

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Flufenpyr-ethyl

MRID # : 451191-20

Matrix: Soil

Analysis: GC/MSD

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VALENT U.S.A. CORPORATION
VALENT TECHNICAL CENTER
Dublin, California

DETERMINATION OF RESIDUES OF S-3153,
S-3153 ACID, S-3153-1-OH, AND S-3153-1-OCH₃,
IN SOIL

Method: RM-36-2

Date: October 26, 1999

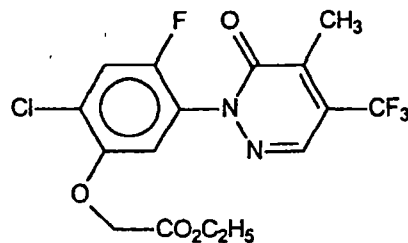
I. INTRODUCTION

This method describes the determination of S-3153 {ethyl [2-chloro-4-fluoro-5-(5-methyl-6-oxo-4-trifluoromethyl-1,6-dihydropyridazin-1-yl)phenoxy]acetate} and its degradates, S-3153 Acid {[2-chloro-4-fluoro-5-(5-methyl-6-oxo-4-trifluoromethyl-1,6-dihydropyridazin-1-yl)phenoxy]acetic acid}, S-3153-1-OH {2-(4-chloro-2-fluoro-5-hydroxy)phenyl-4-methyl-5-trifluoromethyl-2,3-dihydropyridazin-3-one}, and S-3153-1-OCH₃ {2-(4-chloro-2-fluoro-5-methoxy)phenyl-4-methyl-5-trifluoro-methyl-2,3-dihydropyridazin-3-one}, in soil.

The method involves extracting the residues into 4/1 acetone/0.01 N HCl (v/v); rotary evaporating the acetone to obtain an aqueous residue; adding Hastings-Sendroy buffer to set the pH to 6; partitioning the S-3153, S-3153-1-OH, and S-3153-1-OCH₃ residues into 9/1 hexane/acetone (v/v); resetting the pH to 2 with HCl; and then partitioning the S-3153 Acid residues into methylene chloride. The extract containing the S-3153, S-3153-1-OH, and S-3153-1-OCH₃ residues is then rotary evaporated, the residues are dissolved in 1.0 mL of ethyl acetate, and the final extract is analyzed by GC/MSD. For the S-3153 Acid residues, the methylene chloride is removed by rotary evaporation, the residues are dissolved in ethanol, the ethanol mixture is acidified with concentrated HCl, and the acidic mixture is refluxed for 30 minutes. After cooling the reaction mixture, water is added and the S-3153 (formed by the ethylation of the S-3153 Acid) is extracted with methylene chloride. The methylene chloride extract is washed with buffer to remove traces of acid, the methylene chloride is then removed by rotary evaporation, and the residues are dissolved in 1.0 mL of ethyl acetate. Analysis for S-3153 Acid (as S-3153) is performed by GC/MSD.

II. ANALYTICAL STANDARDS

S-3153 (or V-3153) reference standard
- Valent U.S.A. Corporation

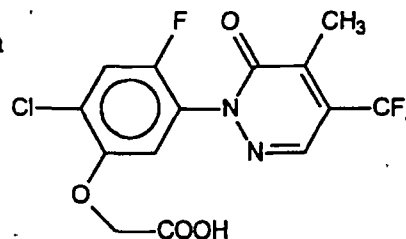


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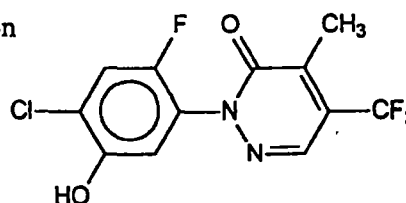
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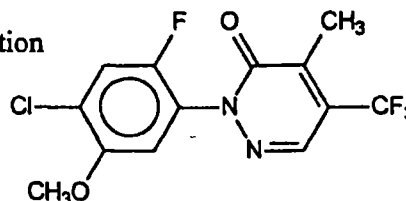
S-3153 Acid reference standard - Valent U.S.A. Corporation



S-3153-1-OH reference standard - Valent U.S.A. Corporation



S-3153-1-OCH₃ reference standard - Valent U.S.A. Corporation



S-3153 standard, 0.25 mg/mL Stock solution.

Weigh 0.025 grams (to ensure a 0.25 mg/mL concentration, correct the amount of standard weighed for the purity of the standard) into 100 mL volumetric flask. Dilute to volume with acetone, and store refrigerated.

S-3153 Acid standard, 0.25 mg/mL Stock solution.

Weigh 0.025 grams (to ensure a 0.25 mg/mL concentration, correct the amount of standard weighed for the purity of the standard) into 100 mL volumetric flask. Dilute to volume with acetone, and store refrigerated.

S-3153-1-OH standard, 0.25 mg/mL Stock solution.

Weigh 0.025 grams (to ensure a 0.25 mg/mL concentration, correct the amount of standard weighed for the purity of the standard) into 100 mL volumetric flask. Dilute to volume with acetone, and store refrigerated.

S-3153-1-OCH₃ standard, 0.25 mg/mL Stock solution.

Weigh 0.025 grams (to ensure a 0.25 mg/mL concentration, correct the amount of standard weighed for the purity of the standard) into 100 mL volumetric flask. Dilute to volume with acetone, and store refrigerated.

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Mixed Standard (S-3153, S-3153-1-OH, & S-3153-1-OCH₃), 50 µg/mL solution (in acetone).
Pipet 10.0 mL of the each of the three Stock solutions into a 50 mL volumetric flask and dilute to volume with acetone. Store refrigerated.

Mixed Standard (S-3153, S-3153-1-OH, & S-3153-1-OCH₃), 2.0 µg/mL solution (in acetone).
Pipet 4.0 mL of the Mixed Standard 50 µg/mL solution (in acetone) into a 100 mL volumetric flask and dilute to volume with acetone. Store refrigerated.

Mixed Standard (S-3153, S-3153-1-OH, & S-3153-1-OCH₃), 0.5 µg/mL solution (in acetone).
Pipet 25.0 mL of the Mixed Standard 2.0 µg/mL solution (in acetone) into a 100 mL volumetric flask and dilute to volume with acetone. Store refrigerated.

S-3153 Acid Standard, 50 µg/mL solution (in acetone).
Pipet 10.0 mL of the Stock solution into a 50 mL volumetric flask and dilute to volume with acetone. Store refrigerated.

S-3153 Acid Standard, 2.0 µg/mL solution (in acetone).
Pipet 4.0 mL of the S-3153 Acid Standard 50 µg/mL solution (in acetone) into a 100 mL volumetric flask and dilute to volume with acetone. Store refrigerated.

S-3153 Acid Standard, 0.5 µg/mL solution (in acetone).
Pipet 25.0 mL of the S-3153 Acid Standard 2.0 µg/mL solution (in acetone) into a 100 mL volumetric flask and dilute to volume with acetone. Store refrigerated.

Mixed Standard (S-3153, S-3153-1-OH, & S-3153-1-OCH₃), 1.0 µg/mL in ethyl acetate.
Pipet 2.0 mL of the Mixed Standard 50 µg/mL solution (in acetone) into a 100 mL volumetric flask and dilute to volume with ethyl acetate. Store refrigerated.

Mixed Standard (S-3153, S-3153-1-OH, & S-3153-1-OCH₃), 0.5 µg/mL in ethyl acetate.
Pipet 1.0 mL of the Mixed Standard 50 µg/mL solution (in acetone) into a 100 mL volumetric flask and dilute to volume with ethyl acetate. Store refrigerated.

Mixed Standard (S-3153, S-3153-1-OH, & S-3153-1-OCH₃), 0.1 µg/mL in ethyl acetate.
Pipet 5.0 mL of the Mixed Standard 2.0 µg/mL solution (in acetone) into a 100 mL volumetric flask and dilute to volume with ethyl acetate. Store refrigerated.

Mixed Standard (S-3153, S-3153-1-OH, & S-3153-1-OCH₃), 0.04 µg/mL in ethyl acetate.
Pipet 2.0 mL of the Mixed Standard 2.0 µg/mL solution (in acetone) into a 100 mL volumetric flask and dilute to volume with ethyl acetate. Store refrigerated.

Note: Similar serial dilutions may also be performed to generate the appropriate standards.

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III. REAGENTS

Acetone - Pesticide quality

Disodium Hydrogen Phosphate·7H₂O [Na₂HPO₄·7H₂O] - Reagent grade

Ethyl Acetate - Pesticide quality

Hexane - Pesticide quality

Hydrochloric Acid, Concentrated [12 N]- Reagent Grade

Methylene Chloride - Pesticide quality

Potassium Phosphate, Monobasic [KH₂PO₄] - Reagent grade

Sodium Sulfate - AR grade, granular (acetone-washed and air dried)

Water - Deionized

IV. REAGENT SOLUTIONS

Acetone/0.01 N HCl, 4/1 (v/v).

Combine 4 parts acetone with 1 part 0.01 N HCl. For example, add 800 mL of acetone and 200 mL of 0.01 N HCl sequentially to a reagent bottle. Store at room temperature.

Hastings-Sendroy Buffer.

Combine 611 mL of 1/15 M disodium hydrogen phosphate with 389 mL of 1/15 M potassium phosphate in an erlenmeyer flask (or reagent bottle). Mix well. [The amounts used may be scaled, as appropriate.] Store at room temperature.

Hexane/Acetone, 9/1 (v/v).

Combine 9 parts hexane with 1 part acetone. For example, add 900 mL of hexane and 100 mL of acetone sequentially to a reagent bottle. Store at room temperature.

Hydrochloric acid, 1 N

Dilute 80 mL of 12 N hydrochloric acid to 960 mL with deionized water. For example - add approximately 500 mL deionized water into a large beaker or flask, add the 12 N acid, transfer the dilute solution into a 1000 mL graduated cylinder, and dilute to volume with deionized water. [Similar ratios of acid to water may also be used.] Store at room temperature. *Use caution when handling 12 N HCl as it is corrosive - avoid breathing fumes, and avoid contact with skin or clothing.*

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Hydrochloric acid, 0.01 *N*

Add 10 mL of 1 *N* HCl to 990 mL of deionized water. Or, add 0.8 mL of 12 *N* hydrochloric acid to 960 mL of deionized water. [Similar ratios of acid to water may also be used.] Store at room temperature.

Potassium Phosphate, 1/15 *M*

Prepare a 1/15 *M* aqueous solution by adding 9.07 grams KH_2PO_4 into 1 L of deionized water in an erlenmeyer flask (or reagent bottle). Cover (or stopper) and stir (or shake) the mixture to dissolve the KH_2PO_4 . [The amounts used may be scaled, as appropriate.] Store at room temperature.

Disodium Hydrogen Phosphate, 1/15 *M*

Prepare a 1/15 *M* aqueous solution by adding 17.9 grams $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$ into 1 L of deionized water in an erlenmeyer flask (or reagent bottle). Cover (or stopper) and stir (or shake) the mixture to dissolve the $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$. [The amounts used may be scaled, as appropriate.] Store at room temperature.

V. EQUIPMENT

Balances, Analytical and Top Loading

Büchner Funnels - 9 cm

Filter Flasks, Vacuum - 500 mL

Filter Funnels (approximately 100 mm diameter)

Filters, Glass Fiber - Whatman GF/A, 9 cm

Graduated Cylinders (1000, 250, 100, 50, 25 mL)

Glass Wool, Pyrex

Heated Water Bath (temperature $<40^\circ\text{C}$)

Heating Mantles

Linear Shaker - Erbach (or equivalent)

Mason Jars - pint (or equivalent)

Pasteur Pipettes, Disposable - 5: and 9 inch

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Pipettors, Automatic - capable of accurately dispensing volumes of 0.1 to 2.0 mL

Pipettes, Volumetric - 25.0, 10.0, and 4.0 mL

Round-bottom Flasks (500, 250, 100, and 50 mL)

Reflux condensers

Refrigerator

Rotary Vacuum Evaporators

Separatory Funnels (250 and 125 mL)

VI. INSTRUMENTATION

GAS CHROMATOGRAPH (GC)

Hewlett-Packard Model 5890 GC equipped with a Hewlett-Packard Model 5971 mass selective detector (GC/MSD), autosampler, and integrator (or equivalent). The conditions shown are suggested for this analysis, and these may be changed as appropriate (*see Note 1 for additional parameters*).

Column: DB - 1 (J & W Scientific, Inc.), 30 m x 0.25 mm I.D. , 0.25 μ m film thickness (or equivalent).

Carrier gas : Helium, 0.8 mL/min.

Injector: 1.0 μ L, Splitless, 0.8 min. Purge valve delay

Temperatures: Injector - 240°C
Detector - 280°C
Column Oven - 75°C Initial (Hold 1.0 min.)
30°C/min Ramp to 225°C (Hold 1.5 min.)
25°C/min Ramp to 280°C (Hold 1.0 min.)
35°C/min Ramp to 310°C (Hold 4.0 min.)

Retention Times (approximate): 7.9 minutes (S-3153-1-OH)
8.1 minutes (S-3153-1-OCH₃)
9.7 minutes (S-3153)

Note: A chromatogram of the Mixed Standard is shown in Figure 1.

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VII. ANALYTICAL PROCEDURES

1. Sample Setup

Weight 20 g of the soil into a pint Mason® jar. At this point, if required by the testing facility, a control sample to be used for method recoveries may be fortified with S-3153, S-3153 Acid, S-3153-1-OH, and S-3153-1-OCH₃ (see Note 2).

