

MIL-L-3918  
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## MILITARY SPECIFICATION

# LUBRICATING OIL, INSTRUMENT, JEWEL BEARING, NONSPREADING, LOW TEMPERATURE

*This specification has been approved by the Department of Defense  
 for use of the Departments of the Army, the Navy, and the Air Force.*

### 1. SCOPE

1.1 This specification covers one grade of low-temperature nonspreading lubricating oil for use on jewel bearing instruments.

### 2. APPLICABLE DOCUMENTS

2.1 The following specifications and standards of the issue in effect on date of invitation for bids, form a part of this specification.

### SPECIFICATIONS

#### FEDERAL

NN-B-591 — Boxes, Fiberboard, Wood-Cleated (for Domestic Shipment).  
 NN-B-621 — Boxes, Wood, Nailed and Lock-Corner.  
 NN-B-631 — Boxes, Wood, Wire-bound (for Domestic Shipment).  
 QQ-C-576 — Copper Plates, Sawed Bars, Sheet and Strips.  
 QQ-S-636 — Steel, Carbon (low carbon), Sheets and Strips.  
 VV-L-791 — Lubricants, Liquid Fuels, and Related Products; Methods of Inspection, Sampling and Testing.

LLL-B-631 — Boxes, Fiber Corrugated (for Domestic Shipment).  
 LLL-B-636 — Boxes, Fiber, Solid (for Domestic Shipment).  
 PPP-B-601 — Boxes, Wood, Cleated-Plywood.  
 PPP-B-676 — Boxes, Set-Up, Paperboard.

#### MILITARY

JAN-P-103 — Packaging and Packing for Overseas Shipment — Boxes; Wood-Cleated; Solid Fiberboard.  
 JAN-P-106 — Packaging and Packing for Overseas Shipment — Boxes; Wood, Nailed.  
 MIL-B-107 — Boxes, Wood, Wire-bound (Overseas Type).  
 JAN-P-108 — Packaging and Packing for Overseas Shipment — Boxes; Fiberboard (V-Board and W-Board), Exterior and Interior.  
 JAN-P-120 — Packaging and Packing for Overseas Shipment — Cartons, Folding, Paperboard.  
 MIL-B-138 — Boxes, Wood, Fiberboard-Lined for Overseas Shipment.  
 MIL-B-10377 — Boxes; Wood-Cleated, Veneer, Paper Overlaid.  
 MIL-L-10547 — Liners, Case, Waterproof.

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## STANDARDS

## MIL-STD-129 — Marking for Shipment and Storage.

(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.)

## 3. REQUIREMENTS

**3.1 Qualification.** — Lubricating oil furnished under this specification shall be a product which has been tested and has passed the qualification tests of this specification.

**3.2 Chemical Composition.** — The chemical composition of the oil shall conform to the requirements shown in Table I.

TABLE I. Chemical composition

Ingredient	Weight percent
Benzyl phenylundecanoate .....	59.95+0.05
Diethylene glycol di-n-caproate .....	39.45+0.05
Dodecylpiperidine stearate .....	0.40+0.01
p-tertiary-butyl catechol .....	0.20±0.01

**3.3 Viscosity.** — The viscosity of the oil shall not be less than 9.5 centistokes or more than 10.5 centistokes when measured at 100°F. as specified in 4.4.1 Method 305.2.

**3.4 Neutralization value.** — The neutralization value of the oil shall not exceed 0.60 when tested as specified in 4.4.1 Method No. 5105.3.

**3.5 Evaporation.** — The evaporation loss shall not exceed 2.0 percent when tested as specified in 4.4.1 Method No. 351.1 for 22 hours at a temperature of 210°F. (99°C.)

**3.6 Copper strip corrosion.** — The oil shall not cause appreciable discoloration of copper when tested as specified in 4.4.2 for three hours at 212°F. (100°C.)

**3.7 Corrosion and oxidation stability.** — When tested as specified in 4.4.3, the oil shall meet the following requirements.

**3.7.1 Corrosion.** — The change in weight of copper and steel specimens shall not exceed 0.5 milligrams per square centimeter of surface. There shall be no pitting, etching or visible corrosion of the metal surfaces. A slight discoloration of the metal surface shall not be cause for rejection.

**3.7.2 Oxidation.** — The viscosity of the oil at 100°F. shall not have changed more than ±5 percent from the original viscosity, upon completion of the corrosion oxidation test. The change in the neutralization of the oil at the end of the test shall not exceed 0.15. The volatile acids evolved during the test shall not require more than 0.5 milligram of KOH, per gram of oil, for neutralization.

