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SUPERSEDING

Int. Fed. Spec. O-H-00200 (AGR-ARS)

August 6, 1956

FEDERAL SPECIFICATION

**HERBICIDE, 2,4-DICHLOROPHENOXYACETIC ACID
(SALTS AND ESTERS)**

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.—2,4-Dichlorophenoxyacetic acid (2,4-D) is an organic acid relatively insoluble in water or oil. It is normally compounded before being used as an herbicide. 2,4-D is a selective herbicide. When applied as a post-emergence spray it will kill many broad-leaved weeds and woody plants, with little or no injury to many grasses, sedges, and other monocotyledonous plants. However, when used as a pre-emergence spray or as a foliage spray on seedlings 2,4-D can also be used to control many annual grasses. This specification covers three general types of 2,4-D.

1.2 Classification.

1.2.1 Types.—Formulations of 2,4-D covered by this specification shall be of three general types, as specified:

Type I.—Dry powder, sodium salt, forms which are the least toxic to plants per pound of 2,4-D acid equivalent.

Type II.—Liquid amine salt forms which are intermediate in toxicity to plants per pound of 2,4-D acid equivalent.

Type III.—Liquid ester forms which are the most toxic forms of 2,4-D to plants per pound of 2,4-D acid equivalent.

Class 1.—Volatile alkyl esters of 2,4-D.

Class 2.—Low volatile esters.

2. APPLICABLE SPECIFICATIONS, STANDARDS, AND OTHER PUBLICATIONS

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

Federal Specifications:

PPP-C-96—Cans, Metal 28 Gage and Lighter.

PPP-D-723—Drums, Fiber.

PPP-D-729 — Drums, Metal, 55-Gallon.
(For Shipment of Non-corrosive Materials).

PPP-D-760—Drums and Pails, Metal
(5 and 16.64 Gallon).

Federal Standards:

Fed. Std. No. 102—Preservation, Packaging, and Packing Levels.

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

(Activities outside the Federal Government may obtain copies of Federal Specifications and Standards 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 25, D. C.)

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

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

Military Standard:

MIL-STD-105 — Sampling Procedures and Tables for Inspection by Attributes.

(Copies of Military Standard referenced above, required by contractors in connection with specific procurement functions, should be obtained from the procuring agency or as directed by the contracting officer.)

2.2 Other publications.—The following publications form a part of this specification. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply:

Governmental:

Federal Insecticide, Fungicide, and Rodenticide Act.

(Copies of the Federal Insecticide, Fungicide, and Rodenticide Act May be obtained from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Prices may be obtained from the Superintendent of Documents.)

Association of Official Agricultural Chemists

Official Methods of Analysis, Eighth Edition, 1955.

(Application for copies should be addressed to the Association of Official Agricultural Chemists, P. O. Box 540, Benjamin Franklin Station, Washington 4, D. C.)

3. REQUIREMENTS

3.1 Type I.—The dry powder form shall consist of the sodium salt of 2,4-dichlorophenoxyacetic acid monohydrate and such

modifying and conditioning agents as are needed. The formulated compound shall contain a minimum of 80 percent 2,4-dichlorophenoxyacetic acid as determined in 4.4.1. The product shall be soluble in soft or hard water (600 ppm. calcium carbonate) at the concentrations specified in the directions for use, non-foaming, and contain no ingredients which will inhibit the application of the material at the concentrations normally used for weed control.

3.2 Type II.—The liquid amine salt form of 2,4-dichlorophenoxyacetic acid shall contain a minimum of four pounds of 2,4-D acid per gallon of formulation at 68° F., as determined in 4.4.2. The amine in the formulation shall be either the alkyl or alkanolamine or mixtures of these types. The product shall be soluble in hard or soft water at the concentrations specified in the directions for use, nonfoaming, disperse easily, making a solution that contains no ingredients which will inhibit the application of the material at the concentrations normally used for weed control. The product shall contain no ingredients which will coagulate with water. The material shall contain sequestering agents which facilitate its application in hard or soft water. The product shall remain free of solid material when held at a temperature of 25° F. for a period of 5 days.

