

MIL-STD-1262
31 August 1967

MILITARY STANDARD
DEGRADATION TESTS FOR FUELS AND FUEL CONTAINER
COATINGS (TEST PROCEDURE)



FSC 8010

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DEPARTMENT OF DEFENSE
WASHINGTON, D. C. 20301

Degradation Tests for Fuels and Fuel Container
Coatings (Test Procedure)

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1. This Military Standard is mandatory for use by all Departments and Agencies of the Department of Defense.
2. Recommended corrections, additions or deletions should be addressed to Army Materials and Mechanics Research Center, Attn: AMXMR-TMS, Watertown, Massachusetts 02172.

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FOREWORD

Cargo tanks of tankers, barges, railway tank cars, refueler semi-trailers and trucks, surface and subsurface tanks, and shipping containers used for the bulk transportation and storage of a wide variety of military fuels are being lined with fuel and water resisting protective coatings to maintain internal corrosion control as well as quality and cleanliness of the product.

Many coatings have been developed and used satisfactorily in this service. Other candidate coatings continue to evolve with the state of the art. A suitable coating must adequately protect the containers from corrosion, must not in itself be deteriorated by the environment and must in no way degrade or contaminate the product.

Various military specifications are current which adequately prescribe requirements peculiar to the military services for qualified coatings. However, requirements for degradation of the different military fuels by the coating vary widely among the coating specifications. Additionally, newer developments in military fuels continue to evolve and military requirements for fuel quality and cleanliness are becoming increasingly more rigid.

It is the primary intent of this standard to prescribe a uniform test procedure applicable to all military fuels to establish that any coating intended for lining containers for the bulk transportation and storage of fuels does not adversely degrade or contaminate the product. In addition the coating being considered under the intent of this standard must not in itself show evidence of degradation in the various environments specified herein to which it will be subjected.

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1. SCOPE

1.1 Purpose. This standard establishes the requirements and test methods for determining the compatibility of protective coatings intended for lining the interior surfaces of bulk transportation and storage containers with military petroleum base hydrocarbon fuels.

1.2 Application. This standard is designed to be used as a reference in specifications and related documents where a requirement for compatibility of fuel container coating or coating system with fuels is specified.

2. REFERENCED DOCUMENTS

2.1 Specifications and standards. The following documents of the issue in effect on date of invitation for bids or requests for proposal form a part of this standard to the extent specified herein.

SPECIFICATIONS

FEDERAL

QQ-S-698 - Steel, Sheet and Strip, Low-Carbon
TT-S-735 - Standard Test Fluids, Hydrocarbon

MILITARY

MIL-G-5572 - Gasoline, Aviation: Grades 80/87, 100/130, 115/145
MIL-T-5624 - Turbine Fuel, Aviation, Grades JP-4 and JP-5
MIL-F-25656 - Fuel, Aircraft Turbine and Jet Engine, Grade JP-6
MIL-I-27686 - Inhibitor, Fuel System Icing

STANDARDS

FEDERAL

Fed. Test Method Std. No. 791 - Lubricants, Liquid Fuels, and Related Products; Methods of Inspection, Sampling and Testing

(Copies of specifications, standards, drawings and publications required by contractors in connection with specific procurement functions, should be obtained from the procuring activity or as directed by the contracting officer.)

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2.2 Other publications. The following documents form a part of this standard to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or requests for proposal shall apply.

STEEL STRUCTURES PAINTING COUNCIL (SSPC) SPECIFICATIONS

- SSPC-SP5-63 - Steel Structures Painting Council Surface Preparation Specifications No. 5 White Metal Blast Cleaning
- SSPC-Vis 1-63T - Visual Standards of Surface Preparation

(Application for copies should be addressed to the Steel Structures Painting Council, 4400 Fifth Avenue, Pittsburgh, Pennsylvania 15213.)

