

O-I-514A
 November 12, 1968
~~SUPERSEDED~~
 Fed. Spec. O-I-514
 July 10, 1957

FEDERAL SPECIFICATION

INSECTICIDE, DDT

This specification was approved by the Commissioner,
 Federal Supply Service, General Services Administration,
 for the use of all Federal agencies.

1. SCOPE AND CLASSIFICATION

1.1 Scope. Dichlorodiphenyltrichloroethane (DDT) covered by this specification is intended for the control of certain insects in certain areas. It will not give adequate control of houseflies and roaches. It will not be used as a residue spray for milk rooms.

1.2 Classification. DDT covered by this specification shall be of but one type and grade.

2. APPLICABLE DOCUMENTS

2.1 Specifications and standards. The following specifications and standards, of the issues in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

Federal Specifications:

UU-S-48 - Sacks, Paper Shipping.
 PPP-D-723 - Drums, Fiber.

Federal Standard:

Fed. Std. No. 123 - Marking for Domestic Shipment (Civilian Agencies).

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications and Standards and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U. S. Government Printing Office, Washington, D. C. 20402)

(Single copies of this specification and other Federal specifications required by activities outside the Federal Government for bidding purposes are available without charge from Business Service Centers at the General Service Administration Regional Offices in Boston, New York, Washington, D. C., Atlanta, Chicago, Kansas City, Mo., Fort Worth, Denver, San Francisco, Los Angeles, and Seattle, Washington.)

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

Military Standards:

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

(Copies of Military Specifications and Standards required by contractors 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 a specific issue is identified, the issue in effect on date of invitation for bids or request for proposal shall apply.

Governmental:

Federal Insecticide, Fungicide, and Rodenticide Act.

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

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Specification No. PHS/NCDC-D-200 - Fiber Drums for Shipping DDT 100% Technical.

(Application for copies should be addressed to the U. S. Public Health Service (NCDC), 1600 Clifton Road N. E., Atlanta, Georgia 30330, Attn: Logistics Specialists.)

Nongovernmental:Consolidated Freight Classification Rules.

(Application for copies should be addressed to the Consolidated Classification Committee, 202 Chicago Union Station, Chicago, Illinois 60606.)

American Society for Testing and Materials (ASTM) Standard:

D 96-63 - Water and Sediment in Crude Oils.

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

National Motor Freight Classification Rules.

(Application for copies should be addressed to the National Classification Board, 1616 P Street, N. W., Washington, D. C. 20036.)

3. REQUIREMENTS

3.1 Material. The material shall comprise dichlorodiphenyl trichloroethane free from added modifying agents.

3.2 Chemical and physical properties shall conform to the requirements in table I.

TABLE I. Chemical and physical properties

Properties	Technical grade DDT		Test paragraph
	Minimum	Maximum	
Setting point, °C.	89	---	4.4.1
Organic chlorine, percent by weight	49	51	4.4.2
Ash content, percent by weight	---	0.5	4.4.3
Chloral hydrate, percent by weight	---	0.025	4.4.5
pH by extraction	5.0	8.0	4.4.4
Water soluble material, percent by weight	---	0.25	4.4.6
Cyclohexanone insoluble, milliliter	---	0.2	4.4.7
Monofluorotrichloromethane insoluble, milliliter	---	0.01	4.4.8

3.3 Form. The material shall be a fine-to-medium granular powder, with a white-to-cream color.

Note: When the material is to be used for formulation by contractors of an end item containing technical DDT, requirements in this paragraph will not apply.

3.4 The finished product shall be clean, uniform and free from lumps and foreign matter.

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, the supplier may utilize his own facilities or any commercial laboratory acceptable to 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 that supplies and services conform to prescribed requirements.

4.1.1 Inspection lot. All material of the same grade manufactured as one batch, and offered for delivery at one time shall be considered a lot for purposes of inspection and tests.

4.1.2 Sampling for inspection of filled containers. A random sample of filled containers shall be selected from each lot offered for inspection in accordance with MIL-STD-105, at inspection level I, and acceptable quality level (AQL) equal 2.5 percent defective to verify compliance with this specification regarding pecking, fill, closure, marking, forms, workmanship and any other requirements not involving tests.