2. Extraction with Acetone/0.01 N HCl

Add 100 mL of 4/1 acetone/0.01 N HCl into the pint jar. Place the cover on the jar and then shake on the linear shaker for 30 minutes. Filter the mixture through a Whatman® GF/A glass filter in a Büchner funnel (under suction) into a 500 mL filter flask. Transfer the moist solids (and the glass fiber filter) back into the jar, and re-extract the solids by adding a second 100 mL portion of 100 mL of 4/1 acetone/0.01 N HCl and shaking for 20 minutes. Filter as before, rinse the jar with 50 mL of 4/1 acetone/0.01 N HCl, and pass the rinse through the solids on the glass fiber filter - combining the rinse with the extracts.

3. Partition with 9/1 Hexane/Acetone

Transfer the filtrate into a 500 mL round-bottom flask, and rotary evaporate the mixture to remove the acetone (temperature <40°C).

Transfer the aqueous residue (about 45 mL) into a 250 mL separatory funnel, and add 2.0 mL of Hastings-Sendroy buffer to the aqueous residue. Check that the pH is approximately 6. Rinse the round-bottom flask with 100 mL of 9/1 hexane/acetone, and transfer this rinse into the separatory funnel. Partition the S-3153, S-3153-1-OH, and S-3153-1-OCH₃ residues into the hexane/acetone by shaking vigorously for 1 minute. Allow the phases to separate, and then drain the aqueous layer back into the round-bottom flask. Drain the hexane/acetone layer through approximately 80 g of sodium sulfate (suspended on a glass wool plug in a glass filter; and freshly washed with 9/1 hexane/acetone) into a 500 mL round-bottom flask.

Transfer the aqueous residue from the round-bottom flask back into the separatory funnel, rinse this round-bottom flask with 100 mL of 9/1 hexane/acetone, and then transfer the rinse into the separatory funnel. Partition the sample by shaking for 1 minute. As before, allow the phases to separate, drain the aqueous layer back into the initial 500 mL round-bottom flask, and then drain the hexane/acetone layer through the sodium sulfate - combining this extract with the first. Rinse the sodium sulfate with 10-20 mL of 9/1 hexane/acetone, and collect the rinsate with the combined extracts. *Reserve this combined hexane/acetone extract for concentration - Step 6.*

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4. Partition with Methylene Chloride (S-3153 Acid)

Add 2.0 mL of 1 N HCl to the aqueous residues. Check that the pH is approximately 2. Transfer the solution back into the 250 mL separatory funnel, rinse the round-bottom flask with 50 mL of methylene chloride, and add the methylene chloride rinse to the separatory funnel. Shake the sample vigorously for 1 minute, with venting. After the phases separate, drain the methylene chloride layer (the lower layer) into a clean 500 mL round-bottom flask.

Rinse the round-bottom that had held the aqueous residues with a second 50 mL portion of methylene chloride, transfer this rinse into the separatory funnel, and shake again for 1 minute. Allow the phases to separate, and drain the methylene chloride layer into the 500 mL round-bottom flask - combining the methylene chloride extracts. Repeat the extraction with a third 50 mL portion of methylene chloride - combining the extracts as before.

5. Ethylation (S-3153 Acid)

Rotary evaporate (temperature $<40^{\circ}\text{C}$) the methylene chloride to reduce the volume to 5-10 mL. Transfer the residues to a 50 mL round-bottom flask, using two 10 mL methylene chloride rinses of the 500 mL round-bottom flask. Continue rotary evaporation to remove the methylene chloride (temperature $<40^{\circ}\text{C}$), just to dryness.

Add 10 mL of ethanol to the flask, and sonicate briefly to dissolve the residues. Add 0.5 mL of concentrated HCl to the sample, add a Teflon boiling chip, attach the round-bottom flask to a reflux condenser, and place the flask into a heating mantle. Reflux the mixture for 30 minutes, and then allow the sample to cool (20-30 minutes).

Add 25-30 mL of deionized water to the sample, transfer the residues into a 125 mL (or 250 mL) separatory funnel, rinse the 50 mL round-bottom flask with 25 mL of methylene chloride, and add the rinsate to the separatory funnel. Shake the sample vigorously for 1 minute (with venting) to partition the S-3153 (ethylated S-3153 Acid) into the methylene chloride layer. Allow the phases to separate, and then drain the methylene chloride layer into a clean 100 mL round-bottom flask (or similar container). Rinse the 50 mL round-bottom flask with a second 25 mL portion of methylene chloride, transfer the rinse into the separatory funnel, and repeat the extraction. Drain the methylene chloride layer into the flask containing the initial methylene chloride extract. Discard the aqueous layer, and transfer the methylene chloride extract back into the separatory funnel (rinsing the flask with 5 mL of methylene chloride and adding the rinse to the separatory funnel). Add 5 mL of Hastings-Sendroy buffer to the separatory funnel, and shake the sample vigorously for 30 seconds (with venting). Allow the phases to separate, drain the methylene chloride layer through approximately 20-25 g of sodium sulfate (suspended on a glass wool plug in a glass filter; and freshly washed with methylene chloride) into a 250

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mL round-bottom flask, and then rinse the sodium sulfate with 10-15 mL of methylene chloride - combining the methylene chloride extracts and rinse.

Rotary evaporate the methylene chloride (using a heated water bath, temperature $<40^{\circ}\text{C}$) to reduce the volume to 10-20 mL. Transfer the residues to a 100 mL round-bottom flask, using two sequential 10 mL ethyl acetate rinses. Continue rotary evaporation, just to dryness. Add 1.0 mL of ethyl acetate, stopper and sonicate briefly to dissolve the residues, and then transfer the final extract into an autosampler vial (or vials) or a screw top vial for storage under refrigeration. Samples in autosampler vials may also be analyzed directly.

6. Concentration for S-3153, S-3153-1-OH, and S-3153-1-OCH₃

For the reserved extract from Step 2, rotary evaporate the solvent (temperature $<40^{\circ}\text{C}$) to reduce the volume to 10-20 mL. Transfer the residues to a 100 mL round-bottom flask, using two sequential 10 mL ethyl acetate rinses. Continue rotary evaporation, just to dryness. Add 1.0 mL of ethyl acetate to the flask, and then stopper and sonicate briefly to dissolve the residues. Transfer this extract into an autosampler vial (or vials) or a screw top vial for storage under refrigeration. Samples in autosampler vials may also be analyzed directly.

7. GC Measurement

Condition the instrument with at least three injections of a sample extract (*see Note 3*). Analyze a range of at least four standard concentrations using Mixed Standards (in ethyl acetate) *within the analytical sequence* to establish the linear response of the GC/MSD, including a 0.04 $\mu\text{g/mL}$ standard. [A typical standard sequence would be 0.04, 0.1, 0.5, and 1.0 $\mu\text{g/mL}$, with an injection volume of 1.0 μL .] To verify the linear response, calculate the response factor for each of the four standards [by dividing the peak height (or area) of the standard by its concentration], determine the standard deviation of the four response factors, and then divide this value by the average response factor. The result (the coefficient of variation) must be 15% or less for the instrument response to be considered linear for that range of standards.

It is recommended that the linearity standards be interspersed within the analytical run, as this allows the injection port and column to be conditioned with sample matrix from the preceding injection. A sample sequence is then constructed with the following order: a reference standard (0.5 $\mu\text{g/mL}$), a set of 2 to 4 sample extracts, a reference standard or linearity standard, another set of 2 to 4 sample extracts, ..., and a reference standard. *The sequence must begin and end with reference standards.* The coefficient of variation of the reference standard responses must be 10% or less for the analysis set to be acceptable.

If the peak response for any of the analytes in a sample is greater than the peak response of the highest linearity standard, the sample extract must be diluted and the diluted extract analyzed.

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The sample extract must be diluted (with acetone) such that the peaks obtained are within the documented linear response range of the GC/MSD.

7. Calculations

The amount of S-3153, S-3153-1-OH, and S-3153-1-OCH₃ in each sample is calculated as follows:

$$\text{Sample concentration, ppm } (\mu\text{g/g}) = \frac{A \times B \times C \times D}{E \times F}$$

where:

- A = Sample peak height (or area)
- B = Continuing calibration standard concentration (0.5 $\mu\text{g/mL}$)
- C = Final extract volume (1.0 mL)
- D = Dilution factor, if any
- E = Average calibration standard peak height (or area)
- F = Initial sample weight (20 g)

The amount of S-3153 Acid (after conversion to S-3153) is calculated as follows:

$$\text{Sample concentration, ppm } (\mu\text{g/g}) = \frac{A \times B \times C \times D \times E}{F \times G}$$

where:

- A = Sample peak height (or area)
- B = Continuing calibration standard concentration (0.5 $\mu\text{g/mL}$)
- C = Final extract volume (1.0 mL)
- D = Dilution factor, if any
- E = Conversion Factor ($MW_{\text{S-3153 Acid}}/MW_{\text{S-3153}} = 0.93$)
- F = Average calibration standard peak height (or area)
- G = Initial sample weight (20 g)

VIII. LIMIT OF DETECTION

The limit of detection (LOD) of this method is 0.002 ppm ($\mu\text{g/g}$) for each analyte; and the validated limit of quantitation (LOQ) is 0.005 ppm ($\mu\text{g/g}$).

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IX. DISCUSSION

The method was verified by fortifying soil samples with S-3153, S-3153 Acid, S-3153-1-OH, and S-3153-1-OCH₃. For the S-3153, S-3153-1-OH, and S-3153-1-OCH₃ analyses, the initial set gave recoveries of 90-131%. Although the S-3153 Acid recoveries in this set averaged 76%, two of the samples showed <70% recovery. Subsequent analyses, after a slight modification to the cleanup following esterification, gave acceptable (≥70%) recoveries. The recovery data are presented in Attachment 1. Example chromatograms obtained from control and fortified soil sample extracts are attached (Figures 2-5).

X. NOTES

1. The mass selective detector parameters shown below are suggested for this analysis - similar conditions may be used as appropriate:

Acquisition Mode:	Selective Ion Monitoring (SIM)	
Solvent Delay:	7 minutes	
MSD Off:	11 minutes	
Quantitation Ions:	S-3153	345 + 335
	S-3153-1-OH	322 (parent ion) + 303
	S-3153-1-OCH ₃	336 (parent ion) + 317

2. Valent Standard Operating Procedure (SOP) VR-002 specifies that fortified control samples are to be analyzed with each set of samples. If the testing facility does not require concurrent analysis of fortified control samples, or if an untreated control (UTC) sample is not available, this method requirement may be waived.

The level of fortification is generally 0.005 ppm (the limit of quantitation of the method) and/or 0.025 ppm. Add 0.20 mL of the 0.5 µg/mL Mixed Standard solution (in acetone) and 0.20 mL of the 0.5 µg/mL S-3153 Acid Standard solution (in acetone) to obtain a 0.005 ppm fortification level, and/or add 0.25 mL of the 2.0 µg/mL Mixed Standard solution (in acetone) and 0.25 mL of the 2.0 µg/mL S-3153 Acid Standard solution (in acetone) to obtain a 0.025 ppm fortification level in a 20 g sample. Method recoveries must be 70 to 120% to be acceptable (except for S-3153-1-OH, with acceptable recoveries from 80 to 130%), unless approved by the chemist responsible for the analysis.

