

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Propargite

MRID #: 449686-01

Matrix: Soil

Analysis: GC/FPD/FID

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STUDY TITLE

449686-01

Analytical Method for Propargite and Its Degradates in Soil

Data Requirement

Not Applicable

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Performing Laboratory

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Related Reports

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Analytical method, soil, propargite, Omite, TBPC

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This submission is not considered a "study" as defined by 40CFR 160 and as such falls outside the scope of GLP requirements. It consists of an analytical method which has been compiled and reformatted to conform more closely with data reporting guideline #850.7100 (draft) and EU guidelines under commission directive 96/46/EC of 16 July 1996. Information for this report was taken from previously submitted GLP studies as indicated on the title page.

Certification

This analytical method was compiled from information in the following reports:

- 1) Uniroyal project 8816
- 2) Uniroyal project 8320
- 3) Uniroyal project 8844
- 4) Uniroyal project 8845

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TABLE OF CONTENTS

| | <u>Page</u> |
|--|--------------------|
| Title Page | 1 |
| Statement of Confidentiality | 2 |
| Statement of Adherence to GLP | 3 |
| Certification | 4 |
| Table of Contents | 5-7 |
| Summary | 8 |
| Materials (Equipment, Reagents, Standards) | 8 |
| Equipment | 8 |
| Reagents/Supplies | 9 |
| Analytical Standards | 9 |
| Safety and Health | 9 |
| Analytical Methods | 10 |
| Principle of the Method | 10 |
| Types of Soils | 10 |
| Sample processing | 10 |
| Extraction Method | 11-12 |
| Fortifications | 12 |
| Clean-up | 13 |
| Derivatization | 14 |
| Instrumentation | 14-15 |
| Potential Interferences | 15 |

| | |
|--|-------|
| Confirmatory Techniques..... | 15 |
| Time Required for Analysis..... | 15 |
| Modification or Potential Problems..... | 16 |
| Methods of Calculation..... | 16-20 |
| Copies of Chromatograms..... | 20 |
| Method Validations..... | 20 |
| Accuracy(USA)/Recovery(EU)..... | 20-21 |
| Precision..... | 21 |
| Limit of Quantitation..... | 22 |
| Limit of Detection..... | 22 |
| Specificity..... | 22 |
| Ruggedness..... | 22 |
| Limitations..... | 23 |
| Independent Laboratory Validation(ILV)(USA)/Reproducibility(EU)..... | 23 |
| Conclusions..... | 23 |

| | <u>Page</u> |
|--|-------------|
| TABLES | 24 |
| Table 1... Validation Parameters for the Analysis of Propargite and TBPC in Soil | 25-26 |
| | |
| APPENDICES | 27 |
| Appendix 1..... Certificates of Analysis and MSDS Sheets for Propargite and TBPC | 28-33 |
| Appendix 2..... Original Method for Determining Propargite (Omite®) and TBPC (formerly called Omite glycol ether) in Soil – Method of Sisken (1973) | 34-41 |
| Appendix 3..... Typical raw data sheet for the analysis of propargite in soil from Study 8845 Standard curve for the analysis of propargite in soil from Study 8845 Chromatographic traces for propargite in soil at 0.0 ppm (check), 0.1 ppm & 1.0 ppm (spikes) from Study 8845 | 42-45 |
| Appendix 4..... Typical raw data sheet for the analysis of TBPC in soil Chromatographic traces for TBPC in soil at 0.0ppm (check), 0.1 ppm and 1.0 ppm (spikes) from Study 8845 | 46-48 |

SUMMARY

Propargite and its metabolite 2-(p-t-butylphenoxy)-cyclohexanol [TBPC] were extracted from soil with acetone. The extract was freed from water and then analyzed by GC using a flame photometric detector in sulfur mode for propargite and a flame ionization detector for TBPC.

A. MATERIALS

A.1 Equipment

Balance

Glass jars (1 liter)

#4 filter paper

Graduated cylinder (500 ml)

Separatory funnel (500 ml)

Round Bottom flask (100 ml)

Rotovap

Assorted test tubes

Steam bath

Glass vials with Teflon lined lids.