3.3 Type III.—The liquid ester forms of 2,4-dichlorophenoxyacetic acid.

3.3.1 Class 1, volatile esters of 2,4-dichlorophenoxyacetic acid.—The alkyl liquid esters of 2,4-D shall contain a minimum of two pounds of 2,4-D acid per gallon of formulation as determined in 4.4.3. The esters in this class shall belong to the alkyl group such as methyl, ethyl, propyl, isopropyl, butyl, amyl, and pentyl, or mixtures of these alkyl esters. The formulation shall be a clear solution readily miscible with oil and emulsifiable when mixed with water. It shall contain the necessary solvents, carrying and emulsifying agents, such that the emulsion formed re-

quires a minimum of agitation to maintain intimate mixture with the diluent during the mixing and application period. The oil carrier for the formulation shall be of such gravity and viscosity, not detracting from the killing power of the active ingredients, to offer maximum penetration and spread of the spray solution. The product shall remain free of solid material when held at a temperature of 25° F. for a period of 5 days.

3.3.2 Class 2, low volatile esters.—These include the glycol, poly-glycol and their ether ester derivatives of 2,4-D, as well as other heavy molecular weight esters of 2,4-D that are known to be low volatile. The low volatile esters of 2,4-D shall contain a minimum of four pounds of 2,4-D acid per gallon of formulation at 68° F., as determined in 4.4.3. This class shall not include esters of the lower alkyl group such as methyl, ethyl, propyl, isopropyl, butyl, amyl, and pentyl, or mixtures of these alkyl esters. The formulation shall be readily miscible with oil and emulsifiable with water. The product shall be a clear solution, and shall include the necessary solvents, carrying and emulsifying agents, such that the emulsion formed requires a minimum of agitation to maintain intimate mixture with the diluent during the mixing and application period. The oil carrier for the formulation shall be of such gravity and viscosity, not detracting from the killing power of the active ingredients, to offer maximum penetration and spread of the spray solution. When tested for volatility as described in 4.4.4 the product shall have an average response of less than 4.0. The product shall remain free of solid material when held at a temperature of 25° F. for a period of 5 days.

3.3.3 The combination of solvents and emulsifiers used in the type III formulations shall not contain more than 0.1 mg. of organic chlorine per gram when analyzed according to 4.4.3.

3.4 Workmanship.—The finished products shall be clean and uniform, and free from any defects which might impair their utility.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 Lot.—A lot shall consist of the material produced by one manufacturer in not more than 24 consecutive hours under essentially the same manufacturing conditions and with no change in materials providing the operation is continuous. In the event the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.1.1 Inspection lot.—For purposes of sampling, a lot shall consist of all material offered for inspection at one time. In case material is produced by a continuous-run process, the lot shall contain material produced in not more than a 24 hour period. Material in the inspection lot shall be identified by order of production (in case of a continuous-run process) or by batch number (in case of batch process) until ultimate action is taken by the Government inspector as to the acceptance or rejection of the lot.

4.1.2 Sampling for inspection of filled containers.—A random sample of filled containers shall be taken from each lot by the Government inspector in accordance with Military Standard MIL-STD-105 at inspection level I, and acceptable quality level (A.Q.L.) = 2.5 percent defective to verify compliance with all stipulations of this specification regarding fill, closure, marking, and other requirements not involving tests.

