duration each should be made at the three-way valve position on the pressure side of the scavenge pump. The oil pressure at the jet should be adjusted using the variable speed drive unit on the test oil pump until the jet oil flow is 600 mL/min. Once the flow rate of 600 mL/min is established for an oil, the flow at this condition is maintained constant during the test.

6.5.3 After the oil jet calibration step, the following conditions are allowed to stabilize:

Oil-in temperature	121° C	250° F
Oil flow rate	600 " 30 mL/min	600 " 30 mL/min
Bearing speed	10,000 " 100 rpm	10,000 " 100 rpm
Radial load	352 gage kPa (2220 N)	51 psig (500 lb)
Support oil-in temperature	71°-82° C	160°-180° F
Support oil pressure	552-689 gage kPa	80-100 psig
Gear box oil pressure	172-207 gage kPa	25-30 psig
Air flow to end cover	9.9 "1.4 L/min	0.35 " 0.05 cfm
Pressure drop across seal	0.8-1.5 cm of test oil (test compartment higher)	0.3-0.6 in. of test oil (test compartment higher)

6.5.3.1 After the foregoing conditions stabilize, the run is continued until the test bearing temperature from the highest reading thermocouple levels off (about one hour). This temperature is then noted and used as an indication of the mechanical condition of the bearing. Previous tests on bearing stabilization at a 121° C (250° F) oil-in temperature to the jet indicate that a temperature range of 149° C to 177° C (300° F to 350° F) is normal for a properly functioning 100-mm Erdco test bearing. Higher stabilization temperatures indicate the test bearing condition is questionable and the bearing should probably be replaced before continuing with the test.

6.5.4 Once the test bearing has reached a satisfactory stabilized temperature, the temperature setting on the temperature controller on the test tank should be increased to raise bulk oil temperatures to about 28° C (50°F) higher than the desired temperature of the oil at the jet (149°C [300°F] for the test of type 1 lubricants, 177°C [350°F] for the type 1-1/2 lubricants, and 204°C [400°F] for the test of type 2 lubricants). In order to reduce the length of warmup time required to reach the desired test oil jet temperature, the Calrod heater should be turned on simultaneously with the change in test tank setting to bring the bearing outer race temperature (max) to 260°C (500°F). At this point, the three thermocouples attached to the bearing outer race should be checked to make sure that the thermocouple indicating the second highest temperature is attached to the Temperature Controller. Improper hookup of the bearing thermocouples will result in poor control of test bearing temperatures.

6.5.5 When the desired test conditions shown in Table I are reached, the test oil flow rate is again checked twice and adjusted if necessary to obtain 600 mL/min flow at the three-way valve position in the scavenge line. Then the 100-hour endurance test is begun. These data are recorded on a multi-channel strip chart continuously during the course of the test.

6.5.6 Test cycle. The 100-hour endurance test is run so that there is at least a seven-hour shutdown period out of every 24 h of operation. Except for emergency stops, all shutdowns should be made in accordance with the detailed procedure as outlined in 6.6.4.Warmup time following shutdown periods is considered part of the 100-hour endurance test time.

6.5.7 Used oil analysis. The following laboratory tests are run on the new and used oil samples:

Test	ASTM method	50 mL samples
Viscosity at:		
37.8°C (100°F), cs	D 445	X
98.9°C (210°F), cs	D 445	X
Neutralization No.	D 664	X

6.5.8 Test oil flow rate checks. During operation the test oil flow rate should be checked at the three-way valve in the scavenge line twice every start-up. For convenience, a 1000-mL, borosilicate glass graduated cylinder should be used to collect the test oil during the flow check. The volume of oil collected in a one- or two-minute period should check the original volume flow in mL/min within "5.0%. In order to prevent too large a drop in test oil level in the tank during flow checks, a maximum continuous flow period should be 2 min. The hot test oil collected in the glass graduate should be dumped back into the test tank after each flow check. With the friction type variable speed drive unit on the test oil pressure pump, it is not possible to obtain a non-slip drive on the pump after oil pressures reach about 276 gage kPa (40 psig). At this point the test oil will usually have become so viscous it can no longer be effectively pumped and the test should be considered ended.

6.5.9 At the completion of the 100-hour endurance test, the test bearing is restabilized at 121°C (250°F) oil-in temperature to determine the relative change in stabilized temperatures compared to the results obtained at the start of the test. These data can be used to determine the change in frictional characteristics of the bearing resulting from metal wear or excessive oil deposits.

6.5.10 The rig is then shut down in accordance with the shutdown procedure described in 6.6.4.

6.5.11 When the system cools to room temperature, the tank and test oil system should be drained into a suitable container so that the volume of test oil remaining can be determined. After the volume of test oil has been measured and recorded, 3.8 L (1 gal) of the test oil should be poured into a clean container and saved for laboratory analyses.

6.5.12 Rig photographs. As soon as possible, the rig should be disassembled. Although color is preferred, black-and-white photographs, measuring at least 7.6 H l0.2 cm (3 H 4 in) or slides should then be taken of the following parts:

a. Bearing compartment with cover removed - front view.
b. Heater mount - rear side.
c. Cover plate - rear side.
d. Seal plate in housing - heater and bearing removed.
e. Test bearing - inner race, rollers and cage assembly.
f. Test bearing - outer race, inside diameter.
g. Test oil tank - baffle plate.
h. Test oil tank - baffle removed.

6.5.13 After the photographs are completed, a more detailed visual inspection is made of these parts to determine the average depth, coverage, consistency, and type of oil deposits, plus the extent of abnormal metal wear on the test bearing cage, rollers, roller pockets, and outer race. Rate deposits according to 6.7.

6.6 PROCEDURES

6.6.1 Flushing procedure.

1. Install a fine mesh (100 mesh) filter at the inlet of the spray nozzle and a coarse mesh (40 mesh) filter at the inlet of the scavenge pump. It is not necessary to preweigh the filters.
2. The same type of oil is used to flush the system as used in the test. Service the test oil tank with 7570 mL (2 gal) of test oil.
3. Insure that the tank is vented by means of an exhauster/blower arrangement and that the vent is open.
4. Insure that the test oil supply valve at the inlet side of the pressure pump is closed.
5. The watt-hour meter reading for the test oil heater should be recorded before the flush is begun.
6. At the control panel turn on the following switches in the order shown:
   a. Control panel power.
   b. Support test oil cart power.
   c. Stirrer.
   d. Timer.
   e. Test oil heater.

   f. Strip chart recorder.
   g. Flowmeter timer.
   h. Doric.
7. Provide full power (220 V) to the test oil heater.
8. When the temperature of the test oil in the tank exceeds 121°C (250°F) turn on the pressure and scavenge pumps. Immediately open the test oil supply valve (on the inlet side of the pressure pump) to allow oil to draw to the pressure pump. The pump speed should be maintained such that the pressure is a minimum of 138 gage kPa (20 psig) and the flow is 600 " 30 mL/min.
9. Adjust the test oil heater as needed to maintain a temperature of 121°C (250°F) in the test oil tank.
10. While the oil is circulating draw two samples of approximately 700 mL each and check for debris. If the samples are clean, return them to the system. If the samples contain debris, drain the oil and begin the flush again.
11. Circulate the oil for at least 20 min and then turn off the tank heater.
12. When the test oil tank temperature has cooled to 38°C (100°F), close the oil supply valve on the inlet side of the pressure pump.
13. Turn off the following switches:
   a. Pumps
   b. Timer.
   c. Stirrer.
   d. Recorder.
   e. Flowmeter.
   f. Doric.
   g. Support test oil cart power.
   h. Control panel power.

14. Drain the test oil tank. Take two samples of the oil (20 and 60 mL). Mark the samples and the remaining oil:

Flush oil
B.R. Test
Test #
Oil Code (PE number)
Date

15. After removing the two flush filters, clean and store them.

6.6.2 Preparation for test.

1. Update the strip chart recorder with the date, test number and oil code.
2. Install two preweighed filters. A fine mesh (100 mesh) filter is installed on the spray nozzle inlet and a coarse mesh (40 mesh) filter is installed on the inlet line of the scavenge pump.

3. Close the oil supply valve. Check the oil level in the test oil and support oil tanks. The test oil tank should be charged with test oil at room temperature until the fluid level is 1.3 cm (1/2 in) above the baffle plate with the pressure and scavenge lines filled. This level should be marked and will establish the "FILL" mark or the minimum operating level during test operation. The expansion of the test oil upon heating to test conditions will establish the "FULL" mark. The support oil tank should contain approximately 23 to 30 L (6 to 8 gal) of a MIL-L-23699 or MIL-PRF-7808 lubricating oil.
4. Insure that the test oil tank vent is open and that any vent line drain is closed.
5. Check the main air supply pressure, adjusting it if necessary to maintain a minimum of 207 gage kPa (30 psig).
6. Open water valves to dynamic and support oil cooler.
7. Check dynamic water pressure: it should be 310 gage kPa (45 psig).
8. Adjust the air pressure for the load cylinder to 207 gage kPa (30 psig).
9. Set the load valve to the zero load position (clockwise direction).
10. Add distilled water to the vapor bottle until the desired level is reached.
11. Open air bleeds on both sides of the differential manometer for the screw thread oil seals which should be filled with test oil.
12. Record the watt hour meter readings from the test oil tank heater and bearing heater meters. Turn on the electric power (breaker boxes) to the test oil tank heater, support oil tank heater, test bearing heater, and the bearing drive motor.
13. Turn off automatic mode switch.
14. Turn on the following switches on the control panel:
    a. Control panel power.
    b. Strip chart recorder.
    c. Support test oil cart power.
    d. Stirrer motor.
    e. Timer (adjust as necessary).
    f. Test oil heater.
    g. Doric.
    h. Water heater.
    i. Load by-pass.

15. When the test oil temperature reaches 121°C (250°F) turn on the support oil heater. The temperature controllers should be adjusted to obtain 138°C (280°F) and 82°C (175°F) in the test oil and support oil tanks, respectively. In the test oil tank, the maximum heater skin temperature should not exceed 29°C (85°F) above the test oil tank target temperature during warmup. Higher temperatures indicate improper electrical hookup of excessively high supply voltage which should be corrected before continuing the test program.
16. When the bulk oil temperature in the test tank reaches 138°C (280°F) turn on the air supply to the end cover and adjust to approximately 9.9 L/min (0.35 ft^3/min). Air pressure should be held at 34.4 gage kPa (5.0 psig).
17. Start the test oil pressure and scavenge pumps. Insure that the flow is 69 gage kPa (10 psig) minimum at the test oil jet. The test oil flowrate is 600 " 30 mL/min.
18. Check the oil supply valve between the test oil tank and the variable speed pressure pump; it should be wide open.

19. Start the support oil pumps; there should be at least 414 gage kPa (60 psig) manifold pressure. At the screw thread seal there should be at least 0.8 cm (0.3 in) of oil positive pressure relative to the support oil side (manometer leg to test compartment lower).
20. Set and maintain the support oil pressure at 552 to 689 gage kPa (80 to 100 psig) by means of a regulating valve.
21. Allow oil temperature at the support scavenge line to reach at 38.8°C least (100°F).
22. Start the bearing drive motor; the bearing speed is preset at 10,000 rpm. (If bearing drive is not constant speed, follow motor operating instructions to bring it up to the necessary speed.)
23. Turn off the load by-pass switch.
24. Apply a load to the test bearing. Increase the load on the bearing gradually with the load controls. The load cylinder pressure should be held at 352 gage kPa (51 psig).
25. Continue running until the following conditions have been stabilized:

Test oil-in temperature	121° " 2.5°C	(250° " 5°F)
Test tank bulk oil temperature	138° " 2.5°C	(280° " 5°F)
Air flow, end cover	9.9 " 1.4 L/min (corrected)	(0.35 " 0.05 ft^3/min) (corrected)

26. Make at least two separate flow checks of one-minute duration each at the three-way valve position on the pressure side of the scavenge pump. The measured volume flow rate should be 600 " 30 mL/min. The hot test oil collected during the flow check should be poured immediately back into the test oil tank through the fill port and the port recapped.
27. Continue running for one hour at these conditions with the bearing heater off. If during or at the end of this period, the maximum bearing temperature has exceeded 177°C (350°F), shut down the rig, install a new test bearing, and repeat items 1 through 26. If the maximum bearing temperature has not exceeded 177°C (350°F) during this period, proceed with the next step.
28. Turn on the test bearing heater and adjust indicator flag to the target temperature on the temperature controller.
29. Check the three test bearing outer-race thermocouples to make sure that the second highest indicating thermocouple is connected to the temperature controller. Maintain 260°C (500°F) on the highest indicating thermocouple
30. Adjust the test oil tank thermal switch so that the target temperature for the type of test being run is met within 30 minutes (minimum) and 60 minutes (maximum). Maximum heater skin temperature should not exceed 29°C (85°F) above test oil target temperature during the warmup period. The nozzle temperature should be maintained at 177° " 2.5°C (350° " 5°F).

Test type	1	1-1/2	2
Target temperature	171° " 2.5°C (340° " 5°F)	199° " 2.5°C (390° " 5°F)	227° " 2.5°C (440° " 5°F)

Oil-in temperature	149° " 2.5°C (300° " 5°F)	177° " 2.5°C (350° " 5°F)	204° " 2.5°C (400° " 5°F)

31. Continue running until the conditions of Table I for the type of test requested have stabilized.
32. Take at least two separate flow checks of one-minute duration at each of the three-way valve position on the pressure side of the scavenge pump. Flow rate should be 600 " 30 mL/min. The hot test oil collected during the flow check should be poured immediately back into the test oil tank through the fill port and the port recapped.

33. Continue endurance testing. At the completion of the 100-hour endurance test, the test bearing is to be restabilized at 121°C (250°F) oil-in temperature to determine the relative change in stabilized temperatures compared to the results obtained at the start of the test. 6.6.3 Daily startup sequence.

1. Update strip chart recorder.
2. Check oil level through visual sight gauge. Add enough oil so that the bottom of the test oil meniscus is even with the mark that was made in step 3 of procedure 6.6.2. Open vent drain valve and remove all oil from the vent line.
3. Repeat steps 4 to 23 for startup procedure.
4. Turn on test bearing heater and adjust as necessary to reach 260° "2.5°C (500° " 5°F) on the highest reading thermocouple.
5. Continue running until the conditions have stabilized for the type of test being run as shown in Table I.
6. Make at least two separate flow checks of one-minute duration each at the three-way valve position on the pressure side of the scavange pump. The measured volume flow rate should be 600 " 30 mL/min.

6.6.4 Daily running procedure.

1. Monitor the strip chart recorder for maintenance of test temperatures within correct operating ranges. Monitor gauges and flowmeters as well.
2. Take two samples of test oil (20 and 60 mL) from the three-way valve in the scavenge line or with an automated system every 10 h (including warmup time along with endurance time). The first test sample should be fresh test oil before the oil is introduced into the system. If the 10-hour sampling period happens to fall during a shutdown period, take the samples just before shutdown or after first reaching type 1, 1-1/2, or 2 test conditions from next startup of rig. Mark each sample:

   B.R Test
   Test #
   Oil Code
   Date
   Hour of Sample

3. Makeup oil should be added immediately after each 80 mL sample is taken by adding sufficient quantity of unused test oil to the tank to bring the oil level in the visual sight gage to the full mark.
4. The test oil filter changes should be made after shutting down on the first day. If the pressure drop across the 100-mesh filter exceeds 21 gage kPa (3 psig) during operation, the rig should be shut down in accordance with the shutdown procedure under 6.6.5, and a new weighed filter reinstalled. The dirty 100-mesh filter element and the 40-mesh filter element are to be weighed and the amount of the weight gain recorded. Prior to weighing, the 100- and 40-mesh filter screens should be allowed to drain for 1 h at room temperature.
5. Actual running time should conform to the following conditions:
    a. 17-hour maximum test time between shutdowns.
    b. Minimum shutdown period of 7 h.
    c. Minimum daily running time of 6 h.

6.6.5 Shutdown procedure.

1. Shut off bearing and tank heaters.
2. Open air regulated loading valve on console and reduce loading pressure to 172 gage kPa (25 psig).
3. Continue running at these conditions until test bearing temperature (maximum) drops to 204°C (400°F).
4. Reduce load completely and shut off drive motor.
5. When the dynamatic output shaft stops turning, shut off test pumps, support pumps and heat, test tank stirrer, main electrical and water switches, and all air valves.

6.6.6 Post-stabilization run.

1. This procedure is done in place of the last shutdown.
2. Repeat steps 1 through 3 of the shutdown procedure.

6.6.5 Shutdown procedure.

1. Shut off bearing and tank heaters.
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3. Continue running at these conditions until test bearing temperature (maximum) drops to 204°C (400°F).
4. Reduce load completely and shut off drive motor.
5. When the dynamatic output shaft stops turning, shut off test pumps, support pumps and heat, test tank stirrer, main electrical and water switches, and all air valves.

6.6.6 Post-stabilization run.

1. This procedure is done in place of the last shutdown.

2. Repeat steps 1 through 3 of the shutdown procedure
3. Reduce load on bearing completely.
4. Allow the test oil tank temperature and the oil-in temperature to reach 138°C (280°F) and 121°C (250°F) respectively. Maintain these temperatures.
5. When the test temperatures stabilize, run for one hour and monitor the maximum temperature of the test bearing.
6. Record the highest bearing temperature.
7. Shut off bearing drive motor.
8. Repeat step 5 of the shutdown procedure.

6.7 Demerit system for rating deposits in Erdco bearing machine.

6.7.1 Outline of method.

6.7.1.1 Cleanliness demerits. Bearing head cleanliness shall be reported in this demerit system by the assignment of values of 0 to 20 to the inspection results, as follows:

0 shall designate a new or thoroughly clean condition.
20 shall represent the worst condition that could be expected.

6.7.1.2 Table III shows the numerical demerits to be assigned to different types and degrees of deposits:

TABLE III. Deposit rating demerit scale

Type of deposit	Degree	Demerit values
Flaked carbon	Heavy	20
	Moderate	19
	Light	18
Blistered carbon	Heavy	17
	Moderate	16
	Light	15
Crinkled	Heavy	14
	Moderate	13
	Light	12
Smooth carbon	Heavy	11
	Moderate	10
	Light	9
Sludge	Heavy	8
	Moderate	7
	Light	6
Varnish	Heavy	5
	Moderate	3
	Light	1

Clean		0

All demerits shall be reported to the nearest whole number

6.7.1.3 Table IV defines deposit types and degrees.

TABLE IV. Description of bearing deposit rating terms.

Type of deposit	Degree	Description
Flaked Carbon		Carbonaceous coating that cannot be removed by wiping with a rag. Broken blisters, peeling
	Heavy	Over 0.12 cm (3/64-in) in thickness.
	Moderate	Ranges from 0.04 cm (1/64-in) to less than 0.12 cm (3/64-in) in thickness.
	Light	Ranges from just covering to less than 0.04 cm (1/64-in) in thickness.
Blistered carbon		Same as for flaked carbon, blistered, bubbled.
	Heavy	Same as for flaked carbon.
	Moderate	Same as for flaked carbon.
	Light	Same as for flaked carbon.
Crinkled carbon		Same as for flaked carbon, ridged, not smooth.
	Heavy	Same as for flaked carbon.
	Moderate	Same as for flaked carbon.
	Light	Same as for flaked carbon.
Smooth carbon		Same as for flaked carbon, ridged, not smooth.
	Heavy	Same as for flaked carbon.
	Moderate	Same as for flaked carbon.
	Light	Same as for flaked carbon.
Smooth carbon		Same as for flaked carbon, smooth coating.
	Heavy	Same as for flaked carbon.
	Moderate	Same as for flaked carbon.
	Light	Same as for flaked carbon.
Sludge		Shiny, oily emulsion of carbon and oil. Can be wiped off with a rag.
	Heavy	Approximately 0.42 cm (1/16-in) thick.
	Moderate	Approximately 0.08 cm (1/32 in) thick.
	Light	Just covering.
Varnish		Varnish or lacquer-like coating shiny.
Clean		Clean - no deposit.

6.7.2 Computation of overall rating of the Erdco high temperature bearing head.

6.7.2.1 An area demerit shall be determined in accordance with the area covered by the deposits as follows:

$$\frac{\textit{Percent area covered}}{\textit{10}} = \textit{Area demerit}$$

6.7.2.2 A rating shall be obtained by multiplying the area demerit by the demerit value assigned in Table III. The rating for each item shall be modified by multiplying by the weight factor assigned by Table V which will yield a demerit rating.

6.7.2.2.1 Bearing rating and demerits are determined from the following categories:

Rollers 1. face
2. rear
3. front
Cage 4. between rollers
5. rear
6. front
Outer ring 7. path
8. rear
9. front
Sum of
Ratings x 5 (Weight Factor)
11
Demerit for Bearing
Inner ring 10. rear
11. front

6.7.2.3 The total bearing head deposit rating shall be the sum of the demerits for each item of Table V.

TABLE V. Bearing head deposit weight factor rating.

No.	Item		Weight factor	Demerit
1.	End cover	Multiply rating by	1	=
2.	Spacer and nut	Multiply rating by	2	=
3.	Heater-front	Multiply rating by	3	=
4.	Heater-rear	Multiply rating by	3	=
5.	Seal plate	Multiply rating by	3	=
6.	Bearing	Multiply rating by	5	=

6.7.2.4 The overall rating of the Erdco high temperature bearing head shall be the total bearing head deposit rating divided by 6.

6.7.2.5 An example of the calculations described is shown in Table VI.

TABLE VI. Example of detailed Rating

Item	Description	Area demerit 6.7.2.1	Demerit Scale 6.7.1.2	Rating 6.7.2.2	Factor 6.7.2.2 Table III	Demerit
End cover	Clean	1	0	0	1	0
Space and Nut	10% L. sludge	1	6	6		
	90% M.	9	3	27	2	66
	Varnish			= 33		
Heater –front	25% Clean	2.5	0	0		
	25% L. Varnish	2.5	1	2.5		
	25%L. Crinkled	2.5	12	30	3	195
	carbon					
	25% M.	2.5	13	32.5		
	Crinkled carbon			= 65		
Heater- Rear	10% L varnish	1	1	1		
	10% M varnish	1	3	3		
	10% H varnish	1	5	5	3	195
	20% M sludge	2	7	14		
	505 L smooth	5	9	45		
	carbon			=68		
Seal-plate	5% M varnish	0.5	3	1.5		
	95% clean	9.5	0	0	1	1.5
				=1.5		
Bearing:						
a. Rollers:						
(1) Face	100% M	10	3	30		
(2) Rear	varnish	5	1	5		
(3) Front	50% L. varnish	5	3	15		
	50% M varnish			=50		
b. Cage:						
(4)Between		10	0	0		
rollers	100%clean					
(5)Rear			0	0		
	50%clean	5	6	30		
(6)Front	50%L sludge	5	0	0		
	50%clean	5	6	30		
	50% L sludge	5		=60		
c. Outer						
ring:	75% clean	7.5	0	0		
(7) path	25% M varnish	2.5	3	7.5		
(8) Rear	30%M varnish	3	3	9		

	40%H varnish	4	5	20		
	30% L smooth carbon	3	9	27		
(9) front	20% clean	2	0	0		
	40%M varnish	4	3	12		
	40%H varnish	4	5	20		
				= 95.5		
d.Inner ring:						
(10) Rear	50% L varnish	5	1	5		
	50% M sludge	5	7	35		
(11)Front	50% L varnish	5	1	5		
	50% L crinkled carbon	5	12	60		
				=105		
Bearing(total)				310.5/11 =28.25		141 = 607.5
Overall ratings (Average of items 1,2,3,4 and 6)				607.6 / 6 =101.2		

7. CALCULATION

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 As required by the specification.

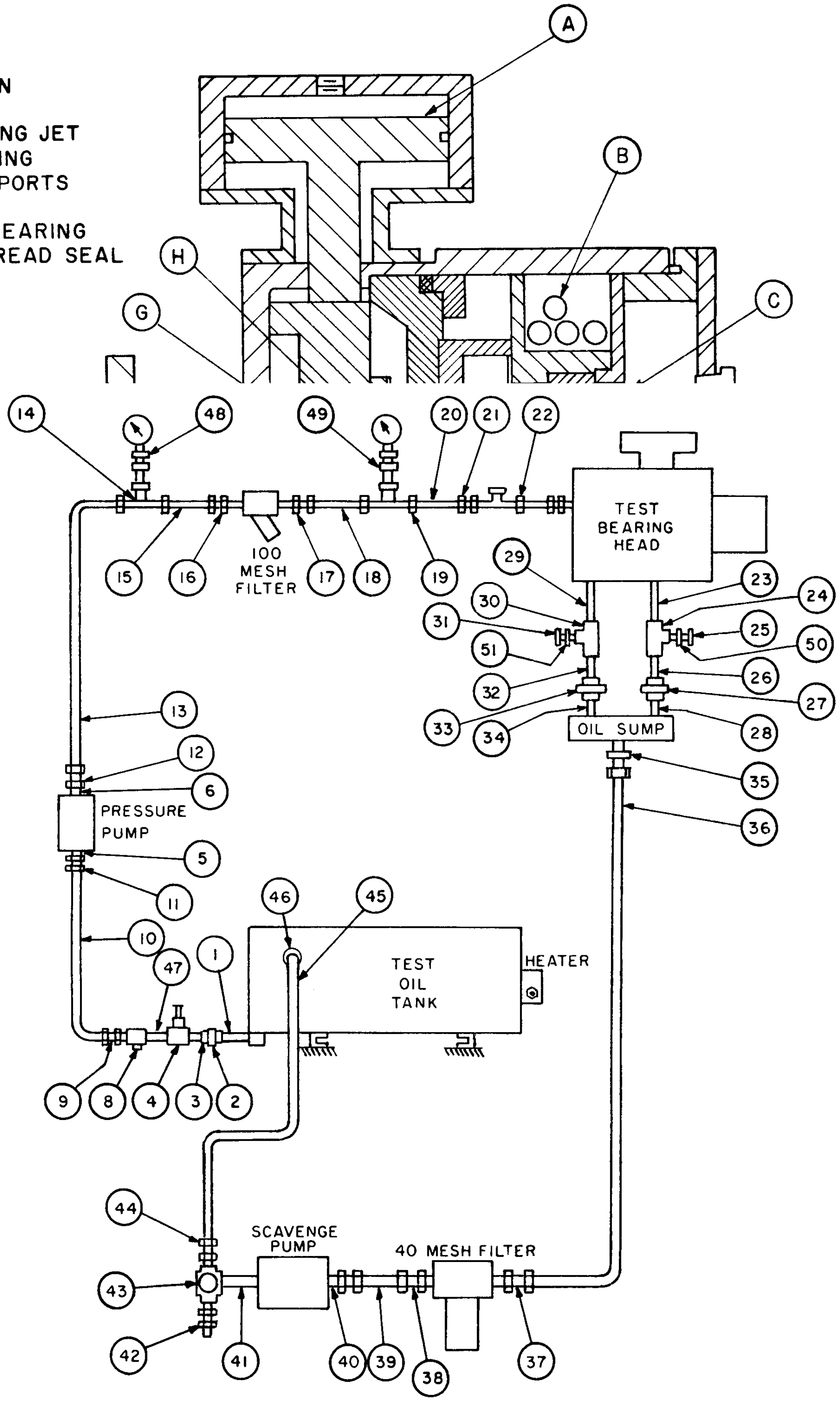
9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Navy - NAPC – 1986

A. LOAD PISTON
B. HEATER
C. TEST BEARING JET
D. TEST BEARING
E. SCAVENGE PORTS
F. SHAFT
G. SUPPORT BEARING
H. SCREW THREAD SEAL



NOTE:
(SEE NOTES ON NEXT PAGE).

FIGURE 2. Schematic of test oil system.

Notes on Figure 2:

1. 3/8 pipe nipple - 1-1/2 long
2. 3/8 pipe union
3. 3/8 pipe nipple - 1-1/2 long
4. 3/8 valve (iron)
5. 3/4 by 3/8 reducer
6. 3/4 by 3/8 reducer
7. omitted
8. 3/8 pipe tee
9. 3/8 tube H 3/8 npt-male
10. 3/8 tubing
11. 3/8 tube H 3/8 npt-male
12. 3/8 tube H 3/8
13. 1/2 tube
14. 1/4 tube tee
15. 1/4 tube
16. 1/4 tube H 3/8 npt-male
17. 1/4 tube H 3/8 npt-male
18. 1/4 tube
19. 1/4 tube tee
20. 1/4 tube
21. 1/4 tube H 1/8 npt-female
22. 3/16 tube H 1/8 npt-male tee (branch T/C modified)
23. 3/8 pipe nipple - 1-1/2 long
24. 3/8 pipe tee
25. 3/16 tube H 1/8 npt-male (T/C modified)
26. 3/8 pipe nipple - 1-1/2 long
27. 3/8 pipe union
28. 3/8 pipe nipple - 1-1/2 long
29. 3/8 pipe nipple - 1-1/2 long
30. 3/8 pipe tee
31. 3/16 tube H 1/8 npt-male (T/C modified)
32. 3/8 pipe nipple - 1-1/2 long
33. 3/8 pipe union
34. 3/8 pipe union - 1-1/2 long
35. 1/2 tube H 1/2 npt-male
36. 1/2 tubing
37. 1/2 tube H 3/8 npt-male
38. 1/2 tube H 3/8 npt-male
39. 1/2 tube1. 3/8 pipe nipple - 1-1/2 long
40. 1/2 tube H 1/2 npt-male
41. 1/2 pipe npl H 3 long
42. 1/2 tube H 1/2 npt-male
43. 1/2 three way valve (iron)
44. 1/2 tube H 1/2 npt-male
45. 1/2 tube
46. 1/2 tube H 3/8 npt-male
47. 3/8 pipe npt - 1-1/2 long
48. 1/4 tube bulkhead connector (steel)
49. 1/4 tube bulkhead connector
50. 3/8 by 1/8 reducing bushing
51. 3/8 by 1/8 reducing bushing

Method 3411.1
September 30, 1986

THERMAL STABILITY AND CORROSIVITY OF AIRCRAFT TURBINE ENGINE LUBRICANTS

1. SCOPE

1.1 This method describes a procedure for determining the thermal stability and corrosiveness of aircraft turbine lubricants in an environment free of air and moisture. The catalytic effects of mild steel on the decomposition of the fluids and the corrosion of the steel by the thermal degradation products of the lubricant are measured.

2. SUMMARY

2.1 A weighed mild steel specimen is placed in a glass thermal stability test cell with a measured amount of test lubricant. Air and moisture are removed from the test cell by heating and shaking under vacuum prior to sealing the tube. The sealed test cell is then heated at a given temperature for a specified period of time. The stability of the lubricant is assessed by determining the change in viscosity, total acid number and by the appearance of the residual lubricant at the end of the heating period. The steel specimen is weighed and examined under a microscope to determine changes due to corrosion.

3. SAMPLE SIZE

3.1 Approximately 10 mL for each steel corrosion test.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Test cell – A borosilicate glass test cell having the dimensions given in Figure l is recommended as the sample container.

4.2 Heating bath, 9.5-L (10-qt) capacity, (manufactured by Sta-Warm Electric, Ravena, Ohio or equivalent). An aluminum block may be used in place of the heating bath if so desired. The resistance wire-heated aluminum block must have a suitable number of openings of the proper diameter (approximately 2.0 cm) and depth (approximately 21 cm) so that the sealed test cell is completely immersed in the heater. The temperature of the aluminum block or heating bath shall be capable of being controlled within . 1°C (. 2°F) at the test temperature. The temperature gradient between oil sample and the aluminum block or bath shall be kept within 1°C (2°F).

4.3 Test cell holder – When a heating bath is used, a heavy-wall steel sample tube holder of the proper size to contain the test cell shall be provided for safety reason.

4.4 Becton, Dickinson and Company Yale Luer-Lock (20YL), or equal 20 mL hypodermic syringe with a No. 14 square-tip needle approximately 20 cm (8 in) long.

4.5. Vacuum pump, capable of reducing the pressure to at least 10 µm of mercury or less.

4.6 McLeod gauge or other suitable pressure measuring device.

4.7 Water bath, capable of maintaining a temperature of 80° ± 3°C (175° ± 5°F) and sufficient size to contain the test cell.

4.8 Laboratory safety shield, constructed of either safety glass or wire reinforced plastic.

4.9 Balance, sensitivity 0.1 mg.

4.10 Microscope, 20X magnification.

4.11 Forceps, stainless steel.

5. MATERIALS

5.1 CAUTION – SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Toluene, reagent-grade [2,3,0].

5.3 Acetone, reagent-grade [1,3,0].

5.4 Nitric-sulfuric acid mixture, approximately 1:10 [3,0,2].

5.5 Abrasive paper, silicon carbide, 240 grit.

5.6 Steel specimens of approximately 0.15 × 0.7 × 7.5 cm made from QQ-S-698 mild steel shall be used.

6. PROCEDURE

6.1 Definitions

6.1.1 Thermal stability is the resistance to permanent changes in properties of the lubricant caused solely by heat.

6.1.2 Corrosion is any surface damage of the steel specimen resulting in a weight change.

6.2 Preparation of apparatus.

6.2.1 Cutting test cells - The cells are cut in two, either on an emery cutoff wheel or by circumscribing the tube with a hot wire about 17 cm from the bottom.

NOTE 1: Circumscribing with a hot wire is preferred because it is cleaner and safer.

6.2.2 Cleaning test cell - Clean the divided test cell by scrubbing with a suitable detergent, rinsing in tap water, heating in hot nitric-sulfuric acid mixture, rinsing with distilled water and drying in an oven at 105°C (225°F) for 1 h. The cells can be stored in a desiccator until used.

6.2.3 Preheat and adjust aluminum block or heating bath temperature to 274° ± 1°C (525° ± 2°F).

6.2.4 Preparation of steel specimens.

6.2.4.1 Cut samples approximately 0.15 × 0.7 × 7.5 cm.

6.2.4.2 Remove pits and other imperfections on a fine grinding wheel.

6.2.4.3 Polish with 240 grit silicon carbide paper to remove all surface blemishes from both sides and all four edges.

NOTE 2: In all succeeding steps, handle the steel specimens with filter paper or forceps.

6.2.4.4 Clean all metal dust from the samples by rubbing vigorously with lint-free tissue.

6.2.4.5 Wash sample strips in warm toluene, then in warm acetone and allow to air dry.

6.2.4.6 Weigh each specimen to within 0.1 mg.

6.3 Test procedure.

6.3.1 Duplicate tests are conducted.

6.3.2 A freshly prepared steel specimen is placed in the lower half of the cleaned cell, which is then fused to the top half.

NOTE 3: Proper annealing of the fused glass cell will prevent rupture and leaks during testing.

6.3.3 Introduce 6 mL of the oil samples below the capillary end of the test cell with the aid of the hypodermic syringe.With this sample volume the metal strip is only partially immersed in the oil leaving approximately 1.3 cm (1/2 in) exposed.

6.3.4 Connect the fused test cell containing the oil sample and metal specimen to the vacuum pump. This can be done with vacuum tubing connected through a Kjeldahl connecting bulb, T_s joint 24/40, and a No. 1 rubber stopper drilled to hold the thermal stability tube.

6.3.5 Mount the test cell, while still connected to the operating vacuum pump, in a water bath maintained at 80° ± 3°C (175° ± 5°F). Degas the cell and its contents while shaking gently for 20 min. A Thomas-Boerner oscillating platform shaking apparatus may be used. Mounting the tubes about 16 cm above the platform. A manifold can be used to evacuate more than one cell at a time. If frothing persists, continue the heating and shaking for another 10 min.

NOTE 4: Certain ester based as well as other type synthetic lubricants are hydrolytically unstable at the test temperature of 274°C (525°F). Therefore, the procedure described in 6.3.5 to remove all water before sealing the cell is necessary. The pressure in the cell, as measured with a McLeod gauge, or other suitable measuring device, shall be no more than 10 μm before sealing.

6.3.6 While the test cell is still under vacuum it is sealed and drawn off at the capillary approximately 3 cm above where it joins the cell body. (See Note 3).

6.3.7 Weigh the test cell to the nearest mg.

6.3.8 Insert the glass test cell in the heater [preheated to 274° ± 1°C (525° ± 2°F)] for period of 96 hours. If a heating bath is used, insert the test cell in the steel holder and place the assembly in the bath.

NOTE 5: Safety shields must always be used as a precaution when placing the test cells in the heater, removing the cells from the heater, and opening the cells. Tremendous pressures are generated during the test period.

6.3.9 At the conclusion of the heating period, remove the test cell and allow to cool to ambient temperature. Wipe down the outside of the cell with suitable solvent.

6.3.10 Visually observe the samples for evidence of insolubles, phase separation, interactions between metal and oil, or changes in color of the oil. A significant difference in final color between duplicate tests will necessitate retesting since color differences indicate improper preparation of one or both of the test cells.

6.3.11 Reweigh the test cell. Any significant loss in weight indicates leakage of the test cell and the test must be repeated.

6.3.12 As a safety measure the cells must be cooled before cutting them open because of the pressure which develops in testing. Any convenient cooling method may be used such as dry ice - acetone, liquid nitrogen, cooling chambers, etc.

6.3.13 Remove the oil sample and steel specimen by carefully breaking the test cell. This may be accomplished by scribing a mark around the test cell near the top and applying a hot wire to the scratch mark. (See Note 5).

6.3.14 Remove the steel specimen with forceps, wash with a stream of toluene from a wash bottle to remove oil then dip in two successive warm toluene washes and a final dip in warm acetone. Wipe with lint-free tissue and air dry. Weigh to the nearest 0.1 mg. Using a 20X microscope, carefully examine each steel specimen and note any evidence of pitting or etching either above or below the liquid level.

6.3.15 Inspect the tube for insolubles and gum deposits.

6.3.16 Determine the viscosity at 37.8°C (100°F) of both the original lubricant and the lubricant after exposure to heat in accordance with ASTM D 445.

6.3.17 Determine the neutralization number on both the original and heated lubricant in accordance with ASTM D 664.

7. CALCULATIONS

7.1

$$Percent\ viscosity\ change = \frac{V_2 - V_1}{V_1} * 100$$

where:
V_1 - viscosity, Initial
V_2 - viscosity after test

7.2 $Total\ Acid\ Number\ change = N_2 - N_1$

where:
N_1 - Total Acid Number, Initial
N_2 - Total Acid Number after test

7.3 $Change\ in\ weight\ of\ the\ steel\ specimen\ in\ mg/cm^2 = \frac{B - A}{12.96}$

where:

B - Metal weight before test in mg
A - Metal weight after test in mg

8. REPORTING

8.1 The report shall include the following:

8.1.1 Visual appearance of test cell, insolubles and gum deposits.

8.1.2 Visual appearance of lubricant after test.

8.1.3 Visual appearance of steel specimen above and below the liquid level.

8.1.4 Change in the neutralization number of the lubricant sample.

8.1.5 Percentage change in viscosity at 37.8°C (100°F).

8.1.6 Change in weight of the steel specimen in mg/cm^2.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Navy - NAPC-1986

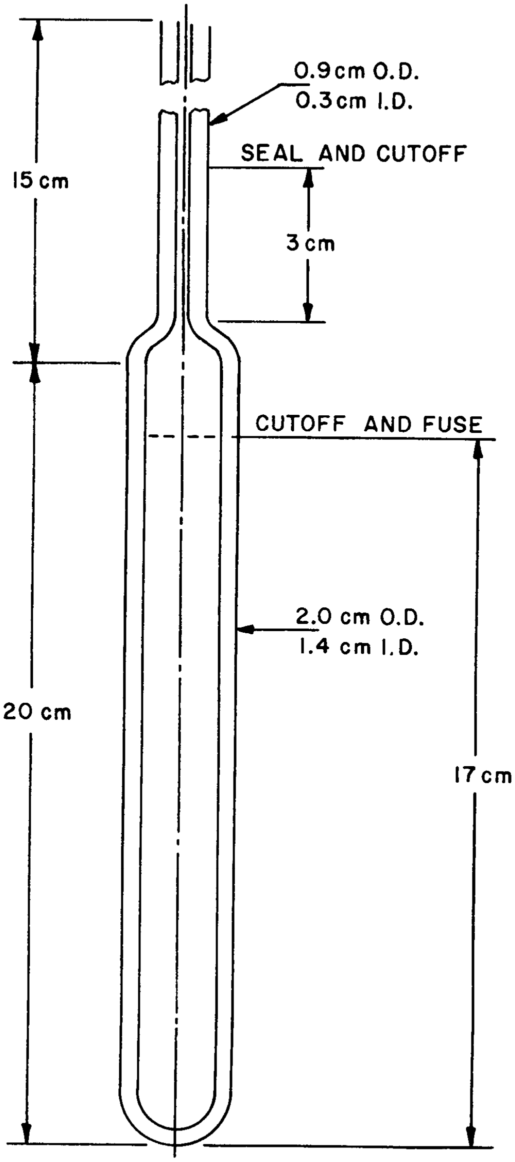


FIGURE 1. Thermal stability-corrosion test cell.

Method 3430.2
September 30, 1986

COMPATIBILITY CHARACTERISTICS OF UNIVERSAL GEAR LUBRICANTS

1. SCOPE

1.1 This method is used for determining the compatibility of a universal gear lubricant when blended with a reference gear lubricant by observing for precipitation of additive material after storage.

2. SUMMARY

2.1 The test consists of mixing the reference gear and test gear lubricants, storing the mixture for 30 days, and centrifuging the mixture to determine the percentage of sludge present.

3. SAMPLE SIZE

3.1 Approximately 4 L (1 gal) of the gear oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 100 mL cone-shaped centrifuge tubes as in ASTM D 96.

4.2 Centrifuge with a diameter of swing (tip-to-tip of whirling tubes) 38.1 to 43.2 cm (15 to 17 in) and shall be capable of being controlled at a speed of 1500 " 25 rpm.

NOTE 1: If the available centrifuge does not conform dimensionally to the preferred form, the speed of rotation of the available centrifuge must be adjusted to give the same centrifugal force at the tips of the tubes as that obtained with the prescribed instrument when operated at 1500 " 25 rpm. The speed to operate the available centrifuge shall be calculated from the formula.

$$rpm = \sqrt{y} * 1500$$

where:
y = The 40.7 divided by the diameter in cm of the swing (tip-to-tip of the whirling tubes) of the available centrifuge. Use 16 as the dividend when the diameter is measured in inches.

4.3 Balance capable of weighing to "1 mg.

4.4 Stirring apparatus capable of stirring the contents of a 400-mL tall-form beaker at approximately 200 rpm.

4.5 Beakers, 400-mL capacity, tall form, heat resistant glass.

4.6 Forced circulation oven capable of being controlled at 104° " 2.5°C (220° " 5°F) and 121° " 1°C (250° " 2°F).

4.7 Desiccator capable of holding several centrifuge tubes.

4.8 Graduated cylinders, 250-mL capacity.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Naphtha, in accordance with TT-N-95B, type II [1,4,0]. Caution, Naphtha is flammable.

5.3 Cleaning solution, consisting of concentrated sulfuric acid saturated with potassium dichromate.

5.4 Distilled water.

5.5 Denatured ethyl alcohol.

5.6 Reference oils. Information concerning reference oils can be obtained from:

SAE
400 Commonwealth Drive
Warrendale, PA 15096-0001

6. PROCEDURE

6.1 Make two compatibility tests with each reference oil.

6.2 Determine the residue in each of the reference oils and the sample oil by subjecting each of the oils alone (not mixed with any other oil) to the procedures described in 6.7 through 6.15. This data will be used in 7.1.

6.3 Preparation of centrifuge tubes. Clean one centrifuge tube for each compatibility test to be made in the following manner:

a. Rinse tubes with naphtha.
b. Clean tubes with cleaning solution.
c. Rinse tubes with distilled water.
d. Rinse tubes with denatured ethyl alcohol.

6.4 Support the centrifuge tubes in an inverted position in an oven maintained at 121° ‟ 1°C (250° ‟ 2°F) for at least one-half hour.

6.5 Remove the tubes from the oven, place them in a desiccator, and permit them to cool to room temperature.

6.6 Number each tube, weigh to the nearest mg, and replace tubes in the desiccator until they are to be used.

6.7 Thoroughly shake sample and reference oils prior to sampling.

6.8 Place 110 ‟ 1 mL of a reference oil and sample oil in a 400-mL beaker using the same graduated cylinder each time the sample is measured, but a clean graduated cylinder for each of the reference oils.

6.9 Heat the beaker containing the reference oils and test oil mixture in an oven at 121° ‟ 1°C (250° ‟ 2°F) for 20 ± 1 min. Remove the beaker from the oven and stir (with a mechanical stirrer) the contents while still hot for 5 min.

6.10 Transfer 100 mL of the contents to the weighed centrifuge tubes.

6.11 Cork the centrifuge tubes and store them in an upright position in a darkened area such as a drawer or cupboard at room temperature for a period of 30 ‟ 1 days.

6.12 At the conclusion of the storage period, remove the centrifuge tubes from the storage area, place it in the centrifuge, and operate the centrifuge at 1500 ‟ 25 rpm for a period of 30 ‟ 1 min.

NOTE 2: In transferring the centrifuge tubes from the storage area to the centrifuge, care must be taken not to disturb any material which may have separated from the oil.

6.13 Remove the centrifuge tubes from the centrifuge and decant and discard the supernatant oil. Permit the centrifuge tubes to drain in an inverted position at room temperature for a period of 2 h. Discard the drainings. If the residue is a solid, wash it with naphtha a sufficient number of times to insure that it is free of oil. decanting is possible

NOTE 3: If the separated material is not sufficiently compacted by the centrifuging to permit decanting the supernatant oil, continue the centrifuging for 15-minute intervals until decanting is possible. If the material is a liquid at the conclusion of the 30-minute centrifuging period, or if it cannot be compacted, stopper the centrifuge tube and replace it in storage for an additional 30 days. At the end of the second storage period, proceed as directed in 6.12.

6.14 Place the centrifuge tube in an upright position in an oven controlled at 104° " 2.5°C (220° "5°F) for approximately 2 h.

6.15 Remove the centrifuge tube from the oven and place it in a desiccator to cool to room temperature. Weigh the tube and contents to the nearest mg. Subtract the weight of the empty centrifuge tube to determine the weight of the separated material.

NOTE 4: If the residue remains a liquid at the end of the second 30-day storage period, centrifuge the tube as specified in 6.12. Remove the tube from the centrifuge and note the volume of the separated liquids to the nearest 0.05 mL.

7. CALCULATIONS

7.1 Calculate the percent incompatibility for each test, using the following formulas:

$$\text{Theoretical zero incompatibility} = R + T$$

$$\text{Evidence of incompatibility} = X - (R + T)$$

$$\text{Percent incompatibility} = \frac{X - (R + T)}{0.9\ (50\,r + 50\,t)} * 100$$

where:

R = weight of separated material in reference oil (g/50 mL of oil). See 6.2.

T = weight of separated material in sample oil (g/50 mL) of oil). See 6.2

X = weight of separated material found in compatibility test. See 6.15.

r = weight percent of additive in reference oil. This information is supplied by the Test Monitoring Center. See 5.6

t = weight percent additive in sample oil. This information is supplied by the manufacturer of the sample oil.

0.9 = assumed specific gravity of both reference oil and sample.

7.2 Calculate and report incompatibility for each reference oil as an average of the two test values obtained for the reference oil.

NOTE 5: It may be of interest to make a chemical analysis of the residue found in the incompatible oil mixture.

8. REPORTING

8.1 Report the percent incompatibility of the test gear lubricants.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Air Force - AFWAL/POSL-1986

Method 3432.1
September 30, 1986

COMPATIBILITY OF ELASTOMERS WITH AIRCRAFT TURBINE LUBRICANTS

1. SCOPE

1.1 This method is used for determining effects of aircraft turbine lubricants on elastomers.

2. SUMMARY

2.1 It consists of determining the swelling effect, change in elongation, change in tensile strength and change in hardness of standard fluorocarbon and fluorosilicone elastomer specimens after being immersed in the sample for 72 h, at temperatures of 175°C (347°F) for fluorocarbon specimens and 150°C (302°F) for fluorosilicone specimens.

3. SAMPLE SIZE

3.1 Approximately 1000 mL of the lubricant to be tested.

4. APPARATUS

4.1 Bath; aluminum block or oil bath capable of maintaining sample temperature within 1°C (1.8°F) of required test temperature. Aluminum block baths will have 5.0 mm ± 1 cm in thickness of insulation. Oil baths will have a cover with openings for immersion of test tubes.

4.2 Test tubes, heat-resistant, 30 cm length and 3.9 cm outside diameter.

4.3 Hanger, specimen, made of stainless steel or nichrome wire.

4.4 Corks, standard size number 19.

4.5 ASTM Die C as described in ASTM D 412.

4.6 Bench Marker as described in ASTM D 412.

4.7 Micrometer as described in ASTM D 412.

4.8 Testing Machine for elongation and tensile strength measurements as described in ASTM D 412.

4.9 Grips as described in ASTM D 412.
4.10 Durometer, Type A Shore as described in ASTM D 2240.

4.11 Stamp Pad and Ink as described in ASTM D 412.

4.12 Punch, Leather, 0.159 cm (1/16 in) diameter.

4.13 Balance, sensitivity 0.1 mg, suitable for weighing in both air and water.

4.14 Tubing, Pyrex, 0.8 cm O.D.

4.15 Beaker, 400 mL.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Standard test rubber sheets, type F-A, conforming to U.S. Air Force Specification Bulletin 539.

5.3 Standard test rubber sheets, type FS, conforming to U.S. Air Force Specification Bulletin 539.

5.4 Cleaning solution, Turbo Alkaline Rust Remover or equivalent.

5.5 Dry cleaning solvent (A-A-59601D).

5.6 Alcohol, ethyl, ACS grade [0,3,0].

5.7 Acetone, ACS grade [1,3,0].

5.8 Distilled water.

5.9 Filter paper.

6. PROCEDURE

6.1 Clean all glassware as follows:

a. Rinse in dry cleaning solvent, and allow to air dry (see note 1).

NOTE 1: Caution. Dry cleaning solvent is both toxic and flammable. Do not breathe its fumes nor allow it to come in contact with the skin. Keep flames away from the dry cleaning solvent.

b. Soak in glass cleaning solution at a temperature of 71°C (160°F) for 1 h (minimum time), if glassware contains oil deposits from a previous test.
c. Wash glassware with laboratory detergent. Rinse thoroughly with tapwater and then with distilled water.
d. Dry in an oven at 100° to 105°C (212° to 221° F) and store in dust free location.

6.2 Clean suspension device and hooks thoroughly with dry cleaning solvent, wipe with a clean dry cloth, air dry and store in a dust free location.

6.3 Inspect the sheets of standard stock elastomer for flaws. Sections of an elastomeric sheet having a flaw will not be used for specimens. Cut four specimens from sheets of standard elastomer stock, with all sheets being from a single production batch, for each lubricant to be tested. Cut the specimens using ASTM Die C as described in method ASTM D 412. Punch a 0.159 cm (1/16 in) hole in each end of the cut elastomer specimens using a leather punch. The hole shall be centered and 0.64 cm (1/4 in) from the end of the specimen. Weigh each test specimen in air and in water in accordance with method ASTM D 471 (and record weights). Using a type A Shore Durometer obtain four Durometer readings (two on each large end) in accordance with method ASTM D 2240. Determine the initial hardness of each specimen by averaging the four readings.

6.4 Preheat and adjust immersion bath to maintain lubricant sample test temperature of 175° ± 1°C (347° ± 1.8°F) for F-A elastomers specimens and 150° ± 1°C (302° ± 1.8°F) for FS elastomer specimens. A suggested method of controlling these temperatures is to place a similar test tube containing 140 mL of lubricant in the bath at the same depth as the test lubricant. A thermocouple is then placed in this tube at a depth equal to the depth of the small section of the elastomeric specimens when suspended in the test lubricant tubes. The test tube containing the elastomeric specimens and test lubricant is placed into the bath to a depth which places the level of the test lubricant 4.1 cm (1-5/8 in) above the top of the heating medium and not from the top of the insulation of bath covering.

6.5 Immerse the elastomeric specimens in the heated test lubricant by means of a specimen hanger made from stainless steel or nichrome wire. The hanger shall have two hooks at one end permitting one elastomeric specimen to be hung from each hook. The two specimens shall hang parallel and 1.3 cm (1/2 in) apart. Attach the wire specimen hanger to a standard size number 19 cork and fit with two lengths of 0.8 cm Pyrex glass tubing (chimneys). Insert one chimney, 7.6 cm (3 in) in length, through the cork such that the chimney extends 1.3 cm (1/2 in) above the top of the cork. Insert the second chimney 12.7 cm (5 in) in length, through the cork such that the chimney extends 8.9 cm (3 1/2 in) above the top of the cork. Place the cork fitted with the chimneys, specimen hanger and specimens into the pre-heated test tube containing the test oil. Adjust the length of the specimen hanger so that when the cork is placed tightly into the test tube, the specimens will hang with equal distance of test lubricant above and below the specimens using 140 mL of test lubricant. Hang only two elastomeric specimens in one tube and adjust cork such that specimens do not contact the side of the test tube.

6.6 Remove the elastomeric specimens from the test tube after 72 h with the time starting when tubes are first inserted into the bath. Place the elastomeric specimens in a beaker of fresh test lubricant which is at room temperature and allow to cool for a minimum of 30 min. Remove the specimens from the cooling oil and place them into a beaker of acetone for a minimum of 14 and a maximum of 20 s. Dry the specimens with filter paper by blotting. Determine volume swell, hardness, elongation and tensile strength of the elastomeric specimens.

6.7 Determine a base line for elongation and tensile strength measurements for each batch of standard elastomer stock. Cut eight specimens in accordance with 6.3 with no more than two specimens being cut from any one sheet. Determine the elongation and tensile strength of each untreated specimen in accordance with method ASTM D 412 using a 2.5 cm (1 in) bench marker. Use the average of these values in determining elongation change and tensile strength change of the oil treated specimens.

6.8 Weigh each oil treated specimen in air and in water in accordance with method ASTM D 472 and calculate percent volume swell for each specimen. Calculate the percent volume swell of the test lubricant as the average of four individual volume swell determinations.

6.9 Determine hardness readings on the specimens as described in 6.3. Calculate hardness change for the test lubricant as the difference between the initial hardness and the final hardness.

6.10 Determine elongation and tensile strength of the four specimens in accordance with method ASTM D 412 using a 2.54-cm (1-in) bench marker. Average the four elongations measurements. Calculate the percent change in elongation, see 7.1.

6.11 Determine the tensile strength of the four specimens in accordance with method ASTM D 412 and average the four readings. Calculate the percentage change in tensile strength, see 7.2.

7. CALCULATIONS

$$Elongation, \%\ change = \frac{Final\ Elengation\ -\ Initial\ Elongation}{Initial\ Elongation} * 100$$

7.1 Calculate the percent change in elongation as follows:

7.2 Calculate the percent change in tensile strength as follows:

$$Tensile\ strength, \%\ change = \frac{Final\ Tensile\ Strength\ -\ Initial\ Tensile\ Strength}{Initial\ Tensile\ Strength} * 100$$

8. REPORTING

8.1 Report the percentage change in the elongation and tensile strength.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Air Force - AFWAL/POSL - 1986

Method 3433
September 30, 1986

COMPATIBILITY OF SYNTHETIC AIRCRAFT TURBINE LUBRICANTS WITH SILICONE RUBBER

1. SCOPE

1.1 This method is used for determining the compatibility of synthetic aircraft turbine lubricants with a standard silicone rubber composition representative of commercial seal materials. It determines the extent of deterioration of a silicone rubber standard by the lubricating fluids.

1.2 Advanced synthetic lubricant fluids for aircraft turbine engines with modified additive systems must be compatible with materials of construction used in aircraft engines particularly with silicone rubber seals. This test was devised to reveal rubber seal-lubricating fluid compatibility problem areas.

2. SUMMARY

2.1 This method provides a procedure for exposing silicone rubber test specimens to the influence of synthetic lubricants under definite conditions of temperature and time for purpose of measuring the resulting deterioration by noting the changes in physical properties before and after immersion in the test liquid.

3. SAMPLE SIZE

3.1 Approximately 500 mL of the lubricant to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 References.

4.1.1 U.S. Air Force Specification bulletin 539, Standard Elastomer Stocks.

4.2 Apparatus

4.2.1 Oven, gravity convection, 121° ± 1°C (250° ± 2°F).

4.2.2 Beakers, tall-form, heat resistant, 600 mL.

4.2.3 Suspension device, glass, for holding test rubber specimens

4.2.4 Hook, stainless steel, for suspending test rubber sheets from balance.

4.2.5 Balance, sensitivity, . 0.1 mg.

4.2.6 Dies and cutters - ASTM die A or equivalent so that width of rubber specimen suspended between jaws of tensile tester is 1.27 cm (1/2 in).

4.2.7 Durometer - Standard shore durometer or equivalent.

4.2.8 Micrometer - To measure thickness of rubber.

4.2.9 Tension testing machines - Scott tensile tester model L or equivalent for rubber testing.

4.2.10 Borosilicate tubing - thermocouple holder closed at one end, 18 cm length □ 0.3 cm inside diameter □ 0.5 cm outside diameter.

4.2.11 Thermocouple and recorder - to record temperature of test oils.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Standard silicone test rubber sheets, may be obtained from Naval Air Propulsion Test Center, Trenton, New Jersey 08628. Test sheets should be 15 □ 15 cm (6 □ 6 in) by 0.19 cm thick (0.075 in) as per U.S. Air Force Specification bulletin 539, Standard Elastomer Stocks.

5.3 Cleaning solution, glass (concentrated sulfuric acid [3,0,2] saturated with potassium or sodium dichromate [1,0,1]).

5.4 Methyl alcohol, ACS, particle-free [1,3,0].

5.5 Solvent - naphtha [1,4,0] or toluene [2,3,0] ACS.

5.6 Acetone, ACS, particle free [1,3,0].

6. PROCEDURE

6.1 Definitions

6.1.1 Tensile stress - the applied force per unit of original cross sectional area of specimen.

6.2 Test specimen preparation

6.2.1 Test specimens are to be cut using ASTM die A or equivalent.

6.2.2 Straight specimens may be prepared where it is not practical to cut ASTM dumbbell specimens. The minimum length must be 10.2 cm (4 in). The width should be 1.27 cm (1/2 in) between the jaws which are 2.54 cm (1 in) apart.

6.3 Test procedure

6.3.1 Volume swell, change in hardness and change in tensile strength are all determined relative to original properties, from the post-test condition of the same specimens. The original tensile strength and hardness shall be determined, using duplicate specimens cut from the same sheet from which the test specimens were obtained. The duplicate specimens should be cut from an area of the sheet which is immediately adjacent to the area from which the test specimens were cut. The original rubber specimen volume is determined from the test specimen prior to immersion in the oil sample.

6.4 Preparation

6.4.1 Clean all glassware.

6.4.1.1 Rinse in solvent, rinse with acetone and air dry.

6.4.1.2 Soak in glass cleaning solution for 2 h (minimum).

6.4.1.3 Rinse thoroughly with tap water, and then with distilled water.

6.4.1.4 Dry in oven at 100°C (212°F) and store in dust-free location.

6.4.2 Preheat and adjust oven to maintain a temperature of 121° ± 1°C (250° ± 2°F).

6.4.3 Prepare standard test silicone rubber specimens.

6.4.3.1 Do not buff.

6.4.3.2 Cut three pieces (per 6.2) from the sheet. Pieces must be cut with the grain of the rubber as indicated by two grain marks at the top of the slab.

6.4.3.3 Punch a 0.3175-cm (1/8-in) diameter hole at the center of the 10.16-cm (4-in) side, about 1.905 cm (3/4 in) from the top edge.

6.4.3.4 Mark each piece for identification by cutting small pieces from one or more corners.

6.4.3.5 Attach the wire suspension device to each piece.

6.4.4 Determine original volume of silicone test specimens.

6.4.4.1 Weigh each rubber test specimen and its hook in air to the nearest 0.1 mg by suspending it from the balance by its hook. Record the weights.

6.4.4.2 Reweigh (to the nearest 0.1 mg) each sheet while suspended in distilled water at room temperature, being careful to remove all air bubbles from the sheet and prevent the sheet from touching the water container. Record the weights.

6.4.4.3 Determine (and record) the displacement weight of each piece by subtracting the weight in water from the weight in air.

6.5 Testing.

6.5.1 Fill a 600-mL beaker with 500 mL of the oil sample.

6.5.2 Attach the rubber specimen to the suspension device, and submerge the sheets in the sample so that the top edges are no less than 0.635 cm (1/4 in) below the surface. Make certain that the sheets do not touch each other or con-tact the sides or bottom of the beaker.

6.5.3 Place the beaker in the oven at 121° ± 1°C (250° ± 2°F) for 96 h.

6.5.4 Insert a thermocouple in a glass thermocouple holder and place assembly into oil. The temperature of each oil should be recorded during the test.

<u>NOTE 1:</u> Test liquids must not be reused.

6.5.5 At the end of 96 h, remove the beaker from the oven and allow it to cool at room temperature for 30 ± 5 min.

6.6 Post test determination of physical properties.

6.6.1 Remove the rubber test specimens from the sample and wash in a fresh beaker of methyl alcohol, then rub gently with the fingers to remove all oil from the surface and rinse with a jet spray of methyl alcohol. Caution: Wear protective gloves.

6.6.2 Allow the rubber pieces to air dry for several minutes.

6.6.3 Volume swell.

6.6.3.1 Measure the displacement weight of the rubber test specimens per 6.4.4

6.6.4 Hardness.

6.6.4.1 The hardness of the rubber specimen shall be determined in accordance with ASTM Method D 2240, Type A durometer.

6.6.4.2 The thickness of the rubber specimen for the hardness test should be at least 0.3 cm. Therefore, a single-ply thickness of silicone rubber may be used as a cushion.

6.6.4.3 After swell testing, hardness readings shall be conducted on the end of each test specimen outside of the area to be used for tensile testing.

6.6.4.4 Apply the pressure foot to the specimen. Read the scale 15 s after the pressure foot is in firm contact with the specimen.

6.6.4.5 The hardness value of the test specimen shall be the average value obtained for the three test specimens.

6.6.5 Tensile strength.

6.6.5.1 Determine the loss in tensile strength by use of Method D 412, Standard Method of Tensile Testing of Vulcanized Rubber.

6.6.5.2 Three measurements shall be made for thickness; one at the center and one at each end of the specimen. The median of the three measurements shall be used as the thickness in calculating the cross sectional area.

6.6.5.3 When rubber specimens of standard thickness are used as per section 5.2 of this test method and the difference between the maximum and minimum thickness does not exceed 0.0762 cm (0.003 in), it is not necessary to calculate the cross-sectional area for each piece.

6.6.5.4 Place a specimen in the grips of the testing machine, using care to adjust it symmetrically in order that the tension will be distributed uniformly over the cross section. If tension is greater on one side of the specimen than the other, maximum strength of the rubber will not be developed. Start the machine and record the stress at the time of the rupture.

7 CALCULATIONS

7.1 Calculate the percentage change in volume (V) of each rubber sample as follows:

$$V = \frac{(A - B)100}{B}$$

where:
V = change in volume, percent.
A = displacement weight at conclusion of test, mg.
B = displacement weight before immersion in sample, mg.

7.2 Calculate the percentage change in hardness (H) of each rubber sample as follows:

$$H = \frac{(C - D)\,100}{C}$$

where:
H = change in hardness, percent.
C = hardness before test.
D = hardness after test.

7.3 Calculate the change in tensile strength.

7.3.1 Observed tensile strength.

F/E = tensile strength in mPa (psi).
F = observed force at break in N (lb).
E = Cross-sectional area of the unstretched specimen in cm^2 (in^2).

7.3.2 Change in tensile strength, percent

$$T = \frac{(J - K)\,100}{J}$$

where:
T = change in tensile strength, percent.
J = tensile strength before test.
K = tensile strength after test.

NOTE 2: See section 6.6.5.3

8. REPORTING

8.1 The report shall include the following:

8.1.1 Results calculated in accordance with paragraph 7, Calculations.

8.1.2 All observed and recorded data on which the calculations are based.

8.1.3 Date of test.

8.1.4 Temperature of test.

8.1.5 Type and dimensions of specimens used.

8.1.6 Immersion fluid used.

8.1.7 Exposure period.

8.1.8 Condition of exposed specimen from visual and manual examination.

8.1.9 Test apparatus used for hardness.

8.1.10 Type of tensile test apparatus.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Navy - NAPC - 1986

Method 3440.1
September 30 1986

STORAGE SOLUBILITY CHARACTERISTICS OF UNIVERSAL GEAR LUBRICANTS

1. SCOPE

1.1 This method is used for determining the storage solubility characteristics of universal gear lubricants.

2. SUMMARY

2.1 Universal gear lubricant is heated at 20°C and observed for separation of additive material after storage at room temperature for a period of 30 days.

3. SAMPLE SIZE

3.1 400 mL (one pint) of the gear oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Apparatus.

4.1.1 100-mL cone-shaped centrifuge tubes as specified in ASTM D 96.

4.1.2 Centrifuge with a diameter of swing (tip-to-tip of whirling tubes) 38 to 43 cm (5 to 7 in) and shall be capable of being controlled at a speed of 500 " 25 rpm.

<u>NOTE 1:</u> If the available centrifuge does not conform dimensionally to the preferred form, the speed of rotation of the available centrifuge must be adjusted to give the same centrifugal force at the tips of the tubes as that obtained with the prescribed instrument when operated at 500 " 25 rpm. The speed to operate the available centrifuge shall be calculated from the formula:

$$rpm = \sqrt{\frac{40}{d}} * 1500$$

where:
d = the diameter of the swing (tip-to-tip whirling tubes) of the available centrifuge in centimeters.

4.1.3 Balance capable of weighing to one mg.

4.1.4 Beakers, 400-mL capacity, heat resistant glass.

4.1.5 Desiccator capable of holding several centrifuge tubes.

4.1.6 Forced circulation oven capable of being controlled at 105° " 3°C

4.1.7 Forced circulation oven capable of being controlled at 20° " 1°C.

4.1.8 Cylinder, graduated, 100 and 400 mL.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Naphtha (TT-N-95, type II) [1,4,0].

NOTE 2: Caution. Naphtha is flammable.

6. PROCEDURE

6.1 Perform three solubility tests (three centrifuge tubes) for each oil.

6.2 Clean centrifuge tubes and dry them in oven at 105° " 3°C (220 °" 5°F) and permit them to cool to room temperature in a desiccator.

6.3 Number each tube and weigh it to the nearest mg.

6.4 Place 320 mL of the oil into a 400-mL beaker and heat in an oven at 120° " 1°C for 20 " 1 min.

6.5 Remove the beaker from the oven, allow the beaker to cool to 25° " 3°C. Transfer 100 " 1 mL of the oil from the beaker to each of the tarred centrifuge tubes. Cork the tubes.

6.6 Place the centrifuge tubes in an upright position in a darkened area such as a drawer or cupboard. The temperature should be maintained at 25° " 3°C for the length of the storage period, 30 " 1 days.

6.7 In transferring the centrifuge tubes from the storage area to the centrifuge, care must be taken not to disturb any material which may have separated from the oil. Remove the tubes from the storage area, place them in a centrifuge, and operate the centrifuge at 500 " 25 rpm for 30 " 1 min. If the residue is a solid, wash it several times with naphtha, a sufficient number of times to assure that it is free of oil.

6.8 Remove the centrifuge tubes for the centrifuge and decant and discard the supernatant oil. Permit the centrifuge tubes to drain in an upside down position at 25° " 3°C for a period of 2 h.

NOTE 3: If the separated material in not sufficiently compacted by the centrifuging to permit decanting the supernatant oil, continue the centrifuging for 5-minute intervals until decanting is possible. If the material is a liquid at the conclusion of the 30-minute centrifuging period, or if it cannot be compacted, cork the centrifuge tube and replace them in storage for an additional 30 days. At the end of the second storage period, proceed as directed in paragraph 6.7. If there is no visual liquid or solid residue after 30 days, stop the procedure and report zero residue.

6.9 Place the centrifuge tubes in an upright position in an oven controlled at 05° " 3°C for approximately 2 h.

6.10 Remove the centrifuge tubes from the oven and place them in a desiccator to cool to 25° " 3°C. Weigh the tubes and contents to the nearest mg. Subtract the weight of the empty centrifuge tubes to determine the weight of the separated material.