3. The analysis of extracts and standards by GC/MSD is noticeably influenced by the condition of the quartz liner, the fused-silica wool in the liner, and the gold-plated seal at the base of the injection port. Care should be taken to ensure that the liner is

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deactivated (e.g.- by treatment with Sylon CT from Supelco) and that a minimum amount of fused-silica wool is inserted about half to two-thirds of the distance from the top of the liner. Maintenance relating to the injection port is very important for the analysis of both S-3153-1-OH and S-3153. If system maintenance is neglected, apparent enhancement of analytes in samples (relative to standards) will be observed in the order S-3153-1-OH > S-3153 > S-3153-1-OCH₃. *Conditioning of the injection port liner prior to the analysis of a sample sequence is essential to obtain reproducible data.*

XI. SIGNATURES

Written by

Charles Green

Charles Green

Date 11/2/99

Reviewed by

Glenn Fujie
Glenn Fujie, Laboratory Manager

Date

11/4/99

Reviewed by

Linda Macey
QAU

Date

10/25/99

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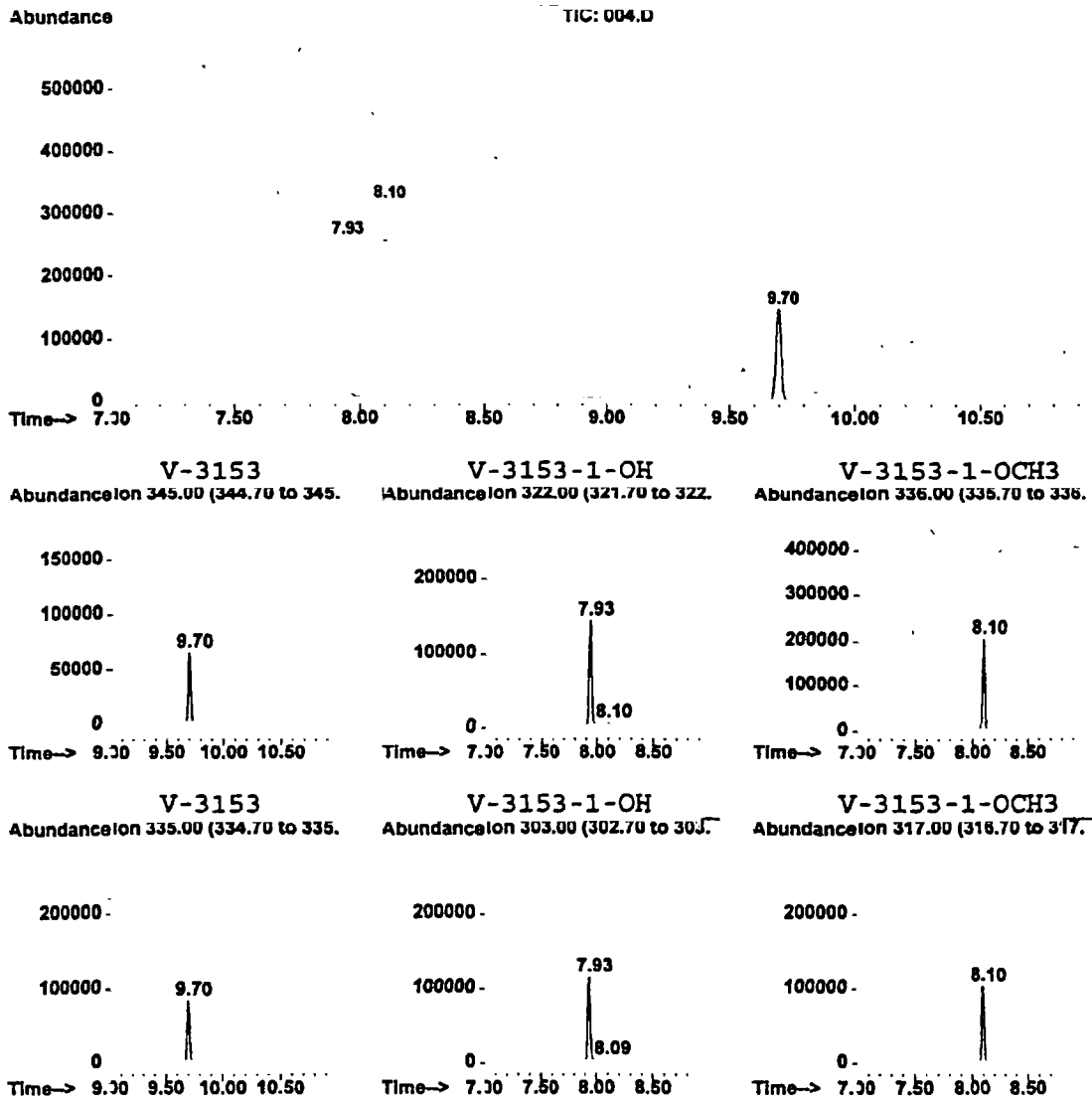


Figure 1. Mixed Standard for Calibration (0.5 µg/mL S-3153, S-3153-1-OH, & S-3153-1-OCH₃ in Ethyl Acetate; 1.0 µL injection)

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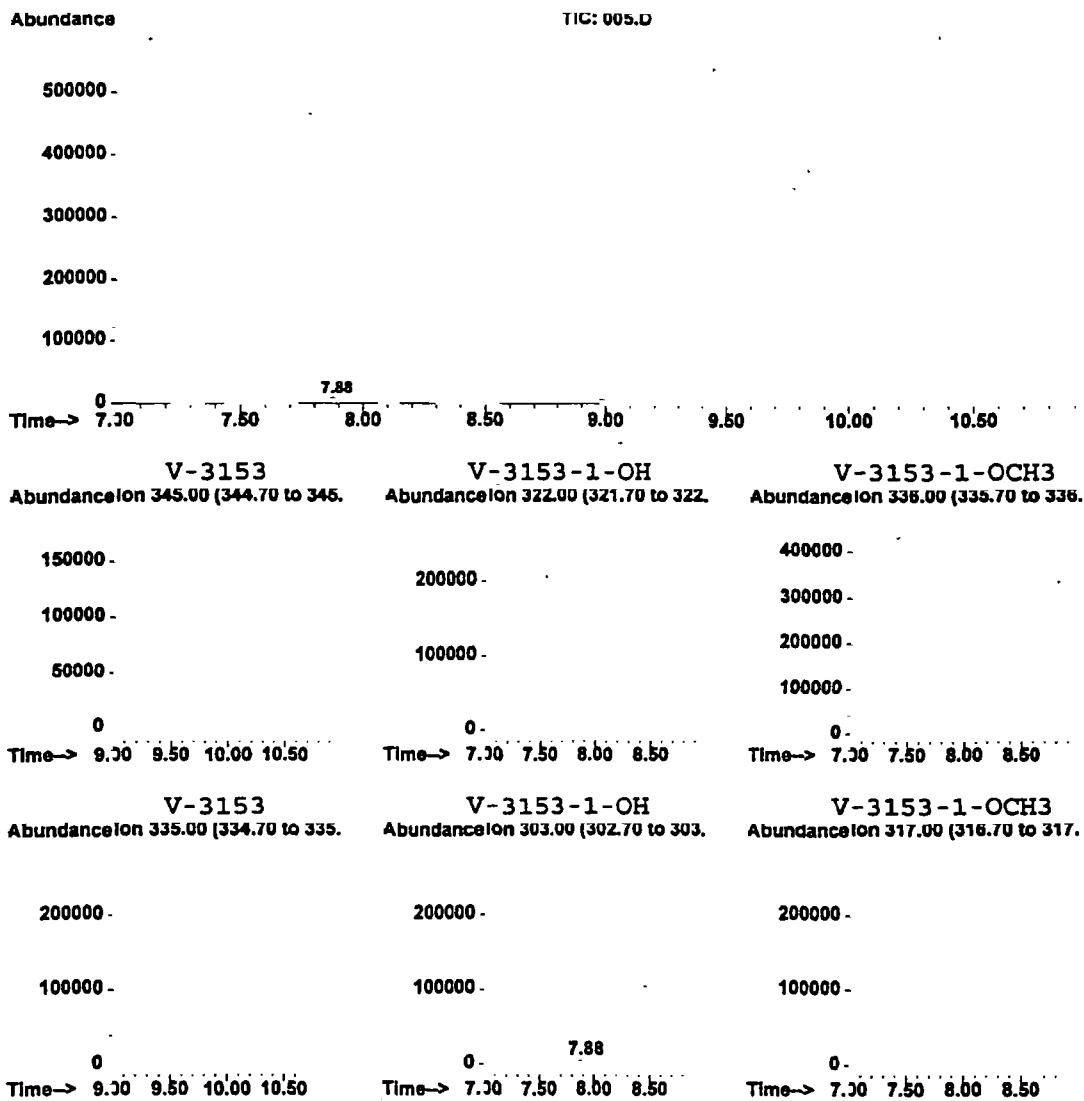


Figure 2. Soil Control Sample (Analyzed for S-3153, S-3153-1-OH, & S-3153-1-OCH₃; 1.0 μL injection)

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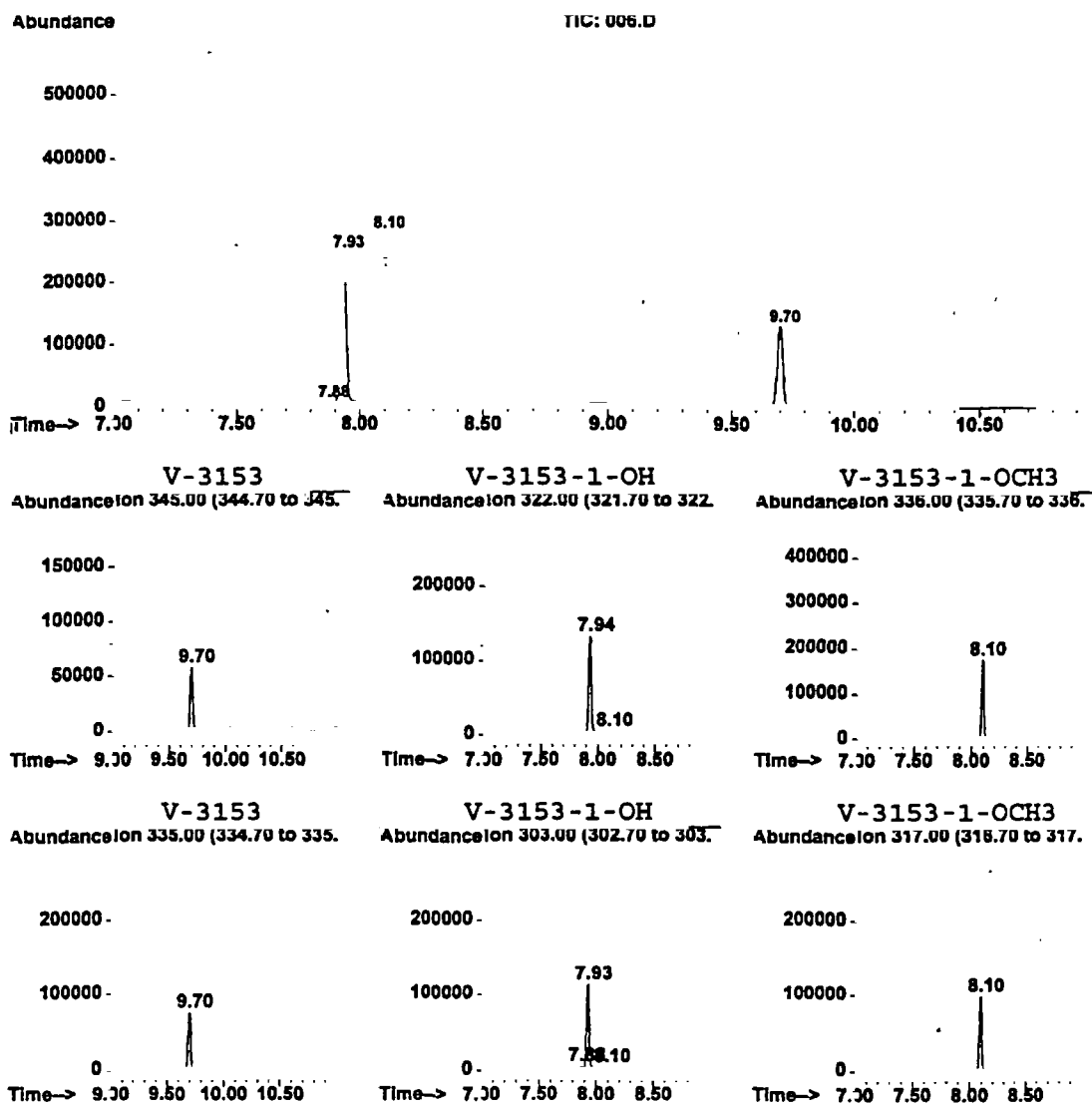


Figure 3. Fortified Soil Sample (0.025 ppm S-3153, S-3153-1-OH, & S-3153-1-OCH₃; 1.0 μ L injection)

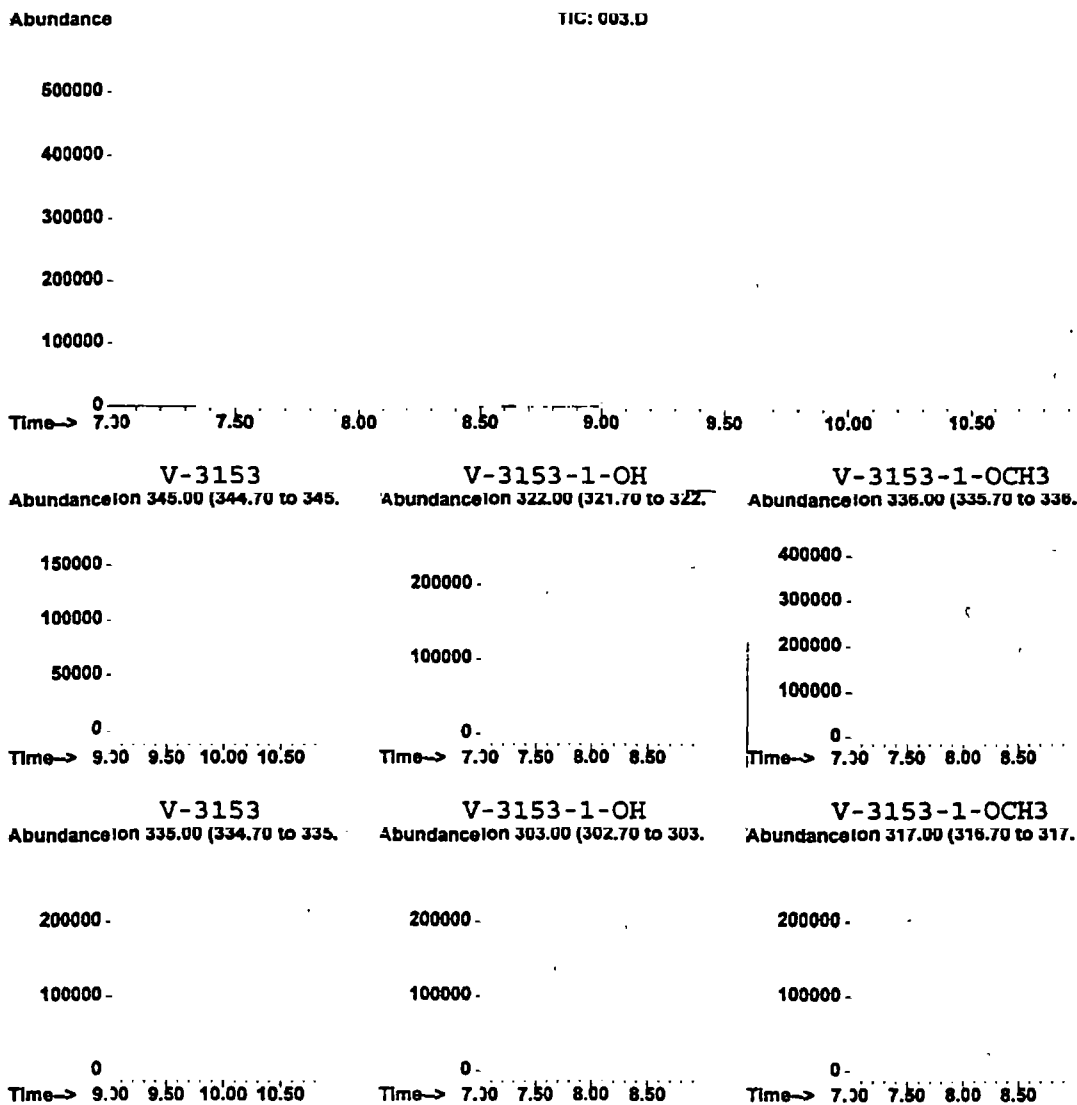


Figure 4. Soil Control Sample (Analyzed for S-3153 Acid as S-3153; 1.0 μ L injection)