AMERICAN SOCIETY FOR TESTING AND MATERIALS ASTM STANDARDS

(Application for copies should be addressed to American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

3. DEFINITIONS

(Not applicable)

4. GENERAL REQUIREMENTS

4.1 Compatibility of test fuels and coating system. The coating shall be tested in accordance with 5.1 in the following test fuels:

- a. JP-4, MIL-T-5624
- b. JP-5, MIL-T-5624
- c. JP-6, MIL-F-25656
- d. 115/145 Avgas, MIL-G-5572

4.1.1 Acceptance criteria. The coating shall not degrade the test fuels beyond the limits specified in table I when tested as specified in 5.1. Degradation of the test fuels shall be based on analyses of the test fuels exposed to the coating and analogous samples of fuels not exposed to the coating, except where otherwise specified (see 5.1.7 and 6.1). After exposure to the test fuels, the coating shall show no evidence of degradation and shall be physically comparable to a control panel of the same coating not exposed to the test fuels.

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Characteristic	Test Fuels			
	MIL-T-5624 JP-4 JP-5		MIL-F-25656 JP-6	MIL-G-5572 115/145
Thermal stability:				
Pressure drop, in. Hg, max. Temperature 425/525°F	--	--	10	--
Deposit rating, max.	--	--	3	--
Water separation index Modified - min.	70	85	--	--
Existent gum				
Unwashed, mg/100 ml, max.	7	--	--	4
Washed, mg/100 ml, max.	3	--	--	2
Bromine depletion, % max.	--	--	--	10

4.2 Immersion resistance of coating to test fluids. The coating shall be immersed in accordance with 5.2 in the following test fluids:

- | | |
|--|--------|
| a. Icing inhibitor, MIL-I-27686 | 195 ml |
| Distilled water | 275 ml |
| b. Icing inhibitor, MIL-I-27686 | 1 ml |
| JP-4, Turbine fuel, MIL-T-5624 | 450 ml |
| c. Standard test fluid, TT-S-735, type II
(60% isooctane, 5% benzene, 20% toluene,
15% xylene by volume) | 350 ml |
| Distilled water | 150 ml |

4.2.1 Acceptance criteria. The coating shall exhibit satisfactory resistance to the test fluids in 4.2 when tested as specified in 5.2 and shall show no evidence of degradation.

5. TEST PROCEDURES

5.1 Compatibility of test fuels and coating system. The coating under consideration shall be applied to steel test panels as specified in 5.1.1 exposed to the applicable test fuels in suitable sealed containers and stored under specified conditions. An analogous set of containers of the test fuels shall be stored concurrently without test panels. Subsequent analyses of the expose

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and unexposed test fuels, except where otherwise specified, shall determine whether the coating has contributed to fuel degradation. After exposure to the test fuels the panels shall be examined for compliance with 4.1.1.

5.1.1 Panel preparation. Use 5 by 10 and 10 by 12-1/2 inch panels cut from 18 gauge (50 ± 5 mils) hot rolled steel conforming to QQ-S-698. Vapor degrease and then abrasive blast both sides of the panels to basis metal to produce a clean white blast finish in conformance with SSPC-SP5-63/SSPC-Vis 1-63T standards, preparatory to coating. Where the coating under consideration is covered by military specification, apply coating in accordance with applicable specification requirements as to method of application, drying time between coats, and dry film thickness. For proprietary coatings not covered by military specification, apply coating in accordance with manufacturers instructions as to method of application, drying time between coats, and dry film thickness. Dip coat all panel edges with the coating under test using extra coats where necessary to assure adequate protection. Air dry the completely coated panels for not less than 18 hours at $50 \pm 5\%$ relative humidity and $75 \pm 5^\circ\text{F}$ then age the panels in an air circulating oven at $140 \pm 5^\circ\text{F}$ for 4 days prior to exposure to the test fuels and test fluids.

5.1.2 Test containers. Use commercially available 2 and 10 gallon unlined open head round steel pails with lug covers or covers secured by means of a bolted or snap-on-ring. Thoroughly clean all interior surfaces by vapor degreasing or solvent wiping prior to use. Replace the cover gasket with one that is fuel resistant (Viton or Buna-N Rubber) or use a sheet of metal foil over top of pail prior to securing cover.