Pasteur pipette (9 inch)

Glass wool

A.2 Reagents/Supplies

All solvents should be pesticide residue grade.

| | |
|---------------------------------|---|
| Propargite analytical standard | Obtain from Uniroyal Chemical Co., Inc. |
| TBPC analytical standard | Obtain from Uniroyal Chemical Co., Inc. |
| Acetone | Various suppliers. |
| Toluene | Various suppliers. |
| Chloroform | Various suppliers. |
| Decanol | Various suppliers. |
| Hexane | Various suppliers. |
| Dichloromethane | Various suppliers. |
| Activated Alumina (80-200 mesh) | Alcoa Type F-20. |

A.3 Analytical Standards

Analytical standards of propargite and TBPC are available from Uniroyal Chemical Company, Inc. division of CK Witco. Standards are kept frozen. Certificates of Analysis (COAs) for these two standards are shown in Appendix 1. The COAs also show the structures of the standards and their typical purities. Appendix 1 also contains MSDS sheets for the standards. One should obtain MSDS sheets for the solvents directly from their suppliers.

B. SAFETY AND HEALTH

This method should be performed by trained chemical personnel. Hazards associated with the chemicals used in this analytical method are shown in the MSDS sheets in Appendix 1.

C. ANALYTICAL METHODS

C.1 Principle of the Methods

Soil samples are extracted with acetone. Chloroform is then added to allow separation of any water that may have been present in the soil sample. The chloroform-acetone layer (lower) is separated and a small amount of decanol "keeper" is added to it. The solvents are removed by evaporation on a rotovap and finally on a steam bath. For the analysis of propargite, hexane is added to bring up to volume and analysis is done by GC with a flame photometric detector in sulfur mode. For TBPC dichloromethane is added to bring up to volume and analysis is done by GC with a flame ionization detector.

C.2 Types of Soils

This method is predicted to be applicable to most soil types. A number of propargite field dissipation studies have been done using this method (*see title page for related studies*). In Uniroyal Chemical Co., Inc. Study 8845 soil from a Georgia, USA location was analyzed to a depth of 36 inches. The soil composition varied depending upon the depth from sandy loam (0-1 ft depth), sandy clay loam (1-2 ft. depth), and clay (2-4 ft depth). The analytical method worked equally well on all these types of soil.

C.3 Sample Processing

The soil samples are normally received frozen and are stored frozen at $-20^{\circ}\pm 2^{\circ}\text{C}$. Before analysis they are thawed and hand mixed.

C.4 Extraction Method

GENERAL NOTE: Weigh out and extract the soils on an "as received" (wet) sample basis. Conduct an air dry moisture determination on a separate portion of each sample. Calculate residues on both the "as received" and on the air dried soil basis.

1. Weigh out a 200 g sample of soil into a quart jar. Spike at this point if applicable. (Note: The weighed sample is "as received" or wet sample basis. Do not pre-dry the soil.)
2. Add 200 mL acetone and shake, by hand every 10 minutes for the next two hours.
3. Decant the acetone through #4 filter paper into a 500 mL graduated cylinder.
4. Add 100 mL acetone to the soil remaining in the jar. Shake twice at 5-minute intervals. Decant the acetone again into the graduated cylinder through the same filter paper.
5. Transfer the soil into the filter paper. Rinse the jar with 75 mL of acetone and pour both washings and the remaining soil into the filter paper.
6. Rinse the soil in the filter paper with 25 mL of acetone. Continue rinsing the jar and the soil with additional acetone until the volume in the cylinder reaches the 400 mL mark. Mix well.
7. Transfer a 200 mL aliquot of the extract to a 500 mL separatory funnel. Add 200 mL chloroform and shake for 1 minute. Let stand for 5 minutes.
8. Drain the chloroform-acetone layer into a 1000 mL round bottom flask through #4 filter paper. Discard the water layer. (Note: Amount of water layer depends on the moisture content of the soil.)
9. Add 0.1 mL of 1% decanol in acetone as a keeper, and evaporate using a rotovap to 2-3 mL. Transfer to a test tube with chloroform.
10. Adjust the volume to 4 mL, mix well and divide the extract into two equal parts of 2 mL each in test tubes (one tube for propargite and one tube for TBPC). Add 1 drop of 1% decanol to each tube.