6.11 If the residue remains a liquid at the end of the second 30-day storage period, centrifuge the tubes as specified in 6.7. Remove the tubes from the centrifuge and note the volume of the separated liquids to the nearest 0.05 mL.

7. CALCULATIONS

7.1 Calculate the percent insoluble residue in the sample from the formula:

$$\textit{Percent insoluble residue} = \frac{\textit{Average amount of separated residue (grams) the three centrifuge tu}}{\textit{0.9 * weight percent of additive in sample}}$$

where:
0.9 = assumed specific gravity of the sample:

NOTE 4: The weight percent of additive in the sample can be obtained from the manufacturer of the sample.

7.2 Calculate the volume percent liquid separation after the second storage period from the formula:

$$\text{Percent volume liquid separation} = \frac{\text{Average separated liquid (mL) from the three centrifuge tubes}}{\text{volume percent of additive in the sample}}$$

NOTE 5: The volume percent of additive in the sample can be obtained from the manufacturer of the gear lubricant.

8. REPORTING

8.1 Report the average amount of separated residue found in the three test samples.

9. PRECISION

9. Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986

Method 3450
September 30, 1986

BEARING DEPOSITION OF AVIATION TURBINE ENGINE LUBRICANTS

1. SCOPE

1.1 This method describes detailed procedures used to conduct the bearing deposition test for evaluating deposit and degradation characteristics of aircraft turbine engine lubricants at various severity levels of bulk-oil temperature and bearing temperature for 48 h duration.

2. SUMMARY

2.1 A sample of the lubricant is subjected to the required temperature levels for a controlled number of hours. At periodic intervals during the test and at the end of test, samples of the oil are examined for both physical and chemical changes when compared to the sample of new oil. A visual inspection is made at the end of test of the accumulated deposits on the bearing and other machine components. A weighted numerical rating system is used for rating individual areas as well as for obtaining an overall rating. Additional data on relative sludge forming tendencies of oils are obtained by weighing a 100-mesh filter element and a 40-mesh filter element.

3. SAMPLE SIZE

3.1 Approximately 10 L of test oil.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

Military specifications:

Military Specification	Title
SAE AMS 5518	Steel, Corrosion Resistant (18-8), Plate, Sheet and Strip

(Copies of specifications, standards, handbooks, drawings, publications, and other Government documents required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting activity.)

Drawings:

Air Force Aero Propulsion Laboratory

Drawing	Title
A-B-67043	Test-Oil Pickup Line, Bearing Test Rig
A-B-67044	Thermocouple, Bearing Test Rig
A-B-67045	Test-Oil Jet Plug, Bearing Test Rig
A-B-67046	Test-Oil Jet Assembly, Bearing Test Rig
A-B-67047	Test-Oil-Out Filter Housing Assembly, Bearing Test Rig
A-B-67049	Metal Specimen Shaft, Bearing Test Rig
A-B-67051	Oil Level Rod Guide, Bearing Test Rig
A-B-67052	Test-Oil Pump Shaft Extension, Bearing Test Rig
A-B-67053	Thermocouple Connector, Bearing Test Rig
A-B-67054	Test-Oil Sump Thermocouple, Bearing Test Rig
A-B-67055	Scavenge Pump Discharge Line, Bearing Test Rig
A-B-67059	Metal Specimen Spacer, Bearing Test Rig
A-B-67060	Sump Vent Port, Bearing Test Rig
A-B-67061	Metal Specimens, Bearing Test Rig
A-B-67062	Scavenge Pump Modification, Bearing Test Rig
A-B-67063	Test-Oil Pump Modification, Bearing Test Rig
A-B-68114	Oil Level Indicator Tube, Bearing Test Rig
A-B-68115	Test-Oil-In-Line, Bearing Test Rig
A-B-69002	Test-Oil Pump Standoff Gasket, Bearing Test Rig
A-B-69008	Cup-Vent Pipe Blower Assembly, Bearing Test Rig
B-B-67039	Test-Oil-Filter Housing Details, Bearing Test Rig
B-B-67040	Test-Oil Accumulator Assembly, Bearing Test Rig
B-B-67058	Test-Oil Accumulator Details, Bearing Test Rig
B-B-68049	Top Plate, Stand, Bearing Test Rig
B-B-68050	Door, Stand, Bearing Test Rig
B-B-68051	Side Panel, Stand, Bearing Test Rig
B-B-68052	Rear Panel, Stand, Bearing Test Rig
B-B-68053	Air Cooler, Bearing Test Rig
B-B-68055	Blower Modification, Bearing Test Rig
B-B-68056	Vent-Pipe Blower, Bearing Test Rig
B-B-68057	Flange-Blower Vent Pipe, Bearing Test Rig
B-B-68058	Gasket-Blower Vent Pipe, Bearing Test Rig
B-B-68060	Test-Oil-In Filter Housing Assembly, Bearing Test Rig
B-B-68061	Test-Oil-In Filter Housing Details, Bearing Test Rig
B-6-69001	Test-Oil Pump Standoff, Bearing Test Rig
B-6-69009	Sump-Oil Fill Port Adapter, Vent Pipe Blower Assembly, Bearing Test Rig
C-B-68047	Stand Assembly, Bearing Test Rig
C-B-68048	Frame, Stand, Bearing Test Rig
C-B-68054	Blower Assembly, Bearing Test Rig

D-B-67016	Test-Oil Sump Components, Bearing Test Rig

(Application for copies should be addressed to the Air Force Aero Propulsion Laboratory, Attn: AFWAL/POSL, Wright-Patterson Air Force Base, Ohio 45433.)

4.1.1 Other publications. The following documents form a part of this method to the extent specified herein. Unless otherwise indicated the issue in effect on date of invitation for bids or request for proposal shall apply.

Society of Automotive Engineers (SAE)

AMS 3652, 3656, 3660, 3667	Polytetrafluoroethylene
AMS 5040	Carbon Steel Sheet and Strip, Deep Forming Grade
SAE J1966	Lubricating Oil, Aircraft Piston Engine (Non-dispersant mineral oil)

(Application for copies should be addressed to the Society of Automotive Engineers, Incorporated, 400 Commonwealth Drive, Warrendale, PA. 15096.)

American Society for Testing and Materials (ASTM)

ASTM D 445	Viscosity of Transparent and Opaque Liquids (Kinematic and Dynamic Viscosities)
ASTM D 664	Neutralization Number of Potentiometer Titration

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

4.2 The apparatus used in this method is the bearing deposition rig which consists of a 100-mm roller bearing machine, a suitable drive and support-oil system, and a specially designed test-oil system.

4.2.1 100-mm roller bearing head. The bearing head is divided internally into two main sections, as shown on Figure 1. The front, or test section with its separate test-oil system, houses the unshielded 100-mm straight-roller test bearing, A. Heating of the test bearing is achieved by supplying heat to the bearing outer race. The outer race is secured in a bearing mount assembly containing a circular-wound tubular heating element, B, packed in aluminum oxide. Heater control is provided by a West Instrument Corporation type JP controller, or equivalent, in conjunction with a manually variable voltage transformer. A watt-hour meter is also contained in the heater circuit and is connected between the heater and controller to monitor total power input. The rear or support section of the bearing head houses the externally loaded bearing, D, and main shaft support bearing, P, together with a separate oil system. The test and support sections of the bearing head are separated by a screw-thread seal, H, to prevent mixing of the test and support oil.

4.2.1.1 Test-oil jet. The test-oil jet, F, is a nominal 0.1-cm (0.040-in) diameter jet located at the 12 o'clock position on a 6.668-cm (2.625-in) radius from the center of the bearing machine end cover. Prior to test, the size of the jet is checked with standard plug pin gages, 0.099-cm (0.039-in) diameter gage go, and 0.104-cm (0.041-in) diameter gage no-go. In addition, the condition of

the jet is observed under flow condition by connection to a pressurized water line. Water flow through the jet is maintained by a 103.4 kPa gage (15-psig) pressure. The jet should deliver an essentially solid stream in axial alignment with the jet orifice. Corrections for size and axial straightness of jet flow are made by replacing the jet plug or the entire test-oil jet as required.

4.2.2 Test bearing. An unshielded 100-mm straight-roller bearing (Rollway P/N RCS 120-560, or equivalent) is mounted on the free end of the shaft in the test-oil compartment of the bearing head. Load is applied to the bearing shaft, C, and hence to the test bearing, by means of a hydraulically controlled load pis-ton, E, acting through the load bearing, as shown in Figure 1. The test bearing is lubricated by a single jet, F, which supplies essentially a solid stream of oil midway between the I.D. of the outer race and the O.D. of the roller retainer at the top or unloaded position of the test bearing.

4.2.3 Test-bearing mount. The bearing is made with an outer race removal feature which permits the removal of the bearing outer race for cleaning and inspection. Three spot-type thermocouples are located 120° apart (radially) around the I.D. of the bearing mount and indicate the skin temperature on the periphery of the outer race of the test bearing, which is pressed into contact with these thermocouple junctions when it is installed in the bearing mount. One of these three thermocouple leads is connected to the West bearing temperature controller, or equivalent.

4.2.3.1 Test bearing outer race thermocouples. Insert fiber glass sheath, 20-gage, iron-constantan thermocouple wire through the three drilled holes in the heater mount from outside to inside. Silver-solder each thermocouple junction leaving spherical bead on the junction about 0.32 cm (1/8 in) in diameter. Peen each bead back into the hole. Remove excess silver solder leaving just enough material to insure contact between the heater mount and the bearing outer race. Each of the three thermocouple leads is brought out directly through the bearing housing at 120 degree intervals.

4.2.3.2 Thermocouple packing. Polytetrafluoroethylene, or equivalent, is used to insulate all thermocouple leads where they pass through the housing or end cover fitting. This also applies to the two bearing heater terminals where they pass through the end cover.

4.2.4 Test-oil system. A schematic of the test-oil system is shown on Figure 2. The test-oil cycle starts at the sump where external heaters maintain the desired bulk-oil temperature. Sump heaters, except for the uppermost band heater which is controlled by a manual switch, are controlled by a West type JP controller, or equivalent, with a watt-hour meter connected between the heaters and the controller to monitor total power input. The test oil is pumped from the sump by the variable-speed pressure pump to the 100-mesh filter housing. The lubricant then flows to a tubing cross attached to the filter housing. One leg of the cross contains a bayonet thermocouple measuring the test-oil-in temperature. One leg transmits fluid pressure to an indicating gage and two pressure safety switch units connected in series. The latter units are set with one unit at a minimum pressure of 34.5 kPa gage (5 psig) and one at a maximum pressure of 241.3 kPa gage (35 psig). In the event of a test-oil pressure excursion outside this range, the applicable safety switch deactivates the main drive motor. The system protects against rig tieup as a consequence of lubricant starvation in the event of test-oil pump failure, jet plugging, or other equipment malfunction. The third leg of the cross delivers test oil to the jet and then to the test bearing. The test oil is drained from the bearing head at two locations, G, as shown on Figure 1. The front drain collects splash oil which does not pass through the bearing, whereas the rear drain collects only oil that has gone through the bearing. Both drains feed into a fluid accumulator via a single line leading to a three-way valve used for in-line sampling. The test-oil line continues beyond this valve to a second three-way valve which permits routing of the oil to an incorporated test-oil flow measuring system. The test oil next passes through a 40-mesh filter and enters the scavenge pump prior to discharging into the sump.

4.2.4.1 Heat loss from the test-oil system is minimized by placing insulation on the sump sides, base, and cover as identified on Figure 2. The test-oil-in lines and filter housing are wrapped with a single thickness of Fiberfrax insulation, or equivalent, to reduce the differential between test-oil-in temperature and sump temperature. Insulating in this manner will normally result in a test- oil-in temperature slightly below the required bulk-oil (sump) temperature. If this differential exceeds the allowed maximum, additional insulation thicknesses are applied. In operation, the test-oil sump is vented to atmosphere.

4.2.4.2 The test-oil entering the sump is normally hotter than the bulk-oil in the sump. A forced-air cooler, shown on Figure 2, is placed around the test- oil line section preceding the test-oil-out filter to cool the lubricant as required. In some cases, it may be necessary to insulate a portion of the scavenge line with insulating tape to maintain the desired oil temperature entering the sump.

4.2.4.3 Only stainless steel tubing or pipelines and fittings are used in the test-oil system. The fluid accumulator, filter housings, and sump are similarly constructed of stainless steel.

4.2.4.4 Test-oil sump. The overall configuration and construction details of the test-oil sump conform to the detailed drawings specified in 4.1.

4.2.4.4.1 Sump cover seal. A 0.16-cm (1/16-in) thickness gasket of suitable dimension is cut to seal between the sump flange and sump cover. Raybestos-Manhattan A-56 gasket material, or equivalent is employed.

4.2.4.4.2 Metal specimens. Metal specimens are mounted within the test-oil sump in order to provide information concerning possible metal attack by the lubricant. The specimen holder is a 0.95-cm (3/8-in) rod mounted on the sump lid and extending 20.32 cm (8 in) into the sump (or approximately 5.08 cm (2 in) below the cold oil level). The specimens are secured to the rod by a 10-32 size screw, 3.81 cm (1-1/2 in) in length. The specimens are individually placed on the screw and separated by spacers of 0.32-cm (1/8-in) thickness and 0.79-cm (5/16-in) O.D. The mounting rod is tapped at the lower end to accept the screw such that the specimens are mounted with their major axis in a vertical plane. The metal specimens are 0.081-cm (0.032-in) thick, 1.905-cm (3/4-in) diameter disks with 0.635-cm (1/4-in) center holes. A five-specimen set is used consisting of the following materials:

Aluminum	ASTM B 209, temper T-3 or T-4
Titanium	8Mn Annealed, 110 ksi (758 MPa) yield strength-UNS R56080
Silver	99.9% purity
Steel	AMS 5040
Stainless Steel	SAE AMS 5518

A sketch of the metal specimen assembly, indicating the metal specimen order, is shown on Figure 3.

4.2.5 Air system. Water saturated air is metered to the test-oil compartment in the bearing head. The air supply for the test-bearing compartment is filtered shop air connected in the following sequence: air supply to pressure regulator to flow control valve to rotameter to water saturator to water separator column to end cover fitting on bearing head. The air directed through the end cover (at 1 o'clock on the same diameter as the test-oil jet) into the bearing compartment is used primarily to control the pressure drop across the seal so as to insure against oil transfer from the support section into the test-oil section. A 0.64- to 0.32-cm (1/4- to 1/8-in) tubing to pipe fitting, J of Figure 1, should be used for the metered air supply fitting at the end cover. The water saturator consists of a 1-in-diameter spherical gas diffuser stone submerged in distilled water within a 2 L filtering flask. The flask is initially charged with 1200 mL of water and is refilled to this level at each shutdown period during the test. A water separator column is connected downstream of the saturator to remove entrained droplets. The column consists of a 61-cm (24-in) length of 5.08-cm (2-in) borosilicate or equivalent, glass pipe flanged at both ends. The flanges are constructed of suitable aluminum plate with a polytetrafluoroethylene gasket seal between the glass pipe and flange, (see AMS 3652, 3656, 3660, 3667). The column is loosely, but completely, packed with glass wool fiber, Corning Cat. No. 3950, or equivalent.

4.2.5.1 Air to the test head is metered through a rotameter. Prior to each run, the rotameter is calibrated against a wet-test gas meter for a flow of 9.91 ± 0.57 L/sec (0.35 ± 0.02 cfm). The procedure is to disconnect the air supply line at the fitting where the air enters the head and then connect the supply line to a wet-test gas meter which in turn discharges to atmosphere. A mercury manometer is located immediately downstream from the rotameter. The settings of the manometer and rotameter are observed and recorded for a wet-test gas meter reading of 9.91 L/sec (0.35 cfm). The air line is then reconnected to the bearing head and the previously observed manometer and rotameter settings are maintained throughout the test.

4.2.5.2 Screw seal. The screw type seal is vented to atmosphere at both the top and bottom seal housing connections.

4.2.5.3 Connection for seal differential pressure measurement. The lines of the manometer used to measure the differential pressure across the seal should be connected to the top, front of the bearing head, and to the rear vertical surface of the bearing housing between 11 and 1 o'clock, 2.54 cm (1 in) above the junction of the rear case with the main housing. After startup, there should be no venting of the manometer lines. In this connection, the pipe plug in the top of the gearbox should be removed and replaced with a small valve to allow adjustment of the pressure in the gearbox and, hence, in the support system. The adjustment of the pressure of the atmosphere of the support-oil system controls the pressure drop across the screw seal, which is used to insure against oil transfer from the support section to the test-oil section. The required differential is controlled by maintaining a slightly higher test-oil compartment pressure of 0.76 to 1.27 cm (0.3 to 0.5 in) of test-oil column relative to the support-oil compartment pressure.

4.2.5.4 Sump air venting. The sump is vented to the atmosphere through a sump cover port as shown on Figure 2. A line connected to the port terminates at a valve which permits connection of the line to a gas-flow meter. When the required test conditions are attained after each startup, the flow rate of the gas discharging from the sump is measured at the valve employing a portable gas flow rate meter. The speed control of the scavenge pump motor is set to provide a sump gas discharge of 14.16 L/sec (0.5 cfm). During a test, additional measurements of the sump vent gas flow rate should be made when excessive scavenge pump wear or test-oil system air leaks are suspected.

4.2.5.4.1 The procedure for the sump-air venting discussed above is modified for bearing tests employing sump bulk-oil temperature above 176.7°C (350°F). The valve is closed to the atmosphere and vent pipe-blower assembly is connected to the sump oil-fill port, as shown on Figure 4 to condense the increased oil vapor created in the sump by the elevated oil temperature. Oil vapor/mist condenses in the blower housing and is drained back into the sump. The measurement of the sump vent gas flow rate is made at the valve as previously discussed. However, the vent pipe-blower assembly is removed and the sump-oil port is capped each time the airflow rate is to be measured. After each measurement, the vent pipe blower assembly is replaced at the sump-oil fill port.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Dry cleaning solvent (CID A-A-59601D).

5.3 Carbon remover (Penetone Corporation 74 Hudson Avenue, Tenafly, New Jersey, Penetone ECS or equivalent) [u,u,u].

5.4 *n*-Heptane [1,3,0].

5.5 Acetone [1,3,0].

5.6 SAE J1966, lubricating oil.

5.7 Polytetrafluoroethylene tape and gasket material [0,0,0].

5.8 Fiberfrax insulation [0,0,0].

5.9 Raxbestos-Manhattan A-56 gasket material [0,0,0].

5.10 Metal specimens [0,0,0].

Aluminum	ASTM B 209
Titanium	8Mn Annealed, 110ksi (758 MPa) yield strength-UNS R56080
Silver	99.9% purity
Steel	AMS 5040
Stainless Steel	SAE AMS 5518

5.11 Glass wool fiber, Corning Cat. No. 3950 [0,0,0].

5.12 400 grit silicon carbide paper [0,0,0].

5.13 30 grit aluminum oxide [0,0,0].

6. PROCEDURE

6.1 Cleaning procedure.

6.1.1 Test-oil sump. The test-oil sump assembly is completely dismantled for cleaning of all internal components after each test. The sump interior is thoroughly cleaned with a suitable solvent, hand scrubbing, or mild abrasive blast to remove all traces of deposits from the previous test. A new test-oil pumpshaft seal is installed prior to each test.

6.1.2 Test-oil pressure and scavenge pumps. The test-oil pressure and scavenge pumps are completely disassembled and then cleaned to remove all deposits from the previous test.

6.1.3 Test-oil accumulator. The test-oil accumulator located directly below the bearing head is disassembled and then cleaned with a stainless steel brush in dry cleaning solvent, or equivalent.

6.1.4 Test-oil lines. All of the straight run test-oil lines and fittings (elbows, tees) are removed from the test-oil system and hand reamed with stainless steel wire brushes in a bath of dry cleaning solvent, or equivalent.

6.1.5 Oil filter assemblies. All oil filter housings in the test-oil system are removed and cleaned with a stainless steel brush in dry cleaning solvent, or equivalent. A soft fiber brush is usually adequate in scrubbing the filter elements after a short soaking in a solvent bath.

6.1.6 Pressure gage line. The test-oil lines leading to the pressure gage and safety switch units are flushed with approximately 25 mL of test oil prior to each test. Prior to starting the drive motor and to insure correct pressure transmission, the drain valve at the end of the line is opened momentarily while the system is under pressure.

6.1.7 Bearing head. The bearing head should be disassembled as far back as the seal and all of the removable parts, except the test bearing, are cleaned with a suitable carbon remover followed by scouring with No. 400 emery paper and solvent to remove all traces of deposits from the previous test. The entire seal assembly should be removed and cleaned when the level of deposits on the rear seal plate exceeds a light varnish condition.

6.1.8 Test bearing. A new 100-mm bearing is used for each 48-hour test. The new bearing is cleaned in solvent to remove preservative oil. In order to facilitate deposit rating, the bearing cage is thoroughly cleaned at three points using a stainless steel wire brush. This procedure removes the brown coating applied to the cage and allows for a more accurate evaluation of cage deposits by initially exposing the bright base metal. The three positions on the cage are arbitrarily selected for brushing but are approximately equally spaced. All exposed sides of the cage are cleaned at each position, covering a circumferential distance equal to that between identical points on two successive rollers. The bearing is recleaned in solvent taking care to remove all adhering wire bristles. The bearing is dipped in test oil prior to installation in the machine.

6.1.9 Metal specimens. Clean and pre polish the specimens by appropriate means of choice. If the specimens are being reused from a previous test, no pits, etchings, or signs of corrosion should be visible at this point. Finish with No. 400 grit silicon carbide paper, removing all marks that may have been left by previous polishing. The specimens should not be handled with bare fingers from this point; handle them only with a clean cloth or paper, or with tongs or forceps. Cotton swab wash the specimens with solvents, first with *n*-heptane and then with acetone, final rinse in clean acetone, air dry and weigh to the nearest 0.1 mg. If there is to be any delay before weighing, store under *n*-heptane until ready.

NOTE 1: As a practical polishing procedure, place a sheet of the abrasive paper on a flat surface and rub the specimen against the paper with longitudinal strokes, protecting the specimen from contact with the fingers with clean cloth or ashless filter paper. An individual abrasive paper should be employed for each metal. Several specimens of the same metal may be polished with one paper.

6.2 Bearing head assembly.

6.2.1 Main shaft. If the main shaft is not solid, its front end should be plugged with an aluminum plug.

6.2.2 Screw-thread seal. In installing the seal, the right-hand threads on the O.D. of the seal must be toward the front (test compartment) end of the rig.

6.2.3 Test-bearing heater mount. The bearing outer race is lightly pressed into the bearing heater mount with the serial number facing the front of the rig. The recommended fit on the bearing outer race is -0.0005 . 0.0018 cm (-0.0002 to . 0.0007 in). On repeated usage, the I.D. of the bearing mount may become worn and result in a loose fit which might tend to cock the outer race. It may also result in erroneous temperature readings of the bearing outer race. When this happens, the I.D. of the mount may be chrome plated and reground to proper dimension, or that section of the mount replaced.

6.2.3.1 Test-bearing heater assembly. In reassembling the heater mount, care should be exercised to insure that the heater coil is centered and that at no point should the heater coils come within 0.32 cm (1/8 in) of the inside surfaces of the assembly faces. A method of accomplishing this is to assemble the unit without retainer ring; this allows visual inspection of the location of the heater-coils within the assembly. Areas where the coils come within 0.32 cm (1/8 in) of either front or rear inner faces are marked. During final assembly, Transite spacers, or equivalent, (0.32 □ 0.64 □ 1.27 cm [1/8 □ 1/4 □ 1/2 in]) are placed at these marked points. The heater mount cavity is repacked with new No. 30-grit aluminum oxide after each test. Suitably small portions of the oxide are added in increments, followed by brisk tapping of the mount exterior to eliminate voids. After adding a portion of the oxide, the mount is raised to the vertical position and tapped with a compositon head hammer over front and rear surfaces working from bottom to top. The procedure is repeated at frequent intervals until the cavity is filled.

NOTE 2: Prior to installation of the heater mount and test-bearing assembly, the oil-jet alignment procedure of 6.2.5 is accomplished.

6.2.3.2 Test-bearing heatermount positioning. Care is exercised to insure that the front and rear vertical faces are parallel to each other and square with the I.D. of the mount. If these conditions are not maintained, the concentricity of the bearing's outer race in relation to the shaft axis may be affected. Before installing the heater mount in the rig, the bearing's outer race thermocouples are checked for continuity.

6.2.4 Test bearing. Before installing the test bearing on the hub, it is examined visually for any obvious mechanical defects. If the bearing appears satisfactory, the inner race and cage assembly is pressed onto the hub with the serial number facing the front of the rig. The recommended fit of the bearing inner race on the hub is 0.002 to 0.0036 cm (0.0008 to 0.0014 in) tight. After repeated usage, the O.D. of the bearing hub may become worn and result in a loose bearing fit. When this happens, the hub may be chrome plated and reground to proper dimension or replaced. After the bearing has been installed in the bearing head, the shaft is turned by hand to make sure it turns freely. Bearing roughness can usually be detected in this manner. When properly installed, the rollers should be evenly centered in the outer race. If the rollers are more than 0.32 cm (1/8 in) off center on the outer race, check for missing shaft assembly parts or an improperly installed bearing outer race. It is important that the lock nut on the test-bearing retainer be tight since it imparts an axial pinch on all of the shaft assembly parts back to the load bearing and in addition helps to prevent the bearing inner race from turning on the hub during operation.

6.2.5 End cover. Before installing the end cover on the rig, the test-oil jet tube alignment is verified. The jet positions midway between the outer race I.D. and the cage O.D. Alignment is verified by assembling (outside the machine housing) the heater mount with the outer race installed, the pinch plate, and end cover. The heater mount is fixed by installing the heater terminal nuts and making certain that the mount O.D. coincides with the pinch plate diameter. The end cover mounting bolts are inserted through the cover and corresponding pinch plate holes. The test-oil jet is inserted through its holder and the jet position verified by visual observations from the rear of the heater mount. The test- bearing roller and cage assembly is installed in the outer race through the heater mount rear side and the jet position noted with respect to the cage O.D. Upon subsequent assembly of the bearing machine for testing, a gasket of Raybestos-Manhattan A-56 material, or equivalent, is installed between the end cover and pinch plate.

6.2.6 Check of seal operation. After assembly of the bearing head, the screw thread seal operation is checked under a 2224 N (500-pound) radial load to the load bearing by applying 351.6 kPa (51-psig) shop air pressure to the loading cylinder and hand rotating the shaft to detect rubbing. If any rubbing occurs, the bearing head is disassembled and a slight amount of material removed from either the screw threads or the seal plate, depending on the location and severity of the rubbing. The material is removed from the seal plate if the rubbing is localized, and the screw thread is ground if the rubbing is severe and occurs over a considerable area.

6.2.7 Miscellaneous joint compounds. No pipe dopes, thread compounds, or gasket sealants are used in the assembly of the rig or test-oil system. Polytetrafluoroethylene tape, or equivalent (no adhesive) is an effective sealant when applied to pipe threads.

6.3 Support-oil system. SAE J1966, lubricating oil is used in the support-oil system and is not normally changed after each run. Drain periods on the support oil are governed by the amount of contamination with test oil, which can be determined from periodic laboratory tests on support-oil samples.

6.4 Critical test items. Before starting a test, care should be taken that critical test items conform to the tolerances listed in Table I.

TABLE I. Critical test items.

Item	Dimensional tolerances in cm (in)	
	Minimum	Maximum
Test-bearing outer race fit in heater mount	-0.0005 (0.0002)	0.00018 (+0.0007)
Test-bearing inner race fit on hub	-0.002 (-0.0008)	-0.0036 (-0.00014)
Screw seal diametral clearance	0.046 (0.018)	0.056 (0.022)
Test-oil jet	0.099 (0.039)	0.104 (0.0041)

6.5 Operating conditions.

6.5.1 Throughout the entire test, the test-oil-in temperature, bulk oil temperature, test-bearing temperature, and airflow to the end cover are controlled at the values required by the applicable lubricant specification.

6.5.2 Initial test-oil flow setting. The test-oil sump is charged with 7.75 L (2 gal) of the test lubricant at room temperature and heated to a temperature of 126.7°C (260°F). Air to the end cover is set at 0.165 L/sec (0.35 cfm). The test- and support-oil pumps are turned on and the main drive motor actuated to a speed of 10,000 rpm. The rig speed should be brought up immediately after oil circulation is started as the screw-thread seal depends on relative motion for its sealing action. The previous setting on the test-oil pressure pump or a minimum of 68.9 kPa gage (10-psig) oil pressure at the jet is used to insure against possible oil starvation during this initial startup. The pressure drop across the seal is maintained at 0.76 to 1.27 cm (0.3 to 0.5 in) of test oil by making fine adjustments to the gearbox vent valve. As soon as a 121.1°C (250°F) oil temperature at the jet has stabilized, a minimum of two separate flow checks of 1-min duration each should be made at the incorporated test oil flow measuring system. The oil pressure at the jet should be adjusted using the variable speed drive unit on the test-oil pump until the jet oil flow is 600 mL/min. Once the flow rate of 600 mL/min is established for an oil, the flow at this condition is maintained constant during the test. The observed test-oil flow rate is checked and recorded once every 3 h, or when indicated by any irregularities in test operation.

6.5.3 After the oil flow is set, the following conditions are allowed to stabilize:

Oil-in temperature	121.1° . . 4°C (250° . 1 2°F)
Oil flow rate	600 . 20 mL/min
Bearing speed	10,000 rpm . 200
Radial load	351.6 kPa (2224 N) [51 psig (500 lb)]
Support-oil-in-temperature	71.1° to 82.2°C (160° to 180°F)
Support-oil pressure	551.6 to 689.5 kPa gage (80 to 100 psig)
Gearbox-oil pressure	172.4 to 206.8 kPa gage (25 to 30 psig)
Airflow to end cover	0.165 . 0.01 L/sec (0.35 . 0.02 cfm)
Pressure drop across seal	0.76 to 1.27 cm (0.3 to 0.5 in) of test oil (test compartment higher)

After the foregoing conditions stabilize, the run is continued until the test- bearing temperature from the highest reading thermocouple stabilizes (about 1 h). This temperature is then noted and used as an indication of the mechanical condition of the bearing. Previous tests on bearing stabilization at a 121.1°C (250°F) oil-in temperature to the jet indicate that a temperature range of 148.9°C (300°F) is normal for a properly functioning 100-mm test bearing. Higher stabilization temperatures indicate the test-bearing condition is questionable, and the bearing should probably be replaced before continuing with the test.

6.5.3.1 Once the test bearing has reached a satisfactory stabilization temperature, the setting on the West controller for the test-oil heaters is increased to raise the bulk-oil temperature to required value. The uppermost sump band heater is controlled by a manual on-off switch, not the West controller. This heater is used only to accelerate the heating rate when a sump temperature of 232.2°C (450°F), or above, is employed. The heater is not actuated until a sump temperature of 204.4°C (400°F) is reached, and is deactivated when the desired sump temperature is achieved. The bearing heater should be turned on simultaneously with the change in sump heater setting to bring the bearing outer race temperature (max) to the required temperature. At this point, the three thermocouples attached to the bearing outer race should be checked to make sure that the thermocouple indicating the second highest temperature is attached to the West temperature controller.

6.5.3.2 When the required bearing and sump oil temperatures are reached, the test-oil flow rate is again checked and adjusted as necessary to maintain 600 mL/min. The sump vent-gas flow is also measured at this time. The scavenge pump motor speed is adjusted to obtain a flow of 0.24 . 0.01 L/sec (0.5 . 0.02 cfm).

6.5.4 <u>Test cycle</u>. The 48-hour test is conducted in three 16-hour operating periods. A fixed shutdown period consisting of a minimum of 4 h and a maximum of 8 h is observed between each 16-hour operating period. Run time begins counting when the test-oil heaters are turned on, including bearing stabilization time. Test time stops when the test-oil heaters are deactivated. Except for emergency stops, all shutdowns are made in accordance with the detailed procedure subsequently outlined.

6.5.5 Used oil analysis. The following laboratory tests are run on the new and used oil samples:

Test	ASTM method
Viscosity at 37.8°C (100°F), cSt	D 445
Viscosity at 98.9°C (210°F), cSt	D 445
Neutralization No., mg KOH/g	D 446, using a pH 11 endpoint

6.5.6 Test-oil flow rate check. During operation, the test-oil flow rate is checked at the incorporated test-oil flow rate measuring system once every 3 h. The system is located in the scavenge line as shown on Figure 2. It consists of a three-way valve located in the scavenge line which permits routing of the oil into an open 1000-mL separatory funnel. The funnel stopcock tube leads to a two-way valve which connects to the scavenge line.

6.5.6.1 The test-oil flow measuring procedure is carried out by setting the three-way valve to route the oil into the funnel for a period of 1 min, at which time the valve is reset to its normal position. After the oil volume is measured, the two-way valve and stopcock are opened to drain the oil in the funnel back into the scavenge line. The two-way valve and stopcock are closed after the oil is drained in preparation for succeeding test-oil flow rate determinations. Care is taken to prevent air entering the scavenge line upon draining the separatory funnel.

6.5.6.2 At the completion of the test, while the test oil is still hot, the sump and test-oil system is drained into a suitable container so that the volume of test oil remaining can be determined. After the volume of test oil has been measured (room temperature) and recorded, 1 L (1 t) of the test oil should be poured into a clean container and saved for laboratory analyses.

6.5.7 Rig photographs. As soon as practical, the rig is disassembled and 10 × 12.7 cm (4 × 5 in) color photographs are taken of the following components:

a. Bearing compartment with cover removed - front view.
b. Heater mount - rear side.
c. End cover - rear side.
d. Seal plate in housing - heater and bearing removed.
e. Test bearing - inner race, rollers, and cage assembly close-up, 45° angle, 0° position.
f. Test bearing - inner race, rollers and cage assembly close-up, 45° angle, 180° position.
g. Test bearing - outer race close-up, inside diameter, 0° position (adjacent to test-oil jet).
h. Test bearing - outer race close-up, inside diameter, 180° position.

A typical set of photographs, reduced and in black and white, is shown on Figure 5.

6.5.7.1 After the photographs are completed, a detailed visual inspection is made of these parts to determine the average depth, coverage, and consistency and type of oil deposits, plus the extent of abnormal metal wear on the test-bearing cage, rollers, roller pockets, and outer race.

6.6 Test Procedure.

6.6.1 <u>Preparation for test</u>.

1. Turn on rig power switches.
2. Turn on support-oil tank heaters and set at 82.2°C (180°F).
3. Turn on multipoint temperature recorder.
4. Charge the test-oil sump with 7.57 L (2 gal) measured at room temperature. The test-oil expansion upon heating to the required sump bulk-oil temperature will establish the "full" mark which should be noted on the level indicator tube.
5. Turn on test-oil heaters and adjust controller to obtain 126.7°C (260°F).
6. Open air bleeds on both sides of the differential manometer which is filled with test oil.
7. Checkloading valve on console. It should be in the open or zero-load position.
8. When the bulk-oil temperature in the test tank reaches 126.7°C (260°F), turn on the air supply to end cover and adjust to 0.165 L/sec (0.35 cfm).
9. Start the test-oil pressure and scavenge pumps. Test-oil pressure should be at least 68.95 kPa gage (10 psig) at test-oil jet.
10. Start the support-oil pumps.
11. Set and maintain support-oil pressure at 551.6 to 689.5 kPa gage (80 to 100 psig).
12. Adjust the load pressure to 68.95 kPa gage (10 psig) and start the drive motor.
13. Increase rig speed immediately to 10,000 rpm.
14. Close loading valve on console until 351.6 kPa gage (51 psig) is obtained on gage.
15. Close air bleeds on both legs of manometer used for pressure differential checks across the screw-thread seal. The required differential is controlled by maintaining a slightly higher test-oil compartment pressure of 0.76 to 1.27 cm (0.3 to 0.5 in) of test-oil column relative to the support-oil compartment pressure.
16. Continue running until the following conditions are stabilized:

Test-oil-in temp	121.1 ± 1.4°C (250° ± 2°F)
Test-tank bulk-oil temp	126.7° ± 2.8°C (260° ± 5°F)
Airflow to end cover	0.165 ± 0.01 L/sec (0.35 ± 0.02 ftm)

17. Make at least two separate flow checks of 1-minute duration each at the test oil flow measuring system. The measured volume flow rate should be 600 ± 20 mL/min.
18. Continue running for 1 h at these conditions with the bearing heater off. If during, or at the end of this period, the maximum bearing temperature has exceeded 176.7°C (350°F), shut down the rig, install a new test bearing, and repeat steps 1 through 17.

If the maximum bearing temperature has not exceeded 176.7°C (350°F) during this period, proceed with the next step.

19. Turn on test bearing heater and adjust controller to obtain the required bearing operating temperature. Adjust bearing heater voltage to achieve consistent, but not accelerated, temperature rise. Upon reaching the required bearing operating temperature, the heater voltage is again adjusted to minimize the bearing temperature variation above and below the control point.
20. Check the three test bearing outer-race thermocouples to make sure the second highest indicating thermocouple is connected to the West temperature controller. However, maintain the required bearing operating temperature on the highest indicating thermocouple connected to a temperature recorder.
21. Adjust the test-oil heater controller to obtain the required bulk-oil temperature.
22. Continue running until the test-oil-in temperature, bulk-oil temperature, test-bearing temperature, and airflow to end cover are stabilized to the applicable specification values.
23. Mark the level indicator tube to show the full position. All subsequent oil level checks are made in reference to this point.
24. Take at least two separate flow checks of 1-minute duration, each at the test-oil flow measuring system. Flow rate should be 600 ± 20 mL/min.
25. Measure the sump vent-gas flow at the exhaust valve and adjust the scavenge pump motor speed to obtain 0.24 ± 0.01 L/sec (0.5 ± 0.2 cfm) vent-gas flow.
26. Adjust airflow to air cooler to maintain specified test-oil return temperature.
27. Continue running to complete the 16-hour operating period, shut down the rig in accordance with 6.6.3 and observe a shutdown period.

6.6.2 Operating Procedure.

6.6.2.1 Record the following operational data at 30-min intervals:

a. Test hours (count from the time when the test-oil heaters are turned on until turned off).
b. Time and date.
c. Load-oil pressure.
d. Test-oil pressure.
e. Ratiotrol setting - test-oil pump.
f. Ratiotrol setting - scavenge pump.
g. Seal differential pressure - test versus support section.
h. Airflow to end cover - rotameter scale.
i. Test-bearing temperature (3 thermocouples).
j. Test-oil sump temperature.
k. Test-oil-in temperature.
l. Test-oil-out temperature - end cover side.
m. Test-oil-out temperature - seal side.
n. Test-oil return temperature.
o. Support-oil-in temperature.
p. Support-oil-out temperature.

q. Watt-hour meter, test-oil sump heater.
r. Watt-hour meter, test-bearing heater. 6.6.2.2 Take 40-mL samples of test oil from three-way valve in scavenge line every 4 h (including warm-up time along with endurance time).

6.6.2.3 Makeup oil should be added immediately after each 40-mL sample is taken by adding sufficient quantity of unused test oil to bring the oil level to the full mark. Makeup oil is not added after the sample withdrawal at 16 and 32 h. The rig is shut down at these times, and makeup oil is added after stabilization of test conditions for the subsequent 16-hour running period.

6.6.2.4 Test-oil-in and test-oil-out filters are replaced with clean, pre-weighed elements during each 4-hour down period. The used filter elements are allowed to drain for 1 h at 85°C (185°F), weighed, and the weight gain recorded.

6.6.3 Shutdown sequence.

a. Shut off test-bearing and sump heaters.
b. Adjust loading valve on console and reduce load pressure to 172.4 kPa gage (25 psig).
c. Continue running at these conditions until test-bearing temperature (maximum) drops to 204.4°C (400°F).
d. Reduce load to 68.95 kPa gage (10 psig) and shut off drive motor.
e. When drive shaft stops, shut off test-oil pumps, support-oil pumps, and heater, main electrical and water switches, and all air valves.

6.6.4 Startup sequence.

a. Turn on rig power switches and support-oil heaters in sufficient time to attain the 82.2°C (180°F) temperature prior to startup.
b. Turn on multipoint temperature recorder.
c. Follow step Nos. 5 through 17 of 6.6.1.
d. Continue running until the maximum bearing temperature stabilizes within 2.8°C (5°F) between successive readings taken at 2-min intervals. This stabilization normally occurs within 20 to 30 min after starting the drive motor.
e. Follow step Nos. 19 through 22 of 6.6.1.
f. Add sufficient new oil to bring the sump level indicator to the full mark.
g. Follow step Nos. 24 through 27 of 6.6.1.

6.7 Deposit rating system.

6.7.1 Outline of method.

6.7.1.1 Deposit demerits. Bearing machine cleanliness is reported in this deposit demerit system by the assignment of values of 0 to 20. Identify the different types and thicknesses of deposits as follows: 0 shall designate a new or clean condition; 20 shall represent the worst condition that could be expected.

6.7.1.2 Table II shows the numerical demerits to be assigned to different types and degrees of deposits.

TABLE II. Demerit rating numbers used for numerically describing deposits.

Deposit type	Demerit rating number		
	Light	Medium	Heavy
Varnish	1	3	5
Sludge	6	7	8
Smooth carbon	9	10	11
Crinkled carbon	12	13	14
Blistered carbon	15	16	17
Flaked carbon	18	19	20

6.7.1.3 Table III defines deposit types and severities.

6.7.1.4 The following six major items in the test bearing section of the bearing machine are visually inspected and rated to obtain the overall deposit demerit rating:

a. End cover.
b. Spacer and nut (considered to be one piece).

Table III. Description of deposit types and degrees.

Deposit type	Degree	Description
Varnish	-----	Varnish or lacquer-like coating, shiny
	Light	Light gold or yellow in color, translucent
	Medium	Brown or dark brown in color, translucent
	Heavy [1]	Black in color, opaque
Sludge	-----	Shiny, oil emulsion of carbon and oil usually light brown in color. Remove by wiping with a rag.
	Light	Less than 1/64 in thickness
	Medium	1/64 to 3/64 in thickness
	Heavy	3/64 in thickness or more
Smooth carbon	-----	Carbonaceous coating not removable by wiping with a rag
	Light [1]	Less than 1/64 in thickness
	Medium	1/64 to 3/64 in thickness
	Heavy	3/64 in thickness or more

Crinkled carbon	-----	Same as for smooth carbon, ridged or uneven surface, not smooth
	Light	Less than 1/64 in thickness
	Medium	1/64 to 3/64 in thickness
	Heavy	3/64 in thickness or more
Blistered carbon	-----	Same as for smooth carbon, blistered, bubbled
	Light	Less than 1/64 in thickness
	Medium	1/64 to 3/64 in thickness
	Heavy	3/64 in thickness or more
Flaked carbon	-----	Same as for smooth carbon, flaked or broken blisters, peeling
	Light	Less than 1/64 in thickness
	Medium	1/64 to 3/64 in thickness
	Heavy	3/64 in thickness or more

1 Some difficulty may be encountered in distinguishing between light smooth carbon deposit and heavy varnish. The varnish deposit appears shiny and glossy and upon scraping indicates a tacky consistency or thin, brittle flaking. The smooth carbon deposit appears dull and lusterless and upon scraping reveals a grainy consistency.

c. Heater-mount front.
d. Heater-mount rear.
e. Seal plate.
f. Test bearing.

6.7.1.5 The specific areas rated on each of the six major items are as follows:

a. End cover: The surface of the end cover normally exposed to test oil.
b. Spacer and nut: The entire surface of the spacer, nut, lock-washer, and flat washer (all considered to be one piece) normally exposed to test oil.
c. Heater-mount front: The surface of the heater-mount front which is parallel to the plane of the end cover and normally exposed to test oil. The portion of the heater-mount front which contacts the heater-mount retainer is not included in the area rated.
d. Heater-mount rear: The surface of the heater-mount rear which is parallel to the plane of the end cover and normally exposed to test oil.
e. Seal plate: The visible surface of the seal plate, with the slinger in place, normally exposed to test oil.
f. Test bearing: The test bearing is divided into four section for rating purposes. These four sections are in turn broken down into eleven specific areas as follows:

Rollers	1. Front 2. Rear

	3. Contact Surface
Cage	4. Front 5. Rear 6. Outside diameter surface
Outer race	7. Front 8. Rear 9. Contact surface
Inner race	10. Front 11. Rear

6.7.2 Computation of overall deposit rating.

6.7.2.1 An area demerit rating is determined according to the area covered by the deposit as follows:

$$\frac{\textit{Percent area covered}}{10} = \textit{area demerit covering}$$

Surface deposits are not normally subdivided into areas smaller than 5%.

6.7.2.2 A rating for each inspected item is obtained by multiplying the area demerit rating by the demerit value assigned in Table II and summing all such results to account for 100% of the item being inspected. In the event that more than one type of deposit is present on the area being inspected, the rating for that area item is the total of the individual rating values. The deposit rated is that which is visible without the removal of another deposit. Double ratings, such as sludge over varnish, are not used. The rating for the test bearing is obtained by taking a sum of the 11 rated areas and dividing by 11. The rating for each item is then modified by multiplying by the assigned weight factor which will yield a demerit rating as follows:

Major item	Rating	Factor	Demerits
End	X_1	1	X_1
Spacer and nut	X_2	2	$2X_2$
Heater mount front	X_3	3	$3X_3$
Heater mount rear	X_4	3	$3X_4$
Seal plate	X_5	1	X_5
Test bearing	X_6	5	$5X_6$

6.7.2.3 The overall deposit rating is the sum of the major item demerits divided by six.

6.7.2.4 After test, a rating of the interior test-oil sump wall and bottom is made. This rating, which is not included in the calculation of the overall test rating, is reported in terms of the type of surface deposits present and the area covered by each deposit rather than numerical demerits.

6.8 Metal Specimen Preparation.

6.8.1 After test, the metal corrosion specimens are disassembled from the holder and solvent-rinsed to remove residual test oil. (The specimens are stored in *n*-heptane if final processing is delayed). The individual specimens are *n*-heptane swabbed using a series of cotton swabs or pads until clean pads are noted. The coupons are then rinsed in clean *n*-heptane and acetone, air-dried, and weighed to the nearest 0.1 mg.

6.8.2 Significant specimen weight change is reported in mg/cm^2 ignoring edge areas in the calculation of exposed surface area. A significant weight change is defined as a weight variation of . 0.20 mg/cm^2 or more.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 As required by the specification.

9. PRECISION

9.1 Precision data for this test is reported in AFAPL-TR-70-8, Volume I, Deposition and Degradation Characteristics of Aircraft Turbine Engine Lubricants.

Copies are available through DDC (No. AD-871991).

Method prepared by:

Air Force-AFWAL/POSL - 1986

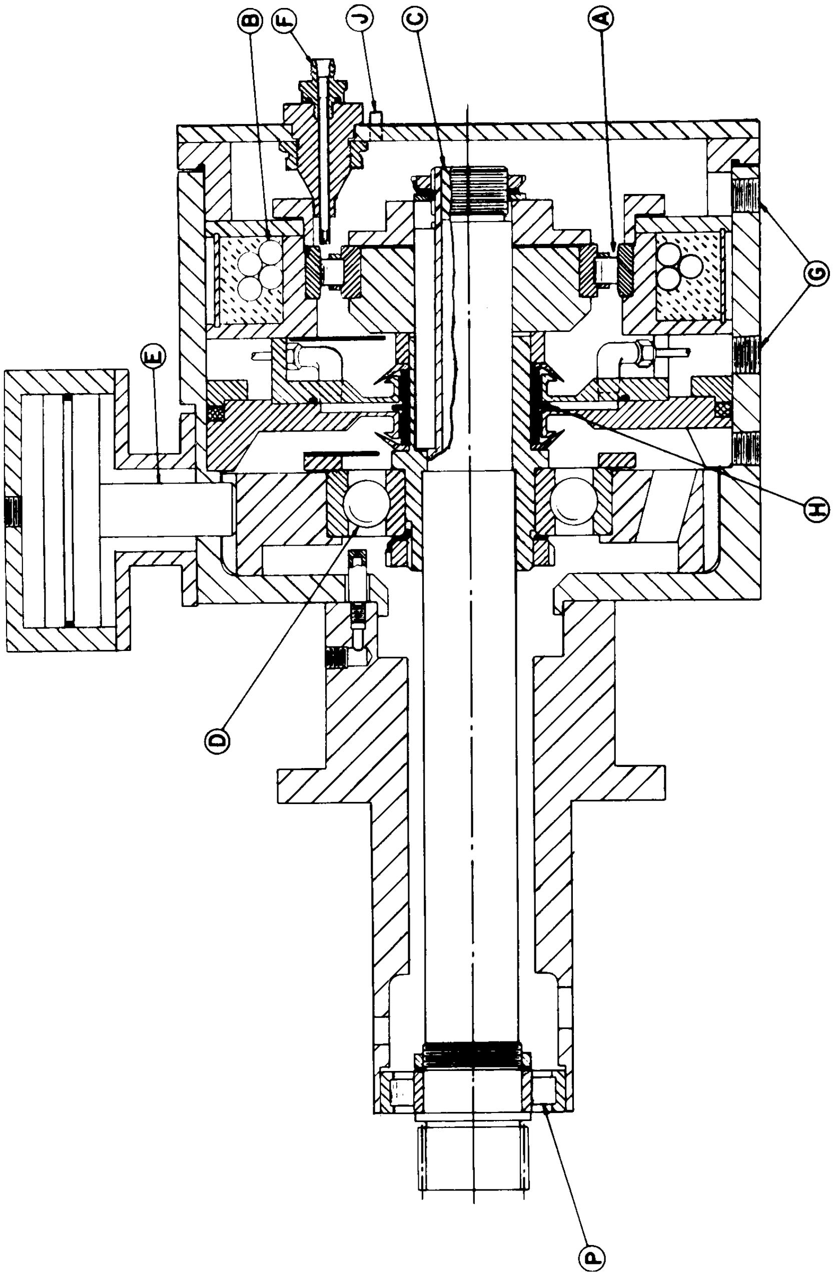


FIGURE 1. Cross section of 100-mm roller bearing machine.

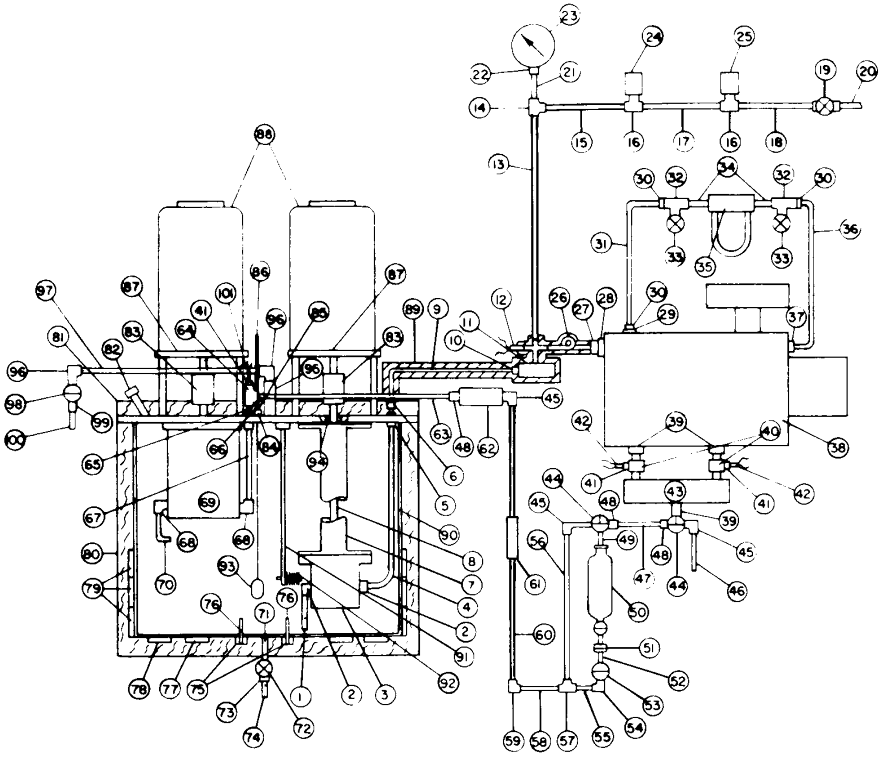


FIGURE 2. Schematic of test-oil system.

Identification of test-oil system parts.

Item No.	Description	Number required
1	3/8-in. tube, (A-B-67043)[1]	1
2	3/8-in tube, X 3/8-in NPT male elbow	2
3	Test-oil pump, Brown & Sharp, Model 00, [2] modified (A-B-67063)	1
4	3/8-in tube, approximately 6-1/2 in long	1
5	3/8-in tube bulkhead union	1
6	3/8-in tube ¼-in tube reducer	1
7	Standoff, test-oil pump (B-6-69001) & (A-6-69002)	1
8	Shaft extension (A-B-67052)	1
9	¼-in tube, approximately 8 in long	1
10	¼-in tube 1/4-in NPT male connector	1
11	Test-oil-in filter (B-B-68060)	1
12	Thermocouple (A-B-67044)	1
13	¼-in tube, as required for installation	1
14	¼-in tube tee	1
15	¼-in tube, as required for installation	1
16	¼-in tube X ¼-in tube X 1/8-in NPT male branch tee	2
17	¼-in tube, as required for installation	1
18	¼-in tube, as required for installation	1
19	Valve, 43S4-316 ball valve, Whitey Research Tool Co.[2]	1
20	1/4-in tube, as required for installation	1
21	1/4-in tube, as required for installation	1
22	¼-in tube X ¼-in NPT female connector	1
23	0 to 60-psi pressure gage	1
24	Pressuretrol, L404A, 5- to	1

	150-psi range, Minneapolis-Honeywell Co.[2]	
25	Pressuretrol, L404B, 5- to 150-psi range, Minneapolis-Honeywell Co.[2]	1
26	¼-in tube (A-B-68115)	1
27	Test-oil jet (A-B-67046)	1
28	Test-oil jet guide, IRBB-6214, Erdco Engineering Co. [2]	1
29	Vent plug, IRBB-6067, Erdco Engineering Co. [2]	1
30	¼-in tube X 1/8-in NPT male connector	3
31	¼-in tube, as required for installation	1
32	1/8-in NPT branch tee	2
33	Valve, 43F2-316 ball valve, Whitey Research, Tool Co. [2]	2
34	1/4-in X 1/8-in NPT hex reducing nipple	2
35	Manometer, 6 in Meriam Model 10AA25WM **	1
36	Manometer, 6 in Meriam Model 10AA25WM **	1
37	1/4-in tube X 1/8-in NPT 45" male elbow.	1
38	100-mm bearing machine IRBB-6200, Erdco Engineering Co.	1
39	**	3
40	3/8-in NPT hex close nipple	2
41	3/8-in NPT tee, modified (B-B-67040) 1/4-in tube X 3/8-in NPT male connector, modified	3
42	(A-B-67053)	2
43	Thermocouple (A-B-67044)	1
44	Accumulator (B-B-67040) 3-way valve, 3229T, stainless steel, 3/8-in NPT, Quality	2
45	Controls, Inc. **	3
46	1/2-in tube X 3/8-in NPT male elbow	1

47	1/2-in tube (as required for sampling)	1
48	1/2-in tube, 8 in long	3
49	1/2-in tube X 3/8-in NPT male connector	1
50	3/8-in nipple, 4 in long	1
51	Funnel, 7253-20, Ace Glass Co. **	1
52	Clamp, 7666-15, Ace Glass Co. **	1
53	Stainless steel socket, 7658-40, Ace Glass Co. ** 2-way valve, 2229S, stainless steel, 1/2-in NPT, Quality	1
54	Controls, Inc., **	1
55	1/2-in tube X 1/2-in NPT male elbow	1
56	1/2-in tube, approximately 2-1/2 in long	1
57	1/2-in tube, approximately 23 in long	1
58	1/2-in tube union tee	1
59	1/2-in tube, approximately 2-1/2 in long	1
60	1/2-in tube union elbow	1
61	1/2-in tube, approximately 42 in long	1
62	Air cooler (B-B-68053)	1
63	Test-oil-out filter (A-B-67047)	1
64	1/2-in tube, approximately 2-1/2 in long	1
65	1/2-in tube X 3/8-in NPT female X 1/2-in long tee	1
66	1/2-in tube, approximately 2-in long	1
67	1/2-in tube bulkhead union. 1/2-in tube, approximately 7-1/2 in long	1
68	1/2-in tube X 1/2-in NPT male elbow	2
69	Scavenge pump, Brown & Sharpe, Model 2S** equipped with Viton O-ring in mechanical seal and special	1

	Graphalloy carbon bearings, modified (A-B-67062)	
70	1/2-in tube (A-B-67055)	1
71	1/4-in NPT hex nipple, 2 in long	1
72	2-way valve, 2229S, stainless steel, 1/4-in NPT, Quality Controls, Inc. **	1
73	3/8-in tube X 1/4-in NPT male connector	1
74	3/8-in tube, as required for installation	1
75	1/8-in tube X 1/8-in NPT thermocouple connector, 2-2 FH4BZ-SS Parker CPI	2
76	Thermocouple (A-B-67054)	2
77	Ring heater, Edwin L. Wiegand Co., Chromalox** No. A-70-660 watts, 240 V	1
78	Ring heater, Edwin L. Wiegand Co., Chromalox** No. A-90, 1000 watts, 240 V	1
79	Band heater, Edwin L. Wiegand Co., Chromalox** No. SE-3801, 1000 watts, 240 V, bent lengthwise to clamp on 12-in-diameter cylinder with mounting tabs bent out to allow securing element to cylinder with one bolt	3
80	Insulation, Johns-Manville Co., Spinglas Type ** 102, 1-1/2-in thick	
81	Insulation, Johns-Manville Co., Marinite 23A, ** 2-in thick	
82	1-1/2-in NPT pipe cap	1
83	Coupling, Lovejoy, Model L070, 5/8-in bore **	2
84	Oil level rod guide (A-B-67051)	1
85	5/16-in tube X 1/4-in NPT male connector with Teflon ferrule	1
86	Oil level indicator tube	1

	(A-B-68114)	
87	Motor stand (D-B-67016)	2
88	Variable speed motor, Boston Gear Works, ** V-33B Ratiotrol motor Speed Control, 1/3-hp, 115-volt and No. V-93300, face mounted, shunt-wound DC drip-proof motor	2
89	Insulation, Carborundum Co., Fiberfrax Lo-Con, ** 6-lb/cu ft density, 1/4-in thick	
90	Test-oil sump (D-B-67016)	1
91	Metal specimen shaft (A-B-67049)	1
92	Metal specimens (see Fig. 16)	1
93	Oil level float and rod, Chicago Float Works. ** Type L, 2-in X 3-in, 302 stainless steel with 1/8-in tube rod, 14-in long	1
94	Pump shaft seal, Torostel 50009 or equivalent	1
95	Sump vent port (A-B-67060)	1
96	3/8-in tube X 1/4-in NPT male elbow	2
97	3/8-in tube approximately 8-in long	1
98	2-way valve, K-702-GG6D aluminum 1/4-in NPT, Kohler Co.**	1
99	3/8-in tube X 1/4-in NPT male connector	1
100	3/8-in tube, as required for installation	1
101	Thermocouple (A-B-67044)	1
*All tubing, tubing fittings, and pipe fittings listed are AISI type 304 or 316 stainless steel. **Or approved equivalent.		

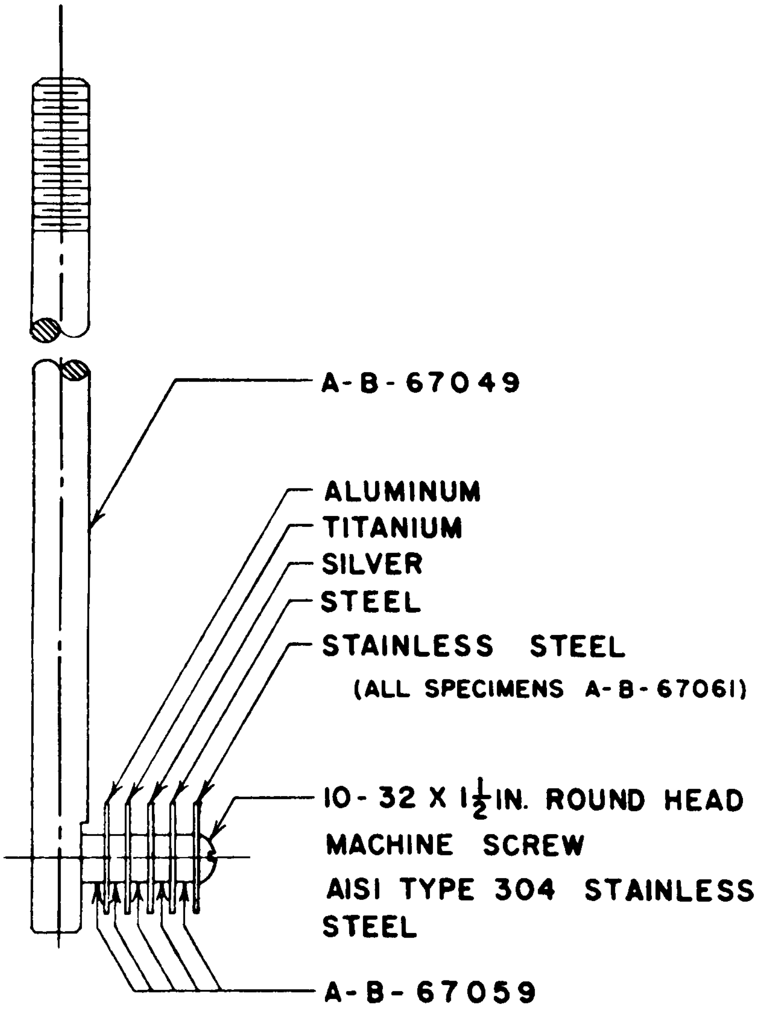
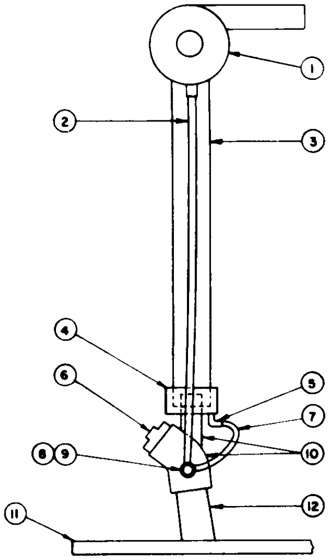


FIGURE 3. Metal specimen assembly.

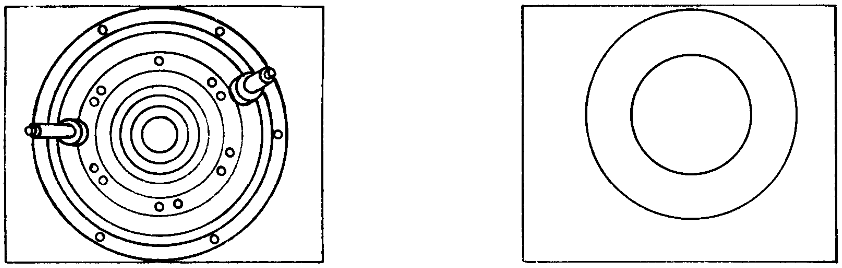


IDENTIFICATION OF VENT PIPE BLOWER ASSEMBLY PARTS

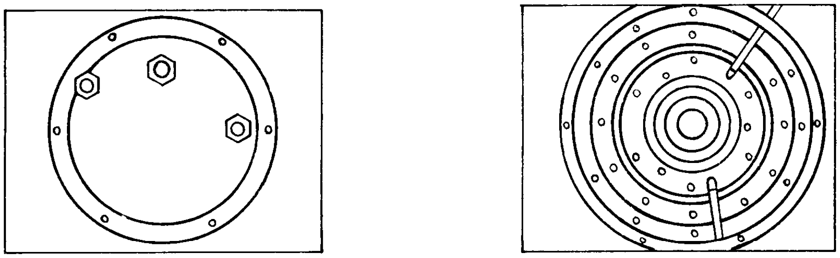
Item No.	Description	No. Required
1	Blower (B-B-68055)	1
2	3/8-in. tube, approximately 28-in. long	1
3	Vent pipe (B-B-68056)	1
4	Vent pipe cup (A-6-69008)	1
5	1/4-in. tube X 1/8-in. male elbow	1
6	1-1/2-in. NPT pipe cap	1
7	1/4-in. tube, approximately 6 in. long	1
8	3/8-in. male run tee	1
9	3/8-in. X 1/2-in. tube end reducer	1
10	Sump-oil fill port adapter (B-6-69009)	1
11	Sump cover	1
12	Sump-oil fill port	1

All tubing, tubing fittings, and pipe fittings listed are AISI Type 304 or 316 stainless steel.

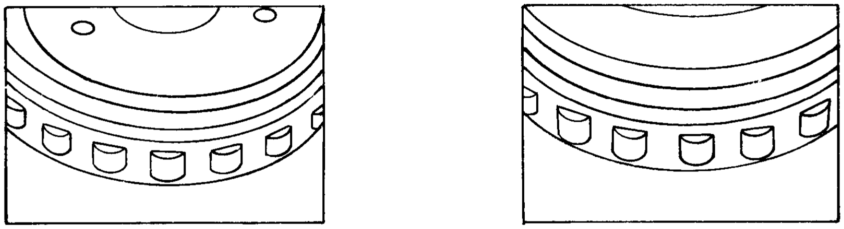
FIGURE 4. Schematic of vent pipe blower assembly.



BEARING COMPARTMENT HEATER MOUNT



END COVER SEAL PLATE



TEST BEARING - 0° POSITION TEST BEARING - 180° POSITION

TEST BEARING - OUTER RACE, 0° POSITION TEST BEARING - OUTER RACE, 180° POSITION

FIGURE 5. Typical diagrams of disassembled rig after test.

Method 3452.2
September 30 1986

BEARING COMPATIBILITY OF TURBINE OILS

1. SCOPE

1.1 This method is used for determining the bearing compatibility (lacquering, deposits, and corrosion) and stability of MIL-L-17331 turbine lubricating oils when subjected to an endurance test.

2. SUMMARY

2.1 A Journal is operated at specified speed in a babbitt metal bearing lubricated with the test lubricant for 200 h; examining the test bearings and checking the lubricant for changes in viscosity, total acid number, and carbon residue; and computing the work factor from the changes noted.

3. SAMPLE SIZE

3.1 4 L (1 gal) of the oil to be used in each test. (An additional 20 L (5 gal) should be on hand for use in repeating the 3-L (3-qt) flushing procedure as required.)

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Test equipment illustrated schematically in Figure 1 consist of:

a. Journal (H), shaft material, 4340 steel, 2.750-inch diameter (at bearing surfaces) and hardened to a Rockwell "C" hardness of 40-45, finish ground not to exceed 0.041 µm (16 µin) rms.
b. Bearings (I, J, and K) babbitt metal in split bearing housing (to accommodate journal), size 6.9886-6.9936 cm (2.7514-2.7534 in) (vertical diameter when installed) by 4.60 cm (1-13/16 in) long. (Bearings should be free of scratches or other blemishes). The bearing material over steel backing is 0.013 to 0.025 cm (0.005 to 0.010 in) thick tin base babbitt (SAE 12 babbitt); composition as per SAE standard 1460 is: Sn, 88.25% (minimum); Sb, 7.0-8.0%; Pb, 0.50% (maximum); Cu, 3.0-4.0% (maximum); Fe, 0.08% (maximum); As, 0.10% (maximum); Bi 0.08 (maximum), Zn, 0.005 (maximum); Al, 0.005 (maximum); and others 0.20 (maximum) total. Bearings of this size and composition may be obtained from Mogul-Bower Bearings, Incorporated, Detroit 13, Michigan under the following part numbers: Upper half B-10994, Lower half B-10995.
c. Loading Device (O), hydraulic cylinder or other suitable unit capable of maintaining a load of 1520 . 20 gage kPa (220 . 3 psig) of projected bearing area on the loading bearing (j). (This loading equivalent to approximately 4,400 N [1000-lb force] on the loading yoke.)
d. Drive unit (f), to drive journal at a speed of either 1750 or 3500 rpm.

e. Oil system, recirculating, 4-L (l-gal) capacity sump, equipped with 3.8 L/min (l gpm) pump and bypass line to sump, to circulate 1.9-2.3 L/min (0.5-0.6 gpm) of test lubricant to support and test bearings.

f. Oil temperature control. A thermistor (L) or other suitable temperature sensing device mounted to contact steel shell of upper half of test bearing; this controls the sump heater. Bearing temperature is monitored by spring-loaded thermocouple (M) in contact with steel shell of upper half of test bearing. Temperature of oil supply to bearing is monitored from thermocouple (P). High-temperature cutoff switch (C) to sump is to insure against overheating of oil. Temperature of oil in sump is recorded from thermocouple (D). High temperature cutoff in the recorder is set for 150°C (300°F).