5.1.3 10-Gallon test assemblies. Use 10-gallon pails as in 5.1.2 for analyses required of JP-6 test fuel (see table I). Insert two 10 by 12-1/2 inch panels coated and aged as in 5.1.1, diagonally in the pail resting the panel tops on opposite walls of the container and the panel bottoms adjacent to one another in the center of the bottom of the pail. Fill the pail with 10 gallons of the JP-6 test fuel and secure cover as in 5.1.2.

5.1.4 2-Gallon test assemblies. Use 2-gallon pails as in 5.1.2 for analyses required of JP-4, JP-5 and 115/145 Avgas (see table I). Insert one 5 by 10 inch panel, coated and aged as in 5.1.1 diagonally in the pail resting the panel top on the wall of the container and the panel bottom along the bottom edge of the opposite wall. Fill the pail with 2 gallons of applicable test fuel and secure cover as in 5.1.2.

5.1.5 Control test assemblies. Fill and seal a duplicate control set of 10-and 2-gallon pails with the applicable test fuels as in 5.1.3 and 5.1.4 but without coated test panels.

5.1.6 Test conditions. Store the 2 and 10 gallon container assemblies with and without coated test panels at ambient temperature ($80 \pm 10^\circ\text{F}$) and 35 days and gently swirl each container assembly every 7 days to agitate the contents.

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5.1.7 Panel examination and analyses of test fuels. At the completion of the 35 day storage period as in 5.1.6 examine the exposed panels and compare with unexposed coated panels for compliance with 4.1.1. Analyze the exposed (5.1.3 and 5.1.4) and control (5.1.5) test fuels in accordance with the test methods and procedures specified in table II and 5.1.7.1 except as follows. Thermal stability analysis of JP-6 test fuel shall be made on the exposed fuel only. In the event the exposed fuel fails thermal stability requirements, thermal stability analysis of the unexposed fuel shall be made to establish whether the coating has contributed to degradation of the JP-6.

Table II. Test methods

Test method	Test method number	
	Fed. T.M. Std. No. 791	ASTM
Thermal stability of gas turbine fuels	3464	D1660
Water separation characteristics of aviation turbine fuels (modified)	3256	--
Gum existent in fuel (jet evaporation)	3302	D381

5.1.7.1 Bromine depletion. Bromine shall be determined on the exposed and control aviation gasolines as follows:

5.1.7.1.1 Apparatus. Assemble apparatus in a hood as in figure 1 for decomposition of bromides. Fit a teflon sealed corrosion resistant stirrer in the center neck of the 250 ml flask. Fit a rubber stopper in the side neck equipped with a tube for delivery of liquid ammonia and a vent tube of Drierite. Assemble bromide titration apparatus, as in figure 2, capable of accurate slip-drop delivery of silver nitrate. Use a continuous indicating pH meter (Beckman Model H-2 or equal) with a glass and a silver reference electrode to follow the titration (a heavy gage silver wire may be used as the reference electrode provided the immersed contact area of the wire is cleaned with fine emery paper and rinsed with water).

5.1.7.1.2 Materials and reagents. The following materials and reagents shall be required:

dry ice and acetone for cooling
Drierite
ammonia
sodium
alcohol
sodium hydroxide pellets
ammonium persulfate
sodium arsenite (10%)
nitric acid, approx. 20%
silver nitrate, 0.05N