11. Evaporate each tube to dryness using a steambath, then bring up to a final volume of 2 mL each: one tube for propargite in hexane; the other tube for TBPC in dichloromethane. (For each fraction, the final volume = 2 mL) (1 mL = 25.0 g)
12. Submit for GC analysis.
13. To determine the air dry soil moisture, proceed as follows:
 - Using a top loader balance, weigh a disposable aluminum weighing dish to one decimal accuracy.
 - Add 10.0 g \pm 1.0 g of "as received" wet soil and record the weight.
 - Place the dish, uncovered on a counter or cabinet top for 24 hours. Temperature is to be "room temperature" (usual range is 60-75° F).
 - Reweigh the dish plus dry soil.

C.5 Fortifications

During the course of analyzing the samples for Uniroyal study 8845, a number of method spikes were used ranging from 0.1 ppm to 2.0 ppm. Fortifications were carried out by adding the appropriate standard solution directly onto a weighed portion of an untreated soil sample check in the extraction container and allowing it to dry prior to adding the extraction solvent. The checks and fortified (spiked) samples were extracted along with each set of treated samples.

A minimum of one spike was run for each sample set. This is approximately one spike for every ten treated samples.

Fortifications were made using diluted stock solutions of propargite or TBPC dissolved in acetone. Stock solutions containing 1040 $\mu\text{g}/\text{ml}$ of propargite and 1000 $\mu\text{g}/\text{ml}$ of TBPC in acetone were prepared for study 8845. Diluted solutions containing 100 $\mu\text{g}/\text{ml}$ were then made from the stock solutions and were used to spike the soil samples. The number of μg used to spike the soil samples is shown in the residue raw data sheets in Appendices 3 and 4.

C.6 Clean-up

Interferences in soil often require a special clean-up procedure after step #11, (see C.4-extraction method) in order to do the analysis of TBPC. This clean-up was not required for analysis of propargite and was not always required for TBPC. When required, the following clean-up procedure is used:

REAGENTS: Alumina, Activated (80-200 mesh, Alcoa Type F-20) MCB AXO612-3 kept at 130°C for a minimum of 12 hours then deactivated by adding 4% water and equilibrating in a glass vial with a teflon lined lid for a minimum of 4 hours and a maximum of 8 hours. All solvents are pesticide residue grade.

PROCEDURE:

1. Evaporate the dichloromethane (from Extraction procedure, Step #11, Section C.4) to dryness using a steambath.
2. Redissolve the extract with 2 mL of toluene (1 mL = 25.0 g).
3. Prepare a column by inserting a small glass wool plug into a 9-inch disposable Pasteur pipette. Add prepared alumina to a height of 2-inches.
4. Pipet 1 mL of toluene extract onto the column.
5. As soon as the extract has just passed into the alumina start washing the column with toluene until a total of 5 mL has been collected. (This is a combination of sample extract plus clean toluene.)
6. Move the column to a clean test tube having a minimum capacity of 10 mL.
7. Elute the TBPC from the column with a mixture of 10% acetone in hexane. Elute into the test tube until a total of 8 mL has been collected.
8. Add one drop of 1% decanol in acetone to the eluate, and evaporate just to dryness using a steambath.
9. Pipet 1 mL of dichloromethane into the tube to redissolve the extract (1 mL = 25.0 g)
10. Submit for GC analysis.

C.7 Derivatization

No derivatization is required for the methods described in this report.

D. INSTRUMENTATION

The instrumentation and operating conditions for the analysis of propargite and TBPC are shown below. As shown, it is possible to use different columns for TBPC.

