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FEDERAL SPECIFICATION

HERBICIDE: SILVEX ESTER, AND SILVEX POTASSIUM SALT

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. This specification covers three types of organic acid herbicides known as silvex which are formulated with 2- (2, 4, 5-trichlorophenoxy) propionic acid, and inert ingredients, as either a low volatile liquid ester, or as a liquid or granular potassium salt. The liquid ester, when emulsified with water or an oil-water carrier, is used as a selective translocated foliar spray for the control of many broad-leaved weeds and some woody plants. The liquid or granular potassium salts are used for the control of aquatic weeds by direct application to water surfaces. Further information on use is contained in section 6 of this specification.

1.2 Classification.

1.2.1 Types. The silvex herbicide shall be of the following types as specified (see 6.2):

Type I—Liquid ester formulation: 2- (2, 4, 5-trichlorophenoxy) propionic acid, low volatile esters; (4 pounds acid equivalent per gallon concentration).

Type II—Liquid salt formulation: 2- (2, 4, 5-trichlorophenoxy) propionic acid, potassium salt; (60 percent by weight, or 6 pounds acid equivalent per gallon concentration).

Type III—Granular salt formulation: 2- (2, 4, 5-trichlorophenoxy) propionic acid, potassium salt; (22.8 percent by

weight, or 20 percent acid equivalent concentration).

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 or request for proposal, form a part of this specification to the extent specified herein.

Federal Specifications:

RR-S-366—Sieves, Standard for Testing Purposes.

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

PPP-D-705—Drum: Metal Shipping, Steel (Over 12 and under 55 Gallon).

PPP-D-723—Drums, Fiber.

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

PPP-P-704—Pails: Shipping, Steel (1 Through 12 Gallon).

Federal Standards:

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

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

FSC 6840

(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 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, Washington, D. C., Atlanta, Chicago, Kansas City, Mo., Dallas, Denver, San Francisco, Los Angeles and Seattle, Wash.)

(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 publications 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.

Code of Federal Regulations:

7 CFR 362—Agriculture, Regulations for the Enforcement of the Federal Insecticide, Fungicide, and Rodenticide Act.

(Copies of the above part of the Code of Federal Regulations may be obtained from the Superintendent of Documents, U. S. Government Printing Office, Washington, D. C., 20402.)

American Society for Testing and Materials (ASTM) Publications:

ASTM D 56-57—Test for Flash Point by Tag Closed Tester.

ASTM D 97-57—Test for Cloud and Pour Points.

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

3. REQUIREMENTS

3.1 Type I—Liquid ester formulation.

3.1.1 Composition. The type I silvex ester herbicide shall be a low volatile liquid ester formulation that is readily miscible in diesel oil, fuel oil, or kerosene, and is readily emulsifiable in water. The finished herbicide shall be composed of such active ingredients, inert ingredients, carriers, emulsifying agents, and solvents as are specified herein.

3.1.1.1 Active ingredient. The active ingredient shall be a 2- (2, 4, 5-trichlorophenoxy) propionic acid low volatile ester. It shall be in the minimum concentration of 4 pounds acid equivalent per gallon, when tested as specified in 4.5.1.1. The particular esters used in the formulation shall be the butoxyethanol, iso-octyl, or propylene glycol butyl ether esters, or other heavy molecular weight esters of 2- (2, 4, 5-trichlorophenoxy) propionic acid that are of the low volatile type. However, methyl, ethyl, propyl, isopropyl, butyl, amyl, and pentyl esters or mixtures of these alkyl esters are not acceptable.

3.1.1.2 Inert ingredients. The inert ingredients shall include the related impurities and such diluting, modifying, and conditioning agents as are needed to meet the requirements of this specification.

3.1.1.2.1 Carrier. The carrier of the formulation shall be an oil that has an aromatic content large enough to keep the emulsifier and the ester homogeneous at low temperatures but small enough to avoid contact

action what would kill plant cells and tissues to the extent of inhibiting translocation when a water emulsion is used.

3.1.2 Volatility. The effect of the vapors from the herbicide shall have an activity rating of 3 or less when tested as specified in 4.5.1.2.

3.1.3 Emulsion stability. The herbicide shall contain the necessary emulsifying agents and solvents to form an emulsion with hard or soft water such that, when tested as specified in 4.5.1.3, there shall be no more than slight creaming (0.7 ml. maximum) of the emulsion, and no apparent free oil present. No aliphatic alcohol having two to five carbons in the chain shall be used as solvents since such alcohols could cause transesterification during prolonged periods of storage. This requirement is intended to ensure that the emulsion will be completely stable during the mixing and application period but will break soon after reaching the plant surface.

3.1.4 Sedimentation. The herbicide shall be a clear liquid containing not more than 0.01 percent of sediment or crystalline solids by volume when tested as specified in 4.5.1.4.

3.1.5 Free acid. The herbicide shall contain not more than 1.4 percent free acid when tested as specified in 4.5.1.5.

3.1.6 Flash point. The flash point (Tag Closed Tester) of the herbicide shall be not less than 60°C., when tested as specified in 4.5.1.6.

3.1.7 Pour point. The pour point of the herbicide shall be minus 23°C. or lower, when tested as specified in 4.5.1.7.

3.2 Type II—Liquid salt formulation.

3.2.1 Composition. The type II silvex potassium salt herbicide shall be a liquid potassium salt formulation composed of such active ingredients and inert ingredients as are specified herein.

3.2.1.1 Active ingredient. The active ingredient shall be 2- (2, 4, 5-trichlorophenoxy) propionic acid potassium salt. It shall be in the concentration by weight of not less than 60 percent, and shall have a minimum of 6 pounds acid equivalent per gallon, when tested as specified in 4.5.2.1.