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5.1.7.1.3 Procedure. Cool the flask in a dry ice/acetone slurry. Using a pre-weighed lecture bottle, charge flask with about 20 g of liquid ammonia added at rates up to 2 g/min. Weigh lecture bottle periodically to control the quantity added. Remove rubber stopper assembly, charge flask with a cube of freshly cut sodium (about 0.5 cm in dimensions) and slowly rotate stirrer by hand to partially dissolve the sodium. To avoid excessive boiling, slowly add as from a pipette exactly 50 ml of Avgas under test, insert a one hole rubber stopper fitted with a vent tube of Drierite, remove cooling bath, and start stirrer. As soon as the ammonia begins to evaporate (evidenced by a change in color of the Drierite) remove the tube of Drierite from the vent. Continue stirring until all the ammonia has evaporated (about 22 to 26 minutes). If the blue color of sodium in liquid ammonia disappears during early stages of the evaporation, add another cube of sodium. Next, add 5 ml alcohol to decompose excess sodium, follow with 30 ml water and stir to aid extraction of inorganic salts. Wash down stirrer into flask and transfer contents to a separatory funnel. Collect the aqueous layer and extract the organic layer with two additional 30 ml portions of water. Combine the three aqueous extracts, evaporate to about 50 ml and cool slightly. If the resultant solution is cloudy or contains a precipitate, filter and wash the filter paper with several small portions of hot water. Evaporate the combined filtrate and washings to about 50 ml and cool slightly. Add 2 g sodium hydroxide pellets followed by 2 g ammonium persulphate and stir until dissolved. Boil solution for 15 minutes making certain that the solution is alkaline to litmus or methyl orange during boiling, adding additional sodium hydroxide if necessary. Formation of a reddish solid or dark solution during the boiling stage will not adversely affect the analysis. Cool solution slightly, add 10 ml of 10 percent sodium arsenite solution, boil for 10 minutes and cool to room temperature. Acidify to phenolphthalein with 20 percent nitric acid, start stirrer, introduce pH meter electrodes, adding more nitric acid if necessary to obtain a pH reading of from 0.0 to 1.5. Next, titrate the bromides with 0.05N silver nitrate and record the apparent pH reading obtained per ml of silver nitrate. Plot the data, determine appropriate inflection points and calculate bromides present as follows:

$$\text{g Br/sample} = 0.003996B$$

or

$$\text{g Br/gal/60}^\circ\text{F} = 0.3025B[1+0.00065(t-60)]$$

where

B = ml 0.05N silver nitrate to titrate bromides

t = °F of Avgas when sampled

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5.2 Immersion resistance of coating to test fluids. The coating shall be applied to 2-3/4 by 6 inch steel test panels prepared and coated as specified in 5.1.1, exposed to the specified test fluids (see 4.2) in sealed jars and stored under specified conditions. Subsequent visual and physical examination of the coating exposed to the test fluids compared to panels of the coating not exposed shall determine whether the coating conforms to 4.2.

5.2.1 Test procedure. Use wide mouth quart glass specimen jars (Raytheon Jar)^{1/} nominally 3-7/8 inches in diameter and 6-3/4 inches deep with metal screw caps. The test fluids of the composition as specified in 4.2 shall each be added to separate glass jars. Insert one 2-3/4 by 6 inch coated panel diagonally in each jar and secure with metal screw cap. By this assembly, the lower half of the coated panel will be immersed in the test fluid and the upper half will be subjected to the vapor phase. The glass jars shall be maintained at ambient temperature ($80 \pm 10^{\circ}\text{F}$) for 35 days and panels examined every 7 days through the glass container for visual evidence of coating degradation. After 35 days immersion in the test fluids the panels shall be removed and the coating immediately examined for compliance with 4.2.1.

^{1/}Catalog No. 3956 specimen jar, Macalaster Bicknell Co., Millville, N. J. or equal.

6. NOTES

6.1 Acceptance limitations. Acceptance of a candidate protective coating under this standard merely constitutes an indorsement that military fuels carried in containers lined with the coating will not be adversely degraded or contaminated by the coating and that the coating was found to be adequately resistant to the various fuel and test fluid environments to which it was subjected. Such an indorsement shall not be construed to mean that a coating acceptable under this standard is additionally qualified for lining containers used for the bulk transportation and storage of fuels. Coatings which require qualification for such service must conform to this standard and in addition must conform to specified requirements of applicable military specifications for coatings intended for such service.