4.1.3 Sampling for tests. For purposes of testing, the lot size shall be expressed in pounds of DDT manufactured as a single batch. The sample unit for testing shall be a 1-pound composite obtained by combining equal portions from samples selected at random throughout the lot in the frequency indicated in the table below. The composite shall be placed in a clean, dry, glass sealed container. Care shall be exercised to prevent contamination or alteration of the DDT during the sampling, compositing, storage and testing. All test reports shall contain the individual values utilized in expressing the final results. The lot shall be unacceptable if the composite fails to meet any test requirements specified.

Lot size (in pounds)	Sample frequency
800 or less	2
801 up to and including 22,000	3
22,001 or more	5

4.2 Inspection.

4.2.1 Inspection of filled containers. Each sample filled container selected in accordance with 4.1.2, shall be examined for defects of the container and the closure, for evidence of leakage, and for unsatisfactory markings; each sample filled container shall also be weighed to determine the amount of the contents. Any container in the sample having one or more defects, or under required fill, shall be rejected; and if the number of defective containers in any sample exceeds the acceptance number for the appropriate sampling plan of MIL-STD-105, the lot represented by the sample shall be rejected. Rejected lots may be resubmitted for acceptance tests provided the contractor has removed or repaired all nonconforming containers.

4.2.2 Lot acceptance. The sample taken as specified in 4.1.3, shall be subjected to the tests described in 4.4.

4.4 Test methods.

4.4.1 Setting point. Transfer 30 ± 0.1 grams of sample into a heavy-wall rimless, ignition tube of heat-resistant glass, 25 millimeters in inside diameter by 200 millimeters long (Corning No. 9860 or equivalent). The tube and its contents shall be supported in an oil bath maintained at 115° to 120°C . When the DDT has almost all become molten, a glass ring stirrer and an accurate thermometer graduated in fifths of a degree (ASTM standard thermometer 70° to 160°C . or equivalent) shall be fitted into the tube through a two-hole cork stopper. While the DDT is being heated, a round, wide-mouth, 8-ounce glass sample jar (approximately 2 inches in diameter and 5 inches high), shall be clamped upright in a water bath maintained at $70^{\circ} \pm 2^{\circ}\text{C}$. The jar shall be kept approximately 80 percent immersed throughout the entire determination and its mouth shall be fitted with a cork stopper into which one hole (just large enough to admit the tube containing the molten DDT), has been bored. When the DDT has melted completely and has reach a temperature of 115° to 120°C ., transfer the test tube and its contents from the oil bath to the water-bath sample jar system, fitting the length of the tube down into the jar through the one-holed cork stopper so that the bottom of the tube is approximately 15 millimeters from the bottom of the sample jar. The liquified sample shall now be stirred continuously, using the glass ring stirrer until the point of maximum super-cooling has been reached and the temperature has begun to rise. Thereafter, the stirring shall be by means of the thermometer. All stirring shall be at the approximate rate of 100 strokes per minute (1 up and down = 1 stroke), all strokes to be 3 to 4 centimeters in length, without breaking the upper surface of the sample. The liquified material shall be stirred while it supercools. When the temperature drops to 89°C ., a small amount of the sample of DDT being tested may be added as seed crystals if none have already formed in the test tube. After the material begins to crystallize, the temperature rises. At this point the stirring shall be stopped momentarily every 15 seconds and a temperature reading taken. The highest temperature reading after the point of maximum super-cooling shall be taken as the setting point. The test shall be discontinued after two successively lower readings after the point of maximum supercooling.