4.1.3 Sampling for tests.—From each inspection lot the Government inspector shall take three separate 1-pound or 1-gallon samples. In case the material is produced by a batch process, and the inspection lot contains more than 2 batches, the three samples shall normally be taken from different batches, from time to time; however, at the discretion of the inspector, two or three of the samples shall be taken from the same batch, in which case the samples shall be obtained in a manner calculated to disclose any non-uniformity of the material within the batch. Where material is produced by a continuous-run process

ess the three samples shall be taken so as to represent respectively, the first part, the middle part, and the last part of the run which produced the inspection lot. Each sample shall be thoroughly mixed and divided into three equal portions. The portions shall be placed in separate, clean, dry, metal or glass containers, which shall be sealed and carefully marked. One of the portions of each sample shall be forwarded to a laboratory designated by the bureau or agency concerned, one shall be delivered to the contractor, and one held by the Government Inspector to be used for retests in case of dispute.

4.2 Inspection.

4.2.1 Inspection of filled containers.—Each sample filled container selected in accordance with 4.1.2 shall be examined by the Government Inspector 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 Military Standard MIL-STD-105 the lot represented by the sample shall be rejected. Rejected lots may be resubmitted for acceptance tests provided that the contractor has removed or repaired all nonconforming containers.

4.3 Lot acceptance tests.—The sample specimens selected in accordance with 4.1.3 shall be subjected separately to the tests specified in 4.4. If either specimen fails in one or more of the tests the lot shall be rejected. Rejected lots may be resubmitted for acceptance tests provided the contractor has removed or repaired all nonconforming products.

4.3.1 Packaging, packing and marking.—The inspector shall inspect the packing, packaging, and marking for compliance with section 5.

4.4 Test procedures.

4.4.1 2,4-Dichlorophenoxyacetic acid content in sodium salt of 2,4-dichlorophenoxyacetic acid monohydrate.—Dissolve a sample equivalent to about 1 g. of 2,4-D acid or 1.20–1.25 g. of the sodium salt in 50 ml. of water, transfer to 250-ml. separatory funnel. Neutralize if necessary with 10% H_2SO_4 , and add 10 ml. in excess. Extract the aqueous phase twice with 75 ml. portions of ether. Wash the combined ether extracts free from mineral acid with 3 portions of water exactly 10 ml. each. Avoid slight emulsification by excessive shaking. Filter the ether solution through a funnel containing a small piece of cotton previously saturated with ether into a 400-ml. beaker, rinsing the separatory funnel with ether. Add 25 ml. of water, a few boiling chips, and evaporate off the ether layer on a steam bath until approximately 25 ml. of ether remains. Remove the beaker from the steam bath and evaporate off the remaining portion of ether at room temperature by means of a current of air. Dissolve the aqueous mixture in 100 ml. of neutral ethyl alcohol and titrate with 0.1 N NaOH using 1 ml. of indicators* (1 g. in 100 ml. of alcohol).

Each ml. of 0.1 N NaOH is equivalent to 0.02210 g. of 2,4-dichlorophenoxyacetic acid or 0.02610 g. of sodium dichlorophenoxyacetate. Reference: Methods of Analysis, Association of Official Agricultural Chemists, 8th Ed., par. 5.133 (c), page 75.

4.4.2 2,4-Dichlorophenoxyacetic acid content in amine salts of 2,4-dichlorophenoxyacetic acid.—Transfer a sample equivalent (or a suitable aliquot of a sample diluted with water) to about 1 g. of 2,4-D acid to a 250 ml. separatory funnel. Dilute to 50 ml. of water and proceed as directed in 4.4.1. Calculate the percent 2,4-D acid found to the specific amine present in the sample.

*Either phenolphthalein or thymolphthalein may be used in the titration provided the one selected is used in standardization of the sodium hydroxide.