**3.8 Spreading on metal.** — When tested as specified in 4.4.4 the average diameter increase of three drops of the oil on polished steel shall not exceed 5 percent in 30 days. The residue from the oxidation test shall not spread more than 5 percent in a week.

**3.9 Low temperature stability.** — A dry sample of the oil shall not crystallize, separate, or develop precipitates of any kind when maintained at a temperature of -40°F. (-40°C.) for 48 hours as specified in 4.4.5. A cloud shall not be cause for rejection.

## 3.10 Ingredients. —

**3.10.1 Benzyl phenylundecanoate.** — The benzyl phenylundecanoate shall be a product not boiling below 200°C. at ½ mm. pressure and vaporizing completely below 210°C. at ½ mm. pressure. The neutralization number shall be less than 0.10.

**3.10.2 Diethylene glycol di-n-caproate.** — The diethylene glycol di-n-caproate shall be a product not boiling below 160°C. at 1½ mm. pressure. The neutralization number shall be less than 0.10. As soon as possible after completion of the preparation, 0.0025 grams of the p-tertiarybutyl catechol shall be added per cc. of the diethylene glycol di-n-caproate.

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**3.10.3 Dodecylpiperidine stearate.** — The dodecylpiperidine stearate shall be a reaction product of equi-molar quantities of dodecylpiperidine boiling between 135°C. and 138°C. at 1.0 mm. pressure and stearic acid melting between 69°C. and 70°C.

**3.10.4 Para-tertiary-butyl catechol.** — The p-tertiary-butyl catechol shall melt in the temperature range of 56°C. to 57°C.

### 3.11 Workmanship. —

**3.11.1** The ingredients used in the manufacture of this product shall be carefully procured from responsible sources.

**3.11.2** The component materials shall be intimately mixed and processed to produce a clear uniform product.

**3.11.3** The product shall be clear and free from sediment, perfume, dye or otherwise undesirable materials and shall be generally suitable for the lubrication of jewel bearings in timepieces and other instruments.

## 4. QUALITY ASSURANCE PROVISIONS

**4.1 Classification of tests.** — The inspection and testing of oil under this specification shall be classified as follows:

- (a) **Qualification tests:** Qualification tests performed on samples submitted for approval as qualified products.
- (b) **Acceptance tests:** Acceptance tests are those tests performed on individual lots of oil which have been submitted for acceptance.

**4.2 Qualification tests.** — Qualification tests shall consist of all tests of this specification.

**4.2.1 Qualification Test Sample.** — Qualification test samples shall consist of 10 ounces of the finished oil. Samples shall be appropriately identified with the manufacturer's

own code number and any additional identification required by the letter of authorization (see paragraph 6.3).

**4.3 Acceptance tests.** — Acceptance tests shall consist of all tests of this specification. The inspector may at his discretion release a production lot prior to completion of the SPREADING ON METALS (see 4.4.4) test provided the contractor assumes full responsibility for conformance of the material to the test. The inspector may accept certification from the manufacturer as to composition and conformance of ingredients to the physical properties specified herein, but the right is reserved to supervise the blending of and to conduct tests on the ingredients of the oil.

**4.3.1 Sampling instructions.** — A sample shall consist of eight ounces of oil from each lot.

**4.3.2 Inspection.** — Inspection shall be in accordance with Method 9601 of Specification VV-L-791.

### 4.4 Test Methods. —

**4.4.1** The following listed tests shall be conducted in accordance with the methods described in Specification VV-L-791:

Test	Method No.
Viscosity (Kinematic) .....	305.2
Neutralization Value (Acid and Base Numbers) by Color-Indicator Titration .....	5105.3
Evaporation loss .....	351.1

**4.4.2 Copper strip corrosion.** — Copper strip corrosion shall be determined in accordance with Method 5313 of Specification VV-L-791 except that the test specimen shall be one-fourth inch wide and one inch long and only 15 milliliters of oil shall be placed in the 25 by 150-millimeter test tube. The test temperature shall be  $212^{\circ} \pm 0.5^{\circ}\text{F.}$  ( $100 \pm .2^{\circ}\text{C.}$ )

**4.4.3 Corrosion and Oxidation stability.** —

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**4.4.3.1 Apparatus.** — The glass cell in which the oil is oxidized shall be described in Figure I and auxiliary apparatus arranged similar to the setup described in Figure II. The condenser shall be a straight bore Liebig condenser with a 300 mm. water jacket and modified to have a ground glass joint fitted to the delivery end. The receiving tube shall be an 18 mm x 150 mm pyrex tubes. The heating unit may be either an oil bath or an aluminum block maintained at  $212^{\circ}\text{F.} \pm 0.2^{\circ}\text{F.}$  All glass equipment shall be thoroughly cleaned and dried before each test.