NOTE 1: A suitable cover for the machine is useful for controlling temperatures during winter months. A wooden box can be used. Thermocouples placed inside and outside the box serve for recording these ambient temperatures.

g. Oil flow rate and pressure control. Oil supply to bearings (sum of oil flow to three bearings) is observed at flowrater (T). Oil supply pressure to bearings is observed on gage (Q). A minimum oil pressure to bearings of 13.7 gage kPa (2 psig) pressure is insured by low- pressure cutoff switch (S).

NOTE 2: Detailed drawings of test equipment may be obtained from US Navy Marine Engineering Laboratory, Annapolis, MD.

4.2 The properties of the new lubricating oil will be determined as follows:

a. Viscosity in cSt at 38°C (100°F), 54°C (130°F), and 99°C (210°F), by method D 445, ASTM Standards.
b. Carbon residue, percent, by method D 524, ASTM Standards.
c. Total acid number by method D 974, ASTM Standards.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Naphtha; aliphatic (TT-N-95), type II [1,4,0].

6. PROCEDURE

6.1 Determine and record the following properties of the new lubricating oil:

a. Viscosity
b. Carbon residue
c. Total acid

6.2 Subject the oil to endurance test as follows:

a. Drain any old oil remaining in the system; remove sump and wipe clean with lintless wiping cloths and naphtha; remove bearing caps, bearings, and shaft from machine; wipe shaft with lintless wiping cloths and naphtha to remove stains.
b. Spread film of test oil on journal and three sets of new bearings; install the shaft and bearings; use a torque of 115-129 N·m (85-95 lb-ft) on bearing cap bolts.
c. Flush run. Temporarily install a 10 micrometer filter between the oil sump and pump inlet. (Filter should be a clean or new filter.) Place approximately 3 L (3 qt) of the test oil in sump. Start pump and turn on sump heater, and slowly adjust oil flow to 1.9-2.3 L/min (0.5-0.6 gpm) (to avoid excess oil pressures). Start motor at 1750 rpm. Allow system to operate for 30 min. During the last 10 min apply a load (half that specified for test) to check out the system functioning. Shut down the machine by first unloading the bearing and then stopping the motor drive. Shut off the oil pump and heater. Drain all oil from the system and wipe sump clean with lint-less wiping cloths. Remove the oil filter from the system and install direct connect from pump inlet to sump.

NOTE 3. A single flush run, following the above procedure, will usually provide sufficient system cleaning.

d. Test oil. Place 4 L (l gal) of test oil in clean sump and start heater. Start oil pump and adjust flow with bypass valve to give 1.9-2.3 L/min (0.5-0.6 gpm). Start motor drive at speed of 3500 rpm and gradually load bearing (using 2 or 3 min.) with loading device to yield a load of 1517 gage kPa (220 psig) of projected bearing area. As the system temperature comes to equilibrium, adjust sump temperature controller to give a bearing temperature of 138° ± 0.5°C (280° ± 1°F) which shall be maintained during the test. Operate the machine under these conditions for a period of 200 h. During this time continuously record bearing temperature, temperature of oil supply to bearing, and temperature of oil in sump. Observe and record (daily) oil flow rate and oil pressure to bearings. Oil flow rate to bearings shall be adjusted during test period to maintain 1.9-2.3 L/min (0.5-0.6 gpm) to bearings.

6.3 Inspection of bearings at end of 200-hour endurance test. Dismantle all journal bearings and rinse in naphtha. Observe bearing surfaces for evidence of deposits, corrosion, wiping, or other changes. Particular reference is to be made to loaded (center) bearing. Report the observation for all bearings.

6.4 At the end of the 200 hour endurance test, determine the properties of the used lubricant, as given in paragraph 6.1.

7. CALCULATIONS

7.1 Compute the viscosity, carbon residue, and neutralization number work values of the test oil. The values shall be computed and reported as follows:

$$a.\ Viscosity\ work\ value = 1.00 - \frac{5\,(A - B)}{B}$$

where:
A = viscosity after endurance test, SUS
B = viscosity before endurance test, SUS

Viscosity work value is determined by averaging the three values obtained from individual calculations based on viscosity data at 38°C (100°F), 54°C (130°F), and 99°C (210°F).

$$b.\ Carbon\ residue\ work\ value = \frac{0.80 - D}{0.80 - C}$$

where:
C = carbon residue before endurance test.
D = carbon residue after endurance test.

$$c.\ Total\ acid\ number\ work\ value = \frac{1.01 - F}{1.01 - E}$$

where:
E = neutralization number before endurance test.
F = neutralization number after endurance test.

d. Average the work values obtained and this value is called the work factor.

8. REPORTING

8.1 Report the three work values and the work factor.

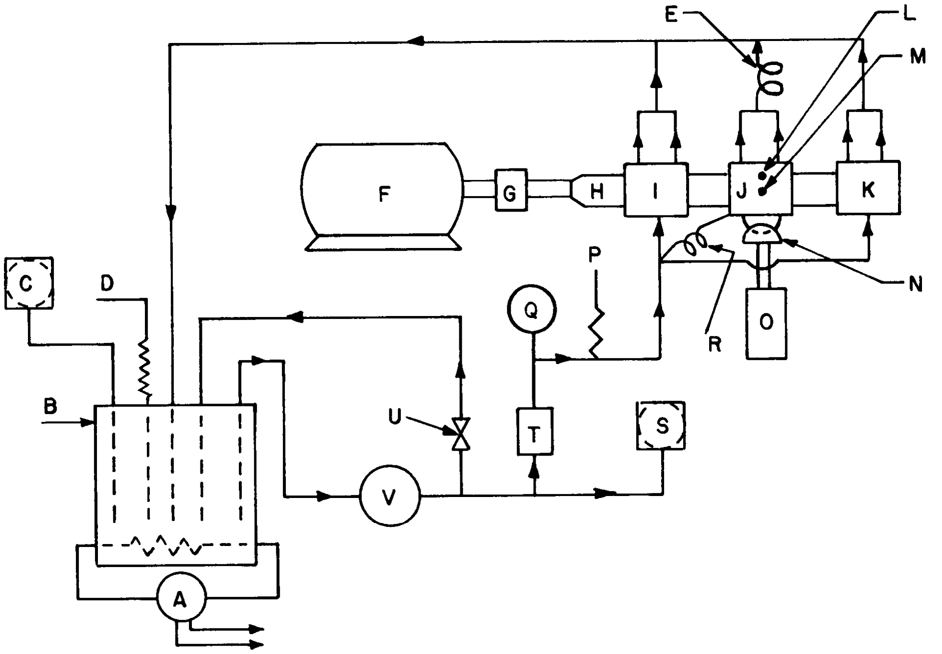
9. PRECISION

9.1 Repeatability. The appearance of the test bearing is of primary importance. A quantitative rating or comparison scale for this characteristic has not yet been established. There has, however been no difficulty in reaching conclusions with respect to excellent, borderline or failing oils. Experience to date indicates that duplicate work factor values can be expected not to differ by more than . 0.02 units from the mean.

9.2 Reproducibility. This has not been established.

Method Prepared by:

Navy - SH - 1986



A	Variac, to temperature controller.
B	Electric glue pot, 110-v ac, 1000 watts (4 L capacity).
C	Temperature control, remote bulb mercury switch, high-temperature cutoff.
D	Thermocouple (sump oil temperature) to reorder.
E	Lube oil return from loading bearing (nonrestraining connection).
F	Motor, 2.5-5.0 hp, 220/440-v ac, 1750 and 3500 rpm (comply NEMA standards).
G	Flexible coupling.
H	Shaft (journal).
I	Support bearing.
J	Loading bearing.
K	Support bearing.
L	Thermistor (bearing temperature) glue pot oil temperature control.
M	Thermocouple (bearing temperature) to recorder, high-temperature cutoff at 149° C (300° F).
N	Bearing loading yoke assembly.
O	Loading device for loading bearing.
P	Thermocouple (oil to bearings) to recorder.
Q	Oil pressure gage.
R	Lube oil to loading bearing (non-restraining connection).
S	Low-pressure cutoff switch.
T	Flowrator, gpm.
U	Bypass valve.
V	Pump-motor unit.

FIGURE 1. Oil stability tests arrangement.

Method 3456.2
September 30, 1986

CHANNELING CHARACTERISTICS OF LUBRICANTS

1. SCOPE

1.1 This method is used for determining the channeling characteristics of lubricants at low temperature.

2. SUMMARY

2.1 The test consists of storing the sample for 18 h at the temperature required by the specification, cutting a channel in the lubricant with a metal strip, and determining whether the lubricant flows together to cover the bottom of the container within 10 s.

3. SAMPLE SIZE

3.1 Approximately 650 mL of lubricant to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Steel strip, square end (channeling tool), approximately 0.3 × 2 × 23 cm.

4.2 Thermometer, HB Instrument Co., No. 22360, or equivalent.

4.3 Container, flat bottom, approximately 9 cm I.D. by 11 cm high, with 2.5 cm (minimum) legs, and with cover to support thermometer in center and steel strip at side of container. (See Figure 1).

4.4 Heating bath, 46° to 48°C.

4.5 Cooling bath (non-liquid), capable of maintaining the specified temperature within 1°C, size sufficient to accommodate container within 3 cm (minimum) clearance on all sides.

5. MATERIALS

5. CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

6. PROCEDURE

6.1 Prepare test setup as follows:

a. Fill container to within 1.3 cm of top with specimen, place in heating bath, and allow sample to reach 46°C (5°F). (If a liquid bath is used, adjust bath level to height of sample.)
b. When sample reaches 46°C, cover container and remove from bath; allow the samples to cool to 22° ± 1°C.
c. Position the thermometer so that the bulb is just below the surface. Insert the steel strip into the slit in the cover so that it is in a vertical position in contact with the wall of the container, with the end resting on the bottom of the container. Immediately place the sample in the cooling bath.

6.2 Store container in cooling bath without interruption for 18 ± 2 h. Begin timing the storage period immediately after placing the sample in the cooling bath.

6.3 At end of storage period, remove container from cooling bath, and within 30 s cut a channel in the sample as follows:

a. Check sample temperature and make sure that it is within ±1°C (±2°F) of specified storage temperature.
b. Without moving steel strip, remove cover and thermometer from container.
c. Note time to nearest second, and within 5 s scrape the vertically-held steel strip all the way across bottom of container.

6.4 Within 10 s after cutting the channel (see 6.3), check to determine whether sample has flowed back and completely covered bottom of container. If it has, note it as non-channeling; if not, note it as channeling.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the result as channeling or non-channeling.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986

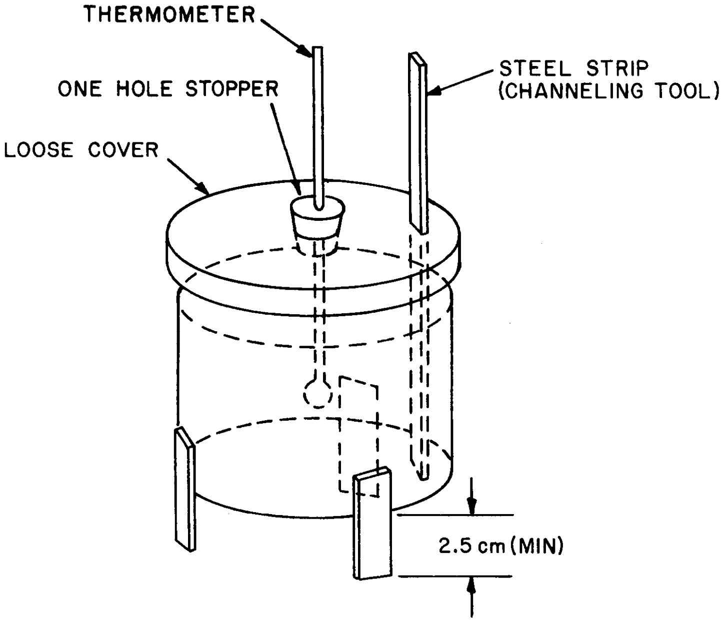


FIGURE 1. Test setup.

Method 3457.2
September 30, 1986

HYDROLYTIC STABILITY

1. SCOPE

1.1 This method is used for determining the resistance of a finished oil to reaction when in contact with water.

2. SUMMARY

2.1 The test consists of tumbling under specified conditions, a mixture of the oil and water in a bottle containing a copper strip, and then testing for changes in the oil, water, and copper.

3. SAMPLE SIZE

3.1 Approximately 125 g of the oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Standards.

4.1.1 ASTM D 445 - Kinematic Viscosity of Transparent and Opaque Liquids.

4.1.2 ASTM D 974 - Neutralization Number by Color - Indicator Titration.

4.1.3 ASTM D 1500 - ASTM Color of Petroleum Products.

4.2 Oven, convection, air; controllable to . 0.5°C (. 1°F) in temperature range of 66° and 93°C (150° and 200°F).

4.3 Fluid container (carbonated beverage bottle), 210 mL (7 oz), capable of being sealed with a cork-lined steel cap.

4.4 Tumbling mechanism (for rotating the fluid container end-over-end at 5 rpm, inside the oven).

4.5 Brush, short-bristled, stiff (typewriter cleaning brush, or equal).

4.6 Gooch crucible.

4.7 Centrifuse, 1500 rpm.

4.8 Centrifuge tubes, cone-shaped, 100 mL.

4.9 Microscope, 20-diameter magnification.

4.10 Balance, sensitivity . 0.2 mg.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Emery paper, 00 or finer.

5.3 Litmus paper.

5.4 Petroleum ether (O-E-751) [1,4,0].

5.5 1,1,1-Trichloroethane (methyl chloroform), stabilized (MIL-T-81533) [2,1,0].

5.6 *n*-Hexane (technical grade) [1,3,0].

5.7 Sodium sulfate, anhydrous (ACS) [u,u,u].

5.8 Copper strip (ASTM B 187/B 187M, ASTM B 272), 16 to 22 gage (B&S), 1.3 □ 5.1 cm.

6. PROCEDURE

6.1 Determine the acid number of the oil by ASTM D 974.

6.2 Determine the kinematic viscosity of the oil by ASTM D 445.

6.3 Heat the oven to the temperature required by the specification.

6.4 Weight (to within 0.5 g) 75 g of the sample and 25 g of distilled water and place in the clean fluid container (see 4.3).

6.5 Using the filter paper to prevent fingerprints during handling, polish all surfaces of the copper strip with the fine emery paper.

6.6 Wash the copper strip in warm petroleum ether or *n*-hexane, dry, and weigh to within 0.2 mg, and immediately place the strip in the container.

NOTE 1: Caution. Petroleum ether and *n*-hexane are both flammable. Use these materials in a well ventilated area. Do not breathe their fumes nor permit them to come in contact with the skin. Keep all flames away from these materials.

6.7 Cap the bottle (with inert seal in place), and mount it in the tumbling mechanism in the oven.

6.8 Allow the mechanism to rotate the container end-over-end at 5 rpm for the time required by the specification.

6.9 After the container has rotated for the required time, filter the liquid contents through a tared Gooch crucible lined with a thin layer of asbestos. Save the filtrate. (The operator should wear heavy asbestos gloves and a face shield while handling the hot beverage bottle). Rinse the copper strip and beverage bottle with 50 mL of distilled water and 50 mL of petroleum ether and filter through the Gooch crucible. Save only the water washings and add them to the filtrate.

6.10 Process any insolubles that remain in the crucible, as follows:

a. Wash the precipitate with 50 mL of distilled water, and add the washings to the filtrate.
b. Wash the precipitate with 50 mL of petroleum ether, and dry the crucible at 66° to 93°C (150° to 200°F).
c. Determine and record the increase in weight of the crucible to within 0.2 mg.

6.11 Process the filtrate as follows:

a. Weigh a clean centrifuge tube to within 0.2 mg.
b. Transfer the filtrate to the tube, and centrifuge the tube for 15 min at 1500 rpm (min.).
c. After centrifuging, decant the oil and water layers to suitable separate containers.

6.12 Process the oil layer from the centrifuge tube as follows:

a. Wash the oil with 25 mL portions of distilled water until the water is neutral to litmus. Save the water from this washing.
b. Examine the oil for insolubles, and remove any that still exist, using a combination of filtration, centrifuging, and solvent extraction as necessary.
c. When all insolubles have been removed, dry the oil with anhydrous sodium sulfate.
d. Determine the acid number of the oil by ASTM D 974, and calculate the change from the original acid number.
e. Determine the kinematic viscosity of the oil by ASTM D 445, and calculate the change from the original viscosity.

f. Determine the color of the oil by ASTM D 1500.

6.13 Process the water layer from the centrifuge tube and the washings as follows:

a. Combine the water from the centrifuge tube with the water from the washings.
b. Determine the acid number of this water (based upon the original weight of the oil sample) by ASTM D 974.

6.14 Determine the weight of any insolubles remaining in the centrifuge tube as follows:

a. Add 50 mL of new test oil to the tube, and centrifuge until the oil is clear.
b. Carefully decant the oil, and dry the tube in the oven to remove all traces of water.
c. Again wash the contents of the tube with new test oil, centrifuge until clear, and decant the oil.
d. Wash the residue with petroleum ether (to remove the remaining oil); centrifuge, decant, and dry the tube in the oven.
e. Determine the increase in weight of the tube to within 0.2 mg.

6.15 Calculate the percentage (by weight) of the insolubles in the oil (see 7.1).

6.16 Remove the copper strip from the fluid container, and process it as follows:

a. Wash the strip with petroleum ether, warm n-hexane, and warm 1,1,1-trichloroethane and allow it to dry in air. See note 1 (see 6.6) and note 2.

NOTE 2: Caution. 1,1,1-Trichloroethane is toxic. Do not permit it to come in contact with the skin or breathe its fumes. Use only in well ventilated area.

b. Determine the weight change of the strip to within 0.2 mg. Calculate the change in weight per square centimeter of total surface.
c. Examine the strip under 20X magnification, and record any visible signs of corrosion.
d. Again wash the strip with petroleum ether, warm *n*-hexane, and warm 1,1,1-trichloroethane. While washing, scrub the strip with the short-bristled brush. Allow the strip to air dry, and reweigh to within 0.2 mg.
e. Repeat this washing until the strip reaches a constant weight.
f. Determine the weight loss of the strip (after brushing) from its originally polished condition.
g. Calculate the loss in weight per square centimeter of total surface.

7. CALCULATIONS

7.1 Calculate the percentage (by weight) of the insolubles in the oil as follows:

$$\text{Percent insolubles} = \frac{100\,(A+B)}{W}$$

where:
A = increase in weight of Gooch crucible.
B = increase in weight of centrifuge tube.
W = original weight of oil sample.

8. REPORTING

8.1 Report the following:

a. weight change of the metal strip before brushing.
b. weight change of the metal strip after brushing.
c. change in acid number of the sample.
d. acid number of the water layer after hydrolysis.
e. change in viscosity of the oil.
f. weight of insolubles formed during hydrolysis.
g. color of the oil after hydrolysis.
h. general appearance of the sample after hydrolysis.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - AR - 1986

Method 3458.1
September 30, 1986

LOW TEMPERATURE STABILITY TEST FOR OIL

1. SCOPE

1.1 This method is used for determining the tendency of components of finished oil blends to be incompatible or to form gels during low-temperature exposure.

2. SUMMARY

2.1 The test consist of refrigerating the sample for 72 h at the temperature required by specification, and examining it visually for evidence of gelling, separation, crystallization, and clouding.

3. SAMPLE SIZE

3.1 Approximately 100 mL of oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Sample bottle, clear-glass 120 mL, with stopper.

4.2 Cold box, capable of maintaining the specified temperature within "6°C.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

6. PROCEDURE

6.1 Pour sample into bottle, and store bottle for 72 h at the temperature of ("6°C) required by the specification.

6.2 At the end of 72 h, remove the container from the cold box, and immediately examine the sample visually for evidence of gelling, separation, or crystallization.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the oil as unstable at the specified temperature, if there is any evidence of gelling, separation, or crystallization; if there is no such evidence, report the oil as stable. Also report the presence of dense cloud, if any, that does not settle out.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986

Method 3459.1
September 30, 1986

LOW TEMPERATURE STABILITY

1. SCOPE

1.1 This method is used for determining the stability (at low temperature) of finished fluid blends.

2. SUMMARY

2.1 The test consists of storing a sample for the time and at the temperature required by the specification, agitating it, comparing its turbidity with a standard, and checking it for evidence of gelling, crystallization, or solidification.

3. SAMPLE SIZE

3.1 Approximately 240 mL of test fluid.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Cooling apparatus, capable of maintaining the specified temperatures.

4.2 Sample bottle (2), clear-glass, 250 mL, with cork stoppers.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Methyl-alcohol-glycerine (technical grade), 1:1 solution (by volume) [1,3,0].

5.3 Turbidity standard, prepared within 30 min of use, as follow:

a. Pour 25 mL of 0.00322 M barium chloride into a 250-mL volumetric flask [0,0,2].
b. Add 125 mL of 0.050 N sulfuric acid, and shake well to insure complete precipitation [3,0,2].
c. Add approximately 25 mL of 1 N sodium hydroxide to make solution alkaline [3,0,1].
d. Add distilled water to bring total volume to 250 mL.

e. Pour solution into a sample bottle, and seal it tightly with stopper.

NOTE 1: For red hydraulic liquids, in place of plain distilled water, use distilled water containing 200 ppm red dye (National Erie Bordeaux B, manufactured by National Aniline Division of Allied Chemicals and Dye Corporation, or equivalent).

6. PROCEDURE

6.1 Clean sample bottles, and dry at approximately 100°C for 24 h (min.).

6.2 Pour sample into bottle, and seal bottle tightly with stopper.

6.3 Store sample at the specified temperature for the specified number of hours.

6.4 Before end of storage period, cool the methyl-alcohol-glycerine solution to -54°C (-65°F).

6.5 Within 5 min of making comparison, shake turbidity standard vigorously for 10 s, and pour into the other sample bottle.

6.6 At the end of the storage period, remove sample from cold storage and shake vigorously for approximately 10 s.

6.7 Dip specimen into methyl-alcohol-glycerine solution to eliminate frosting (if necessary), and within one minute of removal from cold storage, compare sample visually with turbidity standard.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report turbidity of sample as less than, more than, or equal to the standard. Also report any evidence of gelling, crystallization, or solidification of the sample.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:
Army - NADC - 1986

Method 3463.2
September 30, 1986

STABILITY OF GREASE IN HOT WATER (WATER IMMERSION)

1. SCOPE

1.1 This method is used for determining the stability of grease in the presence of hot water.

2. SUMMARY

2.1 The method consists of immersing the sample in boiling water for 10 min, and checking the water for cloudiness or other evidence of emulsification of the sample.

3. SAMPLE SIZE

3.1 Approximately 5 g of grease to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Beaker, 600 mL.

4.2 Stirring rod, glass.

5. MATERIAL

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Distilled water.

6. PROCEDURE

6.1 Place approximately 500 mL of distilled water in the beaker, and heat it to gentle but active boiling.

6.2 Apply a single lump or globule of the sample to one end of the stirring rod.

6.3 Immerse the sample in the boiling water for 10 min while visually examining the water, and note the presence of cloudiness, or other evidence of emulsification of the sample.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the presence of cloudiness, or other evidence of emulsification of the sample.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Navy - NADC - 1986

Method 3465.1
September 30, 1986

STORAGE STABILITY TEST OF FLUIDS AND LUBRICANTS

1. SCOPE

1.1 This method is used for determining the storage stability of aircraft turbine lubricants and hydraulic fluids. It consists of storing the lubricant in darkness for 12 months at 23.8° " 3°C (75° " 5°F), and then inspecting it for changes in homogeneity.

2. SUMMARY

2.1 This method duplicates a typical warehouse environment, and it determines if 12 months of static storage at moderate temperatures can affect product homogeneity by producing cloudiness, sediment, suspended matter, discoloration, etc. The presence or absence of observed visual changes in homogeneity is a reliable and economical indicator of extended storage stability.

3. SAMPLE SIZE

3.1 Approximately 4 L (1 gal) of the lubricant or fluid to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Chamber, constant-temperature 24° " 3°C (75° " 5°F), free of vibration (such as caused by heavy traffic).

4.2 Jar, clear-glass, 3.8 L (1 gal), wide-mouth with cap.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment measures, and spill practices of each chemical are explained.

5.2 Aluminum foil, commercial grade.

5.3 Scouring powder, commercial grade.

5.4 Distilled water.

6. PROCEDURE

6.1 Clean the jar and cap by scrubbing with scouring powder and rinsing with tap water. Complete the rinsing with distilled water, and air dry the jar and cap (using an oven at 104°C if desired).

6.2 Line the cap with aluminum foil.

6.3 Pour approximately 3.8 L of the sample into the jar, and replace and tighten the cap.

6.4 Wrap aluminum foil around the jar to exclude all light from the sample.

6.5 Identify the contents of the glass jar (including type of sample, date stored, and any other useful information).

6.6 Store the sample for one year in the storage chamber 24° " 3°C (75° " 5°F).

6.7 At the end of the 12-month period, remove the jar from the chamber and take off the aluminum wrapper, being careful not to shake or agitate the contents.

6.8 Visually examine the sample, and note the presence of cloudiness, sediment, suspended matter, discoloration, or other changes in homogeneity.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the presence of cloudiness, sediment, suspended matter, discoloration, or other changes in homogeneity.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Air Force - SFTL - 1986

Method 3467.1
September 30 1986

STORAGE STABILITY OF LUBRICATING GREASE

1. SCOPE

1.1 This method covers a technique used for measuring the stability of a lubricating grease after a definite storage period at an elevated temperature.

NOTE: The storage period and temperature at which the grease is to be stored must be given in the specification referencing this test method.

2. SUMMARY

2.1 A determination for consistency (unworked and work penetration) is made after the storage interval and the results compared with data established prior to the storage interval.

3. SAMPLE SIZE

3.1 Approximately 1 kg of grease, sufficient to fill two standard grease cups (ASTM D 217) with test sample.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Two standard grease worker cups, grease worker, penetrometer, etc. (ASTM D 217).

4.2 Oven, gravity - convection.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

6. PROCEDURE

6.1 Fill one standard grease worker cup with the test sample and remove excess grease to insure that the surface of grease is flush with the top of the cup. (Sample for un worked penetration, see 6.6.)

6.2 Fill another cup with sufficient quantity of grease to insure over filling of the cup. (Sample for worked penetration, see 6.6.)

6.3 Cover the samples with aluminum foil.

6.4 Place the grease filled cups in an oven at the designated test temperature for the required storage interval.

6.5 After completion of storage interval, remove the cups from the oven and allow to cool to 25°C (77°F).

6.6 Conduct unworked and worked penetration measurements on the grease samples in accordance with ASTM D 217.

7. CALCULATIONS

7.1 Calculate the change in worked penetration after storage as follows:

$$C = A - B$$

where:

C = change in worked penetration
A = penetration before storage
B = penetration after storage

8. REPORTING

8.1 Report the unworked and worked penetration after storage. Report the difference in worked penetration before and after storage as the change in worked penetration.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Navy - NADC - 1986

Method 3470.1
September 30, 1986

HOMOGENEITY AND MISCIBILITY OF OILS

1. SCOPE

1.1 This test determines if an oil is and will remain homogeneous, and if it is miscible with certain standard reference oils after being submitted to a prescribed cycle of temperature changes.

2. SUMMARY

2.1 Using standard reference oils, an oil sample is tested to determine if it is and will remain homogeneous and if it is miscible with the reference oils. A sample oil is mixed thoroughly with a reference oil and heated. When the oils reach room temperature, they are observed for color and evidence of separation. The pour point is determined for each oil. A series of tests are made in cooling baths at 12°C above the expected pour point. The temperature is read at a multiple of 3°C. The test jar is removed and it is tilted to determine if there is movement of the oil. The test jar is placed in successively lower cooling baths until the oil has ceased to flow. When the oil does not flow when the jar is tilted, hold the jar in a horizontal position for 5 s and observe the oil. If the oil shows any movement under these conditions, place the jar in the cooling bath at the next lower temperature. The test is continued under these conditions until there is no movement when the jar is tilted in a horizontal position. The thermometer reading is recorded at this temperature. The samples are thawed. When the cloudiness has barely disappeared, the color and evidence of separation is recorded. After the samples reach room temperature, the thermometers are removed and the samples are heated to 232°C in an oil bath. The sample jars are corked and stored at their pour points for 18 to 24 h. When the cloudiness has barely disappeared, after heating, the color and evidence of separation is recorded. The last procedure is repeated. The data is recorded on the Homogeneity and Miscibility Test forms.

3. SAMPLE SIZE

3.1 Approximately 300 mL of oil to be tested.

4. REFERENCE, STANDARDS, AND APPARATUS

4.1 Test jar. A test jar of clear glass, cylindrical form, flat bottom, approximately 3.0 to 3.5 cm I.D. and 12 to 13 cm in height.

4.2 Thermometers. -80° to +21°C range, conforming to ASTM E 1.

4.3 Cork. To fit the test jar, bored centrally to take the test thermometer.

4.4 Jacket. Glass or metal, water tight, of cylindrical form, flat bottom, about 12 cm in depth, with inside diameter 0.9 to 1.2 cm greater than the outside diameter of the test jar.

4.5 Disk. Cork of felt, 0.6 cm in thickness of the same diameter as the inside of the jacket.

4.6 Gasket. A ring gasket, about 0.5 cm in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

4.7 Bath. A cooling bath of a type suitable for obtaining the required temperatures.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Six reference oils. Information concerning the availability of the reference oil may be obtained from USA Belvoir Research, Development, and Engineering Center, ATTN: STRBE-VF, Ft. Belvoir, VA 22060-5606.

6. PROCEDURE

6.1 Shake oil sample well and pour into six sample jars to 3.2 cm mark and one sample jar to the 7.5 cm mark. Add a reference oil to each of the six sample jars to the 7.5 cm mark.

6.2 Mix the oil thoroughly and heat to 46°C in a water bath.

6.3 After the oils reach room temperature, observe and record the color and evidence of separation.

6.4 Determine and record the pour point of each oil.

a. Maintain the temperature of the cooling bath at -1° to 2°C. Support the jacket, containing the test jar, firmly in a vertical position in the cooling bath so that not more than 2.5 cm of the jacket projects out of the cooling medium.
b. Starting at a temperature 12°C above the expected pour point remove the test jar from the jacket carefully and tilt it just enough to ascertain whether there is a movement of the oil in the test jar. Take a test thermometer reading when the

temperature is 3°C lower then the previous reading. The complete operation of removal and replacement shall require not more than 3 s. If the oil has not ceased to flow when its temperature has reached 10°C, place the test jar in the jacket in a second bath maintained at a temperature of -18° to -15°C. If the oil has not ceased to flow when its temperature has reached -7°C, place the test jar in the jacket in a third bath maintained at a temperature of -35° to -32°C. For determinations of very low pour points, additional baths should be maintained with successively lower temperature differentials of about 17°C. In each case transfer the test jar when the temperature of the oil reaches a point of 28°C above the temperature of the new bath. At no time place the cold test jar directly in the cooling medium. As soon as the oil in the test jar does not flow when the jar is tilted, hold the test jar in a horizontal position for exactly 5 s, as noted by a stop watch or other accurate timing device, and observe carefully. If the oil shows any movement under these conditions, place the test jar immediately in the jacket and repeat a test for flow at the next temperature 3°C lower.

c. Continue the test in this manner until a point is reached at which the oil in the test jar shows no movement when the test jar is held in a horizontal position for exactly 5 s. Certain lubricating oils tend to move as a whole and should be very closely observed. Record the reading of the test thermometer at this temperature, corrected for error if necessary.

6.5 Allow the samples to thaw; and when the cloudiness has barely disappeared observe and record the color and any evidence of separation.

6.6 When the samples reach room temperature, place them in an oil bath after removing the thermometers. Heat the bath to 232°C and immediately remove the samples.

6.7 Cork the samples and store them at their respective pour points for 18 to 24 h.

6.8 Remove the samples and allow them to thaw. When cloudiness has barely disappeared, observe and record the color and any evidence of separation.

6.9 Repeat the last operation when the samples reach room temperature.

6.10 Note the homogeneity and miscibility of the samples.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the data on the homogeneity and miscibility test forms.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986

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ENGINE OIL HOMOGENEITY AND MISCIBILITY TEST

COMPANY--SAMPLE NO ---------------

VISCOSITY GRADE------------------------TEST NO--

DATE-----------------

Qualified Oil Added To Sample	None	1-30	2-30	3-30	4-30	5-30	6-3-
Before Treatment							
Evidence of Separation							
Location							
Color							
Particle Size							
Color of Oil							
Cooled to Pour Point, º C							
Warmed Just Above Cloud Point							
Evidence Of Separation							
Location							
Color							
Particle Size							
Color of Oil							
Heat to 232º C cool to Pour Point Store 24 Hours Warm above Pour point							
Evidence of Separation							
Location							
Color							
Particle Size							
Color Of Oil							
Warm to Room Temperature							
Evidence of Separation							
Location							
Color							
Particle Size							
Color of oil							

ABBREVIATIONS

EVIDENCE OF SEPARATION
D - DEFINITE
N - NONE OR DOUBTFUL

LOCATION
T - NEAR TOP
B - NEAR BOTTOM
F - FILAMENT
U - UNIFORMLY DISTRIBUTED

COLOR
w - WHITE OR VERY LIGHT
y - YELLOW
B - BLACK

PARTICLE SIZE
s - SMALL, AS IN CLOUD OR HAZE
s_p - SPECKS OR LARGER PARTICLES

OIL PHASE COLOR
d - TOO DARK TO SEE THROUGH
m - TRANSPARENT BUT DARK
l - LIGHT

Method 3480.1
September 30, 1986

VOLATILITY OF BLENDED OILS

1. SCOPE

1.1 This method is used for determining the volatility characteristics of a blended oil.

2. SUMMARY

2.1 The test consists of heating the sample for 24 h at 105°C (221°F), and determining its percentage of weight lost as result of evaporation.

3. SAMPLE SIZE

3.1 Approximately 10 g of test oil.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Oven, gravity-convection, 105° " 0.5°C (221° " 1°F).

4.2 Evaporating disk, aluminum foil, 5.7 cm (2-1/4 in) I.D., 1.6 cm (5/8 in) deep.

4.3 Balance, sensitivity "0.1 mg.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Test oil.

6. PROCEDURE

6.1 Heat oven 105° " 0.5°C (221° " 1°F).

6.2 Weigh evaporating dish to within 0.2 mg.

6.3 Pour sample into evaporating dish, and weigh dish and contents to within 0.2 mg. Then place dish into heated oven for 24 h.

6.4 At the end of 24 h, remove dish, and allow sample to cool to room temperature.

6.5 Determine the evaporation loss of sample within 0.2 mg (see 7.1).

7. CALCULATIONS

7.1 Calculate the evaporation loss of the sample as follows:

$$\text{Evaporation loss, percent by weight} = \frac{S - W}{S} * 100$$

where:
S = initial weight of sample, in g.
W = weight of sample, in g after the test.

8. REPORTING

8.1 Report the evaporation loss to the nearest 0.2 mg.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Air Force - AFWAL/POSL - 1986

Method 3500.1
September 30, 1986

MONOBASIC ACID COMPONANTS OF SYNTHETIC ESTER LUBRICANTS BY GAS CHROMATOGRAPHY

1. SCOPE

1.1 This method covers the determination of monobasic acid components of ester lubricants by gas chromatography. The method is intended for use on any neopentyle polyol ester lubricants where the acid components contain from 4 to 10 carbon atoms.

2. SUMMARY OF METHOD

2.1 The lubricant to be analyzed is hydrolyzed with potassium hydroxide, followed by hydrochloric acid treatment to evolve the acids. After ether extraction of the acids, the acids are physically separated by gas chromatography and are identified by comparing them to corresponding acids separated under identical operating conditions from a reference standard acid mixture of known composition. The acids are determined quantitatively by also using the chromatogram of the sample which is interpreted by comparing peak heights with those obtained on the reference standard mixture of pure acids of known concentration.

3. SAMPLE SIZE

3.1 Approximately 20 g of sample are required for duplicate determination.

4. REFERENCES,STANDARDS, AND APPARATUS

4.1 Chromatograph and Recorder- A chromatograph with a thermal conductivity detector and a strip –type recorder may be used provided that the butyric and isovaleric acid components in the standard acid mixture (see 5.21 can be satisfactorily resolved when the column of 6.1.1 is used and the conditions of 6.1.2 are met. In addition, the equipment shall have sufficient sensitivity to meet the precision requirements of section 9.

Two columns, stainless steel. 183 cm(6ft) long, 0.635 cm(0.25in) O.D packed with the mixture and in the manner described in section 6.11

Erlenmeyer flask, wide mouth 3.7 cm(1.46 in) I.D 250mL

1.1 Reflusk condenser, spiral, block tin

1.2 Electric hot plate

1.3 Steam bath or flash evaporator

1.4 Crystallizing dish, 9 cm diameter H 5.0 cm deep

1.5 Graduated cylinders, 50 and 100 mL

1.6 Separatory funnel , pear shaped ,250 mL

1.7 Erlenmeyer flask, narrow mouth, glass stoppered, 300mL

1.8 Erlenmeyer flask narrow mouth , standard taper joint, 300mL

1.9 Vigreux fractionating column, standard taper joint at lower end complementary to joint of flask described in 4.11

1.10 Syringe, 10-µL capacity, graduated in 0.2µL.

1.11 Analytical balance, 200 g capacity

1.12 Vacuum oven

1.13 Vacuum pump

1.14 Electric stirrer

1.15 Electric vibrator

1.16 Filter , size 4.7 cm diameter, 10 µL.

1.17 Stainless filter holder and funnel

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS . The materials and standards listed in this section must be handled carefully. Federal test method 10000 , Material Handling Safety Precautions, is a reference which lists all toxic and hazardous material cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures and spill practices of each chemical are explained.

5.2 Potassium hydroxide, reagent grade [3,0,1].

5.3 Ethyl alcohol, 95% [0,3,0].

5.4 Distilled water

5.5 Hydrochloric acid, dilute, made by diluting one volume of reagent grade acid, sp.gr. 1.18 with an equal volume of water [3,0,0].

5.6 Ethyl ether, reagent grade [2,4.1]

5.7 Sodium sulfate, anhydrous, reagent grade[u,u.u]

5.8 Diethylene glycol adipate polyester [u,u.u]

5.9 Gas Chrom P, 60 to 80 mesh, acid-based washed, or equivalent

5.10 Chloroform ,reagent grade[2,2,0].

5.11 Phosphoric acid, reagent grade, 85% [2,0,0].

5.12 Helium, oil free.

5.13 Butyric acid, reagent grade, [2,2,0].

5.14 Isovaleric acid, reagent grade [u,u.u]

5.15 Valeric acid, reagent grade [u,u.u]

5.16 Caproic acid, reagent grade [2,1,0].

5.17 Eptylic acid reagent grade [u,u.u]

5.18 Caprylic acid, reagent grade [u,u.u]

5.19 Pelargonic acid, reagent grade [u,u.u]

5.20 Capric acid, reagent grade [u,u.u]

5.21 Standard acid mixture prepared as follows:

5.22 By means of an analytical balance successively weigh into a 125-mL glass-Stopperd bottled aa the acids listed in 5.13 through 5.20 in the amounts indicated in the table below. These listed weights give concentrations of the mixed acids in terms of the indicated individual mole percent.

Acid	Number of carbon [1] Atom in acid	Weight in grams [2]	Mole percent
Butyric	4	4.41	10
Isovaleric	5	5.11	10
Valeric	5	5.11	10
Caproic	6	5.81	10

Heptylic	7	6.21	10
Caprylic	8	10.82	15
Pelargonic	9	11.87	15
Capric	10	17.23	20

[1] For convenience , these acids hereafter in this method will be referred to by the number of their carbon atoms; e.g., butyric as C4 , isovaleric as is - C5.

[2] These weights , or some multiole of them are recommended as giving a satisfactory chromatogram of the acids relative to peak heights.

6. PROCEDURE

6.1 Preparation of apparatus

6.2 Column preparation.

6.3 Packing for chromatographic column prepared as follows

6.4 Dissolve 6.0 g of doethylene glycol adipate polyester in 150 mL of chloroform with rapid stirring.

6.5 Add 1.06 g of phosphoric acid to the mix and continue stirring until the acid is completely dispersed.

6.6 Add 23.1 g of Gas Chrom P to the mix and continue stirring for 10 min.

6.7 Remove bulk of chloroform from mix by flash evaporator or on steam bath.

6.8 Remove remaining chloroform by heating in a vacuum oven for 2 h at 200ºC and 0.1 cm pressure

6.9 Cool and load into chromatographic column using vibrator on column to insure uniform and complete packing.

6.10 Chromatograph- Mount the column in the chromatograph and use the programmed temperature operation. For this operation, set the controls of the chromatograph for an initial column temperature of140ºC (284 ºF), a final column temperature of 210ºC (410º F), a temperature rate of 8ºC (46º F)/min., a hold time of 20 min at the final temperature, an injection port temperature of 220ºC (428ºF),a detector temperature of 220ºC(428ºF), abridge current of 150mA, and the helium flow at 30.5 mL/min. The temperature programming must start immediately after injection.

6.11 Test procedure.

6.12 Hydrolysis of lubricant to be analyzed-Weigh 10 g of the lubricant into a 250 –mL wide-mouth Erlenmeyer flask, add a solution consisting of 8g of potassium hydroxide and 15 mL water, followed by 60mL ethyl alcohol. Swirl to mix contents.

Insert spiral reflux condenser in mouth of flask,connect condenser to cold water line, place flask on hot plate, adjust heat so that contents boil gently and continue refluxing for 16 h.

6.13 Recovery of acid-Transfer contents of flask to crystallizing dish, rinse flask 3 or 4 times with 15 mL water and add washing to dish. Take to complete dryness on steam bath, [1] , add 15 mL water to dish.and warm to dissolve salts. Transfer solution to separatory funnel, rinse dish 3 or 4 times with 15 mL water and add washings to funnel. Place funnel in Ice bath, add 50 mL of cold dilute hydrochloric acid (1:1),cool for 10 min and swirl to complete mixing of contents. Add 50 mL of ethyl ether, shake vigorously for 2 min,venting frequently to relieve pressure. Let phase separate completely and run the lower (water) phase into a second separatory funnel. Run the upper (ether) layer into a 300 mL glass stoppered Erlenmeyer flask. Add 50 mL of ethyl ether to the second funnel and re-extract the water layer as above, adding the ether layer to the 300-mL flask .Repeat the ether extraction of the water layer twice more in individual funnels, collecting the ether layers inthe300mL flask. Discard the water layer. Add 35-40 g of sodium sulfate to the ether extract, shake and let stand a few hours or probably overnight. Filter the ether solution through 10-µm filter membrane (using stainless filter and funnel)in to a 300mL Erlenmeyer flask with tapered joint. Rinse filter and funnel a few times with ethyl ether to ensure complete removal of acids from sodium sulfate.Add 6-10 granules of silicon carbide into the flask to avoid superheating, connect to Vigreux column and distill off the ether in a hot water bath until the remaining solution is 20-25 mL stopper flask and retain for essay. Conduct recovery of acids under hood.

6.14 Preparation of chromatograms- Using standard acid mixture and the recovered acids, prepare chromatograms in the following manner: Inject 1 µL of the standard acid mixture into the chromatograph, mark for reference on recorder chart paper where recorder pen was located at the time of injection, and complete chromatogram. Examine chromatogram for peak heights of the acids. If the greatest peak height is approximately three quarters of the chart width, the dosage and attenuator settings are satisfactory. If not, adjust dosage and attenuator until the desired peak height is attained

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[1] *A flash evaporator may be used as an alternate for removal of the water and alcohol.*

In a similar manner inject 1µL of the recovered acid, obtain chromatogram and proceed as above until the peak height of the scan is satisfactory.

Identification and quantitative determination of recovered acids.

6.15 Identification of the acids recovered from the sample- On the chromatogram of the standard acid mixture, assign C4 acid to first peak from reference mark on chart, designate next peak as is -C5 acid, then the successive peaks to -C5 through C10 acids in numerical order. Measure in mm the distances from reference mark to each peak and record each distance against the corresponding acid. These values are designed as the retention times of the acids. On the chromatogram of the recovered acids, measure distance from reference mark to each peak. Compare with those obtained from the standard acid scan and identify each recovered acid accordingly.

6.16 Quantitative determination of the recovered acids – On the chromatogram of the standard acid mixture , draw a base line from the reference mark to that point on the scan which indicates the C10 acid has been eluted completely. From the base line measure each peak height in mm and sum all peak heights. Normalize each peak height by dividing each by the sum of the heights. Establish a correction factor for each acid by dividing the normalized peak height per mole of C5 acid by the normalized peak height per mole of each component acid. On the chromatogram of the recovered acids, proceed as above up to and including the normalization of each peak height. Multiply the normalized peak height of each acid component by its appropriate correction factor to obtain a "corrected" height for each. To obtain the mole-percent of each acid, divide the "corrected" peak height of each by the sum of all the "corrected" heights times 100. Record each component to the nearest integral value. Examples of the illustrated in Table 1 and 2.

TABLE 1. Standard acid mixture.

Chromatographic retention times and correction factors				
Acid	Retention time	Peak height	Normalized peak area	Correction factor
	(see 6.3.1) in mm (measured)	In mm (measured)	Peak height of one component Sum of peak heights Of all components	Normalized peak Height of C5 acid per mole normalized peak height of component acid per mole
C4	70	168	11.6	1.14
Iso-C5	74	187	12.9	1.02
C6	84	192	13.2	1.00
C7	98	182	12.6	1.05
C8	115	155	10.7	1.23
C9	136	212	14.6	1.36
C10	162	169	11.7	1.69
	196	184	12.7	2.08

TABLE 2. Unknown acid mixture

Chromatographic Identification and mole percent determination						
Identified acid	Retention time ,mm	Peak height Mm (measured)	Normalized peak height	Correction factor (see Table 1)	A corrected height	Mole percent (rounded)
	See 6.3.1		*1*		*2*	*3*
C4	71	5	1.2	1.14	1.4	1.2=1
iso C5	0	0	0	0	0	0=0
C5	84	165	40.0	1.00	40.0	34.4=34
C6	98	84	20.4	1.05	21.4	18.4=18
C7	115	87	21.1	1.23	26.0	22.4=22
C8	136	21	5.1	1.36	36.9	5.9=6
C9	162	50	12.1	1.69	20.4	17.6=18

1. $$\frac{\text{Peak height of one component}}{\text{sum of peak heights of all components}}$$

3. $$\frac{\text{100 times "corrected" height of component}}{\text{sum of all "corrected" heights}}$$

2. $$\text{Peak height times correction factor}$$

7. CALCULATIONS

7.1 This section is not applicable to the test procedure

8. REPORTING

8.1 Report each component to the nearest integral value

9. PRECISION

9.1 Repeatability- Replicate determinations shall not differ from one another by more than ± mole-percent.

9.2 Reproducibility has not been established

Method prepared by:

Navy- NAPC-1986

Method 3603.5
September 30, 1986

SWELLING OF SYNTHETIC RUBBERS

1. SCOPE

1.1 This method is used for determining the effect of petroleum products on synthetic rubber.

2. SUMMARY

2.1 The test consists of determining the volume of three standardized test rubber sheets by water displacement, storing them immersed in the sample for 168 h at 70°C, then determining the average change in volume of the sheets.

3. SAMPLE SIZE

3.1 For liquid products: Approximately 300 mL of the liquid to be tested.

3.2 For greases: Approximately 400 g of grease to be tested.

4. REFERENCE, STANDARDS, AND APPARATUS

4.1 Container, heat-resistant, consisting of:

a. For liquid sample: Test tube, 5 cm O.D. by 40 cm long. A graduated cylinder with these dimensions may be used provided it doesn't have a pouring spout.
b. For grease sample: Petri dish, 10 cm O.D. by 5 cm deep with cover (watch glass).

4.2 Balance, sensitivity "1 mg, suitable for weighing in both air and water.

4.3 Oven, gravity-convention, 70° " 1°C (158° " 2°F).

4.4 Spatula (for grease sample).

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Test sheets (3), rubber standard, type as required by the specification, approximately 2.5 H5.1 H 0.19 cm. Samples of synthetic rubber stock should not be older than 6 mo.

5.3 Alcohol, ethyl or methyl, anhydrous [1,3,0].

5.4 Filter paper.

5.5 Distilled water.

6. PROCEDURE

6.1 Determine the water displacement of each test sheet to the nearest 1 mg by weighing each sheet in air, then in distilled water at 24° " 3°C, recording the weights, and computing the difference between the weight in air and the weight in water for each sheet (see 7.1).

6.2 Fill container with sample, using the following:

a. For liquid sample: Approximately 20 mL of sample for each gram of total weight of test sheets.
b. For grease sample: Sufficient sample to fill container completely.

6.3 Blot dry each test sheet with filter paper, then immerse sheets completely in sample in container. For grease samples, immerse test sheets vertically, with 5 cm edge uppermost, and cover the container.

6.4 Store container in oven at 70° " 1°C (158° " 2°F) for 168 " 0.5 h (approximately one week). For liquid samples, maintain a ratio of 20 mL of oil to each gram of test sheet. Make up oil must be the same temperature as the test fluid when added. For grease samples, check daily (except weekends), that the samples are in complete contact with test sheets and close any voids by using a spatula.

6.5 At the end of the storage period, cool the sample to 24° " 3°C. Then clean test sheets as follows:

a. Remove test sheets from sample and wipe them to remove most of the adhering sample.
b. Dip sheets in alcohol and wipe or blot them dry, using filter paper (or soft cloths). NOTE THE TIME.

6.6 Within 5 min of drying test sheets, redetermine the water displacement of each sheet as specified in 6.1.

7. CALCULATIONS

7.1 Compute the percentage change in volume of each rubber sheet as follows:

$$V = \frac{(A - B)}{B} * 100$$

where:
V = change in volume, percent.
A = displacement weight at conclusion of test, mg.
B = displacement weight before immersion in sample, mg.

8. REPORTING

8.1 Report the average percent volume change of the three rubber sheets.

9. PRECISION

9.1 Repeatability. Test results, obtained by one operator at one laboratory, for each test sheet must not vary from the average by more than the following:

a. If average is 0 to 5% units: 0.5% unit.
b. If average is above 5% units: 1% unit.

Method Prepared by:

Navy - NADC - 1986

Method 3604.2
September 30, 1986

SWELLING OF SYNTHETIC RUBBER BY AIRCRAFT TURBINE LUBRICANTS

1. SCOPE

1.1 This method is used for determining the swelling effect of aircraft turbine lubricants on synthetic rubber. Synthetic lubricating fluids for aircraft turbine engines must be compatible with materials-of-construction, such as elastomers used in the engines. Elastomers are used in seals, o-rings, and related areas. This method is designed to reveal lubricating fluid-elastomer compatibility problems relative to volume swell.

2. SUMMARY

2.1 The volume of three standard test rubber samples is determined by water displacement. The rubber samples are immersed in the test lubricant for a specified time (such as 72, 96, or 168 h) at a given temperature. After storage, the average change in volume is determined.

3. SAMPLE

3.1 Approximately 350 mL of the lubricant to be tested.

4. REFERENCES, STANDARDS AND APPARATUS

4.1 Oven, gravity-convection, 70° ± 1°C (158° ± 2°F).

4.2 Beakers (2), borosilicate, 400 mL.

4.3 Suspension device, glass or stainless-steel (for holding test rubber-sheets completely immersed in specimen in 400-mL beaker so that they do not touch each other or the beaker).

4.4 Hooks, stainless-steel wire (for suspending test rubber sheets from suspension device and from balance).

4.5 Balance, sensitivity, ± 0.1 mg.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully.

Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Standard test rubber sheets, type H or FA, fabricated less than one year prior to use. Test sheets should conform to the properties listed in U.S. Air Force specification bulletin 539, Standard Elastomer Stocks. If stored for more than one year, the elastomer should be retested before use. Storage should be in a dark place at a temperature of less than 25°C (77°F) away from electrical apparatus.

5.3 Cleaning solution, glass (concentrated sulfuric acid [3,0,2] saturated with potassium or sodium dichromate [1,0,1], technical grade).

5.4 Solvent naphtha, technical grade [1,4,0].

5.5 Toluene, technical grade [2,3,0].

5.6 Alcohol, ethyl [0,3,0].

5.7 Emery cloth No. 60.

6. PROCEDURE

6.1 Clean all glassware as follows:

a. Rinse in toluene, followed by naphtha, and allow to air dry. An alternative procedure is to wash in a commercial type dish washer.
b. Soak in glass cleaning solution for 2 h (min.).
c. Rinse thoroughly with tap water, and then with distilled water.
d. Dry in an oven at 100°C (212°F), and store in dust-free location.

6.2 Clean suspension device and hooks thoroughly with (toluene and naphtha) solvents, air dry, and store in a dust-free location.

6.3 Preheat and adjust the oven to maintain the required temperature.

6.4 Prepare standard test rubber sheets as follows:

a. Using No. 60 emery cloth, buff both sides of the rubber sheet, using strokes first in one direction and then at 90° to that direction. Buff just enough so that no gloss can be seen on the sheet when viewed by reflected light.
b. Cut three pieces (3.5 × 5 cm) from the sheet.
c. Punch a 0.3-cm (1/8-in) (approximate) hole at the center of the 5-cm side, about 0.6 cm (1/4 in) from the edge.

d. Mark each piece for future identification by cutting small pieces from one or more corners.
e. Attach wire hooks to each piece.

6.5 Weigh each rubber test sheet (and its hook) in air to the nearest 0.1 mg by suspending it from the balance by its hook. Record the weights.

6.6 Reweigh (to the nearest 0.1 mg) each sheet while suspended in distilled water at room temperature, being careful to remove all air bubbles from the sheet and to prevent the sheet from touching the water container. Record the weights.

6.7 Determine (and record) the displacement weight of each piece by subtracting the weight in water from the weight in air.

6.8 Fill a 400-mL beaker with 350 mL of the sample.

6.9 Attach the rubber sheets to the suspension device, and submerge the sheets in the sample so that the top edges are no less than 1.3 cm (1/2 in) below the surface. Make certain that the sheets do not touch each other or contact the sides or bottom of the beaker.

6.10 Place the beaker in oven at the required temperature for the specified time.

6.11 At the end of 72 h, remove the beaker from the oven and allow it to cool at room temperature for 30 . 5 min.

6.12 Remove the rubber test sheets from the sample and wash them in ethyl alcohol, rubbing them gently with the fingers to remove all oil from the surfaces. Caution - wear protective gloves.

6.13 Wipe the rubber pieces dry with soft lint-free cloth, and allow them to air dry for several minutes.

6.14 Repeat 6.5 to 6.7 to determine the displacement weight of the rubber sheets after immersion in oil.

7. CALCULATION

7.1 Displacement weight

$$\begin{array}{c} \textit{Weight of test sheet in air} \\ \underline{-\textit{Weight of test sheet in water}} \\ \textit{Displacement weight} \end{array}$$

7.2 Calculate the percentage change in volume of each rubber sheet as follows:

$$V = \frac{A - B}{B} * 100$$

where:
V = change in volume, percent.
A = displacement weight at conclusion of test, mg.
B = displacement weight before immersion in sample, mg.

8. REPORTING

8.1 Report the average percent volume change of the three rubber sheets.

9. PRECISION

9.1 Repeatability. Results by one operator using one apparatus must not differ by more than 1%.

9.2 Reproducibility. Results from different operators or different apparatus must not differ by more than 2%.

Method prepared by:

Navy - NAPC - 1986

Method 3710.1
September 30, 1986

MOLYBDENUM DISULFIDE PURITY

1. SCOPE

1.1 This method is used for determining the purity of molybdenum disulfide powder.

2. SUMMARY

2.1 This method consists of dissolving the MoS_2 in nitric acid and perchloric acid, removing iron with ammonium hydroxide and precipitating solution with lead acetate. The purity of MoS_2 is determined from the weight of lead molybdate precipitate.

3. SAMPLE SIZE

3.1 10 g of molybdenum disulfide (MoS_2) powder.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Hot plate.

4.2 Beakers, borosilicate, 400 and 600 mL.

4.3 Pipet, 50 mL.

4.4 Volumetric flask, 500 mL.

4.5 Buret, 50 mL.

4.6 Mechanical stirrer.

4.7 Porcelain crucible.

4.8 Muffle furnace capable of maintaining 550° to 600°C (1,020° to 1,110°F).

4.9 Desiccator.

4.10 Analytical balance.

5. MATERIALS

(Unless otherwise indicated, it is intended that all chemicals shall be of reagent grade and shall conform to the specifications established by the Committee of Analytical Reagents of the American Chemical Society, where such specifications are available.)

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Potassium chlorate (reagent grade) [1,0,2].

5.3 Nitric acid, conc. (reagent grade) [2,0,0].

5.4 Hydrochloric acid, conc. (reagent grade) [3,0,0].

5.5 Ammonium hydroxide, conc. (reagent grade) [u,0,u].

5.6 Methyl orange indicator solution. Dissolve 0.1 g methyl orange crystals (reagent grade) in 100 mL of distilled water [u,u,u].

5.7 Ammonium acetate solution. Dissolve 605 g of ammonium acetate (reagent grade) in distilled water and dilute with distilled water to 2 L [u,0,u].

5.8 Glacial acetic acid (reagent grade) [2,2,1].

5.9 Lead acetate solution. Dissolve 20 g of lead acetate, $Pb(CH_3COO)_2 \cdot 3H_2O$ (reagent grade) in distilled water, add 20 mL of glacial acetic acid (reagent grade) and dilute with distilled water to 1 L [u,u,u].

5.10 Tannic acid [0,1,0].

5.11 Tannic acid indicator. Dissolve 0.05 g tannic acid (reagent grade) [0,1,0] in 48 mL of distilled water and add 2 mL of glacial acetic acid (reagent grade) [2,2,1].

5.12 Ammonium nitrate solution. Dissolve 30 g of ammonium nitrate (reagent grade) in distilled water and dilute with additional distilled water to 1 L [0,1,3].

6. PROCEDURE

6.1 Weigh a moisture- and oil-free sample (See Note l) of powder, 2.6 to 2.8 g, on an analytical balance to the nearest 0.1 mg and transfer the sample to a 400-mL beaker.

NOTE 1. Moisture may be removed by heating MoS_2 powder in a drying oven at 105°C for 1h.Oil may be removed by extraction with acetone and petroleum ether.

6.2 Add 1 mL of distilled water to the sample and moisten particles of MoS_2. Add with caution 30 mL of a saturated solution of potassium chlorate in nitric acid.

6.3 After vigorous action ceases, place the beaker on a hot plate and digest the sample slowly until the solution becomes pale yellow or almost white. Add 0.5 g of potassium chlorate to the solution and evaporate it to dryness.

6.4 Add 15 mL of conc. HCl, and boil solution until all brown fumes are expelled. Allow solution to cool and while stirring dilute with distilled water to 50 . 5 mL.

6.5 To this solution add 40 mL of concentrated ammonium hydroxide (this will precipitate iron). Heat to boiling and while hot filter through Whatman No. 40 (or equivalent) paper. Paper pulp may be added to aid filtration. Collect filtrate in 600-mL beaker and retain.

6.6 Wash residue on filter paper 5 times with 10-mL portions of hot distilled water, collecting the filtrate in the 600-mL beaker containing first filtrate. Retain combined filtrate.

6.7 Transfer filter paper with residue to clean 400-mL beaker, add 10 mL of hydrochloric acid. Stir until filter paper is pulped.

6.8 To this solution add 40 mL of distilled water and while stirring add 40 mL of concentrated ammonium hydroxide. Bring solution to a boil. While the solution is hot, filter through Whatman No. 40 (or equivalent) paper, and collect the filtrate in the 600-mL beaker containing the previous filtrate. Wash residue on filter paper with 25-mL portions of hot distilled water 8 times, collecting filtrate in the 600-mL beaker with the combined filtrate.

6.9 Discard filter paper and residue (see Note 2) and retain combined filtrate.

NOTE 2. The filter paper and residue can be used to determine percent total insoluble matter. Place filter paper and residue in a previously ignited and weighed porcelain crucible, carefully burn off filter paper and then place in muffle furnace at 600°C for 1 h. Remove crucible and place in desiccator for 1 h, when cooled remove and weigh. Repeat ignition to constant weight. Calculate percent total insoluble matter (see 7.1).

6.10 Transfer the combined filtrate from the 600-mL beaker to a 500-mL volumetric flask, dilute with distilled water to 500 mL.

6.11 Using a 50-mL pipet, pipet a 50-mL aliquot into a 400-mL beaker, add 2 drops of methyl orange indicator and neutralize the excess ammonium hydroxide in the filtrate aliquot with drops of concentrated hydrochloric acid. Neutralization will be complete at the first sign of pink or red color in the solution.

6.12 Add 30 mL of ammonium acetate to the solution and 10 mL of glacial acetic acid. Heat solution to near boiling.

6.13 Add a previously calculated amount of lead acetate solution (31 to 33 mL/0.26 to 0.28 g of sample) in aliquot respectively, (see Note 3), to the sample solution from a buret, dropwise at the rate of 30 mL in 2 min. While stirring the solution mechanically add an excess of 3 mL of lead acetate and stir for 5 min after complete addition.

NOTE 3. For an accurate check on the quantity of lead acetate solution required, the tannic acid solution can be used as an outside indicator. The end point is reached when a drop of the neutralized aliquot solution added to 0.1 mL of the tannic acid indicator solution on a spot plate fails to give a brown coloration.

6.14 Place sample solution on a hot plate and heat until near boiling and hold at this point for at least 1 hour and allow the precipitated lead molybdate to settle completely.

6.15 Decant the bulk of the solution through a Whatman No. 42 (or equivalent) paper containing a small amount of paper pulp. Transfer the precipitate to the filter paper and wash 4 times with 50-mL portions of hot ammonium nitrate solution. The filtrate can be discarded.

6.16 Place filter paper with residue in a previously cleaned, ignited, cooled and weighed porcelain crucible. Ignite carefully in a muffle furnace at 550° to 600°C (1,020° to 1,110°F) for 1 h.

6.17 Remove crucible and place in desiccator for 30 to 60 min and allow to cool to room temperature. Weigh crucible. Repeat ignitions to constant weight. Calculate the weight percent MoS_2 (see 7.2).

7. CALCULATIONS

7.1 The percent total insoluble matter may be calculated as follows:

$$\text{Percent total insoluble matter} = \frac{c}{w} * 100$$

where:
c = weight of residue in crucible
w = weight of moisture- and oil-free sample

7.2 The percent MoS_2 is calculated using the following equation:

$$\text{Percent } MoS_2 = \frac{c}{w} * 435.9$$

where:
c = increase in weight of crucible
w = weight of moisture- and oil-free sample

8. REPORTING

8.1 Report the percent total insoluble matter and percent MoS_2.

9. PRECISION

9.1 Precision data have not been developed for this method

Method Prepared by:

Navy - NADC - 1986

Method 3720.2
September 30, 1986

MOLYBDENUM DISULFIDE CONTENT OF LUBRICATING GREASE

1. SCOPE

1.1 This method is used to determine the molybdenum disulfide content of lubricating greases. This method does not apply to lubricating greases that contain non-soap thickeners.

2. SUMMARY

2.1 This method consists of dissolving the base oil and soap thickener in a mixture of oleic acid and benzene. The MoS_2 is removed by filtration and MoS_2 content determined by weighing the MoS_2.

3. SAMPLE SIZE

3.1 5 g of lubricating grease to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Desiccator.

4.2 Erlenmeyer flask, 125 mL.

4.3 Filter pump (aspirator).

4.4 Flask, filtering heavy wall.

4.5 Gooch crucible and suitable holder.

4.6 Oven capable of 100°C.

4.7 Steam bath.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 *n*-Hexane, ACS grade [1,3,0].

5.3 Oleic acid, USP grade [0,1,0].

5.4 Petroleum ether (O-E-751) [1,4,0].

6. PROCEDURE

6.1 Perform the test as follows:

a. Weigh 3 to 5 g of the grease to be tested to the nearest mg and distribute as a thin layer in a 125 mL Erlenmeyer flask prior to adding 90 mL of a 50-50 mixture by volume of *n*-hexane and oleic acid. (See Note 1).
b. Warm the flask for approximately 1/2 h on a steam bath with occasional agitation until all lumps have disappeared and the molybdenum disulfide is dispersed.
c. Filter the contents of the flask through a previously prepared and weighed (to the nearest mg) Gooch crucible. Wash the flask and crucible with approximately 10 mL of a 50-50 mixture by volume of oleic acid and *n*-hexane, followed by three washings with petroleum ether. (See Note 1.)

<u>NOTE 1:</u> Caution. *n*-Hexane and petroleum ether are both toxic and flammable. Do not breath their fumes nor allow them to come in contact with the skin. Keep flames away from the *n*-hexane and petroleum ether.

d. Dry the crucible and contents at 100°C for 1/2 h, cool in a desiccator to room temperature and weigh. (See Note 2).

<u>NOTE 2:</u> The purity of MoS_2 powder may be determined in accordance with test method No. 3710 or by means of spectrochemical analysis.

7. CALCULATIONS

7.1 Calculate the percentage of molybdenum disulfide as follows:

$$Percent\ molybdenum\ disulfide\ (as\ insoluble\ matter) = \frac{Increase\ in\ weight\ of\ crucible}{weight\ of\ sample} * 100$$

8. REPORTING

8.1 Report the percent molybdenum disulfide as insoluble matter.

8.2 *n*-Hexane, ACS grade [1,3,0].

8.3 Oleic acid, USP grade [0,1,0].

8.4 Petroleum ether (O-E-751) [1,4,0].

9. PROCEDURE

9.1 Perform the test as follows:

a. Weigh 3 to 5 g of the grease to be tested to the nearest mg and distribute as a thin layer in a 125 mL Erlenmeyer flask prior to adding 90 mL of a 50-50 mixture by volume of *n*-hexane and oleic acid. (See Note 1).
b. Warm the flask for approximately 1/2 h on a steam bath with occasional agitation until all lumps have disappeared and the molybdenum disulfide is dispersed.
c. Filter the contents of the flask through a previously prepared and weighed (to the nearest mg) Gooch crucible. Wash the flask and crucible with approximately 10 mL of a 50-50 mixture by volume of oleic acid and *n*-hexane, followed by three washings with petroleum ether. (See Note 1.)

NOTE 1: Caution. *n*-Hexane and petroleum ether are both toxic and flammable. Do not breath their fumes nor allow them to come in contact with the skin. Keep flames away from the *n*-hexane and petroleum ether.

d. Dry the crucible and contents at 100°C for 1/2 h, cool in a desiccator to room temperature and weigh. (See Note 2).

NOTE 2: The purity of MoS_2 powder may be determined in accordance with test method No. 3710 or by means of spectrochemical analysis.

10. CALCULATIONS

10.1 Calculate the percentage of molybdenum disulfide as follows:

$$\text{Percent molybdenum disulfide (as insoluble matter)} = \frac{\text{Increase in weight of crucible}}{\text{weight of sample}} * 100$$

11. REPORTING

11.1 Report the percent molybdenum disulfide as insoluble matter.

12. PRECISION

12.1 Precision data have not been developed for this method.

Method Prepared by:
Navy - NADC - 1986

Method 3722.2
September 30, 1986

MOLYBDENUM DISULFIDE CONTENT OF NON-SOAP THICKENED LUBRICATING GREASES

1. SCOPE

1.1 This method is used to determine the MoS_2 content of non-soap thickened greases.

2. SUMMARY

2.1 The test consists essentially of extracting the base oil from the grease with n-hexane and determining the molybdenum disulfide content of the remaining solids by precipitation to sulfate with barium chloride.