3.2.1.2 Inert ingredients. The inert ingredients shall include the related impurities and such diluting, modifying, and conditioning agents as are needed to meet the requirements of this specification.

3.2.2 Reaction. The acidity of the herbicide shall be not greater than 2 percent calculated as H_2SO_4 and the alkalinity shall be not greater than 2 percent calculated as NaOH, when tested as specified in 4.5.2.2.

3.2.3 Sedimentation. The herbicide shall be a clear amber liquid containing not more than 0.01 percent of sediment or crystalline solids by volume when tested as specified in 4.5.2.3.

3.3 Type III—Granular salt formulation.

3.3.1 Composition. The type III silvex potassium salt herbicide shall be a granular potassium salt formulation composed of such active ingredients and inert ingredients as are specified herein. It shall be a relatively free-flowing granular product which shall be dry, loose, nondusty, non-gritty, and free from foreign matter. The herbicide shall be prepared by impregnation of an absorbent granular carrier with a solution of the active ingredient. The finished herbicide shall be essentially in the form of dry granular aggregates. Before being packaged, the final herbicide shall be cured for a sufficient time after formulation to allow full absorption of the solution of the active ingredient.

3.3.1.1 Active ingredient. The active ingredient shall be 2- (2, 4, 5-trichlorophenoxy) propionic acid potassium salt. It shall be in the concentration by weight of not less

than 22.8 percent, and shall have a minimum of 20 percent acid equivalent, when tested as specified in 4.5.3.1.

3.3.1.2 Inert ingredients. The inert ingredients shall consist of (1) the granular carrier, (2) the unevaporated solvent remaining permanently as part of the dry finished preparation, and (3) any impurities in the active ingredient used in preparation of the herbicide.

3.3.1.2.1 Carrier. The carrier shall be a granular attapulgite clay or other mineral that has the ability to hold and carry relatively large amounts of oily, waxy, or sticky chemicals while retaining a dry and free-flowing condition in the finished herbicide. It shall be extruded (commonly designated AA), and calcined (commonly designated LVM). It shall produce a stable formulation with the active ingredient either with or without a deactivator, and also have sufficient absorptive capacity to inhibit agglomeration, caking, and lumping.

3.3.2 Chemical stability. Not more than 10 percent of the concentration of 2- (2, 4, 5-trichlorophenoxy) propionic acid potassium salt specified in 3.3.1.1 shall be lost from the herbicide when tested as specified in 4.5.3.2.

3.3.3 Reaction. The acidity of the herbicide shall be not greater than 2 percent calculated as H_2SO_4 and the alkalinity shall be not greater than 2 percent calculated as NaOH when tested as specified in 4.5.3.3.

3.3.4 Grain size distribution. The grain size distribution of the herbicide shall meet the requirements in table I. The distribution shall be determined by the dry sieve test specified in 4.5.3.4.

TABLE I. Grain size distribution (dry sieve)

Sieve		Percent passing	
Micron size	U. S. No.	Minimum	Maximum
2380	8	99.8	—
590	30	—	10
250	60	—	1.5

3.4 Labeling. The herbicides shall be registered and labeled in compliance with the Federal Insecticide, Fungicide, and Rodenticide Act. The following additional information shall also be included in the labeling:

Federal Stock No.
 Specification No.
 Type

3.5 Workmanship. The herbicides shall be free from any defects which would impair their intended use.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 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.

4.2 Lot.

4.2.1 Herbicide. For purposes of inspection, a lot for each type of herbicide shall consist of all herbicide produced by one manufacturer under essentially the same conditions and process, within the following limitations. In the event the herbicide is produced by a continuous-run process, the lot shall contain herbicide produced in a period of not more than 24 hours. In the event the herbicide is produced by a batch process, each batch shall constitute a lot. If the herbicide cannot be identified to a particular 24-hour period or to a particular batch, all herbicide to be offered for delivery at one time under a contract or order shall be considered a lot.

4.2.2 Filled containers. For purposes of inspection, a lot of filled containers shall

consist of all specified containers filled with one type of herbicide which are to be offered for delivery at one time under a contract or order.

4.3 Sampling.

4.3.1 Sampling for examination (type III only) and for tests (all types of herbicide). When herbicide is sampled from a continuous-run process, three separate 1-quart or 1-pound samples, as applicable, shall be taken from each inspection lot, and shall represent, respectively, the first part, the middle part, and the last part of the run which constituted the lot. When herbicide is sampled from a batch process, for type I and type II herbicide one 1-quart sample shall be taken from each batch and shall be representative of all portions of the batch; for type III herbicide, three separate 1-pound samples shall be taken at different locations within each batch. When herbicide is sampled from an inspection lot consisting of all herbicide to be offered for delivery at one time, three separate 1-quart or 1-pound samples, as applicable, shall be taken at random from the lot.

4.3.2 Sampling for examination of filled containers. A random sample of filled containers shall be selected from each lot in accordance with MIL-STD-105, at inspection level I, and an AQL of 2.5 percent defective.

4.4 Examination.

4.4.1 Herbicide (type III only). Prior to conducting the tests specified in 4.5.3, the samples of herbicide selected in accordance with 4.3.1 shall be visually examined for conformance to the appearance requirements of 3.3.1. If any one of the samples fails to pass the examination, the lot represented therein shall be rejected. Rejected lots may be reexamined provided the manufacturer has corrected all defects. The manufacturer shall furnish to the Government full particulars concerning the rejection and the action taken to correct the defects.