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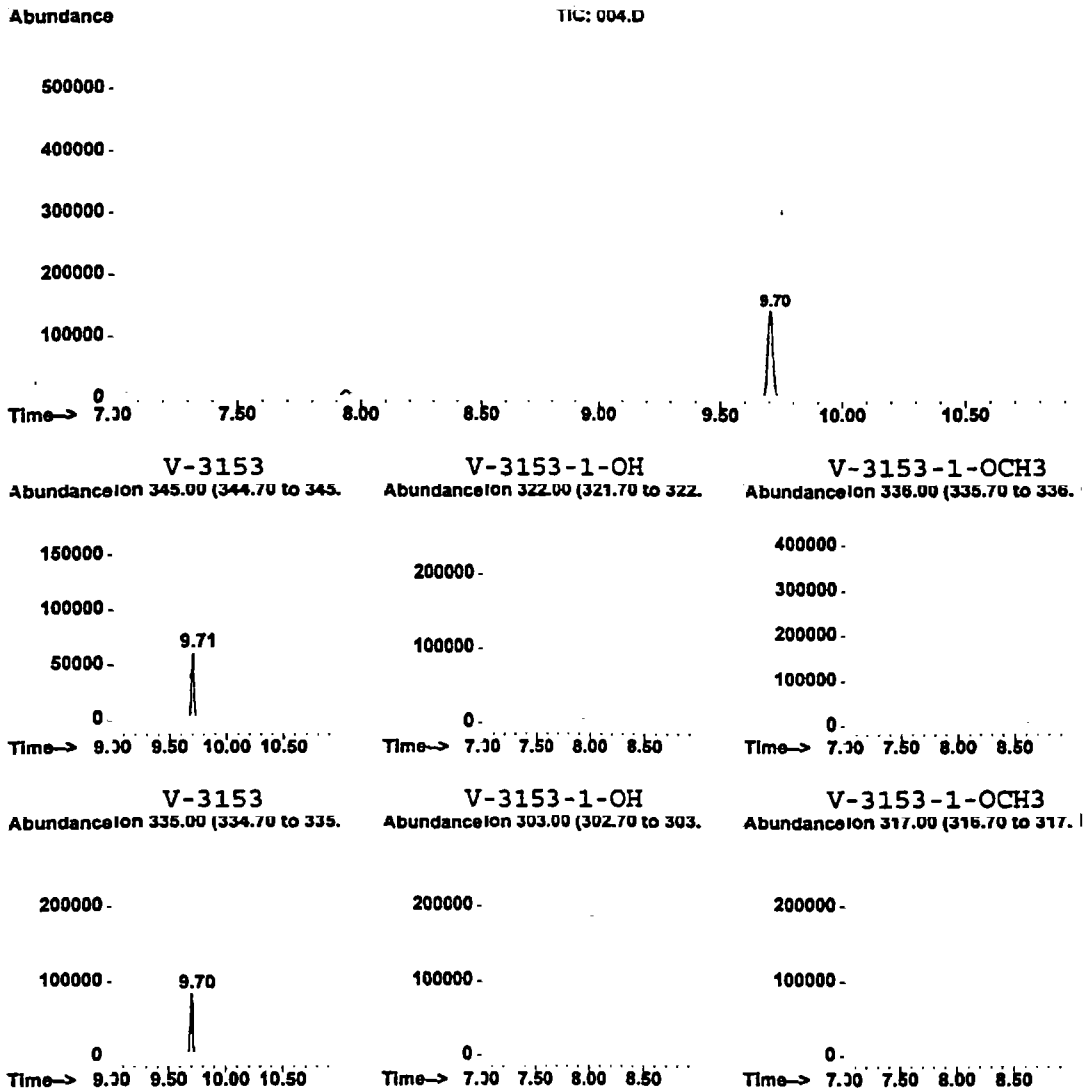


Figure 5. Fortified Soil Sample (0.025 ppm S-3153 Acid; analyzed as S-3153; 1.0 μ L injection)

VALIDATION REPORT FOR RESIDUE METHOD RM-36-2
REPRODUCIBILITY OF ANALYSIS (REF. SOP VR-002)

DETERMINATION OF RESIDUES OF S-3153, S-3153 ACID (M-1),
S-3153-1-OH (M-5), and S-3153-1-OCH₃ (M-9) RESIDUES IN SOIL

CHEMICAL: S-3153

Sample Matrix	Reference Page	Extraction Date	Fortification Level (ppm)	Result (ppm)	Percent Recovery ^a	
Soil	VDL-473-50	5/26/98	0.005	0.0054	108	
Soil	VDL-473-50	5/26/98	0.005	0.0058	116	
Soil	VDL-473-50	5/26/98	0.005	0.0057	113	
Soil	VDL-473-50	5/26/98	0.025	0.0229	91	
Soil	VDL-473-50	5/26/98	0.025	0.0237	95	
Soil	VDL-473-50	5/26/98	0.025	0.0232	93	
Soil	VDL-473-50	5/26/98	0.025	0.0247	99	Mean = 100%
Soil	VDL-473-50	5/26/98	0.025	0.0224	90	CV = 9.9%
Soil	VDL-473-50	5/26/98	0.025	0.0235	94	n = 9

^a Corrected for 0.00003 ppm equivalents in control sample.

CHEMICAL: S-3153 Acid (M-1)

Sample Matrix	Reference Page	Extraction Date	Fortification Level (ppm)	Result (ppm)	Percent Recovery ^b	
Soil	VDL-473-50	5/26/98	0.005	0.0039	78	
Soil	VDL-473-50	5/26/98	0.005	0.0042	82	
Soil	VDL-473-50	5/26/98	0.005	0.0040	79	
Soil	VDL-473-50	5/26/98	0.025	0.0142	57	^c
Soil	VDL-473-50	5/26/98	0.025	0.0194	77	
Soil	VDL-473-50	5/26/98	0.025	0.0170	68	^c
Soil	VDL-473-50	5/26/98	0.025	0.0191	76	Mean = 76%
Soil	VDL-473-50	5/26/98	0.025	0.0209	83	CV = 12%
Soil	VDL-473-50	5/26/98	0.025	0.0215	86	n = 9

^b Corrected for 0.00005 ppm equivalents in control sample.

^c Below 70% Recovery - See Comments section for additional data obtained after modification.

CHEMICAL: S-3153-1-OH (M-5)

Sample Matrix	Reference Page	Extraction Date	Fortification Level (ppm)	Result (ppm)	Percent Recovery ^d	
Soil	VDL-473-50	5/26/98	0.005	0.0070	122	
Soil	VDL-473-50	5/26/98	0.005	0.0068	118	
Soil	VDL-473-50	5/26/98	0.005	0.0075	131	^e
Soil	VDL-473-50	5/26/98	0.025	0.0288	112	
Soil	VDL-473-50	5/26/98	0.025	0.0314	122	
Soil	VDL-473-50	5/26/98	0.025	0.0295	114	
Soil	VDL-473-50	5/26/98	0.025	0.0304	118	Mean = 118%
Soil	VDL-473-50	5/26/98	0.025	0.0278	108	CV = 5.7%
Soil	VDL-473-50	5/26/98	0.025	0.0307	119	n = 9

^d Corrected for 0.0009 ppm equivalent in control sample.

^e Above 130% Recovery.

DETERMINATION OF RESIDUES OF S-3153, S-3153 ACID (M-1),
 S-3153-1-OH (M-5), AND S-3153-1-OCH₃ (M-9) RESIDUES IN SOIL

CHEMICAL: S-3153-1-OCH ₃ (M-9)					
Sample Matrix	Reference Page	Extraction Date	Fortification Level (ppm)	Result (ppm)	Percent Recovery ^e
Soil	VDL-473-50	5/26/98	0.005	0.0053	106
Soil	VDL-473-50	5/26/98	0.005	0.0060	119
Soil	VDL-473-50	5/26/98	0.005	0.0057	114
Soil	VDL-473-50	5/26/98	0.025	0.0244	98
Soil	VDL-473-50	5/26/98	0.025	0.0265	106
Soil	VDL-473-50	5/26/98	0.025	0.0249	99
Soil	VDL-473-50	5/26/98	0.025	0.0258	103
Soil	VDL-473-50	5/26/98	0.025	0.0234	94
Soil	VDL-473-50	5/26/98	0.025	0.0257	103
					Mean = 105%
					CV = 7.6%
					n = 9

^e Corrected for 0.00004 ppm equivalent in control sample.

ppm = mg/kg

COMMENTS:

S-3153 Acid preparation modified to include washing the methylene chloride extract (after partitioning of the acidic ethanolic mixture from the esterification) with buffer solution to remove the acid.

CHEMICAL: S-3153 Acid					
Sample Matrix	Reference Page	Extraction Date	Fortification Level (ppm)	Result (ppm)	Percent Recovery
Soil	V-11989-1	5/28/98	0.025	0.0220	88
Soil	V-11989-2	5/28/98	0.025	0.0189	76
Soil	V-11989-3	5/29/98	0.005	0.0035	70
Soil	V-11989-4	6/2/98	0.005	0.0042	84
					Mean = 80%
					CV = 10%
					n = 4

Julian Kowalski
 Analyst

11/4/99
 Date

Charles Brown
 Approved by

11/2/99
 Date

J. Morris
 Reviewed by

11/4/99
 Date

APPENDIX III

Analytical Data Sheets

Morse Laboratories, Inc.

Residue results for S-3153 in soil

Sid conc (µg/mL)	M.L. Ticket No	Sample ID	Set No	Extraction Date	Injection Date	Sample Wt (g)	Fort Level (ppm)	mL solv	FV mL	GC Dil Fact	Peak Resp	Avg Sid Response	ppm	rept ppm	ppm control	% Rec
0.05	78237	MLI-99-01 cont 4 reagent cont 3	2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	10	251711	245765	0	<0.005	0.000	101
0.05	78237	MLI-99-01 cont 5	2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	10	259336	245765	0	<0.005	0.000	101
0.05	78237	MLI-99-01 fort cont 6	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	20	248737	245765	0.050604643	0.0506	0.000	101
0.01	78237	MLI-99-01 fort cont 7	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	20	46573	245765	0.04890139	0.0489	0.000	98
0.05	78237	MLI-99-01 fort cont 8	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	20	257781	245765	0.052444612	0.0524	0.000	105
0.1	78237	MLI-99-01 fort cont 9	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	20	419874	245765	0.057834924	0.0578	0.000	118
0.05	78237	MLI-99-01 fort cont 10	2-subset 2	19-Jan-00	09-Feb-00	20.0	0.05	250	1.0	20	290747	245765	0.059151425	0.0592	0.000	118
0.05	78237	reagent cont 4	3	09-Feb-00	18-Feb-00	20.0		250	1.0	10	8666	8327	0	<0.005	0.000	
0.05	78237	MLI-99-01 soil cont 6	3	09-Feb-00	18-Feb-00	20.0		250	1.0	10	0	8327	0	<0.005	0.000	
0.004	78237	MLI-99-01 soil cont 7	3	09-Feb-00	18-Feb-00	20.0		250	1.0	10	7937	8327	0.005190945	0.00519	0.000	104
0.05	78237	MLI-99-01 soil fort cont 11	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	546	8327	0	<0.005	0.000	104
0.01	78237	MLI-99-01 soil fort cont 12	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	1729	8327	0.005190945	0.00519	0.000	104
0.05	78237	MLI-99-01 soil fort cont 13	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	8711	8327	0.005190945	0.00519	0.000	104
0.05	78237	MLI-99-01 soil fort cont 14	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	1538	8327	0.004980785	0.00498	0.000	100
0.10	78237	MLI-99-01 soil fort cont 15	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	1995	8327	0.005992554	0.00599	0.000	120
0.05	78237	MLI-99-01 soil fort cont. 15	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	7506	8327	0.005992554	0.00599	0.000	120
0.05	78237	MLI-99-01 soil fort cont. 15	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	15681	8327	0.005821424	0.00582	0.000	116
0.05	78237	MLI-99-01 soil fort cont. 15	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	1696	8327	0.00509187	0.00509	0.000	102
0.05	78237	MLI-99-01 soil fort cont. 15	3	09-Feb-00	18-Feb-00	20.0	0.005	250	1.0	10	8814	8327	0.00509187	0.00509	0.000	102

