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MINISTRY OF DEFENCE

ERDL/TRIM/PROP/RM/8
PROVISIONAL SPECIFICATION FOR LECITHIN

E.R.D.L.
SUTARWADI, PUNE-411 008.

Encl to ERDL/TECH/QARD/6129
Dated: 9·11·92

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0. FOREWORD

0.1 This specification has been prepared by Explosives Research & Development Laboratory, Sutarwadi, Pune-411 008.

0.2 This specification will be approved by the Ministry of Defence after appropriate sealing action by Controllerate of Quality Assurance (Military Explosives) and will be mandatory for use by Defence Services.

0.3 Before sealing action, any queries regarding this specification may be referred to Explosives Research and Development Laboratory, Sutarwadi, Pune-411 008.

1. SCOPE

1.1 This specification is intended to govern supply and Quality Assurance of Lecithin.

1.2 INTENDED USE

This material is intended for use in booster and sustainer propellants of "TRISHUL" Missile.

2. RELATED SPECIFICATIONS AND DOCUMENTS

2.1 The related documents as mentioned in clause 2.2 are those applicable at the date of publication of this specification. It is contractor's/manufacturer's responsibility to confirm their current applicability and to obtain from CQA(ME), Kirkee, the information concerning any change that may be necessary due to cancellation, replacement or supersession of any of these documents.

2.2 The following related specifications have been referred to in the preparation of this specification.

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i) MIL-L-3061 B(OS) LECITHIN (FOR USE IN EXPLOSIVES).
13 June 1969

ii) MIL-L-82661 (OS) LECITHIN, TECHNICAL
31st Jan 77

2.3 Copies of related specifications are obtainable as follows :

Specifications	Source of Supply
MIL Standard Specifications	Directorate of Standardization Ministry of Defence, DHQ PO, NEW DELHI-110 011.

(Any queries regarding the above mentioned MIL Standard specifications may be referred to Explosives Research and Development Laboratory, Pune-411 008).

3. MATERIAL/FINISH

3.1 The material will be vegetable, lecithin amber to reddish colored and in a viscous form.

4. MANUFACTURE

4.1 Lecithin shall be manufactured by a process which will produce the product conforming to this specification.

4.2 Nothing in this specification shall relieve the manufacturer of his responsibility for the safety of his operations.

5. TENDER SAMPLE

5.1 Unless otherwise instructed, the contractor/supplier shall submit free of all charges, two tender samples of 500 g each conforming to this specification in all respects.

5.2 Acceptance of tender will denote that the tender sample has

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been accepted as standard of supply in accordance with terms of this specification.

6. QUALITY ASSURANCE

6.1 INSPECTION

6.1.1 Lecithin and the containers in which it is packed shall be subject to Inspection by and to the approval of Quality Assurance Officer/Quality Assurance Authority.

6.1.2 Samples of the material and of the containers may be taken from any portion of the batch/lot/consignment.

6.1.3 If, on examination, any sample be found not to conform to this specification, the whole batch/lot/consignment may be rejected.

6.1.4 The foregoing provisions shall equally apply to the prime contractors and sub-contractors, if any.

6.2 SAMPLING

6.2.1 Normally two representative samples each of 250 g supplied free of charge shall be drawn from each batch/lot/consignment of supply/manufacture. However, the number of samples to be drawn shall be at the discretion of the Quality Assurance Officer/Quality Assurance Authority.

6.3 TEST REQUIREMENTS

6.3.1 Samples taken from any portion of the batch/lot/consignment of the material shall conform to clause 3 and in addition shall conform to the following test requirements.

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Sl. No.	Characteristics	Passing standard	Reference to Test Method
1	2	3	4
1.	Moisture, percent by mass, Max.	1.0	Appendix-I (Ref. ERDL Method)
2.	Viscosity at 25°C, poise, Max.	130	Appendix-II (Ref.MIL-L-82661(OS)
3.	Benzene Insoluble matter, percent by mass, Max.	0.2	Appendix-III (Ref.MIL-L-82661(OS))
4.	Acid value mg.KOH/g of sample, Max.	32	Appendix-IV (Ref.MIL-L-82661(OS))
5.	Acetone insoluble matter, percent by mass, Max.	62	Appendix-V (Ref.MIL-L-3061 B(OS))
6.	Lecithin, percent by mass, Max.	99	Appendix-VI (Ref.ERDL Method)

7. SUPPLIER'S INSPECTION OF STORES/CONSIGNMENT

7.1 Before tendering the store for inspection, the supplier shall carry out a thorough inspection of each delivery to satisfy himself that the store fully conforms to this specification and shall render a certificate to that effect to the Quality Assurance Officer/Quality Assurance Authority.