4.4.2 Filled containers. Each sample filled container selected in accordance with 4.3.2 shall be examined for any defects of the container and the container closure, for evidence of leakage, and for illegible, incorrect, or missing marking. Each sample filled container shall also be weighed to determine conformance to the quantity (net weight) requirement. Any filled container having one or more defects shall be considered a defective unit.

4.5 Tests. Samples of herbicides selected in accordance with 4.3.1 shall be subjected separately to the tests specified herein as applicable to each type of herbicide. If any one of the samples fails to pass any one of the tests specified, the lot represented therein shall be rejected. Rejected lots may be reinspected provided the manufacturer has corrected all defects. The manufacturer shall furnish to the Government full particulars concerning the rejection and the action taken to correct the defects.

4.5.1 Type I—Liquid ester formulation.

4.5.1.1 Concentration of low volatile liquid ester of 2- (2, 4, 5-trichlorophenoxy) propionic acid. Since an ester type wetting agent is present in the formulation, the ester content cannot be determined by saponification. It is, therefore, calculated from the amount of chlorine present. The sample is decomposed by heating with sodium peroxide in a closed bomb. The chlorine present is converted to inorganic chloride and determined volumetrically by the Volhard procedure. A correction is made for any free acid initially present.

4.5.1.1.1 Reagents.

(a) Sodium peroxide (Na_2O_2), reagent grade. CAUTION: Na_2O_2 is both a strong caustic and an intense oxidizing agent. Nothing but glass or metal should come in contact with the dry powder. If any is spilled, use dry sand in sweeping up. Do not pour Na_2O_2 or any mixture containing Na_2O_2 into water, as it may ignite violently.

(b) Nitric acid (HNO_3). Dilute one volume of reagent grade HNO_3 with an equal volume of water.

(c) Silver nitrate (AgNO_3), 0.1 N standard solution. Dissolve 16.99 g. of reagent grade AgNO_3 in water and dilute to 1 liter. Standardize the solution against recrystallized potassium bromide.

(d) Ammonium thiocyanate (NH_4SCN), 0.1 N standard solution. Dissolve 7.612 g. of recrystallized NH_4SCN in water and dilute to 1 liter. Standardize the solution against 0.1 N AgNO_3 solution.

(e) Volhard indicator. Dissolve 100 g. of ferric ammonium sulfate in 15 ml. of concentrated HNO_3 and enough water to make 1 liter.

(f) Alcohol, 95 percent. Formula SDA-30; 10 gallons methyl alcohol plus 100 gallons ethyl alcohol, or formula SDA-2B; 1/2 gallon benzene or 1/2 gallon rubber hydrocarbon solvent in 100 gallons ethyl alcohol.

(g) Sodium hydroxide (NaOH) solution, 0.1 N. Dilute a solution containing 4.0 g. of low-carbonate NaOH to 1 liter with water and standardize against benzoic acid as primary standard. Prepare a stock solution of low-carbonate NaOH by filtering or allowing the Na_2CO_3 to settle from a 48 percent to 50 percent solution of reagent grade NaOH . Store in a polyethylene bottle.

4.5.1.1.2 Apparatus.

(a) Parr Sodium Peroxide Bomb, Series 2100, Flame Ignition Apparatus, 22 ml. This bomb consists of four parts: (1) A 22 ml. fusion cup, 36 percent or 94 percent nickel, 1-1/16 inch I. D. by 1-5/8 inch deep with a 1/16-inch wall and a 7/16-inch bottom. (2) A nickel-plated brass body. (3) A nickel-plated brass cover fitted with a lead gasket. (4) A nickel-plated brass screw cap which clamps the cup, body, and cover together.

(b) Ignition housing plus a safety glass shield and safety glasses.

(c) Blast lamp for air, gas, and oxygen.

(d) Gelatin capsule, No. 00.

(e) Beckman model G. pH meter (or its equivalent), equipped with a glass-calomel electrode system.

4.5.1.1.3 Procedure.

(a) Weigh about 0.4 g. of the herbicide sample in a gelatin capsule to the nearest tenth of a milligram.

(b) Add about 5 g. of Na_2O_2 to the fusion cup. Place the capsule in the cup, and cover with about 10 g. of Na_2O_2 .

(c) Close the bomb, make sure that the lead gasket is in place, and tighten with a wrench. Place the bomb behind a safety shield. Heat the bottom of the bomb for one minute with an air gas flame and then with the oxygen flame until it is cherry red (about 2 minutes). Allow the bomb to air-cool for several minutes and then cool to room temperature either by allowing the bomb to stand or by placing it under a water tap. Remove the screw cap, without disturbing the cover. Wash the outside of the bomb with distilled water.

(d) Remove the cover and wash any adhering particles into a 600 ml. beaker with 50 ml. of distilled water. Place the fusion cup in an upright position in the beaker. Tip the cup over with a dry stirring rod and immediately cover the beaker with a watchglass. When the reaction has completely subsided, remove the fusion cup with a glass stirring rod and wash thoroughly with a stream of distilled water. If the cake has not completely dissolved, return the fusion cup and heat the solution carefully until dissolution is complete. Cool to room temperature.

(e) Slowly neutralize the cool solution with dilute HNO_3 and add 5 ml. in excess. A very vigorous reaction, due to the liberation of excess oxygen, takes place at the neutral point or in the presence of excess acid. Avoid losses from this reaction. Add 0.1 N AgNO_3 in a measured excess of from 2 to 5 ml. more than the calculated amount required. (The calculated amount of AgNO_3 required will be approximately 50 times the

sample weight in grams.) Heat to coagulate the precipitated silver chloride and digest for 5 minutes.