**4.4.3.2 Preparation of Metal Strips.** — The metal catalyst and corrosion strips shall be approximately  $\frac{1}{2}'' \times 1\frac{1}{2}'' \times \frac{1}{16}''$  prepared from steel conforming to Specification QQ-S-636 and copper conforming to Specification QQ-C-576. Each strip shall be polished, degreased and weighed as follows:

Polish the surface of the strip with No. 150 emery cloth followed by No. 320 grit paper, then place, in a clean glass container filled with c.p. benzene and boil for at least 5 minutes. Transfer the strip directly from the boiling benzene to another clean glass container filled with high grade petroleum ether and boil for approximately one minute. Remove the strip from the boiling petroleum ether and weigh to the nearest tenth of a milligram. Immerse the strip in c.p. benzene until ready to be placed in the oil to be tested. During all operations following polishing the strips shall be handled by forceps or tongs to avoid contamination.

**4.4.3.3 Procedure.** — Weigh a strip of steel and copper cleaned in accordance with section 4.4.3.2 and place them in the bottom of the oxidation cell. Arrange the two strips so that their faces do not touch. Weigh  $25.0 \text{ grams} \pm 0.05 \text{ g.}$  of the oil directly into the cell, then place the cell into the heating unit and assemble the apparatus as described in Figure II. A convenient source of air is first filtered then passed through a combustion furnace containing a copper oxide catalyst followed by an absorber to remove water

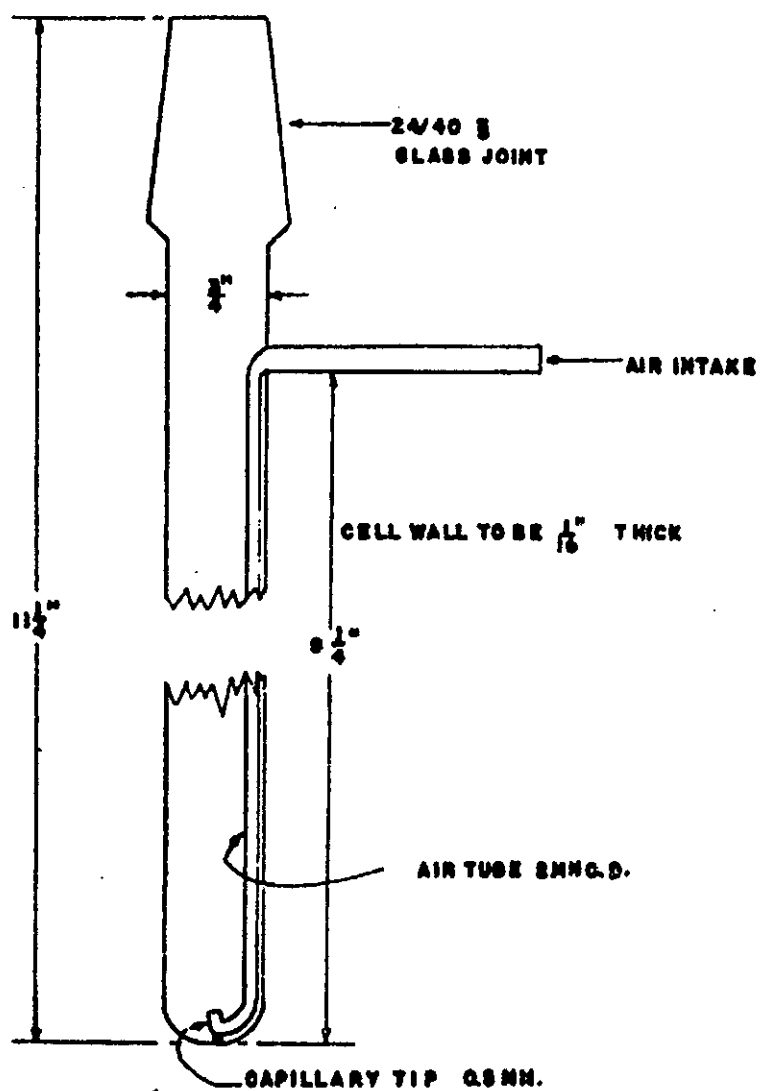
and  $\text{CO}_2$ . Regulate the air flow at 20 ml/min.  $\pm 2 \text{ ml/min.}$  by any suitable device and pass the air into the oxidation cell. Place  $30.0 \text{ ml} \pm 0.02 \text{ ml.}$  of approximately 0.1 N standard potassium hydroxide in the receiving tube. At the conclusion of every 24 hour period a new tube shall be installed and the potassium hydroxide in the removed tube shall be titrated with standard acid. The amount of volatile acids evolved during the 168 hours test shall be calculated as milligrams of potassium hydroxide per gram of oil tested. At the conclusion of the test, 168 hours, the cell shall be removed, and the oil observed for changes in color, odor, and formation of sludges or precipitates. Test the oxidized oil for change in viscosity at  $100^{\circ}\text{F.}$  The neutralization number shall be determined in accordance with Method No. 5105.3 of Specification VV-L-791. Examine the strips for corrosion, oxidation and changes of the metallic surfaces. Wash the strips in precipitation naphtha (B.P.  $125^{\circ}\text{--}135^{\circ}\text{F.}$ ) then in a one to one mixture of reagent grade benzene and acetone, dry, weigh and record the weight loss per square centimeter of surface.