M.L. Project No ML99-0800-VAL
Protocol No ML99-01

Residue results for S-3153-1-OH in soil

Morse Laboratories, Inc

Sid conc (ug/mL)	M L Ticket No	Sample ID	Set No	Extraction Date	Injection Date	Sample Wt q	Fort Level (ppm)	mL solv	FV mL	GC Dil Fact	Peak Resp	Avg Sid Response	ppm	rept ppm	ppm control	% Rec.
0.05	78237	MLI-99-01 cont 4 reagent cont 3	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	10	261045	273140	0	<0.005	0.000	104
0.05	78237	MLI-99-01 cont 5	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	10	298230	273140	0	<0.005	0.000	100
0.004	78237	MLI-99-01 fort cont 6	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	10	20307	273140	0	<0.005	0.000	114
0.05	78237	MLI-99-01 fort cont 7	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	20	284562	273140	0.052090869	0.0521	0.000	120
0.01	78237	MLI-99-01 fort cont 8	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	20	45928	273140	0.04992513	0.0499	0.000	127
0.05	78237	MLI-99-01 fort cont 9	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	20	310124	273140	0.056770154	0.0568	0.000	100
0.1	78237	MLI-99-01 fort cont 10	2-subset 2	19-Jan-00	08-Feb-00	20.0	0.05	250	1.0	20	259504	273140	0.05873142	0.0599	0.000	114
0.05	78237	MLI-99-01 fort cont 11	2-subset 2	19-Jan-00	09-Feb-00	20.0	0.05	250	1.0	20	348779	273140	0.063480083	0.0635	0.000	120
0.05	78237	reagent cont 4	3	09-Feb-00	18-Feb-00	20.0	0.05	250	1.0	10	14863	14088	0	<0.005	0.000	127
0.004	78237	MLI-99-01 soil cont 6	3	09-Feb-00	18-Feb-00	20.0	0.05	250	1.0	10	13761	14088	0	<0.005	0.000	127
0.05	78237	MLI-99-01 soil fort cont 11	3	09-Feb-00	18-Feb-00	20.0	0.05	250	1.0	10	1128	14088	0.004612081	0.00461	0.000	92
0.01	78237	MLI-99-01 soil fort cont 12	3	09-Feb-00	18-Feb-00	20.0	0.05	250	1.0	10	2599	14088	0.004812606	0.00481	0.000	96
0.05	78237	MLI-99-01 soil fort cont 13	3	09-Feb-00	18-Feb-00	20.0	0.05	250	1.0	10	3260	14088	0.005765065	0.00579	0.000	116
0.10	78237	MLI-99-01 soil fort cont 14	3	09-Feb-00	18-Feb-00	20.0	0.05	250	1.0	10	12158	14088	0.005501136	0.00550	0.000	110
0.05	78237	MLI-99-01 soil fort cont 15	3	09-Feb-00	18-Feb-00	20.0	0.05	250	1.0	10	2641	14088	0.004686613	0.00469	0.000	94
			3		18-Feb-00						14934					

Morse Laboratories, Inc

Residue results for S-3153-1-OCH₃ in soil

Std Ticket No	M.L. Project No ML99-0800-VAL Protocol No ML99-01	Sample ID	Set No	Extraction Date	Injection Date	Sample Wt g	Fort Level (ppm)	mL solv	FV mL	GC Dil Fact	Peak Resp	Avg Std Response	ppm	rept ppm	ppm control	% Rec
78237		MLI-99-01 cont 4 reagent cont 3	2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	10	511769	494813	0	<0.005	0	96
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	10	538527	494813	0	<0.005	0	
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	10	44685	494813	0	<0.005	0	
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	473744	494813	0.047871014	0.0479	0.000	96
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	482774	494813	0.047871014	0.0479	0.000	96
78237		MLI-99-01 fort cont 7	2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	98585	494813	0.048525301	0.0485	0.000	87
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	495620	494813	0.050081546	0.0501	0.000	100
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	458755	494813	0.050081546	0.0501	0.000	100
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	892474	494813	0.054208459	0.0542	0.000	108
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	536461	494813	0.054208459	0.0542	0.000	108
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	556501	494813	0.056233466	0.0562	0.000	112
			2-subset 2	19-Jan-00	08-Feb-00	20.0	20.0	250	1.0	20	482240	494813	0.056233466	0.0562	0.000	112
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	18555	17588	0	<0.005	0	
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	17168	17588	0	<0.005	0	
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	1204	17588	0.004974983	0.00497	0.000	99
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	3500	17588	0.004974983	0.00497	0.000	99
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	18311	17588	0.004974983	0.00497	0.000	99
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	3680	17588	0.00487264	0.00487	0.000	97
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	3428	17588	0.005913123	0.00591	0.000	118
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	4160	17588	0.005913123	0.00591	0.000	118
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	15415	17588	0.005516545	0.00552	0.000	110
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	34528	17588	0.004746134	0.00475	0.000	95
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	3881	17588	0.004746134	0.00475	0.000	95
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	3339	17588	0.004746134	0.00475	0.000	95
			3	09-Feb-00	18-Feb-00	20.0	20.0	250	1.0	10	18492	17588	0.004746134	0.00475	0.000	95

M.L. Project No ML99-0800-VAL
Protocol No ML99-01

Residue results for S-3153 Acid in soil

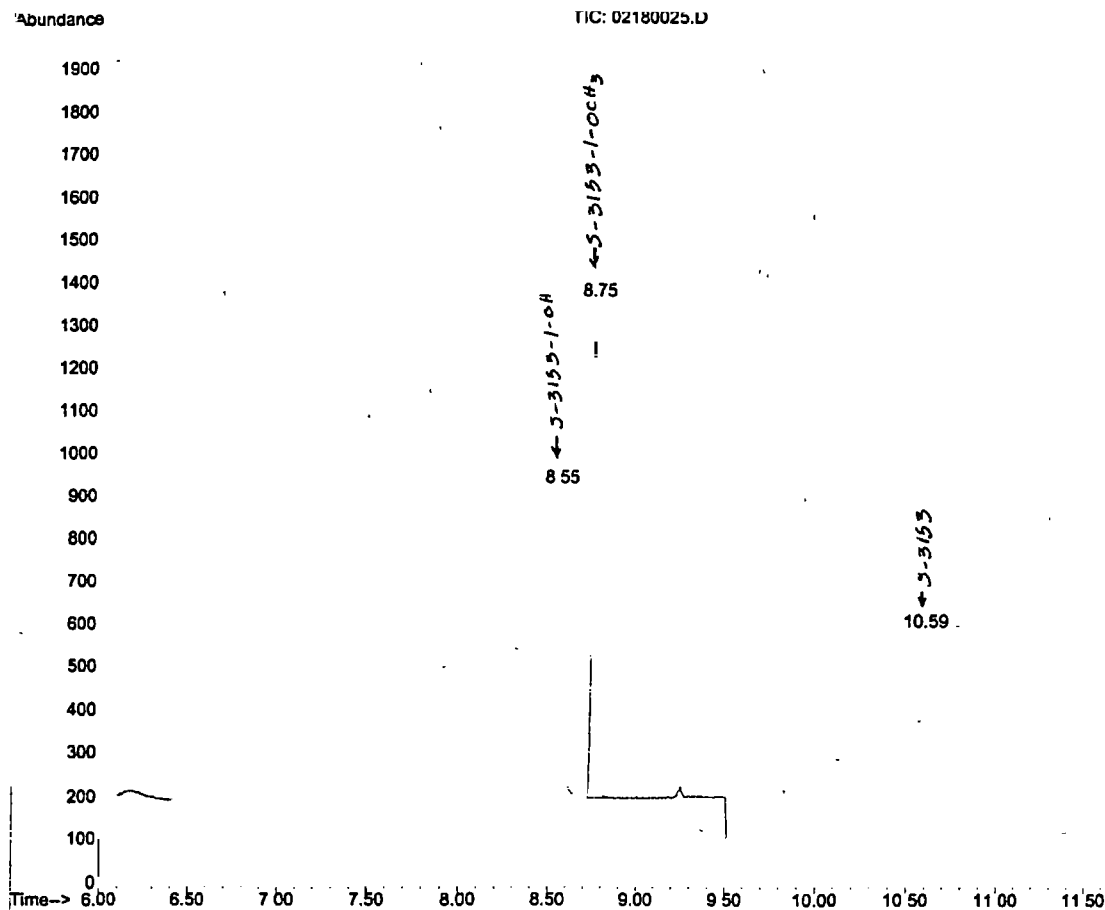
Morse Laboratories, Inc

Std conc (ug/mL)	M L Ticket No	Sample ID	Set No	Extraction Date	Injection Date	Sample Wt (g)	Fort Level (ppm)	mL solv	FV mL	GC Dil Fact	Peak Resp S-3153	Avg Std Response	ppm S-3153 Acid	rept ppm	ppm control	% Rec
0.5			2-subset 2		03-Feb-00						404173					
0.1	78237	MLI-99-01 fort cont 7	2-subset 2	19-Jan-00	03-Feb-00	20.0	0.05	250	1.0	2	72710	390381	0.045661315	0.0457	0.000250	91
0.5	78237	MLI-99-01 fort cont 8	2-subset 2	19-Jan-00	03-Feb-00	20.0	0.05	250	1.0	2	383340	390381	0.045412366	0.0454	0.000250	91
1.0			2-subset 2		03-Feb-00						384547					
0.5	78237	MLI-99-01 fort cont 9	2-subset 2	19-Jan-00	03-Feb-00	20.0	0.05	250	1.0	2	789770	390381	0.048428105	0.0484	0.000250	97
0.5	78237	MLI-99-01 fort cont 10	2-subset 2	18-Jan-00	03-Feb-00	20.0	0.05	250	1.0	2	409613	390381	0.048790808	0.0488	0.000250	98
0.5	78237	MLI-99-01 fort cont 4	2-subset 2	19-Jan-00	03-Feb-00	20.0		250	1.0	1	4927	390381	0.000293438	<0.005		
0.5		reagent cont 3	2-subset 2	19-Jan-00	03-Feb-00	20.0		250	1.0	1	0	390381	0	<0.005		
0.04			2-subset 2		03-Feb-00						405008					
0.5	78237	MLI-99-01 cont 5	2-subset 2	19-Jan-00	03-Feb-00	20.0		250	1.0	1	27569	390381	0.000207974	<0.005		
0.5	78237	MLI-99-01 fort cont 6	2-subset 2	19-Jan-00	03-Feb-00	20.0	0.05	250	1.0	2	3492	390381	0.044017298	0.0440	0.000250	88
0.5			2-subset 2		03-Feb-00						377851					
0.5		reagent cont 4	3	09-Feb-00	17-Feb-00	20.0		250	1.0	1	30661	30480	0	<0.005		
0.5	78237	MLI-99-01 soil cont 6	3	09-Feb-00	17-Feb-00	20.0		250	1.0	1	0	30480	0	<0.005		
0.04			3		17-Feb-00						30713					
0.5	78237	MLI-99-01 soil cont 7	3	09-Feb-00	17-Feb-00	20.0		250	1.0	1	2298	30480	0	<0.005		
0.5	78237	MLI-99-01 soil fort cont 11	3	09-Feb-00	17-Feb-00	20.0	0.005	250	1.0	1	5376	30480	0.00410787	0.00410	0.000	82
0.1			3		17-Feb-00						29906					
0.5	78237	MLI-99-01 soil fort cont 12	3	09-Feb-00	17-Feb-00	20.0	0.005	250	1.0	1	5558	30480	0.004162574	0.00416	0.000	83
0.5	78237	MLI-99-01 soil fort cont 13	3	09-Feb-00	17-Feb-00	20.0	0.005	250	1.0	1	5412	30480	0.004128248	0.00413	0.000	83
1.0			3		17-Feb-00						30849					
0.5	78237	MLI-99-01 soil fort cont 14	3	09-Feb-00	17-Feb-00	20.0	0.005	250	1.0	1	60997	30480	0.00408248	0.00408	0.000	82
0.5	78237	MLI-99-01 soil fort cont 15	3	09-Feb-00	17-Feb-00	20.0	0.005	250	1.0	1	5578	30480	0.004254872	0.00425	0.000	85
0.5			3		17-Feb-00						30273					