PROPARGITE:

Instrument: MicroTek 220, FPD-S
 Column: 15 m x 0.53 mm, DB-1, 5.0 um (J&W)
 Temperatures: Column – 255°C
 Detector – 200°C
 Sampler – 240°C
 Carrier Gas and Flow: N₂ at 32 to 35 mL/min

TBPC:

Instrument: Varian 1400 equipped with FID
 Column: 25 m x 0.53 mm, Methyl Silicone, 5.0 um (Quadrex)
 Temperatures: Column – 210°C
 Detector – 200°C
 Sampler – 230°C
 Carrier Gas: N₂ at 25 psi
 Makeup Gas: N₂ at 15 psi

Instrument: Varian 1400 equipped with FID
 Column: 30 m x 0.53 mm, DB-17, 1.0 um (J&W)
 Temperatures: Column – 190°C
 Detector – 220°C
 Sampler – 230°C
 Carrier Gas: N₂ at 30 psi
 Makeup Gas: N₂ at 10 psi

Instrument: Varian 1400 equipped with FID
Column: 30 m x 0.53 mm, DB-1701, 1.0 μ m (J&W)
Temperatures: Column – 187°C
Detector – 200°C
Sampler – 230°C
Carrier Gas: N₂ at 25 psi
Makeup Gas: N₂ at 10 psi

E. POTENTIAL INTERFERENCES

It is possible that some soil samples may have interferences. While the columns and conditions shown in Section D have worked well historically alternate columns may have to be used for certain soil samples or the GC conditions may have to be changed somewhat to improve resolution. As mentioned in section C.6, a special clean-up of some soil samples may be necessary before analyzing for TBPC. A control soil should always be run along with the samples to determine if interfering substances are present in the soil.

F. CONFIRMATORY TECHNIQUES

No confirmatory techniques were performed in report 8845. Identification depended solely on retention time compared to standards.

G. TIME REQUIRED FOR ANALYSIS

Analyses in report 8845 consisted of one control soil, one method spike, and nine study samples. Preparation and extraction were usually done on one day and analyses on the following day. Thus, it is expected that a set of ten to twelve samples could be completed in two days.

H. MODIFICATIONS OR POTENTIAL PROBLEMS

There is some variance allowed in the soil sample size used for extraction. The original method (Sisken (1973) see Appendix 2) used a 400 g samples and 400 ml of acetone for extraction. The method described in this report (based on Uniroyal study 8845) generally used 200 g of soil and 200 ml of acetone. If smaller or larger acetone extracts are used, it is important to adjust the amount of chloroform used to separate the water so the acetone/chloroform ratio remains at 1:1. The original method also mentions that the rotovap used to remove the acetone/chloroform is run at 40°C under mild vacuum. The conditions used in Uniroyal study 8845 are not mentioned.

There are also some variations allowed in the column type, carrier gas, and flow rates. Additionally, the original method used an increasing oven temperature technique, whereas, the method in report 8845 was performed isothermally.

The methods mentioned in this report were developed ten or more years ago. It is expected that today's practitioner will use electronic integration methods rather than the peak height method described herein.

I. METHODS OF CALCULATION

Calculations are done separately for propargite and TBPC as shown below.

I.1 Calculations for Propargite

1. A four point standard curve, plotted on log-log graph paper is required for each chromatogram. Peak height x attenuation vs nanograms injected is used to calculate nanograms found.
2. All method spikes are corrected by subtracting the amount of analyte found in the control (check) from that found in the fortified control (spike).
3. Residues found in "treated" samples are not corrected for spike recovery.
4. In study 8845, three significant figures were used for calculating residues on the "as received" soil moisture basis.

EXAMPLE FROM STUDY 8845: Sample #74, Rep 2, 0-6" (M.L. #49341) with 14-day Post 3rd Application

Standard Curve:

| <u>ng injected</u> | <u>peak height</u> | <u>attenuation</u> | <u>peak height x attenuation</u> |
|--------------------|--------------------|--------------------|----------------------------------|
| 5 | 15 mm | 16x | 240 mm |
| 10 | 51 mm | 16x | 816 mm |
| 15 | 103 mm | 16x | 1648 mm |
| 20 | 172 mm | 16x | 2752 mm |

Sample:

1. Peak height x attenuation = corrected peak height

$$148 \text{ mm} \times 16 = 2368 \text{ mm}$$

2. From Standard curve find nanograms (FOUND)

$$2368 \text{ mm} = 18.2 \text{ ng}$$

3.
$$\frac{\text{Nanograms Found}}{\text{Amount of Sample Injected (mg)}} = \text{ppm} \quad \frac{18.2 \text{ ng}}{50.0 \text{ mg}} = 0.364 \text{ ppm}$$

1.2 Calculations for TBPC

1. Averaged standard sensitivity (mm/ng) is used to calculate nanograms found.
2. A standard curve (peak height in mm vs nanograms injected) is generated on a regular basis to show linearity for the compound. This standard curve is not used for calculating residues.
3. All method spikes are corrected by subtracting the amount of analyte found in the control (check) from that found in the fortified control (spike).
4. Residues found in treated samples are not corrected for spike recovery.
5. In study 8845, three significant figures were used for calculating residues on the "as received" soil moisture basis.