#### 4.4.4 Spreading on Metal. —

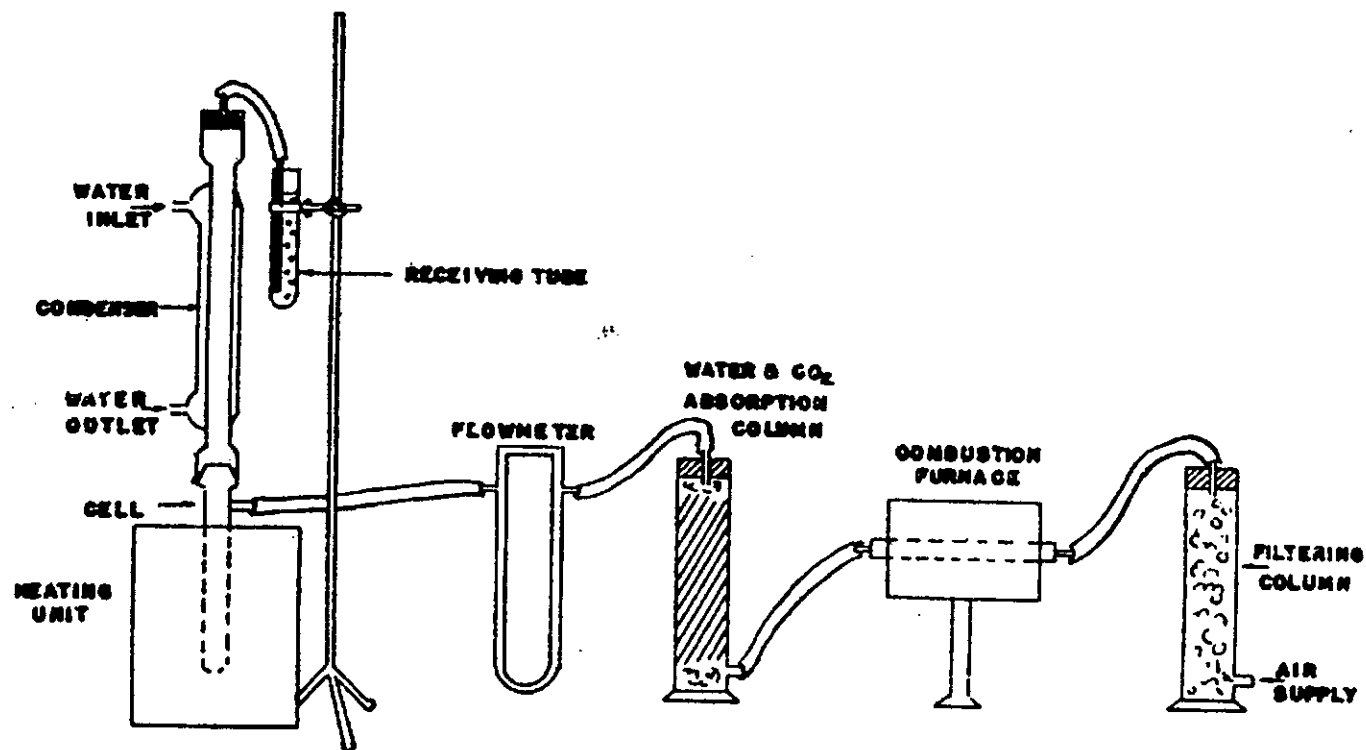
**4.4.4.1 Preparation of Metal Surface.** — A steel surface with a minimum area of 0.25 square inch is polished with metallographic papers Nos. 3/0 and 4/0 in the order named and finally with Fisher's "Gamal" or equivalent polishing alumina on a wheel covered with heavy broadcloth. After polishing, the surface is washed with water, swabbed with wet absorbent cotton, and rinsed thoroughly with distilled water. The surface is then rinsed with 95 percent C.P. methanol and dried for 15 minutes in an oven at  $105^{\circ}\text{C.}$  The specimen is then allowed to cool to room temperature in a desiccator containing magnesium perchlorate.

