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ROUTING AND TRANSMITTAL SLIP

Date

3 OCT 83

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Initials

Date

- 1. *Dr. Young*
- 2.
- 3.
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 FPMR (41 CFR) 101-11.206

H-13-28

MIL-I-51064C
26 March 1973
SUPERSEDING
MIL-M-51064B
20 December 1965

MILITARY SPECIFICATION
INSECTICIDE, MALATHION

This specification is mandatory for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers two grades of malathion insecticide, chemically known as O,O-dimethyl phosphorodithioate of diethyl mercaptosuccinate.

1.2 Classification. Malathion shall be of the following grades as specified (see 6.2):

- Grade A - Premium.
- Grade B - General purpose.

2. APPLICABLE DOCUMENTS

2.1 Government 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.

SPECIFICATIONS

FEDERAL

- PPP-D-729 - Drums: Metal, 55 Gallon (for Shipment of Noncorrosive Material).

STANDARDS

MILITARY

- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
- MIL-STD-129 - Marking for Shipment and Storage.

THIS DOCUMENT CONTAINS ~~4~~ PAGES

FSC 6640

(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 date of invitation for bids or request for proposal shall apply.

FEDERAL INSECTICIDE, FUNGICIDE, AND RODENTICIDE ACT

(The Federal Insecticide, Fungicide, and Rodenticide Act of 1947 is under the jurisdiction of the Environmental Protection Agency, Pesticides Office, Washington, DC 20250.)

UNIFORM FREIGHT CLASSIFICATION RULES

(Application for copies should be addressed to the Uniform Classification Committee, Room 116, 222 South Riverside Plaza, Chicago, Illinois 60606.)

NATIONAL MOTOR FREIGHT CLASSIFICATION RULES

(Application for copies should be addressed to the American Trucking Associations, Inc., Tariff Order Section, 1616 P Street, N.W., Washington, DC 20036.)

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM) STANDARDS

- D891-59 - Specific Gravity of Industrial Aromatic Hydrocarbons and Related Materials.
- D1193-70 - Reagent Water.
- D1533-61 - Water in Insulating Liquids (Karl Fischer Method).

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

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

3. REQUIREMENTS

3.1 Chemical and physical characteristics. Malathion shall conform to the chemical and physical characteristics of table I when tested as specified therein.

Table I. Chemical and physical characteristics

Characteristic	Requirement		Test paragraph
	Grade A	Grade B	
Purity, percent by weight, minimum	95.0	95.0	4.2.4.1
Diethyl fumarate, percent by weight, maximum	1.5	1.5	4.2.4.2
Acidity (as sulfuric acid), percent by weight, maximum	0.6	0.6	4.2.4.3
Water, percent by weight, maximum	0.1	0.1	4.2.4.4
Methyl mercaptan, parts per million, maximum	10.0	---	4.2.4.5
Iron, parts per million, maximum	10.0	10.0	4.2.4.6
Specific gravity at 25°/25° C	1.225+	1.225+	4.2.4.7
	0.010	0.010	

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier 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 specified requirements.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the malathion produced by one manufacturer, at one plant, from the same materials, and under essentially the same manufacturing conditions in a period of no more than 24 hours provided the operation is continuous. In the event the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.2.2 Sampling.

4.2.2.1 For examination of preparation for delivery. Sampling for examination of preparation for delivery shall be conducted in accordance with MIL-STD-105.

4.2.2.2 For test. Sampling for test shall be conducted in accordance with table II. A representative specimen of approximately 1 liter shall be removed from each sample container and placed in a suitable clean, dry container labeled to identify the lot and container from which it was taken.

Table II. Sampling for test

Number of containers in batch or lot	Number of sample containers
2 to 25	2
26 to 150	3
Over 150	5

4.2.3 Inspection procedure.

4.2.3.1 For examination of preparation for delivery. The sample unit shall be one filled shipping container. Sample containers and the preparation for delivery thereof shall be examined for the following defects using an AQL of 1.5 percent defective:

- (a) Contents per container not as specified
- (b) Container not as specified
- (c) Container closure not as specified
- (d) Container damaged or leaking
- (e) Interior coating missing or not as specified
- (f) Exterior paint coating missing or not as specified
- (g) Marking incorrect, missing, or illegible

4.2.3.2 For test. Each sample specimen taken in 4.2.2.2 shall be tested as specified in 4.2.4. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.2.4 Tests. Water in accordance with ASTM D1193 and reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Tests shall be conducted as follows:

4.2.4.1 Purity. (See 6.4) Use chloride-free water in all applications for this test. Standardize the silver nitrate solution potentiometrically and determine the inflection point in the same manner as described in (b) and (c) for the malathion determination.