(f) Filter the solution with suction through a coarse filter paper, such as Whatman No. 40, on a Buchner type porcelain funnel into a 500 ml. pyrex filter flask with a side tube. Wash the beaker and precipitate in the funnel several times to remove the excess of AgNO_3 . Cool, add 5 to 10 ml. of Volhard indicator, and titrate the excess AgNO_3 with 0.1 N NH_4SCN to the first permanent reddish-orange end point.

(g) Run a reagent blank by bombing a capsule containing peroxide and carrying it through the normal analytical procedure. Correct the AgNO_3 titration in paragraph (f) for the blank.

(h) Weigh 10 g. of the herbicide sample into a 250 ml. beaker and add 100 ml. of alcohol. Titrate the free acid potentiometrically with 0.1 N NaOH to pH 7. The correction for free acid initially present will be:

$$\frac{\text{ml. of 0.1 N NaOH} \times 3}{10}$$

\times sample weight (paragraph (a))

This correction is also subtracted from the AgNO_3 titration in paragraph (f).

Calculation:

- (1) Percent 2-(2,4,5-trichlorophenoxy) propionic acid ester.

$$\frac{\text{net ml. of 0.1 N AgNO}_3 \times X \times 100}{\text{Weight of sample}}$$

= Percent acid ester

where

X = equivalent weight in grams of the particular acid ester used in the formulation to 1 ml. of 0.1 N AgNO_3

(Example: Should the formulation be with propylene glycol butyl ether ester, the equivalent weight (X) would be 0.01855.)

- (2) Percent acid equivalent.

$$\text{Percent acid ester} \times \frac{269.5}{Y}$$

= Percent acid equivalent

where

Y = molecular weight of the particular acid ester used in the formulation

(Example: Should the formulation be with propylene glycol butyl ether ester, the molecular weight (Y) would be 406.5.)

- (3) Pounds per gallon acid equivalent.

$$\frac{\text{Percent acid equivalent}}{100}$$

$\times 8.33 \times \text{sp. gr. of herbicide}$

= Pounds per gallon of acid equivalent

The procedure for the determination of the specific gravity of the herbicide at 20°C. is as follows:

Apparatus:

- Pycnometer, 25 ml. vacuum-wall.
- Thermometer, calibrated in 0.1°C., such as ASTM E 1-61.
- Analytical balance having a sensitivity of at least 1/10 milligram at full load.
- Analytical weights, class S.

Procedure:

(a) Clean the pycnometer with a saturated solution of chromic acid in concentrated sulfuric acid. Allow it to stand a few hours, empty, and rinse well with distilled water. Dry the empty pycnometer, using acetone. Weigh and record as E. (This weighing must be in duplicate but need not be repeated for each sample.)

(b) Standardization: Fill the pycnometer with freshly boiled distilled water that has been sufficiently cooled so that, when the pycnometer is filled, the temperature of the water will be 20°C. Insert the plug and wipe the plug and neck dry with filter paper. Put the glass cap in place and weigh at once. Repeat this standardization procedure at least six times. Record the average weight as T. (This need not be repeated for each sample.)

(c) Transfer 50 to 100 ml. of product to a clean 250-ml. Erlenmeyer flask. Adjust the temperature of the sample so that, when the pycnometer is filled, the temperature of the sample will be 20°C. Fill the

pycnometer, insert the plug, clean the outside of the pycnometer, and wipe the plug and neck dry with filter paper. Put the glass cap in place and weigh at once. Record this weight as T' .

Calculation:

$$T' - E = W'$$

$$T - E = W$$

then

$$\frac{W'}{W} = \text{Specific gravity of the herbicide at } 20^{\circ}\text{C. in air}$$

where

T' = weight of pycnometer and sample at 20°C .

T = weight of pycnometer and water at 20°C .

E = weight of pycnometer.

W = weight of water at 20°C .

W' = weight of sample at 20°C .

4.5.1.2 Volatility. This test is a bioassay in which the effect of vapors on test plants is observed. The relative vapor activity of the herbicide can be designated numerically.

4.5.1.2.1 Apparatus and materials.

(a) Polyethylene containers, gastight, 4 by 4 by 18 inches.

(b) 3-inch pots.

(c) Whatman No. 1 filter paper, 9 cm. in diameter.

(d) Pinto bean plants, about 4 inches tall.

(e) Ethyl alcohol, 95 percent.

(f) 2-(2, 4, 5-trichlorophenoxy) propionic acid (chemically pure).

(g) 2-(2, 4, 5-trichlorophenoxy) propionic acid, butyl ester.

4.5.1.2.2 Procedure. Just prior to testing, place a pot containing one growing bean plant in each polyethylene container. Dissolve 2 mg. of the acid equivalent of the herbicide in 10 ml. of alcohol and thoroughly moisten a filter paper by dipping in the solution. Discard the container used in this impregnation. Allow the alcohol to evaporate, insert the impregnated filter paper into the polyethylene container holding the

bean plant, and fasten 6 inches above the leaves. Seal the open end of the container. Place in a dark room maintained at $27^{\circ} \pm 3^{\circ}\text{C}$. Following the same procedure, substitute 2 mg. of the material in (f) for the herbicide as one control, and substitute 2 mg. of the acid equivalent of the material in (g) for the herbicide as the other control. Each test shall be repeated three times.

Conclusions:

After 24 hours, observe the effect of the vapor on the plant and rate as follows:

0—no visible effects — no vapor activity.

1,2,3—plants recover with no reduction in growth, slight epinasty — very low vapor activity.

4,5,6—plants recover, moderate epinasty, moderate terminal bud inhibition and moderate stem curvature — low to moderate vapor activity.