**4.4.4.2 Procedure.** — Place three drops of the oil on the surface with a clean platinum wire. The drops shall be between 1.0 and 2.0 mm. in diameter. (A platinum wire

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OXIDATION TEST CELL  
FIG. 1



**OXIDATION TEST**  
**FIG. 2**



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0.005 inch in diameter is suitable for depositing a drop of this size.) After recleaning the wire, place, two drops of c.p. tri-ortho-cresyl phosphate on the surface in the same manner. Cover the surface and drops to keep out dust and let stand for 15 minutes. Measure and record the diameters of each of the five drops using a suitable measuring apparatus with an accuracy of one percent. transfer the covered test surface to an air bath maintained at  $45 \pm 2^\circ\text{C}$ . Examine the drops after 24 hours. If the oil appears to be spreading appreciably and the tri-ortho-cresyl phosphate standard is not, the oil is definitely not in the nonspreading category. If both the oil and the standard appear to be spreading, the test should be repeated, on a freshly prepared surface. If no evidence of spreading is observed, the test is continued and the diameters of the drops observed at one week intervals.

**4.4.4.3 Reporting Results.** — Calculate the percent increase in the diameter of each drop as follows:

$$\text{Percent increase} = \frac{D_N - D_0}{D_0} \text{ where}$$

$D_N$  = diameter of drop after n days

$D_0$  = diameter of drop after 15 minutes

Report the average of the values for the three drops as the percent of spreading in the specified number of days.

**4.4.5 Low Temperature Stability.** — A dry 50 ml. sample of the oil shall be maintained at a temperature of  $-40^\circ\text{F}$ . ( $-40^\circ\text{C}$ .) for 48 hours in a stoppered flask. At the end of that time the sample shall be examined visually for conformance to the requirements.

## 5. PREPARATION FOR DELIVERY

**5.1 Packaging.** — Unless otherwise specified, the oil shall be packaged in  $\frac{1}{2}$  ounce brown glass bottles fitted with a screw cap. The screw cap shall be fitted with a cork gasket or its equivalent and an uncoated

metal foil inner-liner; materials which react with this oil must not be used. Each bottle shall be packaged in a snug fitting folding carton conforming to Specification JAN-P-120. Not more than 12 cartons shall be further packaged in a set-up box conforming to Specification PPP-B-676.

### 5.2 Packing. —

**5.2.1 Immediate use.** — Unless otherwise specified, boxes shall be packed for shipment in standard commercial containers so constructed as to insure acceptance by common or other carrier for safe transportation at the lowest rate to the point of delivery. The gross weight of the shipping containers shall not exceed approximately 50 pounds.

**5.2.2 Domestic shipment.** — Oil packaged to meet the requirements specified in 5.1 shall be packed in domestic type exterior containers conforming to Specifications NN-B-631, LLL-B-631, LLL-B-636 or MIL-B-10377. Exterior containers shall be of minimum cube and tare consistent with the protection required. As far as practical, exterior containers shall be of uniform shape and size and contain identical quantities. The gross weight of each pack shall not exceed the limitation of the applicable box specification. Strapping and closure shall be in accordance with the appendix of the applicable container specification. When fiberboard containers are used, the fiberboard shall meet the special requirements table of Specifications LLL-B-631 or LLL-B-636, as applicable.