3. SAMPLE SIZE

3.1 Approximately 5 g of grease to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Desiccator.

4.2 Oven capable of 100°C.

4.3 Muffle furnace capable of 760°C.

4.4 Hotplate.

4.5 Steam bath.

4.6 Soxhlet extraction apparatus with 250-mL flask.

4.7 Beakers (2), 600 mL.

4.8 Crucibles (2), 50 mL.

4.9 Funnels (2).

4.10 Burner, Meker (or equivalent).

4.11 Policeman, rubber.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Hexane, normal, ACS [1,3,0].

5.3 Extraction thimble (double thickness 3.3 □ 8.0 cm).

5.4 Filter paper (Whatman No. 1 or equivalent).

5.5 Filter paper (Whatman No. 40 or equivalent).

5.6 Eschka's mixture - 2 parts of calcined ("light") magnesia thoroughly mixed with one part of sodium carbonate [u,u,u].

5.7 Bromine water, saturated [4,0,0].

5.8 Barium chloride, 10% [0,0,2].

5.9 Sodium carbonate, CP [u,u,u].

5.10 Potassium carbonate, CP [u,u,u].

6. PROCEDURE

6.1 Determine the solids content of the grease as follows:

a. Dry an extraction thimble (3.3 □ 8.0 cm) at 100°C, cool in a desiccator and weigh to the 100° C nearest tenth of a mg. Dry thimble to constant weight. Weigh approximately 5 g of the grease to be tested into the extraction thimble.
b. Place 200 mL of *n*-hexane in the flask, place the thimble containing the grease sample in the Soxhlet extraction tube, and assemble the apparatus as shown in Figure 1.
c. By means of a hotplate bring the hexane to boiling and reflux until the base oil has been completely extracted from the grease (see Note 1). Complete removal of the oil may be determined by sampling small (2 mL) quantities of hexane from the extraction tube and evaporating on a clean evaporating dish. An oily film will remain if oil is contained in the sample.

NOTE 1. Caution. *n*-Hexane is both toxic and extremely flammable. Use only in a well ventilated area. Do not breathe its fumes nor allow it to come in contact with the skin. Keep all flames away from the Hexane.

d. Remove the thimble from the extraction tube and dry in an oven at 100°C. Allow to cool in a desiccator. After cooling, remove and weigh to constant weight. Subtract the weight of the thimble from the latter weight to determine the weight of the solids in the grease. Calculate the percent solids (see 7.1).

6.2 Determine percent molybdenum disulfide in the grease as follows:

a. Ignite a clean crucible (50 mL) in the blue oxidizing flame of a Meker burner. Cool in a desiccator. Remove and add approximately 2 g of Eschka's mixture and weigh to the nearest 0.1 mg.
b. Add approximately 0.1 g of the grease solids (6.1 d) and reweigh to the nearest 0.1 mg (throughly mix by stirring sample using a wire rod).
c. Place a layer 0.6 cm (0.25 in) of Eschka's mixture on the sample from 6.2 b. Do not mix.
d. A blank determination using 4 g of Eschka's mixture is conducted in a separate crucible.
e. Place both crucibles in a cold muffle furnace and slowly raise the temperature to 760°C. Maintain a temperature of 760°C for 2 h. Remove the crucibles from the muffle furnace and allow to cool.
f. Remove the contents of each crucible into separate 600-mL beakers and place the crucibles in a horizontal position in the beakers. Cover the crucibles with distilled water.
g. Place on heated steam bath for 30 min. Remove and rinse each crucible so that the rinse water is deposited in the beakers.
h. Pass the contents of beaker through filter paper (Whatman No. l or equal) and rinse the residue 2 or 3 times with distilled water. Discard residue and paper.
i. Add 10 mL of saturated bromine water to the filtrates. Heat gradually to boiling. Continue to boil until the volume is reduced to 200 mL.
j. Cool the solution and add 5 mL of concentrated hydrochloric acid to each beaker and boil until colorless.
k. While boiling, add at a very slow rate 10 mL of 10% solution of barium chloride. Heat on a steam bath for at least 3 h, and allow to stand for 24 h.
l. Pass the solutions through filter paper (Whatman No. 40 or equal). Remove all barium sulfate adhering to the surfaces of the beakers by means of a rubber policeman. Wash the precipitates and discard the filtrates.
m. Clean the crucibles by fusing 50-50 mixture of sodium and potassium carbonates. Do not use potassium bisulfate. Dry and cool the crucibles and weigh to the nearest 0.1 mg.
n. Fold the filter paper and precipitates, and place in the clean weighed crucibles.
o. Place the crucibles in a cold muffle furnace and heat gradually to a temperature of 400°C. Hold at 400°C until the filter papers are thoroughly charred. Slowly

raise the temperature to 760° C and maintain at this temperature until there is no longer any carbon present. This will require approximately 1 h at 760°C. The barium sulfate should be pure white.

p. Allow the crucibles to cool and determine the weight of barium sulfate in each one. Subtract the weight of barium sulfate in the blank from that of the sample and record the difference as the weight of barium sulfate. (See note 2).

q. Calculate the percent MoS_2 in the grease solids (see 7.2).

r.Calculate the percent MoS_2 in the grease (see 7.3).

NOTE 2: A spectrochemical analysis may be conducted to determine purity of MoS_2.

7. CALCULATIONS

7.1 Calculate the percent solids as follows:

$$Percent\ solids = \frac{w_2}{w_1} * 100$$

where:

W_1 = weight of solids in sample
W_2 = weight of sample

7.2 Calculate the percent MoS_2 in the grease solids as follows:

$$Percent\ MoS_2\ in\ grease\ solids = \frac{0.343 * M_2}{M} * 100$$

where:

M = weight of solids from grease sample
M_2 = weight of barium sulfate

7.3 Calculate the percent MoS_2 in the grease as follows:

$$Percent\ MoS_2 = percent\ solid\ (see\ 7.1) * percent\ MoS_2\ in\ grease\ solids\ (see\ 7.2)$$

8. REPORTING

8.1 Report the percent solids, percent MoS_2 in grease solids, and percent MoS_2.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by

Navy - NADC – 1986

Method 3816.1
September 30, 1986

FILM THICKNESS OF DRY SOLID FILM LUBRICANTS

1. SCOPE

1.1 This method provides a means for measuring the film thickness of dry film lubricants.

2. SUMMARY

2.1 The thickness of a coated and uncoated aluminum panel is measured in four specified areas using a template and precision micrometer. The difference in readings represents the film thickness of the lubricant.

3. SAMPLE SIZE

3.1 240 mL (8 oz) of solid-film lubricant in a mixture of sprayable consistency.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Binks Spray Gun, Model 18, with a 66 SD Head attachment (Binks Manufacturing Company, Chicago, Illinois or equal).

4.2 Vernier micrometer capable of measuring to the nearest 0.0025 mm (0.0001 in), or an equivalent measuring device.

4.3 Template - 7.6 × 15.2 cm (3 × 6 in) with four 0.95-cm (3/8-in) holes centered 3.8 cm (1-1/2 in) in from the 7.6-cm side and 1.6 cm (5/8 in) in from the 15.2-cm side (see Figure 1).

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Aluminum alloy test panel conforming to ASTM B 209, 7.6 × 15.2 × 0.051 cm (3 × 6 × 0.020 in) thick, anodized in accordance with MIL-A-8625, Type 1.

5.3. 1,1,1-Trichloroethane (MIL-T-81533) [2,1,0].

6. PROCEDURE

6.1 Prepare test panel as follows:

a. Degrease the test panels in 1,1,1-trichloroethane.
b. Place template in position on the uncoated panel, trace the circumferences of the openings in the template on to the test panel.
c. Measure the thickness of the test panels at the circled areas with a micrometer having 0.0025-mm (0.0001-in) graduations. Note the thickness measurement for each circled area.

6.2 Perform the test as follows:

a. Apply two coats of dry solid film lubricant by spraying the solid lubricant to the unmarked side of the test panel. Application shall be conducted in a well ventilated area or hood, where no flames or ignition sources are present. Care should be taken to insure that the panel has a uniform coating of solid lubricant.
b. Air dry, temperature 25° ± 2°C (77° ± 3°F) test panel for ten minutes between coats. After the final application, allow coated test panel to air dry for fifteen minutes, then cure the film in accordance with specification requirements.
c. Maintain a distance of approximately 30 cm (12 in) between spray gun and test panel while spraying.
d. Use an air pressure of 275 ± 35 gage kPa (40 ± 5 psig)
e. Allow test panel to reach room temperature after curing.
f. Remeasure the thickness of the test panel at the same positions.
g. Duplicate tests shall be conducted.

7. CALCULATIONS

7.1 Calculate the film thickness as follows:

$$F = A - B$$

where:
F = film thickness
A = thickness of the coated panel
B = thickness of the uncoated panel

8. REPORTING

8.1 The average of four readings shall be reported as the film thickness of the dry solid film lubricant.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Navy - NADC - 1986

Method 4001.3
September 30, 1986

CORROSION PROTECTION COATING: SALT-SPRAY (FOG) TEST

1. SCOPE

1.1 This method is intended for the determination of the corrosion protection provided by fluid, semi-solid, and solid coatings.

2. SUMMARY

2.1 The test consists of exposing test panels to either 5 or 20% salt (NaCl) spray as stated in the product specification. When the salt concentration is not stated in the product specification, the 5% salt solution shall be used.

3. SAMPLE SIZE

3.1 Approximately 500 mL of the material to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Exposure chamber, with racks for supporting test panels, and with suitable heaters and controls.

<u>NOTE 1:</u> The chamber and all accessories shall be made of material which will not affect the corrosiveness of the fog (such as glass, hard rubber, plastic, or wood other than plywood.) In addition, all parts which come in contact with the test panels shall be of materials that will not result in accelerated local attack associated with dissimilar metal crevices. The chamber and accessories shall be so constructed and arranged that there is no direct impinging of the spray or dripping of the condensate on the test panels, so that the spray circulates freely about all panels to the same degree and so that no liquid which has come in contact with the panels returns to the salt solution reservoir. The chamber shall be properly vented. Satisfactory cabinets may be obtained from the Industrial Filter and Pump Mfg. Co., Chicago, Illinois.

4.2 Atomizers, including suitable nozzles and compressed air supply (for producing a finely divided, wet, dense fog).

4.3 Humidifier, atomizer-air (for warming and humidifying the air to produce a relative humidity at the nozzle of at least 85%).

NOTE 2: A high humidity insures against clogging the atomizers by salt deposition. A satisfactory method of humidifying the air is to pass it in very fine bubbles through a tower containing heated water. The temperature of the water should be 35°C (95°F) or higher. The permissible temperature increases with increasing volume of air and with decreasing heat insulation of the chamber and temperature of its surroundings. It should not exceed a value above which an excess of moisture is introduced into the chamber (for example, 43°C [110°F] at an air pressure of 82 gage kPa [12 psig]), or a value which makes it impossible to meet the required operating temperature.

4.4 Air supply, compressed, free from impurities such as oil and dirt.

4.5 Reservoir (for salt solution).

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Sodium chloride (A-A-59282).

5.3 Hydrochloric acid (A-A-59282).

5.4 Sodium hydroxide (A-A-59282).

5.5 Bromothymol blue, solution, indicator, 6.0 to 7.6 pH range [u,u,u].

5.6 Distilled water ACS.

5.7 1,1,1-Trichloroethane (ASTM D 4126).

5.8 Cleaning tissue, facial.

5.9 Three test panels made of low-carbon, open-hearth, cold-finished 1010 steel, 7.5 □ 5.0 by 0.32 cm thick (3 □ 2 by 1/8 in thick).

6. PROCEDURE

6.1 Preparation of salt solutions.

6.1.1 5% solution. Dissolve 5 . 0.5 parts by weight of sodium chloride in 95 . 0.5 parts by weight distilled water. Filter the solution if there is any suspended matter.

The specific gravity of this solution shall be between 1.027 and 1.041 when measured at 35° ± 1°C (95° ± 2°F). Maintain the pH of the solution between 6.5 and 7.2 by the addition of either hydrochloric acid or sodium hydroxide as necessary. Determine the pH of the salt solution electrometrically or colorimetrically using bromothymol blue as an indicator.

6.1.2 Twenty percent solution. Dissolve 20 ± 2 parts by weight of sodium chloride in 80 ± 2 parts by weight distilled water. Filter the solution if there is any suspended matter. The specific gravity of this solution shall be between 1.1267 and 1.157 when measured at 35° ± 1°C (95° ± 2°F). Maintain the pH of the solution between 6.5 and 7.2 by the addition of either hydrochloric acid or sodium hydroxide as necessary. Determine the pH of the salt solution electrometrically or colorimetrically using bromothymol blue as an indicator.

6.2 Preparation of panels for testing.

6.2.1 Cleaning of panels. Handle all panels as little as possible, particularly on the significant surfaces. Clean uncoated metal panels with warm 38° to 54°C (100° to 130°F) 1,1,1-trichloroethane and cleaning tissue. Clean all significant surfaces until they are free from water-break. If a panel has an organic coating, do not clean it with an organic solvent. Dry all panels thoroughly. Coat the edges of panels which may come in contact with supports with wax or with some material which is impervious to moisture.

6.2.2 Application of coatings to panels. Fluid, semi-solid, or solid coatings (see 1.1) shall be applied to test panels in accordance with product specification.

6.3 Location of panels in the exposure chamber. Test panels shall be supported in such a manner that the significant surfaces are approximately 15° from the vertical position and are parallel to the principal direction of horizontal flow of fog through the chamber. Whenever practicable, the panels shall be supported from the bottom or from the side. When panels are suspended from the top, suspension shall be accomplished with glass hooks or waxed string. The use of metal hooks is not permitted. The panels shall be positioned so that they do not shield each other from the freely settling fog, and so that corrosion products and condensate from one panel do not fall upon another.

6.4 Operating conditions.

6.4.1 Temperature. The test shall be conducted with a temperature in the exposure zone maintained at 35° +1°, -1.5° C (95° +2°, -3° F). Satisfactory methods for controlling the temperature accurately are by housing the apparatus in a properly controlled constant temperature room, by thoroughly insulating the apparatus and preheating the air to the proper temperature prior to atomization, and by jacketing the apparatus and controlling the temperature of the water or of the air used. The use of immersion heaters for the purpose of maintaining the temperature within the chamber is prohibited.

6.4.2 Atomization. The conditions maintained in all parts of the exposure zone shall be such that a suitable receptacle placed at any point in the exposure zone will collect from 0.5 to 3 mL of solution per hour for each 80 square cm of horizontal collecting area (10-cm diameter) based on an average run of at least 16 hours. The solution thus collected shall have a sodium chloride content of 4.5 to 5.5% for the 5-percent solution (specific gravity 1.027 to 1.041 when measured at 35° ± 1°C [95° ± 2°F]) or a sodium chloride content of 18 to 22% for the 20-percent solution (specific gravity 1.126 to 1.157 when measured at 35° ± 1°C [95° ± 2°F]). At least two clean fog-collecting receptacles shall be used, one placed nearest to any nozzle and one farthest from all nozzles. Receptacles shall be fastened so that they are not shielded by specimens and so that no drops of solution from specimens or other sources will be collected.

NOTE 3: When using nozzles made of material nonreactive to the salt solution, suitable atomization has been obtained in boxes having a volume less than 0.34 m^3 (12 ft^3) with the nozzle pressure 82 to 124 gage kPa (12 to 18 psig) with the orifices of 0.05 to 0.08 cm, and with the atomization of approximately 3 L (3 qt) of salt solution per 0.28 m^3 (10 ft^3 of box volume per 24 h). When using large size boxes having a volume considerably in excess of 0.34 m^3 (12 ft^3), the above conditions may have to be modified to meet the requirements for the operating conditions.

6.4.3 Length of test. The length of the salt spray test shall be as required by the product specification. The test shall be run continuously for the specified test period, or until a definite indication of failure is observed, with interruption only for adjustment of the apparatus and for inspection of the test panels.

6.5 At the end of the test, the test panels shall be examined immediately to determine compliance with the standards of acceptability given in the product specification. If necessary for examination purposes, the test panels may be washed gently or dipped in running water not warmer than 38°C (100°F) and brushed lightly.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the number of specimens tested and the number of passing or failing results.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:
Army - ME - 1986

Method 5003.2
September 30, 1986

DEPOSIT-FORMING TENDENCIES OF AIRCRAFT TURBINE LUBRICANTS

1. SCOPE

1.1 This test method describes a procedure for determining the deposit and sludge forming tendencies of aircraft gas turbine lubricants.

2. SUMMARY

2.1 A sample of the lubricant is circulated under controlled conditions for a prescribed period of time through an aerated test chamber containing an aluminum tube held at constant temperature. From the chamber the oil passes through a cooler, a line filter, a circulating pump and back into the chamber. The weight of solid decomposition products on the heated tube and on the line filter are determined. Changes in the viscosity and neutralization number of the oil can be determined if desired.

3. SAMPLE SIZE

3.1 As needed.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Deposition tester, Wright Air Development Center (Erdco Engineering Corp., Adison, IL) consisting of an oil circulation pump, coking head with tube, oil feeder, oil cooler, line filter, and the necessary instrumentation and supporting structure (see Figure 1 through 5).

NOTE 1: Model 1 is equipped with a pyrometer, Model 2 with a temperature recorder, and Model 3 with a pyrometer, a selector switch, and a differential pressure recorder. A "W" following the model number indicates the inclusion of a watt-hour meter.

a. Cabinet. The test cabinet consists of a test section and an instrument section. Ventilation and heat are provided to maintain the test section at 48.9° ± 3°C (120° ± 5°F) and the instrument section at not more than 6°C (10°F) above room temperature. The sections are separated by a partition.
b. Instrument section. The instruments and their locations are shown in Figure 1. Optional items (differential-pressure recorder, watt-hour meter, pyrometer, and selector switch) are included in this Figure.
c. Thermocouples. All thermocouples and leads are made from precision calibrated iron constantan wire. Two bayonet thermocouples are provided for the coking tube, one of which is connected to the stepless temperature control,

the other to the pyrometer or temperature recorder. Two bare-wire thermocouples are provided for measuring "oil-in" temperature. One is connected to the off-on temperature controller, the other to the pyrometer or temperature recorder. One bare-wire thermocouple, connected to pyrometer or temperature recorder, is provided for measuring "oil-out" temperature. A thermocouple, design optional, is used to measure the test section air temperature and is connected to the pyrometer or temperature recorder.

d. Test section. The test section components and their approximate location are shown in Figure 2. A schematic drawing of the entire rig is shown in Figure 3. The test section components are mounted on a rack that can be removed from the cabinet to permit easy disassembly for cleaning. A spare test section can be advantageously utilized to keep down-time on the rig to a minimum

e. Coking tube. The coking tube consists of an aluminum tube with a shrunk-in 1500-watt cartridge heater. Two thermocouple holes are provided. The configuration and dimensions of the coking tube are shown in Figure 4.

f. Line filter. The filter consists of a two-piece body, a 100-mesh stainless steel screen, and a screen support. These components are shown assembled in Figure 5.

g. Oil cooler. The oil cooler is designed for oil downflow through annular space and water upflow through the inner tube. A continuous water bleed to waste is taken through an orifice just ahead of the inlet to the cooler (see Figure 2, item 32). This acts as a drain for the cooler when the water flow is shut off by the control, air being bled back into the cooler through a check valve (see Figure 2, item 30).

4.2 Balance, capacity 500 g, sensitivity . 10 mg.

4.3 Stop watch, accuracy . 0.1 s.

4.4 Wet test gas meter.

4.5 Brushes, metal, special; 0.195 in diameter, 1/2 in B.P., 10 in; P.A. No. 22 single stem 0.0025 fill (Mill-Rose Co., 1985 East 59th Street, Cleveland, OH).

4.6 Holding fixture, coking-tube (for supporting coking tube by top end without marring or distorting the tube).

4.7 Oven, 99° to 104.4°C (210° to 220°F).

4.8 Thermometer, platinum-resistance or a thermometer certified by the Bureau of Standards (for weekly check of thermocouples at normal operating temperature).

4.9 Compressed-air source.

4.10 Millivoltmeter (precision potentiometer).

4.11 Gooch crucible with asbestos mat.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Silicon carbide paper, No. 400.

5.3 Solvent, dry-cleaning (A-A-59601D).

5.4 Cleaning solution, glass (concentrated sulfuric acid [3,0,2] saturated with potassium or sodium dichromate [1,0,1], technical grade).

5.5 *n*-Hexane, thiophene-free (ACS) [1,3,0].

5.6 Isopropyl alcohol (ACS), [1,3,0].

5.7 Petroleum ether (ACS) [1,4,0].

5.8 Acetic acid, 25% solution [2,2,1].

5.9 Distilled water.

5.10 Scouring powder, commercial grade.

5.11 Steel wool.

5.12 Pipe cleaners.

5.13 Cleaning compound, special (Penetone ECS; Penetone Corporation, 74 Hudson Avenue, Tenafly, New Jersey 07670; [201] 567-3000 or equivalent) [u,u,u].

5.14 New parts to be installed before each test (available form Erdco Engineering Corp.):

a. Pump shaft seal.
b. O-rings for sealing the filter, cooler, and oil-bypass relief valve.
c. Air-diffuser stone for coking head. (The stone in the humidifier is not replaced except as needed).

6. PROCEDURE

6.1 Ascertain (at least once a week) that all thermocouples are operating properly, using a platinum-resistance thermometer (or a thermometer certified by the Bureau of Standards) and immersing only the tip of the thermometer in the bath. Discard any thermocouple that is off by 3°C (6°F) or more; apply a correction factor to the reading if the thermocouple error is 1°C (2°F) or more.

NOTE 2: When the rig is in operation, a continuous check is available on the "oil-in" and coking-tube temperature since two independent thermocouples and instruments are used on each. The two readings on each of the above thermocouples should agree within 2.5°C (5°F). Disagreement in excess of 2.5°C (5°F) should be resolved immediately by a thorough check on instruments, leads, and thermocouples. The "oil-out" and test-section thermocouples should be checked daily with a precision millivoltmeter. This is primarily a check on the leads and measuring instrument which can be eliminated on Models 2 and 2W as the recording millivoltmeter on these models is not affected by variations in lead or contact resistance.

6.2 Clean bayonet thermocouples by swabbing and rinsing in isopropyl alcohol and then air dry. Clean bare-wire thermocouples by rinsing in isopropyl alcohol and using brushes, steel wool or No. 400 silicon carbide paper as necessary for deposit removal. After final rinsing, air dry.

6.3 Prepare the oil system of the tester as follows:

a. Disassemble all components of the test oil system completely (except for pipe-threaded aluminum joints which cannot be removed without damage).
b. Disassemble pump, relief valve, and all valves completely. Disconnect all lines including those from the pressure gage and from the humidifier to the coking tube.
c. Discard used O-rings, pump shaft seal, and air dispersion stone in the coking head.
d. Soak all reusable components of the test oil system, except the coking tube, in dry-cleaning solvent for 20 min.
e. Drain excess solvent, and soak for 4 h in special cleaning compound. Cleaner at 43° to 49°C (110° to 120°F).
f. Remove from cleaner, and rinse thoroughly with running water to remove all cleaner, brushing all parts thoroughly with the special metal brushes to remove all deposits.
g. Rinse all components thoroughly with clean isopropyl alcohol, then with clean petroleum ether, and blow dry with clean, dry air.
h. Inspect the parts for deposits, and repeat the cleaning if necessary (starting with the soak in special cleaning compound), until they all are completely clean.

NOTE 3: It is advisable to cut open lines and critical fittings periodically to check on the efficiency of cleaning. The most critical section is between the coking head and the cooler.

i. Clean glass coker-assembly body and glass oil-filler tube with scouring powder, rinsing afterwards with water then with isopropyl alcohol. Air dry and immerse both in glass-cleaning solution for at least 1 h. Rinse thoroughly using distilled water for the final rinse, then oven dry at 99° to 104.4°C (210° to 220°F).
j. Clean glass feeder flask and its connection by rinsing thoroughly with isopropyl alcohol and air dry.
k. Store all parts in a clean, dust-free location until ready for reassembly.

6.4 Check the water side of the oil cooler for cleanliness, and clean as necessary. (The frequency of the cleaning depends on the hardness of the water used.) With very hard water it may be necessary to de-lime the inside of the cooling-water tube after each test. Cleaning is best accomplished by corking the bottom end of the tube and filling it with dilute acetic acid (25%), allowing it to stand for 15 to 30 min as needed, and then flushing thoroughly with water. This cleaning is in addition to the regular cleaning procedure for the removal of deposits from the oil side.

6.5 Prepare the coking tube as follows:

a. Clean the coking tube by hand abrasion with No. 400 silicon carbide paper holding the tube by the top end in a jig or fixture designed to avoid marring or distorting the tube. (To avoid getting dust in the thermocouple wells it is best to plug them with paper during this operation.)
b. Inspect the surface of the tube for pits. If the tube is pitted, turn it down in a lathe. (However, when the diameter has been reduced from its original 2.984 to 2.883 cm [1.175 to 1.135 in] or less, replace the tube.) Then repolish it by hand.
c. After the final polishing with silicon carbide paper, wipe free of dust with clean rags. Then rinse thoroughly with *n*-hexane (including thermocouple wells). Blow dry with clean, dry air. (Thermocouple wells should also be swabbed out with clean pipe cleaners.)

CAUTION: After final cleaning, the coking tube must be handled only by the top end to avoid contamination of the coking surface.

d. Inspect the thermocouple wells for residual oil. If there is any evidence of oil, repeat the
n-hexane rinse and swab with pipe cleaners until all oil is removed.
e. Install a clean coking-tube thermocouple in one of the wells, and heat the tube to 204°C (400°F) by plugging in the power rig.
f. Allow the tube to cool, and store it in a dust-free location.

6.6 Weigh a clean coking tube (within 10 mg), and record the weight. Handle by the top to avoid contaminating coking surface.

6.7 Weigh a clean filter screen to the nearest 10 mg, and record the weight.

6.8 Clean a new coking-head diffuser stone, a new pump-shaft seal, and a complete set of new O-rings by rinsing in isopropyl alcohol, and then in petroleum ether, and allow them to air-dry.

6.9 Assemble clean components of the test section, installing the previously cleaned new parts (see 6.8)

CAUTION: Do not use compound of any type on threaded fittings, stopcocks, or elsewhere in the system.

6.10 After assembly, check to see that all fittings are tight, all valves are in proper position, bayonet thermocouples are "bottomed" in the coking tube, all thermocouples are registering, and the temperature indicators on all controllers and recorders read correct ambient temperature. (Adjust if necessary.)

6.11 Level coking head by means of adjusting screws.

6.12 Ascertain (at least once a month) that the air flowmeter is calibrated to deliver 300 mL/min of saturated air at atmospheric pressure and 25°C (77°F). In calibrating (to detect leaks in the flowmeter or line), use a definite flowmeter pressure (preferably 69 gage kPa [10 psig]) and a wet test meter, connecting from the discharge line of the humidifier to the inlet of the meter.

6.13 Flush the oil system as follows:

a. Charge the circulating system with unused isopropyl alcohol, and circulate for 5 min, alternating the flow several times between the filter (36, Figures 2 and 3), and the pressure-relief bypass valve (34). This is accomplished by use of valves (35) and (37). Drain thoroughly opening valve (37), and tubing connections as necessary for complete venting and draining. Discard the used isopropyl alcohol.
b. Repeat step (a) using unused petroleum ether. Drain thoroughly, and discard used petroleum ether.
c. If the first flush of petroleum ether comes out dirty or discolored, repeat until petroleum ether remains colorless. Drain thoroughly, and discard all used solvents.

6.14 Check the adjustment of the system as follows:

a. Open valve (29), and charge the system with test oil through oil filler system.

b. Turn on pump, and continue to add oil until level in coking head glass is at or slightly above the mark 3.8 cm ([1-1/2 in] below top of glass).

c. Fill humidifier half full of distilled water, and adjust air flow control valve to give an air flow of approximately 150 mL/min.

d. Turn on the coking tube heater, and set controller for 274°C (525°F). (It is imperative that this temperature is not exceeded during flow check since higher temperatures would cause coke formation). As the test oil heats and expands, drain as necessary to maintain a visible level in the coking head. It is desirable to have the oil level close to the top of the coking head glass. Valve (29) must be kept open.

e. Turn on the cooling water, and check the operation of the water solenoid valve by adjusting "oil-in" temperature controller to open and close solenoid, keeping water by-pass valve (17) closed and water trim valve (16) wide open. (When water flow is shut off, the cooler should drain in 10 s or less.)

f. Set "oil-in" temperature control to 149°C (300°F), keeping water-bypass valve (17) closed and water-trim valve (16) wide open.

g. As soon as "oil-in" temperature is above 121°C (250°F), check the setting of bypass relief valve (34) by cutting off filter flow at valve (35). Adjust the relief valve to give a pressure of 34.5 kPa gage (5 psig) higher than obtained when flowing through the filter.

h. Open valve (35) to direct flow through the filter.

i. Shut off coking-tube heater as soon as the "oil-in" temperature reached 138°C (280°F). Check hot flow rate by measuring the time required for a discharge of 100 mL of oil through three-way valve (28). Pour the 100 mL of oil back into the filler tube immediately, keeping valve (29) open. Calculate the flow rate in mL/min.

j. Turn on the coking tube heater.

k. If first hot flow check shows a rate of 300 . 5 mL/min, record the actual flow rate and proceed to step (1). If the flow rate is outside this limit, readjust the variable-speed-transmission speed control (14) to correct for the difference, and repeat steps (i) and (j) until a flow rate of 300 . 5 mL/min is obtained.

l. Direct oil flow through the bypass valve (34) by blocking the flow through the filter, and again check the hot oil flow as in step (i). If the flow rate is above 255 mL/min, record this value and proceed to 6.15. If the flow rate is below 225 mL/min, the pump is worn or defective and must be replaced or rebuilt before proceeding further. In this event, after replacing or rebuilding the pump, repeat 6.13 and 6.14.

<u>NOTE 4:</u> The entire sequence of flow check procedure (steps j though l) must be completed within 30 min to avoid excessive oil deterioration.

TABLE I. Standard operating conditions.

Condition	Value	
Coking-tube temperature, °C (°F)	310° ± 2.5°	(590° ± 5°)
"Oil-in" temperature, °C (°F)	149° ± 2.5°	(300° ± 5°)
Air temperature (test section), °C (°F)	48.9° ± 2.5°	(120° ± 5°)
Oil flow, mL/min	300 ± 5	
Air flow, mL/min	300 ± 5	
Oil level (aerated), below top of coking-head glass, cm (in)	3.81 ± 0.32	(1-1/2 ± 1/8)

6.15 Using the standard operating condition listed in Table I, conduct a 12-hour (± 10 min) run on the test oil used in adjusting the system (see 6.14). Perform the test as follows:

a. While the system is hot, adjust the oil level in the oil filter tube to about 3.8 cm (1-1/2 in) below the oil level mark on the tube (to allow for an increase in volume as a result of aeration).
b. Start the oil flow at 300 mL/min.
c. Adjust the air flow to 300 mL/min.
d. Fill the oil feeder flask completely with test oil, stopper tightly, and install in oil filler tube.

<u>NOTE 5:</u> The feeder flask must be full at the start of the test run (to minimize oil ejection due to air expansion). It is helpful to heat flask and oil to 54.4°C (130°F) prior to its installation. The stopper on the flask must be tight to avoid air leakage after installation.

e. Set coking tube temperature control to 310°C (590°F).
f. Check setting of "oil-in" temperature control for 149°C (300°F).
g. As soon as "oil-in" temperature reaches 138°C (280°F) start timing the test period by setting the timer (4, see Figure 1) on 12 h. If apparatus is so equipped, set watt-hour meter (5) to zero. Read and record all temperatures and pressures.
h. Adjust height of oil-filler assembly to maintain the aerated oil level in the coking head at the mark on the coking-head glass to within ± 0.32 cm (± 1/8 in). Maintain this level for duration of test. Keep valve (29) open for duration of test.
i. Check the operation of the coking-tube temperature control, and adjust to hold 310° ± 2.5°C (590° ± 5°F) coking-tube temperature. If the control does not hold within these limits, adjustment of the band width is required. (See instruction manual for the control).
j. Check "oil-in" temperature control, and adjust to hold 149° ± 2.5°C (300° ± 5°F) "oil-in" temperature. If the "oil-in" control does not hold within prescribed

limits, it can usually be corrected by adjusting the water supply to the cooler by means of trim valve (16).

k. Keep cabinet door closed throughout the test, opening only when necessary and for as short periods as possible. Check and adjust operation of ventilating and heating equipment to hold test-section air temperature at 48.9°C (120° . 5°F).

(1) Record the following data every 30 min for the duration of the test:

Oil-in temperature, °C (°F)
Oil-out temperature, °C (°F)
Coking-tube temperature, °C (°F)
Cabinet-air temperature, °C (°F)
Heater voltage
Watt-hour meter (total)
Air flow (rotameter setting and pressure), mL/min
Aerated-oil level (above or below mark), cm
Pump pressure, kPa (psig)

6.16 At the end of the 12-hour test run, shut off the coking-tube heater, and immediately shut filler valve (29). Remove feeder flask, and empty all fresh oil from the filler tube.

6.17 Shut off air flow.

6.18 With pump running, drain the used oil into a clean, dry container through drain valve (37). Save used oil if optional analysis is desired.

6.19 Turn off all switches on control panel.

6.20 While the test rig is still hot and as soon after the completion of the test run, check for sludge deposits in the filter as follows:

a. Remove the filter (see Figure 5) as a unit, and allow it to drain for 5 min with the inlet at the top and the filter housing tilted approximately 30° from the vertical.
b. Being careful not to dislodge sludge particles, disassemble the filter housing, and place the screen (deposit side up) on a clean, absorbent paper towel or on a piece of qualitative-grade filter paper.
c. After 10 min replace paper with a new one and allow screen to drain an additional 50 min.
d. Weigh the screen and deposits to the nearest 10 mg, and record.
e. Oils with very high sludge values may leave deposits on the upstream half of the filter housing. These deposits should be removed with petroleum ether and a policeman, and filtered through a tared Gooch crucible. The crucible and contents should be washed thoroughly with petroleum ether until the washings are completely free from discoloration. The crucible and contents are than dried

in an oven at 96°C (205°F) for 1 h, cooled at least 1 h in a desiccator, and reweighed. The resulting deposit weight should be added to that obtained from the filter screen, and the total obtained reported as sludge.

6.21 Check the coking tendency of the oil as follows:

a. Being extremely careful not to dislodge any deposits, remove the coking tube from the coking head.
b. Allow the tube to cool for at least 1 h.
c. Immerse the tube up to its flange in *n*-hexane, and allow it to soak for 10 min.

CAUTION: Handle carefully to avoid dislodging deposit.

d. Repeat the soaking for 10 min in clean *n*-hexane.
e. Swab out the thermocouple wells with pipe cleaners to remove any oil. Then install a clean thermocouple in one of the wells.
f. Connect the heater to the rig power source, and heat tube to 204°C (400°F) maximum.
g. Remove the thermocouple, allow the tube to cool in air at room temperature for at least 1 h, weigh to nearest 10 mg, and record.

6.22 Disassemble and clean the deposition test rig. (Refer to 6.3, 6.4, and 6.5.)

7. CALCULATIONS

7.1 Calculate the following:

a. Sludge is expressed as the difference between the final and initial weights of the filter screen.
b. Coke is expressed as the difference between the final and initial weights of the coking tube.
c. Deposit rating is calculated as follows:

$$Deposit\ rating = Sludge + 10 * coke$$

8. REPORTING

8.1 Report the weight, in g, of the sludge and the coke separately, and the deposit rating of the oil.

8.2 Report the following operating data:

a. Elapsed time (to within 1/2 h) to pump-pressure increase of 34.5 gage kPa (5 psig).

b. Oil consumption (difference between initial and final oil volumes in feeder flask).
c. Unusual operational condition (such as foaming of oil in coking head).
d. Elapsed time (and reasons for termination) if test run must be stopped before 12 h.

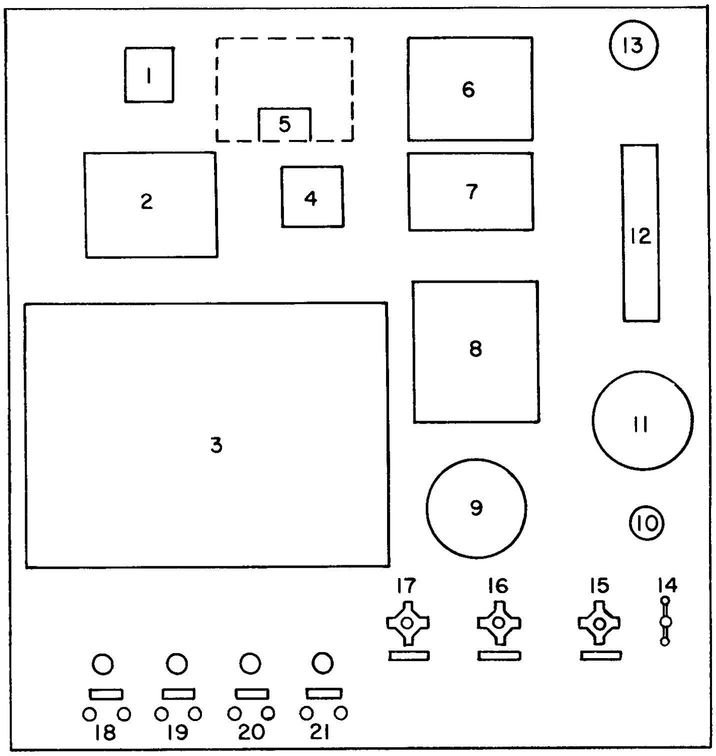
9. PRECISION

9.1 Repeatability. Two or more runs on the same oil in the same apparatus and by the same operator should differ from their mean by no more than 0.75 deposit rating.

9.2 Reproducibility. The average values for the same oil obtained on different apparatus or by different operators (or both) should not differ by more than 0.75 Deposit Rating.

Method prepared by:

Air Force-AFWAL/POSL-1986



1. Voltmeter, 0 to 300 volts
2. Controller (stepless), coking-tube temperature, -18° to 430° C (0° to 800° F)

*3. Strip chart recorder (potentiometer), 6-channel; or differential pressure recorder, 0 to 25 cm (0 to 10) inches of mercury

4. Timer, 24-hour

*5. Watt-hour meter

6. "OFF-ON" controller, oil-temperature, -18° to 320° C (0° to 600° F)

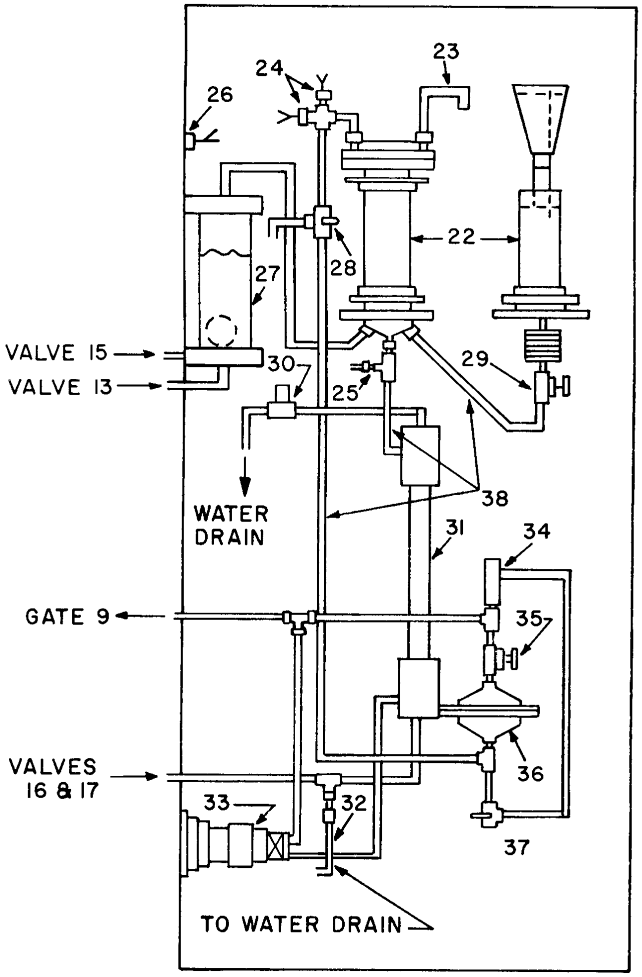
*7. Pyrometer

*8. Selector switch

9. Filter pressure gage, 0 to 69 gage kPa (0 to 10 psig)
10. Pressure regulator, air-flow
11. Air-pressure gage, 69 gage kPa (10 psig) (working)
12. Air flowmeter, 0 to 500 mL/min
13. Air throttle valve
14. Speed control, variable-speed pump-drive
15. Humidifier-water valve
16. Water trim valve (flow regulation through solenoid valve)
17. Solenoid-bypass water valve
18. Pump switch and indicating light
19. Coking-tube heater switch and indicating light
20. Air solenoid-valve switch and indicating light
21. Ambient-air heater switch and indicating light (off-on by thermoswitch)

* Optional.

FIGURE 1. Deposition tester instrument and control section.



22. Coking head and oil reservoir
23. Vent line
24. Oil-in thermocouples
25. Oil-out thermocouple
26. Test-cabinet ambient-temperature thermocouple
27. Humidifier
28. Bypass valve to coking head
29. Shut-off valve, oil reservoir
30. Check valve (air bleed)
31. Oil cooler
32. Orifice (water-drain)
33. Oil pump and variable-speed transmission
34. Pressure-relief bypass valve
35. Shut-off valve, test-filter
36. Test filter
37. Drain valve, test-oil system
38. All test-oil tubing shall be 1/4 inch aluminum tubing

FIGURE 2. Test section components.

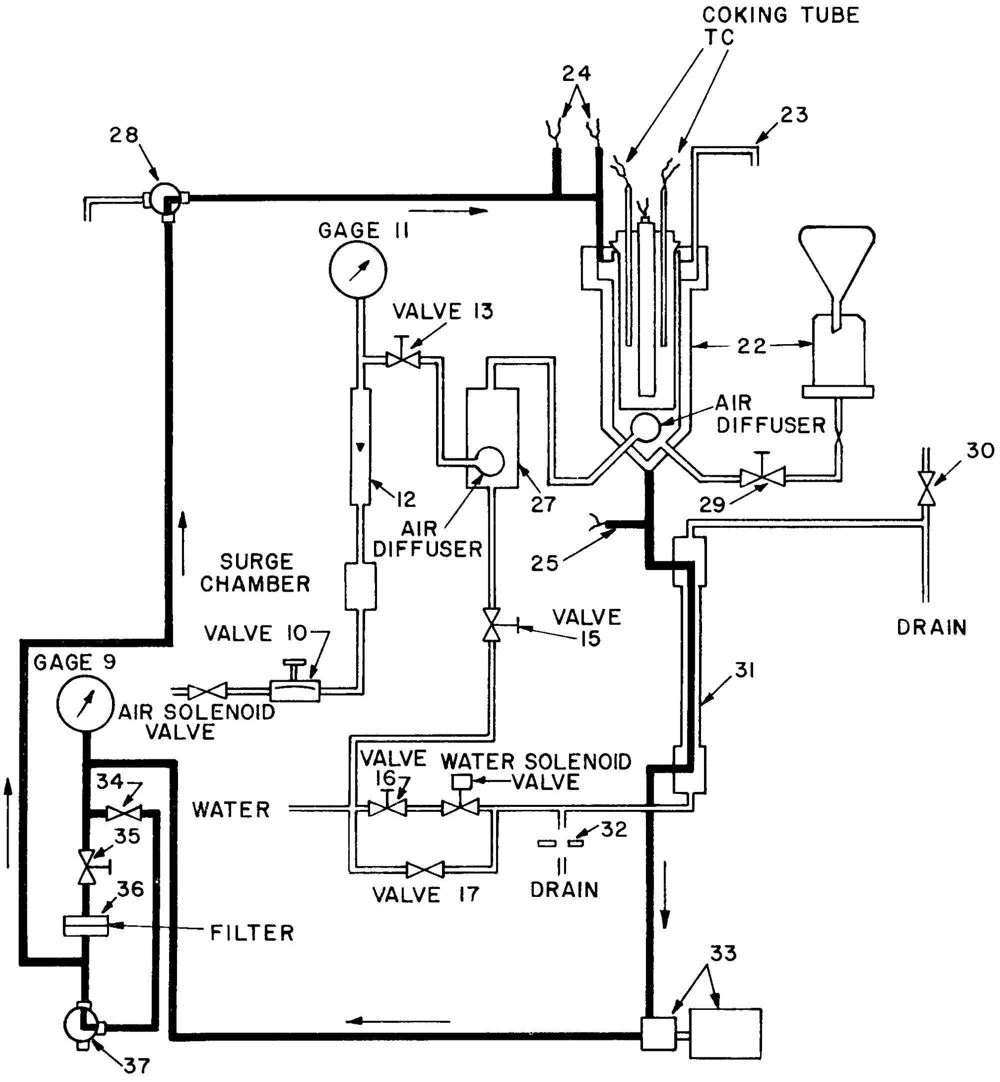
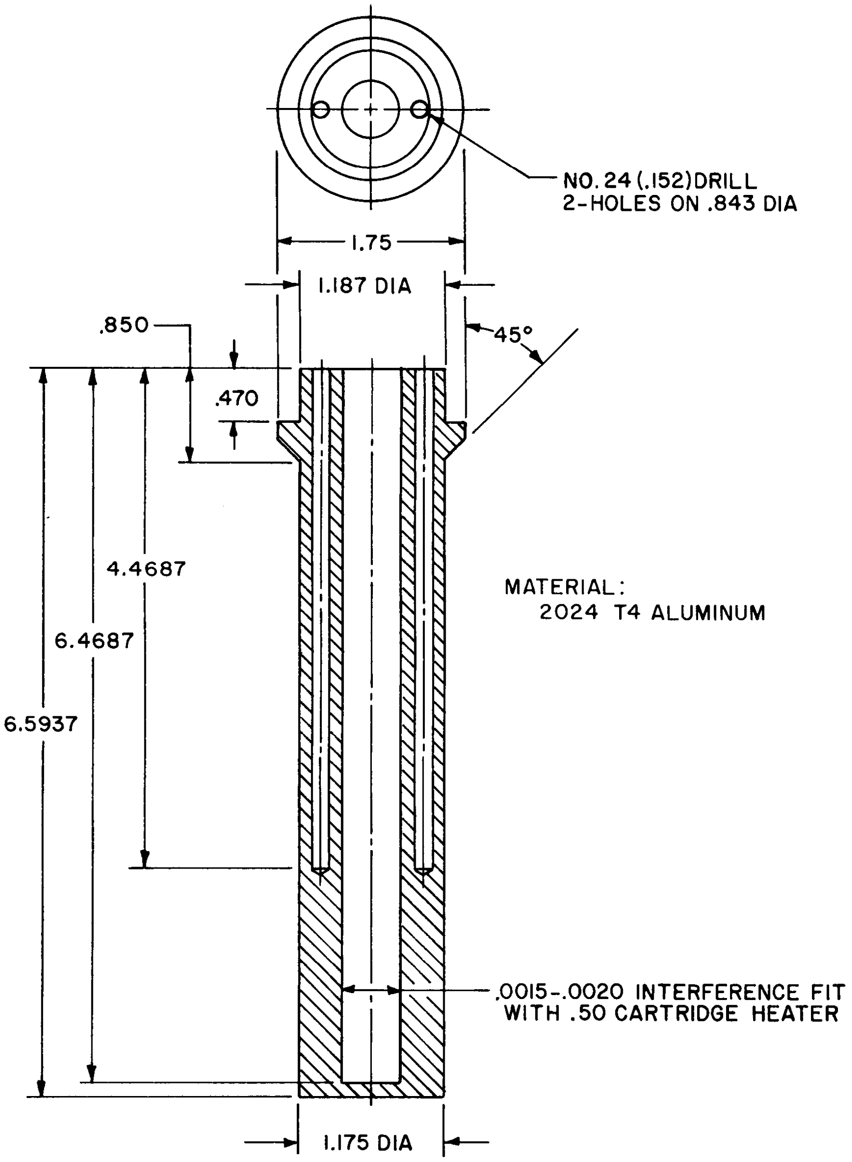


FIGURE 3. WADC deposition tester (schematic).



NOTE: DIMENSIONS IN INCHES.

FIGURE 4. Coking tube.

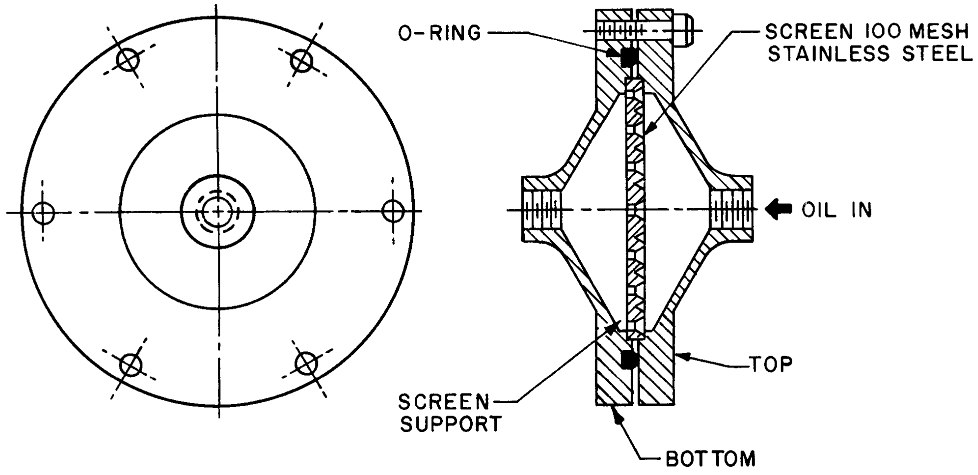


FIGURE 5. Line filter.

Method 5304.5
September 30 1986

CORROSIVENESS OF GREASES OR SEMI-SOLID PRODUCTS AT 25°C

1. SCOPE

1.1 This method is used for determining the presence of corrosive compounds in grease or other semi-solid products at room temperature (25°C; 77°F).

2. SUMMARY

2.1 The method consists of half-covering two copper strips with the product under test and examining for corrosion after 24 h.

3. SAMPLE

3.1 Approximately 5 g of material to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Watch glass for covering sheets.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measure, and spill practices of each chemical are explained.

5.2 Copper strips (2), (ASTM B 152/152M), 2.5 x 2.5 x 0.3 cm (1 x 1 x 1/8 in).

5.3 *n*-Hexane (ACS) [1,3,0].

5.4 Acetone (ASTM D 329).

5.5 Abrasive paper (silicon carbide or alumina), various grade of grit.

5.6 Abrasive paper, silicon carbide, 240 grit.

5.7 Silicon carbide, 150 mesh.

5.8 Cotton, absorbent.

6. PROCEDURES

6.1 Prepare the copper strip as follows:

6.1.1 Using progressively finer grades of abrasive paper, remove all surface blemishes from one side of strip.

6.1.2 Polish strips with the 240-grit paper to remove any marks produced by the coarser abrasive. Do not touch the strips with the fingers. Handle strips only with lint-free paper or tongs. Store strips immersed in acetone until ready for final polishing.

6.1.3 Remove strips from the acetone, and final polish one side to a mirror finish, using 150-mesh silicon carbide grit (held on an acetone-moistened cotton pad).

6.1.4 Clean all metal dust from the strip by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled.

6.1.5 Wash strips in fresh warm *n*-hexane, then in fresh warm acetone, and allow to air dry.

NOTE 1: Caution. *n*-Hexane and acetone are toxic. Use only in a well ventilated area. Do not breathe its fumes nor allow it to come in contact with the skin. Keep all flames away from these materials.

6.2 Place approximately one-half of the sample on each strip, taking care to leave approximately half of the polished surface exposed.

6.3 Cover the strips with inverted watch glasses, and let stand at room temperature for 24 h.

6.4 Remove the watch glasses, and visually examine the sample for a green color.

6.5 Wash the copper strips in non-corrosive *n*-hexane and inspect them for brown stains.

7. CALCULATION

7.1 This section is not applicable to the test procedure.

8. REPORT

8.1 Report the lack or the presence of any evidence of corrosion (green color in sample or stain on copper sheets).

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986

Method 5305.1
September 30 1986

CORROSIVENESS OF LUBRICANTS AT 232°C (450°F)

1. SCOPE

1.1 This method is used for determining the corrosive tendencies of lubricants at high temperatures. It consists of immersing copper and silver strips in the sample at 232°C (450°F) for 50 h, and then examining them for weight change.

2. SUMMARY

2.1 This method duplicates an extended, high-temperature engine or gear box environment. Copper and silver strips are exposed to the heated lubricant for 50 h and carefully cleaned and weighed for loss of weight due to corrosive action resulting from corrosive decomposition products being formed in lubricant.

3. SAMPLE SIZE

3.1 Approximately 400 mL of lubricant to be tested.

4. REFERENCES, STANDARDS AND APPARATUS

4.1 Oven, explosion-proof, 232° . 3°C (450° . 5°F).

4.2 Copper strip (ASTM B 152/B 152M), 3.8 cm x 3.8 cm x 0.16 cm (1-1/2 x 1-1/2 x 1/16 in).

4.3 Silver strip, pure electrolytic (99.9% purity), 3.8 cm x 3.8 cm x 0.32 cm (1-1/2 x 1-1/2 x 1/8 in).

4.4 Beakers (2), borosilicate glass, Griffin low-form, 400 mL.

4.5 Supports (2), glass (for holding copper or silver strip vertically in beaker with point contact, so that strip does not touch the sides or bottom of the beaker).

4.6 Balance, sensitivity 0.1 mg.

4.7 Electrolytic bath, carbon-rod anode, 0.5 A (min.), containing sulfuric acid solution (5% by weight in distilled water) as the electrolyte.

4.8 Tongs, stainless steel.

4.9 Watch glass (2), 10 cm or larger.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Solvent, dry-cleaning (A-A-59601D).

5.3 1,1,1-Trichloroethane (reagent grade) [2,1,0].

5.4 Acetone (ACS) [1,3,0].

5.5 Nitric acid solution (3 parts concentrated nitric acid and 7 parts distilled water) [2,0,0].

5.6 Pumice, FFF grade.

5.7 Eraser (Eberhard Faber "Pink Pearl" No. 100, or equivalent).

5.8 Cleaning solution, glass (concentrated sulfuric acid [3,0,2] saturated with potassium or sodium dichromate [1,0,1], technical grade).

NOTE: If the glass-cleaning solution cannot remove all deposits (see 6.1) use a special cleaner, such as Turco Transpo cleaner (Turco Products Co., Chicago, IL).

5.9 Distilled water.

6. PROCEDURE

6.1 Clean two glass supports and two 400-mL beakers as follows:

a. Rinse with dry-cleaning solvent to remove excess oil.
b. Place each support in its beaker, and fill each beaker with glass-cleaning solution. Let them stand for at least 8 h or until all traces of deposits are removed.
c. Remove from the glass-cleaning solution, and rinse the glassware with running tap water followed by distilled water.

6.2 Handling only with tongs, clean the copper and silver strips in separate bath solutions as follows:

a. Wash with dry-cleaning solvent.

b. Immerse in dilute nitric acid solution for 15 ± 1 s.
c. Rinse the silver strip only in hot 82° to 100°C (180° to 212°F) tap water, and then wet polish with pumice. (No polishing of the copper strip is required.)
d. Rinse the strip in hot tap water, and then in hot distilled water.
e. Dip in acetone, and allow to air dry.

6.3 Handling the strips with tongs, weigh each strip to the nearest 0.1 mg.

CAUTION: In succeeding steps the strips must be handled only with tongs until after they have been weighed (see 6.9) at the end of the test.

6.4 Mount each strip in a glass support then place each support in a separate beaker so that the strip does not touch the sides of the beaker.

6.5 Add 200 mL of the sample to each beaker, and cover with watch glass to avoid evaporation losses.

6.6 Store both beakers in an oven for 50 h at 232° ± 3°C (450° ± 5°F).

6.7 At the end of the heating period, remove both beakers from the oven and allow them to cool to room temperature.

6.8 Remove the copper and silver strips from the supports and clean them as follows:

a. Wash each strip with successive portions of fresh, hot 60°C (approx. 140°F) 1,1,1-trichloroethane, to remove as much discoloration and deposit as practicable.
b. If carbonaceous deposits remain, place the strip (as a cathode) in the electrolytic bath for 10 min at 0.5 A.
c. Wash each strip in hot, running water, while rubbing it with an eraser.
d. Rinse each strip with distilled water, then with acetone, and air dry both strips.

6.9 Weigh each strip to the nearest 0.1 mg, and compute the change in weight of each strip (see 7.1).

6.10 Measure the surface area of each strip (both sides) (see 7.1).

7. CALCULATIONS

7.1 The calculations to determine mg/cm^2 as follows

$$CW = \frac{W_1 - W_2}{2 * W * L}$$

where:
CW = change in mg/cm^2
W_1 = original weight in mg.
W_2 = final weight in mg.
W = width of metal strip in cm.
L = length of metal strip in cm.

8. REPORTING

8.1 Report change in weight in mg/sq cm rounded off to the nearest mg.

9. PRECISION

9.1 Repeatability. Results of each test obtained by one operator using the same apparatus must not vary by more than that shown in Table 1.

9.2 Reproducibility. Results of each test obtained by different operators or different apparatus must not vary by more than that shown in Table 1.

TABLE 1 - Precision.

Average weight change mg/cm^2	Permissible variation	
	Repeatability	Reproducibility
0 to 0.46 Above 0.46	0.046 mg/cm^2 10%	0.09 mg/cm^2 20%

Method prepared by:

Air Force - SFTL - 1986

Method 5306.5
September 30 1986

CORROSIVENESS OF EMULSIFIABLE CUTTING FLUIDS

1. SCOPE

1.1 This method is used for determining the corrosiveness of soluble cutting oils and chemical type metal working fluids on brass, copper, cast iron, and steel.

2. SUMMARY

2.1 The test consists of placing a metal strip and the sample in a test tube. A bubble tower filled with sodium hydroxide is used to permit air into the test tube at a specified rate and temperature. The strip is examined for conformance with specification requirement.

3. SAMPLE SIZE

3.1 Approximately 10 mL of the fluid to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 ASTM D 130 - Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test.

4.2 Source of clean compressed air.

4.3 Air pressure regulator.

4.4 Approximately 7.3 m (24 ft) of 0.6-cm (1/4-in) I.D. rubber tubing.

4.5 Four screw type pinch clamps.

4.6 One bubble tower, capacity 500 mL.

4.7 Four test tubes, approximately 3.2 cm O.D. by 20.0 cm long.

4.8 Four pieces of glass tubing, 0.6 . 0.1 cm I.D. by 23.0 . 1 cm long.

4.9 Forceps, stainless steel.

4.10 Graduated cylinder, capacity, 250 mL.

4.11 Magnifier, 5X.

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 500 mL of sodium hydroxide solution containing approximately 25% NaOH by weight [3,0,1].

5.3 *n*-Hexane, commercial [1,3,0].

5.4 1,1,1-Trichloroethane (ASTM D 4126).

5.5 Distilled water.

5.6 Cleaning tissue.

5.7 Two copper strips, approximately 76 □ 13 cm by 0.32 cm (3 □ l/2 □ 1/8 in) (ASTM B 152/152M, electrolytic tough pitch, cold-rolled, half-hard temper).

5.8 Two brass strips, approximately 76 □ 13 □ 0.32 cm (3 □ 1/2 □ 1/8 in) (ASTM B 121/B 121M, ASTM B 36/ B 36M, composition 2 [cartridge brass], half-hard temper).

5.9 Two cast iron strips, approximately 76 □ 13 □ 0.32 cm (3 □ 1/2 □ 1/8 in) (ASTM A 48, class 20, strip only).

5.10 Two steel strips, approximately 76 □ 13 □ 0.32 cm (3 □ 1/2 □ 1/8 in) (QQ-S-698, physical quality, cold-rolled, half-hard temper - CR half-hard No. 2 temper).

6. PROCEDURE

6.1 Prepare two strips of each type of metal listed in section 4 in accordance with ASTM D 130.

6.2 Using the forceps, place one strip of each type of metal in a separate test tube. Place the duplicate strips in a bottle filled with the normal hexane. Keep this bottle tightly stoppered until time to examine the strips.

NOTE: Caution. Normal hexane is flammable. Use only in a well ventilated area. Keep all flames away from the normal hexane.

6.3 Prepare a solution of the cutting fluid by placing 190 mL of distilled water and 10 mL of the cutting fluid in a 250-mL graduated cylinder. Stopper the graduated cylinder and invert it several times. Continue inverting the graduate until a uniform mixture is obtained.

6.4 Place 50 mL of the cutting fluid solution in each of the four test tubes. Mark the fluid level on the outside of each test tube and maintain this level by the addition of distilled water during the course of this test.

6.5 Fill the bubble tower with the sodium hydroxide solution and connect the tower, through the air pressure regulator, to the source of compressed air by means of the rubber tubing.

6.6 Connect each length of glass tubing, by means of rubber tubing and an appropriate manifold or series of Y-tubes, to the discharge outlet of the bubble tower. Place a screw type pinch clamp on each piece of rubber tubing leading to the glass tubes.

6.7 Insert each glass tube to the bottom of a test tube and adjust the air flow into each tube to produce approximately one bubble per second by means of the air pressure regulator and pinch clamps.

6.8 Permit the air to bubble into the tubes at room temperature 24° . 3°C (75° . 5°F) for a period of 48 h.

6.9 At the end of the test period, remove the metal strips from the test tubes. If the cutting fluid under test is a soluble oil type, wash the metal strips in 1,1,1-trichloroethane and permit them to air dry. If the cutting fluid under test is a chemical solution type, wash the metal strips in distilled water and blot them dry with facial tissue.

6.10 Remove the duplicate metal strips from the bottle of normal hexane and permit them to air dry. Examine the exposed and duplicate metal strips under 5X magnification for conformance with the specification requirement.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Comply with specification requirement.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - AR – 1986

Method 5308.7
September 30, 1986

CORROSIVENESS AND OXIDATION STABILITY OF LIGHT OILS (METAL SQUARES)

1. SCOPE

1.1 This method is used for testing hydraulic oils (spindle oils), and similar, highly refined, light oils to determine their ability to resist oxidation and their tendency to corrode various metals.

2. SUMMARY

2.1 Metal specimens are suspended in a measured amount of lubricant. The oil, at an elevated temperature, is blown with air for a period of time. When the test is completed, the oil is tested to determine the extent of degradation. The metal specimens are examined for corrosion.

3. SAMPLE SIZE

3.1 Approximately 225 mL of oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Test tube, borosilicate, standard-wall; 5.0 ± 0.3 cm O.D., 50.0 ± 1 cm long; with open end formed as a male ground-glass joint, joint is T_s 71/60.

4.2 Condenser, Allihn type. Borosilicate, 3.5 cm O.D. (min.) by 300 ± 2.0 cm long jacket, with the lower end formed as a female ground-glass joint to mate with the test tube; joint is T_s 71/60; drip tip in T_s joint, 60 cm, min length; tabulations are parallel at opposite sides of the condenser, they are about 1.0 cm O.D.; open end has an I.D. of approximately 2.2 cm.

<u>NOTE 1:</u> The test tube (4.5.1) and condenser (4.5.2) may be obtained from:

John A. Glover
8225 Parkview Avenue
Munster, Indiana

4.3 Air tube, borosilicate, standard wall; 0.6 to 0.8 cm O.D., length at least 5.0 cm greater than combined length of assembled test tube and condenser, about 94.0 cm, with one end drawn to a 0.16 ± 0.04 cm (l/16 ± 1/64 in) diameter orifice. A capillary tube with a 0.16 ± 0.04 cm (1/16 ± 1/64 in) bore may be used.

4.4 Bath constant - temperature, capable of maintaining the sample ±0.5°C (±1°F) of the specified temperature.

NOTE 2: A liquid medium bath is not to be used over 135°C (275°F). Above 135°C (275°F) use an aluminum block. The heaters should be placed on the sides of the aluminum block. Base heaters are to be avoided. A satisfactory heater arrangement is described in method 350 of this standard. Unless otherwise specified the bath temperature is 121°C (250°F) for all oils except spindle oils where it is 100°C (212°F).

4.5 Assembly fixture, wood (slotted to hold metal squares for tying with cord, as shown in Figure 1).

4.6 Balance, sensitivity 0.1 mg.

4.7 Microscope, 20 diameter magnification.

4.8 Air-supply, clean, dry, low-pressure, 3-5 ± 0.5 L/hr.

4.9 Forceps, stainless-steel.

4.10 Brush, short-bristled, stiff (typewriter cleaning brush, or equal).

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Metal squares (l each) 0.081 cm (0.032 in) thick by 2.5 cm (1 in) square, with and without two drilled holes (Figure l), as follows:

5.2.1 Copper, electrolytic (ASTM B 152/152M).

5.2.2 Steel, carbon, mild (ASTM 1008A/1008M, class l, commercial bright finish).

5.2.3 Aluminum alloy (ASTM B 209, temper T-3 or T-4).

5.2.4 Magnesium alloy (SAE AMS 4375, 4376, 4377)

5.2.5 Anodes, Cadmium (A-A-51126B).

5.3 Cord (cotton, nylon, or linen), light-weight, clean (for tying metal squares together).

NOTE 3: A fine stainless steel wire may be used to tie the metal squares together when a test temperature of over 204°C (400°F).

5.4 Abrasive paper (silicon carbide or alumina), various grades of grit.

5.5 Abrasive paper, silicon carbide, 240 grit.

5.6 Silicon carbide grains, 150 mesh.

5.7 Cotton, absorbent.

5.8 1,1,1-Trichloroethane (ASTM D 4126).

5.9 Acetone (ACS), sulfur-free [1,3,0].

5.10 Laboratory filter paper.

5.11 Naphtha, Aromatic (TT-N-97) [1,4,0].

6. PROCEDURE

6.1 Determine the acid number of the sample by method ASTM D 664 or D 974, whichever is applicable.

6.2 Determine the viscosity in centistokes of the sample by method ASTM D 445 at 40°C unless otherwise specified.

6.3 Wash a length of the cord by boiling it in distilled water for 10 min, and allow it to dry.

6.4 Prepare the solid cadmium squares by removing any burrs from the drilled holes.

6.5 Prepare the aluminum copper, magnesium, and steel squares. For testing spindle oil, use only metal squares of copper and steel. The squares are prepared as follows:

6.5.1 Using progressively finer grades of abrasive paper, remove all surface blemishes from both sides and all four edges of each square, and any burrs from the drilled holes.

NOTE 4. Caution. In all succeeding steps, handle the squares with tongs or filter paper only, until the final weighing (see 6.11).

6.5.2 Polish each square with the 240 grit paper to remove any marks produced by coarser abrasive. Store squares immersed in separate containers of acetone until ready for final polishing.

NOTE 5: Caution. Acetone is flammable. Use only in a well ventilated area. Keep all flames away from acetone.

6.5.3 Remove each square from the acetone, and final polish both sides to a mirror finish, using 150 mesh silicon carbide grit (held on an acetone-moistened cotton pad).

6.5.4 Clean all metal dust from the square by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled.

6.6 Continue preparation of all five squares as follows:

6.6.1 Wash squares in warm 1,1,1-trichloroethane (approximately 54°C [130°F]), rinse with 1,1,1-trichloroethane, and allow them to air-dry.

NOTE 6: Caution. 1,1,1-Trichloroethane is toxic. Do not breathe its fumes nor allow it to come in contact with the skin.

6.6.2 Immediately weigh each square to within 0.1 mg.

6.6.3 Arrange squares in the wooden assembly fixture in the pattern shown in Figure l, making certain that the magnesium and copper squares do not touch each other. (When testing spindle oil, the metal squares shall be placed in the test tube in such a manner as to form a "V" in the bottom of the test tube.)