APPENDIX IV

Example Chromatograms

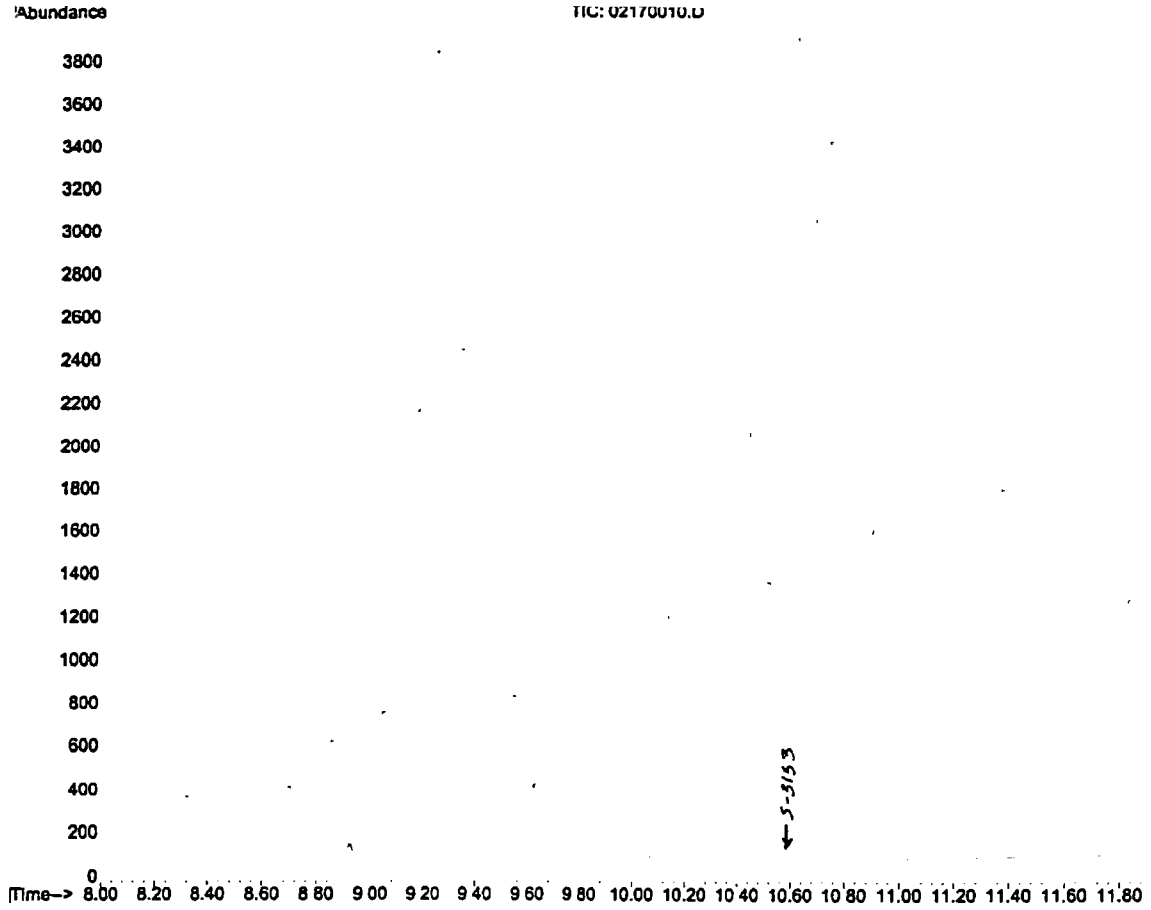
EXAMPLE GC CHROMATOGRAM, STANDARD,
SET# 3



peak #	R. T. min	PK TY	peak height	peak area
1	8.546	M	723	14722
2	8.754	M	1164	18311
3	10.590	M	484	8711

S-3153-1-OH, S-3153-1-OCH3, S-3153
0.05 µg/mL Standard,
1 µL (10X dil. of 0.5 µg/mL)

EXAMPLE GC CHROMATOGRAM, SOIL CONTROL SAMPLE,
SET# 3

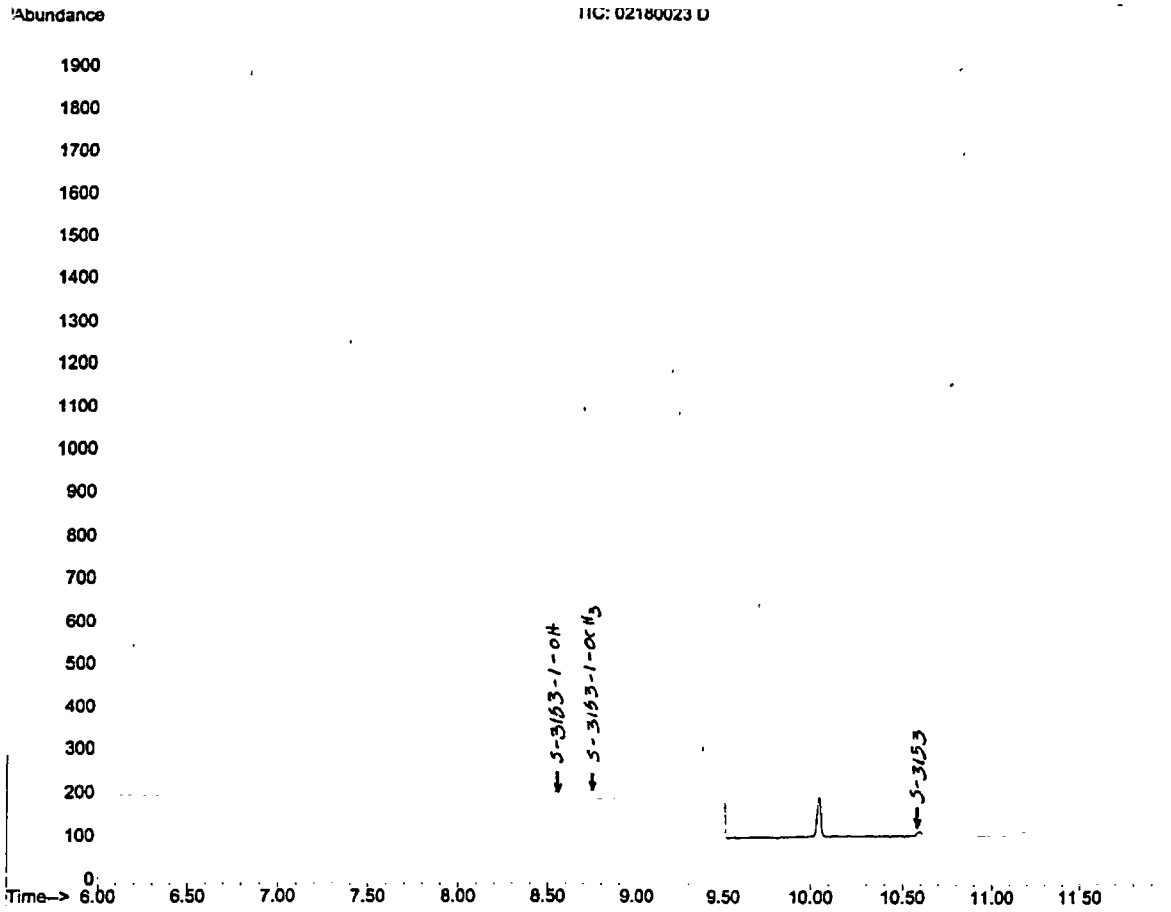


peak #	R.T. min	PK TY	peak height	peak area
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No peaks were detected using the method integration parameters!

MLI-99-01, Control 7
S-3153 Acid (as S-3153)
1 μ L of 20.0 g/mL injected

EXAMPLE GC CHROMATOGRAM, SOIL CONTROL SAMPLE,
SET# 3

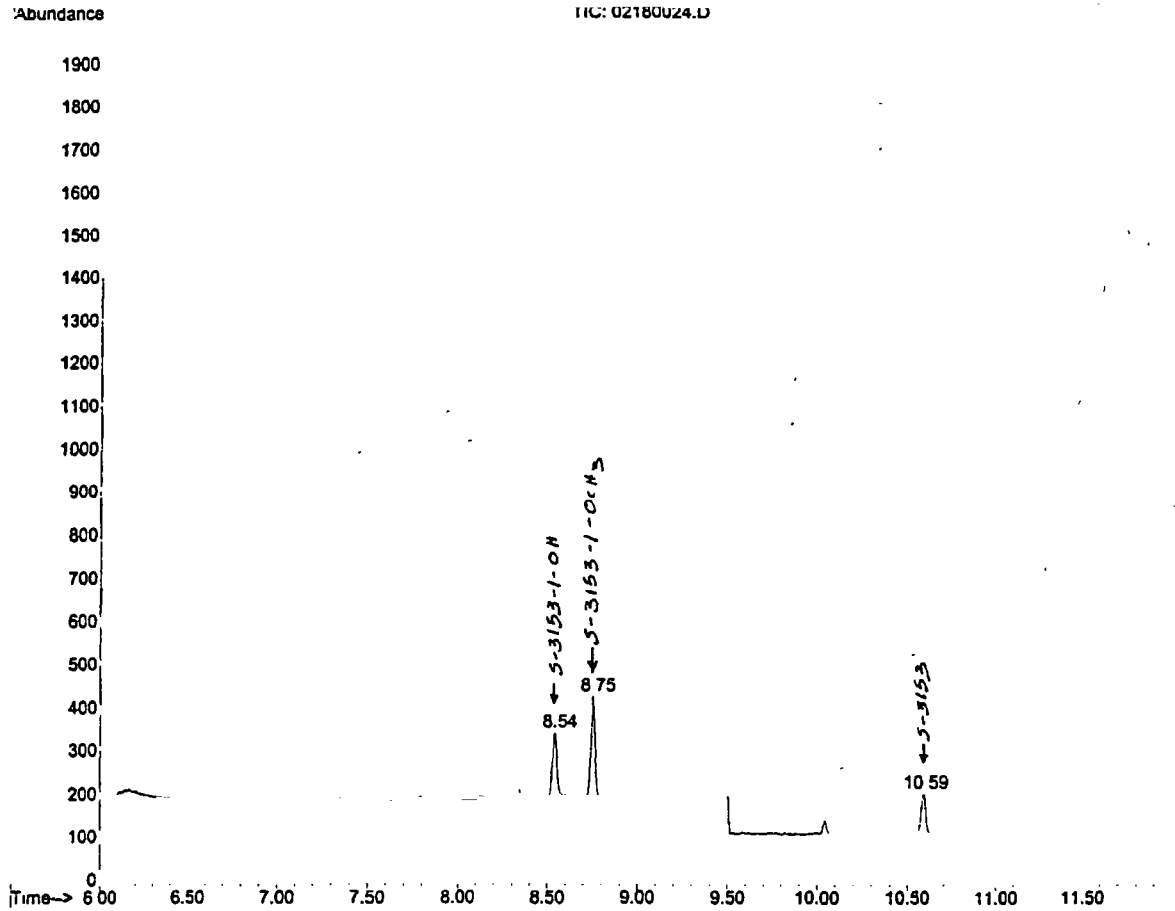


peak #	R.T. min	PK TY	peak height	peak area
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No peaks were detected using the method integration parameters!

MLI-99-01, Control 7
S-3153-1-OH, S-3153-1-OCH3, S-3153
1 μ L of 20.0 g/mL injected (10X dil.)

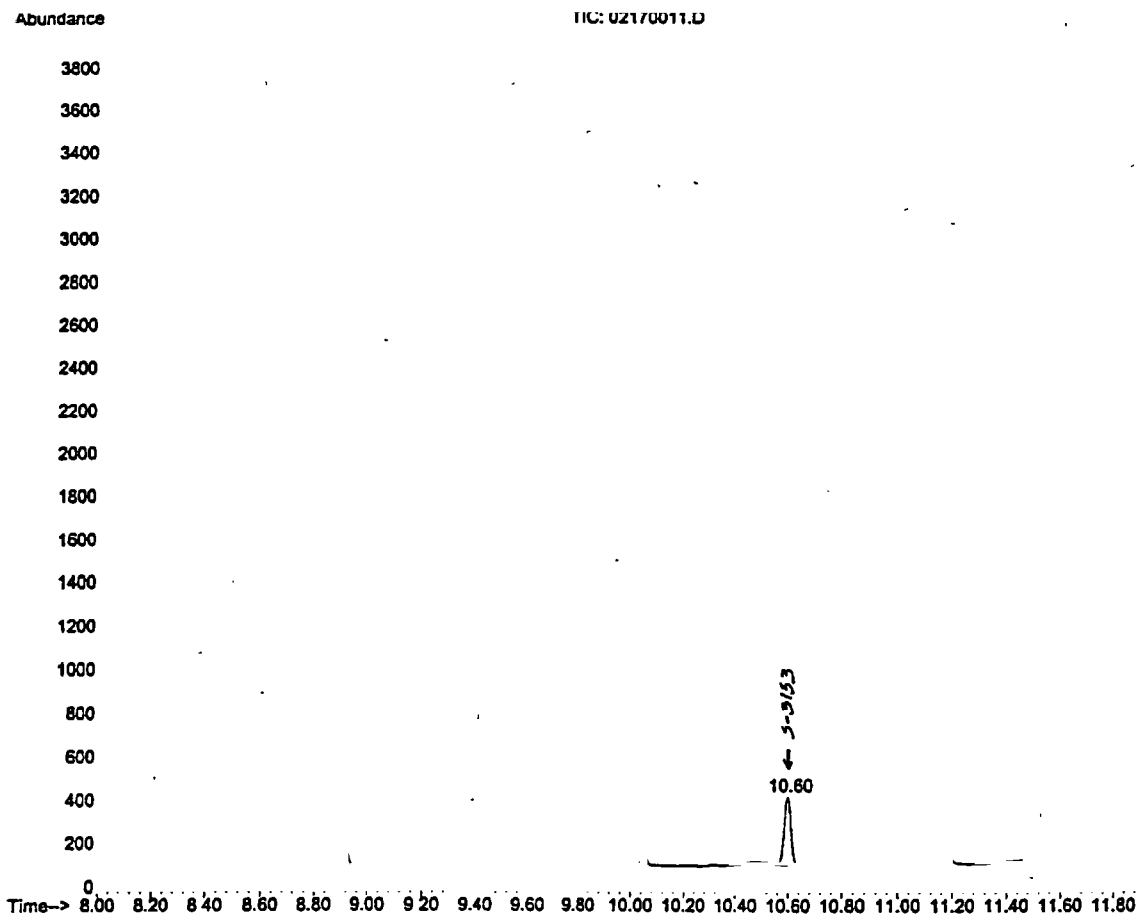
EXAMPLE GC CHROMATOGRAM, SOIL FORTIFIED CONTROL SAMPLE,
SET# 3



peak #	R.T. min	PK TY	peak height	peak area
1	8.541	M	149	2599
2	8.752	M	234	3500
3	10.590	M2	97	1729

MLI-99-01 Fortified Control 11
S-3153-1-OH, S-3153-1-OCH3, S-3153 @ 0.005 ppm each
1 μ L of 20.0 g/mL injected (10X dil.)