(a) Ion exchange resin. Convert "Amberlite" IRA400 (chloride form, see 6.5), 3 to 5 percent crosslinked and 20 to 50 mesh size, to the nitrate form as follows: Transfer the resin to a large chromatographic column and pass approximately 2N nitric acid through the resin bed until all chloride ion is exchanged. (To check completeness of conversion, add 1 milliliter (ml) of 0.1N silver nitrate solution to 10 ml of the eluate. No turbidity should be observed.) Wash the resin bed with water until the pH of the eluated water is the same as that of the water used for washing. Wash the resin bed with four portions of anhydrous ethanol and allow the resin to air dry.

(b) Procedure. Weigh to the nearest 0.1 milligram (mg) approximately 2.0 grams (g) of the specimen into a 50-ml volumetric flask and dilute to the mark with anhydrous ethanol. Shake well to mix. Add approximately 2.0 g of "Amberlite" IRA400 (nitrate form) prepared as specified in (a) to the flask and mix well by moderate shaking for 5 minutes. Allow the resin to settle and transfer a 25-ml aliquot of the supernatant liquid into a 1500-ml beaker. Add 20 ml of 0.5N ethanolic potassium hydroxide solution to the beaker and swirl for exactly 1 minute. Add 100 ml of water to the beaker and 2 drops of phenolphthalein indicator solution (1 g per 100 ml, in ethanol). While stirring mechanically with a magnetic stirring bar, add dropwise 1 to 1 nitric acid until the disappearance of the pink color. Adjust the final pH to 6.5 to 7.0 with 1 to 5 nitric acid or 0.5N ethanolic potassium hydroxide solution, as required, using a pH meter. Carefully add 900 ml of acetone. With mechanical stirring, slowly add, from a buret, 25 ml of 0.1N silver nitrate solution. Stir for 2 minutes. Continue the titration on an automatic recording titrimeter using a Beckman combination electrode, catalog number 39187 or equal (see 6.6). Deliver the silver nitrate solution at a rate no greater than 1 ml per minute until a millivoltage (mv) of -250 is reached. NOTE: 1.000 g of 100 percent malathion consumes about 30.2 ml of 0.1N silver nitrate solution; 1.000 g of 95 percent malathion consumes about 28.7 ml of 0.1N silver nitrate solution.

(c) Data plot. Plot the millivoltage (from about +30 to -250 mv) versus corresponding milliliters of 0.1N silver nitrate solution on linear graph paper. Determine the milliliters of 0.1N silver nitrate solution corresponding to the point of inflection of the plotted curve. If more than one inflection is observable, use the first inflection that occurs between -50 and -150 mv.

volumetric flask, add 5 ml of 3N hydrochloric acid, and make up to volume with water at 25° C. Pour a portion of the acidified solution into a polarographic cell and treat in the same manner as the acidified solutions in (a). Record the current in microamps. Calculate the percent diethyl fumarate as follows:

$$\text{Percent diethyl fumarate} = \frac{C (10^5)}{3W}$$

where: C = Concentration in grams of diethyl fumarate per milliliter, for corresponding current in microamps, of specimen, and

W = Weight of specimen in grams.

4.2.4.3 Acidity. Weigh 10 g of the specimen to the nearest 0.01 g and dissolve in acetone using gentle warming if necessary. Add 75 ml of water and titrate immediately with 0.02N sodium hydroxide solution using methyl red as an indicator. Calculate the percent acidity (as sulfuric acid) as follows:

$$\text{Percent acidity} = \frac{4.5AB}{W}$$

where: A = Milliliters of sodium hydroxide solution used,

B = Normality of sodium hydroxide solution, and

W = Weight of specimen in grams.