6.6.4 Using only forceps to handle the clean cord, tie the squares together as shown in Figure 1.

6.7 Prepare new test set up as follows:

6.7.1 Insert the tied metal squares in the test tube, positioning the squares vertically (so that the air tube can be inserted to touch the bottom of the test tube). Weigh the air tube, test tube, and contents together to within 0.1 g.

6.7.2 Add 100 . 1 mL of sample to the test tube, reweigh the air tube, test tube, and contents together to within 0.1 g, and determine weight of sample added.

6.7.3 Assemble the test tube and condenser and mount the assembly so that 30 . 5 cm of test tube is submerged in the bath operating at specified temperature.

6.7.4 Start the flow of cooling water through the condenser jacket.

6.7.5 Insert the air tube (orifice-end down) through the condenser and into the sample and support it so that its orifice is within 0.3 cm (l/8 in) of the bottom of the tube. (When testing spindle oil, orifice should be within 0.6 cm (l/4 in) of the junction of the metal square.)

6.8 Connect the source of clean, dry air (3 ± 0.2 L/hr for spindle oils; 5 ± 0.5 L/hr for other oils) to the air tube and allow the air to flow for 168 h.

6.9 At the end of the specified period, shut off the air-flow, and disassemble, and check test setup as follows:

6.9.1 Remove air tube and disconnect condenser.

6.9.2 Remove test tube from constant-temperature bath, and wipe off the outside of the tube with a naphtha-dampened cloth.

<u>NOTE 7:</u> Caution. Naphtha is flammable. Use only in a well ventilated area. Keep all flames away from naphtha.

6.9.3 Reweigh the air tube, test tube, and contents to within 0.1 g, determine weight of sample remaining, and compute the percentage of weight loss resulting from evaporation of sample (see 7.1). (If the evaporation loss is greater than 8%, leakage is present. Correct the leak, and repeat the determination, using fresh sample and new metals squares.)

6.9.4 Using forceps, withdraw the metal squares from the test tube, and remove the cords holding them together. (Retain the test tube and sample for further examination.)

6.10 Prepare squares for examination as follows:

6.10.1 Using forceps, wash each square individually in warm 1,1,1-trichloroethane (approximately 54°C [130°F]).

6.10.2 Repeat the washing, using fresh 1,1,1-trichloroethane and scrubbing the squares with the short-bristled brush, until the 1,1,1-trichloroethane shows no additional discoloration, and allow the squares to air-dry.

6.11 Examine each square as follows:

6.11.1 Reweigh each square to the nearest 0.1 mg and compute (in mg/cm^2 of surface) the change in weight of each square (see 7.2).

6.11.2 Using the 20-power microscope, carefully examine each square, and note any evidence of pitting or etching.

6.11.3 Note the color of any stains present on the copper square.

6.12 Examine the sample in the test tube as follows:

6.12.1 Inspect for any evidence of insoluble or gummy material as required by the specification and record.

6.12.2 Determine the acid number by method ASTM D 664 or D 974, whichever is applicable, and calculate the change from the original acid number (see 6.1 and 7.3).

6.12.3 Determine viscosity in centistokes by method ASTM D 445 at 40°C (104°F) (unless otherwise specified), and calculate the percentage change from the original centistoke viscosity (see 6.2 and 7.4).

7. CALCULATIONS

7.1 Evaporation loss:

$$L = \frac{W_1 - W_2}{W_1} * 100$$

where:
L = percentage evaporation loss
W_1 = initial weight
W_2 = final weight

7.2 Change in metal square weight:

$$M = \frac{(M_2 - M_1) * 1000}{2 * (W * L)^2}$$

where:
M = change in metal weight per surface area (mg/cm^2)
M_2 = final weight in g
M_1 = original weight in g
W = width of metal square in cm
L = length of metal square in cm

7.3 Change in acid number:

$$A = AN_2 - AN_1$$

where:
A = change in acid number
AN_2 = final acid number
AN_1 = original acid number

7.4 Change in viscosity:

$$C = \frac{V_2 - V_1}{V_1} * 100$$

where:
C = change in viscosity
V_2 = final viscosity in centistokes
V_1 = original viscosity in centistokes

8. REPORTING

8.1 Report as corrosiveness stability the change in weight of each of the metal squares in mg/cm^2 and the appearance of each of the metal squares.

8.2 Report as oxidation stability of the fluid the result of the insoluble gummy material inspection, the change in acid number, and the change in viscosity.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986

Method 5309.5
September 30, 1986

CORROSIVENESS OF GREASES
(Copper Strip, 100°C)

1. SCOPE

1.1 This method is used for determining the corrosive properties of grease at elevated temperatures.

2. SUMMARY

2.1 The test consists of partially immersing a copper strip in the sample at 100°C for 24 h then visually examining both sample and strip for evidence of corrosion.

3. SAMPLE SIZE

3.1 Approximately 20 mL of grease to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Oven, 100°C.

4.2 Microscope, approximately 60X diameter magnification.

4.3 Test tube, clean, dry, approximately 2.5 cm diameter by 10 cm long.

4.4 Support (beaker) for holding test tube approximately vertical.

4.5 Forceps, stainless steel.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Copper strip (Copper and Brass Research Association No. 110 Electrolytic Grade), soft tempered, annealed, approximately 1.3 × 7.6 × 0.15 cm or 0.3 cm.

5.3 *n*-Hexane (ACS Grade) non-corrosive (as determined by ASTM D 130) [1,3,0].

5.4 Acetone (ACS Grade) [1,3,0].

5.5 Polishing materials.

5.5.1 Fine silicon carbide or alumina grit paper.

5.5.2 240-grit silicon carbide paper.

5.5.3 150-mesh silicon carbide grains.

6. PROCEDURE

6.1 Prepare finish of copper strip as follows:

a. Surface preparation - Remove all blemishes from all six sides of the strip with silicon-carbide or alumina grit paper of such degree of fineness as is needed to accomplish the desired results effectively (see Note 1). Finish with 240-grit silicon carbide paper, removing all marks that have been made by other grades of paper used previously. Immerse the strip in acetone (see Note 2) from which it may be with-drawn immediately for final polishing or in which it may be stored for future use.

NOTE 1: As a practical manual procedure for surface preparation moisten it with kerosene or acetone and rub the strip against the paper with a rotary motion protecting the strip from contact with the finger with an ashless filter paper. Alternatively the strip may be prepared by use of motor driven machines using appropriate grades of dry paper or cloth.

NOTE 2: Caution. Acetone is both toxic and flammable. Use only in a well ventilated area. Do not breathe its fumes nor allow it to come in contact with the skin. Keep all flames away from the acetone.

b. Final polishing - Remove a strip from the acetone. Holding it in the fingers protected with the ashless filter paper, polish first the ends and then the sides with 150-mesh silicon carbide grains picked up from a clean glass plate with a pad of absorbent cotton moistened with a drop of acetone. Wipe vigorously with fresh pads of absorbent cotton and subsequently handle only with stainless steel forceps; do not touch with fingers. Clamp in a vise and polish the main surfaces with silicon carbide grains on absorbent cotton. Rub in the direction of the long axis of the strip, carrying the stroke beyond the end of the strip before reversing the direction (see Note 3). Clean all metal dust from the strip by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled. When the strip is clean, immediately immerse it in the prepared sample.

NOTE 3: It is important to polish the whole strip uniformly to obtain a uniformly strained strip. If the ends show wear (surface elliptical) they will likely show more corrosion than the center. The use of a vise will facilitate uniform polishing.

6.2 Fill test tube with sample to depth of 5 cm. Remove air pockets from the grease by tapping the tube on the bench or table as it is being filled.

6.3 Using stainless steel forceps, insert copper strip until it touches the bottom of the tube.

6.4 Place the test tube (supported approximately vertically in 100°C) oven for 24 h (unless otherwise specified).

6.5 At the end of the specified time, remove test tube from oven and allow it to cool at room temperature.

6.6 Using forceps, remove copper strip, and wash in non-corrosive ACS grade n-hexane. (See Note 4.)

NOTE 4: Caution. *n*-Hexane is both toxic and flammable. Use only in a well ventilated area. Do not breathe its fumes nor allow it to come in contact with the skin. Keep all flames away from the n-hexane.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report whether the section of the strip immersed in the sample is darker in color than the exposed section.

8.2 Report by describing any corrosion of the strip when examined under a microscope of approximately 60 diameters magnification.

8.3 Report development of any green color in the grease.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Navy - NADC - 1986

Method 5321.2
September 30 1986

CORROSION OF LEAD BY LUBRICATING OILS

1. SCOPE

1.1 This method is used for measuring the corrosiveness of lubricating oils on lead in the presence of a copper catalyst.

2. SUMMARY

2.1 It consists of rotating panels of lead and copper in the test lubricant under specific conditions, and determining the degree of corrosion from the change in weight of the lead panel.

3. SAMPLE SIZE

3.1 Approximately 500 mL of the oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Sample tube, SOD (Standard Oil Development), available from Koutes Co., Spruce St., Vineland, NJ 08360 or SGA Scientific, Inc., 735 Broad St., Bloomfield, NJ 07003 (see Figure 1).

4.2 Bath, constant-temperature, 163° " 1°C (325° " 2°F), minimum depth 31 cm (12 in).

4.3 Motor, stirring, 600 " 50 rpm.

4.4 Shaft, stirring, with attaching hardware (see Figure 2).

4.5 Air supply, clean, dry, approximately 7 gage kPa (1 psig) to provide flow of 0.940 " 0.047 L/min (2 " 0.1 ft^3/h).

4.6 Balance, sensitivity "1 mg.

4.7 Forceps, stainless steel.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Test panel, chemical-lead grade (ASTM B 29-55 or equal) 3.5 H 4.13 H 0.16 cm (1-3/8 H 1-5/8 H 1/16 in), conforming to Table I.

TABLE I. Composition of lead test pane.

Element	Content percent	
	Maximum	Minimum
Silver	0.020	0.002
Copper	0.080	0.040
Arsenic, antimony, and tin	0.002	-----
(total)	0.001	-----
Zinc	0.002	-----
Iron	0.005	-----
Bismuth	-----	99.90
Lead (by difference)		

5.3 Test panel, copper, electrolytic (ASTM B 152/152M, 3.5 H 4.13 H 0.130 cm [1-3/8 H 1-5/8 H 0.051 in] [16 gage]).

5.4 Naphtha, petroleum (TT-N-97).

5.5 Acetone (ASTM D 329).

5.6 Steel wool.

5.7 Pads, absorbent-cotton.

6. PROCEDURE

6.1 Preheat and adjust bath to 163° " 1°C (325° " 2°F).

6.2 Pour 500 mL of the sample into a clean sample tube, and place tube into the bath so that the bath level is at least 2.5 cm (1 in) above the sample level.

6.3 Connect air supply line to the aerator intake of the sample tube.

6.4 Allow approximately 30 min for the specimen to come to test temperature.

6.5 Drill a 3/16-inch hole in the center of each test panel (see 5.2 and 5.3).

6.6 Polish and clean the test panels (handling only with forceps) as follows:

a. Polish the faces and edges of the panels with steel wool until a uniform bright surface is produced.

CAUTION: When polishing the lead panel, avoid excessive pressure which may cause the steel wool to become embedded in the lead.

b. Remove all loose particles from the panels, using naphtha-soaked cotton pads.
c. Rinse with fresh naphtha, and allow to air dry.

6.7 Using forceps to handle the panel, record the weight of the lead panel to the nearest mg.

6.8 Clean the stirrer shaft and panel attaching hardware by rinsing with naphtha, using naphtha-soaked cotton pads or steel wool as required. Rinse in clean naphtha, and allow to air dry.

6.9 Using forceps to handle the panels, mount both panels on the shaft as shown in Figure 2.

6.10 Attach stirrer motor to the shaft, and lower the shaft into the sample tube so the center of the lead panel is centered in the tube approximately 7.5 cm (3 in) from the bottom.

6.11 Start the stirrer and immediately adjust the air flow 0.940 " 0.047 L/min (2 " 0.1 ft^3/h). Continue the operation for 60 " 1 min.

6.12 Remove the stirrer assembly, and detach the lead panel from the shaft. Swab the lead panel lightly with naphtha-soaked cotton pads to remove any loose surface deposits. Rinse with fresh naphtha and then with acetone, and allow the panel to air dry.

6.13 Reweigh until there is no change in the weight.

7. CALCULATIONS

7.1 Calculate the change in weight in mg/cm^2 (mg/in^2) of the exposed surface area (including the faces and edges, but excluding the areas covered by the washers).

8. REPORTING

8.1 Report the change in weight to the nearest 1.5 mg/cm^2 (mg/in^2).

9. PRECISION

9.1 Repeatability. Results by one operator using the same apparatus must not differ:

a. By more than 0.15 mg/cm^2 (1 mg/in^2) if the change in weight does not exceed 1.5 mg/cm^2 (10 mg/in^2);

or

b. By more than 10% of the average, if the change in weight exceeds 1.5 mg/cm^2 (10 mg/in^2).

9.2 Reproducibility. Results from different operators or different apparatus must not differ:

a. By more than (2 mg/in^2) if the change in weight does not exceed 1.5 mg/cm^2 (10 mg/in^2),

or

b. By more than 20% of the average, if the change in weight exceed 1.5 mg/cm^2 (10 mg/in^2).

Method prepared by:

Air Force - SFTL - 1986

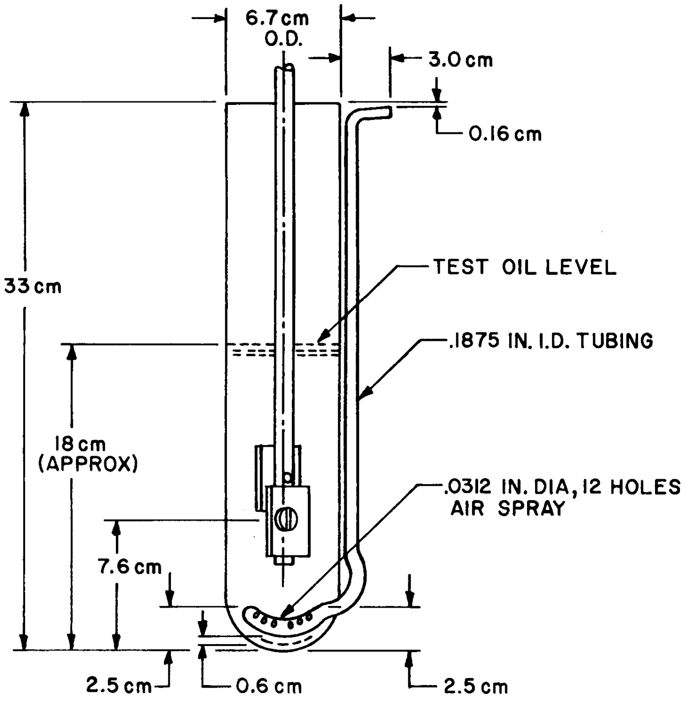
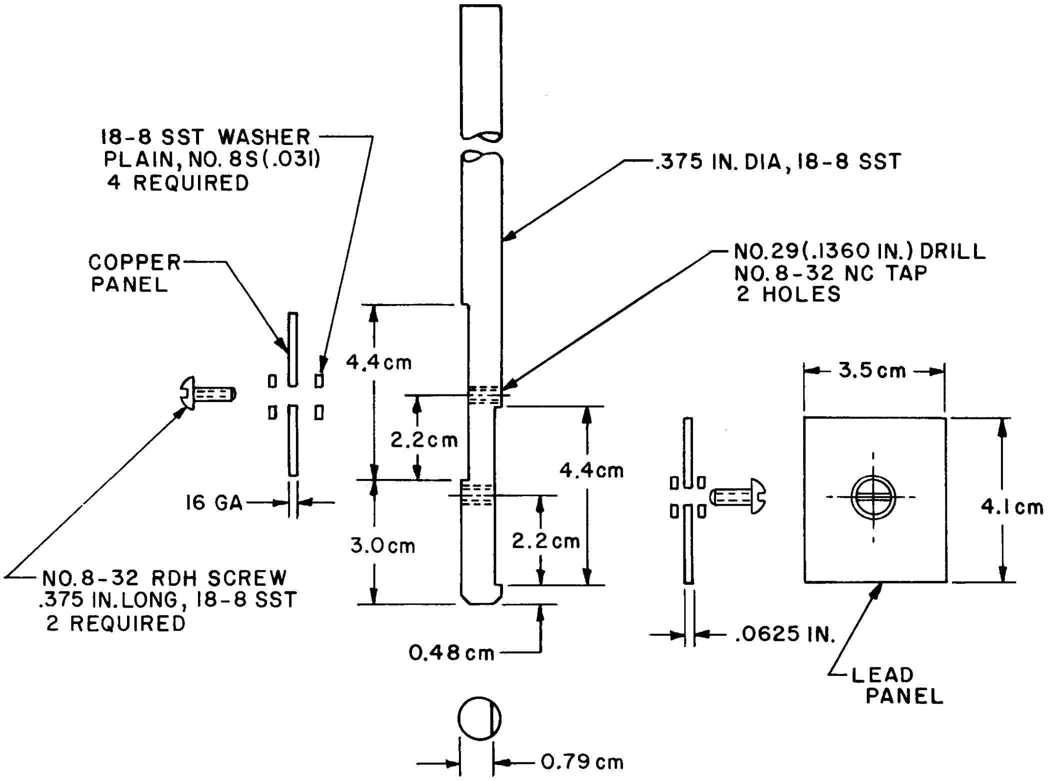


FIGURE 1. SOD sample tube.



NOTES:
1. LENGTH AND TOP CONFIGURATION OF SPINNER SHAFT TO SUIT SHAFT ROTATOR.
2. ALUMINUM MAY BE SUBSTITUTED FOR 18-8 STAINLESS STEEL.

FIGURE 2. Stirring shaft and test panels.

Method 5322.2
September 30, 1986

CORROSIVENESS OF OIL ON A BIMETALLIC COUPLE

1. SCOPE

1.1 This method is used for determining the corrosive effect (chemical or electrochemical) of an oil on a bimetallic couple.

2. SUMMARY

2.1 The method consists of fitting a brass clip to the oil-coated surface of a steel disk, storing the assembly in a humidity cabinet for 10 days, and visually inspecting the assembly for evidence of corrosion.

3. SAMPLE SIZE

3.1 Approximately 2 g of the oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Humidity cabinet, approximately 50% relative humidity.

4.2 Magnifier, 10X.

4.3 Desiccator, containing desiccant.

4.4 Stirring rod, glass.

5. MATERIAL

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Steel disks, (3) (ASTM A 322, composition G52986), heat treated to Rockwell C-62, 2.54 cm (1 in) diameter by 1.25 to 2.54 cm (1/2 to 1 in) thick ground on one side to 0.508 µm (20 µin) rms prior to hand-polishing (see 6.1.2). This treatment need not be repeated prior to each test unless the disks are badly corroded.

5.3 Clips, (3), brass (ASTM B 121/ B 121M, ASTM B 36/B36M, composition 2), spring temper, fabricated to dimensions shown in Figure 1.

5.4 Abrasive papers, silicon-carbide or aluminum oxide, 150, 240, 400, and 600 grit.

CAUTION: Do not use papers containing iron oxide.

5.5 Cloth, lint-free, clean, dry.

5.6 Acetone (ASTM D 329).

5.7 Etching solution, consisting of:

5.7.1 Distilled water, 450 mL.

5.7.2 Nitric acid (ACS), concentrated, 225 mL [2,0,0].

5.7.3 Sulfuric acid (ACS), concentrated, 300 mL [3,0,2].

5.7.4 Hydrochloric acid (ACS), concentrated, 8 mL [3,0,0].

5.8 Methyl alcohol (ACS), absolute [1,3,0].

5.9 Distilled water.

6. PROCEDURE

6.1 Prepare the three steel disks as follows:

6.1.1 Slush the disks in the methyl alcohol to remove traces of coolant used in grinding.

NOTE Caution. Methyl alcohol is both toxic and flammable. Use it only in a well ventilated area. Do not permit it to come in contact with the skin or breathe its fumes. Keep all flames away from this material.

6.1.2 Polish the surface-ground side using 150-, 240-, 400-, and 600-grit silicon carbide or aluminum oxide paper in succession. Perform each polishing operation at right angles to the previous one, and continue until all cross scratches from the previous operation are removed.

6.1.3 Clean the disks by wiping with the clean, dry, lint-free cloth, being careful not to touch the polished surface with bare hands.

6.1.4 Examine with the 10X magnifier for discoloration, etching, pitting, or other signs of corrosion. Discard disks with any sign of corrosion.

6.1.5 Store the disks in the desiccator until use. Immerse the brass clips (see 5.3) in the etching solution for 20 s, and immediately wash successively in cold running water, distilled water, and acetone. Allow to air dry, and store in the desiccator until ready for use. Be careful not to touch (with bare hands) the surface that will be in contact with the steel disks (see Figure 1).

CAUTION: Acetone is flammable. Use it only in a well ventilated area. Keep all flames away from this material.

6.3 Remove the disks from the desiccator (being careful not to touch the finished surface), and using the stirring rod, drip the test sample over the entire polished surface of each disk.

6.4 Remove the brass clips from the desiccator (being careful not to touch the area to be in contact with the polished surface of the disks), and place on the disks as indicated in Figure l so that the center of the clip is in full contact with the disk.

6.5 Place the assemblies in the humidity cabinet for 10 days [relative humidity, 50%; temperature, 27°C (79°F)].

6.6 Remove the assemblies from the cabinet after 10 days, and using a sharp pencil, mark an outline of the clips where they were in contact with the polished surface of the disks. Remove the clips, and wipe the disks clean with lint free cloth.

6.7 Using the 10X magnifier, examine the marked-off area of the disks for discoloration, etching, pitting, or other signs of corrosion.

6.8 Repeat the test (if the test results are questionable), using new disks and extending the exposure time in the humidity cabinet to 20 days. Examine the disks and report as before indicating that the test was repeated.

7. CALCULATION

7.1 This section is not applicable to the test procedure.

8. REPORTING

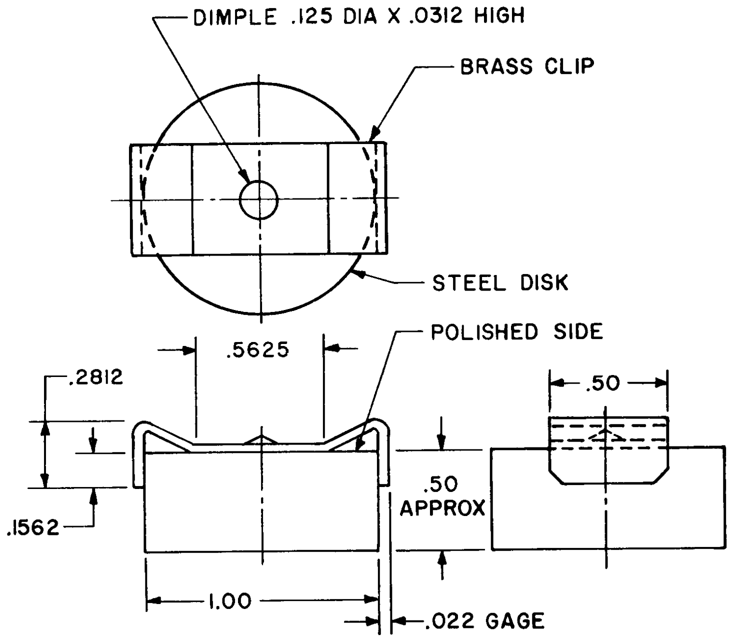
8.1 Report the oil as failing the test if the test areas of more than one disk show signs of corrosion, pitting, or other attack. Also report the oil as failing if two are free of corrosion, but the third shows more than three affected spots. Report the oil as passing if the condition of the disk is other than these.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986



NOTES:
1. DIMENSIONS IN INCHES.
2. TOLERANCE ± .0312.

FIGURE 1. Corrosivity test setup.

Method 5329.2
September 30, 1986

CORROSION-PROTECTION (HUMIDITY CABINET)

1. SCOPE

1.1 This method is used for determining the effectiveness of corrosion-inhibiting oils and compounds under conditions of high humidity.

2. SUMMARY

2.1 The test consists of dipping steel test panels in the test sample and then placing them in a humidity cabinet. After the specified exposure time the panels are removed and examined for the presence of rust.

3. SAMPLE SIZE

3.1 Approximately 500 mL of the oil to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Standards.

4.1.1 ASTM D 1748 Rust Protection By Metal Preservatives In the Humidity Cabinet.

4.2 Humidity cabinet conforming to Appendix I of ASTM D 1748 where the following standard conditions must be maintained.

a. Air temperature:
   Inside the cabinet: 49° ± 1°C (120° ± 2°F).
   Outside the cabinet: 24° ± 5°C (75° ± 10°F).
b. Rate of air to the cabinet: 868 ± 28 L (31 ± 1 ft^3) per hour at 25°C (77°F) and 76 cm of mercury.
c. Water in cabinet:
   Level - 20.3 ± 0.6 cm (8 ± 1/4 in)
   pH - 5.5. to 7.5
   Oil content - clear with no evidence of oil
   Chlorides - less than 20 ppm
   Sulfates & sulfites - less than 20 ppm.
d. Speed of rotating stage - 0.33 ± 0.03 rpm.
e. Cover - close fitting.
f. Cloth layers in cover - shall not be torn, contaminated, nor contain droplets of water.

g. Cover opening - to a height of 35.6 cm (14 in) at the front.
h. Open the cabinet twice each day, except Saturday and Sunday, once for 15 min and again for 5 min. (A period of approximately 5 h should elapse between openings.)
i. Check the rate of air to the cabinet, air temperatures, pH, and water level and regulate, if necessary, in the morning and afternoon of each day.
j. Check remaining standard conditions once each week.

4.3 Desiccator, containing an indicating desiccant.

4.4 Panel hook, stainless steel (for handling panels during processing).

4.5 Hooks, Monel or stainless steel (for supporting steel panels).

4.6 Sandblast cabinet (a suitable apparatus for sandblasting test panels).

4.7 Beakers, tall-form, 500 mL.

4.8 Controlled atmosphere (to provide a temperature of 25° ± 3°C [77° ± 5°F] and a maximum relative humidity of 50%).

4.9 Shaded fluorescent light. (A 15-watt balance illuminator type that will permit the panel to be viewed from all angles at a distance of 7.6 cm [3 in]).

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Naphtha, aliphatic (TT-N-95) [1,4,0].

5.3 Methyl alcohol (O-M-232) [1,3,0].

5.4 Silica sand, white, dry, sharp. (The size shall be such that it meets the following sieve requirements of ASTM E 11.)

a. 100% must pass through a No. 10 sieve.
b. Minimum of 90% must pass through a No. 20 sieve.
c. Maximum of 10% permitted to pass through a No. 50 sieve.

5.5 Test panels, FS 1009 steel, fabricated from open-hearth, "killed" type low carbon, No. 4 soft-temper, cold-rolled sheet or strip conforming to QQ-S-698. Dimensions of the panels shall conform to Appendix I of ASTM D 1748. Cold drawn bar stock is not satisfactory.

5.6 Surgical gauze.

6. PROCEDURE

6.1 Clean the steel panels as follows:

a. Round the edges and ream out the suspension holes in accordance with Appendix I of ASTM D 1748.
b. Wipe the surfaces with solvent soaked rags and scrub with surgical swabs in a beaker of hot naphtha.

NOTE 1: Caution. Naphtha is flammable. Use only in a well ventilated area. Keep all flames away from the naphtha.

c. Rinse in a beaker of hot methyl alcohol.

NOTE 2: Caution. Methyl alcohol is both toxic and flammable. Use only in a well ventilated area. Do not allow it to come in contact with the skin or breathe its fumes. Keep all flames away from methanol.

d. Cool the panels in a desiccator until further processing.
e. Test panels must be handled with a panel hook at all times, avoiding contact with any type of contaminated surface.
f. Maintain the solvents at a temperature high enough to keep the temperature of the panels above the dew point during the cleaning operation.

6.2 Sandblast the panels as follows:

a. Sandblast the edges and lightly sandblast the back of the panels with silica sand.
b. Sandblast the unnumbered side, or test surface, of the panels to a fresh, uniformly abraded surface.
c. Immediately after sandblasting, place the panels in a beaker of anhydrous methanol.
d. Heat the methyl alcohol so that the solvent will evaporate from the panels immediately upon withdrawal from the solvent.
e. Remove remaining residue by holding the panels in a rack at 20° from the vertical and spraying downward with naphtha.
f. Spray the test surface, then the back of the panel and the test surface again.
g. Rinse the panels in hot naphtha and hot methyl alcohol and store in a desiccator until cool.
h. Panels are to be used the same day as prepared.

6.3 Three test panels are processed as follows:

a. Dip the test panels in the test sample at a temperature of 25° ± 3°C (77° ± 5°F) and agitate gently for one minute.
b. Suspend the panels by means of Monel or stainless steel hooks and drain for 2 h at 25° ± 3°C (77° ± 5°F) and a maximum of 50 percent relative humidity.
c. Place the panels in the humidity cabinet described in Appendix I of ASTM D 1748 for the time required by the applicable specification.
d. Dummy panels, fabricated from monel or stainless steel, are used to fill all spaces not occupied by test panels. They are also placed under the three arm supports of the rotating stage.

6.4 At the end of the exposure period, remove the panels and rinse in methyl alcohol. Follow with a rinse in naphtha and methyl alcohol and examine the significant area of the test surface as defined in Appendix I of ASTM D 1748, using a 15-watt, shaded fluorescent light.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Record the extent of corrosion as follows:

a. The number of rust dots, 0.1 cm in diameter or less.
b. The number of rust spots, 0.1 to 0.2 cm in diameter.
c. The number of rust areas, greater than 0.2 cm in diameter.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - AR - 1986

Method 5331.1
September 30, 1986

CORROSION PROTECTION OF STEEL AGAINST SULFUROUS ACID-SALT SPRAY BY SOLID FILM LUBRICANTS

1. SCOPE

1.1 This method is used to evaluate the corrosion preventive properties of dry, solid-film lubricant under the combined conditions of salt, sulfurous acid, and humidity environment.

2. SUMMARY

2.1 A steel disk having solid film lubricant deposited on one surface is subjected to the cycling effect of salt spray and drying. After each cycle of salt spray and drying, the disks are examined for evidence of corrosion.

3. SAMPLE SIZE

3.1 180 mL of solid-film lubricant of a sprayable consistency.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Air Supply - filtered, 69 gage kPa to 103 gage kPa (10 to 15 psig).

4.2 Air flow regulator - capable of regulating air flow at 472 mL/s (1 ft^3/min).

4.3 Spray Nozzle (Figure 1) - corrosion resistant with a 13 cm (5 in) diameter acrylic baffle which prevents direct impingement of the spray, but allows a fine mist full contact with the disks.

4.4 Turntable Assembly which consists of:

a. Turntable - corrosion resistant, 26.7 cm diameter with holes for mounting test holders and specimens.
b. Driving Mechanism - capable of mechanically rotating turntable at 1/3 rpm by an electric motor (slow speed), encased within a protective acrylic box and mounted above jar on an arrangement of flexaframe support rods.

4.5 Specimen Holders - holder cap (Figure 2) and ice jacket (aluminum) that is so placed as to be in direct contact with back of test specimen.

4.6 Jar - 30.5 cm I.D. and 30.5 cm high.

4.7 Support Rods - flexaframe or equivalent.

4.8 Flexible Tygon Tubing - 0.96 cm I.D.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Steel Specimen - disk with 5.4 cm diameter and thickness of 0.16 cm, made of carbon steel conforming to FS 1010 with a 0.08 cm hole for handling disk with wire.

5.3 Synthetic Sea Water-Sulfurous Acid Test Solution - synthetic sea water (Formula ASTM D 1141 - 41.95 g/L, 900 mL with 2 mL sulfurous acid [not less than 6.0% assay as $S0_2$]).

5.4 Aluminum Oxide Cloth - 240 grit.

5.5 1,1,1-Trichloroethane (MIL-T-81533) [2,1,0].

5.6 Distilled Water (ASTM D 1193 - Type III).

5.7 Sulfurous acid, ACS [u,u,u].

6. PROCEDURES

6.1 Prepare test specimens as follows:

a. Degrease test specimens in 1,1,1-trichloroethane (MIL-T-81533).
b. Hand polish surface to be tested with 240 grit aluminum oxide cloth to a surface finish of (0.25 to 0.50 µm).
c. Repeat step (a).
d. Allow specimens to dry and store in a dust-free container.
e. Apply solid-film lubricant to surface of steel specimen by spraying to produce a film thickness between 0.0005-0.0013 cm (0.0002-0.0005 in) and cure film in accordance with specification requirements.
f. Handle the specimen at all times with a hook.

6.2 Prepare the apparatus (Figure 3) as follows:

a. Fill bottom 5 cm of jar with the synthetic sea water-sulfurous acid solution and then lower the turntable assembly into jar.
b. Place disk in specimen holder (Figure 2) with ice jacket on top.
c. Mount the complete specimen assembly in one of the holes of the turntable and start the turntable.
d. Turn on the air and regulate rate of flow to 472 mL/s (1 ft^3/min).
e. Two min after the spray has started, place ice in ice jackets in order to cause a fine condensate to form on the surfaces of disks.

6.3 Perform test as follows:

a. Continue spraying for 2 h with a rate of flow of approximately 0.4 mL/s.
b. Discontinue the spray at the end of two hours; remove the ice jackets; and raise the turntable above the jar to allow specimens to dry for two hours at room temperature. The two-hour spray and two-hour drying period shall constitute one cycle.
c. Rinse the test specimens after each full cycle, while still retained in the holder, with distilled water (ASTM D 1193 - Type III) and dry with a stream of air.
d. After each cycle examine test specimen for evidence of visible corrosion, pitting or staining.
e. Repeat the test cycle until failure occurs. Failure is defined as the first visible sign of corrosion, pitting or staining.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the number of test cycles completed prior to failure. Report the amount and type of corrosion present.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Navy - NADC - 1986

Method 5414.4
September 30, 1986

RESISTANCE OF GREASE TO FUEL

1. SCOPE

1.1 This method is used for determining the resistance of grease to the solvent action of fuel.

2. SUMMARY

2.1 The test consists of determining the solubility of the grease in a standard test fluid, and observing for physical changes caused by an 8-hour immersion in the test fluid.

3. SAMPLE SIZE

3.1 Approximately 4 g of grease to be tested (duplicate 2 g samples).

4. REFERENCE, STANDARDS, AND APPARATUS

4.1 Centrifuge, approximately 650 rpm, swing diameter (tip to tip of whirling tubes) approximately 40 cm, or equivalent.

NOTE: An equivalent centrifuge must be capable of having its speed adjusted according to the following formula:

$$rpm = \sqrt{\frac{40}{d}} * 650$$

where:
d = diameter (tip to tip) of whirling tubes centimeters

4.2 Shaker, mechanical, stroke approximately 3.8 cm (1-1/2 in), speed approximately 1350 rpm.

4.3 Centrifuge tube, 200 mL, with stopper.

4.4 Evaporation dish, 100 mL.

4.5 Pipet, 50 mL.

4.6 Test tube, approximately 1.5-cm (1/2-in) O.D. by 7.5 cm (3 in) long, with stopper.

4.7 Steam bath (to accommodate evaporating dish).

4.8 Oven, approximately 100°C (212°F).

4.9 Balance, sensitivity 0.2 mg.

4.10 Spoon type spatula.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Standard Test fluid (ASTM D 471, Type III).

5.3 Aluminum strip (ASTM B 209), 5.08 □ 1.3 □ 0.04 cm.

6. PROCEDURE

6.1 Determine the solubility of the sample in the standard test fluid as follows:

a. Clean, dry and weigh a centrifuge tube to within . 0.2 mg accuracy. With a spoon spatula spread approximately two g of grease in a thin film inside the tube and weigh to within . 0.2 mg; add 100 mL of the test fluid and stopper the tube.
b. Using the mechanical shaker, shake tube until sample appears to be thoroughly dispersed (approximately 1/2 h).
c. Centrifuge the tube until fluid and sample are completely separated, and the fluid portion is clear and transparent.
d. Weigh evaporating dish to within 0.2 mg.
e. Pipet 50 mL of the clear fluid from the centrifuge tube into evaporating dish. Place dish on steam bath until liquid is evaporated. Then dry dish in oven for 1/2 h at approximately 100°C.
f. After drying, remove dish from oven and reweigh to within 0.2 mg. Determine the weight of residue, and calculate the percent of sample soluble in test fluid (see 7.1).

6.2 Determine the deterioration of the sample due to the test fluid as follows:

a. Fill test tube to a depth of approximately 2.5 cm with test fluid. Spread the grease evenly on both sides of the aluminum strip, and place the strip in the test tube so that it is half immersed in the test fluid. Stopper test tube, and allow it to stand for 8 h at room temperature (approximately 25°C).
b. After 8 h, remove strip from test tube (being careful not to disturb sample), and dip once into fresh test fluid. Examine sample while wet for swelling, loss of adhesion, blistering, cracking, or deterioration, and report observations.
c. Allow strip to dry for 24 h at room temperature (approximately 25°C), then re-examine the sample for the signs of deterioration, and report observations.

7. CALCULATIONS

7.1 Calculate the percent of sample soluble in test fluid as follows:

$$Percent\ solubility = \frac{200\ \ A}{B}$$

where:

A = weight of sample, mg (see 6.1 f).
B = weight of original sample, mg.

8. REPORTING

8.1 Report percent solubility as determined under 6.1 f.

8.2 Report condition of grease on strip as determined under 6.2. b and c.

9 PRECISION

9.1 Precision data have not be developed for this method.

Method prepared by:

Navy - NADC - 1986

Method 5415.1
September 30, 1944

RESISTANCE OF GREASE TO WATER AND A 1:1 WATER-ETHANOL SOLUTION

1. SCOPE

1.1 This method is used for determining the resistance of a grease to the solvent action of water.

2. SUMMARY

2.1 The test consists of storing two portions of grease, one in distilled water and the other in an ethyl alcohol-water solution, for one week, and examining the grease for disintegration.

3. SAMPLE SIZE

3.1 Approximately 4 g of grease to be tested.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Containers (2), glass, 250 mL, with stoppers.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Alcohol, Ethyl, Specially Denatured, Aircraft (A-A-51693).

5.3 Distilled water.

6. PROCEDURE

6.1 Place 200 mL of distilled water into one container, and 200 mL of an alcohol-distilled water solution (1:1 by volume) into the other.

6.2 Place a single globule of grease (approx. 2 g) into each of the two containers, stopper the containers tightly, and allow both to stand for one week.

6.3 At the end of one week, shake each container once or twice, and then visually examine each portion of grease for disintegration.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report the degree of disintegration of the grease in each container.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Navy - NADC - 1986

Method 6050.1
September 30, 1986

NITRATE-TYPE IGNITION IMPROVERS IN DIESEL FUEL

1. SCOPE

1.1 This method of test covers the determination of organic nitrate ester-type cetane improver additives used in diesel fuel. It is intended as a screening test for those diesel fuel inspection test procedures that are affected by the presence of cetane improvers; namely, ASTM D 524 Standard Method of Test for Ramsbottom Carbon Residue of Petroleum Products and D 976 Standard Methods for Calculated Cetane Index of Distillate Fuels.

2. SUMMARY

2.1 A diesel fuel sample is saponified in a potassium hydroxide-1-butanol mixture and then filtered through a glass fiber filter disk. The material remaining on the disc is treated with diphenylamine reagent after drying. The presence of a nitrate ester cetane improver is revealed by the formation of a blue ring or blue-black spot due to oxidation of diplenylamine to intense blue quinoidal compounds by the nitrate salt. No color change confirms the absence of a cetane improver.

3. SAMPLE SIZE

3.1 A 10-mL sample of diesel fuel is required.

4. REFERENCES, STANDARDS, AND APPARATUS.

4.1 Reaction bottle - screw-cap bottle, 29.6 mL (l fl oz) capacity, wide mouth, flint glass, with screw-cap lined with tin or tetrafluoroethylene (TFE) resin.

4.2 Glass fiber filter paper - 3.7 cm diameter, Grade 934 AH (H. Reeve Angel, Inc., Clifton, NJ, or equivalent).

4.3 Pipette - 10 mL capacity, fitted with pipetting bulb. Several types and makes of pipetting bulbs and assemblies are available. One of the following is suggested: Fisher Cat. No. 13-681, Safety Pipet Filler, or equal; Fisher Cat. No. 13-681-50, Pipet Filler, or equal.

4.4 Graduated cylinders - 10 and 25 mL capacity.

4.5 Suction flask with a suitable holder to accommodate a 60-mL glass-fritted crucible.

4.6 Crucible - 60 mL capacity, glass-fritted, medium porosity.

4.7 Oven suitable for drying filter discs at 110°C.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Saponification Mixture (lN) - Prepared by mixing 6.5 g potassium hydroxide (ACS grade) [3,0,1] with 100 mL absolute *n*-butyl alcohol (ACS grade) [1,3,0] and heating to dissolve the KOH. After the solution cools, the mixture is filtered through the glass fiber filter paper.

5.3 Diphenylamine (1%) Solution - Prepared by dissolving 0.250 g diphenylamine (ACS indicator grade) [3,1,0] in 25 mL sulfuric acid (sp gr 1.834) [3,0,2].

5.4 Toluene (ACS Reagent grade) [2,3,0].

<u>NOTE 1:</u> Toluene is flammable and toxic. Avoid breathing vapors or contact with skin.

6. PROCEDURE

6.1 Pipette 10 mL of sample into the reaction bottle and add 5 mL of toluene followed by 10 mL of the saponification mixture.

<u>NOTE 2:</u> Oral pipetting techniques should not be used because of the toxicity of the substances involved. A pipetting bulb or assembly similar to one of those described in 4.3 should be used.

6.2 Affix cap to the reaction bottle tightly and, after mixing the contents, place it in an oven maintained at 110°C for 4 h.

6.3 Remove the reaction bottle from the oven and allow it to cool to 25° . 3°C.

6.4 Filter the contents of the reaction bottle through the 60-mL glass-fritted crucible fitted with the glass fiber filter disc.

6.5 Wash the reaction bottle with 25 mL of toluene and transfer it to the glass-fritted crucible.

6.6 Carefully remove the glass fiber filter disc and dry it in oven at 110°C for 15 min.

6.7 Remove the filter disc and cool it to 25° . 3°C.

6.8 Add 3 drops of diphenylamine solution to the center of the disc and observe whether a blue or blue-black color forms.

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 The presence of organic nitrate ester-type cetane improvers will be reported if the formation of a blue color occurs. Reference samples of diesel fuels containing 0.5% by volume of any one of the approved cetane improvers (amyl nitrate, cyclohexyl nitrate, hexyl nitrate and isopropyl nitrate) give an intense blue to blue-black color throughout the reagent spot whereas those samples containing only 0.1% by volume produce a blue ring at the outer boundary of the reagent. If a positive reaction occurs (i.e., a blue or blue-black coloration), the carbon residue determination (ASTM D524) must be performed on a neat or base fuel blend.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared by:

Army - ME - 1986

Method 6052.1
September 30, 1986

HIGH-TEMPERATURE--HIGH-PRESSURE SPRAY IGNITION

1. SCOPE

1.1 This method is used for determining the relative flammability of liquids.

2. SUMMARY

2.1 The method consists of forcing the test fluid sample through a 0.0368-cm (0.0145-in) orifice at 6.98 MPa gage (1000 psig), attempting to ignite the spray with a torch and noting the characteristics of the resulting flame.

3. SAMPLE SIZE

3.1 Sufficient liquid to be tested to fill cylinder of spray test setup.

4. REFERENCES, STANDARDS AND APPARATUS

4.1 Apparatus.

4.1.1 Spray test setup. A setup similar to Figure 1 shall be assembled so as to supply a constant pressure of 6984 " 69 kPa gage (1000 " 10 psig) to the sample.

4.1.2 Torch. An oxyacetylene torch equipped with a No. 3 tip or equal.

4.1.3 Nozzle. A nozzle 0.163-cm (0.064-in) thick by 0.965-cm (0.38-in) minimum diameter with a centered orifice approximately 0.0368-cm (0.0145-in) diameter having sharp, square edges (see Figure 1).

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The chemical materials listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Nitrogen. A cylinder of nitrogen fitted with a regulator capable of supplying 6890 kPa gage (1000 " 10 psig) of pressure to the system.

6. PROCEDURE

6.1 Fill the fluid cylinder of the spray test setup with test fluid at 15.6° to 37.8°C (60° to 100°F) and adjust nitrogen pressure to produce a liquid pressure of 6894 " kPa gage (1000 " 10 psig).

6.2 Adjust the torch to deliver a neutral flame.

6.3 Open the quick-opening valve at the orifice and attempt to ignite the spray with the torch at the nozzle.

6.4 If the spray does not ignite gradually move the torch away from the nozzle until ignition takes place (45.7 cm max.)

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report whether the spray ignites. If the spray does ignite, also report:

a. The distance from torch to nozzle at ignition.
b. Whether the spray flashes readily or with difficulty.
c. Whether the flame produced is self extinguishing or sustained.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method Prepared By:

Army - ME - 1986

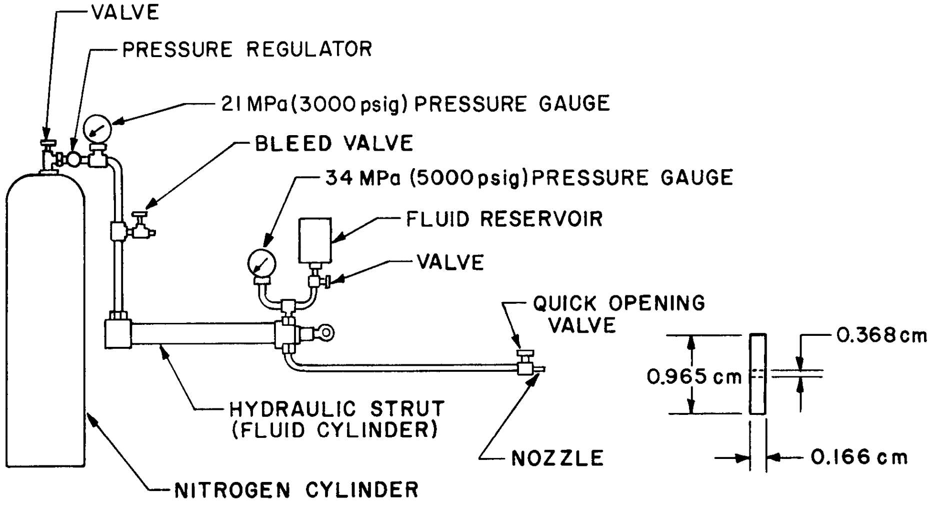


FIGURE I. Spray test setup.

Method 6053.1
September 30, 1944

MANIFOLD IGNITION TEST

1. SCOPE

1.1 This method is used for determining the relative flammability of a liquid in contact with a hot surface.

2. SUMMARY

2.1 The test method consists of dropping the flammable fluid on a hot surface and observing the presence of a flame.

3. SAMPLE SIZE

3.1 15 mL per test run.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Simulated manifold test set up (Figure 1), fabricated from 7.6 cm (3 in) O.D. corrosion-resistant steel tubing of 0.114 (0.045 in) wall thickness. The exterior of the tube shall be sandblasted. A 1/8-in diameter 18-8 corrosion-resistant rod 25 cm (10 in) long shall be tacked to the stack on the exterior surface. Opposite the steel rod, tack, weld or suitably attach a thermocouple, and insulate the leads. Care should be taken in this attachment to provide a minimum additional radiating surface.

4.2 Heating element, electrical, "globar" type or equivalent, (Carborundum Company, AT, 31 □ 12 □ 1, 0.633 Σ unit, or equal), with suitable means for controlling the temperature of the tube to 704°C (1300°F).

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Steel wool.

6. PROCEDURE

6.1 Clean the outside surface of the tube of the test setup (Figure 1), using steel wool.

6.2 Mount the heating element centrally in the tube, and adjust the voltage so that the temperature of the tube is maintained at 704°C (1300°F).

6.3 When the tube has reached the correct temperature, drop 10 mL portions of the test liquid at a rate of 10 mL in 40 to 60 s from various heights onto various points on the tube, and observe the ignition characteristics of the liquid. (Clean the tube with steel wool before each 10 mL application of the liquid.)

7. CALCULATIONS

7.1 This section is not applicable to the test procedure.

8. REPORTING

8.1 Report results as follows:

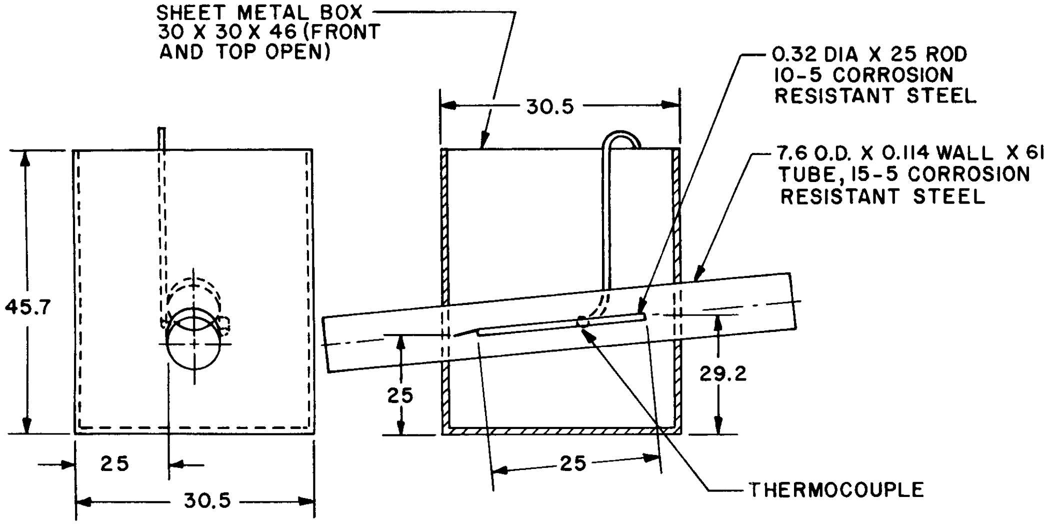
a. Flashes or burns on the tube, but does not after dripping from the tube.
b. Does not flash or burn on tube, but does after dripping from the tube.
c. Does not flash or burn on the tube or after dripping from the tube.

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Army - ME - 1986



NOTE: DIMENSIONS IN CENTIMETERS.

FIGURE 1. Simulated manifold test setup.

Method 6508.2
September 30, 1944

LOAD CARRYING CAPACITY OF LUBRICATING OILS
(Ryder Gear Machine)

1. SCOPE

1.1 This method describes a procedure for determining the load carrying ability of lubricating oils with reference to gears.

2. SUMMARY

2.1 The test consists of incremental increasing the pressure between gears to measure the resultant scuffing. The ability of the test oil to impair scuffing is measured by the maximum pressure when operating at 10,000 rpm.

3. SAMPLE SIZE

3.1 Approximately 2 L of test oil.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Test unit. The apparatus used in this method is the Ryder gear-Erdco universal tester (Figure l), and test gears available from Erdco Engineering Corp., Addison, Ill. This tester comprises a special test machine known as the Ryder gear machine, a drive system, a support and load oil system, a test oil system, and the necessary instruments and controls. Figure 2 illustrates the control console.

4.1.1 Ryder gear machine. The Ryder gear machine operates on the so called "four square" principle. As shown in Figure 3, two parallel shafts (P and Q) are connected by two slave gears (R and S) and two test gears (T and U) to form a "square", so that the power required to operate the machine is only that required to overcome the friction losses in the gear and bearings. The slave gears are helical gears made integral to the shafts, (see Figure 4). The replaceable test gears are spur gears, shown installed on the shaft in Figure 5. Load on the test gears is obtained through the application of a controlled oil pressure in two load chambers (Figure 3, Y and Z) which causes an axial movement of one shaft relative to the other. Because of the integral helical slave gears, a torsional load is thus applied on the shafts thereby loading the test gears. The relation between the tooth load on the test gears and the load oil pressure is obtained by arithmetic calculation, calibration, or both. Arithmetic calculation is used herein.

4.1.2 Test gears. The test gears are special spur gears, made of AMS 6260 steel, case hardened and ground, having 28 teeth, 8.88-cm (3.5-in) pitch diameter, 8 diametral pitch, 22.5° pressure angle, and zero tip relief. Each set of test gears comprises gears of two different widths. The narrow test gear is used for rating purposes. The mating gear is wider in order to maintain a constant contact width between the two gears at different tooth loads. Both sides of the test gear are used for testing. The "A" side of the gear refers to the side obtained by installing the tests gears with the serial numbers facing the end cover as shown in Figure 5. The "B" side is obtained by reversing the gear (serial numbers facing away from the end cover). Test gears can be used once only on each side and then discarded.

4.1.3 Support and load-oil system. The support and load-oil system, shown in Figure 6, serves two purposes: to provide lubrication to all parts except the test gears and to apply load to the test gears. The system is charged with approximately 30 L (8 gal) of lubricating oil, SAE J1966. It is recommended that the oil be changed every 200 h of running time under normal circumstances. A support oil pressure pump and a relief valve furnish oil to the distributing manifold at a specified support oil pressure of 758 ± 69 gage kPa (110 ± 10 psig). The support oil temperature, also measured at the distributing manifold, is maintained at a specified value of 73.9° ± 2.5°C (165° ± 5°F) by electric heaters located in the sump and by a water cooler located after the pump, through the action of thermoswitches. Pressure lines from the distributing manifold lubricate the bearings and helical gears of the machine as well as the adapter housing and the stepup gear box. The pressure to the latter is reduced to 172 to 207 kPa gage (25 to 30 psig) by a throttle valve. Three scavenge pumps scavenge oil from the machine, the adapter housing, and the step-up gear box, back to the support oil sump. The action of the load oil circuit can be seen in Figures 3 and 6. Oil from the distributing manifold (Figure 6) enters the Ryder gear machine at points W and X (Figures 3 and 6). After lubricating the bearings and slave gears, the oil enters the load chambers Y and Z (Figure 3). For a given pressure in the distributing manifold, the pressure in the load chambers is dependent on the flow restriction offered by the circuit downstream of the load chambers. Figure 6 shows that the oil, upon leaving the load chambers at J and J, must pass through a control in the load oil circuit before returning to the support oil sump. Two pressure gages (a low range and a high range) located near the outlet points J and J, read the so called "load oil pressure", i.e. the pressure in the load chambers. The load oil circuit contains three valves. Of these the load bypass valve is an open or shut valve which is closed during the test run. The load oil pressure is controlled by regulating the load oil adjusting valve. The bleeder valve is normally slightly open, its function being to decrease the sensitivity of the load adjusting valve, thus facilitating the control of load oil pressure. The load bypass valve is opened when it is desired to quickly release the load on pressure, such as at the end of a test run.

4.1.4 Test oil system. A schematic diagram of the test oil system is shown in Figure 7. The capacity is 500 mL. Test oil is supplied to the test gears by a pressure pump through a jet located on the unmeshing side of the gears. Oil temperature to test gears is maintained at 73.9° ± 2.5°C(165° ± 5°F) by a line heater actuated by a thermo switch and controlled by a thermocouple close to the oil jet. The bypass circuit is arranged to permit the use of the graduated sump for the determination of the test oil flow rate. The bypass circuit also facilitates inspection of the narrow test gear without stopping test oil circulation.

4.1.5 Drive system. As shown in Figure 1, the Ryder Gear machine (A) is driven by a 50 hp induction motor (B) through a dynamic coupling (C), a step-up gear box (D), and an adapter (E). The test gear speed is controlled by adjusting the field excitation of the dynamic coupling. By this means, accurate speed control from standstill to well over the specified test speed of l0,000 rpm can be attained.

4.2 Instrumentation and controls. All of the instruments and controls that require constant attention during test are located on the control console (Figure 2). Items that do not require frequent attention are located on the test stand. The functions of most of the instruments and controls are apparent. Therefore, only selected items will be explained further.

4.2.1 Speed measurement and control. An electronic counter (Figure 2, item 4), located on the control panel, measures the speed. This instrument is actuated by pulses supplied by a variable reluctance pickup (the variable reluctance being provided by a 60-tooth gear operated from the driven shaft of the step-up gear box). The rpm adjustment control (Figure 2, item 20), located on the con-sole, regulates the field excitation of the dynamic coupling and thus the gear speed.

4.2.2 Control of test run duration. An electric timer (Figure 2, item 10) located on the console, is used to control the duration of each test run. As soon as the desired test load has been reached the timer is started at the determined test duration setting. After the run is over the timer automatically turns off the drive motor and opens the load-bypass valve. The timer switch must be turned to the "stop" position prior to the next run.

4.2.3 Support-oil controls. The support-oil pump buttons (Figure 2, item 11) on the console are connected to the support-oil pressure pump as well as the three scavenge pumps. Pressing the start or stop button controls all four pumps at the same time. The support-oil heater buttons (Figure 2, item 12) control both the electric heaters and the water cooler through the action of thermoswitches (Figure 6). The support-oil chamber vacuum is measured by a water manometer located on the test stand. A valve connecting the chamber to a vent (Figure 6) should be adjusted to give a chamber vacuum of 2.5 to 10.2 cm (1 to 4 in) of water under normal test conditions. If this value cannot be maintained, the shaft and shaft seals should be inspected for leakage.

4.2.4 Control of load oil pressure. The control of load oil pressure has been explained in 4.1.3. Of the three valves in the load oil circuit (Figure 6) only the bleeder valve is not remote controlled. However, this valve need only be set to give the desired sensitivity for load control during installation of the tester. As a rule, adjustments are seldom necessary thereafter. The load adjusting valve is air actuated and is controlled by the load adjust control (Figure 2, item 9) located on the console. The load bypass valve is solenoid actuated and is controlled by the load bypass switch (Figure 2, item 13) on the console. During the test run the load bypass switch is in the "close" position and the load bypass valve is closed. At the end of a run, the solenoid circuit will automatically be opened by the electric timer. This opens the load-bypass valve, but the load bypass switch on the console remains in the "close" position. For this reason, the switch should be turned to the "open" position prior to the next run.

4.2.5 Emergency stop. The emergency stop button (Figure 2, item 18) on the console stops the drive motor and turns off all the controls except those for instruments. The timer switch and the load bypass switch remain in the positions to which they were originally set.

4.3 Scuff rating equipment. A microscope and a light source for visual inspection of the narrow test gear are provided as standard equipment with the Ryder gear-Erdco universal tester. A camera with accessories for photographing the gear teeth is available as optional equipment. To inspect or photograph the narrow test gear the machine is stopped, but the test oil is kept circulating through the bypass circuit. The inspection or photographing can then be accomplished through special inspection holes provided in the gear case without taking off the end cover. These holes are closed with corks during the test run.

4.4 Inspection equipment. The microscope provided with the tester is an 18X stereoscopic microscope. A high intensity light source is provided for use with the microscope. Figure 8 shows the inspection equipment in position for use. The left eyepiece of the microscope contains a net reticule which divides the gear tooth area lengthwise into five 20%-sections as shown in Figure 10. These 20% grids provide a convenient means for the visual estimation of the percentage of gear tooth area that has been scuffed. Scuffing of individual gear teeth is estimated to the nearest 5%.

4.5 Photographic equipment. This is optional equipment consisting of a Leica camera with a copying attachment, a viewer, and an electronic flash unit synchronized with the camera shutter. Figure 9 shows this equipment installed for use.

4.6 Gear tooth indexing rachet. An indexing rachet (Figures 1 and 8) is used to rotate the test gears so that the teeth of the narrow gear can be brought into position one by one for inspection or photography.

4.7 Additional instruments.

a. Stop watch for measurement of test oil flow rate.
b. Micrometer 0. to 2.5 cm (0 to 1 in) for measurement of the gross tooth width of the narrow test gear.
c. Set of conventional mechanical tools as necessary for assembly, disassembly, maintenance and repair.

5. MATERIALS

5.1 CAUTION: SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Lubricating oil, SAE J1966.

5.3 Flushing solvents.

5.3.1 Dry cleaning (A-A-59601D).

5.3.2 Petroleum ether (O-E-751) [1,4,0].

5.4 Test gear retaining nut lock washer, Fafnir WO-6 (Erdco Engineering Corporation, Addison, ILL, or any commercial supplier of Fafnir parts).

5.5 O-rings. An O-ring is used between the main case and the end cover of the Ryder gear machine. National No. 622710 O-rings 0.238 □ 1.27 □ 1.75 cm (3/32 □ 1/2 □ 11/16 in) are recommended. It is recommended that a new O-ring be used each time the end cover is installed. These rings can be obtained from Erdco Engineering Corp., Addison, IL, or any commercial supplier of National O-rings.

5.6 Shaft seals. The shaft seals recommended for the Ryder Gear machine are Garlock seals (obtained from Erdco Engineering Corp., Addison, IL, or the Garlock Packing Co., Palmyra, NY) with the following part numbers and dimensions:

a. Klozure 63 □ 359 with 9478 element and light-weight spring 2.86 □ 5.08 □ 0.952 cm (l-1/8 □ 2 □ 3/8 in).
b. Klozure 63 □ 1200 with 9478 element and light-weight spring 5.08 □ 7.30 □ 1.27 cm (2 □ 2-7/8 □ 1/2 in).

6. PROCEDURE

6.1 Operating condition.

6.1.1 Throughout the entire test, the variables listed in Table I should be controlled within the limits specified. With the tester operating within the specified limits, the test gears are loaded first to 34 kPa gage (5 psig) load oil pressure, and then at successive increments of 34 kPa (5 psi). The duration of each loading period is 10 min . 5 s. At the end of each loading period, the tester is stopped and each tooth of the narrow test gear is examined to determine the percent of tooth area scuffed.

TABLE I. Operating conditions.

Condition	Value	
Test gear speed, rpm	10,000 ± 10	
Test oil inlet temperature	73.9° ± 2.5°C	165° ± 5°F
Test oil flow rate	270 ± 5 mL/min	
Support oil inlet temperature	73.9° ± 2.5°C	165° ± 5°F
Support oil pressure	758 ± 69 kPa gage	110 ± 10 psig
Step-up gear box pressure	172 to 207 kPa gage	25 to 30 psig
Support oil chamber vacuum	2.5 to 10.2 cm of water	1 to 4 in of water

6.2 Preparation for test.

1. Record pertinent information such as test number, test oil code, operator's name, etc., on suitable data sheets (Figure 11).
2. Turn on main electric switch to test cell.
3. Turn on main water valve to test cell.
4. Push instruments button "ON".
5. Push support oil heaters button "ON".
6. Make sure that the test oil system has been thoroughly flushed with Stoddard solvent and petroleum ether after the last test. See 6.3.2 steps (17) through (25) for the flushing procedure. Use the flushing solvent once and discard.
7. Remove gear case end cover.
8. Inspect shaft seals. Replace with new seals if necessary.
9. Inspect shafts for any nicks or other damage in the area of the seals. Damage must be repaired before machine is used for testing.
10. Clean a set of new test gears with dry cleaning solvent and discard solvent. Measure gross tooth width of the narrow test gear, and record the measurement.
11. Install test gears with the serial number facing outward, matching timing marks on shafts and gears. (See Figure 5.) Check for proper seating of the retaining nut, and lock the lock washer.
12. Install new O-ring in position.
13. Reinstall gear case end cover.
14. Start test oil pressure pump.
15. Flush test oil system with test oil, as follows:

    a. Turn 3-way valve L to "DRAIN".
    b. Turn 3-way valve M to running position.
    c. Turn 3-way valve N to running position.
    d. Fill test oil sump with about 750 mL of test oil. Watch drop of level in sump. Allow 400 mL to drain through L.
    e. Drain the next 100 mL by opening drain K also. Then close drain K.
    f. Turn valve L to running position, so as to permit flushing of that section of the test oil system between valve L and sump.

g. Drain the remaining 250 mL by turning valve M to bypass the Ryder gear machine, and turning valve N to "DRAIN".

NOTE 1: The test oil for flushing is to be discarded after use. If the supply test oil is limited, a reduced quantity may be used for flushing.

16. Turn valves L, M, and N to running position.
17. Fill test oil sump with a new charge of test oil (500 mL).
18. Push test oil heaters button "ON".
19. Close valve to support oil chamber vacuum manometer.
20. Allow at least 20 minutes to elapse since the support oil heaters were turned on (step 5). Then start support oil pumps.
21. Turn timer switch to "STOP" position.
22. Set electric timer to 10 min and 0 s. Then lock timer dial.
23. Turn rpm adjust control to "ZERO" position.
24. Check calibration of electronic rpm counter by setting the input sensitivity control to "CHECK" position, and the gate selector switch to "ONE SECOND" position. The counter should read 60. After checking, turn input sensitivity control to about midscale.
25. Turn load bypass control to "OPEN" position.
26. When the test oil and support oil temperature are up to the specified value of 73.9° . 2.5°C (165° . 5°F), check support oil chamber vacuum 2.5 to 10.2 cm ([1 to 4 in], of water, see 4.2.3).
27. Check test oil flow rate as follows:

    a. Turn 3-way valve L to stop flow from the Ryder gear machine. By noting the drop of oil level in the test oil sump, measure the time required for 270 mL of test oil to accumulate in the Ryder gear machine. Then turn valve L back to running position.
    b. Adjust the variable speed transmission to vary the speed of the test oil pressure pump, until the specified flow rate at 270 . 5 mL/min is obtained.

6.3 Test procedure.

6.3.1 Test on "A" side of test gears.

NOTE 2: The "A" side of the gear refers to the side obtained by installing the test gears with the serial numbers facing the end cover. The "B" side is obtained by reversing thegear (serial numbers facing away from the end cover).

1. Check all temperatures and pressures for the specified limits. (See paragraph 3). Recheck test oil flow rate if necessary.
2. Make sure that the gear-tooth indexing rachet is locked in disengaged position.
3. Make sure that the load adjust control is wide open (counterclockwise).

4. Start drive motor.
5. Increase gear speed by turning the rpm adjust control clockwise until the specified 10,000 . 100 rpm is obtained. Measure speed with the electronic rpm counter.
6. Turn load bypass control to "CLOSE" position.
7. Apply load slowly by turning the load adjust control clockwise. Stop when the load oil pressure is 34 kPa gage (5 psig).

NOTE 3: In adjusting load oil pressure, take care not to overshoot the desired pressure.

8. Check gear speed.
9. Turn timer switch to "START" position. The 10-minute test run now begins.
10. Record all pertinent data about midway into the 10-minute test run.
11. At the end of the l0-minute run, the electric timer will automatically stop the drive motor and open the load bypass valve. However, the timer switch on the control console will remain in "START" position, the load bypass control will remain in "CLOSE" position, and the rpm adjust control will remain in running position. To restore these to their respective off positions:

    a. Turn load bypass control to "OPEN" position.
    b. Turn timer switch to "STOP" position.
    c. Turn rpm adjust control to "ZERO" position.

12. Make visual scuff inspection of the narrow test gear, as follows:

    a. Turn 3-way valve M to bypass flow around the Ryder gear machine.
    b. Remove corks from inspection holes on the gear case.
    c. Blow off excess oil from the test gear with compressed air through the inspection hole.
    d. Install microscope and inspection light.
    e. Focus microscope and adjust the 20% grid lines over the contact area of the tooth. (See Figure 10).
    f. Set indexing ratchet wheel to "No. 1" position.
    g. Estimate the percent scuff on No. l tooth to the nearest 5%, and record the value (See Figure 12).

    h. Operate indexing ratchet to give No. 2 tooth, then No. 3 tooth, etc., repeating step (g) until all 28 teeth have been examined
    i. Remove microscope and light. Replace corks in the holes.
    j. Turn 3-way valve M to running position.
    k. Lock indexing ratchet in disengaged position.

13. Repeat steps (3) through (12) at the next test load of l70 kPa (10 psig) load oil pressure.

14. Continue the test at 34 kPa (5 psi) load oil pressure increments, until the average scuffed area for all 28 teeth is closest to 22.5%. At this juncture, visually estimate the percent effective tooth width to the nearest 1 percent, and record the value.
15. Also at this juncture, install photographic equipment and take a photograph of each gear tooth. Make the first frame a test identification frame.

NOTE 4: Step (15) is optional. Photographic records of the scuffed gear teeth are not required in current test specifications. Using the photographic equipment as supplied (see 4.5), and Plus-X film, the camera should be set at a lens aperture ("f" value) of 16. The actual exposure time is controlled by the electronic flash unit; but to insure adequate exposure, the camera speed should be set at 1/25 s.