8. WARRANTY

The stores supplied against the contract shall deem to have been warranted against defective material and performance by the contractor/manufacturer for a period of 24 months from the date of receipt of the store at the consignee's end and if during this period any of the stores supplied is found defective, the sample shall be replaced by the contractor/manufacturer free of charge at the consignee's premises.

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9. PACKAGING

9.1 The material shall be supplied in a suitable polythene (HDPE) containers or other approved packages containing an approved quantity.

9.2 The inclusion of any foreign matter or impurities in any of these packages will render the whole consignment liable for rejection.

10. MARKING

10.1 All packages containing the material shall be durably and legibly marked with the following details (as applicable):

i) Nomenclature and specification number of the material

ii) Name and address of the consignee

iii) S.O. number and date

iv) Consignment number

v) Lot/Batch number and date of manufacture

vi) Gross and net mass

vii) Consecutive number of package and total number of packages in the consignment.

viii) Date of supply

ix) Contractor's initials or recognised trade mark.

x) Storage temperature limit.

10.2 In addition to the above, the Quality Assurance Officer/Quality Assurance Authority may suggest some more markings/identifications considered suitable at the time of inspection.

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10.3 The marking shall be done by a suitable marking ink.

11. DEFENCE STORES CATALOGUE NUMBER

11.1 Defence Stores Catalogue Number allotted to this store is.....

12. SUGGESTIONS FOR IMPROVEMENT

12.1 Any suggestion for improvement in this particular document may be forwarded to Explosives Research & Development Laboratory, Sutarwadi, Pune-411 008.

13. APPENDICES

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APPENDIX-I

MOISTURE CONTENT

General: For moisture content determination, sample is dissolved in appropriate solvent and moisture content is estimated using Karl Fischer Aquameter.

1. APPARATUS

Mettler DL 40 RC Memotitrator or equivalent.

2. REAGENTS

a) Karl Fischer reagent

b) Specially dried Methanol for Karl Fischer titrations (moisture Max. 0.04 %)

c) Disodium tartarate dihydrate, G.R.

3. PROCEDURE

a) Determination of Factor :

Take 35 ml of methanol in Karl Fischer titration flask. Neutralise the moisture in the solvent with Karl Fischer reagent. Add 50 mg of disodium tartarate and stir for 100 seconds. Titrate with Karl Fischer reagent to a point at which the polarization of double pin platinum electrode does not take place beyond 20 mV within a delay of 15 seconds at the constant polarization current of 2.5 mA.

b) Determination of moisture in sample

Take 35 ml of methanol in Karl Fischer titration flask. After neutralization of moisture in solvent with Karl Fischer

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reagent. add 0.5 g of sample and stir for 100 seconds. Titrate with Karl Fischer reagent under exactly similar experimental conditions applied for determination.

4. CALCULATIONS

a) Determination of Factor :

$$\text{Karl Fischer Factor}_{\text{mg/ml}} = \frac{10 \times m \times 15.6467}{V}$$

where V = the volume of Karl Fischer Reagent, ml

m = mass of disodium tartarate in g (dihydrate)

b) Determination of moisture in sample

$$\text{Total moisture content} = \frac{F \times V \times 100}{m} \times \frac{1}{1000}$$

where F = factor Karl Fischer mg H_2O/ml

V = titre volume of Karl Fischer reagent

m = mass of sample taken, g

Report the results of two determinations and their average.

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APPENDIX-II

VISCOSITY DETERMINATION

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

1 APPARATUS

(i) Brookfield Viscometer, Model LV, Brookfield Engineering Laboratories, Stoughton, or equivalent.

(ii) Constant temperature water bath.

2 PROCEDURE

a) Remove the wooden handle and mount the viscometer on a ring stand. Carefully attach the Brookfield No.7 spindle.

b) 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. Exercise care to avoid any inclusion of air bubbles into the sample.

c) 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 determination in the 100 to 2000 poise range. During operation, maintain the spindle depth in the sample so that the polymer level rises midway up the narrowest section of the spindle.

d) Obtain a series of readings until the value is constant.