4.2.4.4 Water content. Determine percent by weight water content in accordance with ASTM D1533.

4.2.4.5 Methyl mercaptan.

(a) Preparation of standard methyl mercaptan solution. Use a 250-ml gas sampling tube with stopcocks at both ends. Set up a gas sampling apparatus by using Tygon tubing to connect one end of the gas sampling tube to a 250-ml gas leveling bulb filled with redistilled mercury. Fasten the tubing securely with copper wire to protect against the strain caused by the weight of the mercury. Use Tygon tubing to connect the other end of the gas sampling tube to a 3-way stopcock and also to connect the 3-way stopcock to a small cylinder of methyl mercaptan. Open both stopcocks of the gas sampling tube and open the 3-way stopcock to the atmosphere. Raise the gas leveling bulb to displace the air from the gas sampling tube and 3-way stopcock. Open the 3-way stopcock to the gas cylinder. Open the valve on the gas cylinder carefully and lower the gas leveling bulb until the gas sampling tube is partially filled with gas (this step fills the tubing between the cylinder and the stopcock with methyl mercaptan). Open the stopcock again to the atmosphere and raise the mercury level to the stopcock to flush the methyl mercaptan and air out of the system. Repeat

this step in order to remove all the air from the system. Open the 3-way stopcock to the cylinder and fill the gas sampling tube with methyl mercaptan. Maintain the mercury level in the gas leveling bulb at a higher level than that in the gas sampling tube to insure positive pressure. Close the upper stopcock in the gas sampling tube, remove the tubing, and place a septum over the tube exit. Displace the air between the stopcock and the septum by inserting a gas syringe, without a plunger, into this space and then opening the stopcock. The positive methyl mercaptan pressure displaces the small pocket of air instantaneously. The methyl mercaptan can now be sampled through the septum with a gas-tight syringe. Pipet 12.5 ml of 99.5+ percent 2,2,4-trimethylpentane into a 10-ml serum bottle (actual volume of 14.5 ml to cap), stopper with a silicone-coated rubber stopper and seal with an open-centered aluminum seal using a seal crimper. Insert a 10-ml gas syringe into the gas sampling tube, rinse several times, and then draw more than 7.5 ml of methyl mercaptan into the syringe. Withdraw the syringe from the gas sampling tube and adjust the volume to 7.5 ml. Immediately inject the methyl mercaptan into the serum bottle containing the 2,2,4-trimethylpentane. Insert the tip of the needle well below the surface of the liquid so that the methyl mercaptan will dissolve as it bubbles up through the liquid. Withdraw the syringe and vigorously shake the serum bottle for 1 minute. Note the barometric pressure and temperature and calculate the concentration of the standard solution as follows:

$$\text{Concentration of methyl mercaptan in} \quad = \frac{267,000}{P(273 + T)}$$

micrograms per microliter

where: P = Atmospheric pressure in millimeters of mercury and
T = Temperature in °C.

(b) Preparation of calibration curve. Repeat the procedure in (a) transferring 5.0, 2.5, 1.0, 0.75, 0.50 and 0.25 ml quantities of methyl mercaptan into separate sealed 10-ml serum bottles containing 12.5 ml of 2,2,4-trimethylpentane. Calculate the concentration of these standard solutions as follows:

$$\text{Concentration of solution in} \quad = \frac{267,000 A}{7.5P(273 + T)}$$

micrograms per microliter

where: A = Milliliters of methyl mercaptan injected into solution,
P = Atmospheric pressure in millimeters of mercury, and
T = Temperature in °C.

Using the conditions specified in (), measure the chromatographic response of a 1-microliter aliquot of each standard solution. Response should be at least 50 percent of full scale deflection. This is obtained by proper adjustment of the attenuation. Plot the response (peak height) against the

concentration (micrograms of methyl mercaptan). Prepare a new calibration curve each day that the analyses are performed. The standard methyl mercaptan solutions should be refrigerated when not in use. Standard solutions may be used for a period of 2 weeks after preparation provided they are refrigerated and no leaks have developed in the system to disturb the liquid-vapor equilibrium in the stoppered and sealed serum bottle.

(c) Chromatographic conditions. Use a flame ionization gas chromatograph. Recommended chromatographic conditions for an F & M Model 1609 chromatograph are shown in table III. These conditions may be modified, as necessary, if other chromatographs are used.

Table III. Chromatographic conditions

Characteristic	Condition
Column dimensions	6 feet by 1/4 inch ID
Column material	Stainless steel
Column support	60 to 80 mesh "Gas Chrom Z"
Column coating	10 percent "XE-60"
Injection port temperature	100°C
Column temperature	30°C
Detector temperature	160°C
Gas flow rates, ml per min:	
Helium	120
Hydrogen	60
Air	250
Range	10
Attenuation	1X
Retention time of methyl mercaptan peak	34 to 36 seconds
Record chart speed	1-1/2 inches per min (high speed No. 1)

NOTES: (1) The range and attenuation are equivalent to 4×10^{-11} amperes for full scale deflection.