EXAMPLE FROM STUDY 8845: Sample #74, Rep 2, 0-6" (M.L. #49341) with 14-day Post 3rd Application.

Average Standard Sensitivity

| <u>ng injected</u> | <u>peak height</u> | <u>sensitivity</u> |
|--------------------|--------------------|--------------------|
| 50 | 97.5 mm | 1.95 mm/ng |
| 50 | 98.5 mm | 1.97 mm/ng |
| 50 | 95.5 mm | 1.91 mm/ng |
| 50 | 91 mm | <u>1.82 mm/ng</u> |
| | | 7.65 mm/ng |

$$\text{AVERAGE SENSITIVITY} = \frac{7.65 \text{ mm/ng}}{4} = 1.91 \text{ mm/ng}$$

Sample:

1. Peak height divided by average standard sensitivity = nanograms found

$$\frac{11.5 \text{ mm}}{1.91 \text{ mm/ng}} = 6.02 \text{ ng}$$

2. $\frac{\text{Nanograms Found}}{\text{Amount of Sample Injected (mg)}} = \text{ppm}$ $\frac{6.02 \text{ ng}}{50.0 \text{ ng}} = 0.120 \text{ ppm}$

As mentioned above for propargite, a four point curve plotted on log-log graph paper was used to calculate the nanograms found. For TBPC a similar standard curve is used to show linearity for TBPC but is not used in calculating the residues. A typical standard curve is shown in Appendix 3.

I.3 Calculation of Soil Moistures

Weight of wet soil plus dish – weight of dish = weight of wet soil

Weight of wet soil plus dish – weight of dry soil + dish = moisture lost

$$\% \text{ Moisture} = \frac{\text{moisture lost (g)}}{\text{weight of wet soil (g)}} \times 100$$

I.4 Calculation of Soil Residues From an "As Received" Basis to an Air Dry Basis

Residues (in ppm) on air dry basis =

$$\frac{\text{Residue (in ppm) on an "as received" basis}}{\text{g air dry soil/100 g "as received" soil}}$$

where: g air dry soil =

$$(100\% - \% \text{ moisture to air dry basis}) \times 100 \text{ g}$$

I.5 Calculation of Recoveries

As an example the 0.1 ppm spike from the propargite stability study residue raw data sheet is used. (See Appendix 3).

The weight of soil used was 200 g. The propargite and TBPC from this soil were extracted into 400 ml of solvent (original extract, plus washes and dilutions)—see Section C.4. One-half this volume of extract or 200 ml was used for the analysis. This is equivalent to using:

$$\frac{200 \text{ ml}}{400 \text{ ml}} \times 200 \text{ g soil} = 100 \text{ g of soil}$$

After further work-up, the analytes are contained in 4 ml of solvent. One-half of this 4 ml is used to analyze for propargite and one-half is used to analyze for TBPC. This is equivalent to using:

$$\frac{2 \text{ ml}}{4 \text{ ml}} \times 100 \text{ g of soil} = 50 \text{ g of soil}$$

In the method, 4 μ l is injected (note: this amount may vary, but is the actual amount as shown on the raw data sheet). This is equivalent to using:

$$\begin{aligned} & \frac{4 \mu\text{l}}{2 \text{ ml}} \times 50 \text{ g of soil} \\ = & \frac{4 \mu\text{l}}{2000 \mu\text{l}} \times 50,000 \text{ mg of soil} \\ = & 100 \text{ mg of soil} \end{aligned}$$

This is the number in column 17 of the raw data work sheet labeled (rather ambiguously) "mg inject".

