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VOLUME 2 OF 2 OF SUBMISSION

MEFENOXAM: Residue Analytical Method Report

TITLE

CONFIRMATORY ANALYTICAL METHOD FOR THE ENANTIOSELECTIVE DETERMINATION OF RESIDUES OF PARENT METALAXYL (CGA-48988) OR MEFENOXAM (CGA-329351) IN CROP SUBSTRATES BY CHIRAL HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH MASS SPECTROMETRIC DETECTION

DATA REQUIREMENT
OPPTS 860.1340

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LABORATORY STUDY IDENTIFICATION
Novartis Number 456-98

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VOLUME 1 OF 1 OF STUDY

STATEMENT OF NO DATA CONFIDENTIALITY CLAIM

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA Section 10 (d) (1) (A), (B) or (C).

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STATEMENT CONCERNING GOOD LABORATORY PRACTICES

This study was conducted in accordance with the applicable EPA Good Laboratory Practice Standards (40 CFR Part 160), with the following exception(s):

1) Novartis Metabolism Study 98JS30, "*Comparative Metabolism of [Phenyl-(U)-14C] CGA-329351 and [Phenyl-(U)-14C] CGA-48988 in Field Grown Lettuce*", is currently in-progress. Although the analytical phase of that study is complete, a final report has not yet been issued. Numerous references have been made in this report to draft data that will be issued in the final report of Study 98JS30.

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QUALITY ASSURANCE STATEMENT

Study Title: Confirmatory Analytical Method for the Enantioselective Determination of Residues of Parent Metalaxyl (CGA-48988) or Mefenoxam (CGA-329351) in Crop Substrates by Chiral High Performance Liquid Chromatography with Mass Spectrometric Detection

Study Director: Mark C. Grunenwald

Study Number: 456-98

Pursuant to Good Laboratory Practice Regulations, this statement verifies that the aforementioned study was inspected and/or audited and the findings reported to Management and to the Study Director by the Quality Assurance Unit on the dates listed below.

Inspection/Audit Type	Inspection/Audit Dates	Reporting Date
Audit Protocol	10/28/98 - 10/28/98	10/28/98
Inspect Analytical	12/16/98 - 12/16/98	12/16/98
Audit Final Report	02/09/99 - 02/10/99	02/11/99

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GENERAL STUDY INFORMATION

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Technical Personnel L. S. Hamilton, A. R. Clayton, T. Blackburn

Test Substance(s) (Refer also to Figure 1)

Code Number	Common Name	Lot Number	Purity	Reassay Date
CGA-48988	Metalaxyl	S94-1763	97.1%	10/2000
CGA-329351	Mefenoxam	S97-2087	98.1%	8/2000

Protocol Number Protocol 456-98[1]

Sample Origin Samples of tobacco, tomatoes, cabbage, and potato foliage were obtained from previously conducted Novartis Crop Protection residue field trial programs. Complete details of sample history, including test formulation, test substance lot, application rates, and data of harvest, are provided in the Residue Test Reports[2,3,4,5].

Samples of lettuce treated with ^{14}C-CGA-48988 and ^{14}C-CGA-329351 were obtained from Study 98JS30[6], Novartis Crop Protection AG, Basel, Switzerland; complete details of sample history will be provided in the final report for Study 98JS30 (in-progress).

Testing Facility Novartis Crop Protection, Inc.
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Study Initiation Date December 8, 1998

Testing Period December 9, 1998 - January 25, 1999

Archive Location The protocol, raw data, and final report will be archived at the Crop Protection Archives, Novartis Crop Protection, Greensboro, North Carolina at the end of the study. After the final report is issued, and with the approval of the Novartis QAU, any remaining samples may be discarded.

Circumstances Affecting Study None.

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I. SUMMARY AND INTRODUCTION

A. SCOPE

This method provides for the enantioselective determination of parent residues of metalaxyl (CGA-48988) or its biologically active (R) enantiomer, mefenoxam, (CGA-329351) in crops. This analytical method has been developed to be compatible with Analytical Method AG-395[7], which is the currently accepted tolerance enforcement methodology for total residues of metalaxyl or mefenoxam and their metabolites determined as 2,6-dimethylaniline (DMA) in crops. This method utilizes the same sample extract generated by the total residue method to determine residues of the parent substance. The limit of detection (LOD) for residues of metalaxyl or mefenoxam, defined as the lowest amount of standard injected, is 0.19 ng for each enantiomer, or 0.38 ng as the metalaxyl equivalent. The limit of quantitation (LOQ), defined as the lowest fortification level used for procedural recoveries, is 0.005 ppm of each enantiomer, or 0.01 ppm as the metalaxyl equivalent. The chemical structures of metalaxyl and mefenoxam are shown in Figure 1.

This method uses a chiral high performance liquid chromatography separation system with mass spectrometric detection (LC/MS) as a highly selective technique to identify the source of parent residues of metalaxyl or mefenoxam. Assuming that total residues as 2,6-DMA have previously been determined in a crop sample using Analytical Method AG-395, this method allows the analyst to identify the source of those residues as having resulted from the application of a commercial formulation containing either metalaxyl or mefenoxam as its applied active ingredient.

The representative substrates studied for the validation of this procedure were tobacco, tomatoes, cabbage, potato foliage, and lettuce. The scope of this validation included the analysis of control crops, control crops fortified with either mefenoxam or metalaxyl, and crop samples containing field-incurred residues of metalaxyl or mefenoxam from side-by-side applications. The samples of tobacco, tomatoes, cabbage, and potatoes used to validate this methodology were obtained from excess sample inventories from magnitude of residue field trials that were previously conducted by Novartis Crop Protection[8,9,10,11]. Detailed information regarding the source and treatment history of these crop samples is provided in Table 2 and in the Residue Test Reports. The ^{14}C-mefenoxam and ^{14}C-metalaxyl treated lettuce samples were obtained from excess sample from Novartis Crop Protection AG Metabolism Study 98JS30 (in-progress).

A summary of validation results, as well as all LC/MS sample chromatograms and raw data worksheets, are included in the body of this method.

For the purpose of consistency, the following terminology will be used to describe the procedures and results presented in this method:

- The term **(RS) racemate** will be used to describe test and reference substances containing metalaxyl, which is a racemic mixture of the (S) and (R) enantiomers. In many of the result tables, the sum of the individual results for the (S) and (R) enantiomers is used to express the result in terms of its metalaxyl equivalent. This term may occasionally be abbreviated as 'RS'.
- The term **(R) enantiomer** will be used to describe test and reference substances containing mefenoxam, which contains primarily the biologically active (R) enantiomer with a trace amount of the biologically inactive (S) enantiomer. This term may be abbreviated as 'R'.
- The term **(S) enantiomer** will be used to describe results obtained for the biologically inactive (S) enantiomer. The (S) enantiomer is present at approximately 50% by weight in metalaxyl, and at trace levels in mefenoxam. This term may be abbreviated as 'S'.

B. PRINCIPLE

A 10-gram crop sample is extracted by refluxing with 100 mL of 80:20 methanol/water for two hours*. A 25-mL aliquot is taken and passed directly through a C18 solid phase extraction cartridge for removal of co-extractives.

The sample is reduced to aqueous, acidified with 0.10N HCl, diluted with water, and partitioned into dichloromethane (DCM). The DCM extract is loaded onto a silica solid phase extraction cartridge. The cartridge is washed with 5% (v/v) diethyl ether in hexane. The analytes are eluted off of the cartridge with 40% (v/v) ethyl acetate in hexane

The ethyl acetate/hexane extract is evaporated to dryness, reconstituted in mobile phase, and if necessary, filtered through a 0.45 µm micro-filter. Analyte determination is made through the use of chiral high performance liquid chromatography followed by mass spectrometric detection (LC/MS).

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* This extraction procedure is identical to that of Analytical Method AG-395, which is the currently accepted tolerance enforcement method for total residues of metalaxyl as 2,6-dimethylaniline. This allows both the total and parent determinations to be made from a single crop extract.

A chiral reverse-phase HPLC system is used for separation of the metalaxyl enantiomers. The reverse phase column is a Chiralcel® OD-R (Chiral Technologies, Inc.), with a mobile phase of 50:50:0.05 (v:v:v) acetonitrile:water:trifluoroacetic acid. Detection is accomplished by monitoring the [M+1] molecular ion (m/z 280) with a mass spectrometer operated in positive-ion, electrospray ionization mode (ES+).

A flowchart of these analytical procedures is presented in Figure 2.

II. MATERIALS AND METHODS

A. APPARATUS/EQUIPMENT

1.0 Glass syringe with LuerLok tip, 20 mL, Becton Dickinson Multifit #2443 (Fisher #14-823-10D, or equivalent)

2.0 Boiling flasks, flat bottom, 125-mL (Fisher #09-552A, or equivalent)

3.0 Rotary evaporator, Buchii or equivalent, with a variable temperature water bath (Buchler Instruments, or equivalent), and aspirator pump (Cole-Parmer, or equivalent). The use of a bump trap is recommended (Fisher #K570200-2524, or equivalent)

4.0 Ultrasonic bath (Fisher Model FS-14, or equivalent)

5.0 Funnel, separatory, 60-mL (Fisher #10-437A, or equivalent)

6.0 Funnels, filtering, 75-mm and 100-mm diameter (Fisher #10-329C, #10-329D, or equivalent)

7.0 Glass wool, Pyrex, Corning #3950 (Fisher #11-388, or equivalent)

8.0 Solid Phase Extraction, C18 SepPak cartridge, 360 mg, (Waters P/N# WAT051910)

9.0 Solid Phase Extraction, Silica SepPak cartridge, 690 mg, (Waters P/N# WAT051900)

10.0 Disposable plastic syringe with LuerLok tip, 3 cc, (Becton Dickinson #9585, or equivalent)

11.0 Syringe filters, 0.45 µm, ,Gelman LC-PVDF Acrodisc #4452, (Fisher #09-730-134, or equivalent)

12.0 Pipettes, volumetric class A, 1-mL, 2-mL, other sizes as needed

13.0 Graduated cylinders, 10-mL, 50-mL, 100-mL, 250-mL, other sizes as needed

14.0 Volumetric flasks, class A, 100-mL, other sizes as needed

15.0 Filter paper, 24-cm, Whatman 2V 1202-240 (Fisher #09-832D, or equivalent)

16.0 Variable power transformer (Powerstat, or equivalent)

17.0 Flask, round bottom, Pyrex, 500-mL or 250-mL (Fisher #10-067G and #10-067F, or equivalent)

18.0 Condenser, Allihn, 500-mm (Fisher #07-735C, or equivalent)

19.0 Heating mantle, 500-mL or 250-mL (Glascol, or equivalent)

20.0 Concentration Tubes, conical-bottom 50-mL capacity (Fisher #05-507-5C, or equivalent)

21.0 Bottles, Boston round amber, 4-oz., with screw cap

22.0 Pasteur pipettes (Kimble 13-678-30B, 13-678-30C, or equivalent)

23.0 Autosampler vials, crimp top borosilicate (Sun Broker, Inc., or equivalent).

24.0 Scintillation vials, 20 mL, with screw cap (Kimble Glass Co., Vineland, NJ, or equivalent).

B. REAGENTS

1.0 Acetonitrile, HPLC grade (Fisher #A998-4, or equivalent)

2.0 Ethyl acetate, HPLC grade (Fisher #E195-4, or equivalent)

3.0 Ethyl ether, HPLC grade (Fisher #E138-1, or equivalent)

4.0 Hexane, HPLC grade (Fisher #H302-4, or equivalent)

5.0 Methanol, HPLC grade (Fisher # A452-4, or equivalent)

6.0 Dichloromethane, Pesticide Residue Analysis grade (Burdick and Jackson #300-4, or equivalent)

7.0 Trifluoroacetic acid, reagent grade (Fisher #04902-100, or equivalent)

8.0 Hydrochloric acid, Certified ACS grade (Fisher #A144-212, or equivalent)

9.0 Sodium sulfate, anhydrous, Certified ACS grade (Fisher #S421-3, or equivalent)

10.0 Water, HPLC grade (Picopure, or equivalent)

11.0 0.10N HCl - Prepare 1 liter by diluting 8.25 mL hydrochloric acid to 1000-mL with HPLC grade water.

12.0 5% Ethyl ether in hexane (v/v) - Prepare 1 liter by combining 50 mL of ethyl ether with 950 mL of hexane.

13.0 40% Ethyl acetate in hexane (v/v) - Prepare 1 liter by combining 400 mL of ethyl acetate with 600 mL of hexane.

14.0 Acetonitrile:water:TFA 50:50:0.05 (v/v/v) mobile phase - Prepare ~1 liter by adding 500 mL of acetonitrile to 500 mL of water. Add 0.50 mL of trifluoroacetic acid.

15.0 CGA-48988, analytical standard supplied by Analytical and Product Chemistry Department, Novartis Crop Protection, 410 Swing Rd., Greensboro, NC.

16.0 CGA-329351, analytical standard supplied by Analytical and Product Chemistry Department, Novartis Crop Protection, 410 Swing Rd., Greensboro, NC.

17.0 ScintiSafe Gel™ scintillation cocktail (Fisher #SX24-5, or equivalent)

C. ANALYTICAL PROCEDURE

1.0 Extraction

1.1 Sample Preparation

Crop substrates were prepared using the procedures outlined in the Novartis Product Safety Group SOP 7.21,

Revision 2[12]. The crop samples analyzed in this study were prepared as follows:

Cabbage, Tobacco, Tomato Foliage, and Potato Foliage
Decomposed leaves were removed and discarded. Heads were quartered. Samples were composited, and chopped in a Hobart food cutter, using dry ice as necessary to keep the sample frozen. Prepared samples were stored in polyethylene bags or bottles.

Tomato Fruit
Decomposed leaves and stems were removed and discarded, and lightly adhering soil was brushed away. Fruits were halved or quartered, composited, and chopped in a Hobart food cutter, using dry ice as necessary to keep the sample frozen. Prepared samples were stored in polyethylene bottles.

Lettuce
The procedures used for the preparation of the ^{14}C-treated lettuce samples are described in the final report for Novartis Study 98JS30[6] (in-progress). Briefly, frozen samples are manually cut into pieces and further macerated under liquid nitrogen using mechanical cutters.

1.2 Extraction of Substrates

Weigh a 10-g representative sub-sample of a homogenized crop sample into a 500-mL round bottom flask. Add 100 mL of 80:20 (v/v) methanol:water and a few boiling chips. Place the flask in a 500-mL heating mantle, attach a water-cooled Allihn condenser to the flask, and reflux for two hours. Allow the solution to cool to room temperature and filter through a Whatman 2V filter paper into a 4-oz. amber bottle.

2.0 Solid Phase Extraction: C18 SepPak Cartridge

2.1 Column Conditioning

Attach a C18 SepPak cartridge (360 mg) to the tip of a 20-mL LuerLok syringe with the plunger removed. Condition the syringe and cartridge assembly with 5 mL of 80:20 (v/v) methanol:water. Allow the methanol:water to drain through the cartridge. Discard the eluate, but do not

allow the C18 cartridge to go dry before performing the next step.

2.2 Sample Addition and Collection

Place a 125-mL boiling flask below the syringe and cartridge assembly and load 25 mL of sample extract to the syringe, collecting the eluate.

Refer also to Section II.H.1, "MODIFICATIONS AND POTENTIAL PROBLEMS", for a discussion of extract flow rate through the C18 cartridge.

3.0 Liquid-Liquid Partition of Extract

3.1 Reduce the sample volume to approximately 5 mL on a rotary evaporator with a water bath maintained at approximately 35°C.

3.2 Add 25 mL of HPLC grade water to the 125-mL boiling flask and 1.0 mL of 0.10N hydrochloric acid. Swirl to mix, using an ultrasonic bath as necessary to loosen any residue that might adhere to the walls of the boiling flask. Carefully transfer the aqueous sample to a 60-mL separatory funnel.

3.3 Add 10 mL of dichloromethane (DCM) to the 125-mL boiling flask, stopper, and shake well. Transfer the DCM rinsate to the 60-mL separatory funnel.

3.4 Stopper the separatory funnel and shake vigorously for 30 seconds. Allow the layers to separate for at least 2 minutes.

3.5 Plug the stem of a 75-mm filtering funnel with a small amount of glass wool. Add approximately 10 grams of anhydrous sodium sulfate to the funnel. Pre-rinse this apparatus with 15-mL of DCM, discarding the rinsate.

3.6 Drain the DCM extract through the funnel containing the pre-rinsed sodium sulfate pad and collect into a 125-mL boiling flask. Minimize the transfer of any water droplets to the sodium sulfate pad.

3.7 Repeat the DCM partition as in steps 3.3 through 3.6 three times, draining each extract through the sodium sulfate pad, for a total of four DCM partitions.

3.8 After the fourth DCM partition, rinse the sodium sulfate pad with an additional 15 mL of DCM and collect.

3.9 Evaporate the DCM extract to dryness and reconstitute with approximately 5 mL of DCM.

4.0 Solid Phase Extraction: Silica SepPak Cartridge

4.1 Column Conditioning

Attach a Silica SepPak cartridge (690 mg) to the tip of a 20-mL LuerLok syringe with the plunger removed. Condition the syringe and cartridge assembly with 10 mL of DCM.

- Do not allow the silica cartridge to run dry at any point during this procedure.
- The flow rate through the silica cartridge should be about 1 drop per second.
- Do not assist the solvent flow through the silica cartridge by applying air pressure with a rubber bulb.

4.2 Sample Addition

Transfer the sample from Section II.C.3.9 from the 125-mL boiling flask to the syringe and cartridge assembly using a Pasteur pipette. Rinse the 125-mL boiling flask with 10 mL of DCM and add to the syringe and cartridge assembly. Allow the sample and rinsate to elute to the top of the cartridge. Discard the load eluate.

4.3 Column Wash

Add 10 mL of 5% ethyl ether:hexane to the 125-mL boiling flask. Rinse the flask thoroughly and transfer the ether/hexane rinsate to the syringe and cartridge assembly. Allow the ether:hexane to elute to the top of the cartridge. Discard the rinse eluate.

4.4 Sample Elution

Add 20 mL of 40% ethyl acetate:hexane to the 125-mL boiling flask and swirl; transfer this elution solvent to the syringe and cartridge assembly. Collect the entire eluate into a 50-mL concentration tube.

5.0 Preparation of Final Sample

5.1 Evaporate the ethyl acetate:hexane sample from Section II.C.4.4 to dryness using a vacuum rotary evaporator set at ~35°C (fitted with a bump trap). Reconstitute the samples to the desired final volume with mobile phase (50:50:0.05 ACN:H_2O:TFA) using a volumetric pipette. Sonicate or shake the samples thoroughly to re-dissolve any sample residue from the sides of the concentration tube.

A 4-mL final volume is used for all samples screened at the method LOQ of 0.01 ppm of the (RS) racemate (0.005 ppm of the (R) enantiomer plus 0.005 ppm of the (S) enantiomer). The volume may be adjusted as appropriate to bring the residue concentrations within the range of the standards.

Refer also to Section II.H.2, "MODIFICATIONS AND POTENTIAL PROBLEMS", if any insoluble particulate material is visible in the final sample extract

After transfer to an autosampler vial, the sample is ready for analysis by chiral LC/MS.

D. INSTRUMENTATION

1.0 Description and Operating Conditions

1.1 Reverse Phase Chiral HPLC

Detection of residues is performed with a chiral reverse-phase HPLC column system. The analytical column is a Chiralcel® OD-R (Chiral Technologies, Inc., Exton, PA), with a mobile phase of 50:50:0.05 ACN:H_2O:TFA, operated at a flow rate of 0.50 mL/minute.

Detection is accomplished by monitoring the [M+1] molecular ion at m/z=280 with a mass spectrometer operated in positive-ion, electrospray ionization mode (ES+). Complete operating conditions for the LC/MS system are provided in Table 1.

The run time for this system to elute both enantiomers is approximately 25 minutes; the (S) enantiomer is retained for approximately 10.8 minutes, and the (R) enantiomer is retained for approximately 16.1 minutes. The run time may be increased appropriately if late eluting peaks are observed.

Refer also to Section II.H.3, "MODIFICATIONS AND POTENTIAL PROBLEMS", for a discussion of the use of the gradient elution to mitigate the effects of late eluting peaks.

2.0 Standardization

Standardize the LC/MS system by injecting 75-µL aliquots of standard solutions containing the (R) and (S) enantiomers in the range of 0.1875 ng to 75.00 ng per injection. Generate a calibration curve by comparing detector response versus ng injected.

See Section II.I.1.0 for preparation of analytical standards. It is necessary to run a set of standards with each analytical set to achieve accurate quantitation of residues.

E. INTERFERENCES

The use of chiral chromatography, in conjunction with mass spectrometric detection, provides a highly selective and specific technique for the analysis of the (S) and (R) enantiomers. No interferences were encountered during the validation of this method.

F. CONFIRMATORY TECHNIQUE

This method is a complementary technique to Analytical Method AG-395, which is the currently accepted US EPA tolerance enforcement method for total residues of metalaxyl or mefenoxam, determined as 2,6-dimethylaniline. The procedures described in this method are used to confirm the presence or absence of either parent metalaxyl or parent mefenoxam in a crop sample.

G. TIME REQUIRED

The extraction and cleanup of a set of 6 samples may be completed within a time period of approximately 4-6 hours. A complete LC/MS analysis sequence consisting of six samples and six calibration standards may take an additional 6 hours when using an automated injection system.

H. MODIFICATIONS AND POTENTIAL PROBLEMS

1. Sample Addition and Collection/C18 (Section II.C.2.2): The flow rate through the cartridge may slow considerably as the C18 sorbent bed begins to swell from the solvent and retain particulate matter from the extract. It may take more than 30 minutes for the entire sample extract to pass through the C18 cartridge. Flow rate through the cartridge may be increased, if desired, by applying light to moderate air pressure to the syringe barrel with a rubber laboratory squeeze bulb. The flow rate should be kept to less than approximately 1 mL per second. The application of excessive air pressure to the syringe barrel may result in extracts that have not been cleaned up sufficiently to permit successful analysis by LC/MS. After all of the extract has passed through the cartridge, apply a final charge of air pressure to the syringe barrel to recover any liquid extract retained by the void volume of the sorbent bed.

2. Preparation of Final Samples (Section II.C.5.1): If any insoluble particulate material is visible in the final sample extract, attach a 0.45 µm syringe filter to a 3-cc disposable syringe with the plunger removed. Pour the sample extract from Section II.C.5.1 into the syringe barrel, insert the plunger, and gently force the sample through the micro-filter directly into an autosampler vial. Analyze by LC/MS.

3. Reverse Phase Chiral HPLC (Section II.D.1.1): Late-eluting peaks from the HPLC analytical column may interfere with linear response of the mass spectrometer. This effect can be mitigated by using a gradient elution, as described in Table 1. This approach may improve analytical results if chromatographic or mass spectral interferences are encountered when analyzing test substrates other than those investigated during this validation.

I. PREPARATION OF STANDARD SOLUTIONS AND SAMPLE FORTIFICATION PROCEDURES

The **metalaxyl reference material**, which is a 50% plus 50% mixture of the (S) and the (R) enantiomers, is used in this method to calibrate the LC/MS measurement system. This approach is preferable to calibrating with reference standards of the (R) enantiomer and the (S) enantiomer

prepared independently, since individually prepared reference standards would contain predominantly one enantiomer with trace amounts of the other, which unnecessarily complicates the calibration process. Small variations from the assumed 50/50 enantiomeric distribution do not significantly introduce error into the results obtained. Furthermore, enantiomeric purity of the metalaxyl reference material is information that is not readily available in most cases.

When using metalaxyl to prepare a calibration curve, the nominal concentration of the metalaxyl calibration standard is corrected for enantiomeric purity, which is assumed to be 50%, to obtain concentrations for each of the (R)- and (S)- enantiomers. For example, a metalaxyl calibration standard that is prepared at 2.00 ng/µL is entered into the LC/MS or other data acquisition system as 1.00 ng/µL of the (S) enantiomer plus 1.00 ng/µL of the (R) enantiomer. The same is true for procedural recovery samples that are fortified with metalaxyl. For example, a control sample fortified with 1.00 ppm of metalaxyl is input into the data system as 0.50 ppm of the (S) enantiomer plus 0.50 ppm of the (R) enantiomer. Consequently, the result obtained from the data system will be expressed in terms of the individual isomers. To express the results in terms of the (RS) racemate (metalaxyl), the results of the individual enantiomers, in ppm, are added together.

The **mefenoxam reference material** used in this method consists primarily of the (R) enantiomer with a trace amount of the (S) enantiomer. This solution is used only for the fortification of procedural recoveries; residual amounts of the (S) enantiomer, if present, will be accounted for when calculating the percent enantiomeric excess (EE%) in Section II.J.3.0.

1.0 Preparation of Fortification and Analytical Standards

1.1 Preparation of Metalaxyl Analytical Standards

Weigh 100.0 mg of metalaxyl, corrected for chemical purity, into a 100-mL volumetric flask and dilute to the mark with methanol. This standard contains 1,000 ng/µL of the (RS) racemate. This solution should be corrected for enantiomeric purity, which is assumed to be 50% of the (S) enantiomer plus 50% of the (R) enantiomer. This provides a mixed reference standard equivalent to approximately 500 ng/µL of the (S) enantiomer plus 500 ng/µL of the (R) enantiomer.

Make serial dilutions of this stock solution with 50:50:0.05 ACN:H_2O:TFA mobile phase solution to give a series of analytical standards in a range of 0.0050 ng/µL to 2.00 ng/µL of the (RS) racemate, equivalent to 0.0025 ng/µL* to 1.00 ng/µL of each individual (R) or (S) enantiomer.

Store these standard solutions in a refrigerator in a capped amber bottle when not in use.

1.2 Preparation of Mefenoxam Analytical Standard

Weigh 50.0 mg of mefenoxam, corrected for chemical purity, into a 100-mL volumetric flask and dilute to the mark with methanol. This provides a reference standard equivalent to approximately 500 ng/µL of the (R) enantiomer, assuming enantiomeric purity of 100%.

Store this standard solution in a refrigerator in a capped amber bottle when not in use.

1.3 Preparation of Fortification Standards

Metalaxyl 10.0 ng/µL Fortification Standard - Add 1.0 mL of the 1,000 ng/µL metalaxyl stock standard solution to a 100-mL volumetric flask and dilute to the mark with methanol to give a 10.0 ng/µL metalaxyl fortification solution (5.0 ng/µL of the (S) enantiomer plus 5.0 ng/µL of the (R) enantiomer).

Metalaxyl 0.10 ng/µL Fortification Standard - Add 1.0 mL of the 10.0 ng/µL metalaxyl fortification solution to a 100-mL volumetric flask and dilute to the mark with methanol to give a 0.10 ng/µL metalaxyl fortification solution (0.05 ng/µL of the (S) enantiomer plus 0.05 ng/µL of the (R) enantiomer).

Mefenoxam 5.00 ng/µL Fortification Standard - Add 1.0 mL of the 500 ng/µL mefenoxam stock standard solution to a 100-mL volumetric flask and dilute to the

* The lowest reference standard typically used for calibration of the analytical instrument was prepared at 0.0050 ng/µL of the (RS) racemate, equivalent to 0.0025 ng/µL of each enantiomer. However, a calibration standard prepared at 0.0030 ng/µL of the (RS) racemate, equivalent to 0.0015 ng/µL of each enantiomer, was run with the 7-day PHI cabbage wrapper leaves (injected 29-Dec-1998). This standard exhibited acceptable signal-to-noise ratio and linearity.

mark with methanol to give a 5.00 ng/µL mefenoxam fortification solution.

Mefenoxam 0.05 ng/µL Fortification Standard - Add 1.0 mL of the 5.00 ng/µL mefenoxam fortification solution to a 100-mL volumetric flask and dilute to the mark with methanol to give a 0.05 ng/µL mefenoxam fortification solution.

Store these fortification solutions in a refrigerator in capped amber bottles when not in use.

2.0 Sample Fortification Procedures

Metalaxyl Fortifications at 1.00 ppm: Add 1.0 mL of 10.0 ng/µL metalaxyl fortification solution to 10 grams of crop substrate to obtain a sample fortified at 1.00 ppm of metalaxyl, equivalent to 0.50 ppm of the (R) enantiomer, plus 0.50 ppm of the (S) enantiomer.

Metalaxyl Fortifications at 0.01 ppm: Add 1.0 mL of 0.10 ng/µL metalaxyl fortification solution to 10 grams of crop substrate to obtain a sample fortified at 0.01 ppm of metalaxyl, equivalent to 0.005 ppm of the (R) enantiomer, plus 0.005 ppm of the (S) enantiomer.

Mefenoxam Fortifications at 0.50 ppm: Add 1.0 mL of 5.00 ng/µL mefenoxam fortification solution to 10 grams of crop substrate to obtain a sample fortified at 0.50 ppm of the (R) enantiomer.