7,8,9—Severe injury, plant does not recover, pronounced epinasty, pronounced stem curvature — high vapor activity.

10—plant killed — very high vapor activity.

The materials in (f) and (g) are used as standards. The former is rated 0 and the latter is rated 9.

To be designated as low volatile, an ester must have a vapor activity rating of 3 or less.

4.5.1.3 Emulsion stability. One percent (by volume) samples of herbicide in water of known hardness are agitated mechanically under controlled conditions. The emulsions formed are immediately transferred to an ASTM long-form centrifuge tube and examined at the end of 30 minutes for the degree of creaming and for presence of free oil.

4.5.1.3.1 Reagents. Use three types of water for these tests.

(1) Standard hard water, having a total hardness equivalent to 342 p.p.m. of calcium carbonate. It is made by dissolving the following salts in distilled water:

Calcium chloride, anhydrous0.804 gram
 Magnesium chloride hexahydrate ..0.189 gram
 Distilled water to make1 liter

(2) Average water, having a total hardness equivalent to 115 p.p.m. of calcium carbonate. Mix one volume of hard water with two volumes of distilled water. This concentration represents the greatest degree of hardness expected from municipal water-treating systems.

(3) Soft water, having a total hardness equivalent to 35 p.p.m. of calcium carbonate. Mix one volume of hard water with nine volumes of distilled water. This concentration represents a reasonable value for falling rainwater and could be assumed to represent average cistern water.

4.5.1.3.2 Apparatus.

(a) Eberbach Variable Speed Mechanical Shaker No. 5109.

(b) Bottles, round jar with metal screw cap and aluminum foil liners, 8-ounce size, approximately 2 by 5-1/8 inches.

(c) Centrifuge tubes, ASTM long form type, 100 ml. capacity.

(d) Centrifuge tube rack.

(e) Lamp, microscope substage with 15-watt bulb and daylight glass filter.

(f) Syringe, 2 ml. capacity, Luer, resistance glass.

(g) Thermometer.

4.5.1.3.3 Procedure. Into an 8-oz. round jar, pour 99 ml. of hard water at a temperature of 18° to 24°C. By means of a 2 ml. glass syringe without needle, add 1 ml. of the herbicide sample to the water at the rate of 1 ml. in 5 to 10 seconds.

Hold the outlet of the syringe 1 inch above the water surface and direct the flow of the concentrate towards the center (away from the side) of the jar. Cap the jar immediately. Invert and right the jar once and observe for dispersibility.

Immediately place the test jar and dispersed emulsion on a mechanical shaker

er with the long dimension of the jar parallel to the direction of movement. Agitate for 3 minutes at 2 cycles per second and with a stroke travel of 1-1/4 inch of the shaker bed.

Pour the emulsion immediately into a centrifuge tube supported in a rack.

Conclusions:

Observe and record the condition of the emulsion at the end of 30 minutes, noting the location and volume of any creaming or the presence of free oil. Inspect the emulsion by placing the microscope lamp behind the centrifuge tube to be observed.

Note: Creaming is a well-defined layer of concentrated emulsion occurring either at the top or bottom of the tube.

Repeat the procedure with average and soft water.

Following each use, thoroughly clean all jars, centrifuge tubes, etc., to remove all traces of emulsifier. Finally, rinse at least three times with distilled water and dry in an oven at 105°C.

4.5.1.4 Sedimentation. Thoroughly shake the herbicide sample in its container, turning from top to bottom. Pour 100 ml. into a 100-ml. graduated cylinder and allow to stand for 24 hours. The herbicide shall be observed at the end of this period for evidence of sediment or crystalline solids, and the percent determined.

4.5.1.5 Free acid. A procedure is given for the determination of the free acid as 2-(2, 4, 5-trichlorophenoxy) propionic acid by titrating an alcoholic solution of the herbicide with 0.1 N sodium hydroxide. The end-point of the titration is determined potentiometrically.

4.5.1.5.1 Reagents.

(a) Sodium hydroxide (NaOH), standard 0.1 N solution. Dissolve 4 g. of sodium hydroxide in distilled water and dilute to 1 liter. Standardize against benzoic acid of known purity.

(b) Alcohol, 95 percent. Formula SDA-2B; 1/2-gallon benzene or 1/2-gallon rubber hydrocarbon solvent in 100 gallons ethyl alcohol.

4.5.1.5.2 Apparatus. Beckman, model G, pH meter (or its equivalent) equipped with a glass-calomel electrode system.

4.5.1.5.3 Procedure. Add a 10 g. sample of the herbicide to 100 ml. of alcohol and titrate the solution potentiometrically to a pH of 8.0 with 0.1 N NaOH.

Calculation:

$$\frac{\text{ml. of NaOH} \times N \times 0.2695 \times 100}{\text{grams of sample}}$$

= Percent free acid

4.5.1.6 Flash point. The flash point (Tag Closed Tester) of the herbicide shall be determined in accordance with ASTM D 56-57.

4.5.1.7 Pour point. The pour point of the herbicide shall be determined in accordance with ASTM D 97-57.

4.5.2 Type II—Liquid salt formulation.

4.5.2.1 Concentration of liquid potassium salt of 2-(2, 4, 5-trichlorophenoxy) propionic acid. The sample is decomposed by heating with sodium peroxide in a closed bomb. The chlorine present is converted to inorganic chloride and determined volumetrically by the Volhard procedure. A correction is made for any inorganic chloride initially present.

4.5.2.1.1 Reagents. The reagents are the same as those specified in (a) through (e) in 4.5.1.1.1, and the following: Nitrobenzene, technical.