To calculate the ppm found, the following calculation is used:

$$\begin{aligned} & 8.5 \text{ ng were found/100 mg of soil (see above)} \\ \text{Therefore, } & \frac{8.5 \text{ ng}}{100 \text{ mg soil}} = \frac{8.5 \times 10^{-9} \text{ g}}{100 \times 10^{-3} \text{ g soil}} \\ = & \frac{8.5 \times 10^{-9} \text{ g}}{0.1 \text{ g soil}} = \frac{8.5 \times 10^{-9} \times 10^7 \text{ g}}{10^6 \text{ g soil}} \\ = & 0.085 \text{ ppm} \end{aligned}$$

The recovery is:

$$\begin{aligned} & \frac{\text{ppm found}}{\text{ppm added}} \times 100\% \\ = & \frac{0.085 \text{ ppm}}{0.100 \text{ ppm}} \times 100\% \\ = & 85\% \end{aligned}$$

J. COPIES OF CHROMATOGRAMS

Copies of chromatographs for soil spiked with propargite at the 0.0 (control soil), 0.1 ppm and 1.0 ppm levels are shown in Appendix 3.

Copies of chromatographs for soil spiked with TBPC at the 0.0 (control soil), 0.1 ppm and 1.0 ppm levels are shown in Appendix 4

K. METHOD VALIDATIONS

K.1 Accuracy (USA)/Recovery (EU)

Although no formal study to determine accuracy has been done, this report addresses this issue by extracting data from Study 8845 method spikes. Table I summarizes this data for spikes at the 0.1 ppm and 1.0 ppm levels for two portions of Study 8845; the field portion and the freezer stability portion. These two portions were chosen to give a larger sample of observations over a period of time. This choice should improve the statistics.

Table 1 summarizes data on the method spikes used in Study 8845. The number of observations, mean recoveries, standard deviation (SD), relative standard deviation (RSD), the range of recoveries, and the \pm confidence limits for 95% confidence are shown for the 0.1 ppm and 1.0 ppm levels. The data indicates that average recoveries of both propargite and TBPC at both levels are between 70-110% as required by the EU (70-120% as required by the USA).

In Table 1, the RSD was calculated as:

$$\text{RSD} = \frac{\text{SD}}{\text{Average}} \times 100\%$$

The 95% confidence limits (CL) were calculated as:

$$\text{CL} = \frac{t \times \text{SD}}{\sqrt{n}}$$

Where SD = standard deviation

n = the number of observations

t = the value t for n-1 degree of freedom at 95% confidence as taken from table C.3, page 267 of Quality Assurance of Chemical Measurements, John K. Taylor, Lewis Publishers, Inc., 1987

K.2 Precision

The USA requires a calculation of the relative standard deviation (RSDs) at the various concentration levels. These RSDs are shown in Table 1 and are less than or equal to 20%, as required by the EPA, at both the 0.1 ppm and 10 ppm spiking levels.

The EU requires a repeatability study where the same sample is used at least five times on the same instrument with the same operator within a short time interval. Although Table 1 indicates that a number of method spikes at the 0.1 ppm and 1.0 ppm levels were done over a relatively short period of time (April 7, 1989 to May 2, 1989), it was impossible to confirm from the raw data that the analyses were done by the same operator or that the same stock solution was used for the spikes. The latter does seem likely, however, since report 8845 only shows one stock solution and one dilution having been made up between 1/12/89 and 5/2/89 for both propargite and TBPC (see Section C.5 - fortifications). The recoveries shown in Table 1 do suggest that if a formal repeatability study were done, the data would show the method to be repeatable.

K.3 Limit of Quantitation (USA)/Limit of Determination (EU)

The lowest spike level used in study 8845 was 0.1 ppm. As indicated in Table 1, at this level the mean recoveries for propargite and TBPC were both between 70 and 110%, and both relative standard deviations were less than or equal to 20%. Thus, the limit of quantitation is 0.1 ppm.