Mefenoxam Fortifications at 0.005 ppm: Add 1.0 mL of 0.05 ng/µL mefenoxam fortification solution to 10 grams of crop substrate to obtain a sample fortified at 0.005 ppm of the (R) enantiomer.

Other volumes and standard concentrations may be used for fortification, although the total volume should not exceed 2 mL.

J. METHODS OF CALCULATION

1.0 Determination of Sample Residues

Inject 75-µL aliquots of the final extract fractions prepared in Section II.C.5.1 into the LC/MS system under the same conditions as for the standards. Develop a calibration curve on the LC/MS data system that plots the peak area of the

abundance of the [m/z 280] positive ion against the amount of (R)- or (S)- enantiomer reference material injected to determine the nanograms of (R)- or (S)- enantiomer in the injected sample extract.

To calculate the residue results, the mg injected must first be calculated as follows:

$$(1)\ \text{mg inj.} = \frac{(G)\,(V_a)\,(V_i)}{(V_e + W\,(M/100))\,(V_f)}$$

G = amount of sample extracted (mg)
V_a = aliquot volume (mL)
V_e = extraction volume (mL)
V_i = injection volume (mL)
V_f = volume of final injection solution (mL)
M = % moisture of sample
W = weight of sample (g)

To determine the corrected ppm of each analyte in the samples, use equation 2:

$$(2)\ \text{ppm} = \frac{(\text{ng analyte found}) \times 100}{(\text{mg sample injected})\,(R\%)}$$

R% = recovery factor given by equation (5)

2.0 Determination of Procedural Recoveries

2.1 Determine the ng of the (R) or (S) enantiomer in the aliquot of the samples injected, and calculate the mg injected using the procedures described in Section II.J.1.0. Calculate the final ppm values of the control and fortified samples according to the following equation:

$$(4)\ \text{ppm} = \frac{\text{ng analyte found}}{\text{mg sample injected}}$$

Determine the recovery factor by first subtracting the background detector response, if any, in the control sample from the analyte response in the recovery sample. Calculate the recovery factor as a percentage (R%) by the equation:

$$(5)\ R\% = \frac{(\text{ppm analyte found} - \text{ppm control})}{\text{ppm analyte added}} \times 100$$

Note: Analysis of controls, correction of recovery results for small control values, and use of the recovery factor is not required for tolerance enforcement purposes. They are used for method validation and generation of residue data where applicable.

3.0 Determination of Percent Enantiomeric Excess (EE%)

The Percent Enantiomeric Excess (EE%) parameter provides the analyst with a measurement of the relative amounts of the (R) enantiomer and the (S) enantiomer in the crop sample. The EE% can be calculated after determination of the parts per million of each enantiomer in the crop sample as follows:

$$EE\% = \frac{[R-S]}{[R+S]} \times 100$$

R = Amount of (R) enantiomer found in sample, ppm
S = Amount of (S) enantiomer found in sample, ppm

The EE% parameter can assist the analyst in making a determination as to whether a given crop was treated with a commercially available formulation containing either metalaxyl or mefenoxam. Samples that contain close to the theoretical enantiomeric ratios would produce EE% values similar to those shown in the following example:

Example of EE% Calculations Using Theoretical Enantiomeric Ratios Found in Commercial Formulations Containing Either Mefenoxam or Metalaxyl

Approximate EE%	Amount of each enantiomer found (ppm)		Formulation Used for Treatment
	(R) enantiomer	(S) enantiomer	
+100%	0.50 ppm	<0.005 ppm	mefenoxam
0%	0.50 ppm	0.50 ppm	metalaxyl
-100%	<0.005 ppm	0.50 ppm	- -

Complete results of the EE% calculation for samples containing incurred residues are presented in Table 4. Refer also to Section III.A, "ACCURACY", for a summary of the EE% data.

4.0 Determination of Treatment with Mefenoxam

Treatment with mefenoxam is inferred by noting the relative absence of a chromatographic peak at the (S) enantiomer retention time window, while confirming the presence of a significant peak at the (R) enantiomer retention time window. Refer also to Section III.E, "ENANTIOMERIC EXCESS".

5.0 Determination of Treatment with Metalaxyl

Treatment with metalaxyl is inferred by the presence of significant and approximately equal-sized peaks at both the (S) enantiomer and (R) enantiomer retention time windows. Refer also to Section III.E, "ENANTIOMERIC EXCESS".

6.0 Determination of ^{14}C-Labeled Metalaxyl and Mefenoxam

Sample extracts were radioassayed by aliquoting an appropriate volume into a 20-ml scintillation vial and adding ~15 ml of Fisher ScintiSafe Gel™ scintillation cocktail. Radioactivity (dpm) was quantitated using a Beckman liquid scintillation counter (LSC). The radioactivity per gram of sample was calculated as follows:

$$\text{dpm per sample gram} = \frac{\text{radioactivity (dpm)} \times \text{total extract volume (mL)}}{\text{LSC aliquot volume (mL)} \times \text{total sample weight (g)}}$$

The radioactivity in the sample final fraction was also measured by LSC analysis. The total radioactivity in the sample extracts was calculated according to the following equation:

$$^{14}\text{C residue, ppm} = \frac{\text{dpm per sample gram}}{\text{specific activity } (\mu\text{Ci/mg}) \times \left(2.22 \times 10^{6}\,\text{dpm}/\mu\text{Ci}\right)} \times \frac{1000\ \mu\text{g}}{\text{mg}}$$

The lettuce samples used in this study were generated under Novartis Study 98JS30[6], and were treated with radiolabeled mefenoxam and metalaxyl. The specific activities of the test substances were: 2.54 MBq/mg for the ^{14}C-mefenoxam, and 2.10 MBq/mg for the ^{14}C-metalaxyl (equivalent to 68.65 µCi/mg for the ^{14}C-mefenoxam, and 56.76 µCi/mg for the ^{14}C-metalaxyl). The specific activities were converted between measurement units as follows:

$$\text{specific activity, } \mu\text{Ci/mg} = \frac{\text{specific activity MBq}}{\text{mg}} \times \frac{27.02703\ \mu\text{Ci}}{\text{MBq}}$$

III. RESULTS AND DISCUSSION

The objective of this study was to evaluate the precision, accuracy, extractability, and accountability of this analytical method for the determination of residues of the (R) and (S) enantiomers of metalaxyl or mefenoxam in crops. Additionally, this study investigated whether the Percent Enantiomeric Excess parameter is a suitable means of identifying whether a crop was treated with either metalaxyl or mefenoxam.

This report contains all experimental data collected during the conduct of this study. A summary of the results of analysis of procedural recovery samples is presented in Table 3. A summary of the results of analysis of samples containing field-incurred residues of either metalaxyl or mefenoxam are presented in Table 4. A summary of the results of analyses to determine extractability and accountability are presented in Table 5.

All Raw Data Worksheets used for calculation of sample results are presented in Tables 6 through 31. Included in the Raw Data Worksheets are sample weights, extraction volumes, aliquot volumes, final volumes, injection amounts, fortification levels, calibration standards, nanograms of analyte found, and uncorrected and corrected sample results (ppm).

The LC/MS chromatograms for every tobacco, tomato, cabbage, potato, and lettuce sample analyzed in this study are shown in Figures 3-15. In these chromatograms, each injection or sample is illustrated by two ion chromatograms: one showing the (S) enantiomer and one showing the (R) enantiomer. In each chromatogram, the peak of interest is highlighted with an arrow and labeled with the retention time and response (in peak area). Each set of chromatograms in Figures 3-15 contains the following:

- one chromatogram of a calibration standard (with the output from the linear regression)
- one chromatogram of an untreated control substrate
- one chromatogram of an untreated control (procedural recovery) fortified with metalaxyl
- one chromatogram of an untreated control (procedural recovery) fortified with mefenoxam
- at least two chromatograms of samples containing field-incurred residues of metalaxyl
- at least two chromatograms of samples containing field-incurred residues of mefenoxam

Figure 16 shows a typical set of calibration standards in the range of 0.0025 ng/µL to 1.00 ng/µL (0.1875 ng to 75.00 ng injected) of the (S) and (R) enantiomers. Figure 17 shows the calibration curves for the (S) and (R) enantiomers obtained from the ion chromatograms of the standards in Figure 16. Figure 18 is a graphical presentation of the EE% values calculated from the analysis of samples containing incurred residues of either metalaxyl of mefenoxam.

A. ACCURACY

Untreated tobacco, tomato, cabbage, potato, and lettuce substrates were fortified with either metalaxyl (CGA-48988) or mefenoxam (CGA-329351) at levels of 0.005 ppm or 0.500 ppm. Pooled procedural recovery results from fortified control samples were used to calculate accuracy in terms of a mean, standard deviation (sd), and coefficient of variation (CV% = sd/mean x 100). Procedural recovery data from fortified control samples from the validation sets analyzed herein (Table 3) were compiled to assess the overall accuracy of Analytical Method 456-98.

Procedural recoveries were calculated by dividing the concentration of either the (S) enantiomer or the (R) enantiomer measured in each fortified sample by the concentration added to the sample. Substrate, fortification levels, and residues in the fortified controls are presented in Table 3 and are summarized below:

Statistical Summary for Procedural Recovery Samples Fortified with Metalaxyl

(R) Enantiomer		(S) Enantiomer	
Minimum =	65%	Minimum =	74%
Maximum =	116%	Maximum =	113%
Average =	98%	Average =	98%
SD =	12.5%	SD =	10.4%
%CV =	12.7%	%CV =	10.7%
n =	13	n =	13

Statistical Summary for Procedural Recovery Samples Fortified with Mefenoxam

(R) Enantiomer	
Minimum =	75%
Maximum =	117%
Average =	98%
SD =	9.3%
%CV =	9.5%
n =	13

The acceptable procedural recovery results in Table 3 confirm that the analytical procedure described in Analytical Method 456-98 is an accurate method for the quantitative determination of residues of the (R) and/or (S) enantiomers.

No significant interferences (>0.005 ppm) were found at or near the retention time of either the (S) enantiomer or the (R) enantiomer in the control substrates*.

The data presented in Table 4 are the results of the use of this method to analyze crop samples containing field-incurred residues of either metalaxyl or mefenoxam. Table 4 lists the result for each sample reported as "ppm (S) enantiomer", "ppm (R) enantiomer", and as the equivalent "ppm (RS) racemate". Refer also to Section III.E, "ENANTIOMERIC EXCESS", for a discussion of the performance of this

* Tomato foliage did exhibit detections of both the (S) and (R) enantiomers in the untreated control at approximately 0.02 ppm as the (RS) racemate. The approximately equal amounts of the (S) and (R) enantiomers in the control sample (EE% = +5%) indicate that the sample contains traces of parent metalaxyl. These detections were insignificant relative to the levels of metalaxyl (~7 ppm) and mefenoxam (~3 ppm) determined in the field treated samples, and will not impact the conclusions of the study.

method for its intended use in determining the source of incurred residues.

B. PRECISION

Precision of this method was determined by calculating the mean, standard deviation, and coefficient of variation of results from the analysis of fortified control samples. Table 3 presents the statistical results for recovery values from all fortification experiments; the %CV results show that this method demonstrates adequate analytical precision.

C. EXTRACTABILITY

The extractability of the total radioactive residue (TRR) by Analytical Method 456-98 was determined by comparing the ppm of ^{14}C-residue in the method's initial sample extract (as measured by liquid scintillation counting) to the total ppm of ^{14}C-residue in the original crop sample (as measured by combustion analysis). The formula for the determination of percent extractability is as follows:

$$\text{percent extractability} = \frac{\text{total } ^{14}\text{C residue (ppm) in sample extract by LSC}}{\text{TRR in crop by combustion analysis (ppm)}} \times 100$$

Table 5 summarizes TRR values, ^{14}C-residues in sample extracts, and percent extractability. The TRR values were determined in Novartis Metabolism Study 98JS30. The concentration of the ^{14}C-extractable residue was calculated by LSC analyses of the extracts and is expressed in ppm of the (RS) racemate equivalents. The total extractabilities were 125%, 78% and 97% for the 0-day, 14-day, and 21-day PHI lettuce treated with ^{14}C-mefenoxam, and 106%, 109%, and 112% for the 0-day, 14-day, and 21-day PHI lettuce treated with ^{14}C-metalaxyl, respectively.

D. ACCOUNTABILITY

The accountability of Analytical Method 456-98 is determined by comparing the ppm of ^{14}C-residue in the method's residue in the final extract (as determined by LC/MS analysis) to the total ppm of ^{14}C-residue in the crop sample (as measured by combustion analysis).

The formula for the determination of percent accountability is as follows:

$$\text{percent accountability} = \frac{\text{parent residue (ppm) in sample extract by LC / MS}}{\text{TRR in crop by combustion analysis (ppm)}} \times 100$$

The total accountabilities were 75%, 6%, and 3% for the 0-day, 14-day, and 21-day PHI lettuce treated with ^{14}C-mefenoxam, and 75%, 14%, and 7% for the 0-day, 14-day, and 21-day PHI lettuce treated with ^{14}C-metalaxyl, respectively. These accountability results show a decline in parent residues of the (R) and (S) enantiomers as a function of increasing PHI, which is consistent with the accountability of this method as calculated from the results of 2-dimensional thin layer chromatography performed in Novartis Metabolism Study 98JS30. Accountability results for this method are summarized in Table 5.

E. ENANTIOMERIC EXCESS

The validity of using the enantiomeric excess (EE%) parameter as a means of determining whether a given crop was treated with either metalaxyl or mefenoxam was investigated as part of this study.

Table 4 contains the results of the EE% calculations that were derived from samples containing field-incurred residues of either metalaxyl or mefenoxam. This data is summarized below:

Summary of Percent Enantiomeric Excess Results Obtained from the Analysis of Crops Containing Field-Incurred Residues of Metalaxyl or Mefenoxam

Treatment Substance:	Metalaxyl	Mefenoxam
Range of Results =	-56% to +20%	+43% to +100%
Mean =	-8%	+91%
Standard Deviation =	± 18.4%	± 14.0%
n =	29	29

A graphical representation of the distribution of all EE% results for the samples analyzed is presented in Figure 18.

The highest EE% obtained in this study for a crop treated with metalaxyl was +20% (mature tomato fruit, 0.025 ppm); the lowest EE% for a crop treated with mefenoxam was +43% (mature tobacco leaf, 0.007 ppm). There were no instances where any sample treated with metalaxyl had a higher EE% than any sample treated with mefenoxam.

These data, generated through the analysis of samples containing incurred residues, demonstrate that the EE% measurement is a reliable and unbiased means of determining if incurred residues can be attributed to applications made with a formulation containing either metalaxyl or mefenoxam.

IV. CONCLUSION

Novartis Analytical Method 456-98 is demonstrated to be a valid method for the determination of residues of parent metalaxyl or mefenoxam, determined as the sum of the (S) and (R) enantiomers, in tobacco, tomato, cabbage, potato, and lettuce.

The validity of this method has been demonstrated by acceptable precision and accuracy results obtained for procedural recovery samples, and by the extractability and accountability results obtained by the analysis of weathered radioactive substrates using this method.

The percent enantiomeric excess (EE%) parameter allows a reliable and unbiased determination to be made as to whether a crop was treated with a formulation containing either metalaxyl or mefenoxam as its active ingredient.

V. TABLES AND FIGURES

TABLE 1: OPERATING PARAMETERS FOR THE ENANTIOSELECTIVE DETERMINATION OF METALAXYL OR MEFENOXAM BY CHIRAL REVERSE PHASE LC/MS

Sample Introduction: Hewlett-Packard Model 1090 High Performance Liquid Chromatograph (HPLC) system, with autosampler, quaternary solvent gradient elution pump, and fixed wavelength UV detector.

Detector: Micromass Platform™ Mass Spectrometer with electrospray ionization (EI)/atmospheric pressure chemical ionization (APCI) interface. Data processed using Masslynx™ software (Rev. 3.1 Build 4).

HPLC Column: Chiralcel™ OD-R, 10 µm packing, 250 mm × 4.6 mm, (Chiral Technologies, Exton, PA).

Mobile phase: 50:50:0.05 acetonitrile:water:trifluoroacetic acid (TFA)

HPLC Conditions: **Flow Rate**: 0.50 mL/min. **Inj. Volume:** 75 µL **Column Temp.**: 30°C

The HPLC system has a post-column "splitter" installed between the C18 column exit and the detector, which directs ~0.375 mL/min of the column flow to waste and ~0.125 mL/min to the MS detector (approximately 4:1 split ratio).

MS Detector: **Cone**: 22V **Probe**: 110°C **Needle**: 3.5kV

Acquire the [M+1] molecular ion at 280 m/z, using positive-ion, electrospray ionization mass spectrometry.

If desired, the UV detector absorbance signal can be monitored at 216 nm.

Retention Time: (S) enantiomer ~10.8 minutes
(R) enantiomer ~16.1 minutes

These run times may vary slightly with column batch and mobile phase composition.

If late eluting peaks are observed, the run time should be increased to remove the late eluting compounds from the analytical column. A gradient elution, using an increased percentage of acetonitrile in the mobile phase, can be used to allow the late eluting peaks to be carried off of the analytical column. Depending upon the crop substrate and the retention time of the late eluting peak, exact gradient elution conditions can be developed as appropriate by monitoring a UV absorption trace at 216 nm or the acquisition signal at 280 m/z. (The LC/MS system is ideally suited for gradient elutions at trace levels due to its rapid re-equilibration time).

Run Time: ~25 minutes

Data Acquisition: The LC/MS Masslynx software has the capability to calculate the parts per million of analyte in a crop substrate if the milligram amount of crop-equivalent injected is supplied to the instrument. However it is typically easier for an analyst to obtain a calibration curve from the LC/MS system that lists "ng found". The "ng foung" data from the LC/MS data system is then transcribed into the Multichrom Worksheet program (version ws.pas 1.3.1) to completely calculate parts per million of analyte in the test substrate.

TABLE 2: SOURCE OF CONTROL AND TREATED CROP SAMPLES ANALYZED AS PART OF THIS STUDY*

Original Study No.	Original Field Test No.	Crop	Substrate	PHI	Sample Code	Treatment Substance †	Maximum Months Stored
105-95	0S-FR-108-95[2]	**tobacco**	mature leaf ‡	62 days	1-3-B	- -	42
			green leaf	0 days	2-2-A	metalaxyl	42
					2-2-B	metalaxyl	42
					3-2-A	mefenoxam	42
					3-2-B	mefenoxam	42
			mature leaf	62 days	1-3-B	- -	40
					2-3-A	metalaxyl	40
					2-3-B	metalaxyl	40
					3-3-A	mefenoxam	40
					3-3-B	mefenoxam	40
100-95	02-FR-039-95[3]	**tomato**	fruit	0 days	1-2-A	- -	39
					2-2-A	metalaxyl	39
					2-2-B	metalaxyl	39
					3-2-A	mefenoxam	39
					3-2-B	mefenoxam	39
			leaves	1 days	1-3-A	- -	39
					2-3-A	metalaxyl	39
					2-3-B	metalaxyl	39
					3-3-A	mefenoxam	39
					3-3-B	mefenoxam	39
			fruit §	0 days	1-2-A	- -	39
			mature fruit	5 days	2-4-A	metalaxyl	39
					2-4-B	metalaxyl	39
					3-4-A	mefenoxam	39
					3-4-B	mefenoxam	39

* Complete details of the source, treatment, and harvest are contained in the Residue Test Reports.

† Refer to the Residue Test Reports (RTR's) for complete information regarding treatment substance and application rates.

‡ Control mature tobacco leaf is substituted here because bulk sample of control immature tobacco leaf was depleted or unavailable.

§ Control immature fruit is substituted here because bulk sample of control mature fruit was depleted or unavailable.

TABLE 2: SOURCE OF CONTROL AND TREATED CROP SAMPLES ANALYZED AS PART OF THIS STUDY* (Continued)

Original Study No.	Original Field Test No.	Crop	Substrate	PHI	Sample Code	Treatment Substance †	Maximum Months Stored
106-95	0S-FR-305-95[4]	cabbage	heads	0 days	1-2-A	- -	42
					2-2-A	metalaxyl	42
					2-2-B	metalaxyl	42
					3-2-A	mefenoxam	42
					3-2-B	mefenoxam	42
			wrapper leaves	0 days	1-3-A	- -	42
					2-3-A	metalaxyl	42
					2-3-B	metalaxyl	42
					3-3-A	mefenoxam	42
					3-3-B	mefenoxam	42
			trimmed head	0 days	1-4-A	- -	42
					2-4-A	metalaxyl	42
					2-4-B	metalaxyl	42
					3-4-A	mefenoxam	42
					3-4-B	mefenoxam	42
			heads	7 days	1-5-A	- -	42
					2-5-A	metalaxyl	42
					2-5-B	metalaxyl	42
					3-5-A	mefenoxam	42
					3-5-B	mefenoxam	42
			wrapper leaves	7 days	1-6-A	- -	42
					2-6-A	metalaxyl	42
					2-6-B	metalaxyl	42
					3-6-A	mefenoxam	42
					3-6-B	mefenoxam	42
			trimmed head	7 days	1-7-A	- -	41
					2-7-A	metalaxyl	41
					2-7-B	metalaxyl	41
					3-7-A	mefenoxam	41
					3-7-B	mefenoxam	41

* Complete details of the source, treatment, and harvest are contained in the Residue Test Reports.

† Refer to the Residue Test Reports (RTR's) for complete information regarding treatment substance and application rates.

TABLE 2: SOURCE OF CONTROL AND TREATED CROP SAMPLES ANALYZED AS PART OF THIS STUDY* (Continued)

Original Study No.	Original Field Test No.	Crop	Substrate	PHI	Sample Code	Treatment Substance †	Maximum Months Stored
101-95	0W-FR-615-95[5]	**potato**	foliage	0 days	1-2-A	- -	39
					2-2-A	metalaxyl	39
					2-2-B	metalaxyl	39
					3-2-A	mefenoxam	39
					3-2-B	mefenoxam	39
					4-2-A	metalaxyl	39
					4-2-B	metalaxyl	39
					5-2-A	mefenoxam	39
					5-2-B	mefenoxam	39
98JS30	98JS30	**lettuce**	head	0 day	SUBPR. 1, INT 1	^{14}C-mefenoxam	‡
				14 day	SUBPR. 1, INT. 2	"	
				21 day	SUBPR. 1, INT. 3	"	
				0 day	SUBPR. 2, INT. 1	^{14}C-metalaxyl	
				14 day	SUBPR. 2, INT. 2	"	
				21 day	SUBPR. 2, INT. 3	"	
				- -	CONTROL	- -	

* Complete details of the source, treatment, and harvest are contained in the Residue Test Reports.

† Refer to the Residue Test Reports (RTR's) for complete information regarding treatment substance and application rates.

‡ Refer to the final report for Novartis Study 98JS30 for complete information regarding test substance, application rates, storage intervals, and other information.

TABLE 3: RECOVERY RESULTS FOR CONTROL SUBSTRATES FORTIFIED WITH METALAXYL OR MEFENOXAM *

Substrate	Sample ID	Fortification Substance	Amount Added (ppm)	(R) Enantiomer Determined (ppm)	(R) Enantiomer Percent Recovery	(S) Enantiomer Determined (ppm)	(S) Enantiomer Percent Recovery	EE%
Tobacco								
green leaf	1-3-B	control	-	0 002	-	0 000	-	-
	1-3-B REC	metalaxyl	0.500	0 509	101%	0.517	103%	-1%
	1-3-B REC	mefenoxam	0.500	0 483	96%	0 009	-	96%
mature leaf	1-3-B	control	-	0 000	-	0.000	-	-
	1-3-B REC	metalaxyl	0.005	0 005	97%	0 005	88%	0%
	1-3-B REC	mefenoxam	0 005	0 005	93%	0 000	-	100%
Tomato								
fruit	1-2-A	control	-	0.001	-	0 000	-	-
	1-2-A REC	metalaxyl	0 500	0.478	95%	0 450	90%	3%
	1-2-A REC	mefenoxam	0 500	0 473	94%	0 009	-	96%
leaves	1-3-A	control	-	0 011	-	0 010	-	-
	1-3-A REC	metalaxyl	0 500	0 471	92%	0 474	93%	0%
	1-3-A REC	mefenoxam	0.500	0 529	104%	0 018	-	93%
mature fruit	1-2-A	control	-	0.002	-	0 000	-	-
	1-2-A REC	metalaxyl	0.005	0 005	65%	0 004	74%	11%
	1-2-A REC	mefenoxam	0 005	0 008	117%	0 000	-	100%

* Wherever finite incurred residues of the (S)- or (R) enantiomers were detected at less than the method limit of determination (LOD, <0.005 ppm), "uncorrected" residue values (ppm) are reported.

TABLE 3: RECOVERY RESULTS FOR CONTROL SUBSTRATES FORTIFIED WITH METALAXYL OR MEFENOXAM * (Continued)

Substrate	Sample ID	Fortification Substance	Amount Added (ppm)	(R) Enantiomer Determined (ppm)	(R) Enantiomer Percent Recovery	(S) Enantiomer Determined (ppm)	(S) Enantiomer Percent Recovery	EE%
Cabbage								
heads	1-2-A	control	-	0 002	-	0.002	-	-
	1-2-A REC	metalaxyl	0 500	0.499	99%	0 489	98%	1%
	1-2-A REC	mefenoxam	0.500	0 484	96%	0 009	-	96%
wrapper leaves	1-3-A	control	-	0 002	-	0 000	-	-
	1-3-A REC	metalaxyl	0 500	0 560	112%	0.563	113%	0%
	1-3-A REC	mefenoxam	0.500	0 499	99%	0 013	-	95%
trimmed heads	1-4-A	control	-	0 000	-	0 000	-	-
	1-4-A REC	metalaxyl	0 500	0.513	103%	0 518	104%	0%
	1-4-A REC	mefenoxam	0.500	0.481	96%	0.010	-	96%
heads	1-5-A	control	-	0 001	-	0 000	-	-
	1-5-A REC	metalaxyl	0 005	0 006	88%	0 006	107%	0%
	1-5-A REC	mefenoxam	0 005	0 005	75%	0 000	-	100%
wrapper leaves	1-6-A	control	-	0 001	-	0.000	-	-
	1-6-A REC	metalaxyl	0 500	0 524	105%	0.518	104%	1%
	1-6-A REC	mefenoxam	0.500	0 475	95%	0 011	-	95%
trimmed heads	1-7-A	control	-	0 001	-	0 001	-	-
	1-7-A REC	metalaxyl	0 005	0 007	116%	0.006	93%	8%
	1-7-A REC	mefenoxam	0 005	0 006	102%	0 000	-	100%

* Wherever finite incurred residues of the (S)- or (R) enantiomers were detected at less than the method limit of determination (LOD, <0.005 ppm), "uncorrected" residue values (ppm) are reported.

TABLE 3: RECOVERY RESULTS FOR CONTROL SUBSTRATES FORTIFIED WITH METALAXYL OR MEFENOXAM * (Continued)

Substrate	Sample ID	Fortification Substance	Amount Added (ppm)	(R) Enantiomer Determined (ppm)	(R) Enantiomer Percent Recovery	(S) Enantiomer Determined (ppm)	(S) Enantiomer Percent Recovery	EE%
Potato								
foliage	1-2-A	control	-	0 003	-	0.000	-	-
	1-2-A REC	metalaxyl	0 500	0 475	95%	0 474	95%	0%
	1-2-A REC	mefenoxam	0 500	0 499	99%	0 008	-	97%
Lettuce								
head	CONTROL	control	-	0.000	-	0.000	-	-
	RECOV	metalaxyl	0.500	0 516	103%	0.548	110%	-3%
	RECOV	mefenoxam	0 500	0 510	102%	0.022	-	92%

Statistical Summary for Samples Fortified with Metalaxyl

	(R) Enantiomer		(S) Enantiomer
Min =	65%	Min =	74%
Max =	116%	Max. =	113%
Avg =	98%	Avg =	98%
SD =	12.5%	SD =	10 4%
%CV =	12.7%	%CV =	10.7%
n =	13	n =	13

Statistical Summary for Samples Fortified with Metalaxyl

	(R) Enantiomer
Min. =	75%
Max. =	117%
Avg =	98%
SD =	9.3%
%CV =	9 5%
n =	13

* Wherever finite incurred residues of the (S)- or (R) enantiomers were detected at less than the method limit of determination (LOD, <0.005 ppm), "uncorrected" residue values (ppm) are reported.