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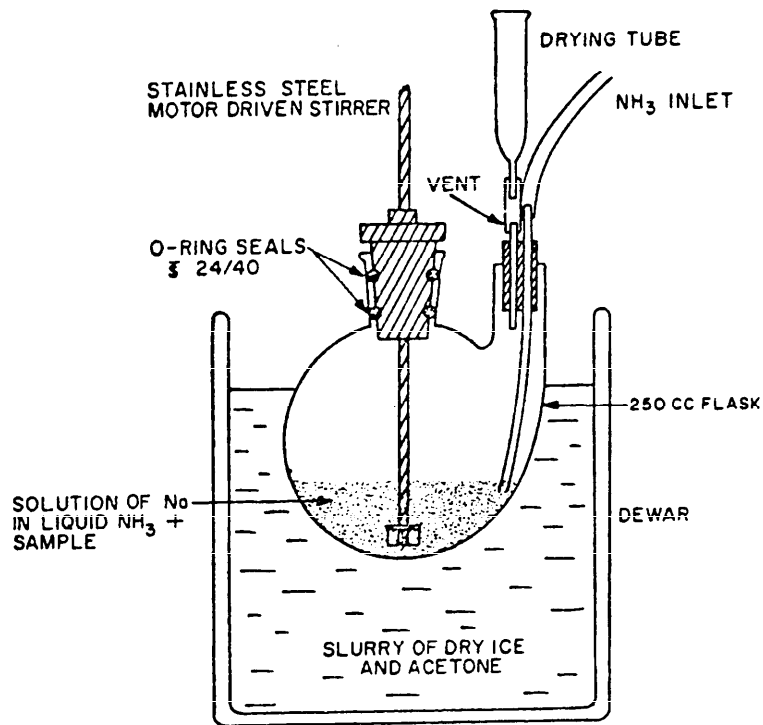


Figure 1 - Apparatus for the decomposition of organic halides

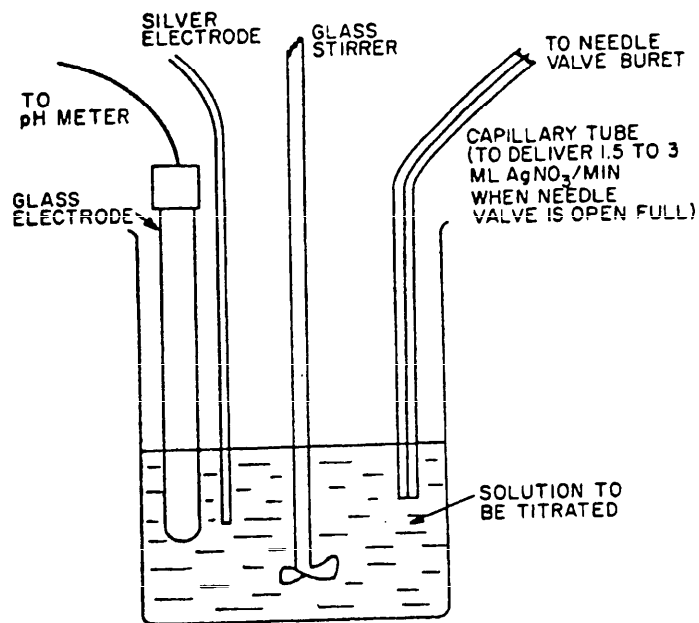


Figure 2 - Potentiometric titration assembly

SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 119-R004
INSTRUCTIONS		
This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity.		
SPECIFICATION MIL-STD-1262, Degradation Tests for Fuels and Fuel Container Coatings (Test Procedure)		
ORGANIZATION		CITY AND STATE
CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT \$
MATERIAL PROCURED UNDER A		
<input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
1 HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE? A GIVE PARAGRAPH NUMBER AND WORDING		
B RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES		
2 COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
3 IS THE SPECIFICATION RESTRICTIVE? <input type="checkbox"/> YES <input type="checkbox"/> NO IF "YES" IN WHAT WAY?		
4 REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)		
SUBMITTED BY (Printed or typed name and activity)		DATE

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