(2) Convert the methyl mercaptan peak height observed to height at attenuation 1 by multiplying peak height by attenuation.

(d) Procedure. Condition the column before using by injecting a 1.0-microliter aliquot of the specimen into the gas chromatograph at 160° C (temperature of column and injection port) and temperature programming the chromatograph at 5° C per minute until the specimen is completely eluted. Repeat this operation from three to five times. Pipet 15 ml of specimen in a 27-ml serum bottle (actual volume of 32 ml to cap), stopper with a silicone-coated rubber stopper, and seal with an open-centered aluminum seal

using a seal crimper. Place in an oven at 56° C for 2 hours. Using a 1-ml gas syringe, inject 0.5 ml of the vapors directly into the chromatograph and measure the chromatographic response using attenuation IX. Calculate the parts per million (ppm) of methyl mercaptan as follows:

$$\text{Methyl mercaptan, ppm} = 63.3A$$

where: A = Micrograms of methyl mercaptan in the 0.5-ml aliquot of specimen vapors as determined from the calibration curve.

4.2.4.6 Iron.

(a) Standard iron solution. Dissolve 0.100 g of pure iron wire in 10 ml of 10-percent sulfuric acid and 3 ml of nitric acid. Cool, transfer to a 1-liter volumetric flask, and dilute to volume with water. Transfer a 10-ml aliquot to a 100-ml volumetric flask and dilute to volume. One ml of this standard iron solution contains 0.01 mg of iron.

(b) Standard graph preparation. By means of a buret, measure 0.5, 1.0, 2.0, 7.0, 10.0, 15.0, and 20.0 ml portions, respectively, of the standard iron solution into seven 100-ml volumetric flasks. Add to each 10 ml of 1 molar hydroxylamine hydrochloride solution. Heat to just boiling, cool, add a small piece of congo red paper, and add sufficient ammonium hydroxide solution to change the color of the congo red paper to a bluish-red. Add 10 ml of 0.1-percent 1,10-phenanthroline solution. Allow the color to develop for 10 minutes, dilute to volume with water, and mix. Place a blank of the reagents in a suitable spectrophotometer having a 525-millimicron filter and a 2-centimeter cell path. Adjust the instrument to zero. Record the dial readings for each of the above standards and plot them against the concentration in milligrams per 100 ml. Use the resulting graph for all subsequent iron determinations.

(c) Procedure. Weigh 2.0 ± 0.01 g of the specimen into a 100-ml silica evaporating dish which has previously been washed with hydrochloric acid. Using a hood, gently ignite using a small flame and avoiding application of excessive heat to the bottom of the dish. When the specimen ceases burning, heat gently with a meeker burner until easily combustible matter is destroyed. Transfer to a muffle furnace at 800° C. When the fumes cease, close the furnace door and leave the dish in the furnace 10 to 15 minutes to destroy the remaining carbon. Remove the dish and cool in a desiccator. Add 3 ml of hydrochloric acid and 3 ml of water. Cover with a clean watch glass and heat 30 minutes on a steam bath. Cool to room temperature and filter through double acid washed dense texture filter paper. (If the filtrate is not clear, refilter using double paper if necessary to make the filtrate clear.) Rinse the filter several times with small portions of water. Add 10 ml of 1 molar

hydroxylamine hydrochloride solution and proceed as in the preparation of the graph in (b). Zero the instrument with a reagent blank. Record the dial reading. From the graph prepared in (b), determine the milligrams of iron in 100 ml for the specimen.

$$\text{Iron, ppm} = \frac{1000 A}{W}$$

where: A = Milligrams of iron and
W = Weight of specimen in grams.

4.2.4.7 Specific gravity. Determine the specific gravity in accordance with ASTM D891.

5. PREPARATION FOR DELIVERY

5.1 Packaging, level C. A quantity of malathion as specified (see 6.2) shall be packaged in a manner which will assure that the purity of the malathion is maintained and that the contents of the package are protected from the supply source to the first receiving activity for a limited time of storage.