4.4.3 Esters of 2,4-dichlorophenoxyacetic acid by determination of total chlorine.—Weigh and mix 1.5 g. of boric anhydride (Eastman Kodak Co., Cat. #2685 or equivalent), 1.0 g. finely powdered potassium nitrate, and 0.4 g. finely powdered sucrose. Transfer approximately one-fourth of this mixture to a 42-ml. Parr bomb, electric ignition type, and add from a small weighing buret about 0.25 – 0.30 g. of sample containing from 0.030 – 0.034 g. chlorine. (When a sample larger than 0.30 g. is required, 2.5 g. of boric anhydride should be used. In no cases should a sample larger than 0.6 g. be taken.) Mix well with a thin stirring rod. Add the remainder of the boric anhydride, potassium nitrate and sucrose mixture in small portions and thoroughly mix after each addition. Measure 15 g. of calorimetric grade sodium peroxide in a standard measuring dipper, add a small portion to the contents of the bomb, and stir. Add the balance of sodium peroxide and thoroughly mix by stirring with the rod. Withdraw the rod and brush free of adhering particles. Quickly cut or break off the lower 1½ inches of the stirring rod and imbed it in the fusion mixture. Sprinkle on the top of the fusion mixture a small quantity of finely ground sucrose. Prepare the head by heating the fuse wire momentarily in a flame and immersing it into a small quantity of sucrose. One milligram of the substance is sufficient to start the combustion. Assemble the bomb and ignite in the usual manner with a satisfactory shield between the operator and apparatus.

Place about 100 ml. of distilled water in 600-ml. beaker and heat nearly to boiling. After cooling of the bomb, dismantle it and dip the cover in the hot water to dissolve any of the fusion which may be adhering to its under side. Wash cover with a fine jet of distilled water catching the washings in the beaker. With a pair of tongs lay the fusion cup on its side in the same beaker of hot water, covering it immediately with a watch glass. After the fused material has been dissolved, remove the cup and rinse with hot water, cool the solution, add several drops of

phenolphthalein indicator, neutralize with concentrated nitric acid and add 5 ml. in excess. From this point, the chlorine may be determined by electrometric titration or by the Volhard procedure as directed in the Methods of Analysis, Association of Official Agricultural Chemists, 8th Ed., page 80, par. 5.153 (a) (c).

Note 1.—The combination of materials used in a sodium peroxide bomb has explosive properties if wrongly handled, and the operator should remain fully aware at all times of the precautions that must be observed and the steps which must be taken to avoid damage to the apparatus and possibly personal injury. It is suggested that the instructions and precautions given in the "Parr Manual Number 121—Peroxide Bomb Apparatus and Methods," Parr Instrument Company, Moline, Illinois, be observed.

Note 2.—A flame fired bomb may be used in place of the electric ignition type, but in case of dispute the electric ignition type will govern.

4.4.4 Volatility test—relative vapor activity.—The vapor activity test is conducted with gas tight cellophane cases approximately 3½ x 3½ x 16 inches in size. Young rapidly growing Pinto bean plants about 4 inches in height are used as test plants. A single bean plant growing in a 3 inch pot is placed in each cellophane case just prior to testing the ester.

4.4.4.1 Two milligrams of acid equivalent as the ester is dissolved in 10 milliliters of 95 percent ethyl alcohol and a Whatman No. 1 filter paper (9 cm. diameter) is thoroughly moistened by dipping in the solution. (Do not reuse the container used in this impregnation.) The alcohol is then allowed to evaporate and the filter paper impregnated with the ester is inserted into the cellophane case containing the bean plant and fastened to the inside of the case 6 inches above the leaves of the test plant. The open end of the case is then sealed.

4.4.4.2 The case containing the test plant and treated filter paper is then placed in a dark room for a period of 24 hours. The temperature range of the room should be 80° to 90° F. Control plants are also sealed in separate cases. The experimental design is a randomized block with three replications and each test is repeated three times. The evaluations shall be made following an exposure period of 24 hours.