16. Further continue the test at 34 kPa (5 psi) load oil pressure increments, until an approximate average scuffed area of 40% or more is obtained.
17. Push test oil heaters button "OFF".
18. Drain all test oil by first opening 3-way valve L and then valves K and N.
19. Close all drains.
20. Stop test oil pressure pump.
21. Push support oil heaters button "OFF".
22. Stop support oil pumps.

NOTE 5: Leave other controls on if test on "B" side will proceed after a short midday break. Otherwise, push instruments button "OFF", and turn off main electric switch and main water valve to test cell.

6.3.2 Test on "B" side of test gears.

NOTE 6: The "B" side refers to the side obtained by reversing the gear (serial numbers away from end cover).

1. Push support oil heaters button "ON".
2. Remove gear case end cover.
3. Remove O-ring.
4. Blow out test oil jet with compressed air.
5. Blow out line screen filters with compressed air.
6. Reverse test gears so that the serial numbers face inward, again matching timing marks on shafts and gears. Check for proper seating of the retaining nut, and lock the lock washer.
7. Install new O-ring.
8. Reinstall gear case end cover.
9. Start test oil pressure pump.
10. Flush test oil system with test oil, in accordance with 6.2, step (15).

NOTE 7: If the supply of test oil is limited, this flushing between tests on "A" and "B" sides of the gears may be omitted.

11. Turn valves L, M, and N to running position.
12. Fill test oil system with a new charge of test oil (500 mL). Then push test oil heaters button "ON".
13. Allow at least 20 min to elapse since the support oil heaters were turned on (step 1). Then start support oil pumps.
14. When the test oil and support oil temperatures are up to the specified value of 73.9° ± 2.5°C (165° ± 5°F) check support oil chamber vacuum 2.5 to 10.2 cm (1 to 4 in) of water.
15. Check test oil flow rate as described in 6.2, step (27).
16. Proceed on test on "B" side of the test gears, by following the procedure outlined in 6.3.1 steps (1) through (18).
17. Flush test oil system with Stoddard solvent, as follows:

    a. Turn 3-way valve M to running position.
    b. Turn 3-way valve N to running position.
    c. Fill test oil sump with about 750 mL of the solvent. Watch drop of level in sump. Slowly turn the test gears by operating the indexing ratchet. Allow about 400 mL to drain through L.
    d. Drain the next 100 mL by opening drain K also. Then close drain K.
    e. Turn valve L to running position, so as to permit flushing of that section of the test oil system between valve L and sump.
    f. Drain the remaining 250 mL by turning valve M to bypass the Ryder gear machine, and turning valve N to "DRAIN".

NOTE 8: The flushing fluid is to be used only once and then discarded.

18. Flush test oil system with petroleum ether, by repeating step (17).
19. Operate test oil pressure pump in reverse for about 60 s, and then resume normal rotation. To reverse pump, adjust the variable speed transmission.
20. Stop test oil pressure pump.
21. Remove gear case end cover.
22. Remove O-ring.
23. Remove used test gears.
24. Blow out gear case and test oil jet with compressed air.
25. Blow out line screen filters with compressed air.
26. Reinstall gear-case end cover (without test gears).
27. Push support oil heaters button "OFF". Then stop support oil pumps.
28. Push instruments button "OFF".
29. Turn off main electric switch to test cell.
30. Turn off main water valve to test cell.

6.3.3 Sequence for repeat test. Repeat test on the same oil may be conducted by omitting steps (26) through (30) of the sequence for "B" side of the test gears (6.3.2.

Instead, proceed from step (8) of the sequence for preparation (6.2), and continue through the complete test sequence (6.3)).

6.3.4 Emergency stop.

1. Push emergency stop button.
2. Push instruments button "OFF".
3. Turn off main electric switch to test cell.
4. Turn off main water valve to test cell.
5. Investigate and take appropriate action.

6.4 Inspection and rating.

6.4.1 Scuffed area. The scuffed area of a gear tooth is that area from which the axial grinding marks have been removed by scratching, scoring, abrasion, and wear. As shown in Figure 10, areas A and B are scuffed; area C is not scuffed. Note that only the actual working portion (areas A plus B plus C) of the tooth surface is considered in the definition of areas.

6.4.2 Percent of tooth area scuffed. The percent of tooth area scuffed is that portion of the working surface of a gear tooth that has been scuffed. Referring to Figure 10,

$$\text{Percent tooth area scuffed} = \frac{\text{scuffed area} * 100}{\text{working area}} = \frac{A+B}{A+B+C} * 100$$

The percent of tooth area scuffed is estimated visually as accurately as possible, but at least to the nearest 5%, for each individual tooth, with the aid of a net reticule mounted in the eyepiece of the inspection microscope.

6.4.3 Average percent of tooth area scuffed. The average percent of tooth area scuffed is the algebraic average of the percent of scuffed area of all 28 teeth of the narrow test gear. Calculate this value to the nearest 1%.

6.4.4 Effective tooth width. The effective tooth width is the actual width of the narrow gear tooth that is in contact with the tooth of the wide gear. To determine the effective tooth width, measure the gross tooth width by means of a micrometer, and estimate the ratio of effective tooth width to gross tooth width by visual examination or with the aid of a suitable scale, referring to Figure 10, and 7.1.

6.4.5 Load carrying ability. The load-carrying ability, or the scuff limited load, of a lubricant is defined as the gear tooth load at which the average per-cent of tooth area scuffed is 22.5% for the narrow test gear as deter-mined by the method outlined herein.

6.4.6 Data sheets. The suggested data sheets are shown in Figures 11 and 12. Note that the gear teeth inspection data sheet (Figure 12) has provision for computing the average percent of tooth area scuffed. This sheet also contains a column for entering the results obtained from the examination of the optional photographic records taken at a load giving approximately 22.5% scuff.

6.4.7 Determination of load carrying ability.

1. Plot the average percent of tooth area scuffed for the narrow test gear versus the load oil pressure. The use of a semilogarithmic paper is recommended for this purpose. Figure 13 is a suggested plot and calculations sheet, showing typical results from tests on both sides of a test gear.
2. Determine from the plotted curve the load oil pressure at which the average percent of tooth area scuffed is 22.5%. In Figure 13, this condition is represented by point X for "A" side, and point Y for "B" side of the test gear.
3. Calculate load carrying ability (see 7.2).

6.4.8 Relative rating procedure. This procedure consists of reporting the load carrying ability of an oil under test as a percentage of the load carrying ability of a standard reference oil, both ratings being determined by the procedure outlined in 6.4.7. Details of this comparative procedure are given in the following paragraphs.

NOTE 9: Standard reference oil can be obtained from Southwest Research Institute, P.O. Drawer 28510, San Antonio, TX 78228.

6.4.9 Determination of the average rating on standard Ryder gear test reference oil.

1. For a new rig, at least eight determinations (four gears) shall be made, according to the foregoing procedure, on the standard reference oil. If the high and low values obtained do not differ by more than 1400 N/cm (800 lb/in) the average is taken as the reference oil rating. If the high and low values differ by more than 1400 N/cm (800 lb/in) four additional determinations (two gears) shall be made and the average of the 12 determinations taken as the reference oil rating.
2. After a major overhaul to a rig such as a bearing or shaft replacement, four determinations (two gears) shall be made on the standard reference oil. If the average of these ratings does not differ from the average obtained prior to the overhaul by more than 525 N/cm (300 lb/in) and the high and low values obtained do not differ by more than 1400 N/cm (800 lb/in) this average will be taken as the reference oil rating. If the ratings obtained do not meet the above criteria, four additional determinations (two gears) shall be made and the average of all eight determinations taken as the reference oil rating.
3. At least two determinations (one gear) shall be made on the standard reference oil for each 20 determinations made on oils under test. The reference oil rating is a progressive rating in that it will consist of the average

value obtained on the last eight determinations, i.e., when two new determiations are made, the two oldest determinations of the eight determinations used in determining the previous reference oil rating are dropped, the two new ratings replacing them and a new reference oil average rating obtained. The sole exception to the above is where it is necessary to run 12 determinations to establish an average on a new rig. (See item l, 6.9).

NOTE 10: Maximum and minimum acceptable average ratings for the reference oil are 5780 N/cm (3300 lb/in) and 4030 N/cm (2300 lb/in), respectively. Rigs which do not give an average reference oil rating within these limits shall not be considered as being satisfactory. Faulty maintenance practices, improper test procedures, and/or the need for major overhaul can be indicated if the average on the reference oil does not fall within these limits.

4. Determinations, as necessary, shall be made on the oil under test and an average obtained. Should the high and low values of these determinations differ by more than 1400 N/cm (800 lb/in) additional determinations should be made to establish an acceptable average.

7. CALCULATIONS

7.1 Calculate the effective tooth width (cm, in)

$$W_2 = W_1 * R$$

where:
W_2 = effective tooth width in cm or in.
W_1 = measured gross tooth width in cm or in.
R = W_2/W_1 = estimated ratio.

7.2 Calculate load carrying ability by the following formula:

$$P = \frac{KL}{W_2}$$

where:
P = load carrying ability (or scuff limited load) of the lubricant, N/cm (lb/in).
K = Ryder gear machine constant (4.1.1).
L = Load oil pressure, gage Pa (psig).
W_2 = effective tooth width, cm (in).

7.3 Calculate the percent relative rating using the following formula:

$$\textit{Relative rating, percent} = \frac{\textit{test oil average rating}}{\textit{reference oil average rating}} * 100$$

8. REPORTING

8.1 Report the percent relative rating (see 7.3).

where:
P = load carrying ability (or scuff limited load) of the lubricant, N/cm (lb/in).
K = Ryder gear machine constant (4.1.1).
L = Load oil pressure, gage Pa (psig).
W_2 = effective tooth width, cm (in).

8.2 Calculate the percent relative rating using the following formula:

$$\textit{Relative rating, percent} = \frac{\textit{test oil average rating}}{\textit{reference oil average rating}} * 100$$

9. REPORTING

9.1 Report the percent relative rating (see 7.3).

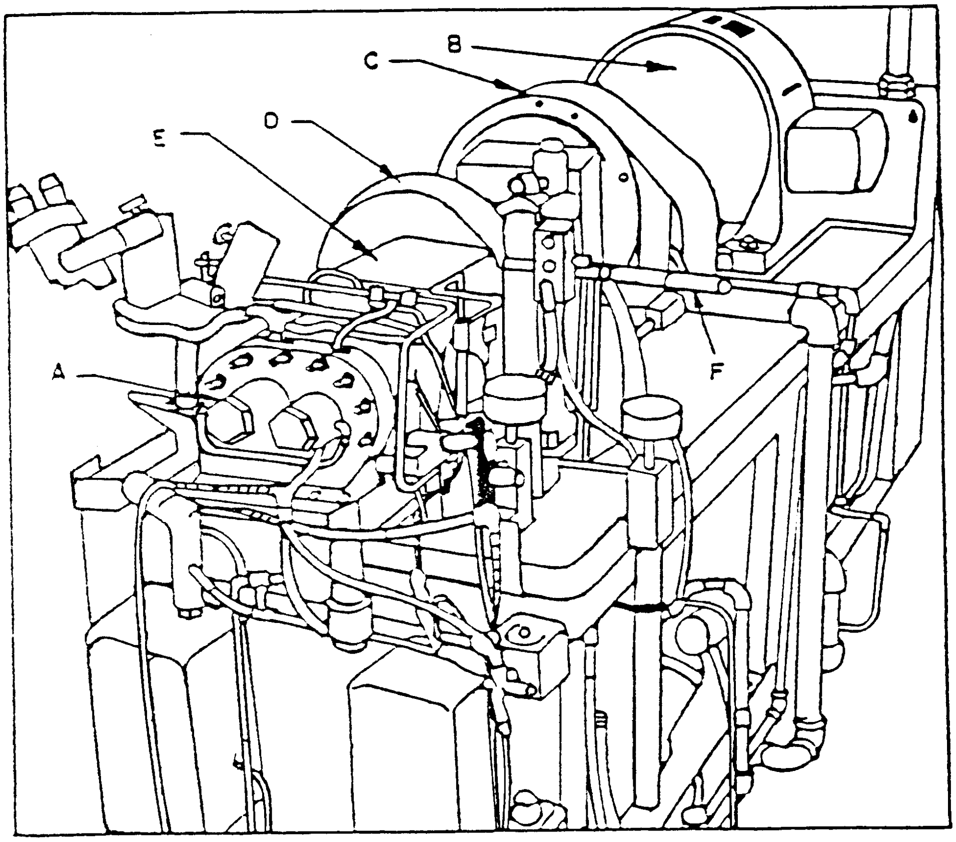
10. PRECISION

10.1 Repeatability. Results from one apparatus and operator should be considered suspect if the relative ratings differ from their mean by more than 10%.

10.2 Reproducibility. Results from different apparatus and/or operators should be considered suspect if the relative ratings from each apparatus and/or operator differ from their mean by more than 5%.

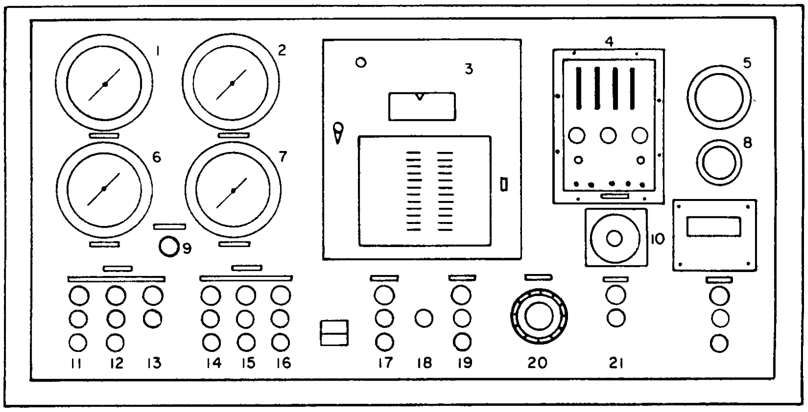
Method prepared by:

Navy - NAPC - 1986



A. RYDER GEAR MACHINE
B. DRIVE MOTOR
C. DYNAMATIC COUPLING
D. STEP-UP GEAR BOX
E. ADAPTOR
F. INDEXING RATCHET

FIGURE I. Ryder gear - ERDCO universal tester.



1.	Support-oil pressure gage	12.	Support-oil heaters on-off buttons
2.	Test-oil pressure gage	13.	Load bypass open-close switch
3.	Multi-point temperature indicator	14.	Test-oil pressure pump start-stop buttons
4.	Electronic rpm counter	15.	Test-oil scavanger pump start-stop buttons
5.	Gear-box pressure gage	16.	Test-oil heaters on-off buttons
6.	Load-oil pressure gage (high range)	17.	Instruments on-off button
7.	Load-oil pressure gage (low range)	18.	Emergency stop button
8.	Running-time meter	19.	Drive motor start-stop buttons
9.	Load-adjust control	20.	RPM-Adjust control
10.	Electric timer	21.	Timer start-stop switch
11.	Support--oil pumps start-stop buttons		

FIGURE 2. Gear machine control console.

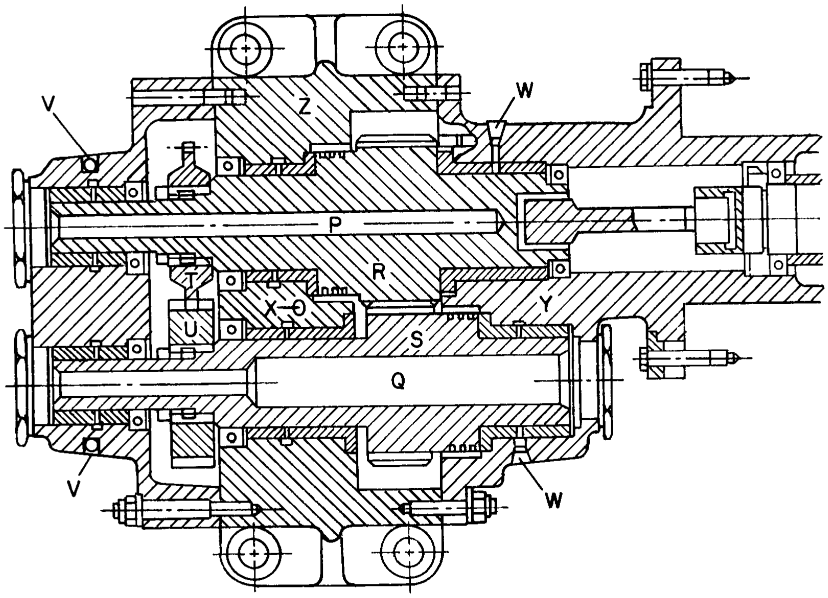


FIGURE 3. Ryder gear machine (section).

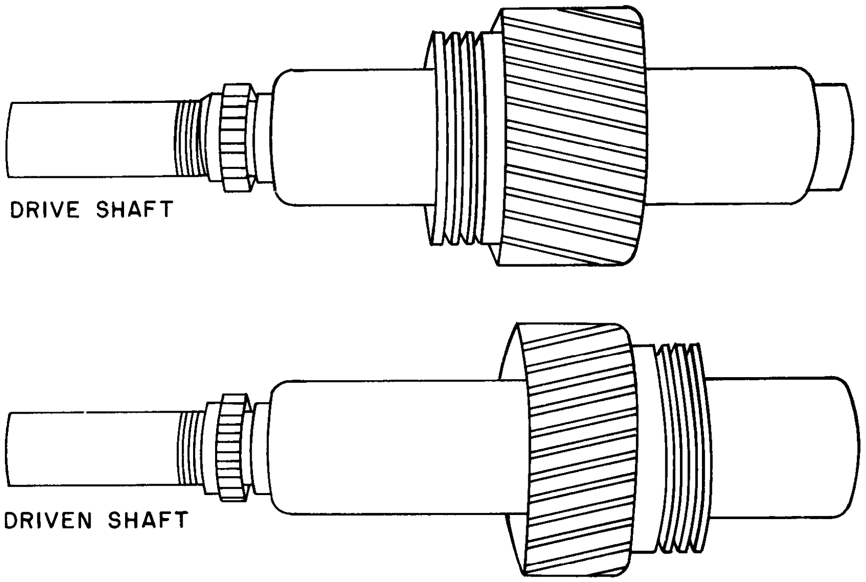


FIGURE 4. Slave gears.

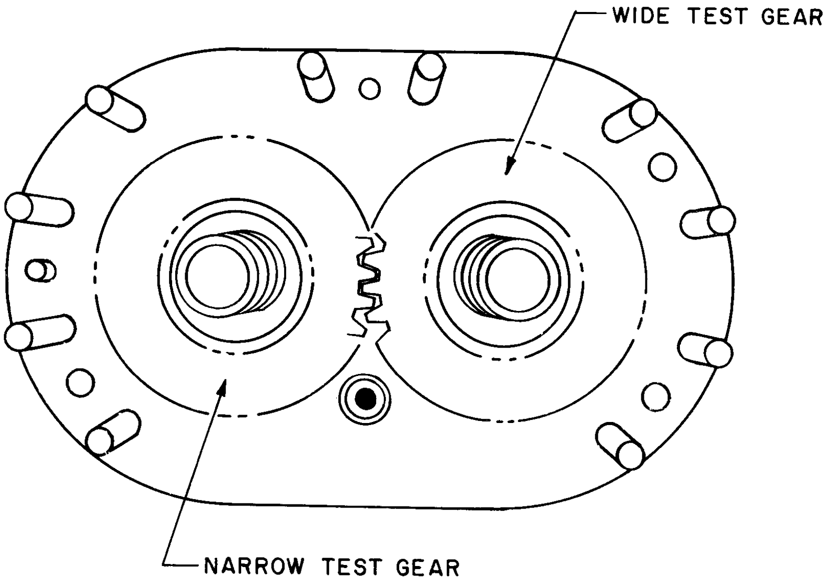


FIGURE 5. Test gears installed.

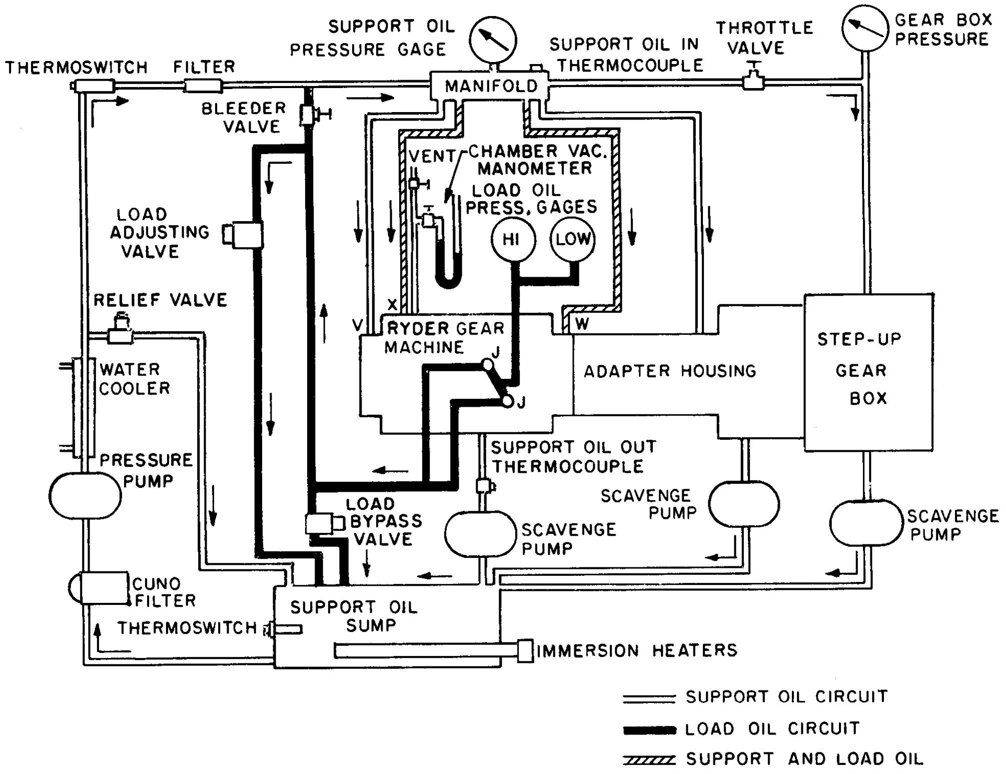


FIGURE 6. Support and load oil system.

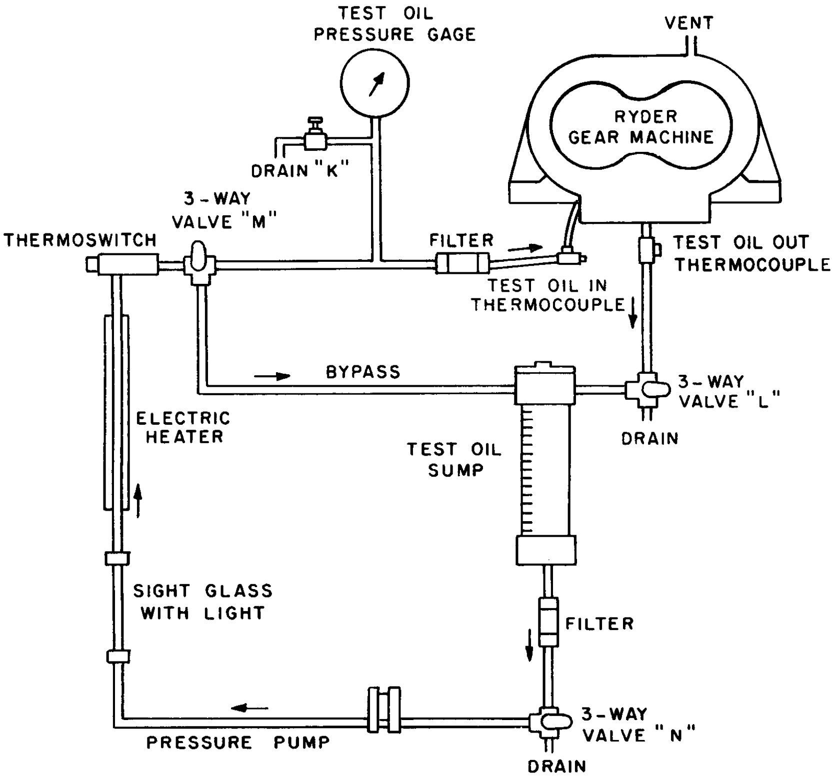


FIGURE 7. Test oil system.

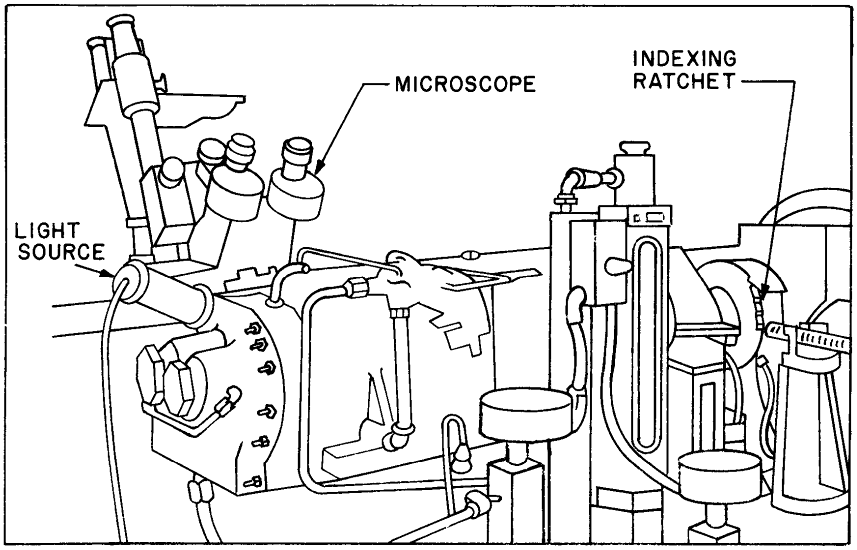


FIGURE 8. Inspection equipment.

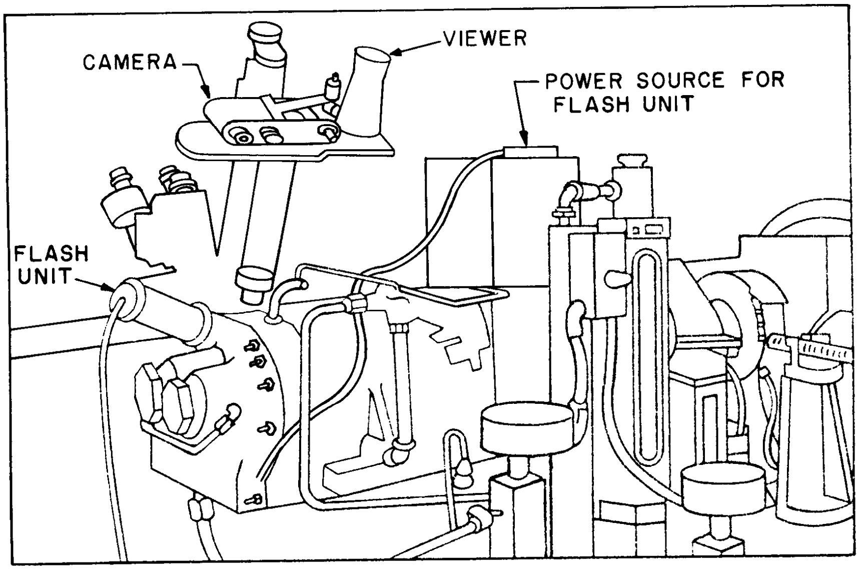
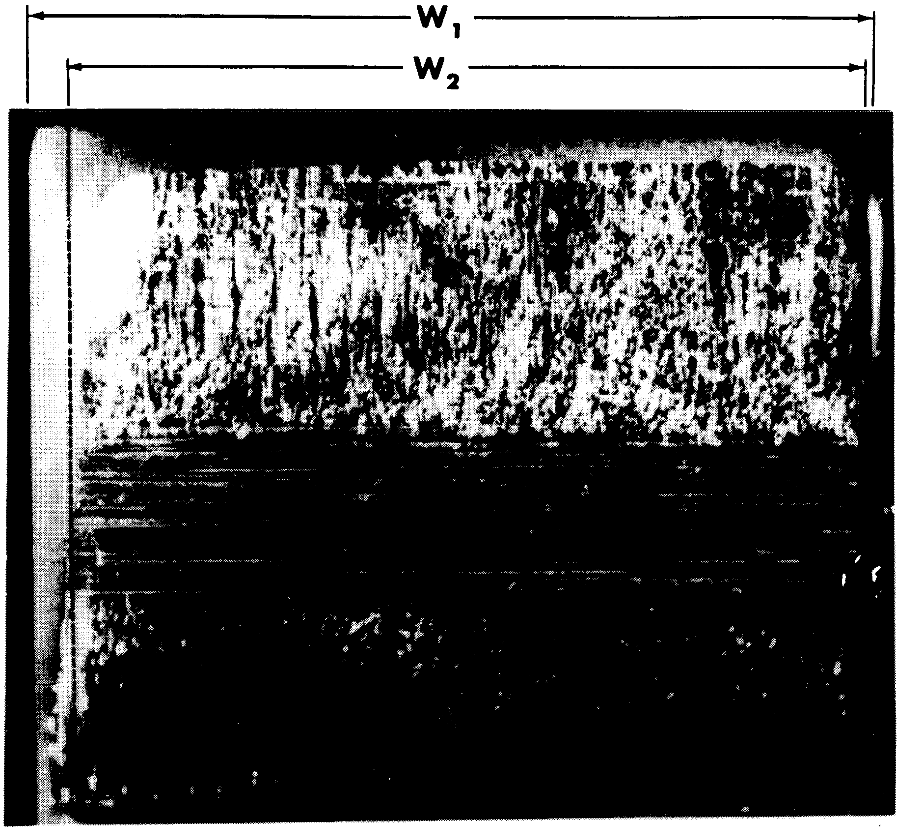


FIGURE 9. Photographic equipment.



Area "A" .... Scuffed
Area "B" .... Scuffed
Area "C" .... Not Scuffed

W_1 = Gross tooth width
W_2 = Effective tooth width

Figure 10. Scuffed Tooth (Enlarged)

Test No. _______
Sheet No. ______

LOAD-CARRYING ABILITY TEST
OPERATING CONDITIONS DATA SHEET

OIL CODE ____________________ TEST SPECIFICATION ____________________

DATE ____________________ OPERATOR ____________________

TESTER NO. ____________________ MACHINE NO. ____________________
TEST GEARS: NARROW GEAR T-116286 _______ SERIAL NO. ____________________
WIDE GEAR T-116287 _______ SERIAL NO. ____________________
GEAR SIDE: ______________ "A" SIDE
______________ "B" SIDE

	Record the following after 5 minutes operation at each load														
Load Oil Pressure Gage kPa (psig)	34 (5)	69 (10)	103 (15)	138 (20)	172 (25)	207 (30)	241 (35)	276 (40)	310 (45)	345 (50)	379 (55)	414 (60)	448 (65)	483 (70)	517 (75)
Time															
R.P.M.															
Support Oil Pressure Gage kPa (psig)															
Test Oil Pressure Gage kPa (psig)															
Test Oil "In" Temp. °C (°F)															
Test Oil "Out" Temp. °C (°F)															
Support Oil "In" Temp °C (°F)															
Support Oil "Out" Temp. °C (°F)															

TEST OIL FLOW TIME FOR 270 mL ____________ MINUTES ____________ SECONDS

TEST OIL SUMP LEVEL, mL: START OF TEST ____________________
END OF TEST ____________________
CONSUMPTION ____________________

CHAMBER VACUUM, cm (IN.) OF WATER: START OF TEST ____________________
END OF TEST ____________________

REMARKS: ____

____

____

FIGURE 11. Operating conditions data sheet.

Test No. ________
Sheet No. ________

LOAD-CARRYING ABILITY TEST
GEAR TEETH INSPECTION DATA SHEET

OIL CODE ______________________ TEST SPECIFICATION ______________
DATE ______________________ OPERATOR ______________
GROSS TOOTH WIDTH ____________ cm (INCHES) GEAR SIDE _____ "A" SIDE
PERCENT EFFECTIVE TOOTH WIDTH ____________ _____ "B" SIDE

Tooth No.	Percent of Tooth Area Scuffed at Load Oil Pressure Gage kPa (psig) of															Photo-graphs
	34 (5)	69 (10)	103 (15)	138 (20)	172 (25)	207 (30)	241 (35)	276 (40)	310 (45)	345 (50)	379 (55)	414 (60)	448 (65)	483 (70)	517 (75)	
1																
2																
3																
4																
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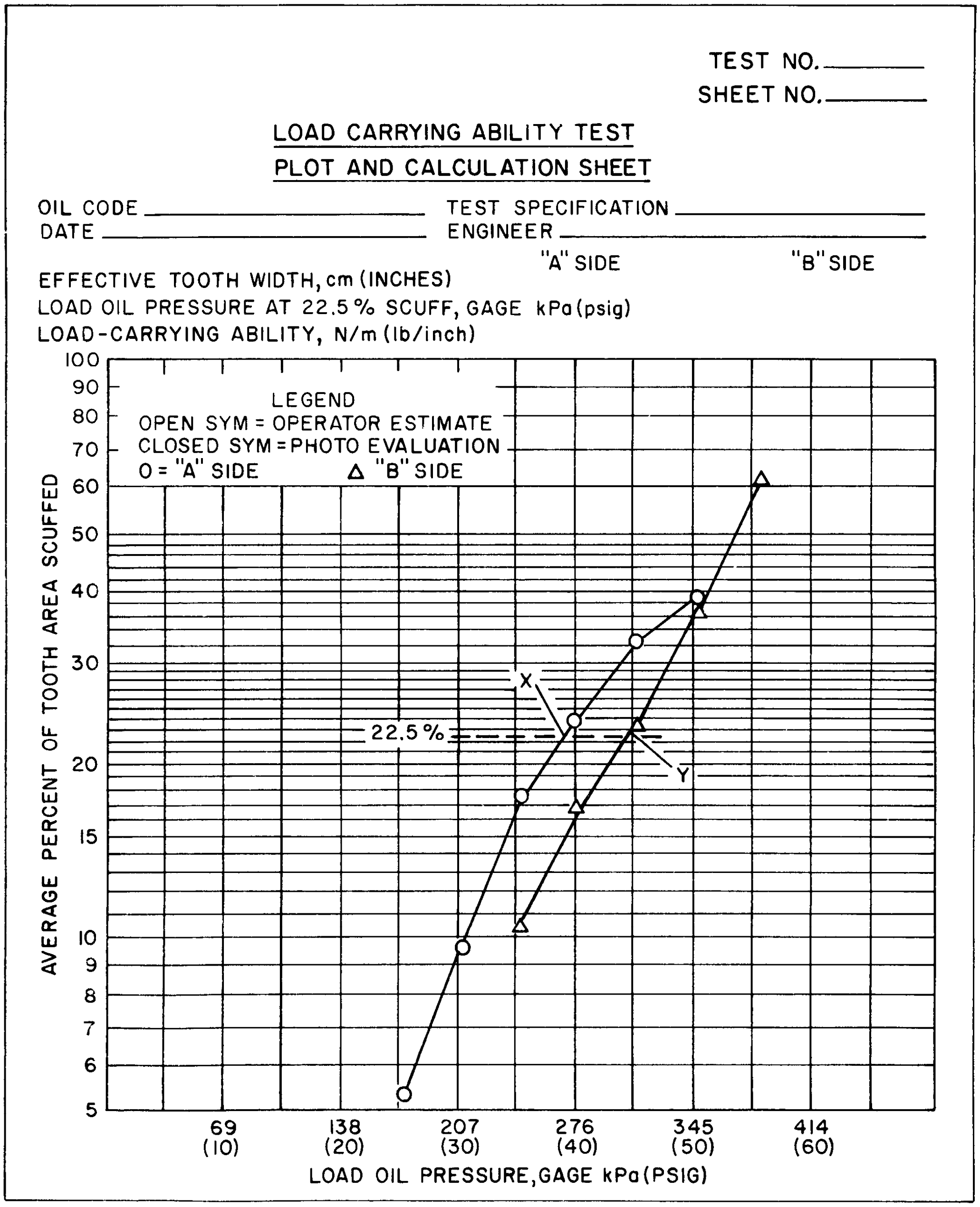
22																
23																
24																
25																
26																
27																
28																
Sum of Percent																
Average Percent																

FIGURE 12. Gear teeth inspection data sheet.



TEST NO. ________
SHEET NO. ________

LOAD CARRYING ABILITY TEST
PLOT AND CALCULATION SHEET

OIL CODE ____________________ TEST SPECIFICATION ____________________
DATE ____________________ ENGINEER ____________________

	"A" SIDE	"B" SIDE
EFFECTIVE TOOTH WIDTH, cm (INCHES)		
LOAD OIL PRESSURE AT 22.5% SCUFF, GAGE kPa(psig)		
LOAD-CARRYING ABILITY, N/m (lb/inch)		

FIGURE 13. Plot and calculation sheet.

Method 6517
September 30, 1986

HIGH-TEMPERATURE GEAR LOAD-CARRYING CAPACITY OF LUBRICATING OILS

1. SCOPE

1.1 This method describes a procedure for determining the gear load-carrying capacity of lubricating oils at controlled test gear temperatures of 220°C (428°F) through 371°C (700°F).

2. SUMMARY

2.1 The method consists of subjecting the lubricant to a series of controlled temperature tests at increasing gear-tooth loads using a set of special spur test gears. Each tooth of one of the gears is examined to determine the working area scuffed after each load step. The load-carrying capacity of the lubricant is determined by the load, in pounds per inch of tooth width, required to produce 22.5% average scuff of the total working area of the examined test gear.

3. SAMPLE SIZE

3.1 Approximately 2 L of test oil.

4. REFERENCES, STANDARDS, AND APPARATUS

4.1 Test unit. The apparatus in this method is the WADD gear machine, or equivalent, available from Erdco Engineering Corp., Addison, IL. This apparatus consists of a WADD gear machine adapted to a modified Erdco universal tester drive stand, a support and load-oil system, a test-oil system, a radiometer, and induction heater, and the necessary instruments and controls.

4.1.1 WADD gear machine. The WADD gear machine operates on the same principle as the Ryder gear machine, the so-called "four-square" principle. However, improvements in material and design permit its operation at test gear temperatures up to 371°C (700°F). Double-row roller bearings are used to support the two parallel shafts, and screw-thread type nonrubbing seals are used to separate the test oil and support oil chambers. As shown on Figure 1, the two parallel shafts (P and Q) are connected by two slave gears (R and S) and two test gears (T and U) to form a "Square" so that the power required to operate the machine is only that required to overcome the friction losses in the gears and bearings. The slave gears are helical gears and are made as integral parts of the two shafts; the test gears are spur gears and are replaceable. Load on the test gears is obtained through the application of a controlled oil pressure in the load chamber (X), which causes an axial movement of one shaft relative to the other. A torsional load is thus applied upon the shafts due to the helical slave gears, thereby loading the test gears. The relation between the tooth load on the test gears and the load-oil pressure can be obtained by arithmetic calculation, calibration, or both. Arithmetic calculation is used herein.

4.1.2 Test gears. The test gears are special spur gears, made of AMS 6475 steel, nitrided and ground, having 28 teeth, 8.89-cm (3.5-in) pitch diameter, 8 diametral pitch, 22.5° pressure angle,0.0000- to 0.00025-cm (0.000- to 0.0001-in) tip relief, and 0.0279- to 0.0356-cm (0.011- to 0.014-in) backlash. (Sier-Bath Gear Co., Inc., North Bergen, N.J., 07047, part No. SB-19293-AN, narrow gear; SB-19469-AN, wide gear; or equivalent.)

NOTE 1: The working area of the gear teeth is finish ground to the required profile after nitriding to remove the "white-layer" formed during the nitriding process.

NOTE 2: Each set of test gears comprises gears of two different widths. The narrow test gear is used for rating purposes. The mating gear is wider in order to maintain a constant contact width between the two gears at different tooth loads. Both sides of the test gears are used for testing. The "A" side of the gears refers to the side obtained by installing the test gears with the serial numbers facing the end cover. The "B" side is obtained by reversing the gears (serial numbers facing away from the end cover). Test gears can be used only once on each side and then discarded.

4.1.2.1 The entire web of each side of the narrow test gear must be electroplated with black chromium. The black chromium rings thus formed, extending from the root diameter of the gear teeth approximately 0.64 cm (1/4 in) toward the center of the gear, provides a continuous black body radiation surface for accurate test gear temperature measurement during test.

4.1.3 Drive system. An Erdco universal drive system, or equivalent, is used to drive the WADD gear machine. A 50-hp induction motor drives the machine through a variable speed dynamatic coupling, a step-up gearbox, and an adapter block to which the WADD gear machine is attached. The only modification required to the drive system is the relocation of the mounting studs in the end of the adapter block to match the holes in the WADD gear machine mounting flange. The test gear speed is controlled by adjusting the field excitation of the dynamatic coupling. By this means accurate speed control can be obtained at the specified test speed of 10,000 rpm.

4.1.4 Support and load-oil system. The support and load-oil system used with the WADD gear machine serves two purposes: To provide lubrication to all necessary parts except the test gears, and to supply load-oil pressure to the load chamber, and thus, the application of load to the test gears. A schematic dia-gram of the support and load-oil system is shown on Figure 2.

4.1.4.1 The system is charged with approximately 30 L (8 gal) of lubricating oil, type MIL-PRF-7808 or SAE J1966. The support oil temperature measured at the distributing manifold, is maintained at a specified value of 74° ± 2.8°C (165° ± 5°F), by means of electrical heaters located in the sump, and a water cooler located after the pump, through the action of thermoswitches. Pressure lines from the distributing manifold lubricate the bearings and helical gears, as well as the adapter housing and the step-up gearbox back to the support oil sump.

4.1.4.2 The action of the load oil circuit for the WADD gear machine is shown on Figures 2 and 3. Oil from the load adjusting valve, Figure 2, enters the WADD gear machine through the load chamber located at the rear of the driven shaft. Sufficient oil is provided to maintain a constant "load-oil pressure," and to allow for leakage past the load chamber seal. The load bypass valve is opened when it is desired to release the load-oil pressure quickly, such as at the end of a test run. The optional automatic recording load oil system, shown on Figure 3, affords automatic loading of the machine and a continuous recording of the load-oil pressure. In operation, the desired load is set on the indicator, the load is then automatically changed at a constant rate to the preset value and is recorded. The load is automatically released at the end of each test run by solenoid valves which release the control air pressure to the pneumatic valves allowing the valves to return to their respective normally closed and normally open positions.

4.1.5 Test-oil system. A test-oil system capable of maintaining a test-oil temperature of 205°C (401°F) is used with the WADD gear machine for high-temperature gear load-carrying capacity evaluations. A schematic diagram of the test-oil system is shown on Figure 4. The capacity of the test-oil system is 1 L. Test oil is supplied to the test gears by means of a pressure pump through an inline filter and then to the jet, located on the unmeshing side of the gears. The oil is gravity drained from the test section through the flow check chamber and returned to the sump. Oil temperature to the test gears is maintained at 205° ± 2.8°C (401° ± 5°F) by means of two electrical band heaters located on the outside of the test-oil sump.

4.1.6 Radiometer. A Barnes R-4D1 industrial radiometer, or equivalent, is used in conjunction with the WADD gear machine to measure the narrow test gear temperature during test. The industrial radiometer consists of two components: (1) the temperature sensing optical head which is located inside the test cell, approximately 137 cm (54 in) from the end cover of the WADD gear machine, and is aimed at the web of the narrow test gear through the hole provided in the gear machine end cover, and (2) the electronics and readout unit which is located in the control console of the test rig.

4.1.7 Induction heater. A Lepel T-5N-3, or equivalent, induction heater having a 5 kW output at 450 kHz with a wide range continuous grid control is used to heat the test gears to the desired test temperature.

4.1.7.1 A load coil transformer is used in conjunction with the induction heater to minimize power loss from the induction heater. The load coil transformer is mounted inside the test cell approximately 38 cm (15 in) to one side of the WADD gear machine. The test gear heating coil, shown in Figure 5 without the end cover, is mounted inside the end cover of the WADD gear machine and is connected directly to the load coil transformer output terminals by removable, silver-plate connectors.

4.1.8 Instrumentation and controls. All except one of the instruments and controls that require constant attention during test are located on the control console, as shown on Figure 6. The one item requiring constant attention and not included in the control console is the induction heater. The induction heater power supply is normally located abutting the control console for convenience. Attention should be directed to the instruction manual for the specific induction heater used to determine the exact control method to use. Items that do not require frequent attention are located inside the test cell. The functions of most of the instruments and controls are apparent. Therefore, only selected items will be explained further.

4.1.8.1 Speed measurement and control. An electronic counter (Figure 6, item 10), located on the control panel, measures the speed. This instrument is actuated by pulses supplied by a variable reluctance pickup (the variable reluctance being provided by a 60-tooth gear operated from the driven shaft of the step-up gearbox). The rpm adjustment control (Figure 6, item 28), located on the con-sole, regulates the field excitation of the dynamic coupling and thus the gear speed.

4.1.8.2 Control of test-run duration. An electric timer (Figure 6, item 16), located on the console, is used to control the duration of each test run. As soon as the desired test load and test gear temperature has been reached, the timer is started. After the ten-minute run is over, the timer automatically turns off the drive motor and opens the load-bypass valve. The timer switch must be moved to the "stop" position immediately following coast down of the machine.

4.1.8.3 Support-oil controls. The support-oil pump buttons (Figure 6, item 18) on the console are connected to the support-oil pressure pump as well as the scavenge pumps. Pressing the start or stop button controls all pumps at the same time. The support-oil heater buttons (Figure 6, item 19) control both the electric heaters and the water cooler through the action of thermoswitches. The support-oil chamber vacuum is measured by a water manometer located on the test stand. A valve connecting the chamber to a vent (Figure 2) should be adjusted to give a chamber vacuum of approximately 2.54 cm (1 in) of water under normal test conditions.

4.1.8.4 Control of load-oil pressure. The control of load-oil pressure has been explained in 4.1.4.2. The load adjusting valve is air actuated and is controlled by the load-adjust control (Figure 6, item 6 or 15) located on the console. The load-bypass valve, is solenoid actuated and is controlled by the load-bypass switch (Figure 6, item 20) on the console. At the end of a run, the solenoid circuit will automatically be open by the electric timer. This opens the load-bypass valve, but the load-bypass switch on the console remains in the "close" position. For this reason, the switch must be turned to the "open" position prior to the next run.

4.1.8.5 Emergency stop. The emergency stop button (Figure 6, item 26) on the console stops the driver motor and turns off all the controls except those for instruments. The timer switch and the load-bypass switch remain in the positions to which they were originally set.

4.1.8.6 Scuff rating equipment. A microscope and a light source are used for visual inspection of the narrow test gear. An optional closed circuit television camera and monitor (Figure 6, item 4) may be used for inspection of the narrow test gear. To inspect the narrow gear, the machine is first stopped. The inspection can then be made through special inspection holes provided in the gear-case end cover. These holes are closed by corks during the test run.

4.1.8.7 Inspection equipment. The standard 18X microscope furnished with the Erdco Ryder gear machine, or equivalent, is slightly modified by the addition of a supplementary lens in the right objective-lens opening. The right eyepiece of the microscope contains a net reticule that divides the gear-tooth area lengthwise into seven sections. The grid lines provide a convenient means for the visual estimation of the percent of gear-tooth area that has been scuffed.

4.1.8.8 Gear-tooth indexing ratchet. An indexing ratchet is normally used for rotating the test gears so that the teeth of the narrow test gear can be inspected individually. If the optional closed circuit television camera and monitor are used, a television antenna rotor may be adapted to engage the output shaft of the step-up gearbox and provide the rotation of the narrow test gear for remote inspection.

4.1.8.9 Additional instruments:

a. Stop watch for oil flow-rate check.
b. Micrometer 0 to 2.5 cm (0 to 1 in) for the measurement of the gross tooth width of the narrow gear.
c. Scale with divisions of 0.04 cm (1/64 in).
d. Set of conventional mechanical tools as necessary for assembly, disassembly, maintenance, and repair.

4.2 Test gear plating procedure.

4.2.1 The following procedure is used to plate the black chromium rings on the web of the narrow test gears:

1. Clean the gear with dry-cleaning solvent and rinse with petroleum ether.
2. Mask the area to be plated with masking tape. (The area to be plated includes the flat area on both sides of the narrow test gear between the root diameter of the gear teeth and the OD of the test gear hub, not including the fillet.)
3. Place the gear in a wire holder and immerse in hot platers wax. Dip the gear successively until the wax has formed a coating approximately 0.32 cm (1/8 in) thick on all gear surfaces.

4. Allow the wax coating to cool and harden. Cut a groove in the wax coating with a knife to expose the masking tape.
5. Remove the masking tape to expose the area to be plated.
6. Lightly sand blast the exposed portion of the test gear using No. 30 grit aluminum oxide.
7. Immediately following the sand blast, the gear must be rinsed thoroughly with distilled water and placed in the nickel strike for 2 min.
8. Rinse the gear again with distilled water and place in the nickel plate bath. The nickel is plated to a thickness of approximately 0.0025 cm (0.001 in) (approximately 6 min).
9. Rinse the gear thoroughly with distilled water. It is important that all of the liquid nickel solution be removed from the gear and wire holder prior to proceeding to the chrome plate bath.
10. After thoroughly rinsing the gear in distilled water, place it in the chrome plate bath and plate until the nickeled area is uniformly covered with black chromium (approximately 20 min).
11. Rinse the gear thoroughly with distilled water. Remove the platers wax from the gear.
12. Clean the gear with dry cleaning solvent and rinse with petroleum ether.
13. Dip the cleaned test gear in standard reference oil and store in suitable container until required for test.

NOTE 3: Organic contamination of the nickel solution will produce a wrinkled, brittle-plated surface which flakes easily. Organic or chloride contamination of the chrome bath will produce a yellowish-brown plate which is unacceptable. If contamination of either bath exists, discard the solution and prepare new plating solution.

4.2.2 Test gear nickel strike and plating solution. Approximately 4 L of nickel strike and plating solution is required, as follows:

a. Prepare the nickel strike and plating solution in a clean borosilicate glass beaker.
b. Add 3 L distilled water to 896 g purified crystalline nickelous chloride ($NiC1_2$) and dissolve the crystals.
c. Add 308 g reagent grade hydrochloric acid ($HC1$) to the solution.
d. Add additional distilled water to make approximately 3.785 L of solution.

4.2.3 Black chromium plating solution. Approximately 3.8 L (1 gal) of black plating solution is required, as follows:

a. Prepare the chromium plating solution in a clean borosilicate glass beaker.
b. Dissolve 1 kg reagent grade crystalline chromium trioxide ($Cr0_3$) in 3.785 L distilled water.
c. Add 1/4 g reagent grade crystalline silver nitrate ($AgNO_3$). Stir solution, cover and place in a dark area for 4 h.
d. Decant and discard precipitate.

e. Add 1 g reagent grade barium carbonate ($BaCO_3$). Stir solution, cover and place in dark area for 4 h.
f. Decant and discard precipitate.
g. Add 1 g hydrochloric acid. Stir solution, cover and store in dark area until bath solution is required for plating.

4.2.4 Plating apparatus. The following apparatus is required for plating the test gear:

a. Ring stand or other suitable rack from which the wire gear holder may be suspended.
b. DC power supply (6 to 8 V with variable power output of 0 to 20 A).
c. Nickel bar stock, approximately 5 □ 15 □ 0.64 cm (2 □ 6 □ 1/4 in), is used for electrodes in the nickel strike and nickel plate bath.
d. Sheet lead, approximately 15 □ 20 □ 0.32 cm (6 □ 8 □ 1/8 in) thick, is used for electrodes in the black chrome plate bath.
e. Plastic covered, 9 gage, copper wire is used to connect the power supply to the electrodes.

4.2.5 Plating conditions. The following conditions are used for the strike and plating baths:

	Solution temperature	Plating time, min	Power, A	Gear polarity
Nickel strike	68.3° . 2.8°C (155° . 5°F)	2	0.5	Positive
Nickel plate	68.3° . 2.8°C (155° . 5°F)	6	0.5	Negative
Black chrome plate	29.4° . 2.8°C (85° . 5°F)	20	13.0	Negative

5. MATERIALS

5.1 CAUTION - SOME MATERIALS ARE TOXIC AND HAZARDOUS. The materials references and standards listed in this section must be handled carefully. Federal Test Method 10000, Material Handling Safety Precautions, is a reference which lists all toxic and hazardous materials cited in FTMS 791. The synonyms, life hazard, flammability, handling and storage precautions, emergency treatment and measures, and spill practices of each chemical are explained.

5.2 Lubricating oil, MIL-PRF-7808 or SAE J1966.

5.3 Dry cleaning solvent (A-A-59601D).

5.4 Petroleum ether (60° - 90°C)[1,4,0].

5.5 Platers wax [u,u,u].

5.6 No. 30 grit aluminum oxide.

5.7 Purified crystalline nickelous chloride ($NiC1_2$)[u,u,u].

5.8 Reagent grade hydrochloric acid ($HC1$)[3,0,0].

5.9 Reagent grade crystalline chromium trioxide ($Cr0_3$)[u,u,u].

5.10 Reagent grade crystalline silver nitrate ($AgNO_3$)[u,u,u].

5.11 Reagent grade barium carbonate ($BaCO_3$)[u,u,u].

5.12 Hydrochloric acid [3,0,0].

5.13 Nickel bar stock [u,u,u].

5.14 Sheet lead [u,u,u].

5.15 Standard reference oil to be obtained from USAF Aero Propulsion Lab, AFWAL/POSL, Wright-Patterson AFB, OH 45433-6563 [u,u,u].

6. PROCEDURE.

6.1 Operating Conditions

6.1.1 Throughout the entire test, the test gear temperature is controlled at 220° ± 2.8°C (428° ± 5°F), or the value required by the applicable lubricant specification. Table 1 presents a list of additional operating conditions which are also controlled within the limits specified. With the tester operating within the specified limits, the test gears are loaded first to 34.5 kPa gage (5 psig) load oil pressure (nominal 230 lb/in tooth load), and then at successive increments of 34.5 kPa gage (5 psi). The duration of each loading period is 10 min ± 5 s. At the end of each ten-minute loading period, the tester is stopped and each tooth of the narrow test gear is examined to determine the percent of tooth area scuffed.

TABLE I. Operating Conditions.

Condition	Value
Test gear temperature	220° ± 2.8°C (428° ± 5°F)
Test gear speed, rpm	10,000± 10
Test-oil inlet temperature	205° ± 2.8°C (401°± 5°F)
Test-oil flow rate, mL/min	(271± 5)
Test-oil pressure	103.4 ± 34.5 kPa gage (15 ± 5 psig)
Support-oil inlet temperature	73.9° ± 2.8°C (165°± 5°F)
Support-oil pressure To load-oil system	827.4 ± 69 kPa gage (120 ± 10 psig)
To step up gearbox To WADD gear machine	241 ± 34.5 kPa gage (35 ± 5 gage)
Airflow to seals	0.33 ± 0.047 L/sec (0.70 ± 0.10 cfm)

6.1 Test Procedures

6.2 Preparation for test.

1. Turn on main electric switch and main water valve to test cell.
2. Push instrument button to “ON” position.
3. push support oil sump heater button to “ON” position.
4. Turn radiometer function switch to “READY” position.
5. Turn on power supply to induction heater and place induction heater filament switch in “ON” position.
6. Inspect test –oil system to see that it was thoroughly cleaned and dried after previous test.
7. Remove gear –case end cover.
8. Clean a set of new plated test gears with petroleum ether, and dry with air.
9. Measure gross tooth width of the narrow test gear with a micrometer, and record

10. Install test gears on proper shafts with the serial number facing out ward (narrow gear on drive shaft, wide gear on driven shaft), matching timing marks on shafts and gears. Check for proper setting of the retaining nuts and lock washers. After tightening the retaining nuts, bend one tang of each lock washer to lock the retaining nuts in position.
11. Loosen the lock nut on the driven –shaft adjusting pin (at rear of gear case) , and unscrew adjusting pin until driven shaft is free to move to the extreme rearward position without touching the adjusting pin. Using a scale set adjusting pin so that the driven shaft can move through only one –half of the normal free travel (from extreme rearward position to extreme forward position). Tighten lock nut on adjusting pin.
12. Re install gear-case end cover with induction heating coil
13. Connect induction heating coil in end cover to load coil transformer. Visually check position of induction heating coil relative to the test gear hubs.

NOTE 4. The induction heating coil should not touch either test gear and should be insulated from the test end cover. An ohm meter may be used to insure that the coil is insulated from the end cover and test gears.

14. Turn on water supply to induction heater load coil transformer and induction heating coil.
15. Turn on ventilating blower in load coil transformer.
16. Turn on ventilating blower connected to gear-case end cover.
17. Turn on air to non rubbing seals. Adjust to approximately 6.9 kPa gage (1 psig).

<u>NOTE 5:</u> Approximately 0.33 L/sec (0.7 cfm) of air (total for both air seals) is required for the nonrubbing air seals to function correctly.

18. Fill test-oil sump with approximately 1 L of test oil.
19. Start test-oil pump motor, and immediately open test-oil drain line from test-oil pressure gage and pressuretrol to allow new test oil to fill the line to the gage and pressuretrol. Close test-oil drain line.
20. Push test-oil heater button to "ON" position.
21. Allow approximately 20 min to elapse after turning support oil sump heaters on (step 3). Then start support oil pumps.
22. Turn timer switch to "STOP" position.
23. Check electric timer to insure it is set to 10 min and 0 s, then lock timer dial.
24. Turn rpm-adjust control to "ZERO" position.
25. When the support-oil temperature is up to the specified value of 73.9° . 2.8° C (165° . 5°F), adjust the support oil supply pressure to the required values.
26. Check calibration of the rpm counter by setting the input-sensitivity control to the "CHECK" position and the gate-selector switch to the "ONE SECOND" position. The counter should read "60." After checking, turn the input-sensitivity control to about midscale.

27. When the test-oil temperature is up to the specified value of 205° ± 2.8°C (401° ± 5°F), check the test-oil flow rate as follows: Adjust the speed of the test-oil pump motor until the test-oil flow is approximately 270 mL/min. Turn flow-check valve (Figure 4) to the closed position, and measure the time required to fill the flow-check chamber to the tip of the wire indicator. The time required should be 30 s. Turn flow-check valve to the open position to allow the test oil to drain to the test-oil sump. If the time required to fill the flow-check chamber to the tip of the wire indicator varies from the 30-second requirement by more than 1 s, readjust the test-oil pump motor speed and recheck test-oil flow rate as above.

<u>NOTE 6</u>: The test-oil in pressure is maintained at 103.4 ± 34.5 kPa gage (15 ± 5 psig). If the test-oil pressure is outside the specified range, a different size test-oil jet must be used. The test-oil jet sizes normally used are 0.066 and 0.081 cm (0.26 and 0.032 in) diameter, depending upon the test oil viscosity.

28. Turn radiometer function switch to "NORMAL RANGE" position.
29. Check radiometer temperature detector spot position on narrow test gear. Refocus temperature detector if necessary.

<u>NOTE 7:</u> The radiometer temperature detector must be focused accurately on the black chromium ring of the narrow test gear to insure correct test gear temperature indication.

6.2.1 <u>Test sequence on "A" side of test gears</u>.

<u>NOTE 8:</u> The "A" side of the gear refers to the side obtained by installing the test gears with the serial numbers facing the gear-case end cover. The "B" side is obtained by reversing the test gears such that the serial numbers face the gear case.

1. Turn radiometer function switch to "READY" position.
2. Recheck test-oil flow rate as in step 27 of 6.2.1.
3. Determine radiometer reference temperature using null meter on radiometer

<u>NOTE 9:</u> The reference temperature must be subtracted from the predetermined test gear operating temperature. After determining this difference, use this value to determine from a temperature/radiometer reading calibration chart the radiometer reading which must be maintained during the test run.

4. Set radiometer scale selector to appropriate scale for test temperature desired.
5. Turn radiometer function switch to "NORMAL RANGE" position.
6. Turn on air supply to automatic load-control unit.
7. Check to see that the rotating device of the gear-tooth inspection equipment is in the disengaged position.

8. Check automatic load-control set point to see that load set point is on "O."
9. Start drive motor.
10. Increase gear speed by turning rpm-adjust control clockwise until the specified 10,000 . 100 rpm is obtained.
11. Turn on power to induction heater plate and slowly adjust plate control until the radiometer reading determined in step 3 is obtained.
12. Set automatic load-control set point to 34.5-kPa gage (5-psi) load-oil pressure.
13. Check gear speed, and adjust if necessary.
14. Check gear temperature, and adjust plate control if necessary.
15. Turn timer switch to "START" position. The ten-minute run now begins.
16. Maintain the test gear temperature and speed at the predetermined specified values.
17. Record all pertinent data about midway of the test run.
18. At the end of the ten-minute run, the timer will automatically turn the power off to the drive motor and reduce the load to zero. However, the timer switch will remain in the "START" position, the rpm-adjust will remain in the running position, and the load set point will remain at the previously set load position. Restore these to their respective "OFF" positions.
19. Turn induction heater plate power off.
20. Make visual scuff inspection of each tooth of the narrow test gear as follows:
    a. Turn 3-way valve (Figure 4) to bypass the gear machine and return the test oil to the sump.
    b. Remove corks from inspection holes in the gear-case end cover.
    c. Install microscope or television camera and inspection light.
    d. Focus microscope and adjust grid lines over the contact area of the tooth.
    e. Set indexing-rachet wheel to "No. 1" position.
    f. Estimate the percent scuff of No. 1 tooth as accurately as possible but at least to the nearest 5%, and record the value (see Figure 7).
    g. Operate the test gear rotating device to give No. 2 tooth, and repeat (f). Repeat for No. 3 tooth, etc, until all 28 teeth have been examined.
    h. Remove inspection equipment and replace corks in holes.
    i. Lock test gear rotating device in disengaged position.
    j. Turn 3-way valve to running position.

21. Repeat step 3 of 6.2.2.
22. Repeat steps 7 through 20 at the next test load of 69 kPa gage (10 psig) load-oil pressure.
23. Continue the test at 34.5-kPa gage (5-psi) load-oil pressure increments until the average scuffed area for all 28 teeth is close to 22.5%. At this juncture, visually estimate the percent effective tooth width to the nearest 1%, and record the value.
24. Further continue the test at 34.5-kPa gage (5-psi) load-oil pressure increments, until an approximate average scuffed area of 30% or more is obtained.
25. Push test-oil heater button to "OFF" position.

26. Push support-oil heater button to "OFF" position.
27. Push support-oil pressure button to "OFF" position.
28. Turn radiometer function switch to "READY" position.
29. Switch induction heater power supply to "OFF" position.
30. Turn off water supply to induction heater load coil transformer and induction heating coil.
31. Turn off ventilating blower connected to gear-case end cover.
32. Remove test-oil sump, take 50-mL test-oil sample, and measure the amount of test-oil remaining. Record the amount of test-oil measured plus the 50-mL sample.
33. Disconnect induction heating coil from load coil transformer.
34. Disconnect ventilating blower from gear-case end cover.
35. Remove gear-case end cover.
36. Reverse test gears so that the serial numbers face toward the gear case again matching the timing marks on the shafts and test gears. Check for proper seating of the retaining nuts and lockwashers. After tightening the retaining nuts, bend one tang of each lockwasher to lock the retaining nuts in position.
37. Adjust driven shaft in accordance with 6.2.1, step 11.
38. Rinse face of gear case and test gears with petroleum ether and blow off with compressed air.
39. Rinse inside of gear-case end cover, including the test-oil jet, with petroleum ether and blow off with compressed air.
40. Blow out test-oil jet with compressed air.
41. Reinstall gear-case end cover and connect induction heating coil to load coil transformer.
42. Reinstall ventilating blower to gear-case end cover.
43. Clean test-oil line screen filter with petroleum either. Blow out line screen filter with compressed air.
44. Rinse test-oil sump, lines, pump, and inlet filter with petroleum ether and blow off with compressed air.
45. Reassemble test-oil system.

6.2.2 Test sequence on "B" side of test gears.

1. Turn on water supply to induction heater load coil transformer and induction heating coil.
2. Turn on ventilating blower connected to gear-case end cover.
3. Proceed on "B" side of the test gears by following the procedure outlined in 6.2.1, steps 18 through 29, and 6.2.2, steps 1 through 35.
4. Remove used test gears.
5. Rinse gear-case face and shaft ends with petroleum ether.
6. Rinse gear-case end cover and test-oil jet with petroleum ether.
7. Reinstall gear-case end cover (without test gears).
8. Turn radiometer function switch to "OFF" position.
9. Place induction heater filament switch in "OFF" position.
10. Turn off power supply to induction heater.

11. Turn rpm counter to "OFF" position.
12. Push instruments button "OFF".
13. Turn off main electric switch to test cell.
14. Turn off main water valve to test cell.

6.2.3 Emergency stop.

1. Push emergency stop button.
2. Push instruments button "OFF."
3. Turn off induction heater power supply.
4. Turn off main water valve to test cell.

6.3 Inspection and rating.

6.3.1 Scuffed area. The scuffed area of a gear tooth is that area from which the axial grinding marks have been removed by scratching, scoring, abrasion, and wear. As shown on Figure 7, area A and B are scuffed; area C is not scuffed. Note that only the actual working portion (areas A plus B plus C) of the tooth surface is considered in the definition of areas.

6.3.2 Percent of tooth area scuffed. The percent of tooth area scuffed is that portion of the working surface of a gear tooth that has been scuffed.

Referring to Figure 7,

$$Percentage\ tooth\ area\ scuffed = \frac{scuffed\ area * 100}{working\ area} = \frac{A \pm B}{A \pm B \pm C} * 100$$

The percent of tooth area scuffed is estimated visually to the nearest 5% for each individual tooth, with the aid of a net reticule mounted in the eyepiece of the inspection microscope, or a grid placed over the monitor tube.

6.3.3 Average percent of tooth area scuffed. The average percent of tooth area scuffed is the algebraic average of the percent of scuffed area of all 28 teeth of the narrow test gear. Calculate this value to the nearest 1%.

6.3.4 Effective tooth width. The effective tooth width is the actual width of the narrow-gear tooth that is in contact with the tooth of the wide gear. For test gears currently available, the percentage effective tooth width may be taken as 98% of the gross tooth width by means of a micrometer. See 7.1 for calculation.

6.3.5 Load-carrying capacity. The load-carrying capacity of a lubricant is defined as the gear-tooth load at which the average percent of tooth area scuffed is 22.5% for the narrow test gear as determined by the method outlined herein.

6.3.5.1 Determination of load-carrying capacity. Using semi-log paper (Figure 10), plot the average percent of tooth area scuffed for the narrow test gear versus the load-oil pressure. Determine from the plotted curve the load-oil pressure at which the average percent of tooth area scuffed is 22.5%. Calculate load-carrying capacity, see 7.2.

6.4 Relative rating procedure. This procedure consists of reporting the load-carrying capacity of an oil under test as a percentage of the load-carrying capacity of a standard reference oil obtained using the same test apparatus. Details of this relative rating procedure are given in the following paragraphs:

6.4.1 Determination of reference oil average rating.

NOTE 10: All load-carrying capacity tests on the standard reference oil are conducted with Test #6517, using AMS 6260 steel test gears at 165°F test temperature conditions.

6.4.1.1 For a new rig, at least eight determinations (four gears) shall be conducted on the standard reference oil. If the high and low values obtained do not differ by more than 140 N/m (800 lb/in), the average is taken as the reference oil rating. If the high low values differ by more than 140 N/m (800 lb/in), four additional determinations (two gears) shall be conducted and the aver-age of the 12 determinations taken as the reference oil rating.

6.4.1.2 After a major overhaul to a rig such as a bearing or shaft replacement, four determinations (two gears) shall be conducted on the standard reference oil. If the average of these ratings does not differ from the average obtained prior to the overhaul by more than 52.56 N/m (300 lb/in), and the high and low valves obtained do not differ by more than 140 N/m (800 lb/in), this average will be taken as the reference oil rating. If the ratings obtained do not meet the above criteria, four additional determinations (two gears) shall be conducted and the average of all eight determinations taken as the reference oil rating.

