Kentucky Method 64-242-0~~83~~
Revised ~~1/7/03~~04/15/08
Supersedes 64-242-~~00~~03
Dated ~~1/13/00~~01/17/03

# HERBICIDES

1. SCOPE: The following test methods cover material for control of weeds, brush and other noxious vegetation on highway right of way. Herbicides shall meet specifications indicated on plans, proposals or bidding invitations.

2. APPARATUS AND MATERIALS:

   2.1. 500 ml Separatory Funnel

   2.2. Ethyl Ether

   2.3. Electric Fan

   2.4. Formula 3A Alcohol

   2.5. Phenolphthalein Indicator

   2.6. 0.1N Sodium Hydroxide

   2.7. Stirring Bar and Magnetic Stirrer

   2.8. Centrifuge

   2.9. pH Meter

   2.10. Hydrometer

3. SAMPLE: A 0.95 liter (quart) sample of each type per source shall be submitted to the Division of Materials. Samples shall be taken with care using clean equipment and clean containers.

4. PROCEDURES FOR 2,4,-D (ALKANOLAMINE SALT OF 2,4-D):

   4.1. Total 2,4-D Acid:

      4.1.1. Weigh by difference from a stoppered vial 1.5 to 2 g sample. Add sample to 150 ml beaker. Add 50 ml of distilled water. Transfer to 500 ml pear-shaped separatory funnel. Rinse beaker with water, and add to funnel. Add 10 ml of $H_2SO_4$ (10%) to funnel, and swirl to mix thoroughly. Extract with 75 ml of ether. Shake vigorously. Invert funnel and open stopcock to relieve pressure. Set funnel in ring holder until layers separate. Draw off bottom layer into another separatory funnel. Save ether

layer in first funnel. To water layer in second funnel add 75 ml of ether, and extract as before. Draw off bottom layer, and discard. Combine two ether layers. Rinse empty funnel with ether and add to other funnel. Add 10 ml of distilled water and shake vigorously. Draw off bottom layer and discard. Repeat this procedure two more times. Place a plug of cotton in a filter funnel, and saturate with ether. Filter sample through the funnel into a clean 400 ml beaker. Rinse separatory funnel with ether and filter through same cotton. To the filtrate add 25 ml of distilled water and a few glass beads. Evaporate at a very low heat until about 25 ml of ether remains above the water layer. Remove from hot plate, and complete the evaporation of the ether layer by placing sample in front of a medium speed electric fan. When ether layer is completely evaporated, add 100 ml of Formula 3A ethyl alcohol, and stir until the 2,4-D has dissolved. Add 5 - 7 drops of phenolphthalein indicator, and titrate with 0.1N NaOH carefully until 1 drop of NaOH gives a pink end point.

4.1.2. Calculations:

$$\frac{mL\ NaOH\ x\ 2.21\ x\ N\ \frac{NaOH}{0.1}}{Wt.\ of\ Sample} = \%\ 2,4\text{ - }D\ Acid$$

4.2. Foam Test: Add 5 ml of sample to 100 ml of tap water in a 250 Erlenmeyer flask, stopper, and shake vigorously. At the instant the shaking is stopped, start a stop watch. Stop the watch the moment the foam breaks. This usually requires 4 - 5 seconds.

4.3. Dilution Test: Use the solution prepared for the foam test. Add teflon covered stirring bar, and stir for two hours on magnetic stirrer. The solution should be clear after 2 hours.

4.4. 2, 4-D Acid Equivalent in Grams Per Liter (Pounds Per Gallon multiplied by 119.8264):

4.4.1. Determine specific gravity of sample at 25°C (77°F) by using a hydrometer.

4.4.2. Calculations: % 2, 4-D x Sp. Gr. x 8.33 x 119.8264 = grams/liter

4.5. Sediment: Fill a Goetz centrifuge tube to the 100 ml mark with the sample. Counter balance with another tube and water. Centrifuge at 1500 RPM for about 30 minutes. The first graduation on the stem of the centrifuge tube is 0.01 ml. If the sediment is below this mark, report sediment as less than 0.01 percent.

4.6. pH Determination: Determine pH of sample at 25°C (77°F) using a regular laboratory pH meter, and using standard procedure for operating the meter.

5. CALCULATIONS FOR 2,4-D: Calculations are included in the Procedures.

6. PRECAUTIONS FOR 2,4-D: The usual precautions, such as avoiding open flames, must be taken

when using ether.

7. REPORT FOR 2,4-D:

7.1. % 2,4-D

7.2. Results of Foam Test in Seconds

7.3. Results of Dilution Test

7.4. 2,4-D Acid Equivalent in Grams per Liter (Pounds per Gallon multiplied by 119.8264)

7.5. % Sediment

APPROVED _____
DIRECTOR
DIVISION OF MATERIALS

DATE 04/15/08

~~APPROVED _____~~
~~Director~~
~~DIVISION OF MATERIALS~~

~~DATE 1/7/03~~

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