

MIL-L-82661(OS)
31 January 1977
 Superseding
 (see section 6)

MILITARY SPECIFICATION

LECITHIN, TECHNICAL

This specification is approved for use by the Naval Sea Systems Command, Department of the Navy and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers one type of lecithin referred to herein as "the material".

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. 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.

STANDARDS

Military

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129	Marking for Shipping and Storage
MIL-STD-1218	ACS Chemicals

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

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

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Ordnance Station, Standardization Division (611), Indian Head, Maryland 20640 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 6810

MIL-L-82661(OS)

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D 70-72

Specific Gravity of Semi-solid Bituminous
Materials

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

INTERNAL REVENUE SERVICE

IRSP No. 368

Formulas for Denatured Alcohol and Rum

(Application for copies should be addressed to the Superintendent
of Documents, Government Printing Office, Washington, DC 20402.)

NATIONAL MOTOR FREIGHT TRAFFIC ASSOCIATION, INC., AGENT
National Motor Freight Classification

(Applications for copies should be addressed to American Trucking
Associations, Attn: Tarriff Order Section, 1616 P Street, Washington,
DC 20036.)

UNIFORM CLASSIFICATION COMMITTEE, AGENT
Uniform Freight Classification

(Applications for copies should be addressed to the Uniform Classi-
fication Committee, Room 1106, 222 South Riverside Plaza, Chicago, IL
60606.)

(Technical society and technical association specifications and
standards are generally available for reference from libraries. They
are distributed among technical groups and using Federal agencies.)

3. REQUIREMENTS

3.1 Material. The material shall be vegetable lecithin amber to
reddish colored in a viscous liquid form.

3.2 Chemical and physical properties. The chemical and physical
properties shall be in accordance with TABLE I.

3.3 Workmanship. The material shall be uniform, free from contami-
nation, foreign material, or any other defect that would prevent its use
for the purpose intended.

4. QUALITY ASSURANCE PROVISIONS

TABLE I. Chemical and physical properties.

Property	Values	
	Minimum	Maximum
Viscosity at 25°C, poise	--	150
Specific gravity, 25°/25°C	1.02	1.04
Water content, wt%	--	1.0
Acid value, mg KOH/g	--	32
Benzene insoluble, wt%	--	0.2
Acetone insoluble, wt%	62.0	--

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Inspection conditions. Unless otherwise specified (see 6.2), all inspections shall be performed under the following conditions:

- a. Temperature: Room ambient 18 to 35°C (65 to 95°F)
- b. Altitude: Normal ground
- c. Vibration: None
- d. Humidity: Room ambient to 95 percent relative, maximum

4.3 Sampling.

4.3.1 Lot Unless otherwise specified in the contract (see 6.2), a lot shall consist of all material manufactured in one continuous production run or in one batch, under essentially identical conditions, from the same raw materials, and to be offered for acceptance at one time. Several batches, manufactured from the same raw materials, may be blended to form a uniform larger batch which shall then constitute a lot for inspection purposes.

4.3.2 Sampling. Sampling for quality conformance inspection shall be in accordance with inspection level I of MIL-STD-105. The sample unit shall be one unit package or container of material. Each sample shall consist of sufficient material to perform the quality conformance tests as specified in 4.4.

MIL-L-82661(OS)

4.4 Quality conformance inspection. Each sample obtained in accordance with 4.3.2 shall be subjected to the tests and examination of 4.5. The acceptable quality level (AQL) shall be 2.5% defective. When specified in the contract (see 6.2), the contractor shall furnish test reports showing quantitative results for all quality conformance tests specified for each lot of material.

4.5 Tests. Unless otherwise specified herein, all chemicals shall be ACS grade in accordance with MIL-STD-1218.

4.5.1 Visual examination. All samples shall be visually examined to verify conformance to the workmanship requirements.

4.5.2 Viscosity determination at 25°C. The apparatus and procedures for the determination of viscosity at 25°C shall be in accordance with the following:

a. Apparatus:

1. Brookfield Viscometer, Model RVF, Brookfield Engineering Laboratories, Stoughton, Massachusetts, or equivalent.
2. Constant temperature water baths, maintained at 25°C.

b. Procedure:

1. Remove the wood handle and mount the viscometer on a ring stand. Carefully attach the Brookfield No. 7 spindle.
2. Pour the sample into a 600 ml beaker, place in the water bath at 25°C and stir with a thermometer until the sample comes to the test temperature. Use care to avoid stirring air bubbles into the sample.
3. Lower the viscometer on the ring stand until the surface of the sample is in the narrow section of the spindle. Level the viscometer, and operate at 20 RPM for determinations in the 100 to 2000 poise range. During operation maintain the spindle depth in the sample so that the polymer level climbs midway up the narrowest section of the spindle.
4. Obtain a series of readings until the value is constant.

c. Calculations:

1. Using the No. 7 spindle at 20 rpm, and reading the 0 to 100 scale, calculate the viscosities as follows:

$$\text{Viscosity at 25°C, poise} = (20) (\text{scale reading})$$

If a different spindle or speed is used, consult the chart accompanying the instrument for the proper multiplying factor. Convert the resulting centipoise to poise by dividing by 100.

MIL-L-82661(OS)

4.5.3 Specific gravity (25/25°C). Specific gravity shall be determined at $25.0 \pm 0.1^\circ\text{C}$ in accordance with ASTM D 70-72.

4.5.4 Water content. The water content shall be determined in accordance with the following:

a. Reagents:

1. Methanol
2. Carrier solution - 3:1 mixture by volume of pyridine and methanol
3. Stabilized Karl Fischer reagent, diluted to a water equivalent of 2.5 to 3.0 milligram/milliliter (mg/ml) with Karl Fischer Diluent.
4. Sodium tartrate

b. Apparatus: Aquameter, Beckman Model KF-4, or equivalent.

c. Standardization of Karl Fischer reagent: Add 100 ml of methanol to the reaction vessel. Neutralize the methanol with dilute Karl Fischer reagent by automatically titrating to a 30 second end point using the Aquameter. Carefully add 0.09 to 0.11 g of sodium tartrate weighed to the nearest 0.1 mg, to the neutralized methanol. Dissolve the sodium tartrate in the methanol by setting the stirring action to the highest speed which will not cause splashing or bubble formation. Automatically titrate with dilute Karl Fischer Reagent to a 30 second end point. Record the volume of dilute Karl Fischer Reagent. Repeat the standardization procedure until three determinations agree within 0.05 mg/ml.

d. Calculate the water equivalent of the dilute Karl Fischer reagent as follows:

$$A = \frac{156.6B}{C}$$

where: A = water equivalent of the dilute Karl Fischer reagent, mg/ml
 B = weight of sodium tartrate, g
 C = volume of dilute Karl Fischer reagent used to titrate the standard, ml
 156.6 = factor for sodium tartrate

e. Procedure: Add 100 ml of the carrier solution to the reaction vessel. Purge with dry air. Neutralize the carrier solution with dilute Karl Fischer reagent by automatically titrating to a 30 second end point using the Aquameter. Add 6 to 10 g of sample weighed to the nearest 0.01 g to the carrier solution in the reaction vessel. Set the stirring action the same as that used in the standardization. Dissolve the sample and automatically titrate with standardized dilute Karl Fischer reagent to a 30 second end point. Record volume of dilute Karl Fischer reagent.

MIL-L-82661(OS)

- f. Calculate the water content as follows:

$$\text{water content, wt\%} = \left[\frac{AD}{1000E} \right] \times 100$$

where: A = water equivalent of the dilute Karl Fischer reagent (from d. above), mg/ml
 D = volume of standardized dilute Karl Fischer reagent used to titrate sample, ml
 E = sample weight, g
 1000 = multiplication factor to convert g to mg

- g. Report the results of a minimum of 2 determinations and their average.

4.5.5 Acid value. Acid value shall be determined in accordance with the following:

- a. Reagents:

1. Standardized 0.1 normal (N) sodium hydroxide (NaOH)
2. Ethyl alcohol, 95 percent denatured, Formula D-30 conforming to IRSP No. 368 (neutralized to phenolphthalein just prior to use).
3. Petroleum ether.
4. Phenolphthalein indicator, 1 percent in ethanol.