TABLE 4: RESULTS OF ANALYSIS OF SAMPLES CONTAINING FIELD-INCURRED RESIDUES OF METALAXYL OR MEFENOXAM *

Substrate	Sample ID	Treatment Substance	PHI (days)	(R) Enantiomer Determined (ppm)	(S) Enantiomer Determined (ppm)	Total, as (RS) Racemate (ppm)	EE%
TOBACCO							
green leaf	1-3-B	control	-	0 002	0.000	0.002	
	2-2-A	metalaxyl	0	0.097	0.254	0.351	-45%
	2-2-B	metalaxyl	0	0 356	0.535	0 891	-20%
	3-2-A	mefenoxam	0	0 312	0 008	0.320	95%
	3-2-B	mefenoxam	0	0.957	0 020	0 977	96%
mature leaf	1-3-B	control	-	0 000	0.000	0 000	
	2-3-A	metalaxyl	61	0.021	0.068	0 089	-53%
	2-3-B	metalaxyl	61	0 014	0.049	0 063	-56%
	3-3-A	mefenoxam	61	0 005	0 002	0.007	43%
	3-3-B	mefenoxam	61	0 003	0.001	0 004	50%
TOMATO							
fruit	1-2-A	control	-	0 001	0 000	0 001	
	2-2-A	metalaxyl	0	0 065	0.060	0 125	4%
	2-2-B	metalaxyl	0	0.093	0 092	0 185	1%
	3-2-A	mefenoxam	0	0.091	0 003	0 094	94%
	3-2-B	mefenoxam	0	0 102	0.011	0 113	81%
foliage	1-3-A	control	-	0 011	0 010	0 021	
	2-3-A	metalaxyl	1	3.013	3 009	6 022	0%
	2-3-B	metalaxyl	1	3.778	3.903	7 681	-2%
	3-3-A	mefenoxam	1	2.771	0.100	2.871	93%
	3-3-B	mefenoxam	1	2 947	0.594	3 541	66%
mature fruit	1-2-A	control	-	0 002	0.000	0 002	
	2-4-A	metalaxyl	5	0 015	0.010	0 025	20%
	2-4-B	metalaxyl	5	0 033	0 023	0 056	18%
	3-4-A	mefenoxam	5	0 018	0.000	0 018	100%
	3-4-B	mefenoxam	5	0.044	0.003	0 047	87%

* Wherever finite incurred residues of the (S)- or (R) enantiomers were detected at concentration levels of less than the method limit of determination (LOD, <0.005 ppm), "uncorrected" residue values (ppm) are reported to assist in the calculation of "Total, as (RS) racemate". Additionally, data are unrounded.

TABLE 4: RESULTS OF ANALYSIS OF SAMPLES CONTAINING FIELD-INCURRED RESIDUES OF METALAXYL OR MEFENOXAM * (Continued)

Substrate	Sample ID	Treatment Substance	PHI (days)	(R) Enantiomer Determined (ppm)	(S) Enantiomer Determined (ppm)	Total, as (RS) Racemate (ppm)	EE%
CABBAGE							
head	1-2-A	control	-	0.002	0 002	0 004	
	2-2-A	metalaxyl	0	1 502	1 493	2 995	0%
	2-2-B	metalaxyl	0	1 324	1 317	2 641	0%
	3-2-A	mefenoxam	0	1.007	0 019	1 026	96%
	3-2-B	mefenoxam	0	1 119	0 027	1 146	95%
wrapper leaves	1-3-A	control	-	0 002	0 000	0 002	
	2-3-A	metalaxyl	0	2.967	3 142	6 109	-3%
	2-3-B	metalaxyl	0	2.721	2.897	5 618	-3%
	3-3-A	mefenoxam	0	2 198	0 050	2 248	96%
	3-3-B	mefenoxam	0	2.330	0 051	2 381	96%
trimmed head	1-4-A	control	-	0 000	0 000	0 000	
	2-4-A	metalaxyl	0	0.109	0 111	0 220	-1%
	2-4-B	metalaxyl	0	0.055	0 052	0 107	3%
	3-4-A	mefenoxam	0	0 100	0 000	0 100	100%
	3-4-B	mefenoxam	0	0.075	0 000	0 075	100%
head	1-5-A	control	-	0.001	0 000	0 001	
	2-5-A	metalaxyl	7	0.242	0 211	0 453	7%
	2-5-B	metalaxyl	7	0.525	0 454	0.979	7%
	3-5-A	mefenoxam	7	0.352	0 009	0 361	95%
	3-5-B	mefenoxam	7	0 209	0 005	0 214	95%
wrapper leaves	1-6-A	control	-	0 001	0.000	0.001	
	2-6-A	metalaxyl	7	0.497	0 587	1 084	-8%
	2-6-B	metalaxyl	7	0.570	0 657	1 227	-7%
	3-6-A	mefenoxam	7	0 329	0.009	0 338	95%
	3-6-B	mefenoxam	7	0.424	0 012	0 436	94%
trimmed head	1-7-A	control	-	0.001	0.001	0 002	
	2-7-A	metalaxyl	7	0 024	0 026	0.050	-4%
	2-7-B	metalaxyl	7	0 033	0 037	0 070	-6%
	3-7-A	mefenoxam	7	0.017	0 000	0 017	100%
	3-7-B	mefenoxam	7	0.021	0 000	0 021	100%

* Wherever finite incurred residues of the (S)- or (R) enantiomers were detected at concentration levels of less than the method limit of determination (LOD, <0.005 ppm), "uncorrected" residue values (ppm) are reported to assist in the calculation of "Total, as (RS) racemate" Additionally, data are unrounded.

TABLE 4: RESULTS OF ANALYSIS OF SAMPLES CONTAINING FIELD-INCURRED RESIDUES OF METALAXYL OR MEFENOXAM * (Continued)

Substrate	Sample ID	Treatment Substance	PHI (days)	(R) Enantiomer Determined (ppm)	(S) Enantiomer Determined (ppm)	Total, as (RS) Racemate (ppm)	EE%
POTATO							
foliage	1-2-A	control	-	0 003	0.000	0 003	
	2-2-A	metalaxyl	0	0.783	0 861	1 644	-5%
	2-2-B	metalaxyl	0	0 912	0 919	1 831	0%
	3-2-A	mefenoxam	0	0 002	0.000	0 002	100%
	3-2-B	mefenoxam	0	0.378	0 010	0.388	95%
	4-2-A	metalaxyl	0	0.853	0 948	1 801	-5%
	4-2-B	metalaxyl	0	0.923	20	1.843	0%
	5-2-A	mefenoxam	0	0 475	[illegible]10	0 485	96%
	5-2-B	mefenoxam	0	0 319	0 006	0 325	96%
LETTUCE							
head	CONTROL	control	-	0 000	0 000	0 000	
	SUBPR1INT1	mefenoxam	0	6 389	0 137	6 526	96%
	SUBPR1INT2	mefenoxam	14	0 144	0 006	0 150	92%
	SUBPR1INT3	mefenoxam	21	0 019	0 001	0 020	90%
	SUBPR2INT1	metalaxyl	0	2 574	2 868	5 442	-5%
	SUBPR2INT2	metalaxyl	14	0 090	0 165	0 255	-29%
	SUBPR2INT3	metalaxyl	21	0 025	0 047	0 072	-31%

* Wherever finite incurred residues of the (S)- or (R) enantiomers were detected at concentration levels of less than the method limit of determination (LOD, <0.005 ppm), "uncorrected" residue values (ppm) are reported to assist in the calculation of "Total, as (RS) racemate". Additionally, data are unrounded.

TABLE 5: EXTRACTABILITY AND ACCOUNTABILITY DATA FROM ANALYSES OF ^{14}C-RADIOLABELED HEAD LETTUCE USING ANALYTICAL METHOD 456-98

			Novartis Metabolism Study 98JS30			Novartis Study 456-98					
Sample Description	PHI (days)	Treatment Substance	TRR Total Radioactive Residue by LSC (ppm)[1]	Parent Residue by TLC (ppm)[2]	Account-ability by TLC (%)[3]	^{14}C Residue in Initial Extract by LSC (ppm)[4]	Extract-ability of Residues (%)[5]	^{14}C Residues in Final Fraction by LSC (ppm)[6]	Extract-ability of Residues in Final Fraction (%)[7]	Residue in Final Fraction as RS-Racemate by LC/MS (ppm)[8]	Account-ability by LC/MS (%)[9]
CONTROL	-	-	0 000	0 000	-	0 003	-	0 001	-	0 000	-
SUBPR1 INT1	0	^{14}C-mefenoxam	8 725	6 438	74%	10 894	125%	7 220	83%	6 526	75%
SUBPR1 INT2	14	"	2.443	0 274	11%	1 913	78%	0 157	6%	0 150	6%
SUBPR1 INT3	21	"	0 615	0 040	7%	0 600	97%	0 030	5%	0 020	3%
SUBPR2 INT1	0	^{14}C-metalaxyl	7 218	5 895	82%	7 636	106%	5 580	77%	5 442	75%
SUBPR2 INT2	14	"	1.833	0 326	18%	2 000	109%	0 277	15%	0 255	14%
SUBPR2 INT3	21	"	1.070	0 110	10%	1 199	112%	0 087	8%	0 072	7%

1 TRR, Total radioactive residues (ppm) as determined by combustion/LSC analysis (*ref Novartis Metabolism Study 98JS30*)
2 Parent residue by TLC (ppm) determined by thin-layer chromatography (*ref Novartis Metabolism Study 98JS30*)
3 Accountability by TLC (%) = (Parent residue by TLC in sample final fraction (ppm) + TRR (ppm)) x 100 (*ref Novartis Metabolism Study 98JS30*)
4 ^{14}C-Residues in initial extract determined by liquid scintillation counting of aliquots of the starting sample extract
5 Extractability of residues (%) = ^{14}C-Residues in initial extract (ppm) + TRR (ppm) x 100
6 ^{14}C-Residues in final fraction (ppm) determined by liquid scintillation counting
7 Extractability of residues in final fraction (%) = (^{14}C-Residues in final fraction (ppm) + TRR (ppm)) x 100
8 Residue in Final Fraction, as (RS) Racemate, (ppm) determined by LC/MS
9 Accountability by LC/MS (%) = (Residue in final fraction as (RS) racemate by LC/MS (ppm) + TRR (ppm)) x 100

TABLE 6: RAW DATA WORKSHEET - (S) ENANTIOMER IN GREEN LEAF TOBACCO

Worksheet 6561-26 | Limit of Determ 0 005 ppm | Extracted 9-Dec-1998 | Product
Ana Set Name | Corr Factor A 1 0000 | Analyzed | **Analyte .: (S)-enantiomer**
Analysis | Corr Factor B 1 0000 | Channel 34 | Project 456-98
Subject tobacco

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-3-B	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
3	S												75 00	
4	R	1	"	"	90.00	10.03	100 00	25 00			2 300	4 0	75 00	43 123
5	S												75 00	
6	R	1	"	"	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
7	S												75 00	
8	X	1	"	2-2-A	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
9	S												75 00	
10	X	1	"	2-2-B	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
11	S												75 00	
12	X	1	"	3-2-A	90 00	10 04	100 00	25 00			2 302	4 0	75 00	43 162
13	S												75 00	
14	X	1	"	3-2-B	90 00	10 04	100 00	25 00			2 302	4 0	75 00	43 162
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0.180								MIXED STD (R,S)
2*	C	1				0 000	0 000				100 00	<0 005		CTRL tobacco
3	S		0 02000	1 5000		1 450								MIXED STD (R,S)
4	R	1				22 300	0 517	0 517	0 500	103 43				CTRL tobacco +0 500ppm RS
5	S		0 10000	7 5000		7 220								MIXED STD (R,S)
6	R	1				0 370	0 009	0 009	0 000					CTRL tobacco +0 500ppm R
7	S		0 50000	37 5000		36 770								MIXED STD (R,S)
8	X	1				10.950	0 254				100 00	0 254		RS-TRTD A-rep
9	S		1 00000	75 0000		75 440								MIXED STD (R,S)
10	X	1				23.040	0 535				100 00	0 535		RS-TRTD B-rep
11	S		0 20000	15 0000		14 820								MIXED STD (R,S)
12	X	1				0 340	0 008				100 00	0 008		R-TRTD A-rep
13	S		0 05000	3 7500		3.640								MIXED STD (R,S)
14	X	1				0 870	0 020				100 00	0 020		R-TRTD B-rep
15	S		0 00500	0 3750		0 350								MIXED STD (R,S)

Substrates green leaf
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 7: RAW DATA WORKSHEET - (R) ENANTIOMER IN GREEN LEAF TOBACCO

Worksheet 6561-26 | Limit of Determ 0 005 ppm | Extracted 9-Dec-1998 | Product
Ana Set Name | Corr Factor A 1 0000 | Analyzed | **Analyte .: (R) enantiomer**
Analysis | Corr Factor B 1 0000 | Channel 34 | Project 456-98
Subject tobacco

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-3-B	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
3	S												75 00	
4	R	1	"	"	90 00	10 03	100 00	25 00			2 300	4 0	75 00	43 123
5	S												75 00	
6	R	1	"	"	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
7	S												75 00	
8	X	1	"	2-2-A	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
9	S												75 00	
10	X	1	"	2-2-B	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
11	S												75 00	
12	X	1	"	3-2-A	90 00	10 04	100 00	25 00			2 302	4 0	75 00	43 162
13	S												75 00	
14	X	1	"	3-2-B	90 00	10 04	100 00	25 00			2 302	4 0	75 00	43 162
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 160								MIXED STD (R,S)
2*	C	1				0.090	0 002				98 83	<0 005		CTRL tobacco
3	S		0 02000	1 5000		1 440								MIXED STD (R,S)
4	R	1				21 970	0 509	0 507	0 500	101 48				CTRL tobacco +0 500ppm RS
5	S		0 10000	7 5000		7 120								MIXED STD (R,S)
6	R	1				20 810	0 483	0 481	0 500	96 19				CTRL tobacco +0 500ppm R
7	S		0 50000	37 5000		37.850								MIXED STD (R,S)
8	X	1				4 130	0 096				98 83	0 097		RS-TRTD A-rep
9	S		1 00000	75 0000		74 860								MIXED STD (R,S)
10	X	1				15 160	0 352				98 83	0 356		RS-TRTD B-rep
11	S		0 20000	15 0000		15 030								MIXED STD (R,S)
12	X	1				13 310	0 308				98 83	0 312		R-TRTD A-rep
13	S		0 05000	3 7500		3 670								MIXED STD (R,S)
14	X	1				40 840	0 946				98 83	0 957		R-TRTD B-rep
15	S		0 00500	0 3750		0 330								MIXED STD (R,S)

Substrates green leaf

B Blank C : Control F Freezer R Recovery

* Control used for this group Negative controls are treated as 0 0

Notes

S Standard V Solvent X Sample

Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 8: RAW DATA WORKSHEET - (S) ENANTIOMER IN MATURE LEAF TOBACCO

Worksheet 6561-28
Ana Set Name
Analysis
Subject tobacco

Limit of Determ 0 005 ppm
Corr Factor A 1 0000
Corr Factor B 1 0000

Extracted 10 Dec-1998
Analyzed
Channel 34

Product
Analyte .: (S)-enantiomer
Project 456 98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-3-B	90 00	10 04	100 00	25 00			2 302	4 0	75 00	43 162
3	S												75 00	
4	R	1	"	"	90 00	10 03	100 00	25 00			2 300	4 0	75 00	43 123
5	S												75 00	
6	R	1	"	"	90 00	10 03	100 00	25 00			2 300	4 0	75 00	43 123
7	S												75 00	
8	X	1	"	2-3-A	90 00	10 00	100 00	25 00			2 294	4 0	75 00	43 005
9	S												75 00	
10	X	1	"	2-3-B	90 00	10 04	100 00	25 00			2 302	4 0	75 00	43 162
11	S												75 00	
12	X	1	"	3-3-A	90 00	10 02	100 00	25 00			2 298	4 0	75 00	43 083
13	S												75 00	
14	X	1	"	3-3-B	90 00	10 03	100 00	25 00			2 300	4 0	75 00	43 123
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 180								MIXED STD (R,S)
2*	C	1				0 020	0 000				88 13	<0 005		CTRL tobacco
3	S		0 02000	1 5000		1 470								MIXED STD (R,S)
4	R	1				0 210	0 005	0 004	0 005	88 13				CTRL tobacco +0 005ppm RS
5	S		0 10000	7 5000		7 140								MIXED STD (R,S)
6	R	1				0 020	0 000	0 000	0 000					CTRL tobacco +0 005ppm R
7	S		0 50000	37 5000		37 230								MIXED STD (R,S)
8	X	1				2.570	0 060				88 13	0 068		RS-TRTD A-rep
9	S		1 00000	75 0000		75 120								MIXED STD (R,S)
10	X	1				1 880	0 044				88 13	0 049		RS-TRTD B-rep
11	S		0 20000	15 0000		15 260								MIXED STD (R,S)
12	X	1				0 070	0 002				88 13	<0 005		R-TRTD A-rep
13	S		0 05000	3 7500		3 800								MIXED STD (R,S)
14	X	1				0 060	0 001				88 13	<0 005		R-TRTD B-rep
15	S		0 00500	0 3750		0 400								MIXED STD (R,S)

Substrates mature leaf
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 9: RAW DATA WORKSHEET - (R) ENANTIOMER IN MATURE LEAF TOBACCO

Worksheet 6561-28
Ana Set Name
Analysis
Subject tobacco

Limit of Determ 0 005 ppm
Corr Factor A 1 0000
Corr Factor B 1 0000

Extracted 10-Dec-1998
Analyzed
Channel 34

Product
Analyte .: (R) enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S													
2*	C	1	RIMV01298	1-3-B	90 00	10 04	100 00	25 00					75 00	
3	S										2 302	4 0	75 00	43 162
4	R	1	"	"	90 00	10 03	100 00	25 00					75 00	
5	S										2 300	4 0	75 00	43 123
6	R	1	"	"	90 00	10 03	100 00	25 00					75 00	
7	S										2 300	4 0	75 00	43 123
8	X	1	"	2-3-A	90 00	10 00	100 00	25 00					75 00	
9	S		"								2 294	4 0	75 00	43 005
10	X	1	"	2-3-B	90 00	10 04	100 00	25 00					75 00	
11	S										2 302	4 0	75 00	43 162
12	X	1	"	3-3-A	90 00	10 02	100 00	25 00					75 00	
13	S										2 298	4 0	75 00	43 083
14	X	1	"	3-3-B	90 00	10 03	100 00	25 00					75 00	
15	S										2 300	4 0	75 00	43 123
													75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 170								MIXED STD (R,S)
2*	C	1				0 020	0 000				95 09	<0 005		CTRL tobacco
3	S		0 02000	1 5000		1 430								MIXED STD (R,S)
4	R	1				0 230	0 005	0 005	0 005	97 40				CTRL tobacco +0 005ppm RS
5	S		0 10000	7 5000		7 200								MIXED STD (R,S)
6	R	1				0 220	0 005	0 005	0 005	92 77				CTRL tobacco +0 005ppm R
7	S		0 50000	37 5000		37 330								MIXED STD (R,S)
8	X	1				0 840	0 020				95 09	0 021		RS-TRTD A-rep
9	S		1 00000	75 0000		75 110								MIXED STD (R,S)
10	X	1				0 590	0 014				95 09	0 014		RS-TRTD B-rep
11	S		0 20000	15 0000		15 060								MIXED STD (R,S)
12	X	1				0 220	0 005				95 09	0 005		R-TRTD A-rep
13	S		0 05000	3 7500		3 730								MIXED STD (R,S)
14	X	1				0 120	0 003				95 09	<0 005		R-TRTD B-rep
15	S		0 00500	0 3750		0 360								MIXED STD (R,S)

Substrates mature leaf
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 10: RAW DATA WORKSHEET - (S) ENANTIOMER IN TOMATO FRUIT

Worksheet	6561-30	Limit of Determ	0 005 ppm	Extracted	14-Dec-1998	Product	
Ana Set Name		Corr Factor A	1 0000	Analyzed		**Analyte .: (S)-enantiomer**	
Analysis	6561-30	Corr Factor B	1 0000	Channel	. 34	Project	456-98
Subject	tomato						

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-2-A	93 00	10 04	100 00	25 00			2 296	4 0	75 00	43 043
3	S												75 00	
4	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
5	S												75 00	
6	R	1	"	"	93 00	10 02	100.00	25 00			2 291	4 0	75 00	42 965
7	S												75 00	
8	X	1	"	2-2-A	93 00	10.02	100 00	25 00			2 291	4 0	75 00	42 965
9	S												75 00	
10	X	1	"	2-2-B	93 00	10 03	100 00	25 00			2 294	4 0	75 00	43 004
11	S												75 00	
12	X	1	"	3-2-A	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
13	S												75 00	
14	X	1	"	3-2-B	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 210								MIXED STD (R,S)
2*	C	1				0 000	0 000				90 07	<0 005		CTRL tomatoe
3	S		0 02000	1 5000		1 530								MIXED STD (R,S)
4	R	1				19.350	0 450	0 450	0 500	90 07				CTRL tomatoe +0 50 ppm RS
5	S		0 10000	7 5000		7 490								MIXED STD (R,S)
6	R	1				0 380	0 009	0 009	0 000					CTRL tomatoe +0 50 ppm R
7	S		0 50000	37 5000		38 370								MIXED STD (R,S)
8	X	1				2 310	0 054				90 07	0 060		RS-TRTD A-rep
9	S		1 00000	75 0000		74 560								MIXED STD (R,S)
10	X	1				3 550	0 083				90 07	0 092		RS-TRTD B-rep
11	S		0 20000	15 0000		15 060								MIXED STD (R,S)
12	X	1				0 130	0 003				90 07	<0 005		R-TRTD A-rep
13	S		0 05000	3 7500		3 680								MIXED STD (R,S)
14	X	1				0 420	0 010				90 07	0 011		R-TRTD B-rep
15	S		0 00500	0 3750		0 350								MIXED STD (R,S)

Substrates immature fruit

B Blank C Control F Freezer R · Recovery

* Control used for this group Negative controls are treated as 0 0

Notes

S Standard V Solvent X Sample

Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 11: RAW DATA WORKSHEET - (R) ENANTIOMER IN TOMATO FRUIT

Worksheet 6561-30
Ana Set Name
Analysis 6561-30
Subject tomato

Limit of Determ . 0 005 ppm
Corr Factor A . 1 0000
Corr Factor B 1 0000

Extracted 14-Dec-1998
Analyzed .
Channel 34

Product
Analyte .: (R) enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-2-A	93 00	10 04	100 00	25 00			2 296	4 0	75.00	43.043
3	S												75 00	
4	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
5	S												75 00	
6	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
7	S												75 00	
8	X	1	"	2-2-A	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
9	S												75 00	
10	X	1	"	2-2-B	93 00	10 03	100 00	25 00			2 294	4 0	75 00	43 004
11	S												75 00	
12	X	1	"	3-2-A	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
13	S												75 00	
14	X	1	"	3-2-B	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 170								MIXED STD (R,S)
2*	C	1				0 050	0 001				94 85	<0 005		CTRL tomatoe
3	S		0 02000	1 5000		1 530								MIXED STD (R,S)
4	R	1				20.520	0 478	0 476	0 500	95 29				CTRL tomatoe +0 50 ppm RS
5	S		0 10000	7 5000		7 470								MIXED STD (R,S)
6	R	1				20 330	0 473	0 472	0 500	94 40				CTRL tomatoe +0 50 ppm R
7	S		0 50000	37 5000		38 230								MIXED STD (R,S)
8	X	1				2 640	0 061				94 85	0 065		RS-TRTD A-rep
9	S		1 00000	75 0000		74 730								MIXED STD (R,S)
10	X	1				3 800	0 088				94 85	0 093		RS-TRTD B-rep
11	S		0 20000	15 0000		14.600								MIXED STD (R,S)
12	X	1				3 710	0 086				94 85	0 091		R-TRTD A-rep
13	S		0 05000	3 7500		3.550								MIXED STD (R,S)
14	X	1				4.150	0 097				94 85	0 102		R-TRTD B-rep
15	S		0 00500	0 3750		0 330								MIXED STD (R,S)

Substrates immature fruit
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 12: RAW DATA WORKSHEET - (S) ENANTIOMER IN TOMATO FOLIAGE

Worksheet	6561-32B	Limit of Determ	0 005 ppm	Extracted	14-Dec-1998	Product
Ana Set Name		Corr Factor A .	1 0000	Analyzed		**Analyte .: (S)-enantiomer**
Analysis		Corr Factor B	1 0000	Channel	34	Project 456-98
Subject	tomato					

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-3-A	90 00	10 00	100 00	25 00			2 294	4 0	75 00	43 005
3	S												75 00	
4	R	1	"	"	90 00	10 01	100 00	25.00			2 296	4 0	75 00	43 044
5	S												75 00	
6	R	1	"	"	90 00	10 07	100 00	25 00			2 308	4 0	75 00	43 281
7	S												75.00	
8	X	1	"	2-3-A	90 00	10 01	100 00	25 00	4 00	0 50	0 287	5 0	75 00	4 304
9	S												75 00	
10	X	1	"	2-3-B	90 00	10 00	100 00	25 00	4 00	0 50	0 287	5 0	75 00	4 300
11	S												75 00	
12	X	1	"	3-3-A	90 00	10 00	100 00	25 00	4 00	0 50	0 287	5 0	75 00	4 300
13	S												75 00	
14	X	1	'	3-3-B	90 00	9 98	100 00	25 00	4 00	0 50	0 286	5 0	75`00	4 293
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 200								MIXED STD (R,S)
2*	C	1				0 400	0 009				92 88	0 010		CTRL foliage
3	S		0 02000	1 5000		1 620								MIXED STD (R,S)
4	R	1				20 390	0 474	0 464	0 500	92 88				CTRL foliage +0 50 ppm RS
5	S		0 10000	7 5000		8 090								MIXED STD (R,S)
6	R	1				0.800	0 018	0 009	0 000					CTRL foliage +0 50 ppm R
7	S		0 50000	37 5000		39 980								MIXED STD (R,S)
8	X	1				12 030	2 795				92 88	3 009		RS-TRTD A-rep foliage
9	S		1 00000	75 0000		74 200								MIXED STD (R,S)
10	X	1				15 590	3 625				92 88	3 903		RS-TRTD B-rep foliage
11	S		0 20000	15 0000		12 660								MIXED STD (R,S)
12	X	1				0 400	0 093				92 88	0 100		R-TRTD A-rep foliage
13	S		0 05000	3 7500		3 220								MIXED STD (R,S)
14	X	1				2 370	0 552				92 88	0 594		R-TRTD B-rep foliage
15	S		0 00500	0.3750		0.290								MIXED STD (R,S)

Substrates foliage

B Blank C Control F Freezer R . Recovery

* : Control used for this group Negative controls are treated as 0 0

Notes

S . Standard V Solvent X · Sample

Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 13: RAW DATA WORKSHEET - (R) ENANTIOMER IN TOMATO FOLIAGE

Worksheet 6561-32B
Ana Set Name
Analysis
Subject tomato

Limit of Determ 0 005 ppm
Corr Factor A 1 0000
Corr Factor B 1 0000

Extracted 14-Dec-1998
Analyzed
Channel 34

Product
Analyte .: (R) enanti
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-3-A	90 00	10 00	100 00	25 00			2 294	4.0	75 00	43.005
3	S												75 00	
4	R	1	"	"	90 00	10 01	100 00	25 00			2 296	4 0	75 00	43 044
5	S												75 00	
6	R	1	"	"	90 00	10 07	100 00	25 00			2 308	4 0	75 00	43 281
7	S												75 00	
8	X	1	"	2-3-A	90.00	10 01	100 00	25.00	4 00	0 50	0 287	5 0	75 00	4 304
9	S												75.00	
10	X	1	"	2-3-B	90 00	10 00	100 00	25 00	4 00	0 50	0 287	5 0	75 00	4 300
11	S												75 00	
12	X	1	"	3-3-A	90 00	10 00	100 00	25 00	4 00	0 50	0 287	5 0	75 00	4 300
13	S												75 00	
14	X	1	"	3-3-B	90 00	9 98	100 00	25.00	4 00	0 50	0 286	5 0	75 00	4 293
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 190								MIXED STD (R,S)
2*	C	1				0.460	0 011				97 86	0 011		CTRL foliage
3	S		0 02000	1 5000		1 680								MIXED STD (R,S)
4	R	1				20 270	0 471	0 460	0 500	92 04				CTRL foliage +0 50 ppm RS
5	S		0 10000	7 5000		8.100								MIXED STD (R,S)
6	R	1				22 900	0 529	0 518	0 500	103 68				CTRL foliage +0 50 ppm R
7	S		0 50000	37 5000		40 580								MIXED STD (R,S)
8	X	1				12 690	2 948				97 86	3 013		RS-TRTD A-rep foliage
9	S		1 00000	75 0000		73 850								MIXED STD (R,S)
10	X	1				15 900	3 697				97 86	3 778		RS-TRTD B-rep foliage
11	S		0 20000	15 0000		12 900								MIXED STD (R,S)
12	X	1				11 660	2 711				97 86	2 771		R-TRTD A-rep foliage
13	S		0 05000	3 7500		3.150								MIXED STD (R,S)
14	X	1				12.380	2 884				97 86	2 947		R-TRTD B-rep foliage
15	S		0 00500	0 3750		0.280								MIXED STD (R,S)