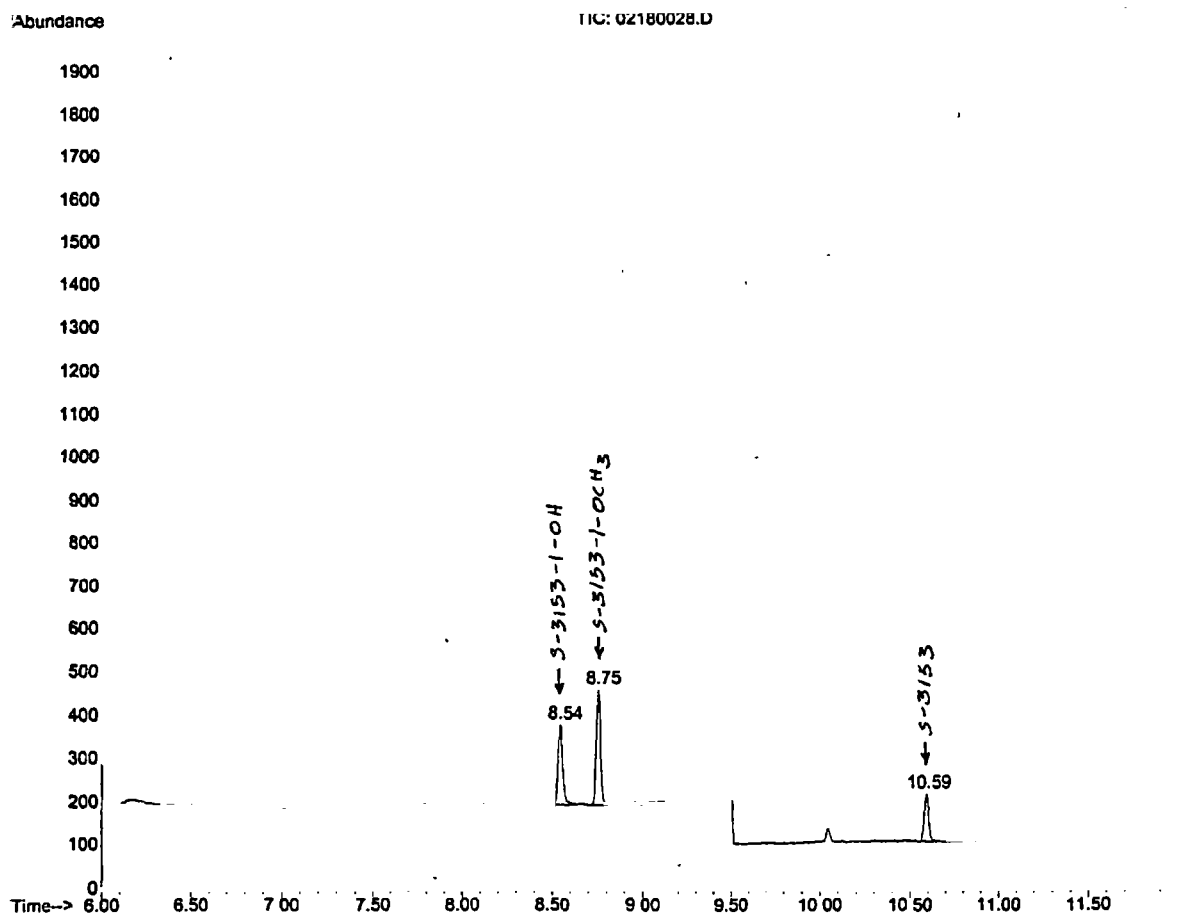
EXAMPLE GC CHROMATOGRAM, SOIL FORTIFIED CONTROL SAMPLE,
SET# 3



peak #	R.T. min	PK TY	peak height	peak area
1	10.598	BV	309	5376

MLI-99-01 Fortified Control 11
S-3153 Acid (as S-3153) @ 0.005 ppm
1 μ L of 20.0 g/mL injected

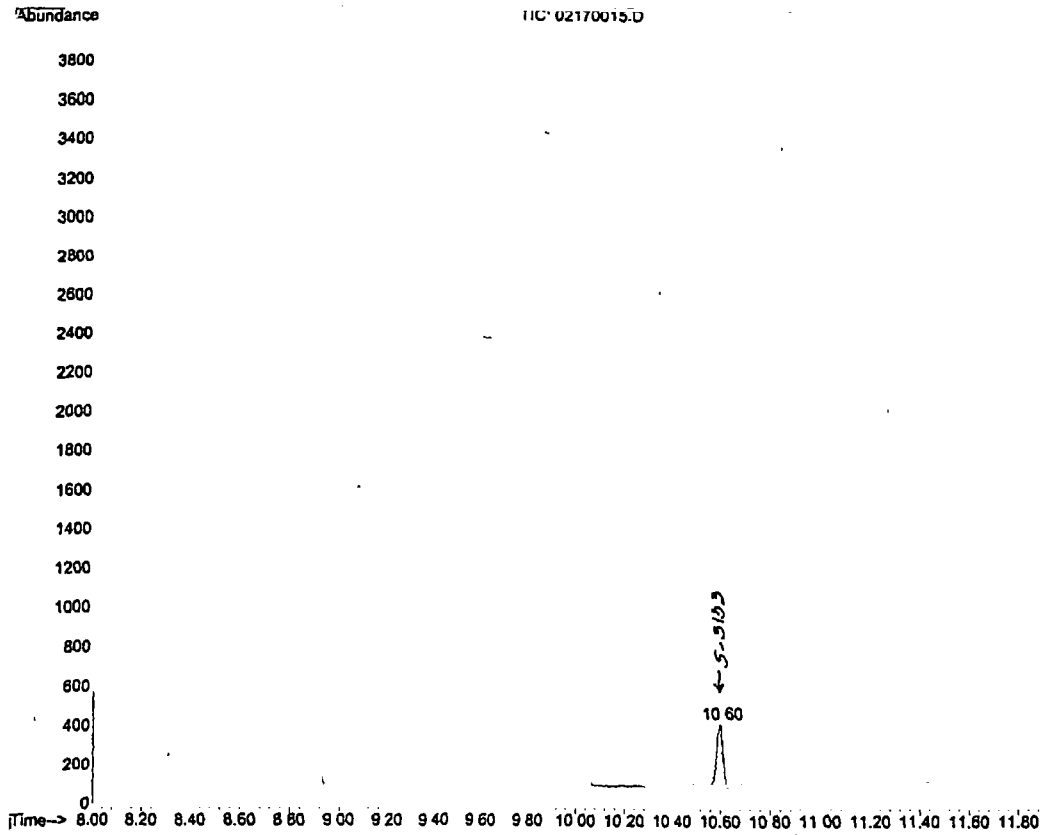
EXAMPLE GC CHROMATOGRAM, SOIL FORTIFIED CONTROL SAMPLE,
SET# 3



peak #	R.T. min	PK TY	peak height	peak area
1	8.538	M2	189	3260
2	8.754	M	267	4160
3	10.590	M2	112	1996

MLI-99-01 Fortified Control 13
 S-3153-1-OH, S-3153-1-OCH₃, S-3153 @ 0.005 ppm each
 1 μL of 20.0 g/mL injected (10X dil.)

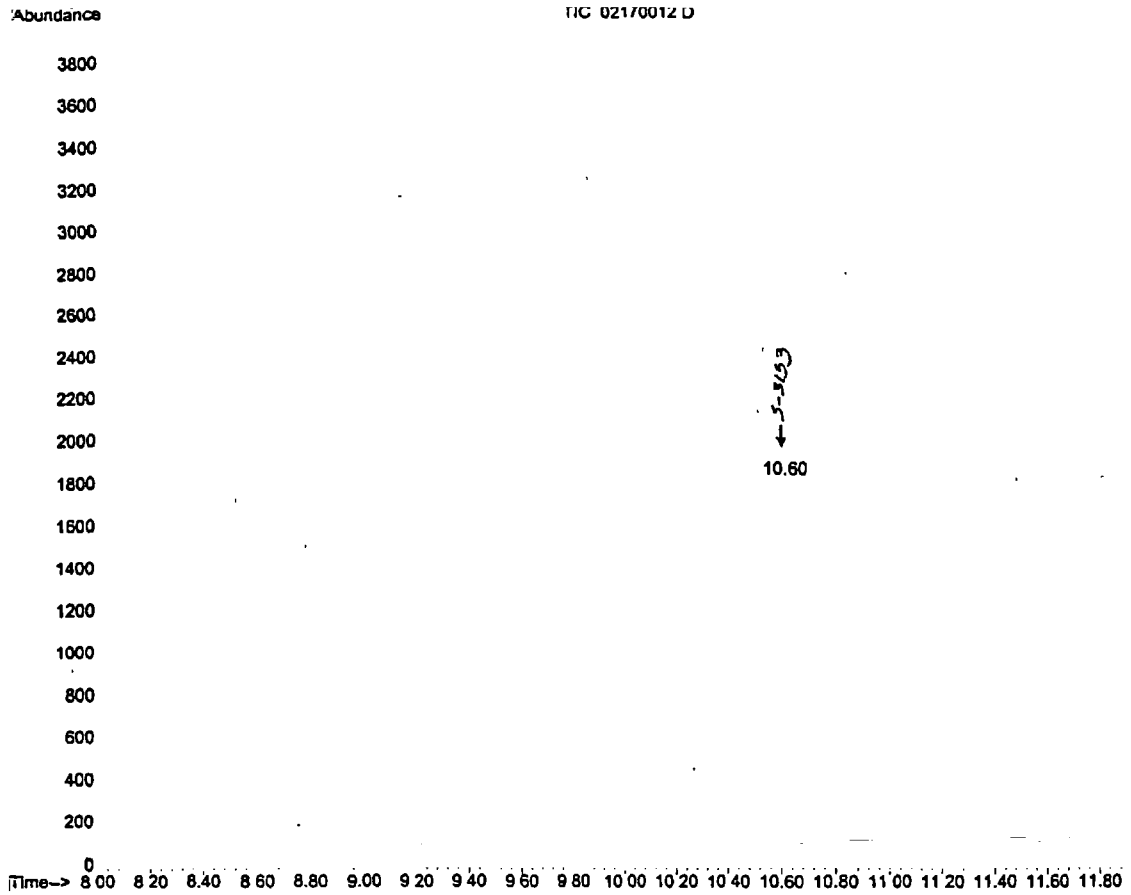
EXAMPLE GC CHROMATOGRAM, SOIL FORTIFIED CONTROL SAMPLE,
SET# 3



peak #	R.T. min	PK TY	peak height	peak area
1	10.598	BV 2	316	5412

MLI-99-01 Fortified Control 13
S-3153 Acid (as S-3153) @ 0.005 ppm
1 μ L of 20.0 g/mL injected