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4.4.2 Organically bound chlorine. Weigh 1 gram of sample of DDT to the nearest 0.1 mg. transfer to a clean, dry 250-milliliter volumetric flask and add 40 milliliters of chlorine-and thiophene-free benzene. Shake until the DDT is dissolved and then make to volume with 99-percent isopropyl alcohol. Mix well and transfer a 25-milliliter aliquot to a clean, dry 300-milliliter flask with a standard tapered neck. Add 2.5 grams of metallic sodium cut into small pieces and swirl the flask in order to mix its contents. Connect to a water-cooled reflux condenser and boil gently at least 1/2 hour. Shake the flask occasionally. Decompose the excess sodium by cautiously adding 10 milliliters of 50 percent isopropyl alcohol through the condenser at a rate of one to two drops per second. Boil for an additional 10 minutes and then add 60 milliliters of distilled water. Cool to room temperature, add two to three drops of phenolphthalein solution, neutralize by adding nitric acid (1:1), and add 10 milliliters of the diluted acid in excess. Add dropwise with stirring of the solution a measured excess (25 ml. approximately) of 0.1N AgNO₃ solution. Coagulate the precipitate by heating on a steam bath for approximately 1/2 hour. Cool to room temperature and filter through a No. 42 Whatman filter paper and wash thoroughly with distilled water, receiving the filtrate in a 500-milliliter Erlenmeyer flask.

Add 5 milliliters of ferric ammonium sulphate indicator and titrate the excess AgNO₃ with 0.1N KCNS solution. Compute the net number of milliliters of 0.1N AgNO₃ consumed by the sample. Calculate percent chlorine as follows:

$$\text{Percent chlorine} = \frac{\text{Ml. 0.1N AgNO}_3 \text{ (consumed)} \times 3.547}{\text{Weight of sample (grams)}}$$

Note: A blank determination (without sample) should be made following the exact procedure given above but limiting the 0.1N AgNO₃ solution to 5 milliliters, in order to obtain a chloride correction value for all reagents used.

4.4.3 Ash. Place a 5-gram sample in a weighed crucible. Burn off or vaporize the DDT under a hood with good ventilation, over a hot plate or from a sand bath. When the organic material has been substantially volatilized, cool the crucible and add an excess of 10 percent sulfuric acid. The crucible shall be heated as above to dryness and then ignited in a muffle furnace for 1 hour at a temperature of 800° + 50°C. Transfer the crucible to a desiccator, cool and weigh. All weighing shall be made to the nearest mg.

4.4.3.1 Calculate the percent of ash as follows:

$$\text{Percent ash} = \frac{A-B}{W} \times 100$$

Where A = combined weight of crucible and ash.
B = weight of crucible.
W = weight of sample in grams.

4.4.4 Determination of pH. Transfer a 20 + 0.1 gram sample of DDT to a 500-milliliter separatory funnel and dissolve in 100 milliliters of benzene. Add 50-milliliter of freshly distilled, cooled, carbon dioxide-free water, stopper, and shake the funnel and contents for 3 minutes. Allow the two phases to separate, and draw off the aqueous layer into a flask. Stopper for flask immediately. Repeat the aqueous extraction twice, using two successive 25-milliliter portions of freshly distilled, cooled, carbon dioxide-free water. Transfer the aqueous layer to a 100 ml. volumetric flask and dilute to mark. Determine the pH of this extract, using any suitable method. However, in the event of dispute, the results obtained with a calibrated pH electrometer shall govern. Reserve the remainder of the extract for the determination of chloral hydrate and water soluble material.

4.4.5 Chloral hydrate. Place 2 milliliters of an aqueous sodium hydroxide solution (40g. in 100 ml. of solution) in a test tube, add 1 milliliter of colorless pyridine and 4 milliliters of the aqueous extract obtained in 4.4.4. Similarly treat 4 milliliters of a standard aqueous solution containing 0.05 milligram of chloral hydrate per milliliter in another tube. Shake the two tubes and heat in a boiling water bath for 1 minute. The red color in the pyridine layer of the sample under test shall not be darker than that of the standard.

4.4.6 Water soluble material. Evaporate 50 milliliters of the aqueous extract obtained in 4.4.4 in a tared evaporating dish or other suitable container. Dry to constant weight at 105°C.

4.4.6.1 Calculate the percent water soluble material as follows:

$$\text{Percent water soluble material} = \frac{2(A-B)}{W} \times 100$$

Where A = combined weight of evaporating dish and residue
B = weight of evaporating dish.
W = weight of sample in grams.