4.5.2.1.2 Apparatus. The apparatus is the same as that specified in (a) through (d) in 4.5.1.1.2.

4.5.2.1.3 Procedure. Procedures (a) through (g) are the same as in 4.5.1.1.3.

(h) Weigh 5 g. of the herbicide sample into a 250-ml. beaker containing 100 ml.

of water and 5 ml. of dilute HNO₃. Add 2 ml. of Volhard indicator. From a burette, add 1 ml. of standard 0.1 N NH₄SCN. Titrate with standard 0.1 N AgNO₃ until the reddish color disappears and then add 2 ml. excess. Add about 2 ml. of nitrobenzene to the flask and agitate thoroughly to coagulate the precipitate. Titrate the excess AgNO₃ with 0.1 N NH₄SCN to a red-brown end-point which persists for about 30 seconds. The correction for inorganic chloride initially present will be:

$$\frac{\text{ml. of 0.1 N AgNO}_3}{5}$$

× sample weight (paragraph (a))

This correction is also subtracted from the AgNO₃ titration in paragraph (f).

Calculation:

(1) Percent 2-(2,4,5-trichlorophenoxy) propionic acid liquid potassium salt.

The molecular weight of the acid potassium salt is 307.6 and the equivalent weight relative to chlorine is 102.5. One milliliter of 0.1 N AgNO₃ is equivalent to 0.01025 g. of acid potassium salt.

$$\frac{\text{net ml. of 0.1 N AgNO}_3 \times 0.01025 \times 100}{\text{weight of sample}}$$

= Percent liquid potassium salt

(2) Percent acid equivalent.

$$\text{Percent acid potassium salt} \times \frac{269.5}{307.6} = \text{Percent acid equivalent}$$

(3) Pounds per gallon acid equivalent.

$$\frac{\text{Percent acid equivalent}}{100}$$

$$\times 8.33 \times \text{sp. gr. of herbicide}$$

= Pounds per gallon of acid equivalent

(The procedure for the determination of the specific gravity of the herbicide at 20°C. is given in 4.5.1.1.3.)

4.5.2.2 Reaction.

4.5.2.2.1 Acidity.

4.5.2.2.1.1 Procedure. Weigh exactly 10 g. of the herbicide sample and disperse in 100 ml. of distilled water with stirring. Filter and titrate the filtrate immediately with 0.02 N sodium hydroxide (NaOH), using methyl red as indicator. Carry out a blank determination on 100 ml. distilled water with 0.02 N NaOH.

Calculation:

$$\text{Acidity as H}_2\text{SO}_4 \text{ (percent)} = 0.0098 \times (a - b)$$

where a = vol. in ml. of 0.02 N NaOH used for the sample

b = vol. in ml. of 0.02 N NaOH used for the blank

Note: The blank may take the form of a small titre with 0.02 N HCl, in which case

$$\text{Acidity as H}_2\text{SO}_4 \text{ (percent)} = 0.0098 \times (a + c)$$

where a = vol. in ml. of 0.02 N NaOH used for the sample

c = vol. in ml. of 0.02 N HCl used for the blank

Alternatively, the end-point may be determined electrometrically.

4.5.2.2.2 Alkalinity.

4.5.2.2.2.1 Procedure. Weigh exactly 10 grams of the herbicide sample and disperse in 100 ml. of distilled water with stirring. Filter, and titrate the filtrate immediately with 0.02 N hydrochloric acid (HCl), using methyl red as indicator. Carry out a blank determination in 100 ml. of distilled water with 0.02 N NaOH.

Calculation:

$$\text{Alkalinity as NaOH (percent)} = 0.008 \times (d + e)$$

where d = vol. in ml. of 0.02 N HCl used for the sample

e = vol. in ml. of 0.02 N NaOH used for the blank

Note: The blank may take the form of a small titre with 0.02 N HCl, in which case

$$\text{Alkalinity as NaOH (percent)} = 0.008 \times (d - f)$$

where d = vol. in ml. of 0.02 N HCl used for the sample

f = vol. in ml. of 0.02 N HCl used for the blank

Alternatively, the end-point may be determined electrometrically.

4.5.2.3 Sedimentation. The sedimentation

test is the same as that specified for the type I herbicide in 4.5.1.4.

4.5.3 Type III—Granular salt formulation.

4.5.3.1 Concentration of granular potassium salt of 2-(2, 4, 5-trichlorophenoxy) propionic acid. The sample is decomposed by heating with sodium peroxide in a closed bomb. The chlorine present is converted to inorganic chloride and determined volumetrically by the Volhard procedure. A correction is made for any inorganic chloride initially present.

4.5.3.1.1 Reagents. The reagents are the same as those specified in (a) through (e) in 4.5.1.1.1, and the following: Nitrobenzene, technical.

4.5.3.1.2 Apparatus. The apparatus is the same as that specified in (a) through (d) in 4.5.1.1.2.

4.5.3.1.3 Procedure.

(a) Grind several portions of the herbicide sample to a fine powder with a mortar and pestle or micropulverizer, to make a total of 20 to 30 g. Weigh about 1 to 1.5 g. of the herbicide sample in a gelatin capsule to the nearest tenth of a milligram.

Procedures (b) through (g) are the same as in 4.5.1.1.3.

