TITLE PAGE

Determination of Folpet and Phthalimide Residues in Soils using Gas Chromatography

METHOD NO. 146-002

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I. SUMMARY/INTRODUCTION

Samples are shaken with acetonitrile/water and filtered. The filtrate is extracted with methylene chloride and the dried organic phase is concentrated. The residues are resuspended in acidified water/acetonitrile and passed through a 6 cc C18 Mega Bond Elut column. The eluate is partitioned into methylene chloride, concentrated, and volume is adjusted to 5 mL. The compounds in the organic concentrate are analyzed by GC using ^{63}Ni electron capture detection.

II. MATERIALS/METHODS

A. Equipment

Orbital Shaker or equivalent
Rotary Evaporator
Glass funnels
Centrifuge bottles, 250 mL
Buchner funnels
Nonadsorbent Cotton
Filter flasks, 500 mL
Separatory funnels, 500 mL
Flat-bottomed flasks, 500 mL and 100 mL, or equivalent
Whatman #4 filters
Whatman glass microfibre filters or equivalent
Mega Bond Eluts, 6 cc C18
Class A volumetric glassware
N-Vap Analytical Evaporator or equivalent

B. Reagents and Standards

1. Reagents

Acetonitrile, Optima Grade
Methylene chloride, Optima Grade
Methanol, Optima Grade
Hexane, Optima Grade
Toluene, Optima Grade
Benzene, Optima Grade
Glacial Acetic Acid, ACS reagent grade or equivalent
Sodium sulfate (anhydrous), reagent grade
Water, Type 1

2. Standards

Folpet, 97% purity
Phthalimide, 97% purity

3. Standard Solutions

a. Folpet stock solution: Weigh 100 mg each of the Folpet standard into a tared 100 mL volumetric flask and dilute to volume with toluene. The solution contains 1000 ug/mL of Folpet.

b. Phthalimide stock solution: Weigh 10 mg each of the Phthalimide standard into a tared 100 mL volumetric flask and dilute to volume with benzene. The solution contains 100 ug/mL of Phthalimide.

c. From the Folpet stock solution, subsequent analytical dilutions, via class A volumetric pipets, are made as follows:

Volume mL	Standard Concentration ug/mL	Final Volume mL	Final Concentration ug/mL
3.0	1000	100	30.00
1.0	1000	100	10.00
10.0	10.00	100	1.000
5.0	10.00	100	0.500
2.0	10.00	100	0.200
1.0	10.00	100	0.100
10.0	0.500	100	0.050
10.0	0.200	100	0.020

Store refrigerated.

d. From the Phthalimide stock solution, subsequent analytical dilutions, via class A volumetric pipets, are made as follows:

Volume mL	Standard Concentration ug/mL	Final Volume mL	Final Concentration ug/mL
1.0	100	100	1.000
0.5	100	100	0.500
20.0	1.000	100	0.200
10.0	1.000	100	0.100
10.0	0.500	100	0.050
10.0	0.200	100	0.020
10.0	0.100	100	0.010
10.0	0.050	100	0.005

Store refrigerated.

C. Analytical Procedure

1. Weigh 10.0 ± 0.05 grams of soil into a 250 mL centrifuge bottle.

2. Prepare fortification samples by pipeting (Class A volumetric) appropriate standard solution onto designated samples.

3. Add 100 mL 30:70:0.2 Type 1 water/acetonitrile/acetic acid and shake for 30 minutes on an orbital shaker.

4. Filter the extract through a Buchner funnel fitted with a Whatman glass microfibre filter and a Whatman #4 filter, collecting the extract in a 500 mL filter flask. Rinse the funnel with 20 mL of 30:70:0.2 Type 1 water/acetonitrile/acetic acid.

5. Transfer the sample extract into a 250 mL separatory funnel, add 100 mL methylene chloride, and shake vigorously for 1 min.

6. Filter the lower organic phase through a funnel, containing 1 inch of anhydrous sodium sulfate supported by a non-adsorbent cotton plug, into a 500 mL flat-bottomed flask.

7. Add 100 mL of methylene chloride to the aqueous layer, shake for 1 min., and combine the lower organic phase with the organic phase from above, using the same sodium sulfate filter.

8. Rinse the funnel with 25 mL methylene chloride.

9. Rotovap to dryness at 30-35°C.

10. Precondition a 6 cc C18 Mega Bond Elut with 2 mL methanol followed by 5 mL Type 1 water.

11. Resuspend the residue from C9 with 5 mL 90:10:0.2 Type 1 water/acetonitrile/acetic acid and transfer to the preconditioned C18 Mega Bond Elut. Discard all eluate to this point.

12. Rinse the flask with 5 mL 30:70:0.2 Type 1 water/acetonitrile/acetic acid and transfer to the C18 Mega Bond Elut, collecting the eluate.

13. Add 10 mL 30:70:0.2 Type 1 water/acetonitrile/acetic acid to the C18 Mega Bond Elut, collecting the eluate.

14. Transfer the eluate to a 60 mL separatory funnel. Rinse the tube with 10 mL methylene chloride, adding it to the separatory funnel.

15. Shake the separatory funnel for 30 seconds, allow to settle, and filter the lower organic phase through a funnel, containing 1/4 inch of anhydrous sodium sulfate supported by a non-adsorbent cotton plug, into a 100 mL flat-bottomed flask.

16. Add 10 mL of methylene chloride to the aqueous layer, shake for 1 min., and combine the lower organic phase with the organic phase from above, using the same sodium sulfate filter.

17. Rinse the funnel with 10 mL methylene chloride.

18. Rotovap to dryness at 30-35°C.

19. Resuspend the residue in 5 mL hexane.

20. Analyze by GC using ^{63}Ni electron capture detection.

D. Chromatography

1. Gas Chromatograph

Perkin Elmer 8500 gas chromatograph equipped with ^{63}Ni electron capture detector, VG Multichrom computer data system and Perkin Elmer AS 8300 autosampler.

GC Column:
J & W Fused silica capillary column
Stationary phase: DB-1701
Film Thickness: 1 um
Column Dimension: 30 m x 0.53 mm

2. Suggested Operating Conditions

Column temperature	100°C, ramp to 200°C at 15°C/min, hold 2 min, ramp to 240°C at 2°C, hold 5 min.
Injector temperature	150°C
Detector temperature	350°C
Volume Injected	2 uL
Carrier gas (Ar/CH_4)	6 mL/min
Quantification Height	External Standards, Peak

E. Soil Moisture

1. Weigh aluminum weigh dish and record. Do not tare.
2. Add soil until weight increases 10 to 11 g. Record weight.
3. Place in oven at 120°C for at least 12 hours.
4. Set in desiccator to cool for 15 minutes.
5. Weigh and record.

M = (Wet Weight - Dry Weight)/(Dry Weight - Al dish Weight)

F. Calculations

1. Calibration

A standard curve is developed for each set of samples analyzed through the analysis of external standards spanning the range of the expected residues. Amounts of Folpet and Phthalimide are calculated from the equation resulting from regression analysis of the calibration data (calibration curve is obtained by plotting ug/mL injected versus peak height).

2. Determination of Sample Residues (ppm)

The concentration of the analyte (ug/mL) in the sample is calculated from the regression analysis of the calibration data.

The sample residue in terms of ppm is calculated using:

Conc (ppm) = Conc (ug/mL) * DF * FV / SW

SW = sample weight (g)
FV = final volume (mL)
DF = dilution factor (if necessary)

The sample residue in terms of ppm adjusted for soil moisture is calculated using:

Adj Conc (ppm) = Conc (ppm) / (1 - M)

M = moisture content of sample