K.4 Limit of Detection

No statistical estimate of the limit of detection (LOD) was made from the data in Table 1. However, if we assume that the LOD is roughly one-third of the LOQ, the LOD would be about 0.03 ppm. In this connection, for propargite, the chromatographic traces showing typical results from the 0.0 ppm (check), 0.1 ppm (spike), and 1.0 ppm (spike) samples in Appendix 3 can be considered. These traces suggest that an LOD of 0.03 should easily be possible. For TBPC, the corresponding chromatographic traces are shown in Appendix 4. These suggest that an LOD at the 0.03 ppm would be pushing the method.

K.5 Specificity

Although the analysis of propargite and TBPC are both done using gas chromatography, it is necessary to use two different detectors to get the best results. The propargite is detected with a flame photometric detector in sulfur mode. This is a fairly specific detector for sulfur compounds so non-sulfur containing compounds will not be seen. The TBPC is detected using a more conventional flame ionization detector. This will detect other compounds that can burn. The soil samples analyzed in Study 8845 had no interfering substances as evidenced by clean checks. It is recommended that non-treated soils always be analyzed to check for interferences.

K.6 Ruggedness

No ruggedness testing was done.

K.7 Limitations

None are known.

K.8 Independent Laboratory Validation (ILV) (USA)/ Reproducibility (EU)

Reproducibility (EU) is defined as an independent lab validation. Reproducibility is not required for soil analytical methods according to EU directive 91/414/EEC, July 16, 1996. An ILV is suggested by the USA EPA. This has not been done in a formal sense. However, four field dissipation studies for propargite in different USA locations (two for California, one each for Florida and Georgia) have been done at various times. Although the same laboratory analyzed the samples, the fact that these analyses were done successfully over a number of years suggests that the method in this report can be considered as having been independently lab validated.

L. CONCLUSIONS

The analytical method AC 6006 described in this report is applicable to the analysis of propargite and its degradate (TBPC) in a variety of soil types. The LOD is about 0.03 ppm and the LOQ is 0.10 ppm. Recoveries and relative standard deviations are excellent and well within the regulatory guidelines of both the EPA and EU.

TABLES

TABLE 1.

Validation Parameters for the Analysis of Propargite and TBPC in Soil

Validation Parameters for the Analysis of Propargite and TBPC in Soil

| DATE | PROPARGITE | | TBPC | |
|--|------------|----------|------------|----------|
| | % RECOVERY | | % RECOVERY | |
| | 0.1ppm | 1.0ppm | 0.1ppm | 1.0ppm |
| 03/15/89 | 81 | 88 | | |
| 03/21/89 | | | 71 | 83 |
| 03/29/89 | | 81 | | |
| 04/03/89 | 90 | | | |
| 04/07/89 | | 86 | | |
| 04/20/89 | 85 | 84 | | |
| 04/21/89 | 75 | 82 | | |
| 04/21/89 | 98 | | | |
| 04/25/89 | | | 120 | |
| 04/27/89 | | | | 81 |
| 04/28/89 | 90 | 82 | 72 | 72 |
| 05/02/89 | | | 101 | |
| 05/23/89 | 86 | 90 | | |
| 05/31/89 | | | | 89 |
| 06/30/89 | 80 | 72 | | |
| 08/01/89 | | | 99 | 74 |
| 08/02/89 | 74 | 78 | | |
| 08/02/89 | | | 106 | |
| 08/03/89 | 94 | | | |
| 09/28/89 | 80 | 80 | | |
| 10/13/89 | | | | 86 |
| 10/19/89 | | | 113 | |
| 10/19/89 | | | 110 | |
| 01/19/90 | 95 | 96 | | |
| 01/24/90 | | | 111 | 86 |
| | | | | |
| | | | | |
| | | | | |
| Count | 12.0 | 11.0 | 9.0 | 7.0 |
| Average % | 85.7 | 83.5 | 100.3 | 81.6 |
| SD | 7.9 | 6.4 | 17.5 | 6.4 |
| RSD | 9.2 | 7.7 | 17.4 | 7.8 |
| Range | 74 to 98 | 72 to 96 | 71 to 120 | 72 to 89 |
| 95 % Confidence | 5.0 | 4.3 | 13.5 | 5.9 |
| Numbers in bold are taken from the field portion of study 8845 | | | | |
| Other numbers are taken from the freezer stability portion of study 8845 | | | | |

Table 1