6.4.1.3 At least two determinations (one gear) shall be conducted on the standard reference oil for each 20 determinations made on oils under test. The reference oil rating is a progressive rating in that it consists of the average value obtained on the last eight determinations, i.e., when two new determinations are conducted, the two oldest determinations of the eight determinations used in determining the previous reference oil rating are dropped, the two new ratings replace them and a new reference oil rating are dropped, the two new ratings replace them and a new reference oil average rating is obtained. The sole exception to the above is where it is necessary to run 12 determinations to establish an average on a new rig. (See 6.4.1.1)

NOTE 11: Maximum and minimum acceptable average ratings for the reference oil are 578.1 N/m (3300 lb/in) and 438 N/m (2500 lb/in), respectively. Rigs which do not give an average reference oil rating within these limits shall not be considered as being satisfactory. Faulty maintenance practices, improper test procedures, or the need for major overhaul can be indicated if the average on the reference oil does not fall within these limits.

6.5 Data Sheets

6.5.1 The suggested data sheets are shown on Figures 8 and 9. These sheets have areas provided to enter all pertinent information obtained during test and inspection.

7. CALCULATIONS

7.1 Calculate the effective tooth width using following equation:

$$W_2 = 0.98\, W_1$$

where:

W_2 = effective tooth width in cm (in).
W_1 = measured gross tooth width in cm (in).

7.2 Calculate the load carrying capacity from the following equation:

$$P = \frac{KL}{W_2}$$

where:
P = load-carrying capacity of the lubricant in N/cm (lb/in) of tooth-face width.
K = WADD gear machine constant (11.5 by definition).
L = load-oil pressure Pa gage (psig).
W_2 = effective tooth-face width in cm (in).

8. REPORTING

8.1 Report the following items:

a. The individual ratings of the test oil obtained from the "A" and "B" sides of all gears tested in N/cm (lb/in) of the tooth-face width (6.3.5.1).
b. The average rating of the test oil in N/cm (lb/in) of tooth-face width.
c. The standard reference oil average rating in N/cm (lb/in) of tooth-face width (6.4.1).

$$d.\ \text{The relative rating of the test oil (percent)} = \frac{\text{Test oil average rating}}{\text{reference oil average rating}} * 100$$

9. PRECISION

9.1 Precision data have not been developed for this method.

Method prepared by:

Air Force - AFWAL/POSL - 1986

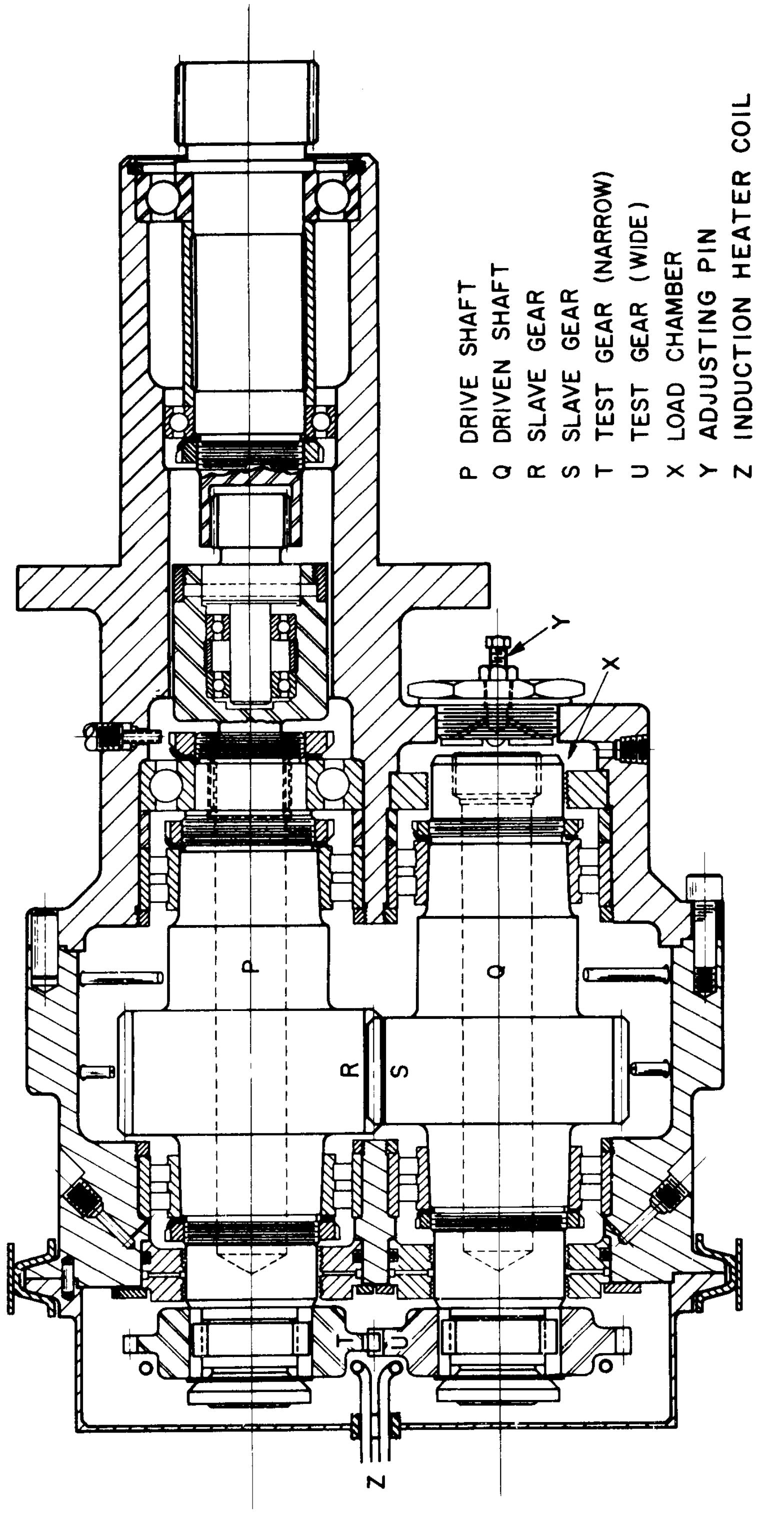


FIGURE I. Cross section of WADD gear machine.

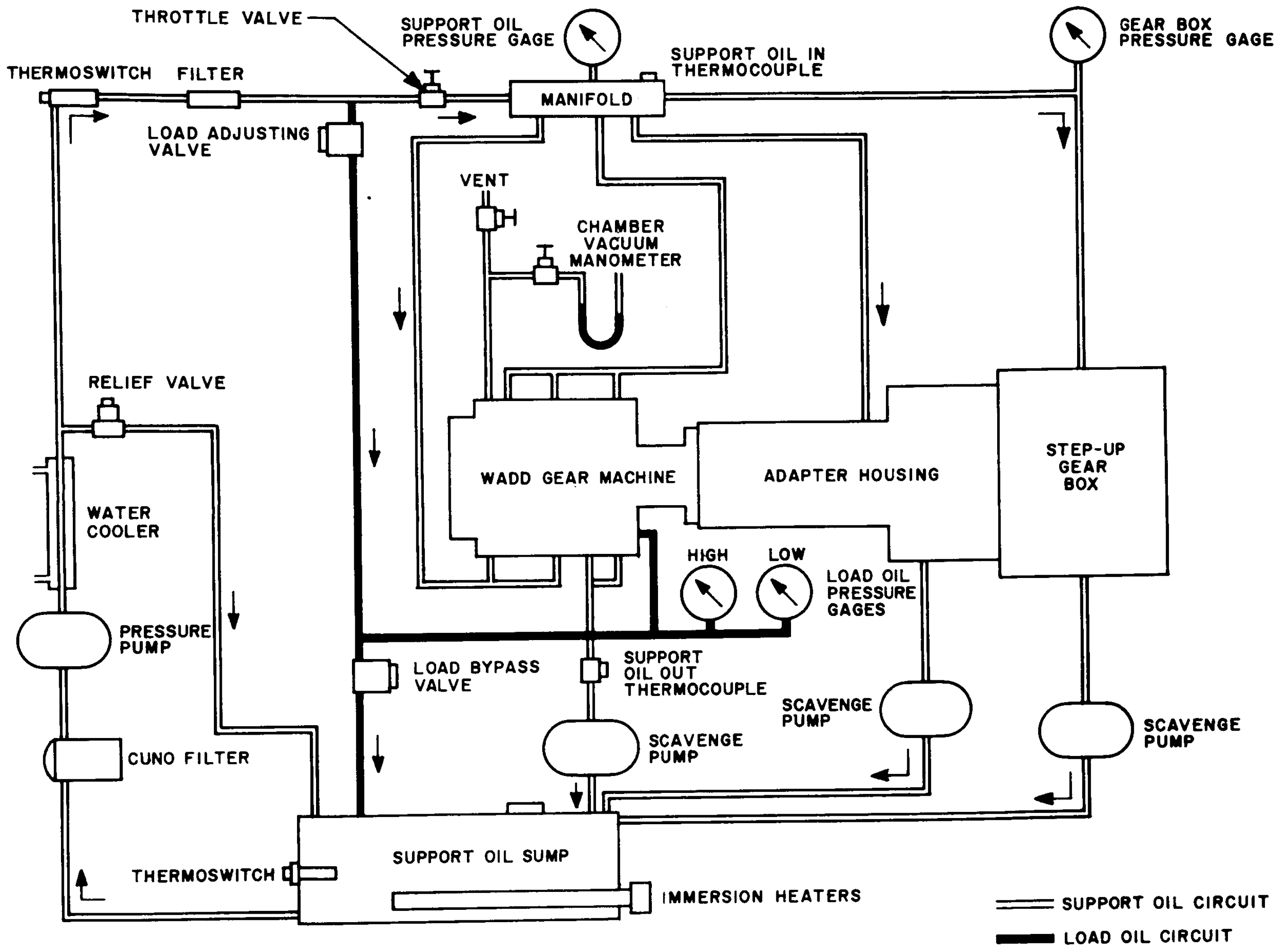


FIGURE 2. Support and load oil system.

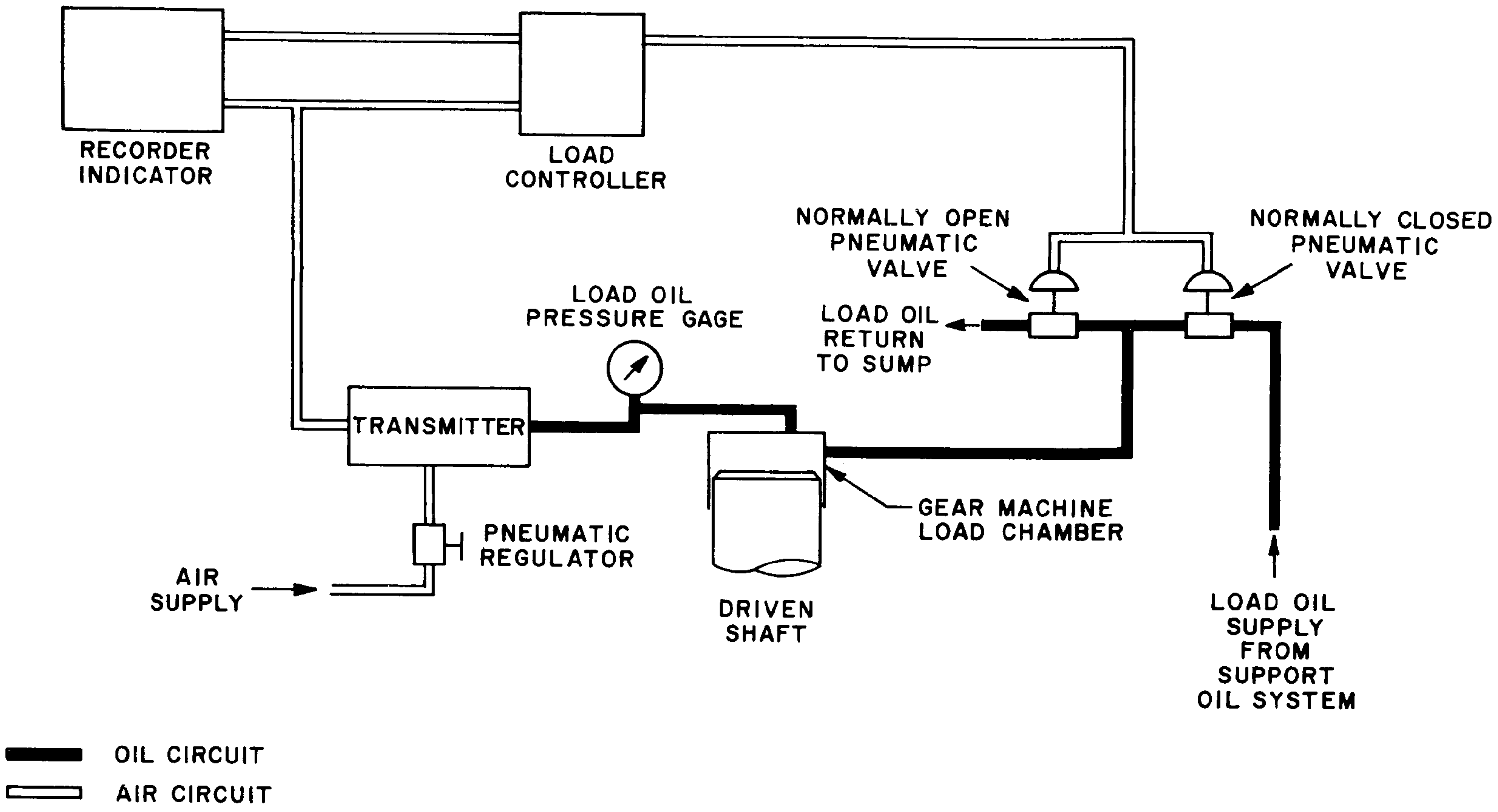


FIGURE 3. Optional automatic-recording load oil system.

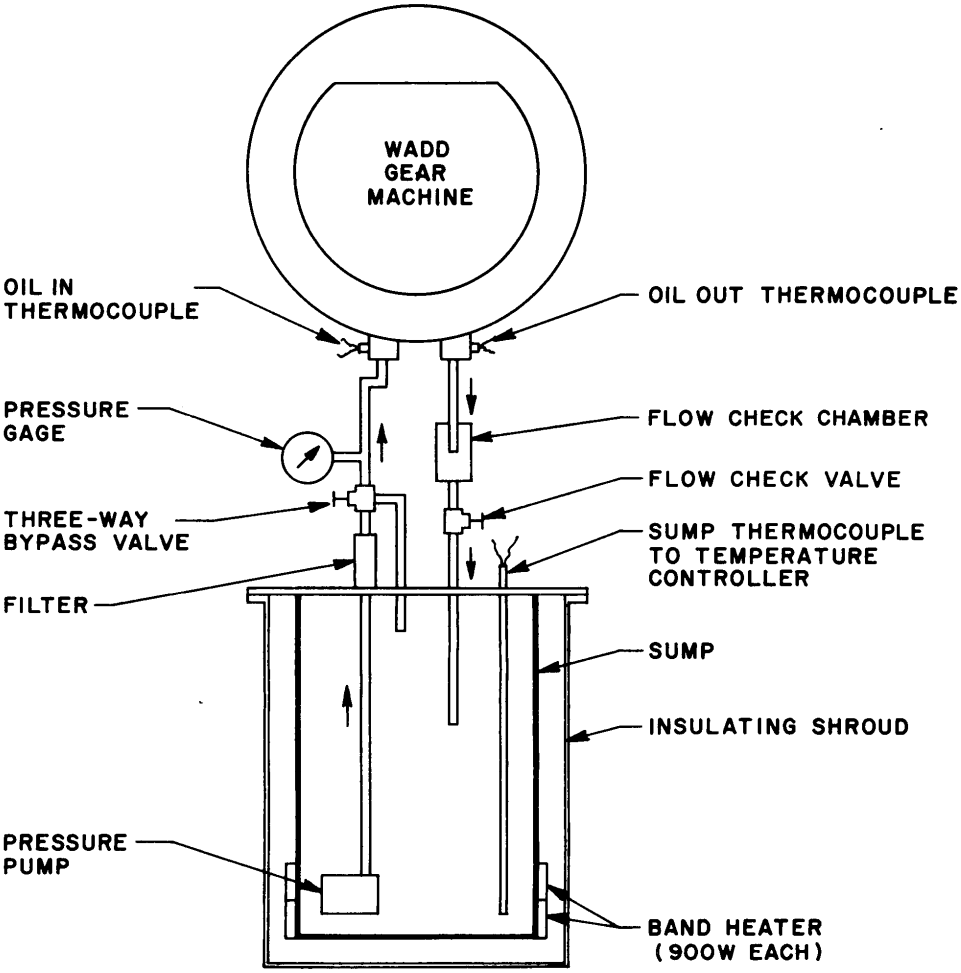
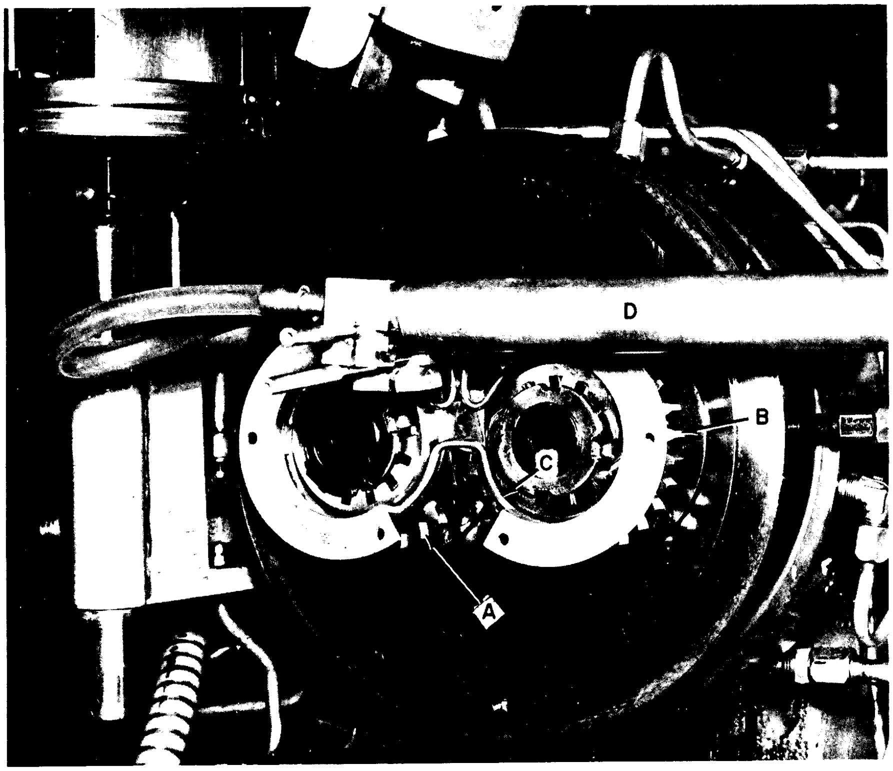
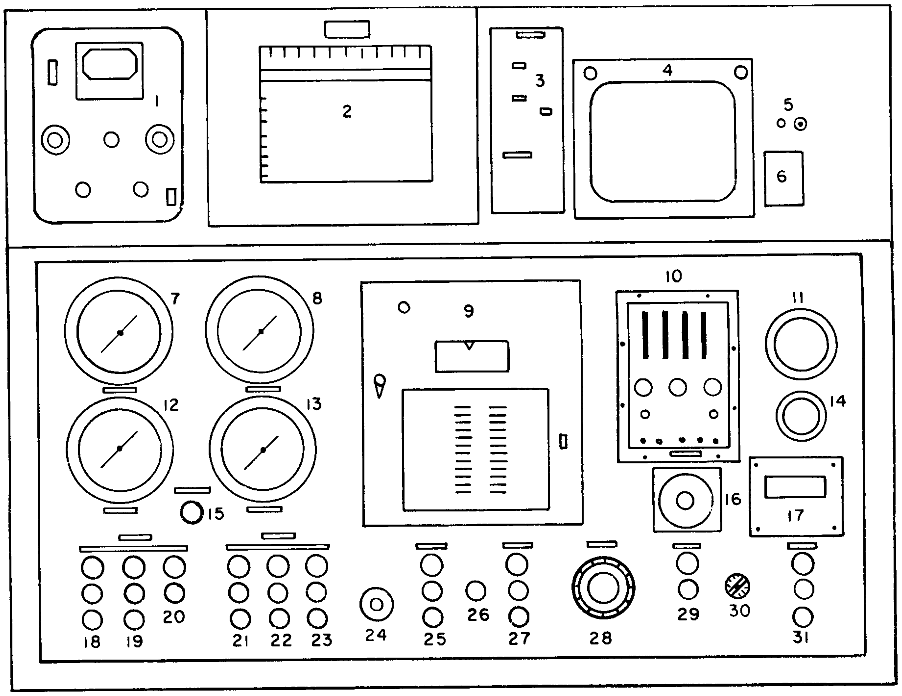


FIGURE 4. Test oil system.



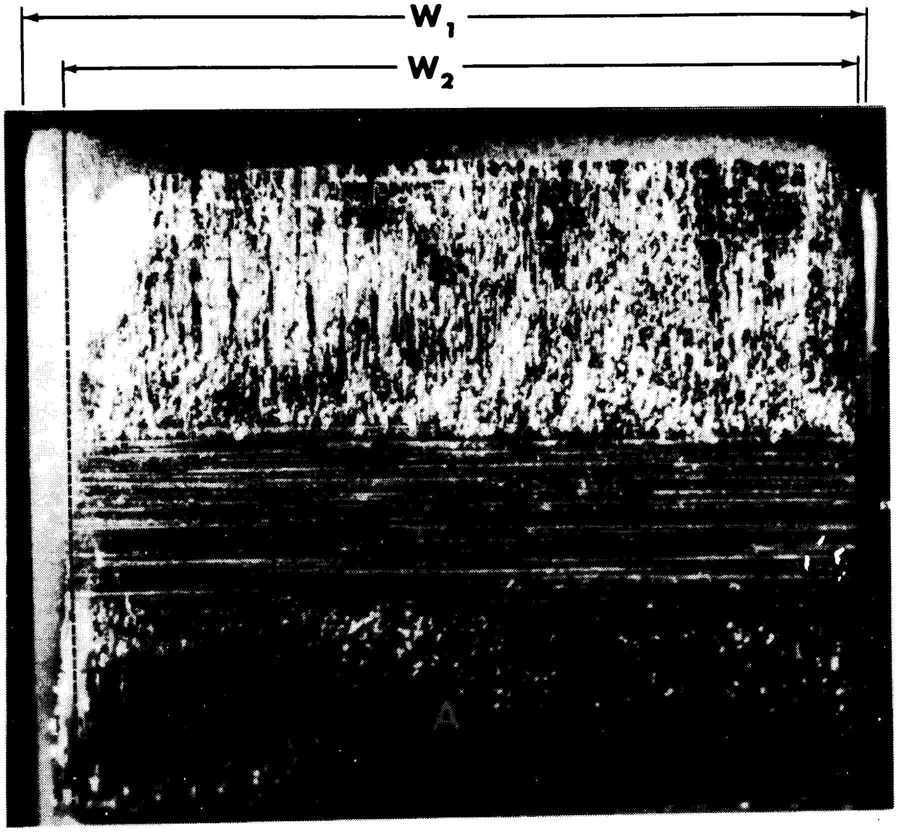
A. Narrow test gear
B. Wide test gear
C. Induction heating coil
D. Connector from load coil transformer

FIGURE 5. WADD gear machine with high-temperature test gears and induction heating coil installed.



1. Radiometer
2. Radiometer output recorder
3. Television monitor controls
4. Television monitor
5. Automatic load-adjust control on-off switch
6. Optional automatic load-adjust control and recorder
7. Support-oil pressure gage
8. Test-oil pressure gage
9. Multi-point temperature indicator
10. Electronic rpm counter
11. Gear-box pressure gage
12. Load-oil pressure gage (high range)
13. Load-oil pressure gage (low range)
14. Running-time meter
15. Standard load-adjust control
16. Electric timer
17. Test-oil sump temperature controller
18. Support-oil pumps start-stop buttons
19. Support-oil heaters on-off buttons
20. Load bypass open-close switch
21. Test-oil pressure pump start-stop buttons
22. Not used
23. Test-oil heaters on-off buttons
24. Air supply valve
25. Instruments on-off buttons
26. Emergency stop button
27. Drive motor start-stop buttons
28. RPM-adjust control
29. Timer start-stop switch
30. Inspection light voltage control
31. Not used

FIGURE 6. WADD gear machine control console.



Area "A" .... Scuffed
Area "B" .... Scuffed
Area "C" .... Not Scuffed

W_1 = Gross tooth width
W_2 = Effective tooth width

Figure 7. Scuffed Tooth (Enlarged)

204°C (400°F) LOAD-CARRYING CAPACITY TEST
OPERATING CONDITIONS DATA SHEET

RIG NO. ____________ MACHINE NO. ____________ TIME METER AT START OF TEST ________ TEST NO. ____________

WIDE GEAR NO. ____________ NARROW GEAR NO. ________ GEAR SIDE ________

RECORD THE FOLLOWING AFTER 5 MINUTES AT EACH LOAD

LOAD GAGE kPa, (PSIG)	34 (5)	69 (10)	103 (15)	138 (20)	172 (25)	207 (30)	241 (35)	276 (40)	310 (45)	345 (50)	379 (55)	414 (60)	448 (65)	483 (70)	517 (75)	551 (80)	586 (85)	620 (90)	655 (95)	689 (100)	723 (105)	758 (110)	792 (115)	827 (120)
TIME																								
S.O. PRESS GAGE kPa, (PSIG)																								
T.O. PRESS GAGE kPa, (PSIG)																								
T.O. "IN" TEMP °C, (°F)																								
S.O. "IN" TEMP °C, (°F)																								
S.O. "OUT" TEMP °C, (°F)																								
T.O. SUMP TEMP °C, (°F)																								
S.O. SUMP TEMP °C, (°F)																								
OPERATOR'S INITIALS																								

TEST OIL FLOW, TIME FOR 270 mL: ____________ SECONDS

REMARKS: ____

____

FIGURE 8. Operating conditions data sheet.

204°C (400° F) LOAD-CARRYING CAPACITY TEST
GEAR TEETH INSPECTION DATA SHEET

TEST OIL CODE ______ DATE ______ WIDE GEAR NO. _____ NARROW GEAR NO. _____ TEST NO. ______

GROSS TOOTH WIDTH ____________ ESTIMATED TOOTH WIDTH __________°/o GEAR SIDE __________

TOOTH NO.	PERCENT OF TOOTH AREA SCUFFED																							
	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100	105	110	115	120
1																								
2																								
3																								
4																								
5																								
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25																								
26																								
27																								
28																								
TOTAL °/o																								
AVE °/o																								

FIGURE 9. Gear teeth inspection data sheet.

204°C(400°F)LOAD CARRYING ABILITY TEST
PLOT AND CALCULATIONS SHEET

OIL CODE ______________ TEST NO. ______________
DATE ______________ ENGINEER ______________

	"A" SIDE	"B" SIDE
EFFECTIVE TOOTH WIDTH, cm (INCHES)	______	______
LOAD OIL PRESSURE AT 22.5% SCUFF, PSI	______	______
LOAD-CARRYING ABILITY, N/m (lb/inch)	______	______

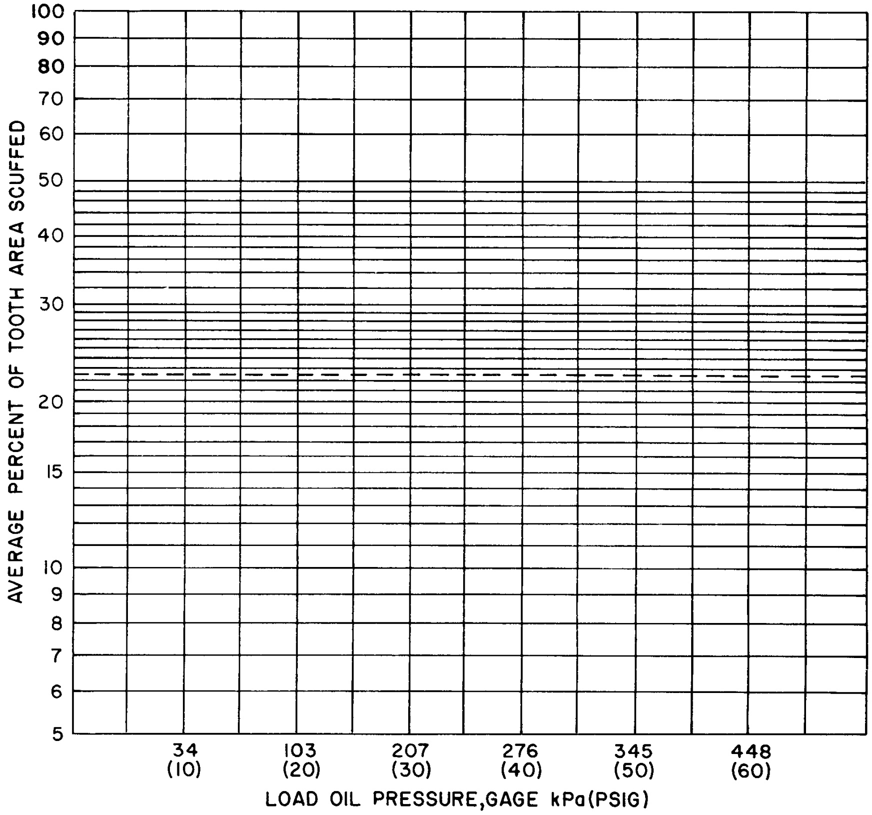


FIGURE 10. Plot and calculations sheet.

Method 7501
August 7 , 2006

LOW TEMPERATURE TORQUE TEST METHOD FOR LUBRICATING GREASES

1. SCOPE

1.1 This test method determines the torque values of lubricating greases using tapered roller bearings when subjected to low temperatures. The method was developed using greases giving torques of less than 80 N·m at -54 °C.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications. The following specification forms a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation (see 6.2).

SPECIFICATIONS

MILITARY

MIL-G-23827 - Grease, Aircraft and Instrument, Gear and Actuator Screw.

(Unless otherwise indicated, copies of federal and military specifications, standards, and handbooks are available from the Naval Publications and Forms Center, (ATTN: NPODS), 5801 Tabor Avenue, Philadelphia, PA 19120-5099, or at http://assist.daps.dla.mil/quicksearch/).

3. SUMMARY

3.1 A worked sample of the test grease is packed into four test specimen assemblies which simulate the automotive front wheel bearing system. These assemblies are mildly heated and then rotated one revolution. The test assemblies are soaked for 16 hours at -54 °C. At the end of this time, the two test assemblies are rotated consecutively at 1 rpm and both breakaway (starting torque) and torque after 5 minutes of rotation (running torque) are then determined. The two remaining test assemblies are tested after the additional four hours cooling to restore the test temperature.

4. SIGNIFICANCE

4.1 This method was developed to assess the low temperature performance of greases at -54 °C and clearly distinguish between differing grease formulations. The torque data obtained from this test method agree with the low temperature performance of military greases in the field. A correlation was found between the breakaway torque and the apparent viscosity range of 5,000 - 30,000 poises at a shear rate of 25 reciprocal seconds.

5. DEFINITIONS

5.1 Breakaway torque. The maximum torque measured at start of rotation.

5.2 Running torque. The torque value after rotation for a specified period of time (5 minutes).

6. APPARATUS

6.1 Low temperature torque test unit. This test unit consists of an ASTM D 4693 test apparatus which is designed with a cold chamber and two spindle drive systems, torque measuring system, test specimen assembly, and data acquisition and control system. The schematic diagram of US Army low temperature torque test unit is shown in figure 1.

6.1.1 The low temperature test chamber consists of a cascade mechanical refrigeration system, a blower, and a solid state electronic temperature controller. This chamber is designed to maintain the test temperature down to -58 °C. The chamber temperature is monitored with a T type thermocouple.

6.1.2 The spindle drive system consists of a 1/3 hp electric motor, a timing belt and pulley, a gear reducer and a solid stainless steel extension shaft (diameter: 2.2 cm, length: 25.6 cm). The gear reducer provides 84 N·m of maximum output torque and approximately 1 rpm of fixed speed. Two spindle drive systems are mounted externally and connected to the inside of the cold chamber using a solid coupling and extension shaft, respectively. These shafts are supported by two sealed roller bearings which are lubricated with MIL-G-23827 grease. Only the outside supporting bearings are rotated with the extension drive shafts during the test time.

6.1.3 The torque measuring system consists of a strain-gage load cell (capacity: 136 kg), a cylindrical rod (diameter: 1.9 cm length: 22.5 cm), a load button and a DC power supply (capacity 20 V). Two torque measuring systems are mounted on a plate which is situated below and external to the cold chamber. To measure the grease torque, rods are connected to the inside of the cold chamber with the load cell. A spherical load button is attached to the top of the rod to allow for the adjustment of the level of the torque arm. A DC power supply is connected to both load cells and applies 10 volts for the input bridge excitation.

6.1.4 The test specimen assembly is as specified in the ASTM D 4693 test method.

6.1.5 The data acquisition and control system consists of a HP data acquisition and control unit, a HP computer as system controller and the user operation program (LOWTEMP). The HP data acquisition and control unit also contains three plug-in assemblies: relay multiplexer assembly for measuring the torque value, relay multiplexer assembly with thermocouple compensation, and high voltage actuator to control the drive system.

6.2 Laboratory oven, gravity convection, 70 ±3 °C.

6.3 Grease packer, as described in the ASTM D 3527 test method.

6.4 Spindle holder or test preparation stand.

6.5 Ultrasonic cleaner.

7. CALIBRATION

7.1 Torque calibration. Attach a support disk (diameter: 4 cm), for holding the dead weight, on the top of the rod. Measure output (millivolt) of load cell as a known force (dead weight: 0.5 kg, 1-10 kg) is applied to load cell at the room temperature. Then, the calibration equation shall be determined using the statistic method. The Method of Regression analysis is very suitable to develop the torque calibration equation. The typical calibration curve is shown in figure 2. This calibration needs be done only at the time of initial setup and when occasional checks indicate that it is required.

7.2 Temperature calibration, as described in ASTM D 4693, or equivalent.

7.3 Spring calibration, as described in ASTM D 4693, or equivalent.

8. TEST BEARINGS.

8.1 Use LM67010-LM67048 and LM11910-LM11949 inboard and outboard bearings, respectively. Timken bearings are suitable. Prior to use in this test, new bearings shall be conditioned by installing any suitable wheel bearing grease in the bearings and running them at room temperature for 48 hours at 1,000 rpm under a normal thrust load of 111 N. ASTM D 3527 test apparatus is suitable for conditioning the bearings.

Note 1. The bearings must be keyed to prevent race rotation on spindle.

9. PROCEDURE

9.1 Clean the test bearings with heptane or equivalent reagent using a ultrasonic cleaner and dry with compressed air.

9.2 Work a grease sample for 60 double strokes using the full scale grease or one-half scale grease worker at room temperature.

9.3 Weigh an inboard and outboard bearing cone to the nearest 0.1g.

9.4 Pack a worked grease sample into the test bearings (inboard: 3.0 ±0.1 g. outboard: 2.0 ±0.1 g) using the following bearing packing procedure: Fill the bearings with the worked test grease using a grease packer utilized in ASTM D 3527 test method. Use care to prevent relative rotation of rollers and bearing components while removing the cones from cups and in all subsequent wiping and handling steps. Strike off excess grease flush using a small spatula. Wipe all grease from cone bore, cone back face, exterior cage surfaces and exposed roller surfaces with a clean lint free cloth or towel and reweigh. Adjust the grease weight in the inboard and outboard cones to 3.0 and 2.0 ±0.1g, by wiping or adding grease to the groove between the cage and cone back face.

9.5 Clean the bearing cups installed in the hub using heptane and apply a thin film of the test grease to the races.

9.6 Set-up a test specimen assembly according to the following procedure: Without any rotation of the test bearing, the inboard bearing is installed on the spindle. Then, this spindle is mounted on the test preparation stand which was designed for holding it in a vertical position. The hub is gently set on the inboard bearing with the torque arm aligned with the spindle set screw. (i.e., pointing in the same direction). Then, the outboard bearing is placed on the hub. The thrust force (400 N) is applied to the test bearings with a calibrated spring-loading device.

9.7 Prepare four test specimen assemblies using the above test assembly preparation procedure.

9.8 Without disturbing the test assemblies, place them in an oven preheated to 70 ±3 °C for 1 hour.

9.9 Rotate the test specimen assemblies for 1 revolution using the test apparatus at room temperature.

9.10 Install two test specimen assemblies on each drive shaft for the first two tests. Without any handling of the test assemblies, the torque arms should be placed vertically with the drive motors.

9.11 Place the other two test assemblies inside the cold chamber in preparation (cold soak) for the next tests.

9.12 Insert two thermocouples (T type) in the test spindles to monitor the test temperature.

9.13 Start 16 hour cold-soak at -54 ±0.5 °C.

9.14 Following a 16 hour cold-soak, the two torque tests shall be consecutively performed using the LOWTEMP computer application program. The following additional test conditions are applied to the torque test runs:

Test temperature: -54 ±0.5 °C
Test time: 5 minutes
Spindle speed: 1 rpm
Data scanning interval: 20 centiseconds
The torque reading starts when torque value is >0.1 N·m

9.15 Store the test results on a floppy disk and plot them on the formatted papers.

9.16 Cut off the cooling system, and remove the tested specimen assemblies from the drive shafts and place them in the preheated oven until completely dried in order to preclude rust formation.

9.17 For the second series of tests, the two remaining test specimen assemblies shall be set up on the drive shafts identically to the previous tests.

9.18 Resume the cooling for the next four hours to restore the test temperature.

9.19 After four hours, two additional torque data shall be generated using the above torque measurement procedure.

10. REPORT

10.1 Breakaway torque (N·m).

10.2 Running torque (N·m) at 5 minutes.

Note 2. Both determinations shall be calculated by the average of two test runs.

11. PRECISION

11.1 Repeatability

Breakaway torque: 10%
Running torque at 5 minutes: 7%

Note 3. Duplicate results by the same operator should be considered suspect if they differ from the mean by more than the above amounts at the 95 percent confidence level.

11.2 Reproducibility has not been established.

Method prepared by

Army – AT - 2006

Test Method 7502
August 7, 2006

SOLVENT CLEANING POWER BY A SOIL TEST METHOD

1. SCOPE

1.1 This test method is used for determining the cleaning power of solvents that have been subjected to soiled mechanical parts. This Appendix is a mandatory part of the specification. The information contained herein is intended for guidance only.

2. REFERENCED DOCUMENTS

2.1 Military Standards:

MIL-PRF-10924, Grease, Automotive, Artillery (GAA)

2.2 Federal Standards:

Federal Test Method Standard 791C, Method 5308.7 Corrosiveness and Oxidation Stability of Light Oils (Metal squares)

3. SUMMARY OF METHOD

3.1 A small amount of grease applied to a steel coupon is immersed in the test solvent and then placed in an ultrasonic cleaner maintained at up to 50 C. The grease is dissolved by the solvent and removed from the steel coupon by the propagation of sonic sound. The solvent power is determined based on the time to completely remove grease from the coupon.

4. SIGNIFICANCE AND USE

4.1 This test method measures the solvency of all types of cleaners and differentiates their cleaning powers.

5. APPARATUS

5.1 Ultrasonic Cleaner

5.2 Beaker, 200ml

5.3 Test specimen assembly

5.3.1 Steel metal square 0.081 cm thick by 2.5 cm square with holding wire (figure 1.). This specimen has been used in Federal Standard Test Method 791C Method 5308.7.

(Note: Paper clips have been successfully used for holding metal square)

5.4 Timer

5.5 Thermometer, having a range of 50 °C.

6. MATERIALS

6.1 Toluene, CP, at least 99 % (Caution- Flammable, vapor harmful)

7. PROCEDURE

7.1 Wash the test assembly with toluene until free from any soil or oil film. Dry the test assembly using laboratory air system. Weight the test assembly to the nearest 0.1 g. Approximately 0.4 g of MIL-PRF-10924 grease is uniformly applied to both sides of the metal coupon. The test assembly is placed in a beaker using a holder. Add the test solvent into the beaker until the test assembly is completely immersed.

7.2 Place the beaker in the ultrasonic cleaner that is maintained water temperatures of 50 C. Start the test using a timer. Observe the test assembly until all grease is removed from metal coupon. Record the time in minutes. If the portion of grease still remains on the metal coupon after 100 minutes, the test is immediately terminated with the testing time being recorded as 100 minutes.

7.3 Repeat two more tests in accordance with 7.1 and 7.2.

8. CALCULATION

8.1 Calculate the cleaning power as follows:

Solvent cleaning power, % = ((100-A)/100) * 100

Where A is average time of three runs obtained from the tests

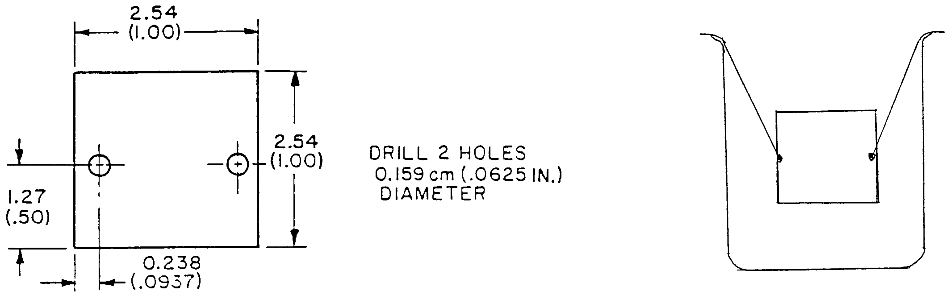
(Note 2. The cleaning power of water is found as zero % while toluene is 92-96 %)

9. REPORT

9.1 Report the calculated solvent cleaning power in percentages to the nearest 0.1.

10. PRECISION

10.1 Precision data have not yet been developed for the method.



(a) Metal square

(b) Test assembly

Figure 1: Metal square dimensions and test assembly

Method prepared by :

Army – AT - 2006

Method 10000
September 30, 1986

MATERIAL HANDLING SAFETY PRECAUTIONS

1. SCOPE

1.1 This reference is used to list all toxic and hazardous material cited in Federal Test Method Standard (FTMS) 791. The relative hazard identification is listed in the material index (4.4). The synonyms, life hazard, flammability, handling and storage precautions emergency treatment and measures, and spill practices of each chemical are explained in the material list (4.5).

2. SUMMARY

2.1 For each material cited in FTMS 791, the first three digits after the material's name indicate the relative hazard identification of health, flammability, and reactivity, respectively. The hazard classification is from the National Fire Protection Agency (NFPA). Refer to the material index (4.4) to locate the safety precautions of each material within this text.

3. SAMPLE

3.1 Acetone - 1, 3, 0 - 002.

3.2 Acetone has a health hazard identification of 1.
Acetone has a flammability hazard identification of 3.
Acetone has a reactivity hazard identification of 0.
The Acetone index is 002.

4. RELATIVE HAZARD IDENTIFICATION

4.1 Health.

4 A few whiffs of the gas or vapor could cause death; or the gas, vapor, or liquid could be fatal on penetrating the fire fighters' normal full protective clothing which is designed for resistance to heat. For most chemicals having a Health 4 rating, the normal full protective clothing available to the average fire department will not provide adequate protection against skin contact with these materials. Only special protective clothing designed to protect against the specific hazard should be worn.

3 Materials extremely hazardous to health, but areas may be entered with extreme care. Full protective clothing, including self-contained breathing apparatus,

impermeable rubber gloves, boots and bands around legs, arms and waist should be provided. No skin surface should be exposed.

2 Materials hazardous to health, but areas may be entered freely with self-contained breathing apparatus.

1 Materials only slightly hazardous to health. It may be desirable to wear self-contained breathing apparatus.

0 Materials which on exposure under fire conditions would offer no health hazard beyond that of ordinary combustible material.

U Unknown.

4.2 Flammability.

4 Very flammable gases, very volatile flammable liquids, and materials that in the form of dusts or mists readily form explosive mixtures when dispersed in air. Shut off flow of gas or liquid and keep cooling water streams on exposed tanks or containers. Use water spray carefully in the vicinity of dusts so as not to create dust clouds.

3 Liquids which can be ignited under almost all normal temperature conditions. Water may be ineffective on these liquids because of their low flash points. Solids which form coarse dusts, solids in shredded or fibrous form that create flash fires, solids that burn rapidly, usually because they contain their own oxygen, and any material that ignites spontaneously at normal temperatures in air.

2 Liquids which must be moderately heated before ignition will occur and solids that readily give off flammable vapors. Water spray may be used to extinguish the fire because the material can be cooled to below its flash point.

1 Materials that must be preheated before ignition can occur. Water may cause frothing of liquids with this flammability rating number if it gets below the surface of the liquid and turns to steam. However, water spray gently applied to the surface will cause a frothing which will extinguish the fire. Most combustible solids have a flammability rating of 1.

0 Materials that will not burn.

U Unknown.

4.3 Reactivity.

4 Materials which in themselves are readily capable of detonation or of explosive decomposition or explosive reaction at normal temperatures and pressures. Includes materials which are sensitive to mechanical or localized thermal shock. If a chemical with this hazard rating is in an advanced or massive fire, the area should be evacuated

3 Materials which in themselves are capable of detonation or of explosive decomposition or of explosive reaction but which require a strong initiating source or which must be heated under confinement before initiation. Includes materials which are sensitive to thermal or mechanical shock at elevated temperatures and pressures or which react explosively with water without requiring heat or confinement. Fire fighting should be done from an explosion-resistant location.

2 Materials which in themselves are normally unstable and readily undergo violent chemical change but do not detonate. Includes materials which can undergo chemical change with rapid release of energy at normal temperatures and pressures or which can undergo violent chemical change at elevated temperatures and pressures. Also includes those materials which may react violently with water or which may form potentially explosive mixtures with water. In advanced or massive fires, fire fighting should be done from a protected location.

1 Materials which in themselves are normally stable but which may become unstable at elevated temperatures and pressures or which may react with water with some release of energy but not violently. Caution must be used in approaching the fire and applying water.

0 Materials which are normally stable even under fire exposure conditions and which are not reactive with water. Normal fire fighting procedures may be used.

U Unknown.

4.4 Material Index

Index Number	Compound=s Name	Health Hazard	Flammability Hazard	Reactivity Hazard
001	Acetic Acid	2	2	1
002	Acetone	1	3	0
003	Ammonium Acetate		0	
004	Ammonium Hydroxide		0	
005	Ammonium Nitrate	0	1	3
006	Barium Chloride	0	0	2
007	Barium Hydroxide			
008	Benzoic Acid			

009	Bromine	4	0	0
010	*n*-Butyl Alcohol	1	3	0
011	Butyric Acid	2	2	0
012	Capric Acid			
013	Caproic Acid	2	1	0
014	Caprylic acid			
015	Chloroform	2	2	0
016	Cleaning Compound Special			
	Cleaning Solution, Glass	See Sulfuric Acid		
017	Detergent, Liquid, Water Soluble, Comm. Grade			
018	Diesel Fuels	0	2	0
019	Diethylene Glycol	1	1	0
020	Diethylene Glycol Monomethyl Ether			
021	Diethylene Glycol Adipate			
022	Diphenylamine	3	1	0
023	Drycleaning Solvent	0	2	0
	Eschka=s Mixture	See Magnesium Oxide, Sodium Carbonate		
024	Ethyl Alcohol	0	3	0
025	Ethyl Ether	2	4	1
026	Ethylene Glycol	0	0	0
027	Ethylene Glycol Monomethyl Ether			
028	Ferrous Ammonium Sulfate			
	Freon-12	See 1,1,2-Trichlorotrifluoroethane		
029	Gasoline	1	3	0
	Glacial Acetic Acid	See Acetic Acid		
030	Glycerine	1	1	0
031	*n*-Heptane	1	3	0
032	Heptylic Acid			
033	*n*-Hexane	1	3	0
034	Hydrochloric Acid	3	0	0
035	Isooctane	0	3	0
036	Isopropyl Alcohol	1	3	0
037	Kerosene			
038	Lead Acetate			
039	Magnesium Oxide			
040	Mercuric Iodide			
041	Methyl Alcohol	1	3	0

	Methyl-Alcohol-Glycerine	See Methyl Alcohol and Glycerine		
042	Methyl Ethyl Ketone	1	3	0
043	Methyl Orange			
044	Naphtha	1	4	0
045	Neopentyl Polyol Ester			
046	Nitric Acid	2	0	0
	Nitric-Sulfuric Acid Mixture	See Sulfuric Acid		
047	NO-Chromix Cleaning Solution			
048	Oleic Acid	0	1	0
049	Pelargonic Acid			
	Petroleum Ether	See Naphtha		
050	1,10-Phenanthroline			
051	Phenolphthalein			
052	Phosphoric Acid	2	0	0
053	Potassium Carbonate			
054	Potassium Chlorate	1	0	2
055	Potassium Dichromate	1	0	1
056	Potassium Hydroxide	3	0	1
057	Potassium Hydrogen Phthalate			
058	Potassium Iodide			
059	Sebabic	0	0	0
060	Silicon Sulfate			
061	Soda Lime			
062	Sodium Carbonate			
063	Sodium Chloride	0	0	2
064	Sodium Dichromate	1	0	1
065	Sodium Hydroxide	3	0	1
066	Sodium Sulfate			
067	Sodium Thiosulfate			
068	Sulfuric Acid	3	0	2
069	Sulfurous Acid			
070	Tannic Acid	0	1	0
071	Teflon			
072	Tetrachloroethylene	2	0	0
073	Tetra Ethyl Lead	3	2	3
074	Toluene	2	3	0
075	1,1,1-Trichloroethane	2	1	0
076	1,1,2-Trichloro-1,2,2-Trifluoroethane			
077	Valeric Acid			
078	Xylene	2	3	0
079	Barium Carbonate			
080	Chromium Trioxide			

081 082	Nickelous Chloride Silver Nitrate			

4.5 Material List

001 **Acetic acid** CH_3COOH

Synonyms: Ethanoic acid, Vinegar acid, Methanecarboxylic acid.

Life Hazard: May produce severe acid burns to skin and eyes. Prolonged breathing of concentrated vapor may be harmful. Threshold limit value, l0 parts per million.

Flammability: Flash point 43°C (open cup), 40°C (closed cup); ignition temperature 427°C; flammable limits 4-16%. Vapors form explosive mixtures with air. Dangerous in contact with chromic acid, sodium peroxide, nitric acid, or other oxidizing materials.

Handling: Separate from oxidizing materials and avoid storage near combustible materials. Expands upon freezing. Wear chemical goggles, chemical cartridge respirator, rubber protective clothing.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilation.
b. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. Call a physician.
c. Fire Precautions (In case of outbreak): Use water spray, dry chemical, alcohol foam or carbon dioxide. Use water to keep fire-exposed containers cool.

Spills: Cover with soda ash or sodium bicarbonate. Mix and add water. Neutralize and drain into the sewer with sufficient water.

002 **Acetone** CH_3COCH_3

Synonyms: Dimethylketone, Beta-Ketopropane, 2-Propanone, Pyroacetic ether.

Life Hazard: Acetone is narcotic in high concentrations. Dangerous, due to fire and explosion hazard; can react vigorously with oxidizing materials.

Flammability: Flammable; flash point -9.4°C (open cup), -20°C; ignition temperature 537.8°C; flammable limits 2.6-12.8 volume percent.

Handling: Keep away from plastic eyeglass frames, jewelry, pens, pencils, and rayon garments. Wear rubber gloves, rubber aprons, chemical goggles, and self-contained breathing apparatus.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilation.
b. First Aid:Irrigate eyes with water. Wash contaminated skin with soap and water. Call a physician if swallowed.
c. Fire Precautions (In case of outbreak): Use dry chemical, carbon dioxide, or alcohol foam. Water spray may be ineffective, but water should be used to keep fire-exposed containers cool.

Spills: Absorb on paper. Evaporate on a glass or iron dish. Burn the paper.

003 **Ammonium acetate** $(CH_3COO)NH_3$

Synonyms: N/A

Life Hazard: Ammonium acetate tends to lose ammonia upon standing.

Flammability: Not flammable. Irritating gases may be produced when heated.

Handling: N/A

Emergency Treatment and Measures:

a. Hygienic Precautions: Avoid inhalation of dust.
b. First Aid: If swallowed and victim is conscious, have victim drink water or milk. If swallowed and victim is unconscious or having convulsions, keep victim warm and nothing else. In both cases call a physician.

Spills: Wash down the drain with excess water.

NH_4OH

Synonyms: Water of ammonia, Ammonium hydrate, Aqua ammonias, Aqua ammonium.

Life Hazard: Dangerous; emits irritating fumes and liquid can inflict burns. Hazardous reactions with inorganic acids immediately boil and are dangerous.

Flammability: Not flammable.

Handling: Bottles with glass, plastic or rubber plug must not be filled completely to the brim and must be stored in cool place. Wear rubber gloves, large face shield and general purpose gas mask.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilation. Preclude from exposure those individuals with eye and pulmonary diseases.

b. First Aid:Irrigate eyes with water. Wash contaminated areas of body with soap and water. Supply oxygen, with use of intermittent positive pressure breathing apparatus. Call a physician.

Spills: Neutralize with dilute sulfuric acid. Wipe with mop or use water aspirator. Drain into the sewer with sufficient water.

005 **Ammonium nitrate** NH_4NO_3

Synonyms: Nitric acid, Ammonium salt, Nitram.

Life Hazard: The oxides-of-nitrogen gases (except nitrous oxide - N_2O) emitted on decomposition of ammonium nitrate are extremely toxic. Any person exposed to such fumes should receive immediate medical attention. Hazardous reactions: ignites by heating its mixture with combustible materials; ignites in the presence of zinc powder and water; explodes by impacting its mixture with ammonium sulfate.

Flammability: Oxidizing agent; supports combustion and is capable of undergoing detonation if heated under confinement or if subjected to strong shocks. Melts at about 150°C with slow decomposition; near 300°C, the decomposition accelerates with sudden rushes of light-brown to orange-copper fumes, indicating that the decomposition may become explosive. Self-ignition of mixtures of ammonium nitrate with easily oxidizable organic materials or easily oxidizable, finely divided metals may take place at moderately elevated temperatures.

Handling: Separate from all organic materials or other contaminating substances such as flammable liquids, acids, corrosive liquids, organic chemicals, chlorates, sulfur and finely divided metals or charcoal, coke, and sawdust. Wear rubber gloves and chemical goggles.

Emergency: Treatment and Measures:

a. First Aid: When exposed to nitrogen oxides due to decomposition at high temperatures, individuals should be promptly given treatment by physician.
b. Fire Precautions (In case of outbreak): Flood with sufficient amount of water. Exercise extreme caution in application of water on fused or molten material to avoid eruptive scattering and spread of fire.

Spills: Sweep into a beaker. Dilute with sufficient water. Add soda ash. Mix and neutralize with dilute acid. Drain into the sewer with abundant water.

006 **Barium chloride** $BaCl_2 \cdot 2H_2O$

Synonyms: Barium chloride dihydrate.

Life Hazard: Highly toxic. Yields toxic fumes when involved in fire. Threshold limit value of dust, 0.5 milligrams per cubic meter.

Flammability: N/A

Handling: Wear rubber gloves.

Emergency Treatment and Measures:
a. Hygenic Precautions: N/A
b. First Aid: If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): N/A

Spills: Cover any spills with sufficient amount of sodium bicarbonate. Remove the mixture into a container such as a fiber drum, plastic bag or carton box for easy disposal in an incinerator.

007 **Barium Hydroxide** $Ba(OH)_2$

Synonyms: Barium hydrate, caustic baryta.

Life Hazards: Highly toxic by ingestion.

Flammability: N/A

Handling: Keep containers closed and store. Wear rubber gloves, safety glasses, and working clothes.

Emergency Treatment and Measures:
a. Hygienic Precautions: Adequate ventilation, protectors. Preclude from exposure those individuals with pulmonary diseases
b. First Aid: Irrigate eyes with water: wash skin with soap and water. If swallowed, call a physician.
c. Fire Precautions (In case of outbreak):

Spills: To a solution of the product in water, add an excess of dilute sulfuric acid. Let stand overnight. Filter to separate the precipitate and filtrate. Add the precipitate slowly to excess ice located in the hood behind a safety shield. The resulting mixture should then be neutralized and washed down the drain with excess water. The filtrate should be washed down the drain with excess water.

008 **Benzoic Acid** C_6H_5COOH

Synonyms: Phenylformic acid, benzenecarboxylic acid, carboxylbenzene.

Life Hazard: Low toxicity. Dust may be irritating to nose and eyes. At elevated temperatures, fumes may cause irritation of eyes, respiratory system, and skin.

Flammability: Flashpoint (121°C), autoignition temperature (574°C).

Handling: Wear rubber gloves, a mask, coveralls, a body shield, and self-contained breathing apparatus.

Emergency Treatment and Measure:
a. Hygienic Precautions: N/A
b. First Aid: Flush affected areas with plenty of water. If in eyes, hold open and flush wit plenty of water. If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): Use water, carbon dioxide, or dry chemical fire extinguisher.

Spills: Carefully mix the acidic compound with dry sodium bicarbonate. Dilute slowly with water and wash down the drain with excess water.

009 **Bromine** Br_2

Synonyms: N/A

Life Hazard: Highly dangerous; when heated, it emits highly toxic fumes; it will react with water or steam to produce toxic and corrosive fumes; and it can react vigorously with reducing materials.

Hazardous reactions with:
a. Hydrogen, methane, ethylene, sulfur, antimony, arsenic phosphor, sodium, potassium and other metallic powder: ignition and explosion.
b. Combustible materials and metallic powder: explosion by contact.

Flammability: Nonflammable, but a strong oxidizing material and reactive with many substances. Heat of reaction may cause ignition of combustible materials.

Handling: Store in cool and dry areas out of direct sunlight. Separate from combustible, organic or other readily oxidizable materials. Keep above -6.7°C to prevent freezing but avoid heating above atmospheric temperatures as vapor pressure increase could rupture the containers. Wear rubber gloves, face shield, coveralls and self-contained breathing apparatus.

Emergency Treatment and Measures:
a. Hygienic Precautions: Adequate ventilation. Preclude from exposure those individuals with pulmonary diseases.
b. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. Burns may be treated with a sodium bicarbonate paste. If swallowed, call a physician.

c. Fire Precautions (In case of outbreak): Use large amounts of water to cool containers and wash away spills.

Spills: Cover with weak reducing agents such as hypo, bisulfites or ferrous salts. Bisulfites or ferrous salts need additional promoter of some 3M-H2SO4 to accelerate reaction. Transfer the slurry (or sludge) into a large container of water and neutralize with soda ash. Drain into a sewer with abundant water.

010 ***n*-Butyl Alcohol** $CH_3(CH_2)_2CH_2OH$

Synonyms: 1-Butanol; n-Butanol.

Life Hazard: Moderately dangerous because of its low volatility; when heated to decomposition it emits toxic fumes; it can react with oxidizing materials.

Flammability: Moderate fire risk. Flash point (open cup) 45.5°C, (closed cup) 28.9°C; ignition temp 365°C; flammable limits 1.4-11.2%. Less reactive; no adverse reaction takes place on fire.

Handling & Storage: Store in cool, unlighted place away from sources of ignition, and from explosives, oxidizing materials, organic peroxides, poisons and radioactive materials. Preferably provide electrical equipment with spark-resistant construction. Wear safety glasses and rubber protective clothing.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilations.
b. First Aid: Flush eyes thoroughly with water. Wash out contaminated skin with soap and water. If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): Use dry chemical, or carbon dioxide: alcohol foam may be ineffective. Water spray may help to keep containers cool, to dilute leaked butanol, and to diffuse vapors.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper. Large quantities should be burned in a solvent burner.

011 **Butyric Acid** $CH_3(CH_2)_2COOH$

Synonyms: Butanic Acid, Butanoil Acid, Ethylacetic Acid, Propanecarbonic Acid.

Life Hazard: Inhalation causes irritation of mucous membranes and respiratory track; may cause nausea and vomiting. Ingestion causes irritation of mouth and stomach. Contact with eyes can cause serious injury. Contact with skin may cause burns; chemical is readily absorbed through the skin and may cause damage by this route.

Flammability: Flash point (76°C) Autoignition temperature (450°C), flammability limits in air (2.19-13.9%). Handling: May attack aluminum or other light metals with the formation of flammable hydrogen gas. Protect containers against physical damage. Store in a cool, dry, well-ventilated place away from sources of ignition and heat.

Emergency Treatment and Measure:

a. Hygienic precautions: N/A
b. First Aid: If inhaled: remove victim to fresh air, give oxygen if breathing is difficult; call a physician. If ingested, give large amounts of water and induce vomiting, call a physician. If eye contact: irrigate with water for 15 min and seek medical attention. Skin contact: flush affected area immediately and thoroughly with water.
c. Fire Precautions: (In case of outbreak): Use dry chemical, "alcohol" foam, or carbon dioxide fire extinguisher. Water may not be effective.

Spills: Carefully mix the acidic compound with dry sodium bicarbonate. Dilute slowly with water and wash down the drain with excess water.

012 **Capric Acid** $CH_3(CH_2)_8COOH$

Synonyms: Decanoic Acid, Decoic Acid, Decylic Acid.

Life Hazard: Moderately toxic.

Flammability: Flashpoint (112°C)

Emergency Treatment and Measures:
a. Hygienic Precautions: N/A
b. First Aid: N/A
c. Fire Precautions (In case of outbreak): N/A

Spills: Carefully mix the acidic compound with dry sodium bicarbonate. Dilute slowly with water and wash down the drain with excess water.

013 **Caproic Acid** $C_6H_{12}O_2$

Synonyms: Hexanoic Acid.

Life Hazard: N/A Flammability: Flash point (104°C).

Handling: A corrosive material.

Emergency Treatment and Measures:

a. Hygienic Precautions: N/A

b. First Aid: N/A
c. Fire Precautions (In case of outbreak): Use foam, carbon dioxide, or dry chemical fire extinguisher.

Spills: Carefully mix the acidic compound with dry sodium bicarbonate. Dilute slowly with water and wash down the drain with excess water.

0l4 **Caprylic Acid** $CH_3(CH_2)CH=CHCOOH$

Synonyms: Octanoic Acid.

Life Hazard: Low toxicity, yields irritability vapors which could cause coughing.

Flammability: Flash point 270°F, open cup.

Handling: N/A

Emergency Treatment and Measures:
a. Hygienic Precaution: N/A
b. First Aid: N/A
c. Fire Precautions (in case of outbreak): N/A

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper. Large qualities should be burned in a solvent burner.

0l5 **Chloroform** $CHCl_3$

Synonyms: Trichloromethane

Life Hazard: Prolonged exposure will induce anesthesia. Poisonous and irritating gases are produced when heated. Suspected carcinogen.

Flammability: Not flammable, but will burn on prolonged exposure to flame or high temperature.

Handling: Protect containers against physical damage. Keep containers closed and store in a dark place away from direct sunlight and moisture. Wear safety glasses, self-contained breathing apparatus, protective clothing and rubber gloves.

Emergency Treatment and Measures:

a. Precaution: Adequate ventilation. Physical examination annually to study liver and kidney functions. Preclude from exposure individuals with diseases of liver, kidney and central nervous system.
b. First Aid: If it has been ingested, or there has been great over-exposure, call a physician.

c. Fire Precaution (in case of outbreak): N/A.

Spills: Compound should be redistilled or otherwise repurified and reused.

016 Cleaning Compound, Special

Synonyms: Penetone ECS.

Life Hazard: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures: N/A

Spills: N/A

017 Detergent, Liquid, Water Soluble, Commercial Grade

Synonyms: N/A

Life Hazard: N/A

Handling: N/A

Emergency Treatment and Measures: N/A

Spills: Wash down the drain with excess water.

018 Diesel Fuels

Synonyms: Fuel Oil, Diesel Oil.

Life Hazard Vapors are considered to be moderately poisonous. Dangerous: when exposed to heat or flame; can react vigorously with oxidizing materials.

Flammability: Flash point (greater than 38°C). Keep away from open flame.

Handling: Keep bottles, cans and drums closed and avoid direct sunlight. No fire. Outdoor or detached storage is preferred. For indoor storage, use standard combustible liquid storage rooms or cabinets. Wear goggles, rubber gloves, a chemical cartridge respirator and coveralls.

Emergency Treatment and Measures:

a. First Aid: Irrigate eyes with running water. Wash contaminated areas of body with soap and water. Administer oxygen, if indicated. If swallowed, call a physician.
b. Fire Precautions (In case of outbreak): Use carbon dioxide, dry chemical, or "alcohol" foam. Water spray is not effective for extinguishing, but effective to keep fire-exposed containers cool. If a leak or spill has not ignited, use water to disperse the vapor and to protect workers attempting to stop the leakage.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper. Large quantities should be burned in solvent.

019 **Diethylene Glycol** $(CHOCH_2)_2O$

Synonyms: Diglycol, DEG, 2,2 - oxybiethanol, 2-Hydroxyethyl ether.

Life Hazard: Suspected carcinogen of bladder.

Flammability: Flashpoint (l43°C), autoignition temperature (229°C) flammable limits in air (1.6-10.8%).

Handling: N/A

Emergency Treatment and Measures:

a. Hygienic precautions: No eating in work areas. Preclude from exposure those individuals with diseases of liver, kidneys or nervous system.
b. First Aid: Ingestion: Induce vomiting, no known antidote - treat symptomatically. Eye and skin contact: flush with water. Call a physician.
c. Fire precautions (in case of outbreak): Use "alcohol" foam carbon dioxide, or dry chemical fire extinguisher. Water and foam may be ineffective.

Spills: Wash down the drain with excess water or dissolve in benzene, naphtha or higher alcohol such as butanol, then dispose by burning the solution in a hood.

020 **Diethylene Glycol Monomethyl Ethane**

Synonyms:

Life Hazards:

Flammability:

Handling:

Emergency Treatment and Measures:

a. Hygienic Precautions.

b. First Aid:
c. Fire Precaution (in case of outbreak):

Spills:

021 **Diethyl glycol adipate**

Synonyms: N/A

Life Hazards: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:

a. Hygienic Precautions: N/A
b. First Aid: N/A

c. Fire Precautions (In case of outbreak): N/A

Spills: N/A

022 **Diphenylamine**

Synonyms: Phenylaniline, DPA, anilinobenzene, N-phenylaniline.

Life Hazard: Dangerous: When heated to decomposition, it emits toxic fumes; can react with oxidizing materials.

Flammability: Flash point 153°C; ignition temperature 635°C.

Handling: Store in a cool and dark place. Wear safety glasses, rubber gloves, rubber clothing.

Emergency Treatment and Measures:
a. Hygienic Precautions: Adequate ventilation.
b. First Aid: Flush eyes with water. Wash body with soap and water. Remove and dry clothing, then wash with soap or scrap. Burn shoes in an incinerator. If swallowed, call a physician.
c. Fire precautions (in case of outbreak): Use foam, dry chemical, or carbon dioxide fire extinguishers.

Spills: Spread over with the 9:1 mixture of sand and soda ash. After mixing, transfer into a paper carton, stuffed with ruffled paper. Burn in an open furnace with the utmost care or in the furnace with afterburner and scrubber.

023 **Dry cleaning Solvent**

Synonyms: Stoddard Solvent, Drycleaner Naphtha, Spotting, Naphtha Petroleum Solvent.

Life Hazard: High concentration of vapors may cause intoxication. If liquid is swallowed, it may get into lungs by aspiration, not very irritating to skin or eyes.

Flammability: Flash point (43 ° C), Autoignition temperature (382 ° C) Flammable limits in air (0.8-5.0 %)

Emergency Treatments and Measures:

a. Hygenic Precautions: Adequate ventilation.

b. First Aid: Inhalation: remove patient from exposure, treat symptoms. Ingestion: Do not induce vomiting, call a physician. Eyes: Flush with water for 15 min. Skin: Wipe off and wash with soap and water.

c. Fire Precautions (In case of outbreak): Use foam, dry chemical, or carbon dioxide fire extinguisher.