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4.4.7 Cyclohexanone insoluble matter. Place 71 milliliters of freshly redistilled cyclohexanone (refractive index 1.4503 through 1.4507) in a calibrated cone shaped centrifuge tube either size described in ASTM method D 96-63. Add 33 ± 0.1 g. DDT, stopper and shake until material is dissolved as completely as possible. Centrifuge for 10 minutes at a speed to yield a relative centrifugal force of between 500 to 800 r.p.m., at the tip of the tubes. (Calculate required revolutions per minutes from equation in the ASTM method.) The volume of the apparent separated solids should not exceed the maximum listed in table I.

4.4.8 Monofluorotrichloromethane insoluble material. Place 5.0 grams of DDT and 10.0 grams of cyclohexanone in a calibrated centrifuge tube, as above. Stopper, shake until the material is dissolved as completely as possible. Dilute to 100 milliliters with monofluorotrichloromethane. Stopper, shake and centrifuge as described in 4.4.7. The solution in the tube should be clear and give not more than 0.01 milliliter of sediment.

5. PREPARATION FOR DELIVERY

5.1 Packing. Packing shall be level A, or C, as specified (see 6.1).

5.1.1 Level A. The insecticide shall be furnished in 50 to 100-pound containers as specified (see 6.1).

5.1.1.1 Fifty-pound containers. The insecticide shall be furnished in a 50-pound sack conforming to UU-S-48, level A, type I, sack no. 13, except that the moisture-barrier wall shall not be an asphalt laminated barrier.

5.1.1.2 One-hundred-pound containers. The insecticide shall be furnished in a fiber drum conforming to Public Health Service specification PHS/NCDC-D-200 or a fiber drum conforming to type III, grade A of PPP-D-723.

5.1.2 Level C. Insecticide, in the quantity specified, shall be packed in containers which will provide protection to the product during shipment from the supply source to the first receiving activity. The containers shall conform to the requirements of the Consolidated Freight Classification Rules or National Motor Freight Classification Rules, as applicable.

5.2 Marking. In addition to the markings specified in 5.2.1 and any special markings required by the contract or order (see 6.1), shipping containers shall be marked in accordance with Fed. Std. No. 123 or MIL-STD-129, as applicable.

5.2.1 Special markings. Each container shall be labeled to include: U.S.D.A. registration no., identification marking, precautionary marking, net weight, lot number and date of pack. The label shall be cleared for compliance with the Federal Insecticide, Fungicide and Rodenticide Act. Application to the Pesticides Regulation Division, U. S. Department of Agriculture, Washington, D. C. 20250, must be made for registration of the label prior to shipment in interstate commerce. Additional markings shall be specified in the contract or order. Each shipping container shall be durably and legibly labeled with the following information:

ACTIVE INGREDIENT

Dichloro Diphenyl Trichloroethane (DDT) 100%

CAUTION: KEEP OUT OF THE REACH OF CHILDREN.

PRECAUTIONS: DDT is toxic and when in oil solution can be absorbed through the skin. Harmful if swallowed. In case of skin contact wash with soap and water. Avoid contamination of feed and food stuffs. Avoid inhaling dust and mists from sprays. Do not reuse empty container. Destroy it by burying with waste or burning. Stay away from smoke or fumes. This product is toxic to fish and wildlife. Keep out of lakes, streams or ponds.

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6. Notes

6.1 Ordering data. Purchasers should select the preferred options permitted herein and include the following information in procurement documents:

- (a) Title, number and date of this specification.
- (b) Level of packing required (see 5.1).
- (c) Quantity to shipping container (see 5.1.1 and 5.1.2).
- (d) Additional markings required (see 5.2).

MILITARY CUSTODIANS:

Army - GL
Navy - SH
Air Force - 68

Military Coordinating Activity

Army - GL

Review Activities:

Army - GL, MD, MU
Navy - SH
Air Force - 68

User Activities:

Army - ME
Navy - AS, MC

Order for this publications are to be placed with the General Services Administration, acting as an agent for the Superintendent of Documents. See section 2 of this specification to obtain extra copies and other documents referenced herein. Price 10 cents each.