**5.2.3 Overseas shipment.** — Oil packaged to meet the requirements specified in 5.1 shall be packed in export type shipping containers conforming to Specifications JAN-P-103, JAN-P-106, JAN-P-108, MIL-B-138, MIL-B-107, or PPP-B-601. As far as practical, exterior containers shall be of uniform shape and size, be of minimum cube and tare consistent with the protection required, and contain identical quantities. The gross weight of each pack shall be

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limited to approximately 50 pounds. Strapping and closure shall be in accordance with the appendix of the applicable container specification. Containers, except those conforming to Specification JAN-P-108, shall be provided with a case liner conforming to Specification MIL-L-10547, and shall be sealed in accordance with the appendix thereto.

**5.3 Marking.** — Marking of all containers shall be done in accordance with MIL-STD-129. In addition the following requirements will apply.

**5.3.1 Interior containers.** — Each bottle, carton, and set-up box shall be labeled as follows:

Lubricating Oil, Instrument, Jewel-  
Bearing, Non-Spreading, Low-  
Temperature  
Specification MIL-L-3918  
Stock No.  
Contract No.  
Manufactured (Date)  
Name of Manufacturer

**5.3.2 Exterior Containers.** — Each exterior container shall be marked "Glass — Handle with Care".

**6. NOTES**

**6.1 Intended Use.** — The oil covered by this specification is intended for the lubrication of steel pivot and jewel bearing combinations in timepieces and other fine instruments. It does not spread on highly polished clean metal or jewel surfaces but may tend to spread when used on unpolished metal surfaces, especially when contaminated with dust or other foreign materials. It will allow operation of most instrument mechanisms at temperatures as low as  $-40^{\circ}\text{F}$ .

**6.2 Ordering data.** — Procurement documents should specify the following:

- (a) Title, number, and date of this specification.

- (b) Quantity of oil required (the unit of purchase being the fluid ounce).

- (c) Whether oil is to be packed for domestic shipment (immediate use), domestic shipment (storage), or overseas shipment.

**6.3 Provisions for Qualification test.** — In the procurement of products requiring qualification, the right is reserved to reject bids on products that have not been subjected to the required tests and found satisfactory for inclusion on a Qualified Products List. The attention of suppliers is called to this requirement, and manufacturers are urged to arrange to have the products that they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or orders for the products covered by this specification. Requests for qualification of products under this specification should be addressed to the Chief of the Bureau of Aeronautics, Navy Department, Washington 25, D. C. Attn: Aer-AE-424.

**6.4 Notice.** — When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any 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 manufacture, use, or sell any patented invention that may in any way be related thereto.

**Custodians:**

Army—Ordnance Corps  
Navy—Bureau of Aeronautics  
Air Force

**Other interest:**

Army—EMQSig  
Navy—OrShS



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## APPENDIX I

## PREPARATION OF LUBRICANT N-22a

## 1. INTRODUCTION

The composition of Lubricant N-22a, a synthetic non-spreading instrument oil, is given, together with the procedure used in compounding it. The laboratory procedures used in the synthesis of the ingredients employed in the compounded lubricant are detailed, since most of these compounds are not commercially available. Some possible commercial sources of supply for these intermediates are suggested.

## 2. COMPOSITION

This lubricant, which was developed under the Mellon Institute Fellowship No. 303, is a mixture of about sixty percent benzyl phenylundecanoate and forty percent diethylene glycol di-n-caproate, to which is added small amounts of dodecylpiperidine stearate (for oiliness) and p-tert-butyl catechol (as an anti-oxidant). The exact composition of the fluid on a *weight percent* basis is as follows:

Ingredient	Percent by weight
Benzyl phenylundecanoate .....	59.95 ± 0.05
Diethylene glycol di-n-caproate ....	39.45 ± 0.05
Dodecylpiperidine stearate .....	0.40 ± 0.01
p-tert-butyl catechol .....	0.20 ± 0.01

## 3. BLENDING

The esters which form the bulk of this lubricant should have passed any desired tests, such as a copper strip test, neutralization number, etc., before the oil is compounded. The required weights of the esters are then mixed in a suitable vessel. One half of the required weight of that oxidation inhibitor (p-tert-butyl catechol) is already present in the mixture since it was dissolved in the diethylene glycol di-n-caproate immediately after the preparation of that ester. The remaining portion of the catechol and the dodecylpiperidine stearate, heated just to melting, can be dissolved in a small portion of the ester mixture which has been warmed to 90-100°C., and this solution added to the main batch of the lubricant. The mixture is stirred until the additives have dissolved; the lubricant is then filtered and bottled.

