

Cover Sheet for

**ENVIRONMENTAL CHEMISTRY METHOD**

***Pesticide Name:*** Pebulate

***MRID #:*** 415568-06

***Matrix:*** Soil

***Analysis:*** GC/NPD

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Method No RRC-85-97

Date 12/4/85

Supervisor \_\_\_\_\_

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TITLE

**GAS CHROMATOGRAPHIC DETERMINATION OF PEBULATE IN SOIL**

**I. SCOPE**

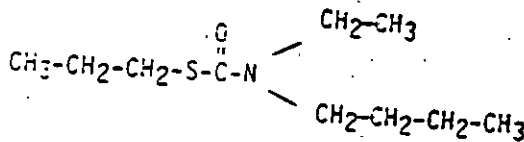
This method is intended for the determination of residues of pebulate in soil. The method has been validated at pebulate levels between 0.01 ppm and 1.0 ppm.

**II. SUMMARY OF METHOD**

A known quantity of sample is extracted with toluene, and the extract is analyzed for pebulate by capillary gas chromatography using nitrogen-phosphorus detection.

**III. INTRODUCTION**

Pebulate is the active ingredient in TILLAM® Selective Herbicide, and has the following chemical structure:



**IV. APPARATUS AND REAGENTS**

**A. Apparatus**

1. Gas Chromatograph. Hewlett-Packard Model 5880A, equipped with nitrogen-phosphorus detector and splitless capillary inlet. An equivalent chromatograph may be used.
2. Chromatographic Column. Fused-silica capillary, 12 m x 0.20 mm I.D., cross-linked methyl silicone, 0.33 micron film thickness. Hewlett-Packard 19091A Opt. 101 or equivalent.
3. Shaker. Reciprocating, Eberbach or equivalent.

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**B. Reagents**

1. Toluene. Nanograde® or equivalent.
2. Acetone. Nanograde® or equivalent.
3. Sodium Sulfate. Reagent grade.
4. Deionized Water. Produced by a Millipore Milli-Q or equivalent system.
5. Pebulate Standard. Available from Stauffer Chemical Co., 1200 S. 47th Street, Richmond, Ca 94804.
6. Calibration Solutions. Prepare  $\approx 1000$   $\mu\text{g/mL}$  stock solution by dissolving pebulate in toluene. Prepare calibration solutions of 0.01, 0.05, 0.1 and 1.0  $\mu\text{g/mL}$  by diluting the stock solution with toluene.
7. Fortification Solutions. Prepare a 1000  $\mu\text{g/mL}$  stock solution by dissolving pebulate in acetone. Prepare fortification solutions at other concentrations as required by diluting the stock solution with acetone.

**V. PROCEDURE**

**A. Extraction**

Place a 50-g sample of thoroughly mixed soil in an 8-ounce wide-mouth glass bottle; add 100 mL water and 50 mL toluene and cap the bottle with a PTFE-lined cap. Shake the bottle for two hours with a mechanical shaker and then centrifuge to separate the phases. Transfer a portion of the upper (toluene) phase to a 1-oz bottle that contains approximately 1 g sodium sulfate.

**B. Determination of Soil Wet and Dry Weights**

Transfer approximately 20 g of soil into a weighed aluminum pan. Determine and record the weight of the pan plus soil before and after drying at 105°C for 12 to 24 hours.

**C. Gas Chromatographic Conditions**

Use the following conditions with a Hewlett-Packard Model 5880A chromatograph:

Column initial temperature      90°C  
Initial time                              1.00 min



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Temperature programming rate 30 C/min  
Column final temperature 130°C  
Injector temperature 220°C  
Detector temperature 300°C  
Carrier gas Helium  
Carrier gas pressure 20 psig  
Make-up gas flow (He total) 30 ml/min  
Air flow 80 ml/min  
Hydrogen flow 3.5 ml/min  
Injection 2 µL splitless  
Purge function activated at 0.5 min  
Quantitation by on-line integrator or peak height

Under the above conditions the elution time of pcpoluate is approximately 3.5 min. The elution time depends on the length and condition of the column, and should be determined empirically by injection of a calibration solution.

D. Calibration and Sample Analysis

Inject the calibration solutions and sample extracts into the gas chromatograph using the conditions given above. For calibration, choose the calibration solution that generates the peak area or height that is closest in size to that in the sample extract. Make replicate injections of this solution until a constant calibration factor results ( $\pm 10\%$ ). Re-inject the calibration after every six sample extract injections. Dilute the sample if necessary to obtain a response within the calibration. If the analyte response produced by the calibration solution during the analysis by more than  $\pm 10\%$ , re-analyze the sample extracts after calibration has been re-established.

VI. CALCULATIONS

A. Calibration Factors

Calculate a response factor, F, for each injection of a calibration solution as follows:

$$F (\text{ng/area counts or ng/cm}) = \frac{C \times V}{H}$$

where C = concentration of calibration solution, ng/µL  
V = volume of calibration solution injected, µL  
H = peak area (counts) or height (cm)



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**B. Analyte in Sample**

Calculate the concentration of the analyte in the original sample as follows:

$$\text{Residue (ppm) as wet weight} = \frac{F \times P}{V \times S}$$

F = calibration factor, ng/area counts or ng/cm

P = peak area (counts) or height (cm) from sample extract

V = volume of sample extract injected,  $\mu\text{L}$

S = concentration of sample in extract injected,  $\text{mg}/\mu\text{L}$

For conversion of wet-weight residues to dry-weight residues use the following formula:

$$\text{Residue (ppm) as dry weight} = \text{Residue (ppm) as wet weight} \times \frac{W}{D}$$

where W = original weight of soil taken, g  
D = weight of soil after drying, g

**VII. DISCUSSION****A. Recoveries**

Soil samples for recovery determinations were fortified at 0.01 to 1.0 ppm by addition of the appropriate amount of fortification solution to a weighed subsample of untreated soil prior to extraction. The mean recovery was 105%. Recovery data are summarized in Table I.