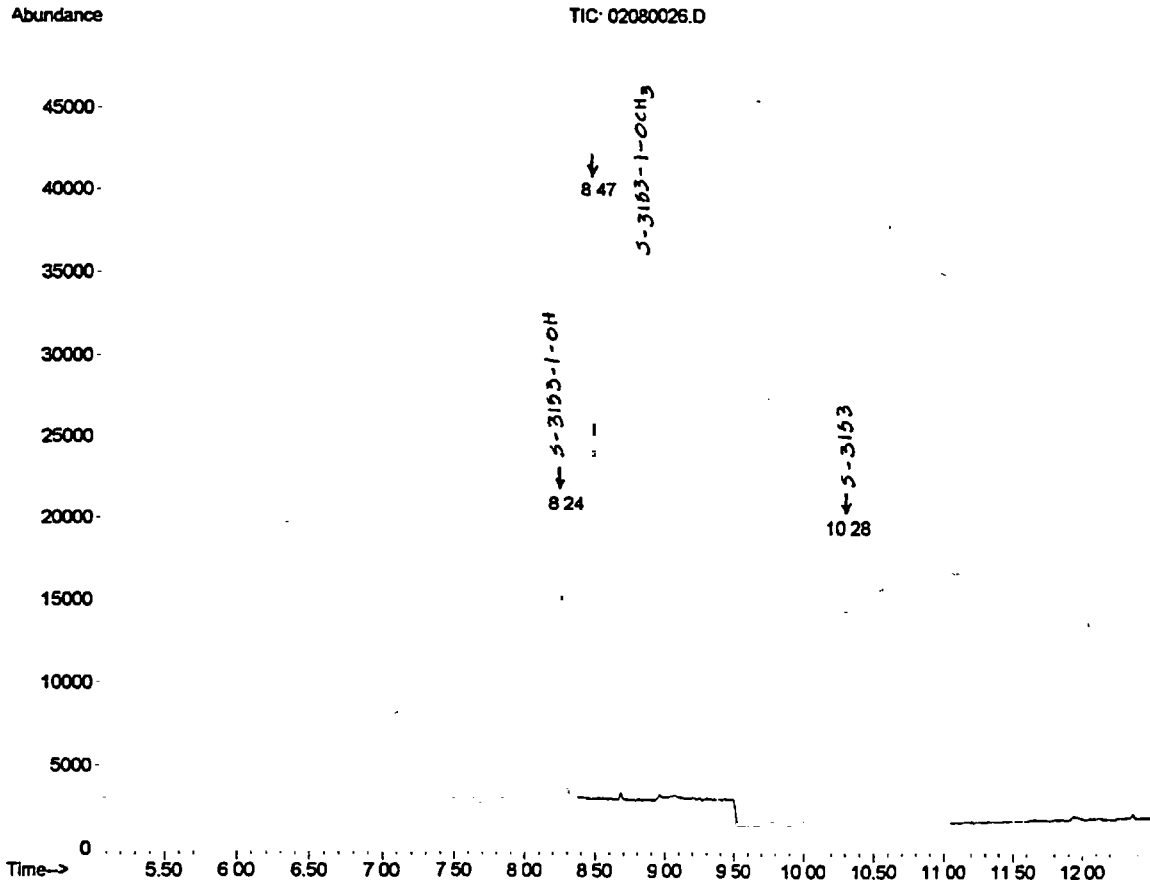
EXAMPLE GC CHROMATOGRAM, STANDARD,
SET# 2



peak #	R.T. min	PK TY	peak height	peak area
1	10.599	BV 2	1720	29906

S-3153
0.5 µg/mL Standard, 1 µL

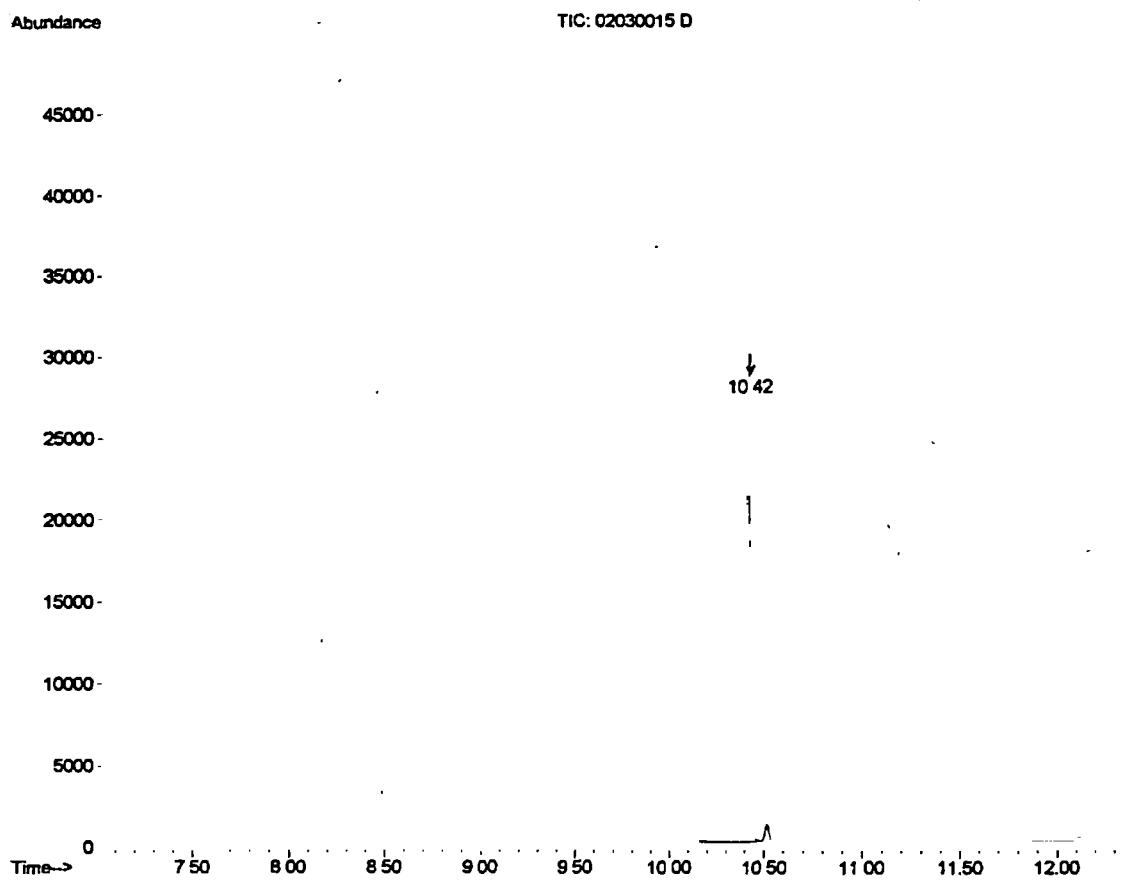
EXAMPLE GC CHROMATOGRAM, SOIL FORTIFIED CONTROL SAMPLE,
SET# 2



peak #	R.T. min	PK TY	peak height	peak area
1	8.243		17217	272731
2	8.468		36283	480219
3	10.283		17266	240365

MLI-99-01 Fortified Control 7
S-3153-1-OH, S-3153, 1-OCH3, S-3153 @ 0.05 ppm each
1 μ L of 20.0 g/mL injected (20X dil.)

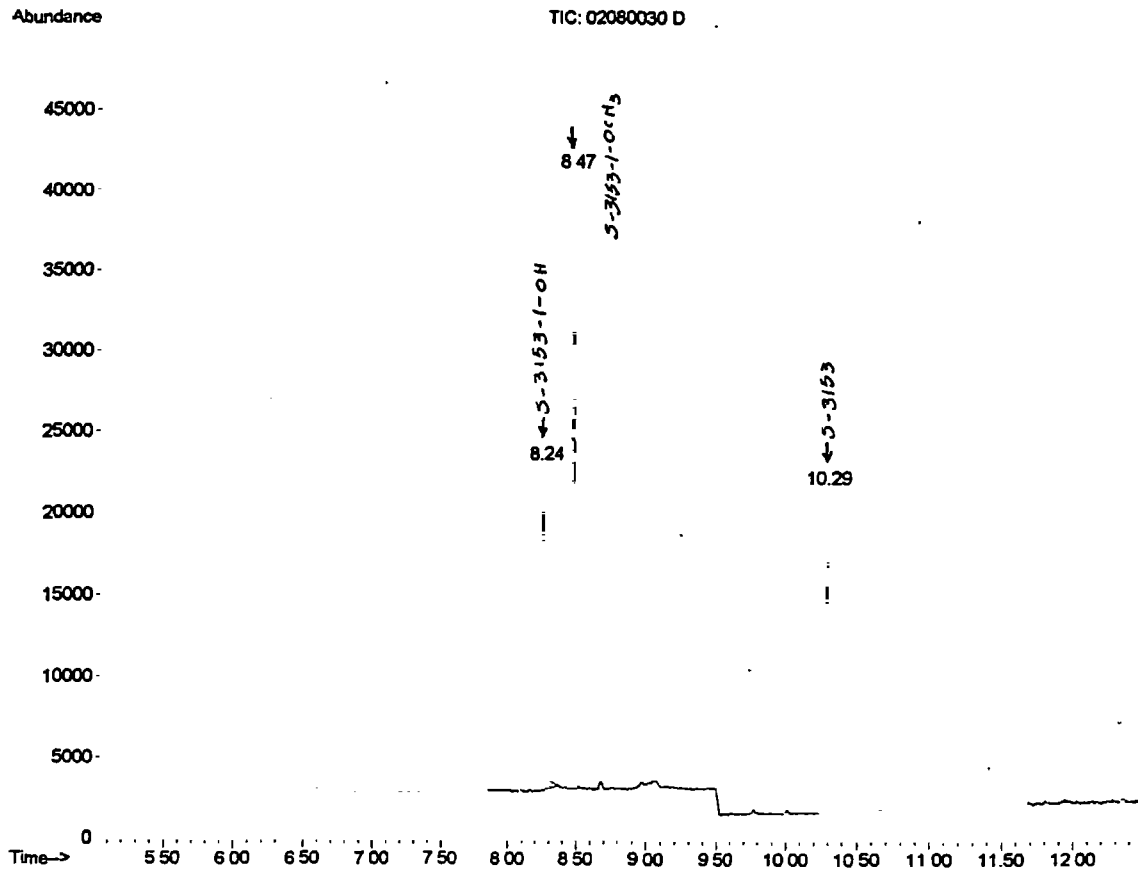
EXAMPLE GC CHROMATOGRAM, SOIL FORTIFIED CONTROL SAMPLE,
SET# 2



peak #	R.T. min	PK TY	peak height	peak area
1	10.415	BB	27100	383340

MLI-99-01 Fortified Control 7
S-3153 Acid (as S-3153) @ 0.05 ppm
1 μ L of 20.0 g/mL injected (2X dil.)

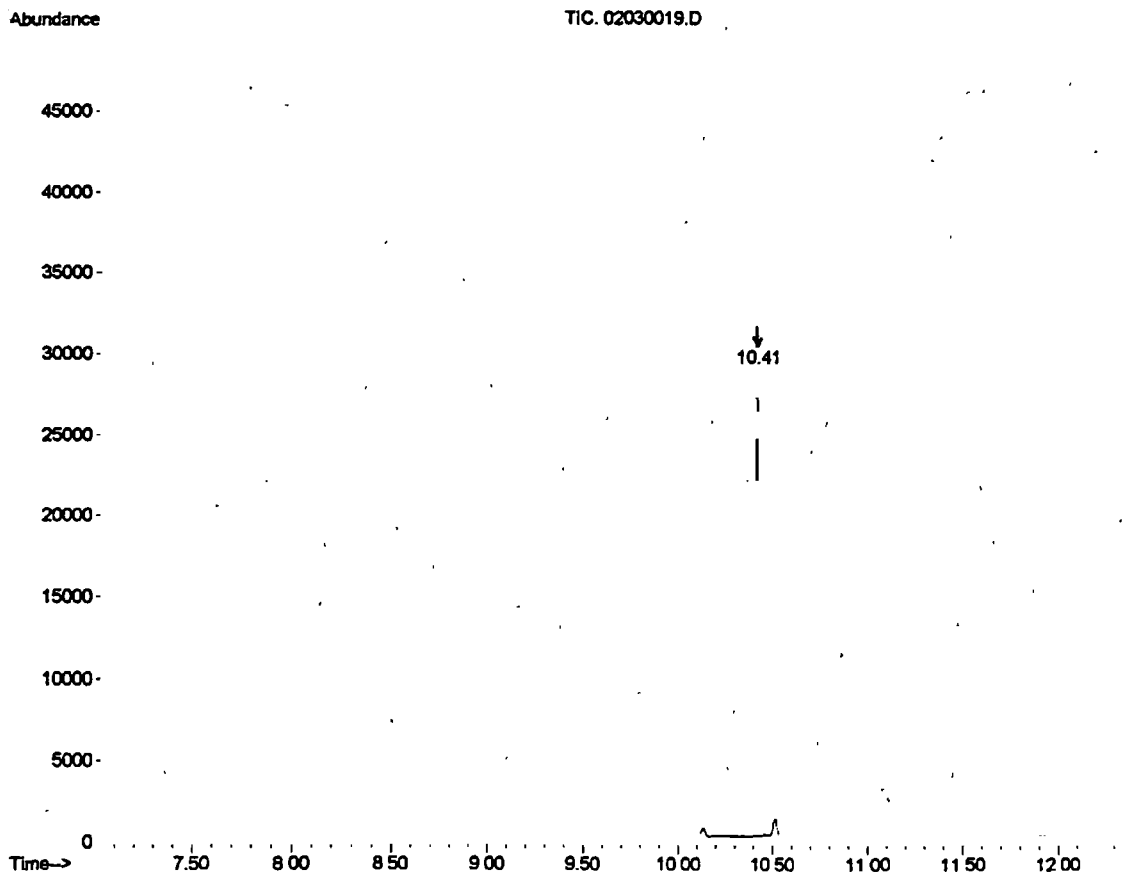
EXAMPLE GC CHROMATOGRAM, SOIL FORTIFIED CONTROL SAMPLE,
SET# 2



peak #	R.T. min	PK TY	peak height	peak area
1	8.246		20264	327075
2	8.471		39169	536461
3	10.283		20302	284276

MLI-99-01 Fortified Control 9
S-3153-1-OH, S-3153-1-OCH3, S-3153 @ 0.05 ppm each
1 μ L of 20.0 g/mL injected (20X dil.)

EXAMPLE GC CHROMATOGRAM, SOIL FORTIFIED CONTROL SAMPLE,
SET# 2



peak #	R.T. min	PK TY	peak height	peak area
1	10.413	BB 2	28600	406568

MLI-99-01 Fortified Control 9
S-3153 Acid (as S-3153) @ 0.05 ppm
1 μ L of 20.0 g/mL injected (2X dil.)