(h) Weigh 15 g. of the herbicide sample into a 250 ml. beaker containing 100 ml. of water and 5 ml. of dilute HNO₃. Add 2 ml. of Volhard indicator. From a burette, add 1 ml. of standard 0.1 N NH₄SCN. Titrate with standard 0.1 N AgNO₃ until the reddish color disappears and then add 2 ml. excess. Add about 2 ml. of nitrobenzene to the flask and agitate thoroughly to coagulate the precipitate. Titrate the excess AgNO₃ with 0.1 N NH₄SCN to a red-brown end-point which persists for about 30 seconds. The correction for inorganic chloride initially present will be:

$$\frac{\text{ml. of 0.1 N AgNO}_3}{15} \times \text{sample weight (paragraph (a))}$$

This correction is also subtracted from the AgNO_3 titration in paragraph (f).

Calculation:

(1) *Percent 2-(2,4,5-trichlorophenoxy) propionic acid granular potassium salt.*

The molecular weight of the acid potassium salt is 307.6 and the equivalent weight relative to chlorine is 102.5. One milliliter of 0.1 N AgNO_3 is equivalent to 0.01205 gram of acid potassium salt.

$$\frac{\text{net ml. of 0.1 N AgNO}_3 \times 0.01205 \times 100}{\text{weight of sample}}$$

= Percent granular potassium salt

(2) *Percent acid equivalent.*

$$\text{Percent acid potassium salt} \times \frac{269.5}{307.6}$$

= Percent acid equivalent

4.5.3.2 Chemical stability. This test is intended for the determination of any chemical breakdown of the granular herbicide when stored at high temperature.

4.5.3.2.1 Apparatus.

(a) Oven, regulated to maintain $55^\circ \pm 3^\circ\text{C}$.

(b) Storage jars, glass, approximately 60 to 120 ml. with screw-caps and aluminum inserts in caps.

4.5.3.2.2 Procedure.

Load a clean dry storage jar approximately half full of the herbicide sample without compaction. Close the jar tightly with the aluminum-insert cap, and store in the oven at $55^\circ \pm 3^\circ\text{C}$. for 14 days.

Remove the jar from the oven and cool to room temperature. Tumble the sample in the jar to redistribute the dust uniformly among the granules, weigh out a 1.0 g. sample, and analyze for potassium salt as described in 4.5.3.1. Calculate the loss, if any, in percent of potassium salt originally present.

4.5.3.3 Reaction. The reaction test is the

same as that specified for the type II herbicide in 4.5.2.2, except that dispersion in the distilled water shall be accomplished by frequent stirring for 1/2 hour.

4.5.3.4 Grain size distribution.

4.5.3.4.1 Sieves. Sieves used in this test shall conform to RR-S-366. Mesh sizes are 2380-micron (U.S. No. 8), 590-micron (U.S. No. 30), and 250-micron (U.S. No. 60). Sieve frame is 8-inch diameter, either full height (inside depth 2 inches) or half height (inside depth 1 inch).

4.5.3.4.2 Sample preparation. Each time any portion is removed from the stock sample, extreme care must be taken to include the proportionate amount of dust with the granules, in order that both the withdrawn sample and the remaining stock be representative in dust content. This necessity is the basis for the following procedure, to be followed in every instance.

(a) Determine the weight of the stock sample.

(b) Tumble the stock sample end-over-end at approximately 30 r.p.m. for 1 minute in a jar whose volume is about twice the apparent volume of the sample.

(c) Separate a 20- to 25 g. portion as a test sample by turntable sampling as follows: A sieve receiver pan, 8-inch diameter, 2-inch depth, is secured in a centered position on a 45 r.p.m. turntable (see 6.4). It is recommended that the drive mechanism for any speeds other than 45 r.p.m. be inactivated to prevent their inadvertent use. A sheet-metal receiver cup, with plain thin unflanged rim is placed within the pan. A 60° angle glass funnel is adjusted on a support so that the outlet tip just clears the top of the cup, and the funnel and cup are set off-center with respect to the turntable by such an amount that the arc projected by the funnel tip across the cup in rotation of the table will bear the same ratio to the complete circle

as the weight of the desired test sample bears to the weight of the whole stock sample. The bore diameter of the funnel stem should be just sufficient to provide free flow for the granular material being treated. With the turntable rotating at 45 r.p.m., the entire stock sample is poured through the funnel so as to be delivered continuously in a steady stream. In case of clogging or bridging, normal flow is restored (the table still turning) by probing with a stout wire flattened on the lower end. The portion for test falls in the cup, while the remainder falls in the pan outside the cup. The cup is removed, any dust clinging to the outside surfaces is brushed back into the pan, and the cup with its load is weighed for determination of the best sample weight; the remainder in the pan is returned to the stock jar.

4.5.3.4.3 Procedure. Screen the test sample through a nest of sieves of the designated mesh sizes, using a single-eccentric type mechanical shaker that imparts to the sieve a rotary motion and tapping action of uniform speed of approximately 300 gyrations and approximately 150 taps per minute. Continue the screening for 15 minutes. Weigh the residues and calculate the percentage passing through each sieve. In the weighing and transferring of the sieve sample and its size fractions, any dust clinging to sieve cloth, sieve wall, scale pan and miscellaneous surfaces, or clogging the sieve-cloth apertures, is to be regarded as part of the finest fraction (sieve-pan fraction). Neither this dust nor the material actually entering the sieve pan need be recovered for weighing; the granules lying on the respective sieve cloths are to be recovered and weighed, and the pan fraction calculated by difference.

4.6 Inspection of preparation for delivery. The packaging, packing, and marking of the herbicides shall be inspected to determine conformance to the requirements of section 5.

5. PREPARATION FOR DELIVERY

(For civil agency use, the definitions and applications of the levels of packaging and packing shall be in accordance with Fed. Std. No. 102.)