Figure 1 shows examples of chromatograms of calibration solution, and of extracts from untreated, fortified untreated, and treated soil samples.

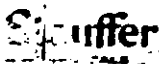
**B. Interferences**

As shown in Figure 1, there were no significant interferences in the chromatograms from extracts of untreated soil.

**VIII. REFERENCES**

1. WRC Notebook No. 9993, pp 35 to 48.

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**IX. SAFETY PRECAUTIONS**

**A. Toluene and Acetone**

- Flammable
- Vapors harmful
- Avoid contact with skin and clothing
- Use with proper ventilation; avoid breathing vapor

**B. Pebulate**

- Avoid contact with skin and clothing



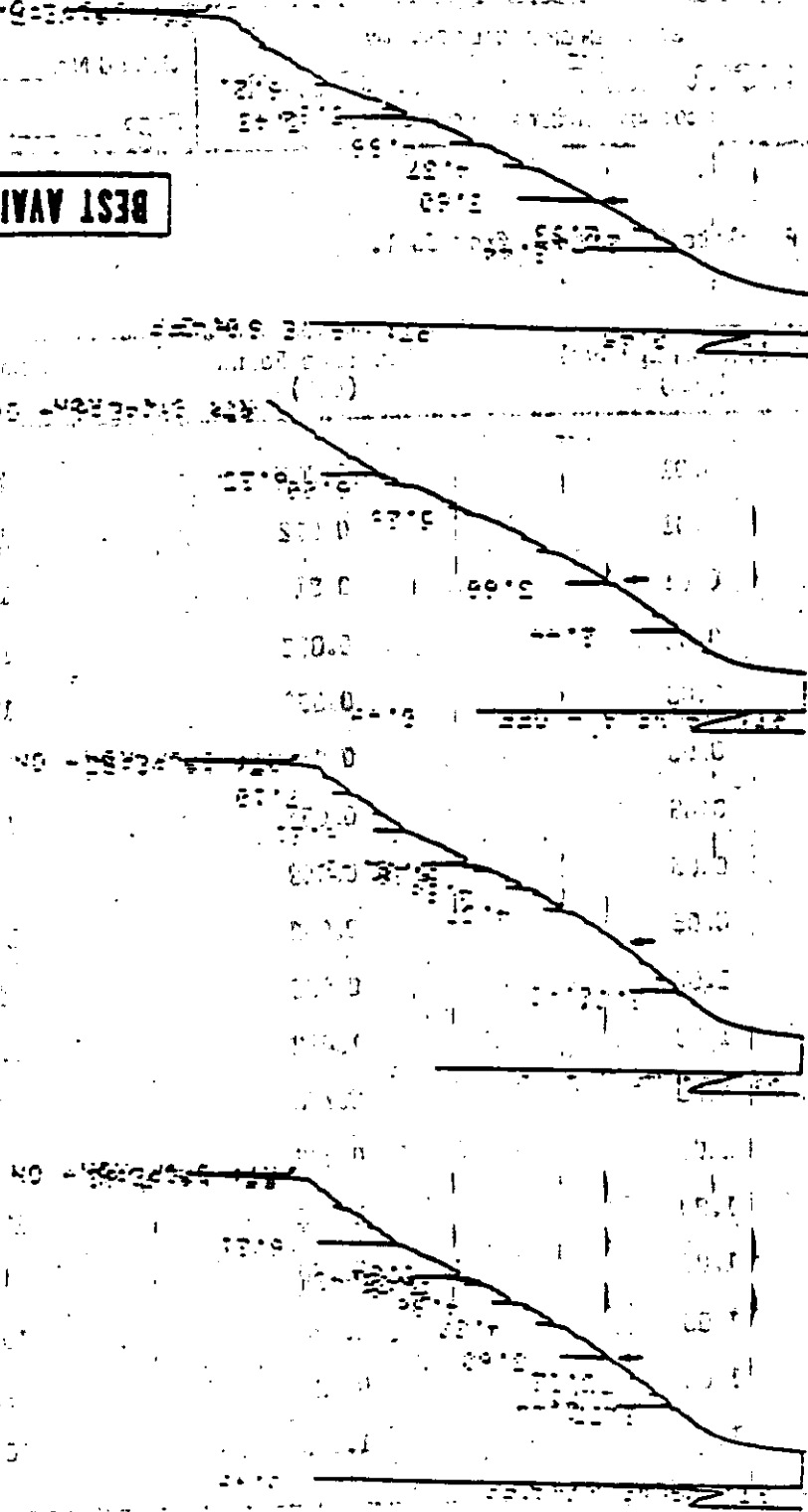


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Table 1. Pebulate Recoveries from Soil:

Fortification Level (ppm)	Pebulate Found (ppm)	% Recovery
0.01	0.011	110
0.01	0.012	120
0.01	0.01	100
0.01	0.012	120
0.05	0.054	120
0.05	0.055	110
0.05	0.054	120
0.05	0.049	98
0.05	0.052	104
1.00	0.965	97
1.00	1.012	101
1.00	0.796	98
1.00	0.955	96
1.00	1.070	107
1.00	0.950	95
1.00	1.028	103
1.00	0.929	93
1.00	1.049	105

Figure 1. Typical Chromatograms of Pebulate in Soil



A. Calibration  
Solution,  
0.01 µg/mL

B. Untreated Soil,  
1 g/mL

C. Untreated Soil,  
1 g/mL fortified  
with pebulate at  
0.01 µg/g

D. Treated Soil,  
1 g/mL

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