Substrates foliage

Notes
B Blank C Control F Freezer R Recovery S Standard V Solvent X Sample
* Control used for this group Negative controls are treated as 0 0 Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 14: RAW DATA WORKSHEET - (S) ENANTIOMER IN MATURE TOMATO FRUIT

Worksheet 6561-34
Ana Set Name
Analysis
Subject tomato

Limit of Determ : 0.005 ppm
Corr Factor A .. 1 0000
Corr Factor B . 1 0000

Extracted 14-Dec-1998
Analyzed
Channel 34

Product
Analyte .: (S)-enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-2-A	93 00	10 06	100 00	25 00			2 300	4 0	75.00	43 122
3	S												75.00	
4	R	1	"	"	93 00	10 05	100 00	25 00			2 298	4 0	75 00	43 083
5	S												75 00	
6	R	1	"	"	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
7	S												75 00	
8	X	1	"	2-4-A	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
9	S												75 00	
10	X	1	"	2-4-B	93 00	10 15	100 00	25 00			2 319	4 0	75 00	43 474
11	S												75 00	
12	X	1	"	3-4-A	93 00	10 02	100 00	25.00			2 291	4 0	75 00	42 965
13	S												75 00	
14	X	1	"	3-4-B	93 00	10 11	100 00	25 00			2 310	4 0	75 00	43 318
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 170								MIXED STD (R,S)
2*	C	1				0 000	0 000				74 28	<0 005		CTRL tomatoe
3	S		0 02000	1 5000		1 500								MIXED STD (R,S)
4	R	1				0.160	0 004	0 004	0 005	74 28				CTRL tomatoe +0 005ppm RS
5	S		0 10000	7 5000		7 360								MIXED STD (R,S)
6	R	1				0 000	0 000	0 000	0 000					CTRL tomatoe +0 005ppm R
7	S		0 50000	37 5000		39.360								MIXED STD (R,S)
8	X	1				0 320	0 007				74 28	0 010		RS-TRTD A-rep
9	S		1 00000	75 0000		74.340								MIXED STD (R,S)
10	X	1				0 740	0 017				74 28	0 023		RS-TRTD B-rep
11	S		0 20000	15 0000		13 860								MIXED STD (R,S)
12	X	1				0 000	0 000				74 28	<0 005		R-TRTD A-rep
13	S		0 05000	3 7500		3 300								MIXED STD (R,S)
14	X	1				0 120	0 003				74 28	<0 005		R-TRTD B-rep
15	S		0 00500	0 3750		0 310								MIXED STD (R,S)

Substrates mature fruit
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 15. RAW DATA WORKSHEET - (R) ENANTIOMER IN MATURE TOMATO FRUIT

Worksheet 6561-34 | Limit of Determ 0 005 ppm | Extracted · 14-Dec-1998 | Product
Ana Set Name | Corr Factor A 1 0000 | Analyzed | **Analyte .: (R) enantiomer**
Analysis | Corr Factor B 1 0000 | Channel 34 | Project 456-98
Subject tomato

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75.00	
2*	C	1	RIMV01298	1-2-A	93 00	10 06	100 00	25 00			2 300	4 0	75.00	43 122
3	S												75 00	
4	R	1	"	"	93 00	10 05	100 00	25 00			2 298	4 0	75 00	43 083
5	S												75 00	
6	R	1	"	"	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
7	S												75 00	
8	X	1	"	2-4-A	93 00	10.00	100 00	25 00			2 287	4 0	75 00	42 887
9	S												75 00	
10	X	1	"	2-4-B	93 00	10 15	100 00	25 00			2 319	4 0	75 00	43 474
11	S												75 00	
12	X	1	"	3-4-A	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
13	S												75 00	
14	X	1	"	3-4-B	93 00	10 11	100 00	25 00			2 310	4 0	75 00	43 318
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 170								MIXED STD (R,S)
2*	C	1				0 080	0 002				90 91	<0 005		CTRL tomatoe
3	S		0 02000	1 5000		1 420								MIXED STD (R,S)
4	R	1				0 220	0 005	0 003	0 005	65 03				CTRL tomatoe +0 005ppm RS
5	S		0 10000	7 5000		7.440								MIXED STD (R,S)
6	R	1				0 330	0 008	0 006	0 005	116 79				CTRL tomatoe +0 005ppm R
7	S		0 50000	37 5000		39 570								MIXED STD (R,S)
8	X	1				0 600	0 014				90 91	0 015		RS-TRTD A-rep
9	S		1 00000	75 0000		74 110								MIXED STD (R,S)
10	X	1				1.300	0 030				90 91	0 033		RS-TRTD B-rep
11	S		0 20000	15 0000		14 360								MIXED STD (R,S)
12	X	1				0 700	0 016				90 91	0 018		R-TRTD A-rep
13	S		0.05000	3 7500		3 600								MIXED STD (R,S)
14	X	1				1.720	0 040				90 91	0 044		R-TRTD B-rep
15	S		0 00500	0 3750		0.330								MIXED STD (R,S)

Substrates mature fruit
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V . Solvent X . Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 16: RAW DATA WORKSHEET - (S) ENANTIOMER IN CABBAGE HEADS, 0-DAY PHI

Worksheet	6561-36	Limit of Determ	0 005 ppm	Extracted	15-Dec-1998	Product
Ana Set Name		Corr Factor A	1 0000	Analyzed		**Analyte .: (S)-enantiomer**
Analysis	. 6561-36	Corr Factor B	1 0000	Channel	34	Project 456-98
Subject	cabbage					

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75.00	
2*	C	1	RIMV01298	1-2-A	93 00	10 10	100 00	25 00			2 308	4 0	75 00	43 279
3	S												75 00	
4	R	1	"	"	93 00	10 03	100 00	25 00			2 294	4 0	75 00	43 004
5	S												75 00	
6	R	1	"	"	93 00	10 10	100 00	25 00			2 308	4 0	75 00	43 279
7	S												75 00	
8	X	1	"	2-2-A	93 00	10 09	100 00	25 00			2 306	4 0	75.00	43.239
9	S												75 00	
10	X	1	"	2-2-B	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
11	S												75 00	
12	X	1	"	3-2-A	93 00	10 09	100 00	25 00			2 306	4 0	75 00	43 239
13	S												75 00	
14	X	1	"	3-2-B	93 00	10 04	100.00	25 00			2 296	4 0	75 00	43 043
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 170								MIXED STD (R,S)
2*	C	1				0 080	0 002				97 53	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1.430								MIXED STD (R,S)
4	R	1				21 050	0 489	0 488	0 500	97 53				CTRL cabbage +0 50 ppm RS
5	S		0 10000	7 5000		7.100								MIXED STD (R,S)
6	R	1				0 390	0 009	0 007	0 000					CTRL cabbage +0 50 ppm R
7	S		0 50000	37 5000		37 780								MIXED STD (R,S)
8	X	1				62 960	1 456				97.53	1 493		RS-TRTD A-rep
9	S		1 00000	75 0000		74 900								MIXED STD (R,S)
10	X	1				55 170	1 284				97 53	1.317		RS-TRTD B-rep
11	S		0 20000	15 0000		15 030								MIXED STD (R,S)
12	X	1				0.810	0 019				97 53	0 019		R-TRTD A-rep
13	S		0 05000	3 7500		3 560								MIXED STD (R,S)
14	X	1				1 150	0 027				97 53	0 027		R-TRTD B-rep
15	S		0 00500	0 3750		0 350								MIXED STD (R,S)

Substrates head, 0-Day

B Blank C Control F Freezer R Recovery

* Control used for this group Negative controls are treated as 0 0

Notes .

S Standard V Solvent X . Sample

Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 17: RAW DATA WORKSHEET - (R) ENANTIOMER IN CABBAGE HEADS, 0-DAY PHI

Worksheet 6561-36
Ana Set Name
Analysis 6561-36
Subject cabbage

Limit of Determ 0 005 ppm
Corr Factor A ... 1.0000
Corr Factor B . 1 0000

Extracted 15-Dec-1998
Analyzed
Channel 34

Product
Analyte .: (R) enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-2-A	93 00	10 10	100 00	25 00			2 308	4 0	75 00	43 279
3	S												75.00	
4	R	1	"	"	93 00	10 03	100 00	25 00			2 294	4 0	75.00	43 004
5	S												75 00	
6	R	1	"	"	93 00	10 10	100 00	25 00			2 308	4 0	75 00	43 279
7	S												75.00	
8	X	1	"	2-2-A	93 00	10 09	100 00	25 00			2.306	4.0	75 00	43 239
9	S												75.00	
10	X	1	"	2-2-B	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
11	S												75 00	
12	X	1	"	3-2-A	93 00	10 09	100 00	25 00			2 306	4 0	75 00	43 239
13	S												75 00	
14	X	1	"	3-2-B	93 00	10 04	100 00	25 00			2 296	4 0	75 00	43.043
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 190								MIXED STD (R,S)
2*	C	1				0 070	0 002				97 94	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1.440								MIXED STD (R,S)
4	R	1				21 450	0 499	0 497	0 500	99 43				CTRL cabbage +0 50 ppm RS
5	S		0 10000	7 5000		7 270								MIXED STD (R,S)
6	R	1				20.940	0 484	0 482	0 500	96 44				CTRL cabbage +0 50 ppm R
7	S		0 50000	37 5000		38 150								MIXED STD (R,S)
8	X	1				63 600	1 471				97 94	1 502		RS-TRTD A-rep
9	S		1 00000	75 0000		74 780								MIXED STD (R,S)
10	X	1				55 730	1 297				97 94	1 324		RS-TRTD B-rep
11	S		0 20000	15 0000		14 680								MIXED STD (R,S)
12	X	1				42 660	0.987				97 94	1 007		R-TRTD A-rep
13	S		0 05000	3 7500		3 570								MIXED STD (R,S)
14	X	1				47 190	1 096				97 94	1 119		R-TRTD B-rep
15	S		0 00500	0 3750		0.320								MIXED STD (R,S)

Substrates . head, 0-Day
B Blank C Control F . Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X · Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 18: RAW DATA WORKSHEET - (S) ENANTIOMER IN CABBAGE WRAPPER LEAVES, 0-DAY PHI

Worksheet 6561-50
Ana Set Name
Analysis
Subject cabbage

Limit of Determ . 0 005 ppm
Corr Factor A . 1 0000
Corr Factor B . 1 0000

Extracted 16-Dec-1998
Analyzed
Channel 34

Product
Analyte .: (S)-enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75.00	
2*	C	1	RIMV01298	1-3-A	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
3	S												75 00	
4	R	1	"	"	93 00	10 00	100 00	25 00			2 287	4 0	75.00	42 887
5	S												75 00	
6	R	1	"	"	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
7	S												75 00	
8	X	1	"	2-3-A	93 00	10.01	100 00	25.00			2 289	25 0	75.00	6 868
9	S												75.00	
10	X	1	"	2-3-B	93 00	10 00	100 00	25 00			2 287	25 0	75 00	6 862
11	S												75 00	
12	X	1	"	3-3-A	93 00	9 99	100 00	25 00			2 285	25.0	75.00	6 856
13	S												75 00	
14	X	1	"	3-3-B	93 00	10 02	100 00	25 00			2 291	25 0	75 00	6 874
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 190								MIXED STD (R,S)
2*	C	1				0 020	0 000				100 00	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 660								MIXED STD (R,S)
4	R	1				24 150	0 563	0 563	0 500	112 53				CTRL cabbage +0 500ppm RS
5	S		0 10000	7 5000		7 920								MIXED STD (R,S)
6	R	1				0 560	0 013	0 013	0 000					CTRL cabbage +0 500ppm R
7	S		0 50000	37 5000		39 250								MIXED STD (R,S)
8	X	1				21 580	3 142				100 00	3 142		RS-TRTD A-rep
9	S		1 00000	75 0000		73.750								MIXED STD (R,S)
10	X	1				19.880	2 897				100.00	2 897		RS-TRTD B-rep
11	S		0 20000	15 0000		16 570								MIXED STD (R,S)
12	X	1				0 340	0 050				100 00	0 050		R-TRTD A-rep
13	S		0 05000	3 7500		4 110								MIXED STD (R,S)
14	X	1				0 350	0 051				100 00	0 051		R-TRTD B-rep
15	S		0 00500	0 3750		0 450								MIXED STD (R,S)

Substrates wrapper leaves, 0-day PHI
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes refer also 6561-38
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 19: RAW DATA WORKSHEET - (R) ENANTIOMER IN CABBAGE WRAPPER LEAVES, 0-DAY PHI

Worksheet · 6561-50
Ana Set Name
Analysis
Subject cabbage

Limit of Determ 0 005 ppm
Corr Factor A 1 0000
Corr Factor B . 1 0000

Extracted 16-Dec-1998
Analyzed
Channel . 34

Product
Analyte .: (R) enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-3-A	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
3	S												75 00	
4	R	1	"	"	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
5	S												75.00	
6	R	1	"	"	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
7	S												75 00	
8	X	1	"	2-3-A	93 00	10 01	100 00	25 00			2 289	25 0	75 00	6 868
9	S												75 00	
10	X	1	"	2-3-B	93 00	10 00	100 00	25 00			2 287	25 0	75 00	6 862
11	S												75 00	
12	X	1	"	3-3-A	93 00	9 99	100 00	25 00			2 285	25 0	75 00	6 856
13	S												75 00	
14	X	1	"	3-3-B	93 00	10 02	100 00	25 00			2 291	25 0	75 00	6 874
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 180								MIXED STD (R,S)
2*	C	1				0 080	0 002				100 00	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 720								MIXED STD (R,S)
4	R	1				24 020	0 560	0 558	0 500	111 64				CTRL cabbage +0 500ppm RS
5	S		0 10000	7 5000		8 410								MIXED STD (R,S)
6	R	1				21 400	0 499	0 497	0 500	99 33				CTRL cabbage +0 500ppm R
7	S		0 50000	37 5000		40 480								MIXED STD (R,S)
8	X	1				20 380	2 967				100 00	2 967		RS-TRTD A-rep
9	S		1 00000	75 0000		73 180								MIXED STD (R,S)
10	X	1				18 670	2 721				100 00	2 721		RS-TRTD B-rep
11	S		0 20000	15 0000		16 040								MIXED STD (R,S)
12	X	1				15 070	2 198				100 00	2 198		R-TRTD A-rep
13	S		0 05000	3 7500		4 200								MIXED STD (R,S)
14	X	1				16 020	2 330				100 00	2 330		R-TRTD B-rep
15	S		0 00500	0.3750		0 420								MIXED STD (R,S)

Substrates wrapper leaves, 0-day PHI
B Blank C Control F Freezer R · Recovery
* Control used for this group Negative controls are treated as 0 0

Notes refer also 6561-38
S . Standard V Solvent X . Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 20: RAW DATA WORKSHEET - (S) ENANTIOMER IN TRIMMED CABBAGE HEADS, 0-DAY PHI

Worksheet	6561-40	Limit of Determ	0 005 ppm	Extracted	17-Dec-1998	Product	
Ana Set Name		Corr Factor A	1 0000	Analyzed		**Analyte .: (S)-enantiomer**	
Analysis	6561-40	Corr Factor B	1 0000	Channel	34	Project	456-98
Subject	cabbage						

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-4-A	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
3	S												75 00	
4	R	1	"	"	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
5	S												75 00	
6	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
7	S												75 00	
8	X	1	"	2-4-A	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
9	S												75 00	
10	X	1	"	2-4-B	93 00	9 98	100 00	25 00			2 283	4 0	75 00	42 808
11	S												75 00	
12	X	1	"	3-4-A	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
13	S												75 00	
14	X	1	"	3-4-B	93 00	10 12	100 00	25 00			2 312	4 0	75 00	43 357
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 210								MIXED STD (R,S)
2*	C	1				0 000	0.000				100 00	<0 005		CTRL cabbage
3	S		0 02000	1 5000		2 270								MIXED STD (R,S)
4	R	1				22 230	0 518	0 518	0 500	103 57				CTRL cabbage +0 50 ppm RS
5	S		0 10000	7 5000		7.460								MIXED STD (R,S)
6	R	1				0 450	0 010	0 010	0 000					CTRL cabbage +0 50 ppm R
7	S		0 50000	37 5000		38 270								MIXED STD (R,S)
8	X	1				4 780	0 111				100 00	0 111		RS-TRTD A-rep
9	S		1 00000	75 0000		74 960								MIXED STD (R,S)
10	X	1				2 220	0 052				100 00	0 052		RS-TRTD B-rep
11	S		0 20000	15 0000		13 360								MIXED STD (R,S)
12	X	1				0 000	0 000				100 00	<0 005		R-TRTD A-rep
13	S		0 05000	3 7500		3 190								MIXED STD (R,S)
14	X	1				0 000	0 000				100 00	<0 005		R-TRTD B-rep
15	S		0 00500	0 3750		0 310								MIXED STD (R,S)

Substrates head, 0-Day

B Blank C Control F Freezer R Recovery S Standard V Solvent X Sample

* Control used for this group Negative controls are treated as 0 0

Notes

Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 21: RAW DATA WORKSHEET - (R) ENANTIOMER IN TRIMMED CABBAGE HEADS, 0-DAY PHI

Worksheet	6561-40	Limit of Determ .	0 005 ppm	Extracted	17-Dec-1998	Product	
Ana Set Name		Corr Factor A	1 0000	Analyzed .		**Analyte .: (R) enantiomer**	
Analysis	6561-40	Corr Factor B .	1 0000	Channel	34	Project	456-98
Subject	cabbage						

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-4-A	93 00	10.02	100 00	25 00			2 291	4 0	75 00	42 965
3	S												75 00	
4	R	1	"	"	93 00	10 01	100 00	25 00			2 289	4.0	75 00	42 926
5	S												75 00	
6	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
7	S												75 00	
8	X	1	"	2-4-A	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
9	S												75 00	
10	X	1	"	2-4-B	93 00	9 98	100 00	25 00			2 283	4 0	75 00	42 808
11	S												75 00	
12	X	1	"	3-4-A	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
13	S												75 00	
14	X	1	"	3-4-B	93 00	10 12	100 00	25 00			2 312	4 0	75 00	43.357
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 150								MIXED STD (R,S)
2*	C	1				0 000	0 000				99 38	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 460								MIXED STD (R,S)
4	R	1				22 020	0 513	0 513	0 500	102 60				CTRL cabbage +0 50 ppm RS
5	S		0 10000	7 5000		7 440								MIXED STD (R,S)
6	R	1				20 660	0 481	0 481	0 500	96 17				CTRL cabbage +0 50 ppm R
7	S		0 50000	37 5000		39 510								MIXED STD (R,S)
8	X	1				4.670	0 109				99 38	0 109		RS-TRTD A-rep
9	S		1 00000	75 0000		74 300								MIXED STD (R,S)
10	X	1				2 330	0 054				99 38	0 055		RS-TRTD B-rep
11	S		0 20000	15 0000		13 640								MIXED STD (R,S)
12	X	1				4 270	0 100				99 38	0 100		R-TRTD A-rep
13	S		0 05000	3 7500		3 350								MIXED STD (R,S)
14	X	1				3 230	0 074				99 38	0 075		R-TRTD B-rep
15	S		0 00500	0.3750		0 300								MIXED STD (R,S)

Substrates head, 0-Day

B Blank C Control F Freezer R Recovery

* Control used for this group Negative controls are treated as 0 0

Notes

S Standard V Solvent X Sample

Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 22: RAW DATA WORKSHEET - (S) ENANTIOMER IN CABBAGE HEADS, 7-DAY PHI

Worksheet 6561-44
Ana Set Name
Analysis
Subject cabbage

Limit of Determ 0 005 ppm
Corr Factor A 1 0000
Corr Factor B 1 0000

Extracted 21-Dec-1998
Analyzed
Channel 34

Product
Analyte .: (S)-enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-5-A	93 00	10 03	100 00	25 00			2 294	4 0	75 00	43 004
3	S												75 00	
4	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42.965
5	S												75 00	
6	R	1	"	"	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
7	S												75 00	
8	X	1	"	2-5-A	93 00	10 01	100 00	25 00			2 289	4 0	75.00	42 926
9	S												75 00	
10	X	1	"	2-5-B	93 00	10.01	100 00	25 00			2 289	4 0	75 00	42 926
11	S												75 00	
12	X	1	"	3-5-A	93 00	10 05	100 00	25 00			2 298	4.0	75 00	43 083
13	S												75 00	
14	X	1	"	3-5-B	93 00	10 01	100 00	25 00	54 00	42 00	1 781	3 0	75 00	44 516
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 170								MIXED STD (R,S)
2*	C	1				0 020	0 000				100 00	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 470								MIXED STD (R,S)
4	R	1				0.250	0 006	0 005	0 005	107 07				CTRL cabbage +0 005ppm RS
5	S		0 10000	7 5000		7 330								MIXED STD (R,S)
6	R	1				0 000	0 000	0 000	0 000					CTRL cabbage +0 005ppm R
7	S		0 50000	37 5000		35 450								MIXED STD (R,S)
8	X	1				9 040	0 211				100 00	0 211		RS-TRTD A-rep
9	S		1 00000	75 0000		76 110								MIXED STD (R,S)
10	X	1				19 470	0 454				100.00	0 454		RS-TRTD B-rep
11	S		0 20000	15 0000		14 670								MIXED STD (R,S)
12	X	1				0.370	0 009				100 00	0 009		R-TRTD A-rep
13	S		0 05000	3 7500		3 750								MIXED STD (R,S)
14	X	1				0 240	0 005				100 00	0 005		R-TRTD B-rep
15	S		0 00500	0 3750		0 380								MIXED STD (R,S)

Substrates head, 7-day
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 23: RAW DATA WORKSHEET - (R) ENANTIOMER IN CABBAGE HEADS, 7-DAY PHI

Worksheet 6561-44 | Limit of Determ . 0 005 ppm | Extracted 21-Dec-1998 | Product
Ana Set Name | Corr Factor A . 1 0000 | Analyzed | **Analyte .: (R) enantiomer**
Analysis | Corr Factor B 1 0000 | Channel 34 | Project 456-98
Subject cabbage

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-5-A	93 00	10 03	100 00	25 00			2 294	4 0	75 00	43 004
3	S												75 00	
4	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42.965
5	S												75 00	
6	R	1	"	"	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
7	S												75 00	
8	X	1	"	2-5-A	93 00	10.01	100 00	25 00			2 289	4 0	75 00	42 926
9	S												75 00	
10	X	1	"	2-5-B	93 00	10 01	100.00	25 00			2 289	4 0	75 00	42 926
11	S												75 00	
12	X	1	"	3-5-A	93 00	10 05	100 00	25 00			2 298	4 0	75 00	43 083
13	S												75 00	
14	X	1	"	3-5-B	93 00	10 01	100 00	25 00	54 00	42 00	1 781	3 0	75.00	44 516
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 160								MIXED STD (R,S)
2*	C	1				0 060	0 001				81 53	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 420								MIXED STD (R,S)
4	R	1				0 250	0 006	0 004	0 005	88 47				CTRL cabbage +0 005ppm RS
5	S		0 10000	7 5000		7 260								MIXED STD (R,S)
6	R	1				0 220	0 005	0 004	0 005	74 60				CTRL cabbage +0 005ppm R
7	S		0 50000	37 5000		37.010								MIXED STD (R,S)
8	X	1				8 460	0 197				81 53	0 242		RS-TRTD A-rep
9	S		1 00000	75 0000		75 420								MIXED STD (R,S)
10	X	1				18 360	0 428				81 53	0 525		RS-TRTD B-rep
11	S		0 20000	15 0000		14 290								MIXED STD (R,S)
12	X	1				12 360	0 287				81 53	0 352		R-TRTD A-rep
13	S		0 05000	3 7500		3 650								MIXED STD (R,S)
14	X	1				7.590	0 171				81 53	0.209		R-TRTD B-rep
15	S		0 00500	0 3750		0 380								MIXED STD (R,S)

Substrates head, 7-day
B Blank C Control F Freezer R · Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 24: RAW DATA WORKSHEET - (S) ENANTIOMER IN CABBAGE WRAPPER LEAVES, 7-DAY PHI

Worksheet 6561-46
Ana Set Name
Analysis
Subject cabbage

Limit of Determ 0 005 ppm
Corr Factor A 1 0000
Corr Factor B 1 0000

Extracted 22-Dec-1998
Analyzed
Channel 34

Product
Analyte .: (S)-enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-6-A	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
3	S												75 00	
4	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
5	S												75 00	
6	R	1	"	"	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
7	S												75 00	
8	X	1	"	2-6-A	93 00	10 01	100 00	25 00			2 289	10 0	75 00	17 170
9	S												75 00	
10	X	1	"	2-6-B	93 00	10 02	100 00	25 00			2 291	10 0	75 00	17 186
11	S												75 00	
12	X	1	"	3-6-A	93 00	10 04	100 00	25 00			2 296	10 0	75 00	17 217
13	S												75 00	
14	X	1	"	3-6-B	93 00	10 02	100 00	25 00			2 291	10 0	75 00	17 186
15	S												75 00	
16	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 180								MIXED STD (R,S)
2*	C	1				0 000	0 000				100 00	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 580								MIXED STD (R,S)
4	R	1				22.240	0 518	0 518	0 500	103 53				CTRL cabbage +0 50 ppm RS
5	S		0 10000	7 5000		7 890								MIXED STD (R,S)
6	R	1				0 490	0 011	0 011	0 000					CTRL cabbage +0 50 ppm R
7	S		0 50000	37 5000		35 860								MIXED STD (R,S)
8	X	1				10 080	0 587				100 00	0 587		RS-TRTD A-rep
9	S		1 00000	75 0000		75 740								MIXED STD (R,S)
10	X	1				11 290	0 657				100 00	0 657		RS-TRTD B-rep
11	S		0 20000	15 0000		15 170								MIXED STD (R,S)
12	X	1				0 150	0 009				100 00	0.009		R-TRTD A-rep
1	S		0 05000	3 7500		3 840								MIXED STD (R,S)
14	X	1				0 200	0 012				100 00	0 012		R-TRTD B-rep
15	S		0 00500	0 3750		0 360								MIXED STD (R,S)
16	S		0 00150	0 1125		0 100								MIXED STD (R,S)

Substrates wrapper leave,7-day
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 25: RAW DATA WORKSHEET - (R) ENANTIOMER IN CABBAGE WRAPPER LEAVES, 7-DAY PHI

Worksheet 6561-46
Ana Set Name
Analysis
Subject cabbage

Limit of Determ 0 005 ppm
Corr Factor A 1 0000
Corr Factor B 1 0000

Extracted 22-Dec-1998
Analyzed
Channel 34

Product
Analyte .: (R) enantiomer
Project . 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-6-A	93 00	10 00	100 00	25 00			2 287	4 0	75 00	42 887
3	S												75 00	
4	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
5	S												75 00	
6	R	1	"	"	93 00	10 01	100 00	25 00			2 289	4 0	75 00	42 926
7	S												75 00	
8	X	1	"	2-6-A	93 00	10 01	100 00	25 00			2 289	10 0	75 00	17 170
9	S												75 00	
10	X	1	"	2-6-B	93 00	10 02	100 00	25 00			2 291	10 0	75 00	17 186
11	S												75 00	
12	X	1	"	3-6-A	93 00	10 04	100 00	25 00			2 296	10 0	75 00	17 217
13	S												75 00	
14	X	1	"	3-6-B	93 00	10 02	100 00	25 00			2 291	10 0	75 00	17 186
15	S												75 00	
16	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 170								MIXED STD (R,S)
2*	C	1				0 050	0 001				99 61	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 620								MIXED STD (R,S)
4	R	1				22 500	0 524	0 523	0 500	104 50				CTRL cabbage +0 005p, RS
5	S		0 10000	7 5000		8 140								MIXED STD (R,S)
6	R	1				20 380	0 475	0 474	0 500	94 72				CTRL cabbage +0 005ppm R
7	S		0 50000	37 5000		38 940								MIXED STD (R,S)
8	X	1				8 500	0 495				99 61	0 497		RS-TRTD A-rep
9	S		1 00000	75 0000		74 270								MIXED STD (R,S)
10	X	1				9 760	0 568				99 61	0 570		RS-TRTD B-rep
11	S		0 20000	15 0000		14 700								MIXED STD (R,S)
12	X	1				5 640	0 328				99 61	0 329		R-TRTD A-rep
13	S		0 05000	3 7500		3.870								MIXED STD (R,S)
14	X	1				7 260	0 422				99 61	0 424		R-TRTD B-rep
15	S		0 00500	0 3750		0 380								MIXED STD (R,S)
16	S		0 00150	0 1125		0.100								MIXED STD (R,S)