- b. Apparatus:

1. Erlenmeyer flask, 250 ml.
2. Microburet, 10 ml.

- c. Procedure: Warm (not over 60°C) the sample, if necessary, to soften the material and then mix thoroughly. Weigh 1.8 to 2.0 g of sample, to the nearest 0.1 mg, into a 250 ml Erlenmeyer flask. Dissolve in 50 ml of petroleum ether by shaking gently. Then add 50 ml of neutral ethyl alcohol and shake to mix. Add four drops of phenolphthalein indicator and titrate while shaking with 0.1 N NaOH to the first pink color which persists for approximately 5 seconds. The end point is fairly easily ascertained by adding the bulk of the NaOH solution rapidly until near the end point, and then slowly down to four, then two drops at a time. During the early addition of the NaOH, the mixture will become clear. As additional NaOH is added, two phases will occur. At this point, slow incremental addition of the NaOH should begin and the lower phase observed for the end point.

MIL-L-82661(OS)

d. Calculation: Acid value, $\text{mg} \frac{\text{KOH}}{\text{g}} = \frac{56.1VN}{W}$

where: V = volume of standardized NaOH, ml
 N = normality of standardized NaOH
 W = weight of sample, g
 56.1 = molecular weight of potassium hydroxide (KOH)

- e. Report the results of a minimum of 2 determinations and their results.

4.5.6 Benzene insolubles. The benzene insolubles shall be determined in accordance with the following:

- a. Apparatus: Filtering crucible, Selas, 30 ml, fine porosity, or equivalent.
 b. Reagent: Benzene.
 c. Procedure: Weigh 10 to 11 g of sample, to the nearest 0.1 mg, into a 250 ml beaker. Add 100 ml of benzene and stir until dissolved. Filter the sample quantitatively through a Selas crucible and wash the remaining insoluble matter into the crucible using two 25 ml portions of benzene. Maintain suction for approximately 5 minutes until the sample is free of benzene. Dry the sample at $105^\circ \pm 5^\circ \text{C}$ for a minimum of 1 hour, cool and weigh.

d. Calculation: Benzene insolubles, $\text{wt}\% = \frac{(A-B)}{W} \times 100$

where: A = weight of crucible and insoluble matter, g
 B = weight of crucible, g
 W = weight of sample, g

- e. Report the results of a minimum of 2 determinations and their average.

4.5.7 Acetone insolubles. Acetone insolubles shall be determined in accordance with the following:

- a. Reagent: Acetone, presaturated with purified acetone insoluble material (phosphatides)
1. Purification of phosphatides - Purify phosphatides from previous acetone insoluble determination by dissolving approximately 5 g in 10 ml of petroleum ether and reprecipitating with 25 ml of acetone. Ascertain the precipitate is free from conglomerates before further processing. Transfer the precipitate to two centrifuge tubes using additional acetone to facilitate the transfer. Stir thoroughly and make to volume of 45 ml, stir, chill for a minimum of 15 minutes in ice bath at 0° to 5°C , stir again and centrifuge for a minimum of 5 minutes. Decant acetone soluble, break up solids with stirring rod, refill tubes

MIL-L-82661(OS)

with acetone, stir, chill, centrifuge and decant as before. The solids, after the second centrifugation, require no further processing and may be used, as is, for presaturating acetone.

2. Saturation of acetone - add purified phosphatides to acetone at 0° to 5°C. 5.0g of the phosphatides are sufficient for about 16 liters of acetone. Maintain at 0° to 5°C for about 2 hours, shaking vigorously at approximately 15 minute intervals. Decant through a rapid filter paper avoiding transfer of any of the solids to the paper and conducting the filtration under refrigerated conditions (0° to 5°C) in order to maintain the same conditions for saturation as described under the procedure.

b. Apparatus:

1. Centrifuge, motor driven with head to hold specified centrifuge tubes and capable of operating at 1900 ± 100 revolutions per minute (rpm).
2. Centrifuge tubes, pyrex, heavy wall, round bottom with lip, 40 ml.
3. Stirring rods, 5mm diameter, convenient length.
4. Buret, 50 ml.
5. Beakers, 250 and 400 ml.