Spills: N/A

024 **Ethyl alcohol** C_2H_5OH

Synonym: Ethanol, Grain Alcohol.
Life Hazard: Dangerous, when exposed to heat or flame.

Flammability: Flash point 12.2°C (closed cup), 15.8°C (open cup); ignition temperature 426°C; flammable limits 3.3-19%.

Handling: Store away from all possible ignition sources. All precautions for the elimination of static electricity should be taken. Should not be stored with perchlorates, peroxides, chromic acid and nitric acid. Protect containers against physical damage. Store indoors in a cool and well ventilated room, limiting the quantity of sealed drums or containers at any one time.

Emergency Treatment and Measures:

a. Hygienic Precautions: Physical examinations of exposed patient periodically including signs of chronic irritation of mucous membranes and chronic alcoholism.
b. First Aid: Irrigate eyes with water. If swallowed, call a physician.

c. Fire Precautions (In case of outbreak): Use dry chemical, alcohol foam or carbon dioxide. Water spray may be ineffective as an extinguishing agent, but water should be used to keep fire-exposed containers cool.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper.

025 **Ethyl Ether** $C_2H_5OC_2H_5$

Synonyms: Sulfuric ether, Anesthesia Ether, Ethyl Oxide.

Life Hazard: A depressant of the central nervous system and is capable of producing intoxication, drowsiness, stupor, and unconsciousness.

Flammability: Flash point (C-40°C), Autoignition temperature (180°C), flammable limits in air (1.85-36.50%).
Handling : N/A

Emergency Treatments and Measures:
a. Hygienic Precautions: Adequate ventilation.
b. First Aid: Inhalation: remove victim to fresh air; if breathing has stopped, apply artificial respiration, call a physician. Eye contact: Flush immediately with water for 15 min. c. Fire precautions (In case of outbreak): Use alcohol foam, carbon dioxide, or dry chemical fire extinguisher.

Spills: Absorb on paper. Burn, but exert extra care in ignition as this material highly flammable.

026 **Ethylene Glycol** $HO(CH_2)_2OH$

Synonyms:
Life Hazard:

Flammability:

Handling:

Emergency Treatment and Measures:
a. Hygienic Precautions:
b. First Aid:
c. Fire Precautions (in case of outbreak):

Spills:

027 **Ethylene Glycol Monomethyl Ether** $CH_3O(CH_2)_2OH$

Synonyms: 2-Methoxyethanol, Methyl cellosolve, Glycolmethyl ether, Dowanol em, Poly-solv em.

Life Hazard: Little health hazard if application doesn't require heating.

Flammability: Flash point (49°C), Autoignition temperature (288°C) Flammable limits in air (2.5-l9.8%).

Handling: N/A

Emergency Treatment and Measures:

a. Hygienic precautions: N/A
b. First Aid: Contact with skin or eyes: flush with water. If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): Use alcohol foam, carbon dioxide, or dry chemical fire extinguisher. Cool exposed containers with water.

Spills: Absorb on paper and burn in hood.

028 **Ferrous ammonium sulfate** $(NH_3)_2Fe(SO_4)_2 \cdot 6H_2O$

Synonyms: Ammonium ferrous sulfate hexahydate, Iron ammonium sulfate, Mohr's salt.

Life Hazard: Toxicity will be that of iron ion. Irritating and toxic ammonia and oxides of nitrogen may form in fires.

Flammability: Not Flammable

Handling: Do not expose to light for extended periods of time.

Emergency Treatments and Measures:

a. Hygienic Precautions.
b. First Aid: Ingestion: Give large amount of water, induce vomiting, call a physician. Eye and skin contact: Flush with water.
c. Fire Precautions (in case of outbreak): N/A

Spills: Wash down the drain with excess water.

029 **Gasoline**

Synonym: Petrol, Motor Spirit, Benzin.

Life Hazard: Vapors are considered to be moderately poisonous. Dangerous: When exposed to heat or flame; can react vigorously with oxidizing materials.

Flammability: Flash point about -45°C; ignition temperature about 250°C; explosive range about 1.3-6.0%.

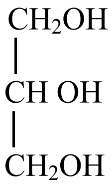
Handling: Keep bottles, cans and drums closed and avoid direct sunlight. No fire. Outdoor or detached storage is preferred. For indoor storage, use combustible liquid storage rooms or cabinets. Wear goggles, rubber gloves, a chemical cartridge respirator and coveralls.

Emergency Treatment and Measures:

a. First Aid: Irrigate eyes with running water. Wash contaminated areas of body with soap and water. If swallowed, call a physician. Do not induce vomiting. Administer oxygen, if indicated.
b. Fire Precautions (In case of outbreak): Use carbon dioxide, dry chemical, or "alcohol" foam. Water spray is not effective for extinguishing, but effective to keep fire-exposed containers cool. If a leak or spill has not ignited, use water to disperse the vapor and to protect men attempting to stop the leakage.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper.

030 **Glycerine**



$$\begin{array}{c} CH_2OH \\ | \\ CH\ OH \\ | \\ CH_2OH \end{array}$$

Synonyms: Glycerol, 1,2,3-Propanetriol, Trihydroxypropane.

Life Hazard: Relatively nontoxic.

Flammability: Flash point 160°C; ignition temperature 429°C. May be explosive in contact with such strong oxidizer as bichromate, potassium chlorate, potassium permanganate.

Handling & Storage: Keep containers well closed.

Emergency Treatment and Measures:

a. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. If swallowed, call a physician.
b. Fire Precautions (In case of outbreak): Use water, dry chemical, alcohol foam, or carbon dioxide. Water or foam may cause frothing. Use water to keep fire-exposed containers cool, to flush spills away from exposures and to dilute spills to noncombustible mixtures.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper.

031 **n-Heptane** $CH_3(CH_2)_5CH_3$

Synonym: Dimethyl pentene, Diethyl propane.

Life Hazard: Narcotic in high concentrations. A central nervous system irritant. Dangerous, upon exposure to heat or flame; can react vigorously, with oxidizing materials.

Flammability: Flash point -4°C (open cup), -22°C (close cup); ignition temperature 223°C; flammable limits 1.25-6.90%.

Handling: Keep containers such as can or bottle closed. Store in cool, dark, well-ventilated place. Avoid every source of ignition. Protect containers against physical damage. Wear rubber gloves, face protecting board, overalls.

Emergency Treatment and Measures:
a. First Aid: Flush eyes with water. Wash contaminated areas of body with soap and water. If swallowed, call a physician.
b. Fire Precautions (In case of outbreak): Use carbon dioxide, dry chemical or "alcohol" foam extinguisher. Water spray may be ineffective as an extinguishing agent, but water should be used to keep fire-exposed containers cool.

Spills: Absorb on paper. Evaporate on glass or iron dish in hood. Burn the paper.

032 **Heptylic Acid** $CH_3(CH_2)_5COOH$

Synonyms: Heptanoic Acid

Life Hazard: N/A
Flammability: Flash point (112°C)

Handling: A corrosive material.

Emergency Treatment and Measures:
a. Hygienic Precautions: N/A
b. First Aid: N/A
c. Fire Precautions (In case of outbreak): N/A

Spills: Carefully mix the acidic compound with dry sodium bicarbonate. Dilute slowly with water and wash down the drain with excess water.

033 ***n*-Hexane** $CH_3(CH_2)_4CH_3$

Synonym: Hexane.

Life Hazard: Dangerous: When heated or exposed to flame; can react vigorously with oxidizing materials. Vapors may explode.

Flammability: Flash point -30°C (close cup); ignition temperature 260°C; flammable limits 1.25-6.90%. Severe fire risk.

Handling: Keep containers closed; store in well-ventilated, cool and dark place. Away from sources of ignition. Inspect frequently existence of leak. Wear rubber gloves, and overalls.

Emergency Treatment and Measures:

a. Hygienic Precaution: Adequate ventilation.

b. First Aid: Skin contact: Wipe off, wash with soap and water. Eye contact: Flush with water. If swallowed, call a physician.

c. Fire Precautions (In case of outbreak): Use carbon dioxide, dry chemical or "alcohol" foam extinguisher. Water is ineffective to fight fire, but is effective to keep fire-exposed containers cool.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn paper.

034 **Hydrochloric Acid** HCl

Synonym: N/A

Life Hazard: Toxic. Eye, skin and respiratory irritant. Inhalation of concentrations of about 1500 parts per million in air are fatal in a few minutes. Hazardous reaction with ethylene.

Flammability: Not flammable.

Handling: Protect containers against physical damage. Store in a cool, well-ventilated place separated from oxidizing substances: Wear rubber gloves, rubber apron.

Emergency Treatment and Measures:

a. First Aid: Irrigate eyes with water. Wash contaminated areas of the body with soap and water. Treat burns as usual. If swallowed; provide oxygen. Do not induce vomiting; call a physician.

b. Fire Precautions (In case of outbreak): Use water. Neutralize chemically with basic substances such as soda ash or slaked lime.

Spills: Cover with sodium carbonate or an equal mixture of soda ash and slaked lime. After mixing, add water if necessary to form a slurry. Dispose by slowly adding soda ash and slaked lime. Discharge the solution with large amount of water into a sink lined with protective matting and filled with dripped marble.

035 **Isooctane** $(CH_3)_2CH(CH_2)_4CH_3$

Synonyms: 2,2,4-trimethylpentane, 2-methylheptane.

Life Hazard: High concentrations can cause narcosis.

Flammability: Flash point (-7°C), autoignition temperature (784°F)

Handling: Keep away from heat and open flame: can react vigorously with reducing materials.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate Ventilation.
b. First Aid: If swallowed, call a physician.
c. Fire Precautions (First Aid): Use foam, carbon dioxide, or dry chemical fire extinguisher.

Spills: Absorb on paper. Burn the paper in a hood.

036 **Isopropyl Alcohol** $(CH_3)_2CHOH$

Synonyms: Isopropanol, Dimethylcarbinol, Sec-propyl alcohol.

Life Hazard: Acts as a local irritant and in high concentrations as a narcotic. Can cause eye burns. Dangerous! Keep away from heat and open flame; can react vigorously with oxidizing materials.

Flammability: Flash point 12°C (closed cup), 21°C (open cup); auto ignition temperature 399°C; explosive range 2-12%.

Handling & Storage: Wear safety glasses, rubber gloves, a face shield and coveralls.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilation.
b. First Aid: Irrigate eyes with water. If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): Use "alcohol" foam or trichloroethane fire extinguisher. Water spray is not effective to extinguish a fire but is effective to cool containers facing fire or to disperse spills before igniting.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper.

037 **Kerosene**

Synonyms: Kerosine

Life Hazard:

Flammability:

Handling:

Emergency Treatment and Measurements:

a. Hygienic Precautions.
b. First aid.
c. Fire precautions (in case of outbreak).

Spills: Absorb on paper. Burn the paper in a hood.

038 **Lead acetate** $Pb(CH_3COO)_2 \cdot 3H_2O$

Synonyms: Lead diacetate.

Life Hazard: A suspected carcinogen. Moderate fire and explosion hazard. When heated it emits highly toxic fumes; can react vigorously with oxidizing materials.

Flammability: Not flammable.

Handling: Keep containers closed. Wear rubber gloves, safety goggles. Separate from feed and foodstuff.

Emergency Treatment and Measurements:

Hygienic Precautions: Protect against dust inhalation. Wash hands and fingers carefully before taking food and drink or smoking.

Spills: Dissolve in a beaker with plentiful water. Add soda ash and mix. Neutralize with dilute acid. Drain into the sewer with abundant water.

039 **Magnesium oxide** MgO

Synonyms: Magnesia, Calcined magnesia.

Life Hazard: Avoid inhalation of fumes. Dangerous when heated, it burns violently in air and emits fumes; will react with water or steam to produce hydrogen; on contact with oxidizing materials, it can react vigorously.

Flammability: May ignite and explode when heated with sublimed sulfur, magnesium powder or aluminum powder.

Handling: Keep containers well closed. Compound is moisture-sensitive. Wear thick gloves, safety glasses and a self-contained breathing apparatus.

Emergency Treatment and Measures:

First Aid: Irrigate eyes with water. Wash contaminated areas with soap and water.

Spills: Bury in a landfill, well away from streams, rivers or wells.

040 **Mercuric Iodide** HgI_2

Synonym:

Life Hazards: A central nervous system toxin. Dangerous: When heated to vaporization, they emit highly toxic fumes of mercury.

Flammability:

Handling & Storage: Keep containers closed. (Small quantities are preferably stored in polyethylene bottle.) Maintain good ventilation. Close attention should be paid to maintenance and cleaning of apparatus. Wear rubber gloves, self-contained breathing apparatus.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilation. Housekeeping in plant must be meticulous and all spills must be cleaned up promptly. Air must be sampled frequently. No eating or smoking in work areas. Protective clothing with special lockers and bathing facilities. Physical examinations of exposed personnel at intervals determined by the degree of exposure, including determinations of mercury in urine as well as neuro-logic evaluations. Preclude from exposure those individuals with diseases of liver, kidneys, lungs and nerves.
b. First Aid: If swallowed, call a physician.

Spills: N/A

041 **Methyl Alcohol** CH_3OH

Synonyms: Methanol, Wood Spirits, Wood Alcohol.

Life Hazard: Narcotic in high concentrations and vapors act as an anesthetic. Dangerous, upon exposure to heat or flame; can react vigorously with oxidizing materials. Is poisonous.

Flammability: Flash point 15.5°C (open cup), 10°C (closed cup), autoignition temperature 464°C; flammable limits 6.0-36.5%.

Handling & Storage: Store containers in well-ventilated place. Large volume storage should be remote from inhabited buildings or structures. Keep away from all sources of ignition. Wear chemical goggles or face shield, rubber gloves, apronsand boots.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilation. Physical examinations of exposed personnel every six months including tests of visual function, careful neurologic evaluation, and studies of kidney and liver function. Preclude from exposure those individuals with diseases of eyes, liver, kidneys and lungs.
b. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. Hospitalize, and call a physician.
c Fire Precautions (In case of outbreak): Use dry chemical, alcohol foam, or carbon dioxide, water spray may be ineffective as an extinguishing agent, but water should be used to keep fire-exposed containers cool.

Spills: Absorb on paper. Evaporate on glass or iron dish in hood. Burn the paper.

042 **Methyl ethyl ketone** $CH_3COC_2H_5$

Synonyms: Methyl Ethyl Ketone; 2-Butanone, MEK.

Life Hazard: Strongly anesthetic. Threshold limit value probably similar to that of ethyl ether, 400 ppm. Wear self-contained breathing apparatus. Highly dangerous upon exposure to heat or flame.

Flammability: Flammable; dangerous fire and explosion risk. Flash point (open cup) -4°C; ignition temperature 516°C; flammable limits 2-10%.

Handling & Storage: Close tightly, store in cool place, and check intermittently for leakage. Avoid fire. Preferably provide electrical equipment with spark-resistant construction. Wear rubber gloves, protective clothing, and face shield. Provide with general-purpose breathing apparatus.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilation. Enforce adequate protective clothing. Make annually physical examinations, including blood cell count. Preclude from exposure those individuals with diseases of skin, blood, and central nervous system.
b. First Aid: Flush eyes with water. Wash out contaminated areas of body with soap and water. If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): Use alcohol foam, powder, and carbon dioxide extinguisher. Water spray is ineffective for extinguishing, but may help to keep fire-exposed containers cool, and to guard fighters who attempt to stop leakage by diffusing vapor of the leaked ketone if not flushed yet.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper.

043 **Methyl orange** $NaOsS\text{-}N{=}N\text{-}N(CH_3)_2$

Synonyms: Helianthine B, Orange III, Bayer 22555, Dimethylaminobenzene-diazosulfonic acid sodium, p-dimethaminoazobenzesulfonate.

Life Hazard: Dangerous: When heated to decomposition, it emits highly toxic fumes of SOx.

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures: N/A

Spills: The compound should be burned. Ideally, all hydrocarbons and related solvents should be burned in a solvent burner. Bury in a land fill, well away from streams, rivers, or wells.

044 **Naphtha**

Synonym: Petroleum ether.

Life Hazard: A recognized carcinogen. Moderate, when exposed to heat or flame; can react with oxidizing materials. Keep containers tightly closed.

Flammability: Flash point -48.3°C; ignition temperature 287.8°C; flammable limits 0.9-6%.

Handling: Keep containers closed, and store in a cool, well-ventilated place. Wear rubber gloves, face shield, overalls and all-purpose canister mask.
Emergency Treatment and Measures:
a. Hygienic Precautions: Adequate ventilation.
b. First Aid: Inhalation; remove to fresh air, treat symptoms. Ingestion: do not induce vomiting, call a doctor. Eyes: flush with water for 15 min. Skin: wipe off and wash with soap and water.
c. Fire Precautions (In case of outbreak): Use foam, dry chemical, or carbon dioxide fire extinguisher.

Spills: Absorb on paper. Evaporate on a glass or iron dish in hood. Burn the paper.

045 **Neopentyl Polyol Ester**

Synonyms: N/A

Life Hazard: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:
a. Hygienic Precautions: N/A
b. First Aid: N/A
c. Fire Precautions (In case of outbreak): N/A

Spills: N/A

046 **Nitric acid** HNO_3

Synonyms: Aqua Fortis, Engraver's Acid, Azotic Acid.

Life Hazard: Dangerous when heated to decomposition, it emits highly toxic fumes of NOx and hydrogen nitrate; will react with water and steam to produce heat and toxic and corrosive fumes. Liquid causes severe tissue burns.

Risks by Contacts under Mixed Conditions

Hazardous Substances if contacts are made	Items	Conditions	Phenomenom	Remarks	
Nitric Acid	Alcohol Inflammables Copper Chloride of β-diketone	Contacts under normal temperature	Ignition Ignition Decomposition explosion	Forms ester of nitric acid	The use of nitric acid is hazardous when selenium is oxidized into selenic acid as it forms an explosive coagulate during the drying up. Phosphorus may explode by heating if it does not dissolve completely during the
Concentrated Nitric Acid	Acetylene Organics Conc. ammonia water Conc. amines Nitrogen dioxide Carbohydrates Organic acid Strong oxidizing agent		Ignition or explosion Ignition or explosion Ignition or explosion Ignition or explosion Ignition or explosion Ignition or explosion	Forms tetranitro-methane ammonium nitrate amine nitrate forms nitrocellulose, nitrostarch & acetyl nitrate	

Fuming thick nitric acid	Turpentine, oil, paper, wood, cloth & other organics		Ignition		preparation of phosphoric acid
Fuming nitric Acid	Thiophene Hydrogen iodide gas Hydrogen selenide Hydrogen phosphide Hydrogen sulfide	Contacts under normal temperature	Explosion Ignition Ignition Ignition Ignition	Forms tetranitromethane ammonium nitrate amine nitrate forms nitrocellulose, nitrostarch & acetyl nitrate	The use of nitric acid is hazardous when selenium is oxidized into selenic acid as it forms an explosive coagulate during the drying up. Phosphorus may explode by heating if it does not dissolve completely during the preparation of phosphoric acid

Flammability: Noncombustible but dangerously reactive with many materials. Reacts explosively with metallic powders, carbides, hydrogen sulfide and turpentine. Increases flammability of combustible, organic and readily oxidizable materials; can cause spontaneous ignition of some materials.

Emergency Treatment and Measures:

a. Hygienic Precautions: Requires proper ventilation and clothings for protection. Those suffering from diseases of lungs and kidney should avoid contact.
b. First Aid: Fully rinse eyes with water. Bodily polluted portions should be fully washed by 5% solution of sodium bicarbonate, or soap and water. Burns should be treated with a cloth immersed in a saturated solution of potassium thiosulfate. If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): N/A.

Spills: Cover with sodium carbonate or an equal mixture of soda ash and slaked lime. After mixing, add water if necessary to form a slurry.

047 **N0-Chromix Cleaning Solution**

Synonyms: N/A

Life Hazard: This solution is a strong oxidizing agent and very corrosive.

Flammability: N/A

Handling: Safety equipment must be worn when preparing and using this solution.

Emergency Treatment and Measures: N/A

Spills: N/A

048 **Oleic acid** $CH_3(CH_2)CH=CH(CH_2)_7$ COOH

Synonyms: cis-Octadecenoic acid, Red oil.

Life Hazard: Slight, when exposed to heat or flame.

Flammability: Flash point 200°C; Autoignition temperature 363°C; combustible.

Handling & Storage: Keep containers closed and store in cool and dark places. Wear rubber gloves, face shield, coveralls, full body shields and self-contained breathing apparatuses should be equipped.

Emergency Treatment and Measures:

a. First Aid: Ingestion: give large amount of water, call a physician. Eyes: flush with water, get medical attention. Skin: wash with soap and water.
b. Fire Precautions (In case of outbreak): Use dry chemical, or carbon dioxide. Water or foam may cause frothing. Water spray may be used to flush spills away from exposures.

Spills: Cover with soda ash or sodium bicarbonate. Mix and add water. Neutralize and drain into a drain with sufficient water.

049 **Pelargonic Acid** $CH_3(CH_2)_7COOH$

Synonyms: Ethyl Heptanoic Acid, Nonanoil Acid, Nonic Acid.

Life Hazard: Limited data suggests low systemic toxicity but high irritation of skin.

Flammability: Flash point (l00 C).

Handling: N/A

Emergency Treatment and Measures:
- a. Hygienic Precautions: N/A
- b. First Aid: N/A
- c. Fire Precautions (In case of outbreak): N/A

Spills: Bury in a land fill, well away from streams, rivers, or wells.

050 **1,10-Phenanthroline**

Synonyms: 4,5-Diazaphenanthrene, Orthophenanthroline, Beta-Phenanthroline, 4,5-Phenanthroline.

Life Hazard: A toxic material

Flammability: N/A

Handling: A hygroscopic material.

Emergency Treatment and Measures: N/A

Spills: Absorb on paper. Burn in a hood.

051 **Phenolphthalein** $C_{20}H_4O_4$

Synonyms: 3-3-bis (p-hydroxyphenyl) phthalide.

Life Hazard: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:
- a. Hygienic Precautions: N/A
- b. First Aid: N/A
- c. Fire Precautions (In case of outbreak): N/A

Spills: Absorb on paper, burn in solvent burner. Bury the ashes in a landfill well away from streams, rivers, or wells.

052 **Phosphoric Acid** H_3PO4

Synonyms: Orthophosphoric Acid.

Life Hazards: When heated to decomposition, it emits toxic fumes of POx.

Flammability: Not flammable. Reacts with metals to liberate flammable hydrogen gas.

Handling: A corrosive material. Wear rubber gloves.

Emergency Treatment and Measures:
- a. Hygienic Precautions: Proper ventilation and protector: preclude from exposure those individuals with pulmonary diseases.
- b. First Aid: Ingestion: do not induce vomiting, give water, milk or vegetable oil, call a physician. Skin or eye contact: flush with water for at least l5 min.
- c. Fire precautions (In case of outbreak): N/A

Spills: Neutralize with lime or sodium bicarbonate. Dilute slowly with water and wash down the drain with excess water.

053 **Potassium carbonate** K_2CO_3

Synonyms: Potash, Pearl ash.

Life Hazard: Toxic. Yields toxic fumes when involved in fire. A strong caustic.

Flammability: N/A

Handling: Wear rubber gloves, safety glasses and coveralls. A hygroscopic material.

Emergency Treatment and Measures: N/A

Spills:
- a. For solid: Sweep into a beaker. Dilute with sufficient water. Add soda ash. Mix and neutralize with dilute acid. Drain into the sewer with abundant water.
- b. For solution: Cover with soda ash. After mixing, transfer into a beaker containing water. Neutralize with dilute acid. Drain into a sewer with abundant water.

054 **Potassium chlorate** $KClO_3$

Synonym: Potcrate.

Life Hazard: Dangerous; shock will induce explosion; when heated to decomposition, it can emit toxic fumes and explode; can react with reducing materials. Hazardous reactions with:

Substance	Condition	Reaction
Sulfur Carbon disulfide	friction, impact, heating	explosion
Organic sulfur, sulfides	friction, impact, heating	explosion
Phosphorus red	friction, impact, heating	explosion
Ammonium rhodanate	friction, impact, heating	explosion
Ammonium rhodanate + copper + alcohol	impact	ignition
Hydrazine, hydroxylamine	friction, impact	explosion
Zinc chloride	contact	explosion
Sugar + ferricyanides	letting stand at atmospheric	heat generation
Sodium hyposulfites	impact	explosion
Powder of combustible materials	impact, friction	explosion
Amines (org, inorg)	impact, friction	explosion
	impact, friction	explosion

Flammability: Powerful oxidizing material. Forms explosive mixtures with combustible, organic or other easily oxidizable materials. These mixtures are easily ignited by friction or heat.

Handling: Protect containers against physical damage. Separate from combustible, organic or other readily oxidizable materials, acids, ammonium salts, sulfur and flammable vapors. Avoid storage on wood floors. Immediately remove and dispose of any spilled chlorate. Wear rubber gloves, face shield and coverall.

Emergency Treatment and Measures:

a. Hygienic Precautions: Encourage personal cleanliness. Provide instructions to exposed personnel.
b. First Aid: If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): Flood with water.

Spills: Cover with weak reducing agents such as hypo, bisulfites or ferrous salts. Bisulfites or ferrous salts need additional promoter of some 3M-H_2SO_4 to accelerate reaction. Transfer the slurry (or sludge) into a large container of water and neutralize with soda ash. Drain into a sewer with abundant water.

054 **Potassium dichromate** $K_2Cr_2O_7$

Synonyms: Potassium bichromate, Red chromate of potash.

Life Hazard: Highly toxic. A recognized carcinogen. A moderate fire hazard by chemical reaction. A powerful oxidizer. Dust may cause skin and nasal irritation. Strong solutions can irritate the skin. Hazardous reaction with:

Substance	Condition	Reaction
Mercury cyanides	Friction	Burning
Flammable materials	Heating	Ignition
Hydrazines, their hydrates	Contact	Ignition, explosion
Hydroxylamines, their salts	Contact	Ignition, explosion
Slaked lime	Mixing	Explosion

Flammability: N/A

Handling: Store in cool, dry place away from easily oxidizable substances, inflammables and organic substances. Avoid storing on wooden floor. Keep away from mercury cyanide, hydrazine and its hydrates, hydroxylamine and the salts, slaked lime. Wear rubber gloves, face shield, work clothing, and entire bodily shield if prevails strong reactions.

Emergency Treatment and Measures:

a. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. Call a physician.
b. Fire Precautions (In case of outbreak): In case of a fire use a flooding amount of water.

Spills: Cover with weak reducing agents such as hypo, bisulfites or ferrous salts. Bisulfites or ferrous salts need additional promoter of some 3M-H_2SO_4 to accelerate reaction. Transfer the slurry (or sludge) into a large container of water and neutralize with soda ash. Drain into a sewer with abundant water.

056 **Potassium hydroxide** KOH

Synonyms: Caustic potash, Potassium hydrate, lye.

Life Hazard: Toxic. A severe eye hazard; solid or concentrated solution destroys tissue on contact. Dangerous; will react with water or steam to produce caustic solution and heat.

Flammability: Not flammable.

Handling: Store in dry place to prevent corrosion of the surface of the drum, absorption of moisture, freezing, etc. For outdoor storage in liquid state, it is advised to protect with some insulation and heating systems. Wear rubber gloves, big face shield and working clothes.

Emergency Treatment and Measures:

a. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. Treat skin burns as usual. If swallowed, call a physician.

   b. Fire Precautions (In case of outbreak): Use large volume of water, but do not splash its solution.

Spills:
   a. For solid: Collect and remove with a broom in a large bucket. Dilute with water and neutralize with dilute acid. Drain into a sewer with sufficient water.
   b. For solution: Neutralize with dilute acid. Wipe with mop or use water aspirator. Drain into a sewer with sufficient water.

057 **Potassium Hydrogen Phthalate** $H00CC_6H_4COOK$

Synonyms:

Life Hazards:

Flammability:

Handling:

Emergency Treatment and Measures:
   a. Hygienic Precautions:
   b. First Aid:
   c. Fire Precautions (In case of outbreak):

Spills:

058 **Potassium Iodide** KI

Synonyms:

Life Hazard: Low toxicity; dietary supplement (up to 0.01% in table salt).

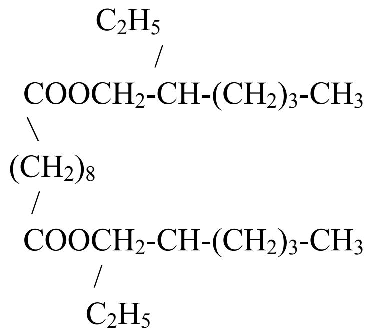
Flammability:

Handling:

Emergency Precautions and Measures:
   a. Hygienic Precautions:
   b. First Aid:
   c. Fire Precautions (In case of outbreak):

Spills:

059 **Sebacic**



$$(CH_2)_8\left(COOCH_2\text{-}CH(C_2H_5)\text{-}(CH_2)_3\text{-}CH_3\right)_2$$

Synonyms: Bisoflex DOS, Decanedioic Acid Bis (2-Ethylhexyl) ester, Di (2-ethylhexyl) sebacate, Dioctyl sebacate, DOS, 2-ethylhexyl sebacate, 1-Hexanol 2-ethyl-sebacate, Monoplex DOS, Octoils octyl sebacate, PX 438, Staflex DOS Bisoflex DOS.

Life Hazard: Highly volatile and can act as asphyxiants or narcotic.

Flammability: N/A

Fire Precautions: Use foam, carbon dioxide, or dry chemical fire extinguisher.

Spills: Absorb on paper and evaporate on glass dish in hood. Burn the paper.

060 **Silicon Sulfate**

Synonyms:

Life Hazards:

Flammability:

Handling:

Emergency Treatment and Measures:
- a. Hygienic Precautions:
- b. First Aid:
- c. Fire Precautions (In case of outbreak):

Spills:

061 **Soda Lime** Na_2CO_3

Synonyms: N/A

Life Hazard: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:

a. Hygienic Precautions: N/A
b. First Aid: N/A
c. Fire Precautions (In case of outbreak): N/A

Spills: N/A

062 **Sodium carbonate** Na_2CO_3

Synonyms: Soda ash, Crystal carbonate.

Life Hazard: Low health hazard, is an irritant.

Flammability: N/A

Handling: Keep containers closed to store. A hygroscopic material. Wear rubber gloves, safety glass and coveralls.

Emergency Treatment and Measures:

First Aid: Flush eyes with water. Wash skin with soap and water. If swallowed, call a physician.

Spills:

a. For solid: Sweep into a beaker. Dilute with sufficient water. Add soda ash. Mix and neutralize with 6M-HCl. Drain into the sewer with abundant water.
b. For solution: Cover with soda ash. After mixing, transfer into a beaker containing water. Neutralize with 6M-HCl. Drain into a sewer with abundant water.

063 **Sodium chloride** NaCl

Synonyms: Salt, Common salt, Halite, Sea salt.

Life Hazard: Low health hazard.

Flammability: N/A

Handling: Store in dry area, is a hydroscopic material.

Spills and Leakage:

a. For solid: Collect, dilute with water, and drain into the sewerwith sufficient water.
b. For solution: Wipe with mop or use water aspiration. Drain into the sewer with sufficient water.

064 **Sodium dichromate** $Na_2Cr_2O_7$ □ $2H_2O$

Synonyms: Sodium bichromate, Sodium acid chromate.

Life Hazard: A recognized carcinogen. Dust may cause skin and nasal irritation. Strong solutions can irritate the skin. Hazardous reaction with:

	Condition	Reaction
Carbide	Mixing	Ignition & explosion
amines such as hydrazine, hydroxylamine, anilin	Mixing	Ignition & explosion
Slaked lime	Mixing	Ignition & explosion
Mercuric cyanides	Friction	Heat evolution & decomposition
Oxidizable materials	Contact	Ignition

Flammability: Decomposes by heating and discharging oxygen, thus promotes the combustion.

Handling: Keep away from inflammables and easily oxidizable substances; tightly close and store in dry place. Wooden floor is not recommendable.

Emergency Treatment and Measures:

a. Hygienic Precations: Adequate ventilation. Chest X-rays every six months up to five years exposure and every three months in excess thereof. Papanicolaou studies of sputum every three months.
b. First Aid: If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): Put out fire by flooding water.

Spills: Cover with weak reducing agents such as hypo, bisulfites or ferrous salts. Bisulfites or ferrous salts need additional promoter of some 3M-H_2SO_4 for rapid reaction. Transfer the slurry (or sludge) into a large container of water and neutralize with soda ash. Drain into the sewer with abundant water.

065 **Sodium hydroxide** $NaOH$

Synonyms: Caustic soda, Sodium hydrate, White caustic, Soda lye.

Life Hazard: Toxic. A severe eye hazard; solid or concentrated solution destroys tissue on contact.

Flammability: Not combustible but solid form in contact with moisture or water may generate sufficient heat to ignite combustible materials. Contact with some metals can generate hydrogen gas.

Handling: Protect against physical damage of container. Store in dry place. Protect against moisture and water. Separate from acids, metals, explosives, organic peroxides and easily ignitible materials. Wear chemical goggles or full face shield, mechanical filter respirator, rubber gloves, aprons and boots.

Emergency Treatment and Measures:

a. Hygienic Precautions: Personal cleanliness.
b. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. Treat skin burns as usual. If swallowed, call a physician.
c. Fire Precautions (In case of outbreak): Flood with water, using care not to splatter or splash this material.

Spills:

a. For solid: Collect and remove with a broom in a large bucket. Dilute with water and neutralize with dilute acid. Drain into a sewer with sufficient water.
b. For solution: Neutralize with HCl. Wipe with mop or use water vacuum. Drain into the sewer with sufficient water.

066 **Sodium Sulfate** Na_2SO_4

Synonyms:

Life Hazards: Is an irritant. Can emit highly toxic SOx when heated to decomposition.

Flammability: Not Flammable.

Handling: Is a hygroscopic material.

Emergency Treatments and Measures:

a. Hygienic Precautions: N/A
b. First Aid: Ingestion, treatment is symptomatic and supportive, call a physician.
c. Fire Precautions (In case of outbreak): N/A

Spills: Wash down the drain with excess water.

067 **Sodium Thiosulfate** $Na_2S_20_3$ □ $5H_20$

Synonyms: Hyposodium subsulfite.

Life Hazards: Low toxicity; used as food additive up to 0.l9.

Flammability:

Handling:

Emergency Treatment and Measures:

a. Hygienic Precautions.
b. First Aid.
c. Fire Precautions (In case of outbreak).

Spills:

068 **Sulfuric acid** H_2SO_4

Synonyms: Oil of vitriol, Dipping acid, Battery Acid, Fertilizer Acid.

Life Hazard: Contact with the body tissue results in rapid tissue destruction, causing severe burns. Threshold limit value of fumes or mist, 1 mg/m^3 moderate fire hazard, by chemical reaction; a powerful oxidizer; can ignite upon contact with combustibles. When heated, it emits highly toxic fumes.

Flammability: Not flammable but highly reactive and capable of igniting finely divided combustible materials on contact. Reacts violently with water and organic materials with evolution of heat. Extremely hazardous in contact with many materials. Attacks and corrodes many metals by releasing hydrogen.

Handling: Protect containers against physical damage and prevent contacts with water. Separate from carbides, chlorates, fulminates, nitrates, picrates, powdered metals and combustible materials. Wear rubber gloves, chemical goggles, rubber boots and chemical cartridge respirator.

Emergency Treatment and Measures:

a. Hygienic Precautions: Adequate ventilation. Preclude from exposure those individuals with pulmonary diseases.
b. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. If swallowed, call a physician. Do not induce vomiting.
c. Fire Precautions (In case of outbreak): Fire involving small amount of combustibles may be smothered with suitable dry chemical. Use water on combustibles burning in vicinity of this material but use care as water applied directly to this acid results in evolution of heat and causes splattering.

Spills: Cover with sodium carbonate or an equal mixture of soda ash and slaked lime. After mixing, add water if necessary to form a slurry.

069 **Sulfurous Acid** H_2SO_3

Synonyms: N/A

Life Hazard: Highly toxic by ingestion and inhalation; strong irritant to tissue.

Flammability: N/A

Handling: Wear self-contained breathing apparatus, eye goggles, rubber gloves, and rubber apron.

Emergency Treatment and Measures:

First Aid: If swallowed, call a physician.

Spills: N/A

070 **Tannic acid** $C_{76}H_{52}O_{46}$

Synonyms: Gallotannic acid, Tannin, Glycerite.

Life Hazard: Moderately toxic by ingestion and inhalation. Combustible. Suspected carcinogen.

Flammability: Flash point 200°C, autoignition temperature 200°C.

Handling: Avoid skin contact and inhalation.

Emergency Treatment and Measures: N/A

First Aid: If swallowed, call a physician.

Spills: Absorb on paper. Burn in a hood.

071 **Teflon**

Synonyms: Polytetra fluoroethylene.

Life Hazards: When heated to above 400 C, it decomposes to yield highly toxic fumes of fluorides.

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:
a. Hygienic Precautions: N/A
b. First Aid: N/A
c. Fire Precautions (In case of outbreak): N/A

Spills: Bury in a landfill well away from streams, rivers, or wells.

072 **Tetrachloroethylene** $Cl_2C{=}CCl_2$

Synonyms: Perchloroethylene. Ethylene tetrachloride, Nema, Carbon dichloride, Tetracap, Perclene.

Life Hazard: Incoordination and impaired judgement may occur at vapor exposures from 300 ppm to 1000 ppm. Dizziness, drowsiness, loss of consciousness and even death can occur at increasing levels of exposure. When involved in fire, tetrachloroethylene emits highly toxic and irritating fumes of chlorides, a suspected carcinogen.

Flammability: The compound has no flash point. Vapor may decompose at high temperature such as open flame, red-heated materials, with evolution of poisonous gases such as chlorine, carbon monoxide, phosgene.

Handling: Store in a closed container in a cool place. Protect against direct sunlight. Do not store in ill-ventilated cellars and hollow places owing to high vapor density. Wear safety goggles, gas mask, rubber gloves. Adequate ventilation.

Emergency Treatment and Measures:

a. Hygienic Precautions: Examine liver and kidneys periodically. Preclude from exposure those individuals with diseases of heart, liver, kidneys and lungs.
b. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. If swallowed, call a physician. Administer oxygen for oxygen-shortage.

Spills: Absorb on paper and evaporate on a glass dish in hood. Burn the paper. The compound should be redistilled and reused.

073 **Tetra Ethyl Lead** $Pb(C_2H_5)_4$

Synonyms: Lead tetraethyl, TEL.

Life Hazards: Powerful poison and a solvent for fatty materials. Suspected carcinogen.

Flammability: Flash point (85°C), Decomposes above 110°C. Violently explodes when heated to more than l70°C.

Handling: Decomposes when exposed to sunlight or allowed to evaporate. Separate from halogenated compounds or oxidzing agents.

Emergency Treatments and Measures:

a. Hygienic Precautions: Avoid skin contact.
b. First Aid: Ingestion: induce vomiting, call a physician Skin: wash immediately with kerosene or similar petroleum distillate followed by soap and water.

c. Fire Precautions (in case of outbreak): Use water, foam, dry chemicals, or carbon dioxide fire extinguisher.

Spills: Absorb the spills with paper towels or like materials. Place in a hood to evaporate. Dispose by burning the towel.

074 **Toluene** C_6H_5Cl

Synonyms: Toluol, Methylbenzene, Phenylmethane, Methacide.

Life Hazard: A recognized carcinogen. Eye and respiratory irritant. Extreme inhalation of vapors may cause death by paralysis of the respiratory center. Threshold limit value, 200 parts per million. Dangerous, when heated, it emits highly toxic fumes; can react with oxidizing materials.

Flammability: Flash point 4.4°C (closed cup); ignition temperature 536°C; flammable limits 1.4-6.7%. Vapors may travel considerable distance to a source of ignition and flash back.

Handling: Protect containers against physical damage. Outdoors or detached or isolated storage is preferable. Indoor storage should be in a standard flammable liquid storage room. Wear chemical goggles, chemical cartridge respirator or self-contained breathing apparatus and impermeable rubber gloves.

Emergency Treatment and Measures:

a. Hygienic Precautions: Physical examinations of exposed personnel annually \with special attention to eyes and nervous system, and including complete blood count and studies of liver function. Preclude from exposure those individuals with diseases of central nervous system and liver.

b. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. An exposed person should be removed immediately to fresh air and kept warm and quiet. If breathing has stopped, artificial respiration should be commenced. Ingestion: do not induce vomiting, call a physician. Eyes; flush with water. Skin: wash with soap and water.

c. Fire Precautons (In case of outbreak): Use water spray, carbon dioxide, dry chemical or foam fire extinguishers.

Spills: Spills are absorbed on paper which is then placed in an iron pan to evaporate it in a hood. Then burn the paper down.

075 **1,1,1-Trichloroethane** $CH_3\ C\ C1_3$

Synonyms: Methylchloroform, Chlorothene, Ethylidyne-chloride.

Life Hazard: Incoordination and impaired judgement may occur at vapor exposures from 500 ppm - 1000 ppm. Dizziness, drowsiness, loss of consciousness and even death can occur at increasing levels of exposure. When involved in fire, 1,1,1-trichloroethane emits highly toxic and irritating fumes.

Flammability: Autoignition temperature 500°C, flammable limits in air 7-l8%.

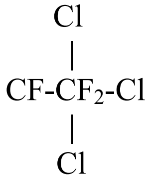
Handling & Storage: Do not store in aluminum containers. Wear safety goggles and rubber gloves.

Emergency Treatment and Measures:

a. Hygienic Precautions: Physical examinations of exposed personnel annually, including studies of liver and kidney function. Preclude from exposure those individuals with diseases of liver and kidneys.
b. First Aid: Irrigate eyes with water. Wash contaminated areas of body with soap and water. Inhalation: oxygen, if necessary. No Adrenaline. Ingestion: have victim drink water and induce vomiting, call a physician.
c. Fire Precautions (in case of outbreak): Use dry chemical, foam, or carbon dioxide fire extinguisger.

Spills: Absorb on paper. Evaporate on a glass dish in hood. Burn the paper in hood.

076 **1,1,2-Trichloro-1,2,2-trifluoroethane**



$$CF\text{-}\overset{\displaystyle Cl}{\underset{\displaystyle Cl}{C}}F_2\text{-}Cl$$

Synonyms: Trifluorotrichloroethane, Freon-113, Freon TF.

Life Hazard: Tolerance, 1000 ppm in air, poisonous gases may be produced in fire.

Flammability: Not flammable.

Handling: Wear safety glasses, working gloves.

Emergency Treatment and Measures:

a. Hygienic Precautions: Wear rubber gloves and apron. Adequate ventilation.
b. First Aid: Skin contact: if frostbite has occured, flush area with warm water. If swallowed, call a physician.

Spills: The compound should be redistilled and reused.

077 **Valeric Acid** $CH_3(CH_2)_3\ COOH$

Synonyms: Pentanic acid.

Life Hazard: A toxic material.

Flammability: Flashpoint (88°C)

Handling: A corrosive material.

Emergency Treatment and Measures:

a. Hygienic Precaution: N/A
b. First Aid: N/A
c. Fire Precaution (In case of outbreak): Use alcohol foam, carbon dioxide or dry chemical fire extinguishers.

Spills: Absorb on paper, then burn in a hood.

078 **Xylene** $C_6H_4(CH_3)2$

Synonyms: Xylol

Life Hazards: Vapors cause headache and dizziness. Liquid irritates eyes and skin. If taken into lungs, causes severe coughing, distress and rapidly developing pulmonary edema. If ingested, causes nausea, vomiting, cramps, headache, and coma; can be fatal. Kidney and liver damage can occur.

Flammability: Flash point (29°-46°C), autoignition temperature (469°-528°C).

Handling: Keep vapors away from open flames.

Emergency Treatments and Measures:

a. Hygienic Precautions: Adequate ventilation, rubber gloves.
b. First Aid: Inhalation: remove to fresh air, administer artifical respiration and oxygen if necessary, call a doctor. Ingestion: Do not induce vomiting, call a doctor. Eyes contact: flush with water for l5 min. Skin contact: wipe off and wash with soap and water.
c. Fire Precaution (In case of outbreak): Use foam, carbon dioxide, or dry chemical fire extinguisher.

Spills: Absorb on paper, then burn in a hood.

079 **Barium Carbonate** $BaCO_3$

Synonyms: N/A

Life Hazard: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:
  a. Hygienic Precautions: N/A
  b. First Aid: N/A
  c. Fire Precautions (In case of outbreak): N/A

Spills: N/A

080 **Chromium Trioxide** CrO_3

Synonyms: N/A

Life Hazard: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:
  a. Hygienic Precautions: N/A
  b. First Aid: N/A
  c. Fire Precautions (In case of outbreak): N/A

Spills: N/A

081 **Nickelous Chloride** $NiCl_2$

Synonyms: N/A

Life Hazard: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:
  a. Hygienic Precautions: N/A
  b. First Aid: N/A
  c. Fire Precautions (In case of outbreak): N/A

Spills: N/A

082 **Silver Nitrate** $AgNO_3$

Synonyms: N/A

Life Hazard: N/A

Flammability: N/A

Handling: N/A

Emergency Treatment and Measures:

a. Hygienic Precautions: N/A
b. First Aid: N/A
c. Fire Precautions (In case of outbreak): N/A

Spills: N/A

APPENDIX A
AUGUST 24, 2006

LIST OF MATERIALS CITED IN EACH TEST METHOD

METHOD	NAME OF THE TEST	MATERIALS
103.6	Color of gasoline	1. gasoline (reference)
202.1	Cloud intensity at low temperature	1.Methanol- glycerol 2.barium Chloride 3. Sulfuric acid 4.Sodium hydroxide 5. Distilled water
203.1	Pour stability of lubricating oils	1. engine crankcase oil
313.3	Penetration of lubricating greases after prolonged working	1. grease to be tested
335.3	Gear wear	1.Dry cleaning solvent (MIL-PRF-680B, Type 1) 2.Petroleum ether(O-E-751) 3.Di-2 ethylhexyl sebscate, commercial grade 4.Test gear set brass and steel
350.2	Evaporation loss of lubricating greases and oils (high temperature)	1. 1,1-Trichloroethane, Technical, inhibited (Methyl Chloroform) ASTM D 4126 2. Tissue Facial
352.1	Effect of evaporation on flammability	1.Pipe cleaner
353.1	Evaporation	1. None
361.4	Stroking properties of hydraulic brake fluids	1.Ethyl alcohol (ACS) 2 Crocus cloth 3.Wheel cylinder assemblies 4. Master cylinder assembly 5. Rubber cups 6.Wheel cylinder pistons 7. Master cylinder pistons
500.1	Induction system deposit(ISD) tendencies of motor gasoline	1. n-Hexane, ACS reagent grade 2. Toluene, ACS reagent grade 3. Acetone, ACS reagent grade
550.1	Emulsification tendencies of petroleum fuels by multiple contact extractions	1.Cleaning solution,concentrated sulfuric acid saturated with technical grade potassium di-chromate 2. Distilled or deionized water

1110.2	Quenching speed	1. Steel test bars, ASTM A 576, A 675, A 675M 2. Abrasive Paper, 280 grit (ANSI B74.18) 3. 1,1,1-trichloroethane (ASTM D 4126) 4. Facial Tissue
1151.2	Explosive vapor in boiler fuel oil	1. Hydrocarbon gas
2508.1	Thermal stability of lubricating and hydraulic fluids	1. Cleaning solution, concentrated sulfuric acid saturated with potassium di-chromate 2. Detergent for cleaning glassware
3005.4	Dirt content of grease	1. None
3007.2	Water displacement and water stability	1. Steel test panels, FS 1009, QQ-S-698) 2. Naphtha, aliphatic (TT-N-95) 3. Methanol (O-M-232) 4. Distilled water 5. Silica sand, white, dry, sharp 6. Surgical gauze 7. Absorbent tissue 8. Emulsion, water and oil
3009.3	Solid-particle contamination in hydraulic fluids	1. Distilled water 2. Alcohol, isopropyl (TT-I-735) 3. Petroleum ether (O-E-751) 4. Detergent ,water soluble, commercial 5. Cleaning solution, glass (concentrated sulfuric acid with saturated potassium or sodium di-chromate, technical grade) 6. Tape, transparent, pressure sensitive 7. Filters, cellulose membrane
3010.9	Solid particle contamination in aircraft turbine engine lubricants (gravimetric procedure)	1. Isopropyl alcohol, reagent grade 2. Peroleum ether, reagent grade boiling point range 30 ℃ to 60 ℃
3012	Determination of particulate matter in aerospace hydraulic fluids	1.AC fine test dust 2.Primary calibration standard 3.Detergent , liquid water soluble, commercial grade. 4.isopropyl alcohol 5.Trichlorotrifluoroethane (MIL-C-81302)

		6. H-515-hydraulic fluid
3013	Determination of particulate contamination in synthetic turbine engine lubricants	1. Isopropyl alcohol , reagent grade 2. Petroleum ether ,30 °C to 60 °C boiling range, reagent grade
3110.1	Fatty oil in cutting fluids by infared spectrophotometry	1. Fatty oil 2. Mineral oil, 0.0-15%
3180.2	Sulfur in cutting fluids	1.Copper powder, ACS 2.Nitric Acid ,5 percent 3.Acetone,ACS 4.Isooctane,Knock test grade 5.Filter paper Whatman No . 4 and 42 or equivalent 6. Firing wire iron approximately26 B and S gage 7. Blue litmus paper 8. Materials required for ASTM D 129
3201.7	Emulsion (petroleum and petroleum like products	1.Emulsifying agent (distilled water, sodium chloride solution or synthetic seawater) as required by the specification.
3205.3	Emulsion (soluble cutting oils)	1. Distilled water
3403.2	Compatibility of turbine lubricating oil	1. Referee lubricant 2. 1,1,1-trichlroethane 3. Cleaning solution ,glass (concentrated sulfuric acid saturated with potassium or sodium dichromate , technical grade)
3410.1	High temperature deposit and oil degradation characteristics of aviation turbine oils	1.Lubricating oil, qualified under MIL-L-23699 2.Dry cleaning solvent, (MIL-PRF-680B, Type 1) 3.No.400 emery paper 4.No. 30 grit aluminum oxide 5.Bi solvent; Isopropyl alcohol: Acetone
3411.1	Thermal stability and corrosivity of air craft turbine engine lubricants	1. Toluene, reagent grade 2. Acetone, reagent grade 3. Nitric-sulfuric mixture, approximately 1:10 4. Abrasive paper, silicone carbide, 240 grit 5. Steel specimens,QQ-S-698 mild

		steel
3430.2	Compatibility characteristics of universal gear lubricants	1. Naphtha,TT-N-95,type II 2. Reference oils
3432.1	Compatibility of elastomers with aircraft turbine lubricants	1.Standard test rubber sheets, type F-A, conforming to US Air Force Specification bulletin 539 2. Standard test rubber sheets type FS conforming to US Air Force Specification bulletin 539 3. Cleaning solution ,turco alkaline rust remover or equivalent. 4.Dry cleaning solvent (MIL-PRF-680B, Type 1) 5. Alcohol, ethyl, ACS grade 6. Acetone, ACS grade 7. Distilled water 8. Filter paper
3433	Compatibility of synthetic aircraft turbine lubricants with silicone rubber	1.Standard test rubber sheets, type F-A, conforming to US Air Force Specification bulletin 539, standard elastomer stocks 2. Cleaning solution , glass (concentrated sulfuric acid saturated with potassium or sodium di chromate) 3. Methyl alcohol, ACS , particle free 4. Solvent-Naphtha or toluene, ACS 5. Acetone, ACS, particle free
3440.1	Storage solubility characteristics of universal gear lubricants	1. Naphtha, (TT-N-95, type 11
3450	Bearing deposition of aviation turbine engine lubricants	1. Dry cleaning solvent, (MIL-PRF-680B, Type 1) 2.Carbon remover (penetone ECS) 3.n- Haptane 4. Acetone 5.Lubricating oil, SAE J1966, grade 1100 6.Polytetrafluoroethylene tape/gasket 7. Fibrefrax insulation 8.Raxbestos-manhattan A-50 gasket materials 9. Metal specimen: - Aluminum; ASTM B 209

		-Titanium; 8Mn Annealed, 110ksi (758 MPa) yield strength-UNS R56080 -Silver; 99.9 purity, grade A -Steel; AMS 5040 -Stainless steel; SAE AMS 5518 10. Glass wool fibre 11. 400 grit silicon carbide paper 12. 30 grit aluminum oxide
3452.2	Bearing compatibility of turbine oils	1. Naphtha; Aliphatic (TT-N-95), type11
3456.2	Channeling characteristics of lubricants	None
3457.2	Hydrolytic stability	1. Emery paper, 00 or finer 2. Litmus paper 3. Petroleum ether (O-E-751) 4. 1,1,1-trichloroethane (methyl chloroform), stabilized (MIL-T-81533) 5. n-Hexane , technical grade 6. Sodium sulfate, anhydrous (ACS) 7. Copper strip (ASTM B 133/ASTM B 272)
3458.1	Low temperature stability test for oil	None
3459.1	Low temperature stability	1. Methyl alcohol-glycerin (technical grade), 1: 1 solution by volume 2. Turbidity standard prepared with barium chloride, sulfuric acid, sodium hydroxide, and distilled water 3. Red dye (National Erie Bordeaux B or equivalent)
3463.1	3463.1Stability of grease in hot water (water immersion)	1. Distilled water
3465.1	Storage stability test of fluids and lubricants	1. Aluminum foil, commercial grade 2. Scouring powder, commercial grade 3. Distilled water
3467.1	Storage stability of lubricating	1. None

	grease	
3770.1	Homogenity and miscibility of oils	1. Six reference oils
3480.1	Volatility of blended oils	1. test oils
3500.1	Monobasic acid components of synthetic ester lubricants by gas chromatography	1.Potassium hydroxide, reagent grade 2. Ethyl alcohol, 95 percent 3. Distilled water 4. Hydrochloric acid , dilute 5. Ethyl ether , reagent grade 6. Sodium sulfate, anhydrous, reagent grade 7. Diethyleneglycol adipate polyester 8.Gas chrom p, 60 to 80 mesh, acid based washed or equivalent 9.Chloroform, reagent grade 10. Phosphoric acid, reagent grade , 85 percent 11. Helium oil free 12. Butyric acid, reagent grade 13. Isovaleric acid, reagent grade 14. valeric acid, reagent grade 15. Caproic acid, reagent grade 16. Heptylic acid, reagent grade 17. caprylic acid, reagent grade 18. pelargonic acid, reagent grade 19. Capric acid, reagent grade
3603.5	Swelling of synthetic rubbers	1. Standard rubber test sheets, as required by the specification. 2. Alcohol, ethyl or methyl, anhydrous. 3. Filter paper 4. Distilled water
3604.2	Swelling of synthetic rubber by aircraft	1.Standard test rubber sheets, type H or FA 2.Cleaning solution , glass(concentrated sulfuric acid saturated with potassium or sodium dichromate , technical grade) 3.Naphtha,technical grade 4.Toluene , technical grade 5.Alcohol, ethyl 6.emery cloth No. 60
3710.1	Molybdenum disulfide purity	1.Potassium chlorate

		2.Nitric acid concentrated 3.Hydrochloric acid ,concentrated 4. Ammonium hydroxide, concentrated 5. Methyl orange indicator solution 6.Ammonium acetate solution Glacial acetic acid 8. Lead acetate solution, 20 g lead acetate,20mL glacial acitic acid and distilled water 9. Tannic acid indicator,0.05g tannic acid, 2mL glacial acetic acid, and 48 mL distilled water 10. Ammonium nitrate solution, 30g ammonium nitrate and distilled water
3720.2	Molybdenum disulfide content of lubricating grease	1. n-Hexane, ACS grade 2. Oleic acid , USP grade 3. Petrolium ether (o-E-751
3722.2	Molybdenum disulfide content of non soap thickened lubricating grease	1.Hexane, normal ACs 2. Extraction thimble (double thickness 3.3 x 8.0 cm) 3.Filter paper (Whatman No. 1 or equivalent) 4. Filter paper (Whatman No. 40 or equivalent 5.Eschka's mixture-2 parts calcined (light) magnesia thoroughly mixed with one part of sodium carbonate 6.Bromine water, saturated 7. Barium chloride 10 percent 8. sodium carbonate CP 9. Potassium carbonate, CP
3816.1	Film thickness of dry solid film lubricants	1. Aluminum alloy test panel (ASTM B 209) anodized in accordance with MIL-T-81533 2. 1,1,1- trichloroethane (MIL-T-81533)
4001.3	Corrosion protection by coating; Salt spray(fog)test	1. Sodium chloride(A-A-59282) 2. Hydrochloric acid (A-A-59282) 3. Sodium hydroxide (A-A-59282) 4. Bromthymol blue solution, indicator, 6.0 to 7.6 pH range 5. Distilled water ACS 6. 1,1,1- trichloroethane 7. Cleaning tissue, facial

		8. Low carbon, open hearth, cold – finished 1010 steel
5003.2	Deposit- forming tendencies of air craft turbine lubricants	1. Silicone-carbide paper. No. 400 2. Solvent, dry-cleaning (MIL-PRF-680B, Type 1) 3. Cleaning solution, glass9Concentrated sulfuric acid saturated with potassium or sodium di chromate technical grade 4.n-Hexane, thiophene-free (ACS) 5.Isopropyl alcohol (ACS) 6. Petroleum ether (ACS) 7.Acetic acid, 25 percent solution 8.Distilled water 9.Scouring powder, commercial grade 10. Steel wool 11.Pipe cleaners 12.Cleaning compounds, special(penetone ECS; Peneton Corporation,74 Hudson ave. Tenafly, NJ 07670 or equivalent
5304.5	Corrosiveness of greases or semi-solid products at 25 °C	1. Copper strips (ASTM B 152/152M) 2. n-Hexane (ACS) 3. Acetone (ASTM D 329), technical grade 4. Abrasive paper, (silicone-carbide or alumina), various grade of grit 5. Abrasive paper , Silicone carbide, 240 grit 6. Silicone –carbide, 150 mesh 7. Cotton, absorbent
5305.1	Corrosiveness of lubricants at 232 ° C or (450 °	1. Solvent, dry cleaning (A-A-59601D) 2. 1,1,1- Trichloroethane (reagent grade) 3. Acetone (ACS) 4. Nitric acid solution (3 parts concentrated nitric acid and 7 parts distilled water0 5. Pumice ,FFFgrade 6. eraser (Eberhard faber “pink

		pearl" No. 100 or equivalent 7. Cleaning solution, glass (concentrated sulfuric acid saturated with potassium or sodium di chromate, technical grade 8. Distilled water
5306.5	Corrosiveness of emulsifiable cutting fluids	1. Sodium hydroxide solution, containing approximately 25 percent NaOH by weight 2. Normal hexane ,commercial 3. 1,1,1-trichloroethane (ASTM D 4126) 4. Distilled water 5. Cleaning tissue 6. Two copper strips (ASTM B 152/152M), electrolytic tough pitch, cold rolled, half hard temper) 7. Two brass strips (ASTM B 121, ASTM B 36, cartridge brass composition 2 half hard temper) 8. Two cast iron strips (QQ-I-652,class 20, strip only) 9. Two steel strips (QQ-S-698), physical quality, cold rolled, half hard temper-CR half hard No. 2 tempers)
5308.7	Corrosiveness and oxidation stability of light oils (metal squares)	1. Copper, electrolytic (ASTM B 152/152M) 2. Steel, carbon, mild (ASTM 1008A/1008M, class 1, commercial bright finish 3. Aluminum alloy (ASTM B 209, temper T-3 or T-4 4. Magnesium alloy (SAE AMS 4375, 4376, 4377, AZ 31B), condition H24 or H26. 5. Anodes, Cadmium(A-A-51126B) 6. Cord (cotton, nylon or linen-, light-weight, clean, for tying metal squares together) 7. Abrasive paper ,(silicon carbide, or alumina) various grade or grit 8. Abrasive paper, silicon carbide

		240 grit 9. Silicon-carbide grains, 150 mesh 10. Cotton, absorbent 11. 1,1,1-trichloroethane (ASTM D 4126) 12. Laboratory filter paper 13. Naphtha, aromatic (TT-N-97)
5309.5	Corrosiveness of greases (copper strip, 100 ° C	1.Copper strip , soft tempered , annealed 2. n-Hexane (ACS grade) 3. Acetone (ACS grade) 4. Fine silicon carbide or aluminum grit paper 5. 240 grit silicon- carbide paper 6. 150 mesh silicon – carbide paper
5321.2	Corrosion of lead by lubricating oils	1. Test panel, Chemical-lead (ASTM B 29-55 or equal) 2. Test panel, copper , Electrolytic (ASTM B 152/152M) 3. Naphtha, petroleum (TT-N-97) 4. Acetone (ASTM D 329) 5. Steel wool 6. Pads, Absorbent- cotton
5329.2	Corrosion Protection (Humidity Cabinet)	1. Naphtha, aliphatic (TT-N-95) 2. Methyl alcohol (O-M-232) 3. Silica sand, white, dry, sharp, (size IAW ASTM E 11) 4. Test panelsFS 1009 steel, fabricated from open-hearth, “ killed ‘’ type low carbon, No. 4 , soft –temper, cold rolled sheet or strip conforming to QQ-S-698 5. Surgical gauze
5331.1	Corrosion protection of steel against sulfurous acid – salt spray by solid film lubricants	1. Steel specimen, carbon steel conforming to fs 1010 2. Synthetic sea water sulfurous acid test solution (formula ASTM D 1141) 3. Aluminum oxide cloth 240 grit 4. 1,1,1- trichloroethane (MIL-T-81533) 5. Distilled water (ASTM D 1193, type 111) 6. Sulfurous acid, ACS

5341	Fuel system icing inhibitor in hydrocarbon fuels (redox method)	1. Crushed ice 2. Ethylene glycol monomethyl ether (EGME) standard solution 3. Potassium di chromate solution, standard 0.2000 N 4. Ferroin indicator (1,10 phenanthroline ferrous complex) 5. Ferrous aluminum sulfate solution 1.1000N . Asolution of ferrous ammonium sulfate, concentrated sulfuric acid, potassium dichromate, ferroin indicator, and water 6. Sulfuric acid , concentrated
5414.4	Resistance of grease to fuel	1. Standard test fluid (ASTM D 471, type111) 2. Aluminum strip (ASTM B 209, tempers T-3 or T04)
5415.1	Resistance of grease to water and a 1:1 water –ethanol solution	1. Alcohol, ethyl, specially denatured, aircraft (A-A-51693) 2. Distilled water
6050.1	Nitrate-type ignition improvers in diesel fuel	1. Saponification mixture 1N. A solution of potassium hydroxide and n-butyl alcohol 2. Diphenylamine (1 percent) solution. A solution of diphenylamine (ACS indicator grade) and sulfuric acid 3. Toluene (ACS Reagent grade)
6052.1	High temperature, high pressure spray ignition	1. Nitrogen gas
6053.1	Manifold ignition test	1. Steel wool
6508.2	Load carrying capacity of lubricating oils (Ryder gear machine)	1. Lubricating oil, SAE J1966 2. Flushing solvents: Stoddard solvent (MIL-PRF-680B, Type 1 and petroleum ether (O-E-751) 3. Test gear, FaFnir WO-6 4. o-rings, National No . 622710 5. Shaft seals
6517	High temperature gear load carrying capacity of lubricating oils	1. lubricating oil, MIL-PRF-7808, SAE J1966, grade 1100 Dry cleaning solvent (A-A-59601D)

		2. Petroleum ether (60-90) °C 3. Platers wax 4. No.30 grit aluminum oxide 5. Purified crystalline nickelous chloride 6. Hydrochloric acid ,reagent grade 7. Crystalline chromium trioxide reagent grade 8. Crystalline silver nitrate reagent grade 9. Barium carbonate 10. Nickel bar stock 11. Lead sheet 12. Reference oil
7501	Low temperature torque test method for lubricating greases	1.None
7502	Solvent cleaning power by soil test method	1. Toluene CP at least 99% 2. Thermometer ,range 50°C
10000	Material handling safety precaution	1. None

APPENDIX B
August 24,2006

CROSS REFERENCE LIST BETWEEN INDEX NUMBER OF TEST METHOD FTM10000, MATERIAL NAME AND TEST METHOD NUMBERS WHERE THE MATERIAL IS CITED

Material Index Number	Compound's Name	Test Method Where the Material is Cited
001	Acetic Acid	5003
002	Acetone	500,3180,3410,3411,3432,3433,3450,5304 5305,5308,5309,5321,5322
003	Ammonium Acetate	3710
004	Ammonium Hydroxide	3710
005	Ammonium Nitrate	3710
006	Barium Chloride	202,3459,3722
007	Barium Hydroxide	
008	Benzoic Acid	
009	Bromine	3722
010	n- Butyl Alcohol	6050
011	Butyric Acid	3500
012	Capric Acid	3500
013	Caproic Acid	3500
014	Caprylic Acid	3500
015	Chloroform	3500
016	Cleaning Compound special, Cleaning Solution, Glass	3450,5003,6511
017	Detergent, Liquid, water soluble, commercial grade	2508, 3012
018	Diesel fuel	
019	Diethylene glycol	
020	Diethylene glycol, mono methyl ether	
021	Diethylene glycol adipate	3500
022	Diphenylamine	6050
023	Dry cleaning solvent, Eschka's Mixture	335,3410,3432,3450,5003,5305,6508,6511,6517
024	Ethyl alcohol	361,3432,3500,3603.5,3604,5415

025	Ethyl ether	3500,3603.5
026	Ethylene glycol	
027	Ethylene glycol monomethyl ether	5341
028	Ferrous ammonium sulfate, Freon-12	5341
029	Gasoline Glacial Acetic Acid	103
030	Glycerin	
031	n-Haptane	3450
032	Heptylic acid	3500
033	n-Hexane	500,3457,3720,3722,5003,5304,5306,5309
034	Hydrochloric acid	3500,3710,40014001,6517
035	Isooctane	3180
036	Isopropyl alcohol	3009,3010, 3012,3013,3410,5003,
037	Kerosine	
038	Lead acetate	3710
039	Magnesium oxide	
040	Mercuric iodide	5327
041	Methyl alcohol Methyl-Alcohol-Glycerine	202,3007,3433,3603.5,5329
042	Methyl ethyl ketone	
043	Methyl orange	3710
044	Naphtha	3007,3013,3430,3433,3440,3452,3604,5308,5321,5329
045	Neopentyl Polyol Ester	
046	Nitric acid Nitric-Sulfuric Acid Mixture	3180,3710,5305
047	N0 –Chromix Cleaning solution	
048	Oleic acid	3720
049	Pelargonic acid, Petroleum Ether	3500
050	1,10- phenanthroline	5341
051	Phenolphthalein	
052	Phosphoric acid	3500
053	Potassium carbonate	3500,3722
054	Potassium chlorate	3710
055	Potassium Dichromate	5341
056	Potassium hydroxide	6050
057	Potassium Hydrogen	

	phthalate	
058	Potassium iodide	
059	Sebabic	335
060	Silicon sulfate	
061	Soda lime	
062	Sodium carbonate	3722
063	Sodium chloride	3201,4001
064	Sodium dichromate	
065	Sodium hydroxide	202,3459,4001,5305
066	Sodium sulfate	3456,3500
067	Sodium thio sulfate	
068	Sulfuric acid	202,3459,5341,6050
069	Sulfurous acid	5331
070	Tannic Acid	3710
071	Teflon	
072	Tetrachloroethylene	
073	Tetraethyl lead	
074	Toluene	500,3411,3433,3604,6050
075	1,1,1-Trichloroethane	350,1110,3403,3457,3816,4001,5305,5306,5308,5331,7502
076	1,1,2-Trichloro-1,2,2-Trifluoroethane	3012
077	Valeric acid	
078	Xylene	6517
079	Barium carbonate	6517
080	Chromium trioxide	6517
081	Nickelous chloride	6517
082	Silvernitrate	6517

Custodians:

Army – AT
Navy – SH
Air Force - 68
DLA – PS (DESC)

Preparing Activity:

Army - AT

Project number 91GP-2007-001

Reviewing Activity:

Air Force - 11

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online, database at http://assist.daps.dla.mil.