Substrates wrapper leave,7-day

B Blank C Control F Freezer R Recovery

* Control used for this group Negative controls are treated as 0 0

Notes

S Standard V Solvent X Sample

Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 26: RAW DATA WORKSHEET - (S) ENANTIOMER IN TRIMMED CABBAGE HEADS, 7-DAY PHI

Worksheet 6561-42 | Limit of Determ 0 005 ppm | Extracted 17-Dec-1998 | Product
Ana Set Name | Corr Factor A 1 0000 | Analyzed | **Analyte .: (S)-enantiomer**
Analysis | Corr Factor B . 1 0000 | Channel 34 | Project 456-98
Subject cabbage

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-7-A	93 00	10 05	100.00	25.00			2 298	4 0	75 00	43 083
3	S												75 00	
4	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
5	S												75 00	
6	R	1	"	"	93 00	10.02	100.00	25 00			2 291	4 0	75 00	42 965
7	S												75 00	
8	X	1	"	2-7-A	93 00	10 04	100 00	25 00			2 296	4 0	75 00	43 043
9	S												75 00	
10	X	1	"	2-7-B	93 00	10 04	100 00	25 00			2 296	4 0	75 00	43 043
11	S												75 00	
12	X	1	"	3-7-A	93 00	10 04	100 00	25 00			2 296	4 0	75 00	43 043
13	S												75 00	
14	X	1	"	3-7-B	93 00	10 01	100 00	25 00			2 289	4.0	75 00	42 926
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 220								MIXED STD (R,S)
2*	C	1				0 040	0 001				93 15	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 680								MIXED STD (R,S)
4	R	1				0 240	0 006	0 005	0 005	93 15				CTRL cabbage +0 005ppm RS
5	S		0 10000	7 5000		7.230								MIXED STD (R,S)
6	R	1				0 000	0 000	-0 001	0 000					CTRL cabbage +0 005ppm R
7	S		0 50000	37 5000		36 880								MIXED STD (R,S)
8	X	1				1 050	0 024				93 15	0 026		RS-TRTD A-rep
9	S		1 00000	75 0000		75 230								MIXED STD (R,S)
10	X	1				1 470	0 034				93 15	0.037		RS-TRTD B-rep
11	S		0 20000	15 0000		15 550								MIXED STD (R,S)
12	X	1				0 000	0 000				93 15	<0 005		R-TRTD A-rep
13	S		0 05000	3 7500		3 650								MIXED STD (R,S)
14	X	1				0 000	0 000				93 15	<0 005		R-TRTD B-rep
15	S		0 00500	0 3750		0 260								MIXED STD (R,S)

Substrates trimmed head, 7-day
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 27: RAW DATA WORKSHEET - (R) ENANTIOMER IN TRIMMED CABBAGE HEADS, 7-DAY PHI

Worksheet 6561-42 | Limit of Determ 0 005 ppm | Extracted 17-Dec-1998 | Product
Ana Set Name | Corr Factor A 1 0000 | Analyzed | **Analyte .: (R) enantiomer**
Analysis | Corr Factor B 1 0000 | Channel 34 | Project 456-98
Subject cabbage

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-7-A	93 00	10 05	100 00	25 00			2 298	4 0	75 00	43 083
3	S												75 00	
4	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
5	S												75 00	
6	R	1	"	"	93 00	10 02	100 00	25 00			2 291	4 0	75 00	42 965
7	S												75.00	
8	X	1	"	2-7-A	93 00	10 04	100 00	25 00			2 296	4 0	75 00	43 043
9	S												75 00	
10	X	1	"	2-7-B	93 00	10 04	100 00	25 00			2 296	4 0	75 00	43 043
11	S												75 00	
12	X	1	"	3-7-A	93 00	10 04	100 00	25 00			2 296	4 0	75 00	43 043
13	S												75 00	
14	X	1	"	3-7-B	93 09	10 01	100 00	25 00			2 289	4 0	75 00	42 926
15	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 220								MIXED STD (R,S)
2*	C	1				0 030	0 001				100 00	<0 005		CTRL cabbage
3	S		0 02000	1 5000		1 740								MIXED STD (R,S)
4	R	1				0 280	0 007	0 006	0 005	116 41				CTRL cabbage +0 005ppm RS
5	S		0 10000	7 5000		7 800								MIXED STD (R,S)
6	R	1				0 250	0 006	0 005	0 005	102 45				CTRL cabbage +0 005ppm R
7	S		0 50000	37 5000		37.530								MIXED STD (R,S)
8	X	1				1 020	0 024				100 00	0 024		RS-TRTD A-rep
9	S		1 00000	75 0000		74 930								MIXED STD (R,S)
10	X	1				1 420	0 033				100 00	0 033		RS-TRTD B-rep
11	S		0 20000	15 0000		15 200								MIXED STD (R,S)
12	X	1				0 730	0 017				100 00	0 017		R-TRTD A-rep
13	S		0 05000	3 7500		3 420								MIXED STD (R,S)
14	X	1				0 890	0 021				100 00	0 021		R-TRTD B-rep
15	S		0 00500	0 3750		0 250								MIXED STD (R,S)

Substrates trimmed head, 7-day
B Blank C Control F Freezer R Recovery
* Control used for this group Negative controls are treated as 0 0

Notes
S Standard V Solvent X Sample
Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 28: RAW DATA WORKSHEET - (S) ENANTIOMER IN POTATO FOLIAGE

Worksheet	6561-48	Limit of Determ	0 005 ppm	Extracted	22-Dec-1998
Ana Set Name		Corr Factor A	1 0000	Analyzed	
Analysis		Corr Factor B .	1 0000	Channel	34
Subject	potato				

Product
Analyte .: (S)-enantiomer
Project 456-98

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj ~ mg
1	S												75 00	
2*	C	1	RIMV01298	1-2-A	15.00	10 02	100 00	25 00			2 468	4 0	75.00	46 273
3	R	1	"	"	15 00	10 02	100 00	25 00			2 468	4 0	75.00	46 273
4	S												75.00	
5	R	1	"	"	15 00	10 01	100 00	25 00			2 465	4 0	75 00	46 228
6	S												75 00	
7	X	1	"	2-2-A	15 00	10.01	100.00	25 00			2 465	4 0	75 00	46 228
8	X	1	"	2-2-B	15 00	9 99	100 00	25 00			2 461	4 0	75.00	46 137
9	S												75 00	
10	X	1	"	3-2-A	15 00	10.01	100 00	25 00			2 465	4 0	75 00	46 228
11	X	1	"	3-2-B	15 00	10 01	100 00	25 00			2 465	4 0	75 00	46 228
12	S												75 00	
13	X	1	"	4-2-A	15 00	10 05	100 00	25 00			2 475	4 0	75 00	46 410
14	X	1	"	4-2-B	15 00	10 05	100 00	25 00			2 475	4 0	75 00	46 410
15	S												75.00	
16	X	1	"	5-2-A	15 00	9 99	100 00	25 00			2 461	4 0	75 00	46 137
17	X	1	RIMV01298	5-2-B	15 00	10 00	100 00	25 00			2 463	4 0	75 00	46 182
18	S												75 00	
19	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 120								MIXED STD (R,S)
2*	C	1				0 010	0 000				94 74	<0 005		CTRL potato
3	R	1				21 930	0 474	0 474	0 500	94 74				CTRL potato +0 50 ppm RS
4	S		0 02000	1 5000		1 570								MIXED STD (R,S)
5	R	1				0 390	0 008	0 008	0 000					CTRL potato +0 50 ppm R
6	S		0 10000	7 5000		8 010								MIXED STD (R,S)
7	X	1				37 720	0 816				94 74	0 861		RS-TRTD A-rep
8	X	1				40 180	0 871				94 74	0 919		RS-TRTD B-rep
9	S		0 50000	37 5000		39 820								MIXED STD (R,S)
10	X	1				0 000	0 000				94 74	<0 005		R-TRTD A-rep
11	X	1				0 450	0 010				94 74	0 010		R-TRTD B-rep
12	S		1 00000	75 0000		73 600								MIXED STD (R,S)
13	X	1				41 670	0 898				94 74	0 948		RS-TRTD A-rep
14	X	1				40 450	0 872				94 74	0 920		RS-TRTD B-rep
15	S		0 20000	15 0000		15,820								MIXED STD (R,S)
16	X	1				0 430	0 009				94 74	0 010		R-TRTD A-rep
17	X	1				0 270	0 006				94 74	0.006		R-TRTD B-rep
18	S		0 05000	3 7500		4 170								MIXED STD (R,S)
19	S		0 00500	0 3750		0 450								MIXED STD (R,S)

Substrates foliage Notes moisture, no reference available, used value for PEANUT HAY (similar dry substrate) OPPTS 860 1000, Table 1
B Blank C Control F Freezer R Recovery S Standard V Solvent X Sample
* Control used for this group Negative controls are treated as 0 0 Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 29. RAW DATA WORKSHEET - (R) ENANTIOMER IN POTATO FOLIAGE

Worksheet 6561-48 | Limit of Determ 0 005 ppm | Extracted 22-Dec-1998 | Product
Ana Set Name | Corr Factor A 1 0000 | Analyzed | **Analyte .: (R) enantiomer**
Analysis | Corr Factor B 1 0000 | Channel 34 | Project 456-98
Subject potato

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	1-2-A	15 00	10 02	100 00	25 00			2 468	4 0	75 00	46 273
3	R	1	"	"	15 00	10 02	100 00	25 00			2 468	4 0	75 00	46 273
4	S												75 00	
5	R	1	"	"	15 00	10 01	100 00	25 00			2 465	4 0	75 00	46 228
6	S												75 00	
7	X	1	"	2-2-A	15 00	10 01	100 00	25 00			2 465	4 0	75 00	46 228
8	X	1	"	2-2-B	15 00	9 99	100 00	25 00			2 461	4 0	75 00	46 137
9	S												75 00	
10	X	1	"	3-2-A	15 00	10 01	100 00	25 00			2 46	4 0	75 00	46 228
11	X	1	"	3-2-B	15 00	10 01	100 00	25 00			2 46	4 0	75 00	46 228
12	S												75 00	
13	X	1	"	4-2-A	15 00	10 05	100 00	25 00			2 475	4 0	75 00	46 410
14	X	1	"	4-2-B	15 00	10 05	100 00	25 00			2 475	4 0	75 00	46 410
15	S												75 00	
16	X	1	"	5-2-A	15 00	9 99	100 00	25 00			2 461	4 0	75 00	46 137
17	X	1	RIMV01298	5-2-B	15 00	10 00	100 00	25 00			2 463	4 0	75 00	46 182
18	S												75 00	
19	S												75.00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 120								MIXED STD (R,S)
2*	C	1				0 120	0 003				96 87	<0 005		CTRL potato
3	R	1				21 990	0 475	0 473	0 500	94 53				CTRL potato +0 50 ppm RS
4	S		0 02000	1 5000		1 690								MIXED STD (R,S)
5	R	1				23 050	0 499	0 496	0 500	99 20				CTRL potato +0 50 ppm R
6	S		0 10000	7 5000		9 750								MIXED STD (R,S)
7	X	1				35 080	0 759				96 87	0 783		RS-TRTD A-rep
8	X	1				40 740	0 883				96 87	0 912		RS-TRTD B-rep
9	S		0 50000	37 5000		38 780								MIXED STD (R,S)
10	X	1				0 070	0 002				96 87	<0 005		R-TRTD A-rep
11	X	1				16 920	0 366				96 87	0 378		R-TRTD B-rep
12	S		1 00000	75 0000		74 000								MIXED STD (R,S)
13	X	1				38 350	0 826				96 87	0 853		RS-TRTD A-rep
14	X	1				41 500	0 894				96 87	0 923		RS-TRTD B-rep
15	S		0 20000	15 0000		15 470								MIXED STD (R,S)
16	X	1				21 230	0 460				96 87	0 475		R-TRTD A-rep
17	X	1				14 260	0 309				96 87	0 319		R-TRTD B-rep
18	S		0 05000	3 7500		4 340								MIXED STD (R,S)
19	S		0 00500	0 3750		0 630								MIXED STD (R,S)

Substrates foliage Notes moisture, no reference available, used value for PEANUT HAY (similar dry substrate) OPPTS 860 1000, Ta ble 1
B Blank C Control F Freezer R Recovery S Standard V Solvent X Sample
* Control used for this group Negative controls are treated as 0 0 Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 30. RAW DATA WORKSHEET - (S) ENANTIOMER IN LETTUCE HEADS

Worksheet	6561-54	Limit of Determ	0.005 ppm	Extracted	4-Jan-1999	Product
Ana Set Name		Corr Factor A	1 0000	Analyzed		**Analyte .: (S)-enantiomer**
Analysis	6561-54	Corr Factor B	1 0000	Channel	34	Project 456-98
Subject	lettuce					

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	CONTROL	96 00	10 00	100 00	25 00			2 281	4 0	75 00	42 769
3	B	1	"	REAG BLK	96 00	10 00	100 00	25 00			2 281	4 0	75 00	42 769
4	S												75 00	
5	R	1	"	CONTROLREC	96 00	10 04	100 00	25 00			2 289	4 0	75 00	42 925
6	S												75 00	
7	R	1	"	"	96 00	10 02	100 00	25 00			2 285	4 0	75 00	42 847
8	S												75 00	
9	X	1	"	SUBPR1INT1	96 00	10 09	100 00	25 00			2 300	40 0	75 00	4 312
10	S												75 00	
11	X	1	"	SUBPR1INT2	96 00	10.16	100 00	25 00			2 314	4 0	75 00	43 393
12	S												75 00	
13	X	1	"	SUBPR1INT3	96 00	10 00	100 00	25 00			2 281	4 0	75.00	42 769
14	S												75 00	
15	X	1	"	SUBPR2INT1	96 00	9 94	100 00	25 00			2 269	40 0	75 00	4 253
16	S												75 00	
17	X	1	RIMV01298	SUBPR2INT2	96 00	10.02	100 00	25 00			2 285	4 0	75.00	42 847
18	S												75 00	
19	X	1	"	SUBPR2INT3	96 00	9 94	100 00	25 00			2 269	4 0	75 00	42 535
20	S												75 00	

Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 230								MIXED STD (R,S)
2*	C	1				0 010	0 000				100 00	<0 005		CTRL lettuce
3	B	1				0 000	0 000				100 00	<0 005		REAG BLK
4	S		0 02000	1 5000		1 820								MIXED STD (R,S)
5	R	1				23 530	0 548	0 548	0 500	109 59				CTRL lettuce +0 500ppm RS
6	S		0 10000	7 5000		8 760								MIXED STD (R,S)
7	R	1				0 950	0 022	0 022	0 000					CTRL lettuce +0 500ppm R
8	S		0 50000	37 5000		39 870								MIXED STD (R,S)
9	X	1				0 590	0 137				100.00	0 137		14C-(R) TREATED, 0-DAY
10	S		0 20000	15 0000		16 960								MIXED STD (R,S)
11	X	1				0.270	0 006				100 00	0 006		14C-(R) TREATED, 14-DAY
12	S		0 05000	3 7500		4 590								MIXED STD (R,S)
13	X	1				0 030	0 001				100 00	<0 005		14C-(R) TREATED, 21-DAY
14	S		1 00000	75 0000		72 890								MIXED STD (R,S)
15	X	1				12 2[illegible]	2 868				100.00	2 868		14C-(RS) TREATED, 0-DAY
16	S		0 20000	15 0000		16 6[illegible]								MIXED STD (R,S)
17	X	1				7 090	0 165				100 00	0 165		14C-(RS) TREATED, 14-DAY
18	S		0 05000	3 7500		4 270								MIXED STD (R,S)
19	X	1				2 010	0 047				100 00	0 047		14C-(RS) TREATED, 21-DAY
20	S		0 00500	0 3750		0 420								MIXED STD (R,S)

Substrates head, 14C-treated

Notes

B Blank C Control F Freezer R Recovery S · Standard V Solvent X Sample

* Control used for this group Negative controls are treated as 0 0 Underline, Strikethrough Manual override, ~~Rejected~~

TABLE 31. RAW DATA WORKSHEET - (R) ENANTIOMER IN LETTUCE HEADS

Worksheet 6561-54 | Limit of Determ 0 005 ppm | Extracted 4-Jan-1999 | Product
Ana Set Name | Corr Factor A 1 0000 | Analyzed | **Analyte .: (R) enantiomer**
Analysis 6561-54 | Corr Factor B 1 0000 | Channel 34 | Project 456-98
Subject lettuce

Bt Nm	T	G	Field Test	Smp Code	Moist %	Smp wt ext g	Ext vol ml	Aliq 1 vol ml	Interm vol ml	Aliq 2 vol ml	Final wt g	Final vol ml	Inj vol ul	Smp Wt Inj mg
1	S												75 00	
2*	C	1	RIMV01298	CONTROL	96 00	10 00	100 00	25 00			2 281	4 0	75 00	42 769
3	B	1	"	REAG BLK	96 00	10 00	100 00	25 00			2 281	4 0	75 00	42 769
4	S												75.00	
5	R	1	"	CONTROLREC	96 00	10 04	100 00	25 00			2 289	4 0	75 00	42 925
6	S												75 00	
7	R	1	"	"	96.00	10 02	100 00	25 00			2 285	4 0	75 00	42 847
8	S												75 00	
9	X	1	"	SUBPR1INT1	96 00	10 09	100 00	25 00			2 300	40 0	75 00	4 312
10	S												75.00	
11	X	1	"	SUBPR1INT2	96 00	10 16	100 00	25 00			2 314	4 0	75 00	43 393
12	S												75 00	
13	X	1	"	SUBPR1INT3	96 00	10 00	100 00	25.00			2 281	4 0	75 00	42 769
14	S												75 00	
15	X	1	"	SUBPR2INT1	96 00	9 94	100 00	25.00			2 269	40 0	75 00	4 253
16	S												75.00	
17	X	1	RIMV01298	SUBPR2INT2	96 00	10 02	100 00	25 00			2 285	4 0	75.00	42 847
18	S												75.00	
19	X	1	"	SUBPR2INT3	96 00	9 94	100 00	25 00			2 269	4 0	75 00	42 535
20	S												75 00	

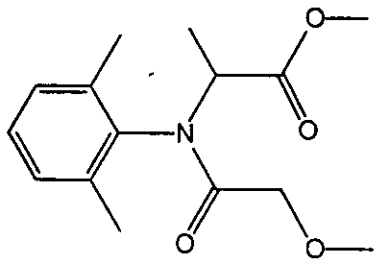
Bt Nm	T	G	Std soln ng/ul	Std Wt Inj ng	Peak Height uV	Analyte Found ng	Residue Uncorr ppm	Value corrt for control	Added Amount ppm	Recov %	Avg Recov %	Corrt Residue ppm	Dry Wt/ (Soil) ppm	Comments
1	S		0 00250	0 1875		0 210								MIXED STD (R,S)
2*	C	1				0 000	0 000				100 00	<0.005		CTRL lettuce
3	B	1				0 000	0 000				100 00	<0 005		REAG BLK
4	S		0 02000	1 5000		1.890								MIXED STD (R,S)
5	R	1				22 140	0 516	0 516	0 500	103 16				CTRL lettuce +0 500ppm RS
6	S		0 10000	7 5000		8 850								MIXED STD (R,S)
7	R	1				21 860	0 510	0 510	0 500	102 04				CTRL lettuce +0 500ppm R
8	S		0 50000	37 5000		39.910								MIXED STD (R,S)
9	X	1				27 550	6 389				100 00	6 389		14C-(R) TREATED, 0-DAY
10	S		0 20000	15 0000		16 940								MIXED STD (R,S)
11	X	1				6.260	0 144				100 00	0 144		14C-(R) TREATED, 14-DAY
12	S		0 05000	3 7500		4 450								MIXED STD (R,S)
13	X	1				0 830	0 019				100 00	0 019		14C-(R) TREATED, 21-DAY
14	S		1 00000	75 0000		73 030								MIXED STD (R,S)
15	X	1				10 950	2 574				100 00	2 574		14C-(RS) TREATED, 0-DAY
16	S		0 20000	15 0000		15 870								MIXED STD (R,S)
17	X	1				3 840	0 090				100 00	0 090		14C-(RS) TREATED, 14-DAY
18	S		0 05000	3 7500		4 170								MIXED STD (R,S)
19	X	1				1 080	0 025				100 00	0 025		14C-(RS) TREATED, 21-DAY
20	S		0 00500	0 3750		0 430								MIXED STD (R,S)

Substrates head, 14C-treated

Notes
B Blank C Control F Freezer R Recovery S Standard V Solvent X Sample
* Control used for this group Negative controls are treated as 0 0 Underline, Strikethrough Manual override, ~~Rejected~~

FIGURE 1. STRUCTURES AND CHEMICALS NAMES

Common Name: Metalaxyl also (RS) Racemate
Novartis Number: CGA-48988
Chemical Name: N-(2,6-dimethylphenyl)-N-(methoxyacetyl)alanine methyl ester
CAS No.: 57837-19-1
Molecular Weight: 279.3 g/mol
Structure:



Common Name: Mefenoxam also (R) Enantiomer
Novartis Number: CGA-329351
Chemical Name: (R)-2-[(2,6-dimethylphenyl)-methoxyacetylamino]-propionic acid methyl ester
CAS No.: 70630-17-0
Molecular Weight: 279.3 g/mol
Structure:

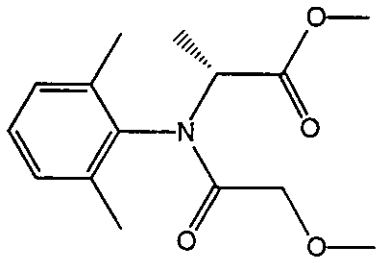


FIGURE 2: PROCEDURAL FLOWCHART FOR STUDY NUMBER 456-98 ANALYTICAL METHOD

EXTRACTION

Weigh a 10-g representative sample.

↓

Reflux for 2 hours in 100 mL methanol:water (80:20).

↓

Filter sample through Whatman 2V filter paper.

↓

Measure 25 mL extract for analysis

C18 SEPPAK CLEANUP

Condition C18 SepPak with 5 mL methanol:water (80:20)

↓

Pass 25 mL sample aliquot through C18 SepPak into 125-mL boiling flask.

↓

Rotary evaporate to aqueous (<35°C).

LIQUID-LIQUID PARTITION

Add 25 mL water and 1 mL 0.10N HCl. Sonicate & swirl to homogenize.

↓

Transfer acidified aqueous extract to a 60-mL separatory funnel.

↓

Wash round bottom flask with 10 mL DCM and transfer to separatory funnel.

↓

Partition for 30 seconds. Pass extract through a 10 gram pad of Na_2SO_4. Collect into a 125-mL boiling flask.

↓

(Repeat partition 3 times – total 4 DCM partitions)

↓

Rinse Na_2SO_4 pad with DCM.

↓

Rotary evaporate to dryness (<35°C). Reconstitute in 5 mL DCM.

↓

(continued next page)

FIGURE 2: PROCEDURAL FLOWCHART FOR STUDY NUMBER 456-98 ANALYTICAL METHOD (Continued)

SILICA SEPPAK CLEANUP

Condition Silica SepPak with 10 mL DCM.

↓

Load the sample extract onto the Silica SepPak; discard the eluate.

↓

Rinse the round bottom flask with 10 mL of DCM and add to the SepPak; discard the eluate.

↓

Rinse the round bottom flask with 10 mL of 5% ethyl ether:hexane and add to the SepPak; discard the eluate.

↓

Elute the Silica SepPak with 20 mL of 40% ethyl acetate:hexane, collecting the eluate into a 50-mL concentration tube.

↓

Evaporate to dryness (<35°C).

↓

Reconstitute to desired final volume (≥2.0 mL) with ACN/HOH/TFA (50:50:0.05). Sonicate to dissolve any residue.

↓

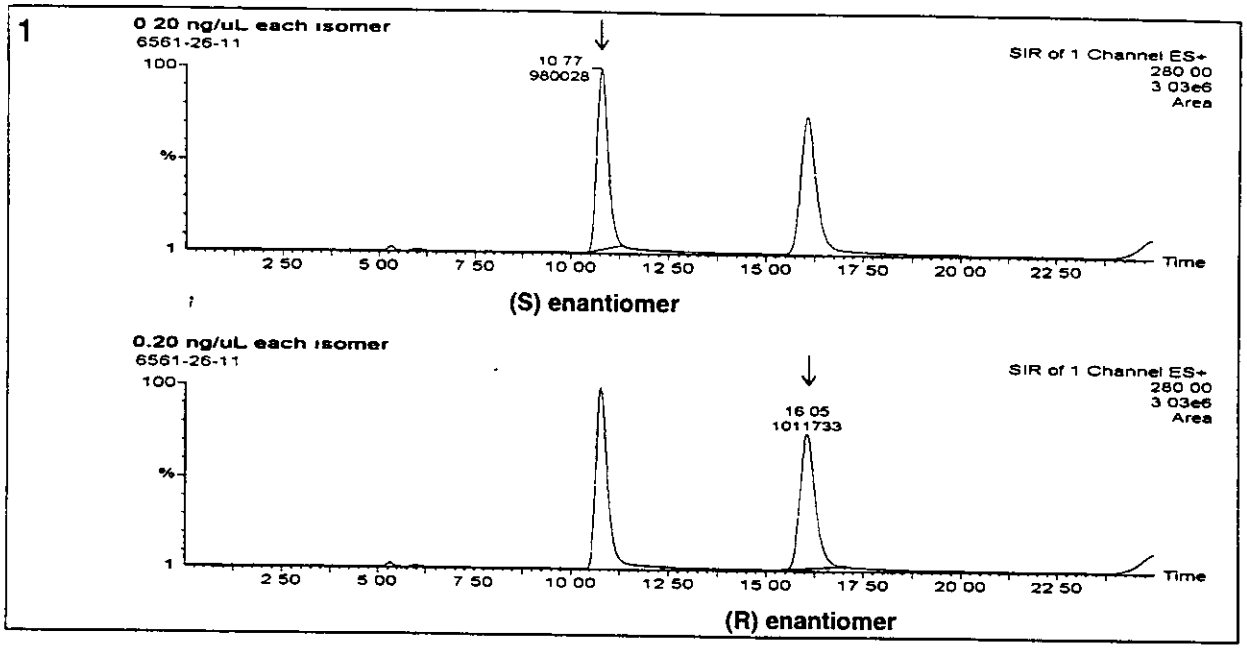
Optional: Filter extract through a 0.45 µm syringe filter directly into an autosampler vial.

↓

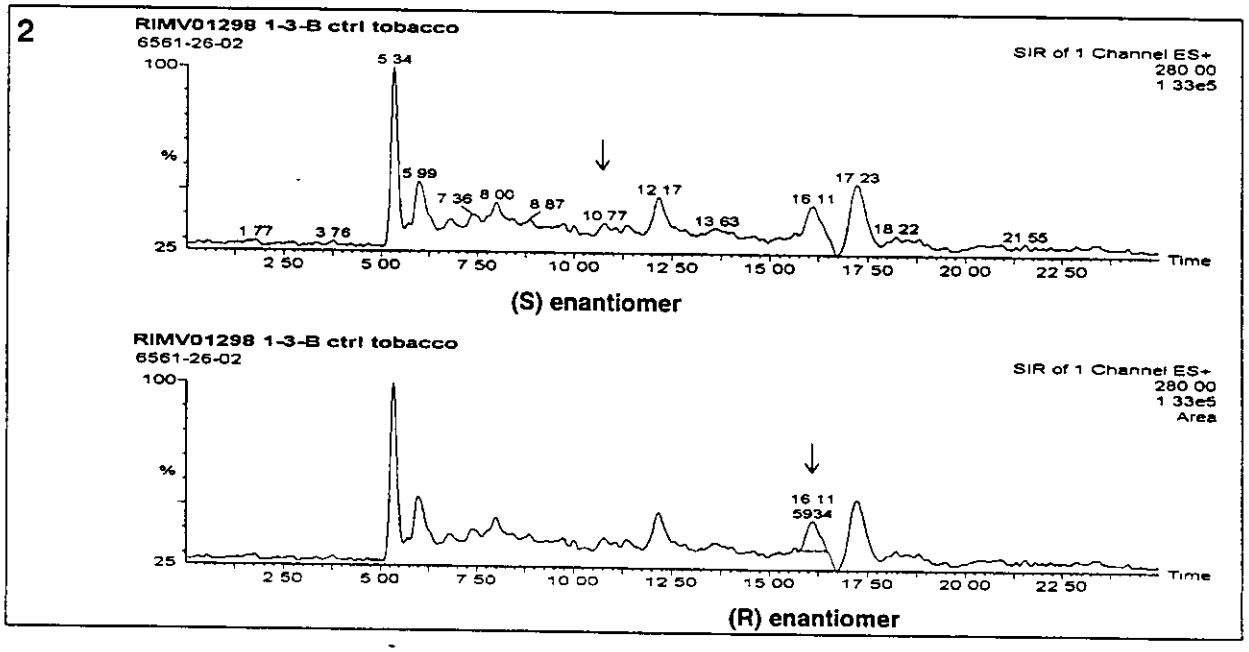
LC/MS ANALYSIS

Analyze the sample by chiral LC/MS in positive ion electrospray mode, acquiring the [M+1] ion at m/z=280.