## 4. VISCOMETRIC DATA

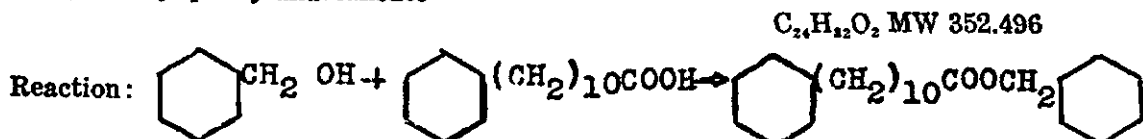
The following information is representative of the viscometric properties of Lubricant N-22a and its components:

Sample	Viscosity, Centistokes			
	100°F.	0°F.	-20°F.	-40°F.
Lubricant N-22a .....	9.96	178*	500*	2600*
(B) Diethylene glycol di-n-caproate .....	16.71	528*	2189*	12000*
(A) Benzyl phenylundecanoate .....	5.24	47.1	99.8	Solid
Blend of 60% (A); 40% (B) .....	9.78	170.2	477*	2365*

\* Non-Newtonian, values shown are only approximate.

## 5. CHEMICAL SYNTHESIS

## (a) Benzyl phenylundecanoate



Twelve hundred and fifty (1250) grams (4.75 moles) of phenylundecanoic acid are placed in a five liter flask together with 576

grams (5.22 moles) of benzyl alcohol, 170 ml. of toluene and 0.8 grams of p-toluene sulfonic acid monohydrate. Chips of porous plate are

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added to prevent bumping and the flask is fitted with a water trap and a reflux condenser. The mixture is boiled under reflux until no more water can be removed, and until titration of a small sample indicates that the percentage of free acid in the esterification mixture has fallen below 0.3 percent. For this test, a 1.00 gram sample is dissolved in 10 ml. of 95 percent ethyl alcohol and titrated with 0.07 N NaOH, using phenolphthalein as the indicator.

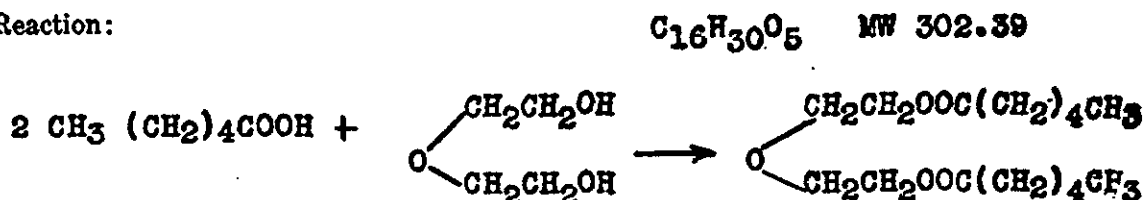
When the esterification is complete, about 5 grams of moist potassium carbonate are added to the reaction mixture and it is distilled. The fraction boiling at 200-210°C. at

1½ mm. is regarded as the product;  $n_{20}^D$  1.5210. The distilled ester must show less than 0.10 neutralization number by the above titration method; the saponification number shall be  $159 \pm 1.0$ . If the percent acid is higher than this, or if the color is poor, the ester can be purified by chromatographic absorption on a column of activated, alumina and fullers earth.

The quantities shown above can be increased several fold without difficulty, the reaction being carried out in glass lined or stainless steel pilot plant kettles of five or ten gallon capacity.

## (b) Diethylene glycol di-n-caproate

Reaction:



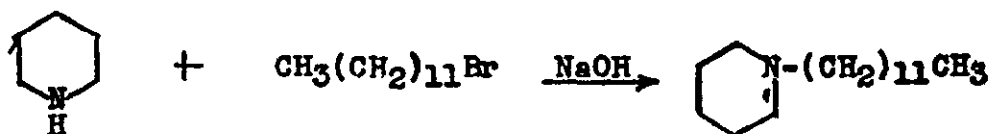
"A mixture of 636 grams (6.0 moles) of diethylene glycol, 1470 grams (12.5 moles) of n-caproic acid, 500 ml. of toluene, and 1.0 gram of p-toluene sulfonic acid monohydrate is placed in a five liter flask. Chips of porous plate are added to prevent bumping and the flask is fitted with a water trap and a reflux condenser. The mixture is boiled under reflux until (no more) water has been removed. Five grams of potassium carbonate are added and the distillation is continued. The fraction boiling at 160-170°C. at 1½ mm. is regarded as the product. The

distillate should be water white;  $n_{20}^D$  1.4400. The neutralization number is determined by the procedure previously described shall be less than 0.10. If the ester is off color or has a higher acid content, it can be purified by chromatography as described above. The product is immediately protected by the addition of 0.0025 gram of tert-butyl catechol per ml. of ester. This reaction has been carried out on a five fold scale, using a ten gallon stainless steel kettle and reflux equipment."