5.1 Packaging. Packaging shall be level A or C, as specified (see 6.2).

5.1.1 Level A.

5.1.1.1 Types I and II herbicides. The herbicides shall be packaged in 5-gallon tight-head metal pails conforming to PPP-P-704, type I, class 3; 30-gallon tight-head drums conforming to PPP-D-705, type I; or 55-gallon nonremovable-cover metal drums conforming to PPP-D-729, type II, as specified (see 6.2).

5.1.1.2 Type III herbicide. The herbicide shall be packaged in 1-pound, 5-pound, or 10-pound quantities in metal cans conforming to PPP-C-96, type V, class 2, or in 50-pound quantities in fiber drums conforming to PPP-D-723, type III, grade A, as specified (see 6.2).

5.1.2 Level C. The herbicides shall be packaged in accordance with the manufacturer's standard practice. The size of containers shall be as specified (see 6.2).

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

5.2.1 Level A.

5.2.1.1 Types I and II herbicides. The herbicides packaged in 5-gallon, 30-gallon, or 55-gallon metal containers as specified in 5.1.1.1 require no overpacking.

5.2.1.2 Type III herbicide. The herbicide packaged in 1-pound, 5-pound, or 10-pound metal cans as specified in 5.1.1.2 shall be packed in accordance with the overseas shipment requirements of the appendix to PPP-C-96. The herbicide packaged in 50-pound fiber drums as specified in 5.1.1.2 requires no overpacking.

5.2.2 Level B.

5.2.2.1 *Types I and II herbicides.* Not applicable.

5.2.2.2 *Type III herbicide.* The herbicide packaged in 1-pound, 5-pound, or 10-pound metal cans as specified in 5.1.1.2 shall be packed in accordance with the domestic shipment requirements of the appendix to PPP-C-96. The herbicide packaged in 50-pound fiber drums as specified in 5.1.1.2 requires no overpacking.

5.2.3 *Level C.* The herbicides shall be packed in a manner which will insure arrival at destination in satisfactory condition and which will be acceptable to the carrier at lowest rates. Containers and packing shall comply with rules and regulations of carriers as applicable to the mode of transportation.

5.3 Marking.

5.3.1 *Civil agencies.* Interior packages and shipping containers shall be marked in accordance with Fed. Std. No. 123.

5.3.2 *Military agencies.* In addition to the requirements of 3.4 and to any special marking required by the contract or order, interior packages and shipping containers shall be marked in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use.

6.1.1 *Type I.* The type I silvex herbicide covered by this specification is intended for use in the selective control of many herbaceous weeds and some woody plants growing in pastures, rangelands, fence rows, right-of-ways, and ditchbanks. It is used much the same way as 2, 4, 5-T herbicide except that (1) it is safer where drift onto cotton is a hazard, (2) it is more effective as a foliage spray on some oaks, maples, salt cedar, redbud, cherokee rose, trumpet creeper, trumpet vine, and yucca, (3) it is not as effective for basal bark and cut sur-

face applications, and (4) it is more effective on mouse-ear, chickweed, and henbit.

Susceptible species in addition to those listed above are: Blackjack oak, northern oaks (pin, red, white, scrub), post oak, sand shinnery oak, maples (red, sugar, big leaf), wild blackberry, black gum, cherry, elm, ground ivy, poison ivy, mesquite, broomweed, cocklebur, cotton weed, curly indigo, lamb's quarters, Mexican weed, pokeberry, ragweed, salt cedar, and sunflower.

Among the species hard to kill are: African rue, ash, hawthorn, hickory, mulberry, Osage-orange, persimmon, prickly pear cactus, rabbit brush, red bay, red vine, multiflora rose, sagebrush, smilax, and sweet gum.

Buckbrush, coralberry, juniper, and wood's rose are resistant.

Although the type I silvex herbicide is intended primarily for use on land, it may also be used for aquatic weed control in ponds and other still waters and is effective against certain submergent weeds such as fanwort, water milfoil, bladderwort, and waterweed.

6.1.2 *Types II and III.* The types II and III silvex herbicides covered by this specification are intended for use in the control of a wide variety of submersed, floating, and emersed aquatic weeds in lakes and ponds. They are less toxic than the ester formulations to fish. They do not injure land plants adjacent to treated areas. They are similar to 2,4-D herbicide in their effect on many species of submersed aquatic weeds and are more effective on some.

Susceptible weeds are: Arrowhead, coontail, naiad, fanwort, mule's ear, parrotsfeather, pickerel weed, primrose willow, spatterdock, water hyacinth, water lilies, water milfoil, water plantain, water shield, water stargrass, and water starwort.

Bulrush, watercrowfoot, waterweed, duckweed, and some pondweeds are harder to kill.

6.2 Ordering data. Purchasers should exercise any desired options offered herein and procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Type of silvex herbicide required (see 1.2.1).
- (c) Level of packaging and level of packing required (see 5.1 and 5.2).
- (d) Size of container (see 5.1.1 and 5.1.2).

6.3 References for use in connection with the active ingredient concentration tests (see 4.5.1, 4.5.2, and 4.5.3).

Parr Manual No. 121, Peroxide Bomb Apparatus and Methods, Parr Instrument Company, Moline, Illinois (1950). Parr, S.W., J. Am. Chem. Soc., 30; 764 (1908). Volhard, J.J., Pract. Chem., 9; 217 (1874).

6.4 Turntable. An approved turntable for use in conducting the grain size distribution test in 4.5.3.4 is the General Industries,

Model TR, motorized 9-inch phonograph turntable, or equivalent.

CUSTODIANS:

Army—MU
Navy—YD
Air Force—68

Review activities:

Army—MU, MO, MD
Navy—YD, MS
Air Force—68

User activities:

Army—SM
Navy—MC

Preparing activity:

Navy—YD