FIGURE 3: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN GREEN TOBACCO LEAF

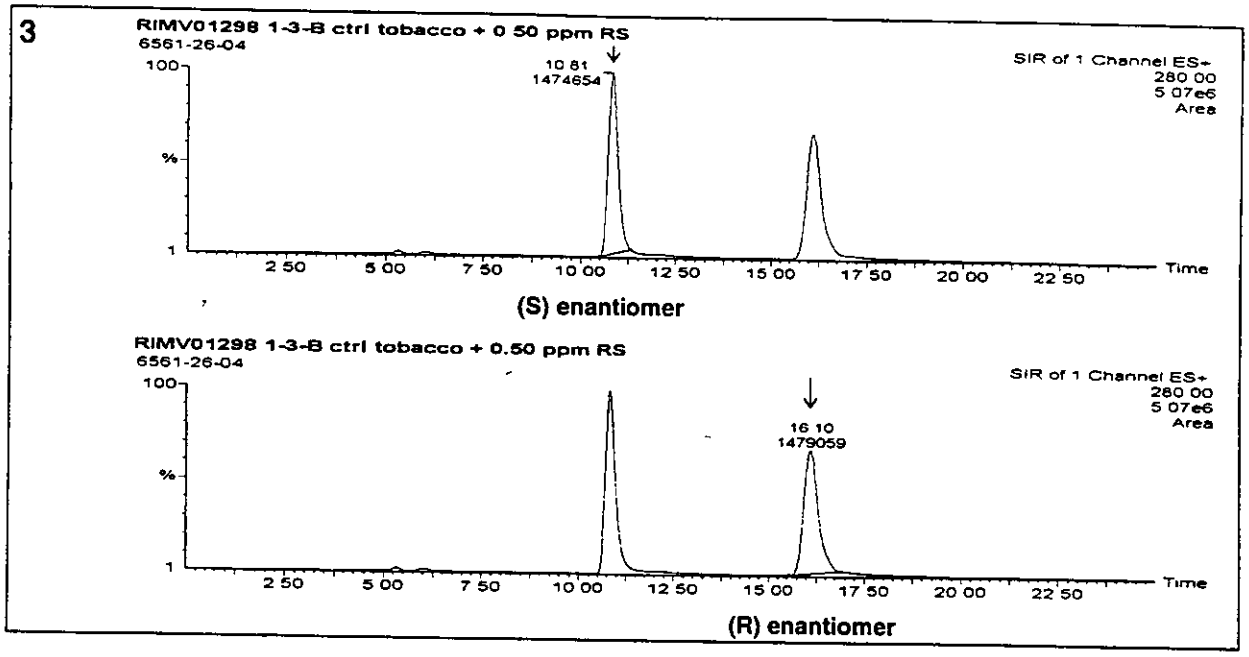


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) enantiomer			(R) enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 200	15 0	66125.6	0	0 9998	67329 9	0	0.9999

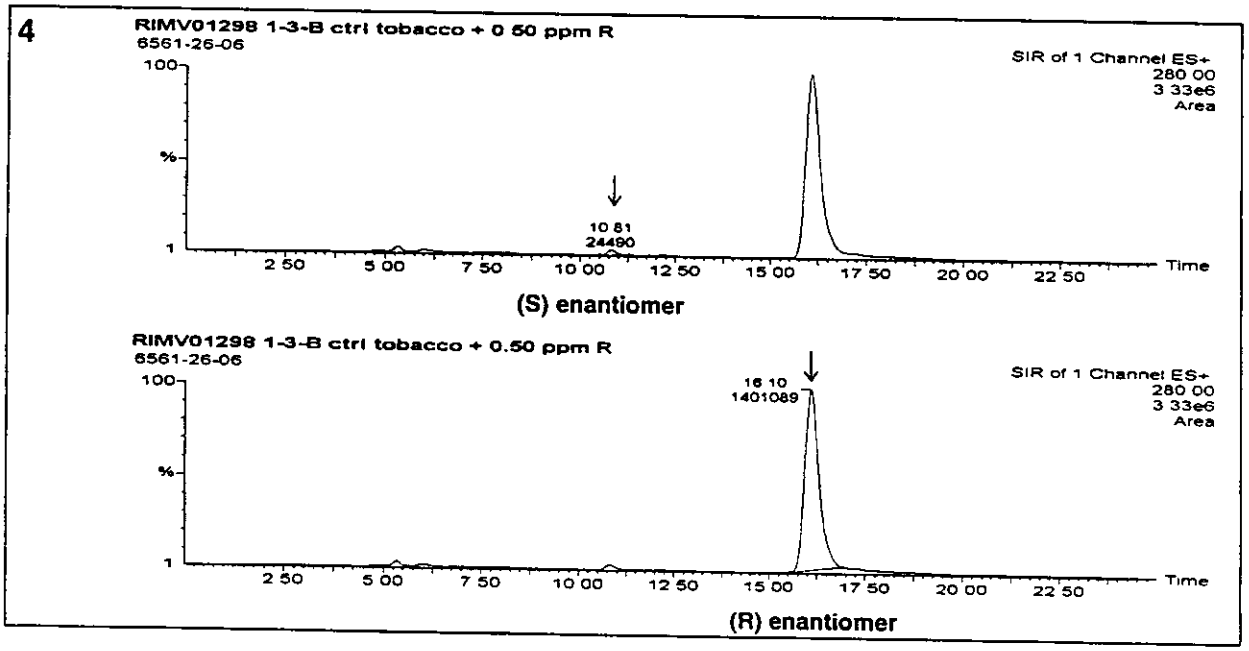


Sample Code	Desc	mg injected	(S) enantiomer ng	ppm uncorr	ppm	(R) enantiomer ng	ppm uncorr	ppm
1-3-B	control	43 083	0 000	0 000	<0 005	0.090	0.002	<0 005

FIGURE 3: CHROMATOGRAMS.
(S) AND (R) ENANTIOMERS IN GREEN TOBACCO LEAF (Continued)

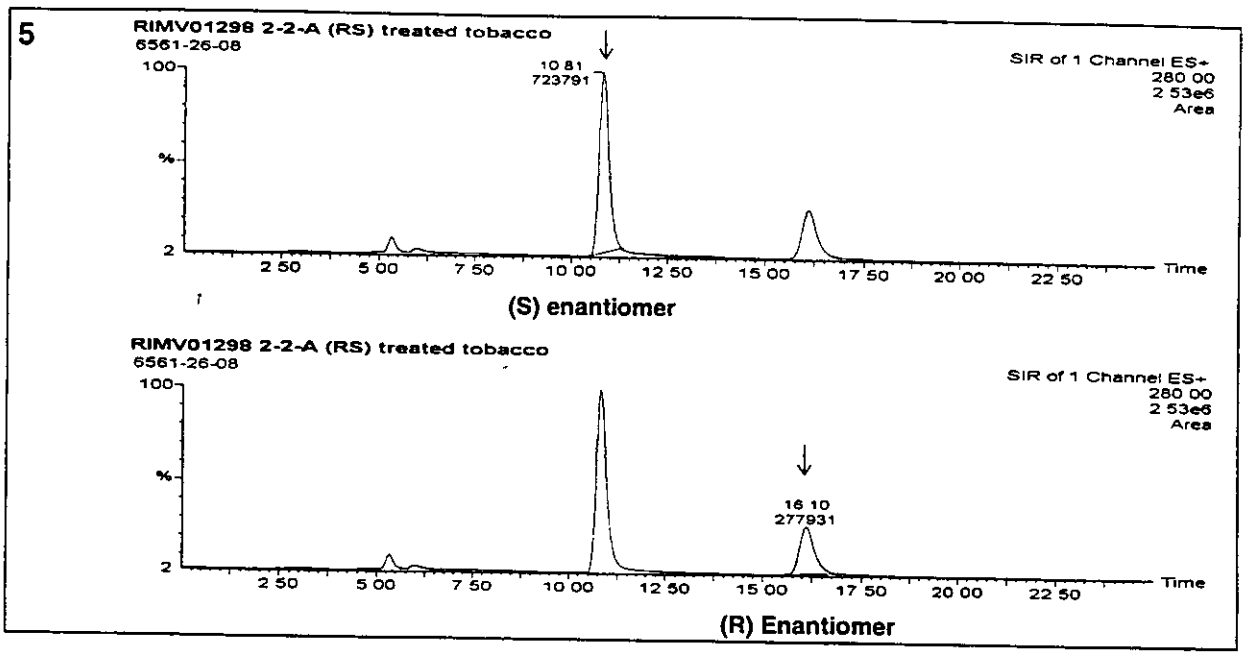


Sample Code	Desc.	Amt. (S), (R) Added	mg injected	(S) enantiomer ng	(S) enantiomer ppm uncorr	(S) enantiomer % Rec.	(R) enantiomer ng	(R) enantiomer ppm uncorr	(R) enantiomer % Rec
1-3-B	recovery	0.50 ppm	43 123	22 300	0.517	103%	21 970	0.509	101%

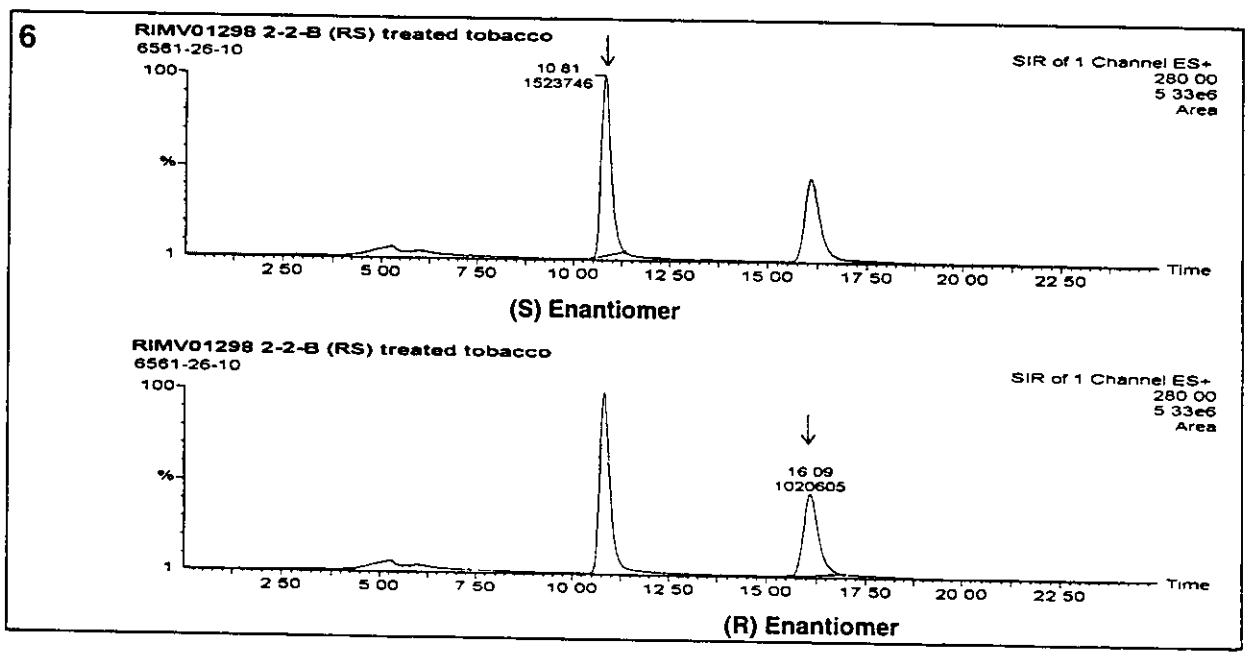


Sample Code	Desc	Amt. (S), (R) Added	mg injected	(S) enantiomer ng	(S) enantiomer ppm uncorr	(S) enantiomer % Rec	(R) enantiomer ng	(R) enantiomer ppm uncorr	(R) enantiomer % Rec
1-3-B	recovery	0 50 ppm	43 083	0.370	0 009	-	20.810	0 483	96%

FIGURE 3: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN GREEN TOBACCO LEAF (Continued)

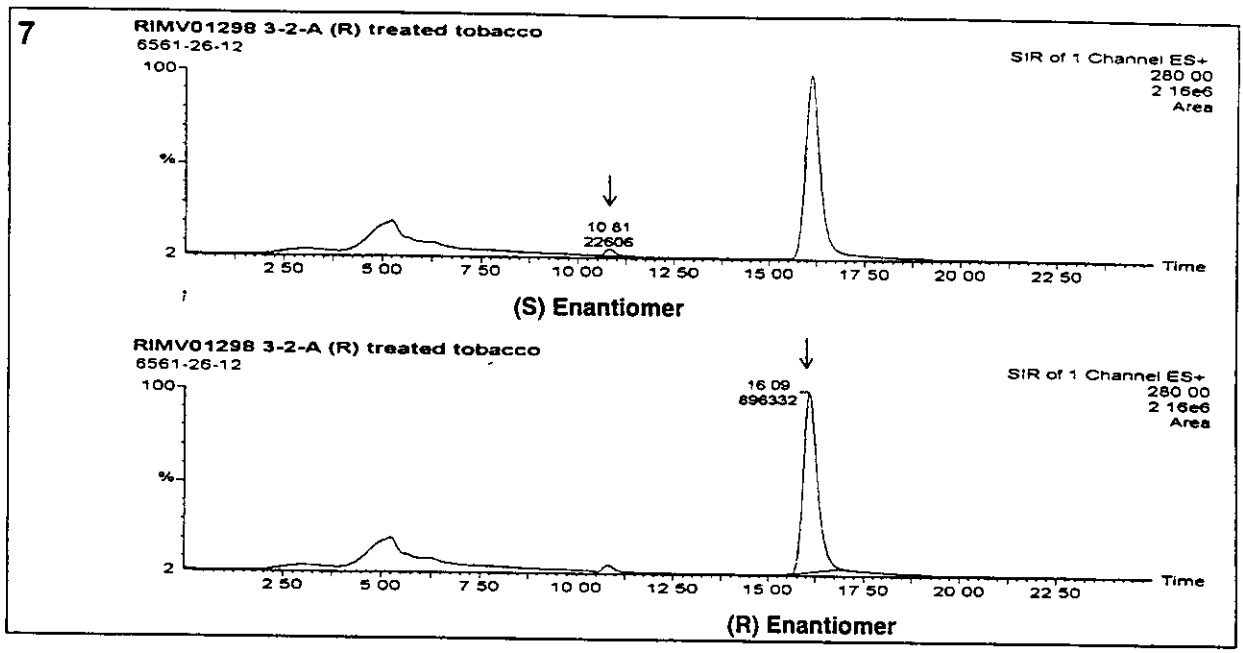


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-2-A	(RS)-treated	0	43 083	10.950	0 254	4 130	0.097	0.351

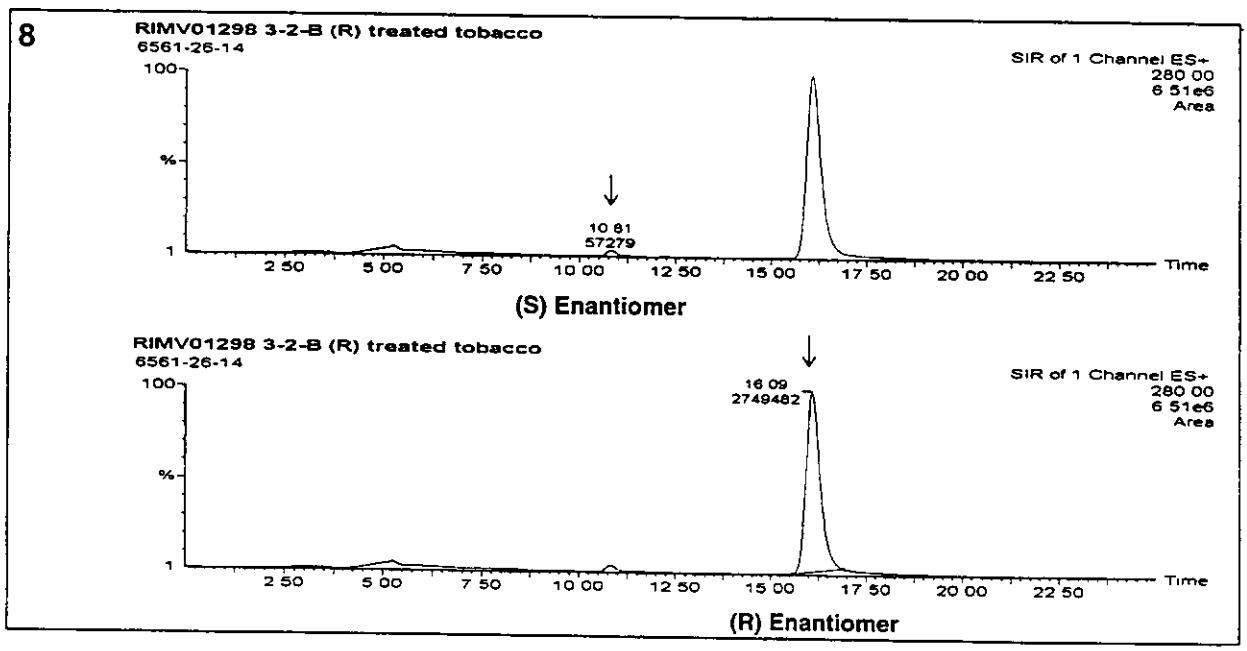


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr.	(RS) Racemate ppm
2-2-B	(RS)-treated	0	43.083	23.040	0 535	15.160	0 356	0.891

FIGURE 3: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN GREEN TOBACCO LEAF (Continued)

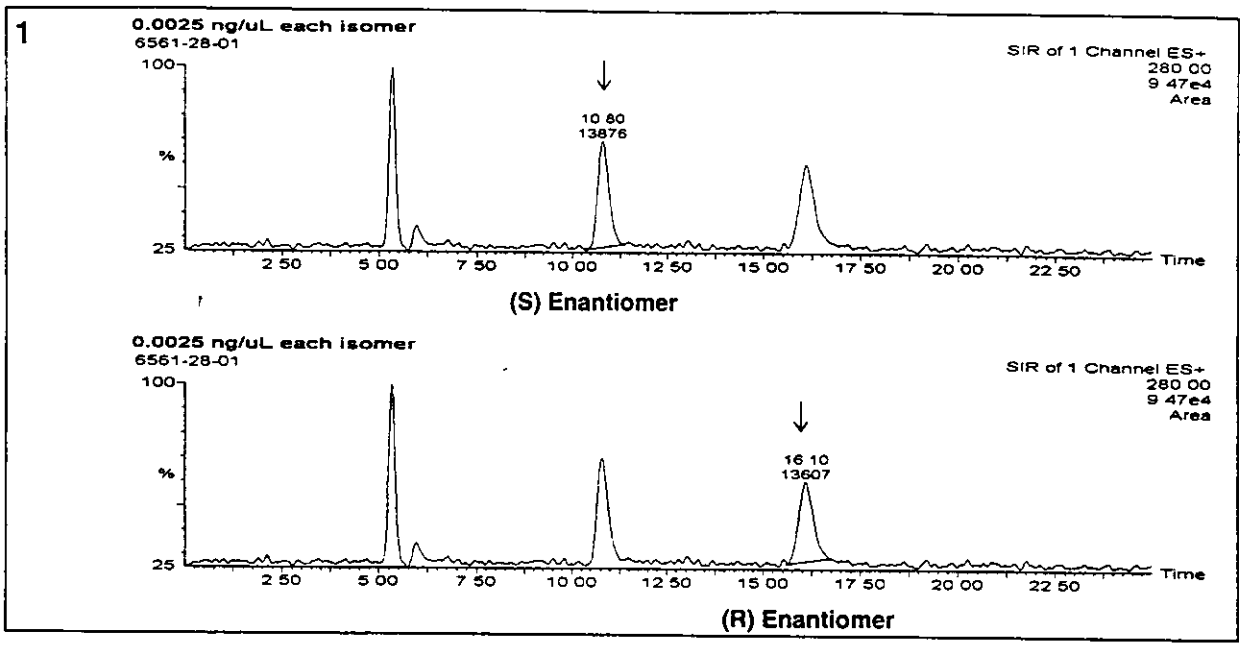


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr.	(RS) Racemate ppm
3-2-A	(R)-treated	0	43 162	0 340	0.008	13 310	0 312	0 320

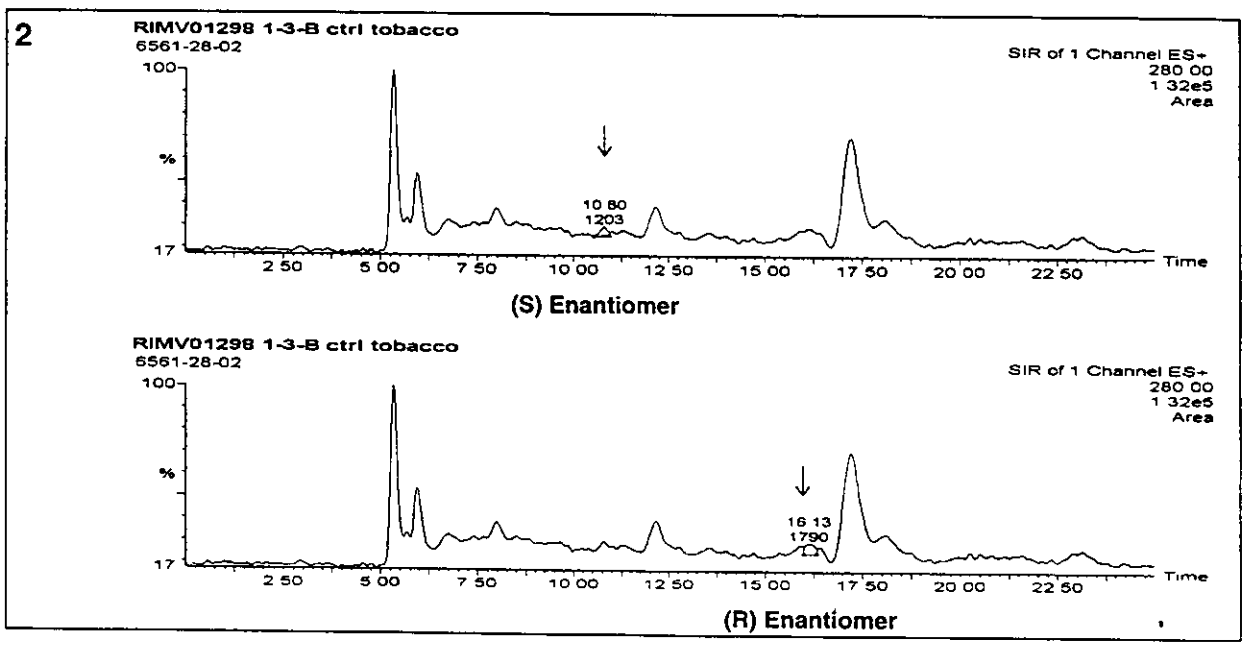


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-2-B	(R)-treated	0	43 162	0 870	0 020	40.840	0.957	0.977

FIGURE 4: CHROMATOGRAMS.
(S) AND (R) ENANTIOMERS IN MATURE TOBACCO LEAF

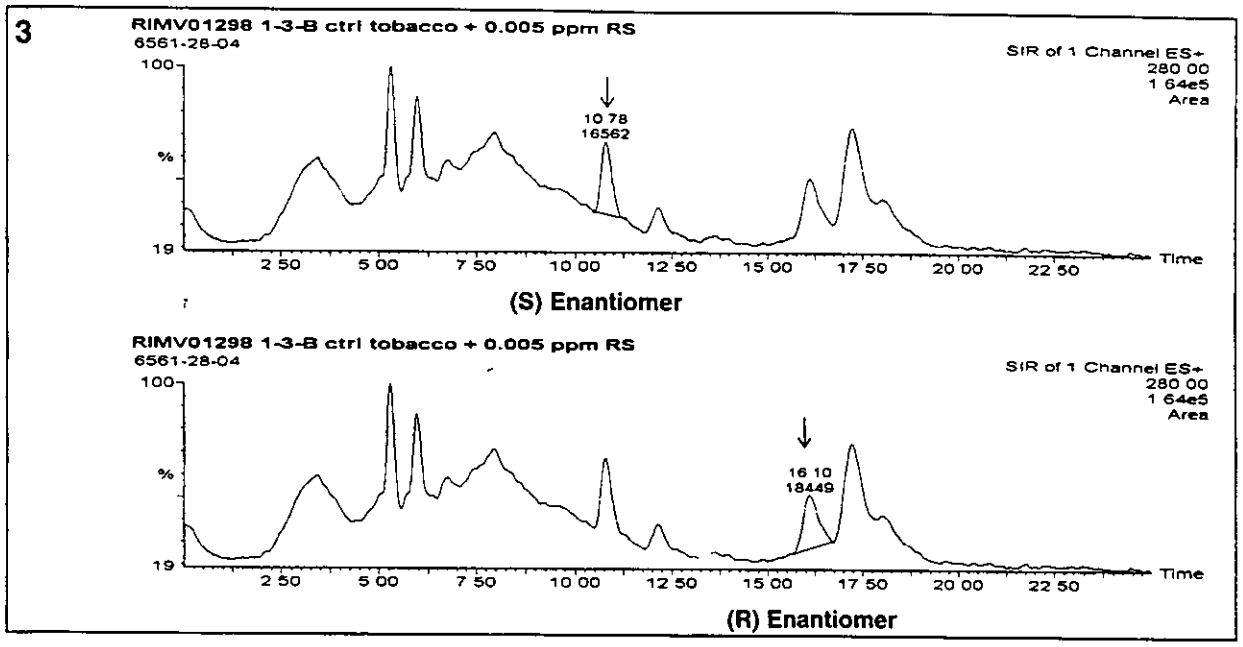


Standard	Concentration	Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 0025	0 1875	77665.3	0	0.9999	81437 0	0	0.9999

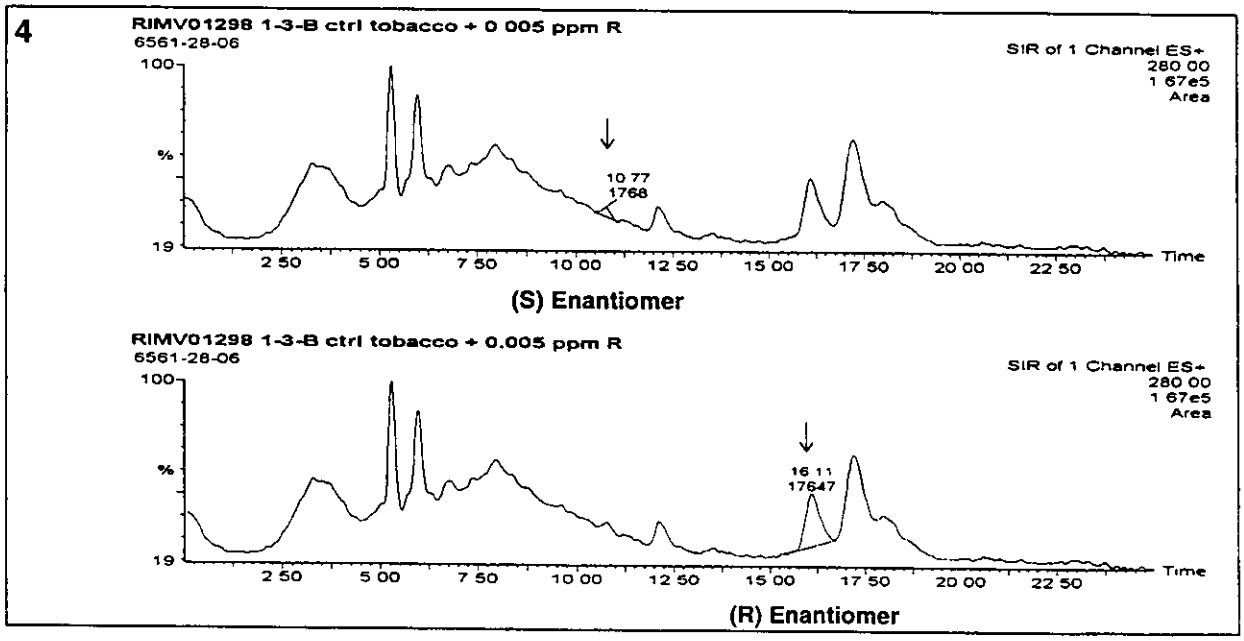


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-3-B	control	43 162	0.020	0 000	<0 005	0 020	0.000	<0 005