## (c) Dodecylpiperidine

$\text{C}_{17}\text{H}_{35}\text{N}$     MW 253.46

Reaction:



Forty-four grams (0.52 moles) of piperidine and 45 ml. of water were mixed in a 500 ml. round bottom flask to which was

attached an efficient reflux condenser and a dropping funnel. Then 140 grams of n-dodecyl bromide was added to the solution

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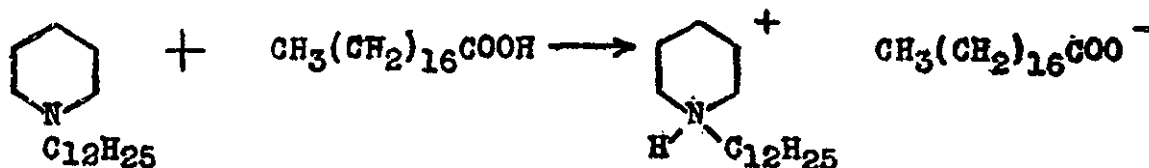
in the flask, and the two-phase mixture which resulted was heated to the boiling point. While the contents of the flask were kept boiling under reflux, there was a slowly added through the dropping funnel a solution of 51 grams (0.9 moles) of potassium hydroxide in 51 ml. of water. The mixture was cooled, the aqueous layer separated and discarded, and the oil layer distilled over

10 grams of solid potassium hydroxide.

"The fraction which boiled over the range 135-150°C at 1 mm. was collected and was redistilled. A fraction boiling at 135-138°C at 1 mm. was collected. The yield is about 105 grams (80 percent);  $n_{20}^D$  1.4588. Since this material deteriorates upon standing, it should be reacted with the stearate immediately.

#### (d) Dodecylpiperidine stearate

Reaction:



Stearic acid (8.86 grams, 0.031 mole) is melted in a 50 ml. beaker on a hot plate and 7.9 grams (0.031 mole) of dodecylpiperidine is added to the well stirred liquid. Heat is evolved and a yellow liquid is formed which becomes a slightly yellow, wax-like product on cooling. The yield is quantitative (16.7 grams).

n-Caproic acid

- (1) The Dow Chemical Company  
Midland, Michigan
- (2) The Edwal Laboratories, Inc.  
732 Federal Street  
Chicago, Illinois
- (3) The Edcan Laboratories  
10 Pine Street  
S. Norwalk, Connecticut
- (4) Carbide and Carbon Chemicals  
Corp.  
30 East 42nd Street  
New York 17, New York

## 6. RAW MATERIALS

One of the difficulties which anyone who attempts to prepare Lubricant N-22a must face, is the fact that the two esters which form the backbone of the composition are not available commercially, and even some of the starting materials for their synthesis are very difficult to obtain. It is with this thought in mind that a suggested list of supplier is presented below in the hope that some of the experience gained here in the search for these raw materials may be of assistance to others.

Neither Benzyl phenylundecanoate nor Diethylene glycol di-n-caproate are available commercially.

Benzyl alcohol and diethylene glycol are available from several sources.

#### Phenylundecanoic acid

This substance is actually a mixture of the 10- and 11-phenylundecanoic acids, and is the product which results from the reaction of benzene with undecylenic acid in the presence of aluminum chloride.

- (1) Sinclair Refining Company  
630 Fifth Avenue  
New York 20, New York
- (2) East Kodak Company  
Rochester 4, New York  
(No. 5352)
- (3) Carbide and Carbon Chemicals  
Corporation  
30 East 42nd Street  
New York 17, New York

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## **Dodecylpiperidine stearate**

**This substance is available from :**

**Bios Laboratories, Inc.  
17 West 60th Street  
New York 23, N. Y.**

**The approximate quantities of the ingredients used in blending to make one gallon of Lubricant N-22a are as follows:**

<b>Benzyl phenylundecanoate.....</b>	<b>4.8 pounds</b>
<b>Diethylene glycol di-n-caproate.....</b>	<b>3.2 pounds</b>
<b>Dodecylpiperidine stearate.....</b>	<b>14.4 grams</b>
<b>p-ter-Butyl catechol.....</b>	<b>7.2 grams</b>