5.2 Packing. Packing shall be level A or C as specified (see 6.2).

5.2.1 Level A. Fifty-five U. S. gallons of malathion shall be packed in a drum conforming to type I of PFP-D-729 except that the film thickness of the dried exterior paint coating on the drum shall be no less than 0.0008 inch. The entire inner surface of the drum shall be lined with two coats of a high-baked resin which shall neither affect nor be affected by the malathion (see 6.8). The closure shall be furnished with cap seal. The gasketing material for the closure shall neither affect nor be affected by the malathion.

5.2.2 Level C. A quantity of malathion as specified (see 6.2), packaged as specified in 5.1, shall be packed in containers which comply with the Uniform Freight Classification Rules, the National Motor Freight Classification Rules, or other carrier rules as applicable to the mode of transportation. Containers shall assure maintenance of malathion purity and safe delivery to destination without leakage or damage to contents. Containers shall be sufficiently rigid to withstand transportation and shall be capable of being stacked in tiers at least 10 feet high without being damaged. Containers shall not exceed the gross weight and size limitations of the applicable regulation and shall be closed or strapped as specified therein.

5.3 Marking. Unit, intermediate, and shipping containers shall be marked in accordance with MIL-STD-129. Containers shall be labeled to

comply with the Federal Insecticide, Fungicide, and Rodenticide Act except that the directions for use shall be printed directly on the front of the label. The label shall be accepted by and registered with the Environmental Protection Agency. In addition, each container shall be labeled to show the manufacturer's lot number and the following information:

FOR MILITARY USE ONLY

NOT FOR RESALE UNDER THIS LABEL.

CAUTION: HARMFUL IF SWALLOWED, INHALED
OR ABSORBED THROUGH SKIN

Use with adequate ventilation.

For external use only.

Use an appropriate respirator (tested and found acceptable by the U. S. Department of Agriculture) and gloves when dispensing.

In the event of a spill, wash contaminated skin with soap and warm water.

NOTE TO PHYSICIANS: Malathion causes cholinesterase inhibition. Intramuscular Atropine USP is the antidote.

6. NOTES

6.1 Intended use. Grade A malathion is intended for use in insecticide formulations, such as louse powder, where it is desirable to minimize odor. Grade B malathion is intended for use in applications where the presence of odor is not an important consideration.

6.2 Ordering data. Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Grade of malathion required (see 1.2).
- (c) Unit quantity required for level C packaging (see 5.1).
- (d) Level of packing required (see 5.2).
- (e) Quantity required for level C packing (see 5.2.2).

6.3 Batch. A batch is defined as that quantity of material which has been manufactured by some unit chemical process or subjected to some physical mixing operation intended to make the final product substantially uniform.

6.4 Purity. The determination of percent malathion is based on the consumption of silver by 0,0-dimethyl phosphorodithioate of diethyl mercaptosuccinate. In the procedure, an ethanolic solution of malathion is treated with a strong anionic resin to remove free 0,0-dimethyl phosphorodithioic acid and other strongly acidic organic phosphorous-sulfur impurities which may consume silver. The ethanolic solution of the insecticide is reacted briefly with alkali causing a quantitative cleavage of the malathion producing sodium 0,0-dimethyl phosphorodithioate, sodium fumarate, and ethanol. The hydrolysis medium is neutralized to a given pH range and diluted with aqueous acetone. The resultant solution of 0,0-dimethyl phosphorodithioic acid is titrated potentiometrically with standardized silver nitrate, using a silver-calomel electrode system. The potentiometric titration is plotted and the malathion concentration determined.

6.5 "Amberlite". "Amberlite" is a trade name of the Rohm and Haas Company. "Amberlite" IRA400 is available from most chemical supply houses.

6.6 Combination electrode. This electrode incorporates the indicator (silver) electrode and the referenced (calomel) electrode into a single probe and eliminates the need for an agar salt bridge as formerly specified for the titration cell.

6.7 Highly purified malathion. Highly purified malathion (diethyl fumarate-free) is available from the American Cyanamid Company, Agricultural Division, Product Development and Registrations, P. O. Box 400, Princeton, New Jersey 08540.

6.8 Container coatings. Two-coat lining systems that have been found satisfactory for use with malathion are as follows:

- (a) American Can Company, Metal and Thermit Division - B124-17.
- (b) Bradley Vrooman Company - A21846.
- (c) "Heresite" - P413D.
- (d) Inland Steel Container Company - IC-26.
- (e) Interchemical Company - 14-474.
- (f) Rheem Container Company - #970.

6.9 Significant places. For the purpose of determining conformance with this specification, an observed or calculated value should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM E29).