FIGURE 4: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN MATURE TOBACCO LEAF (Continued)

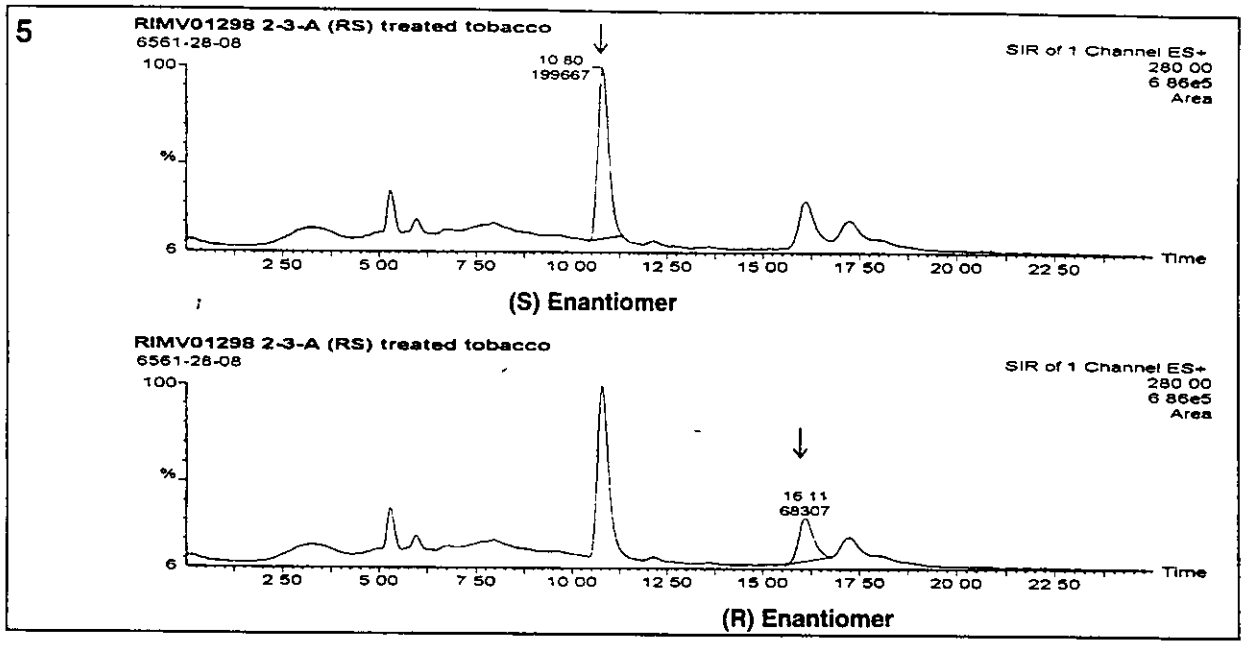


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr.	% Rec
1-3-B	recovery	0.005 ppm	43.123	0.210	0 005	88%	0 230	0 005	97%

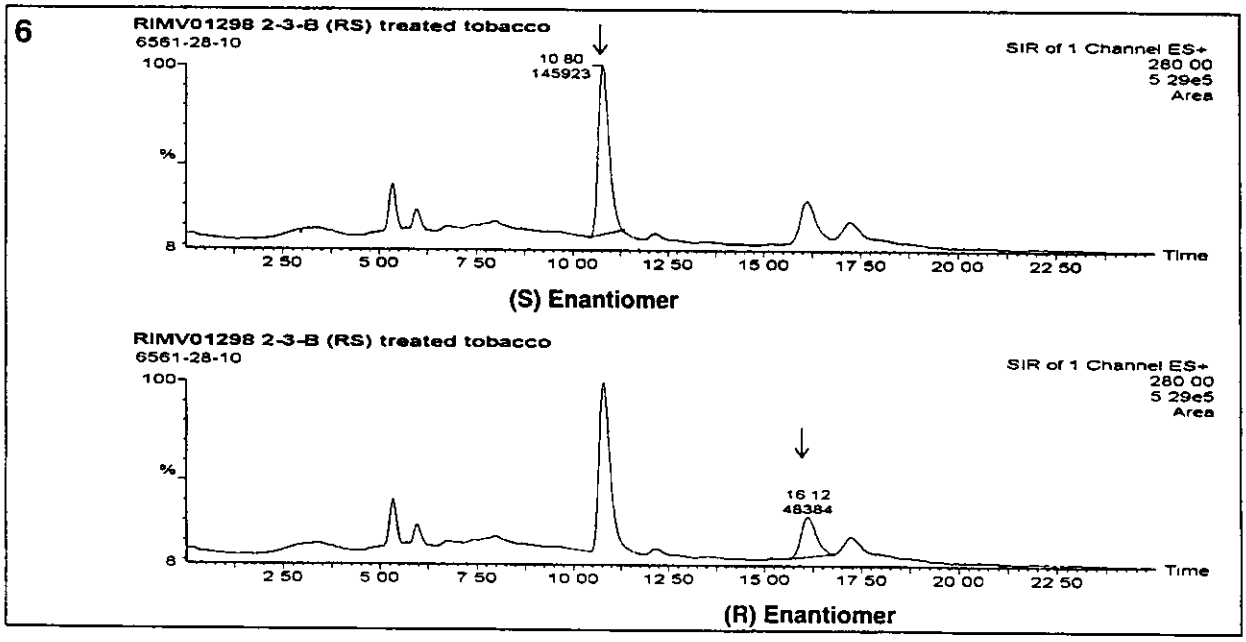


Sample Code	Desc	Amt (S), (R)- Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec
1-3-B	recovery	0 005 ppm	43 123	0 020	0 000	-	0 220	0 005	93%

FIGURE 4: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN MATURE TOBACCO LEAF (Continued)

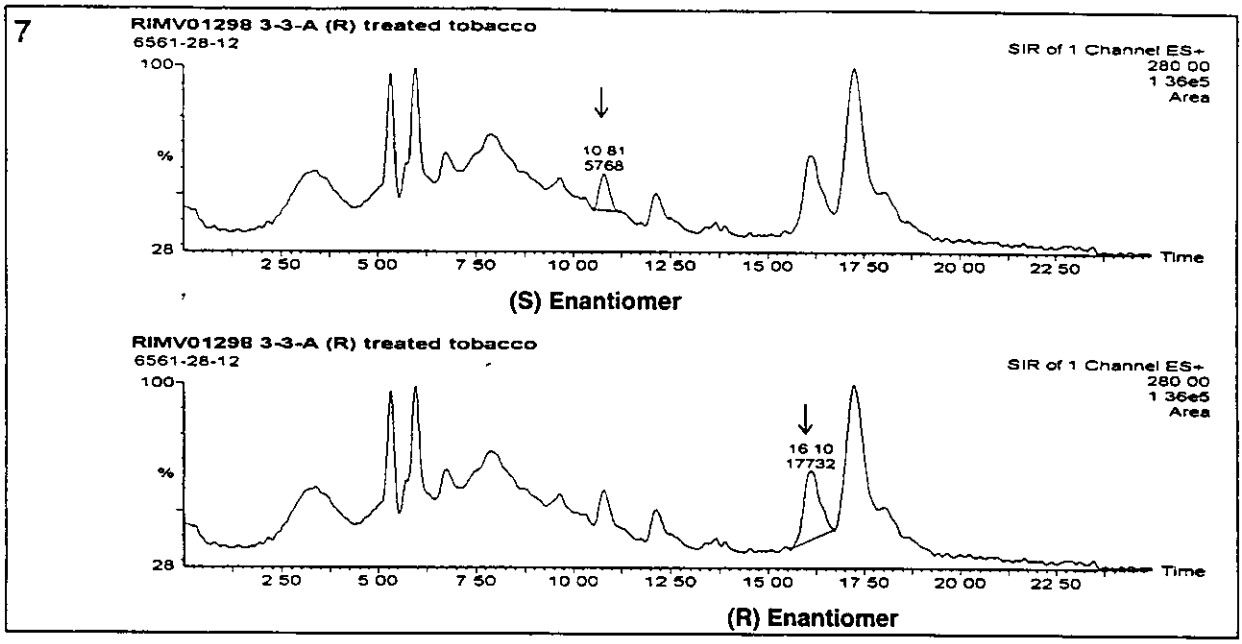


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-3-A	(RS)-treated	61	43.005	2 570	0.068	0 840	0.021	0.089

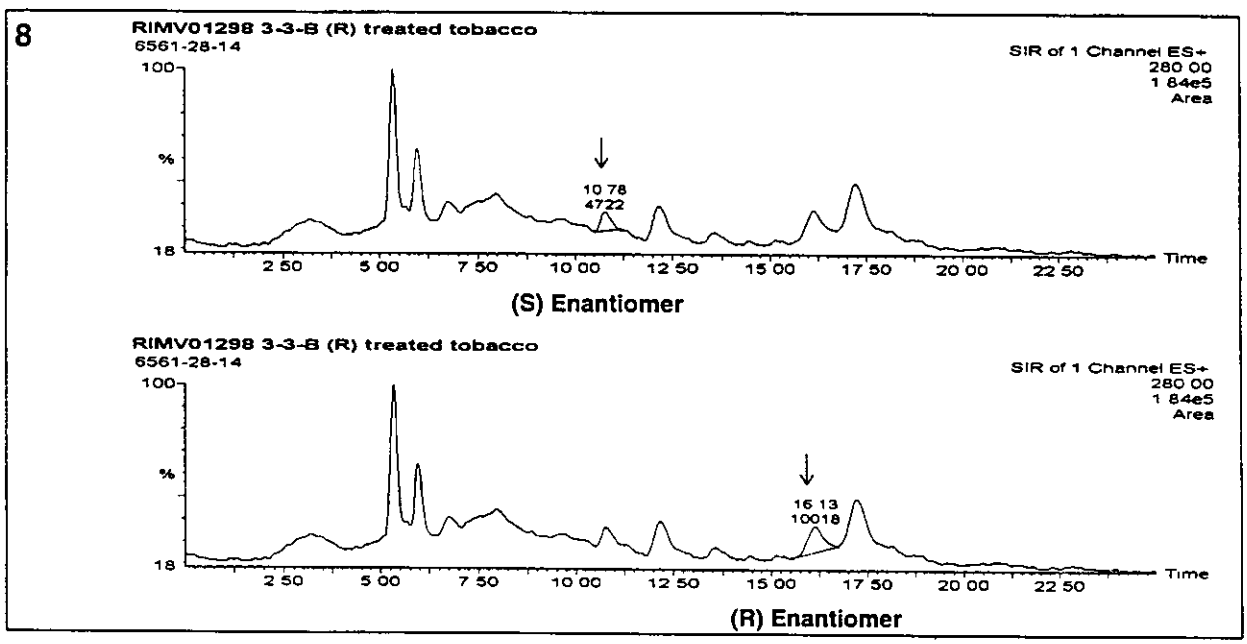


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-3-B	(RS)-treated	61	43 162	1.880	0 049	0 590	0 014	0.063

FIGURE 4. CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN MATURE TOBACCO LEAF (Continued)

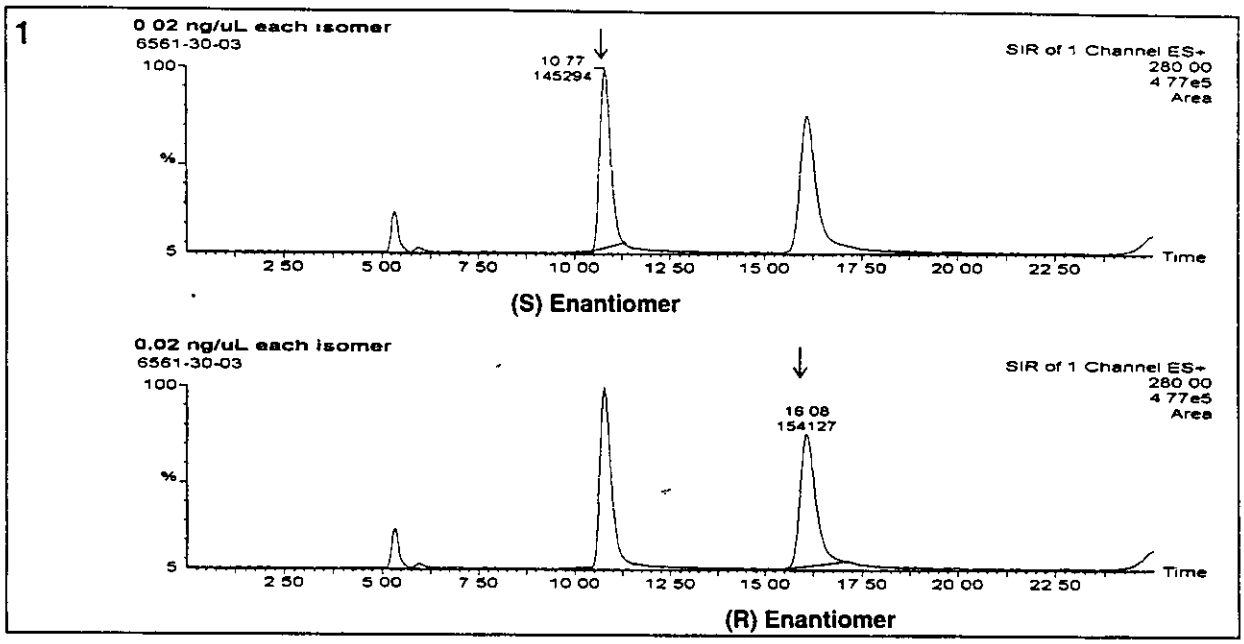


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-3-A	(R)-treated	61	43 083	0.070	0.002	0.220	0.005	0.007

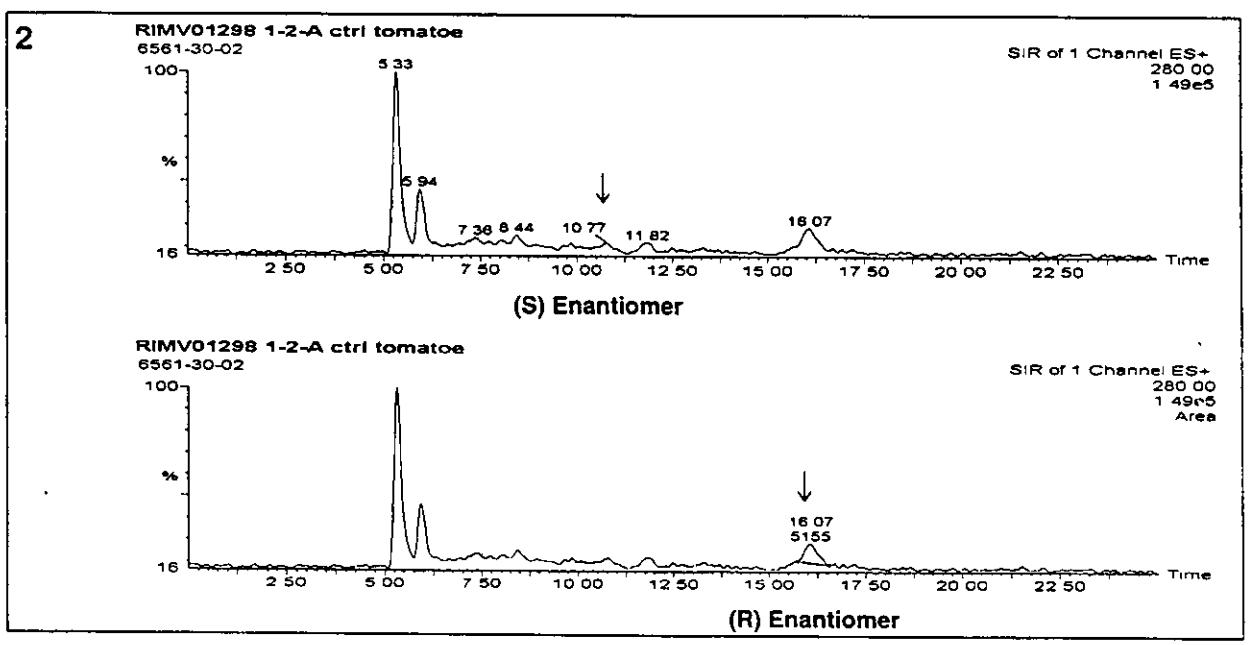


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-3-B	(R)-treated	61	43.123	0.060	0 001	0 120	0.003	0 004

FIGURE 5: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TOMATO FRUIT

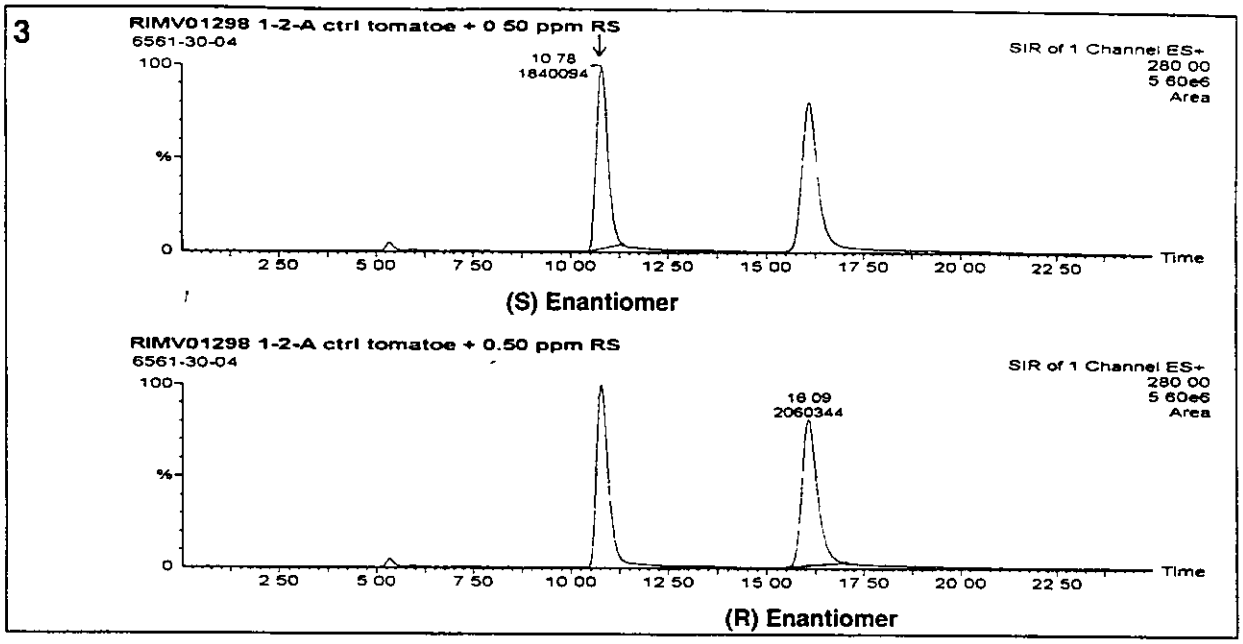


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0.0200	1.500	95073 2	0	0 9998	100417	0	0 9998

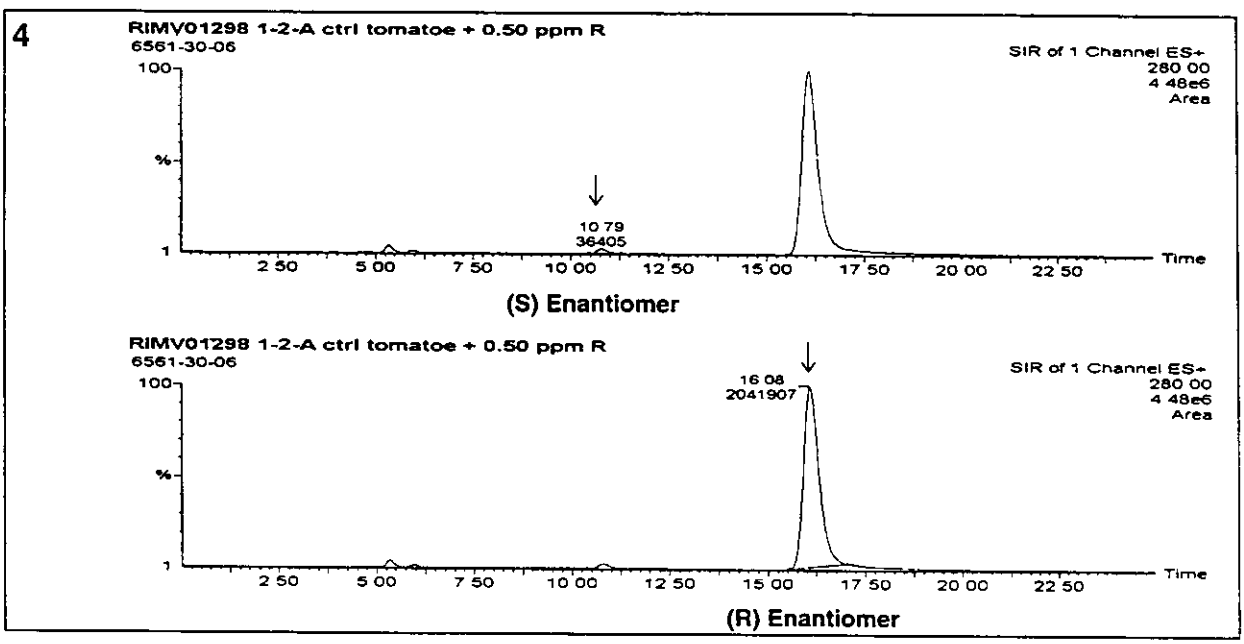


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-2-A	control	43 043	0.000	0 000	<0 005	0 050	0.001	<0 005

FIGURE 5: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TOMATO FRUIT (Continued)

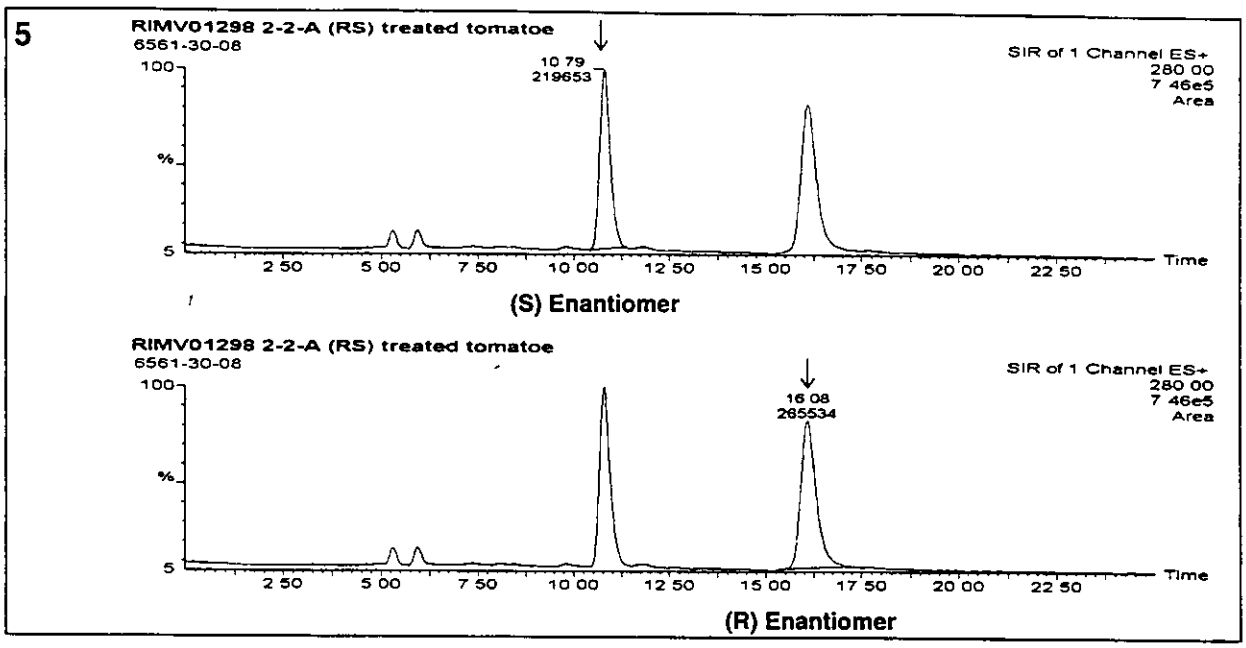


Sample Code	Desc.	Amt. (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec.	(R) Enantiomer ng	ppm uncorr	% Rec.
1-2-A	recovery	0 50 ppm	42.965	19.350	0 450	90%	20 520	0 478	95%

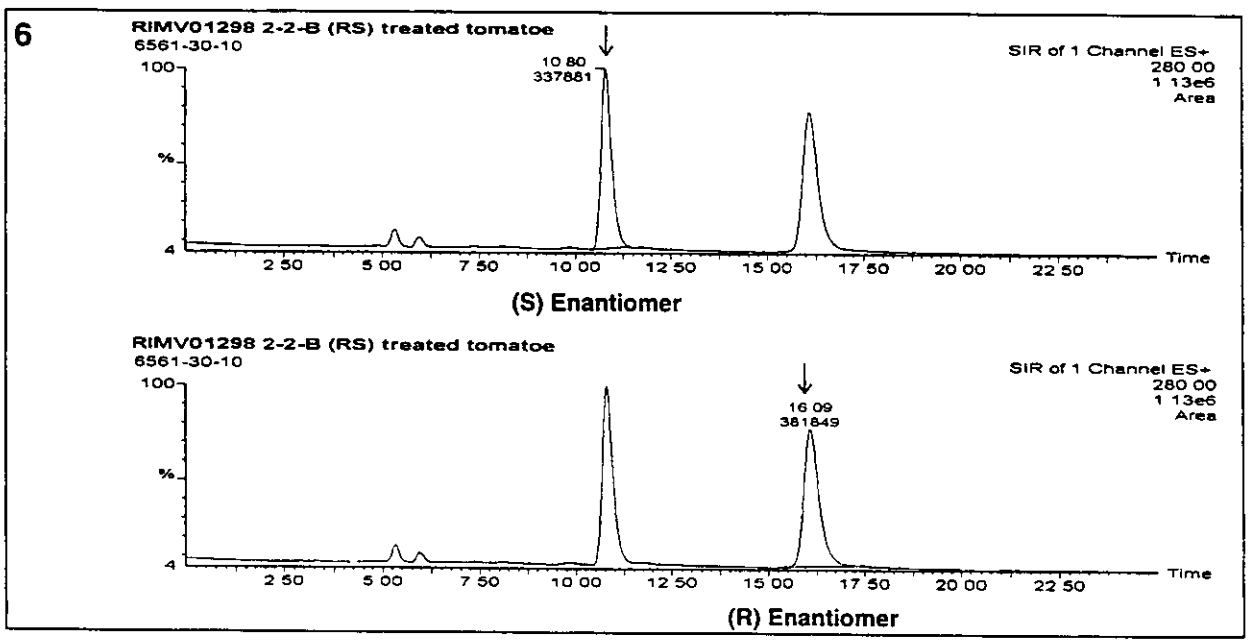


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec
1-2-A	recovery	0 50 ppm	42 965	0.380	0 009	-	20 330	0.473	94%

FIGURE 5: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TOMATO FRUIT (Continued)

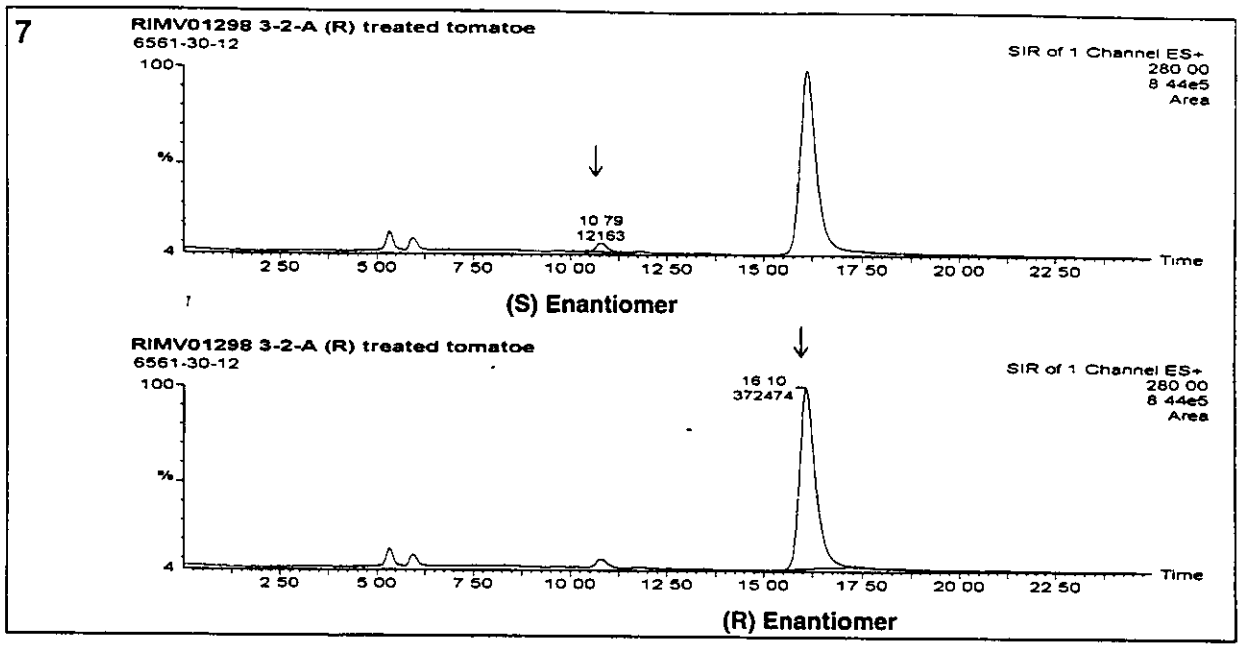


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-2-A	(RS)-treated	0	42.965	2 310	0.060	2 640	0 065	0 125