- c. Procedure: Mix the sample thoroughly. Heat the sample, if necessary, but not above 60°C, to facilitate mixing. Weigh 1.9 to 2.0g of mixed sample to the nearest 0.1 mg, into a centrifuge tube which has been tared with a stirring rod. Add 15 ml of saturated acetone from a buret. Warm in a water bath until the lecithin melts, but avoid evaporation of acetone. Stir until the material is completely disintegrated, then place in ice water bath and chill for a minimum of 5 minutes. Remove the tube from the bath and add about one half of the final required volume of chilled (0° to 5°C) saturated acetone. Stir well to complete dispersion of remaining particles. Make to volume of 45 ml with chilled (0° to 5°C) acetone, stir and return tube and contents to ice bath at 0° to 5°C for a minimum of 15 minutes. At the end of the 15 minutes chilling period stir again while in the bath; remove rod and centrifuge immediately at 1900 ± 100 rpm for a minimum of 5 minutes. Decant the acetone soluble material into a clean beaker. Break up the centrifuged solids with the assigned stirring rod and refill the centrifuge tube to the 45 ml mark with chilled (0° to 5°C) acetone; stir well

and repeat washing and centrifuge steps. Following the second centrifugation, pour off the acetone, return the stirring rod to the tube and break up the solids. Place the tube and contents in a horizontal position on a laboratory bench until the excess of acetone evaporates. Mix again and place in a forced draft oven at $105^{\circ} \pm 3^{\circ}\text{C}$ until constant weight is obtained (usually 30 to 45 minutes). Cool to room temperature in an efficient desiccator and weigh immediately.

d. Calculation: Acetone insolubles, wt% = $\left[\frac{A - B}{W} \right] \times 100$

where: A = weight of dried residue, g

B = percent benzene insolubles (as determined in 4.5.6)

W = weight of sample, g

e. Report the results of a minimum of 2 determinations and their average.

4.6 Packaging inspection. The packaging, packing and marking shall be inspected to verify conformance with the requirements of Section 5.

5. PACKAGING

5.1 Packaging and packing. Unless otherwise specified in the contract (see 6.2), packaging and packing shall be level C.

5.1.1 Level C. Unless otherwise specified in the contract or order (see 6.2), packaging and packing of lecithin shall be in accordance with standard commercial practice applicable to the type of material. The packaging and packing shall be of such construction and materials that the contents will be adequately protected against loss or contamination. Container size shall be as specified in the contract (see 6.2). Containers shall conform to Uniform Freight Classification, National Motor Freight Classification or to rules of other carriers applicable to the mode of transportation and shall be suitable for indoor storage at 32°C (90°F) maximum for a minimum of 2 years.

5.2 Marking. In addition to any special marking required by the contract (see 6.2), each container shall be marked in accordance with MIL-STD-129. Marking shall include, but not be limited to, the following information:

- a. Title, number and date of this specification
- b. Manufacturer's name and location
- c. Material trade name
- d. Net weight
- e. Lot number, batch number(s), and date of manufacture
- f. Storage conditions
- g. Contract or purchase order number

MIL-L-82661(OS)

6. NOTES

6.1 Intended use. Lecithin in accordance with this specification is intended for use as an ingredient in a solid propellant for the CCU-22/A impulse cartridge and other cartridge devices.

6.2 Ordering data. Procurement documents should specify the following:

6.2.1 Procurement requirements.

- a. Title, number and date of this specification
- b. Quantity required
- c. Place of delivery
- d. Inspection conditions when other than as specified (see 4.2)
- e. Lot size if other than as specified (see 4.3.1)
- f. Packaging requirements if other than as specified (see 5.1)
- g. Size of container required (see 5.1)
- h. Any special markings required (see 5.2)

6.2.2 Contract data requirements. Items of deliverable data required by this specification are cited in the following paragraph herein:

<u>Paragraph</u>	<u>Data Requirement</u>	<u>Applicable DID*</u>
4.4	Quality conformance inspection data	-

*DID's (Data Item Description/DD Form 1664) for the above requirements will be documented in the applicable ADL (Authorized Data List). Such data will be delivered as identified on completed (numbered) DID's when specified on DD Form 1423 (Contract Data Requirements Lists) and incorporated into applicable contracts.

6.3 Suggested source of supply. A product that has met the requirements of this specification in past procurement actions is Lecithin, Grade C3F-UB manufactured by Central Soya. This information is for the convenience of the procuring activity and is not to be construed as a waiver of any requirement of this specification nor as any limitation of additional potential sources of supply.

6.4 Supersession information. MIL-L-82661 is intended to be used in lieu of AS 3060 (Code Ident 30003) dated 6 December 1974 for Naval Sea Systems Command procurement.

Custodian:
Navy - OS

Preparing Activity:
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