4.4.4.3 Observations of effect of the vapors on test plants should take into consideration whether or not the plant is slightly, moderately, or severely injured, including such symptoms as degree of stem curvature, terminal bud inhibition and degree of leaf curl. The relative vapor activity of an ester can be numerically designated as follows: 0 - no visible effect; 1, 2, 3 - slight injury - plant usually recovered with little or no reduction in growth, slight epinasty present, stem curvature slight; 4, 5, 6 - moderate injury - plant usually recovered, moderate epinasty, moderate terminal bud inhibition and moderate stem curvature present; 7, 8, 9 - severe injury - plant usually does not recover, pronounced epinasty, together with pronounced stem curvature; 10 - plant killed.

4.4.4.4 Chemically pure 2,4-D acid and the butyl ester of 2,4-D are used as standards. The 2,4-D acid under most conditions is rated 0 (zero) while the butyl ester has a high vapor activity with a rating of 9.0. Esters receiving the following ratings would be classed as follows:

- 0 no vapor activity
- 1,2,3 very low vapor activity
- 4,5,6 low to moderate vapor activity
- 7,8,9 high vapor activity
- 10 very high vapor activity

Esters must receive a vapor activity rating of less than 4 to be designated low volatile.

5. PREPARATION FOR DELIVERY

The definitions and applications of the levels of packaging and packing shall be in accordance with Federal Standard No. 102.

5.1 Packaging.

5.1.1 Level A.

5.1.1.1 *Type I.*—Packaging is not applicable. Material shall be packed directly into fiberboard drums as specified in 5.2.1.1 or 5.2.2.1.

5.1.1.2 *Types II and III.*—One-gallon quantities of material shall be packaged in 1-gallon oblong cans conforming to type V of Federal Specification PPP-C-96 having a plan B coating. Cans shall be closed with either a class 4 screw-cap or a class 5 snap-on cap. Five and fifty-five gallon quantities shall be packed directly into containers, as specified in 5.2.1.2.2 and 5.2.1.2.3.

5.1.2 *Level C.*—Unless otherwise specified, packaging shall be in accordance with the manufacturer's commercial practice.

5.2 Packing.

5.2.1 Level A.

5.2.1.1 *Type I.*—Fifty (50) pounds of material shall be packed in fiberboard drums conforming to type III of Federal Specification PPP-D-723.

5.2.1.2 *Types II and III.*

5.2.1.2.1 *One-gallon.*—Six one-gallon cans shall be packed for overseas shipment in accordance with the overseas shipment requirements Appendix of Federal Specification PPP-C-96.

5.2.1.2.2 *Five-gallon.*—Material shall be furnished in metal drums conforming to type I, class 1 or 2 of Federal Specification PPP-D-760.

5.2.1.2.3 Fifty-five-gallon.—Material shall be furnished in drums conforming to type II or IV of Federal Specification PPP-D-729.

5.2.2 Level B.

5.2.2.1 Type I.—Fifty pounds of material shall be packed in fiberboard drums conforming to type II of Federal Specification PPP-D-723.

5.2.2.2 Types II and III.

5.2.2.2.1 One-gallon.—Six one-gallon cans shall be packed for domestic shipment in accordance with the domestic shipment requirements of the Appendix of Federal Specification PPP-C-96.

5.2.2.2.2 Five and fifty-five-gallon.—Material shall be packed as specified in 5.2.1.2.2 and 5.2.1.2.3.

5.2.2.3 Levels A and B.—The fifty pound fiberboard, and five and fifty-five gallon drums will require no overpacking.

5.2.3 Level C.—Material shall be packed in containers of the type commonly used for the purpose in a manner that will insure acceptance by common carrier and safe delivery at destination. Shipping containers shall comply with the Uniform Freight Classification Rules, or regulations of other carriers as applicable to the mode of transportation.

5.2.4 Level D.—To be inserted by the Department of Defense if required.

5.3 Marking.

5.3.1 Civilian agencies.—In addition to any special marking required by the contract or order, marking for shipment shall be in accordance with Federal Standard No. 123.