3. CALCULATIONS

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

Viscosity at 25°C, centipoise = Factor x dial reading

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

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APPENDIX-III

BENZENE INSOLUBLES

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

1. APPARATUS

a) Sintered glass crucible, G_4

2. REAGENT

a) Benzene (IS 534, 1974)

3. PROCEDURE

Weigh about 10 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 sintered glass crucible (G_4) crucible and wash the remaining soluble 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°C ± 5°C for a minimum of 1 hour, cool and weigh.

4. CALCULATION

$$\text{Benzene insolubles, \% by mass} = \frac{(m_3 - m_2)}{m_1} \times 100$$

where m_1 = mass of sample, g

m_2 = mass of crucible, g

m_3 = mass of crucible and insoluble matter, g

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

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APPENDIX-IV

ACID VALUE

Acid value shall be determined in accordance with the following :

1. REAGENTS

a) Standardized (0.1 N) sodium hydroxide solution(NaOH)

b) Ethyl Alcohol, 95% denatured, neutralised to phenolphthelein.

d) Petroleum ether (40 - 60°C).

d) Phenolphthalein indicator, 1 percent in ethanol

2. APPARATUS

a) Erlenmeyer flask, 250 ml

b) Microburette, 10 ml capacity

3. 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 the 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 the contents. Add four drops of phenolphthalein indicator and titrate, with 0.1N NaOH to the first pink colour 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

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appear. At this point, slow incremental addition of the NaOH solution should begin and the lower phase observed for the end point.

4. CALCULATION

$$\text{Acid value (mg KOH/g of sample)} = \frac{56.1\ VN}{m}$$

where V = Volume of standardized NaOH solution (ml).

N = Normality of standardized NaOH solution.

m = Mass of the sample g

Report the results of a minimum of two determinations and their average.

APPENDIX-V

ACETONE INSOLUBLE MATTER

1. APPARATUS

a) Centrifuge machine

b) Centrifuge tubes

c) Ice water bath

d) 250 ml beaker

2. REAGENTS

a) Petroleum ether (40 - 60°C)

b) Acetone (IS 170, 1986)- The solvent is chilled to 0°C to 5°C before use.

3. PROCEDURE

Warm the sample to soften the product (not over 60°C) and then mix thoroughly. Accurately weigh 2 grams of well mixed sample into the centrifuge tube which has been previously tared with a stirring rod. Add 3 ml of petroleum ether and stir well to dissolve soluble matter. Thorough mixing is essential, and will require about 15 minutes. Add exactly 15 ml of chilled acetone from a burette, mix thoroughly, and place the tube in a dish containing ice water. Add acetone, which has been previously chilled to 0° to 5°C, to the 50 ml mark on the tube, stirring during the addition. Place in an ice water bath at 0°C for 15 minutes, centrifuge for 10 minutes, at a speed sufficient to clear the solution during this period and then decant the acetone into a 250 ml beaker which has been previously dried at

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105° C ± 2°C dess kept in desiccator and weighed. Refill the centrifuge tube to the 50 ml mark with acetone, stir well and repeat as above. Decant the acetone into the 250 ml beaker. Evaporate the acetone on a water bath. A stream of clean dry air may be used to facilitate the removal of the solvent. Dry in an oven at 105° ± 2°C for one hour, cool to room temperature in a desiccator and weigh the acetone soluble matter.

4. CALCULATIONS

Acetone soluble matter,% by mass, A = $\frac{\text{Mass of acetone extract} \times 100}{\text{Mass of sample}}$

Acetone insoluble matter, percent = 100-(A + B + C)

A = Percent acetone soluble matter

B = Percent moisture, from moisture clause

C = Percent Benzene insolubles

105°C ± 2°C dessc kept in desiccator and weighed. Refill the centrifuge tube to the 50 ml mark with acetone, stir well and repeat as above. Decant the acetone into the 250 ml beaker. Evaporate the acetone on a water bath. A stream of clean dry air may be used to facilitate the removal of the solvent. [illegible] oven at 105° ± 2°C for one hour, cool to room temperature in a desiccator and weigh the acetone soluble matter.

4. CALCULATIONS

Acetone soluble matter, % by mass, A = $\frac{\text{Mass of acetone extract} \times 100}{\text{Mass of sample}}$

Acetone insoluble matter, percent = 100 - (A + B + C)

A = Percent acetone soluble matter