5.3.1.1 Labeling.—Unless otherwise specified, each container of 2,4-D formulation shall be labeled with instructions for use and marked in compliance with The Federal In-

secticide, Fungicide, and Rodenticide Act and other applicable existing Federal laws. Date of pack and lot number shall appear on the label. In addition, the cover shall have the stock number and item nomenclature shall be embossed on a metal plate and wired securely to the individual container.

5.3.2 Military marking.—In addition to the marking specified in 5.3.1.1, and any special marking required by the contract or order, shipments shall be marked in accordance with Military Standard MIL-STD-129.

6. NOTES

6.1 Intended use.

6.1.1 Type I.—The monohydrate sodium salt dry powder form of 2,4-D is sparingly water soluble, it is especially adapted for lawns, cemeteries and in other areas where desirable vegetation such as flowers and ornamentals are likely to be injured by spray drift or vapors. The dry powder sodium salt form in water solution is useful on easy-to-kill weeds. The main disadvantage of the powder form of 2,4-D is that it is not as effective as either the amine salts or esters of 2,4-D on hard-to-kill weeds or older weeds. Some nozzle clogging and other application difficulties are likely to result due to incomplete solution of the sodium salt when the dry powder form is applied at high rates with low gallonage sprayers. The effectiveness of the sodium salt of 2,4-D is reduced when rains occur immediately following application. The sodium salt of 2,4-D is the least toxic form per pound of 2,4-D acid to plants as specified in types I, II, or III.

6.1.2 Type II.—The liquid amine forms of 2,4-D are highly soluble in water, making a relatively clear solution. They are quite stable and are effective for easy-to-kill or moderately easy-to-kill weeds. The amine salts of 2,4-D are much less volatile than the ester forms of 2,4-D and are somewhat better adapted for spraying for weed control near plants sensitive to 2,4-D. The liquid amine

salt forms of 2,4-D are well adapted for spraying in lawns, turfed areas, and in tolerant field and horticultural crops for weed control. The amine salts of 2,4-D are not quite as effective on old, semi-resistant weeds and woody species as are the esters. The amine salts of 2,4-D are more toxic to plants than the sodium salt of 2,4-D, but less toxic to plants than the esters of 2,4-D per pound of 2,4-D acid.

6.1.3 Type III.—The liquid ester forms of 2,4-dichlorophenoxyacetic acid.

6.1.4 Class 1.—The lower alkyl esters of 2,4-dichlorophenoxyacetic acid are comparatively volatile. When the lower alkyl esters of 2,4-D are used for weed control in tolerant field and horticultural crops they should be used at lower acid equivalent rates than either the sodium or amine salts of 2,4-D. The lower alkyl esters of 2,4-D are better adapted for the control of deep rooted perennial weeds, harder-to-kill weeds, older semi-resistant weeds and woody species than the sodium or amine salts of 2,4-D. The lower alkyl esters of 2,4-D should not be used in areas near sensitive crops such as cotton, grapes, tomatoes, tobacco, and other sensitive crops.

6.1.5 Class 2.—The low volatile esters of 2,4-D have the same intended use as the ester forms specified in class 1. However, in areas where sensitive crops are grown such as cotton, etc., if an ester form of 2,4-D is necessary, the esters specified in class 2 should be used to reduce the hazard of volatility.

6.2 Ordering data.—Purchasers should exercise any desired options offered herein (see 1.2, 5.1, 5.2, 5.3) (also see 6.3 for basis of award).

6.3 Basis of award.

6.3.1 Type I.—Bids should be evaluated and the award made on the basis of computing the price per pound of 2,4-D acid equivalent contained per pound of bulk. (Supplier should be requested to furnish 2,4-D acid equivalent data.)

6.3.2 Type II and III (classes 1 and 2).—Bids should be evaluated and the award made on the basis of computing the price per pound of 2,4-D acid equivalent contained in each gallon of preparation or concentrate. (Supplier should be requested to furnish 2,4-D acid equivalent data.)

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 way 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.

MILITARY INTERESTS:

Army—C E M Q
Navy—Y
Air Force.