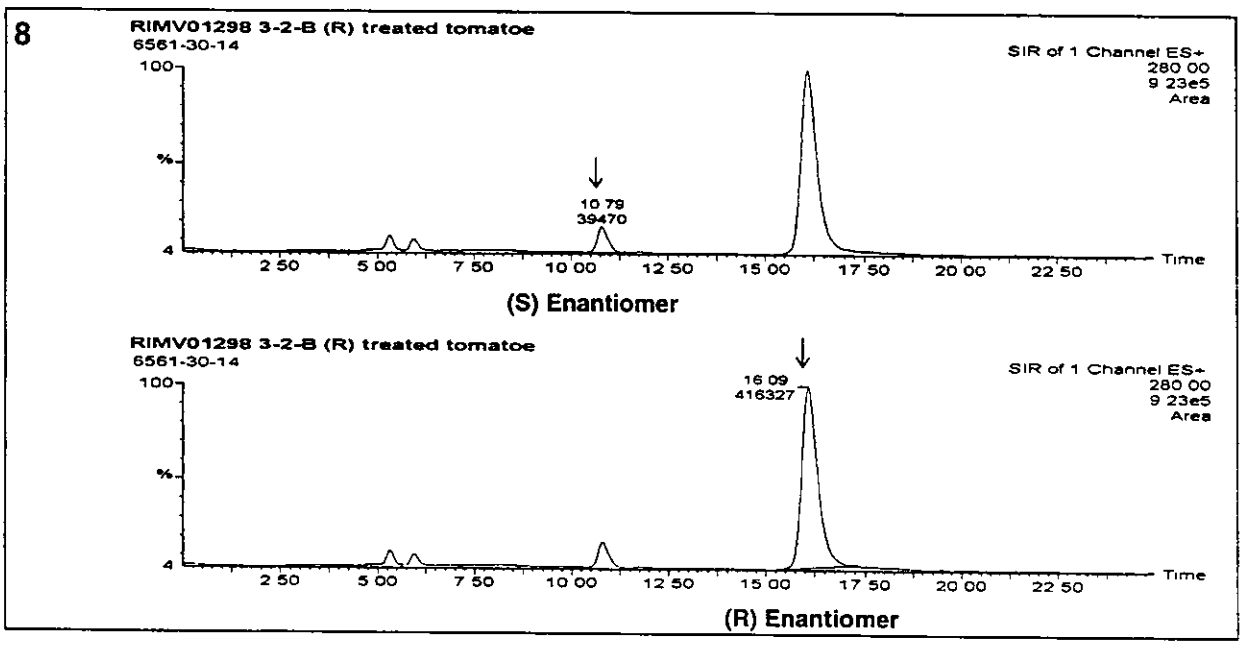


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr.	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-2-B	(RS)-treated	0	43.004	3 550	0 092	3 800	0 093	0 185

FIGURE 5: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TOMATO FRUIT (Continued)

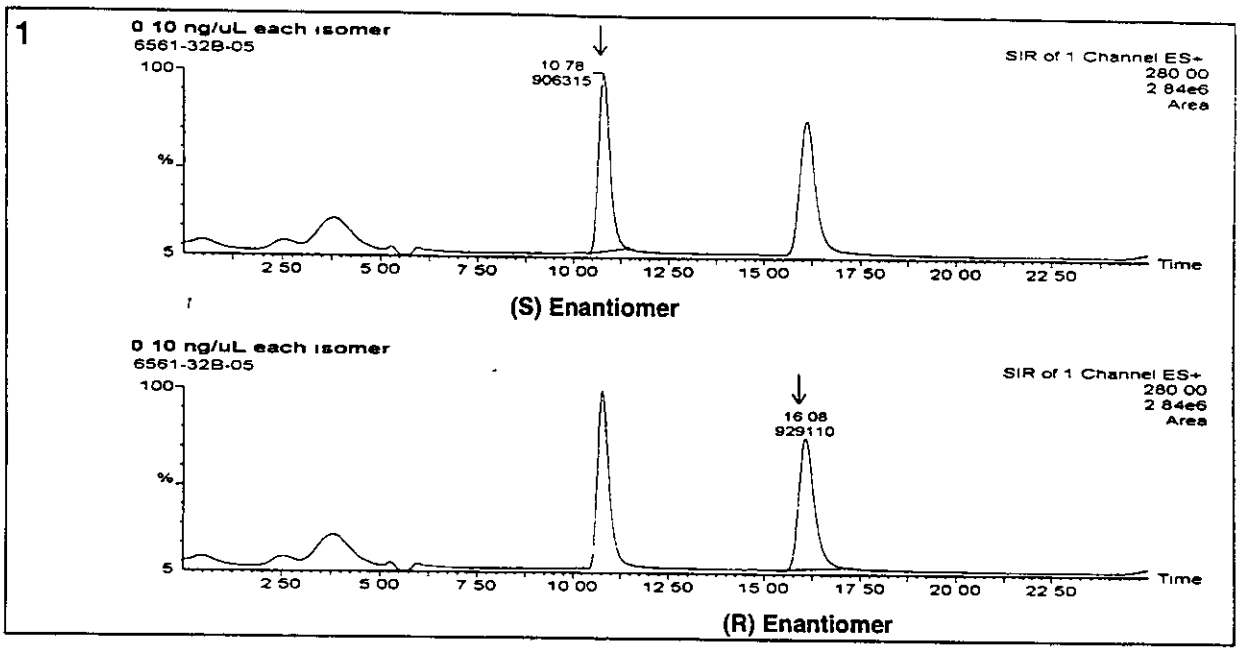


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-2-A	(R)-treated	0	42.965	0 130	<0.005	3 710	0.091	0.094

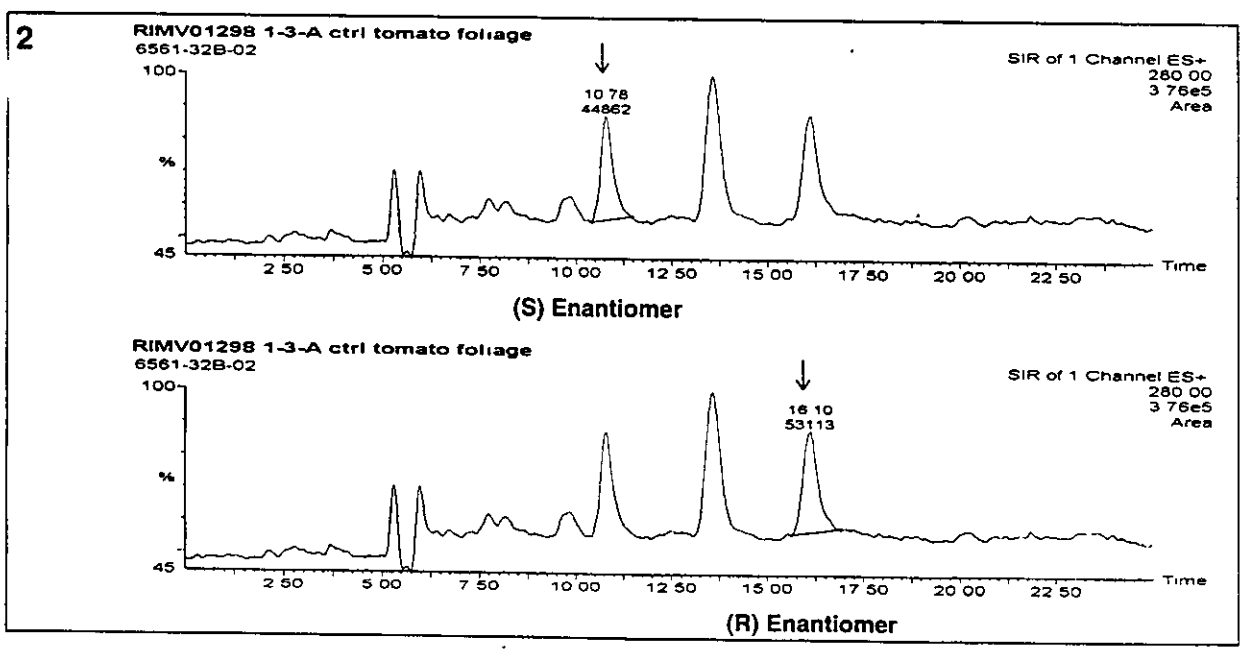


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-2-B	(R)-treated	0	42.887	0 420	0 011	4.150	0.102	0 113

FIGURE 6: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TOMATO FOLIAGE

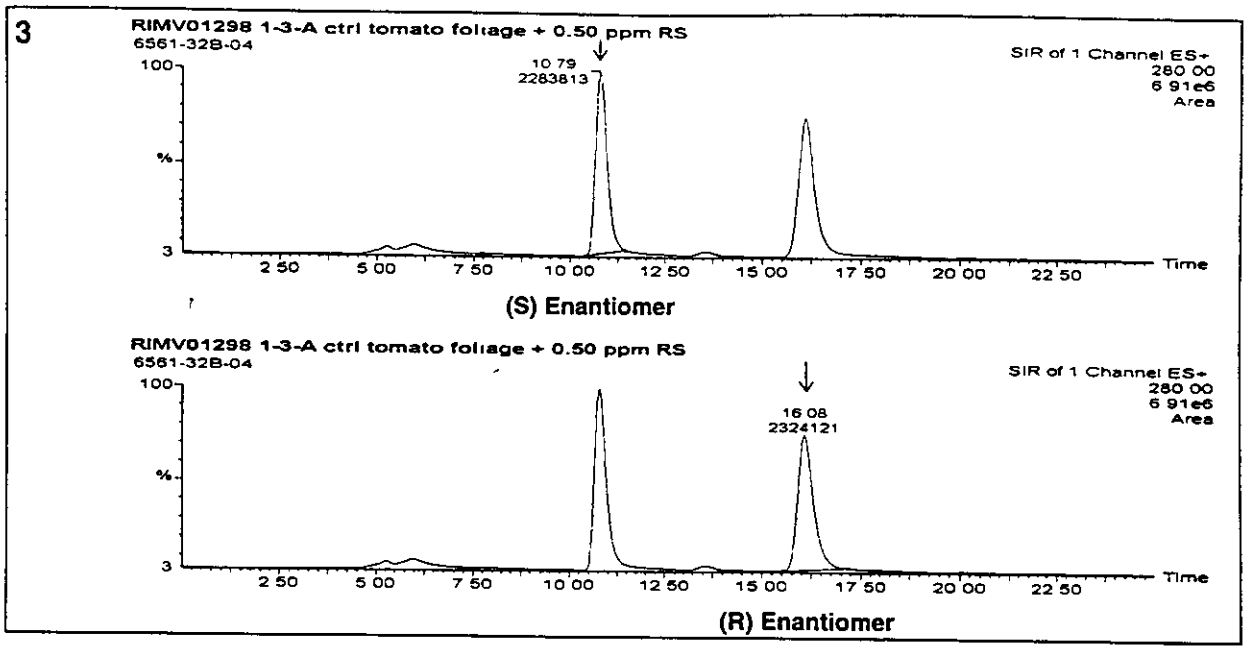


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 1000	7 500	111986	0	0 9973	114647	0	0.9967

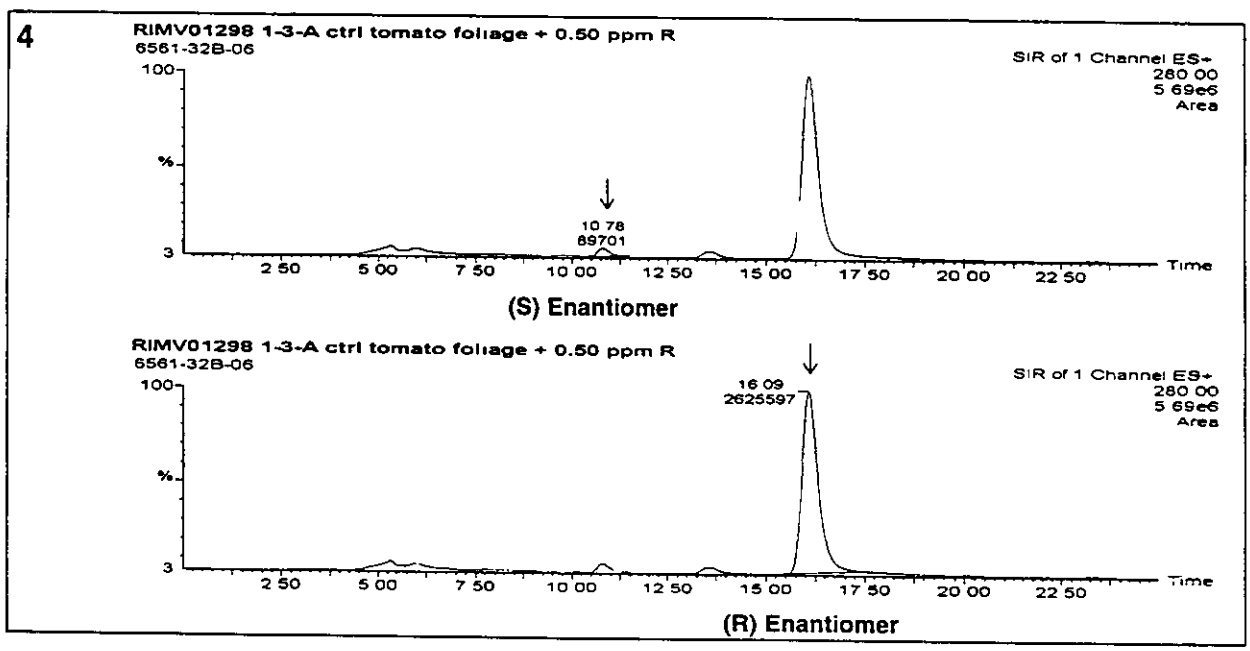


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-3-A	control	43 005	0.40	0 009	0 010	0 46	0 011	0 011

FIGURE 6: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TOMATO FOLIAGE (Continued)

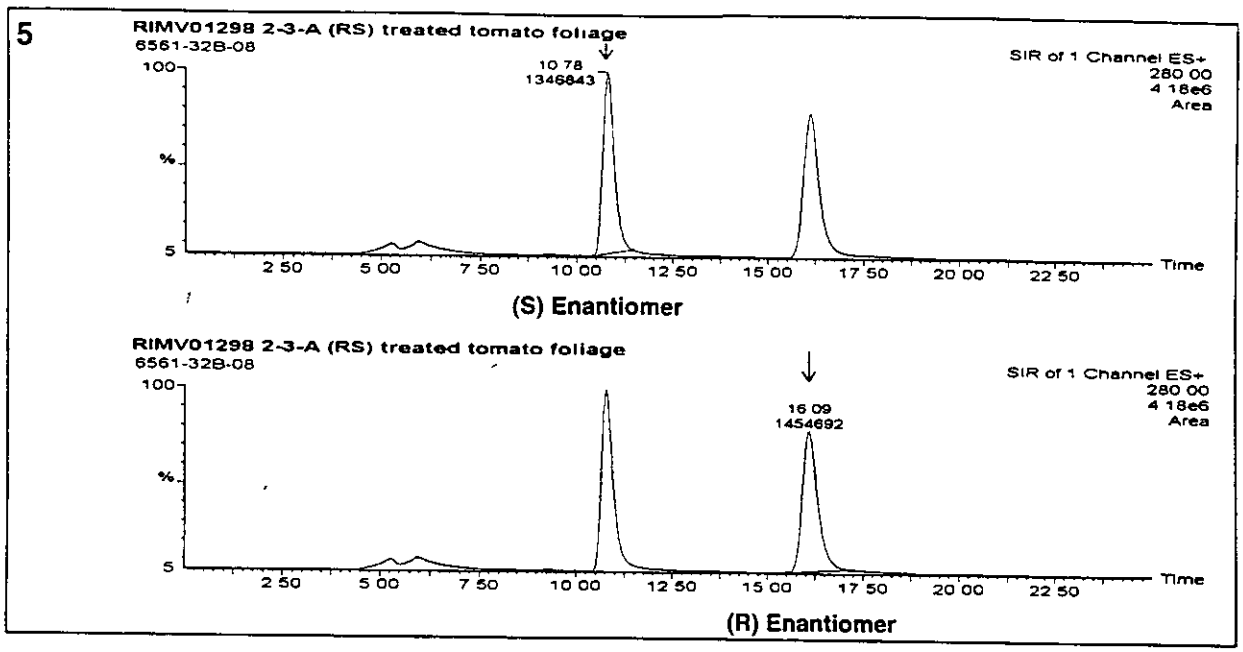


Sample Code	Desc.	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec.
1-3-A	recovery	0 50 ppm	43.044	20.39	0 474	93%	20.27	0.471	92%

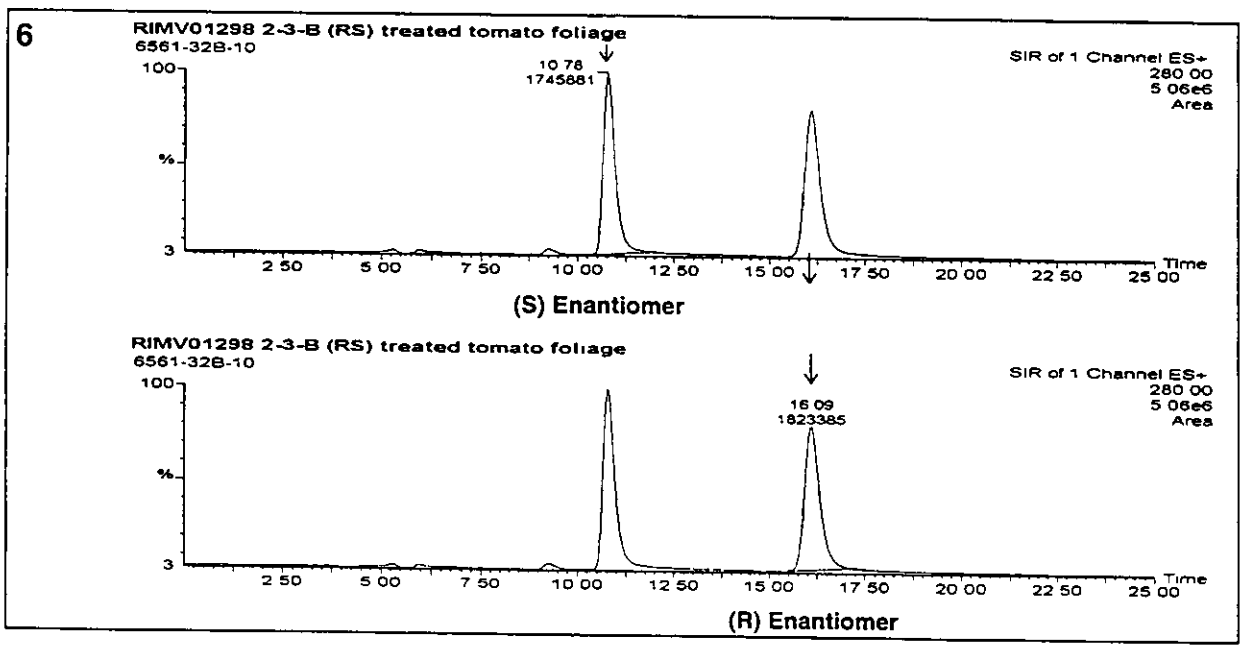


Sample Code	Desc	Amt. (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec.	(R) Enantiomer ng	ppm uncorr	% Rec
1-3-A	recovery	0 50 ppm	43 044	0 80	0 018	-	22 90	0 529	104%

FIGURE 6: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TOMATO FOLIAGE (Continued)

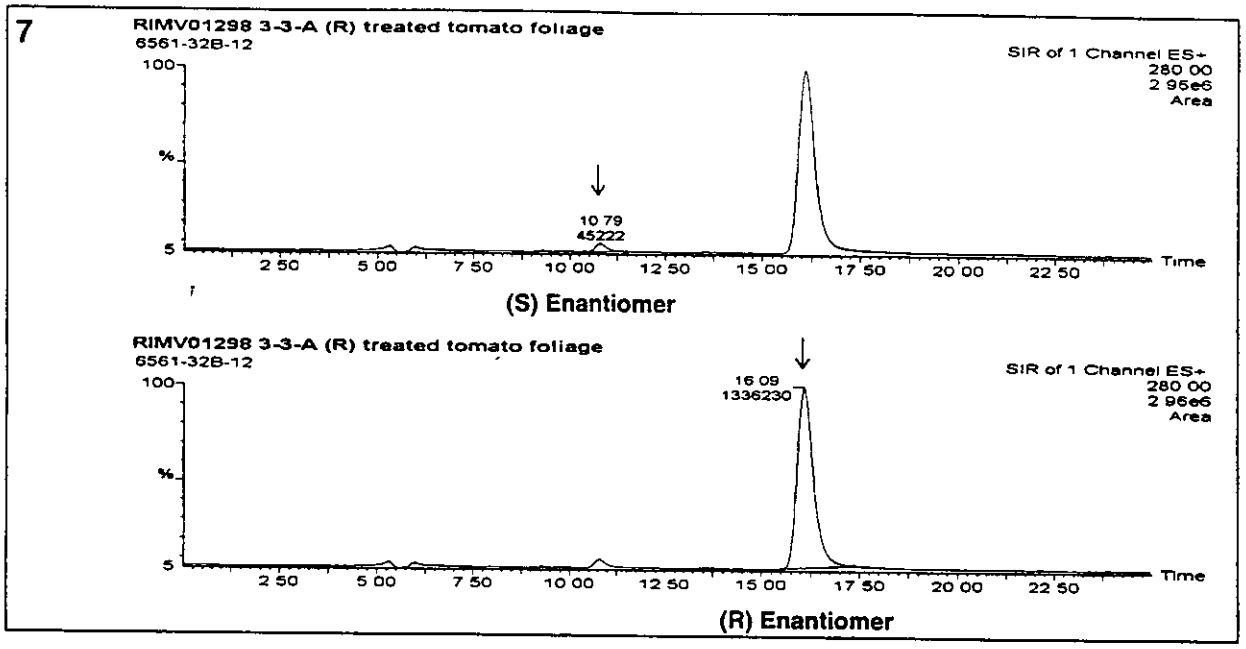


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-3-A	(RS)-treated	1	4 304	12.03	3 009	12 69	3 013	6.022

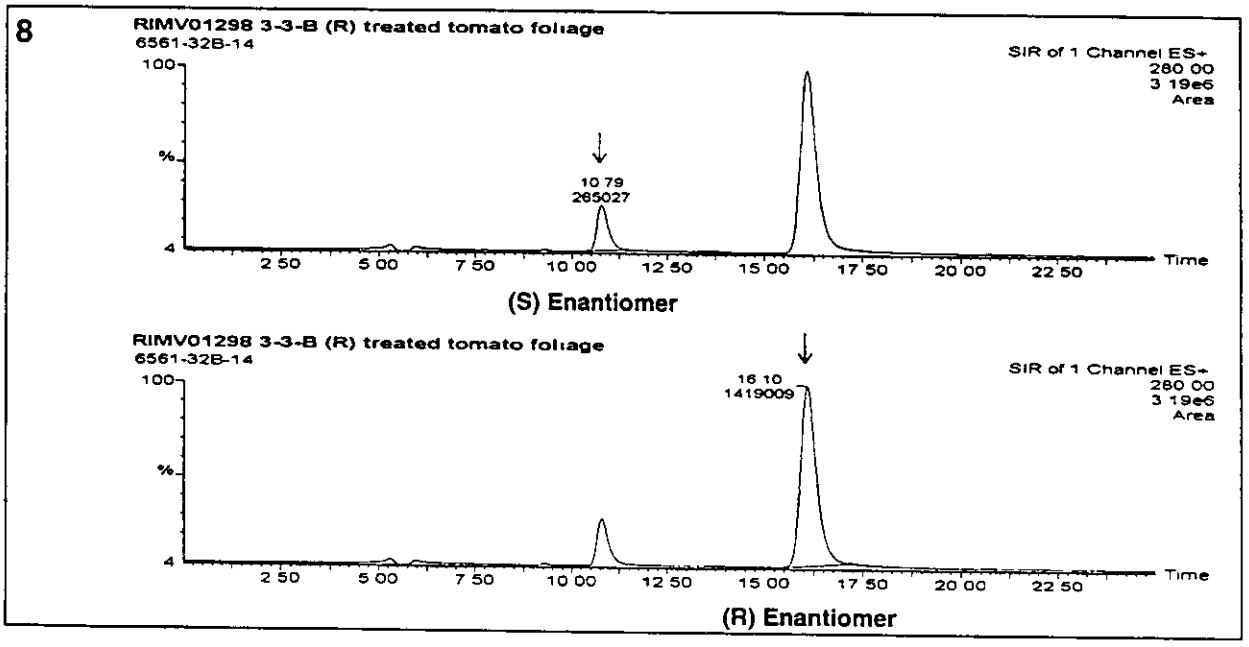


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-3-B	(RS)-treated	1	4 300	15 59	3 903	15 90	3 778	7 681

FIGURE 6: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TOMATO FOLIAGE (Continued)

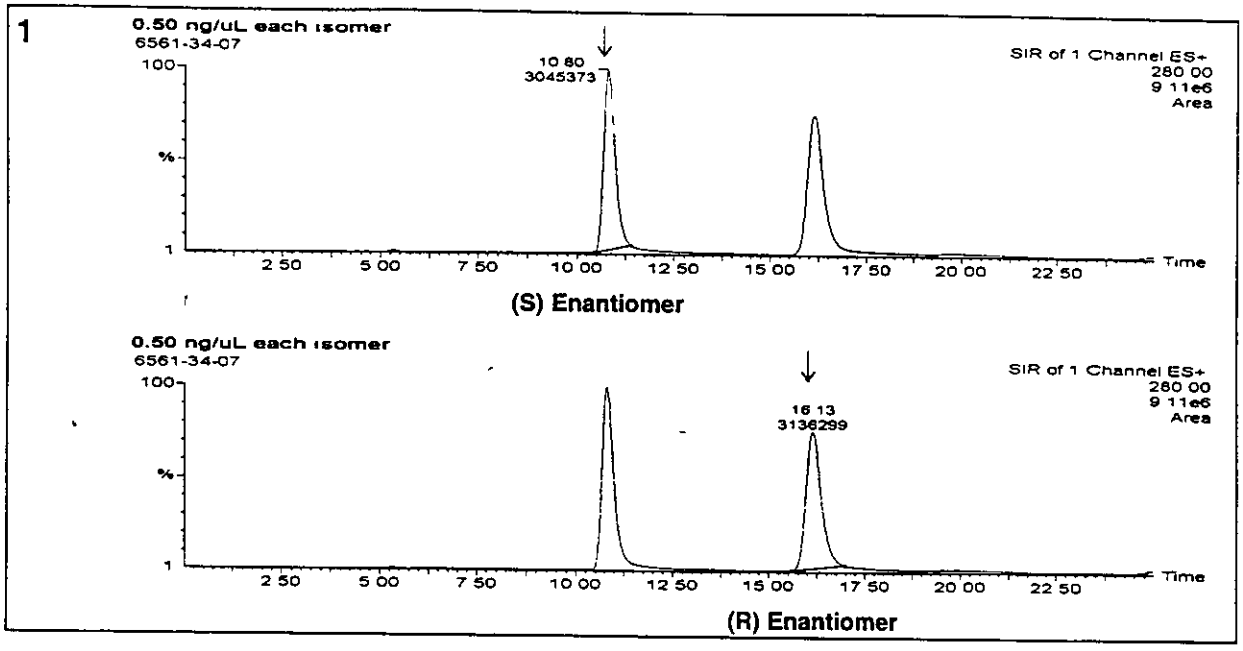


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-3-A	(R)-treated	1	4 300	0.400	0 100	11 66	2.771	2.871

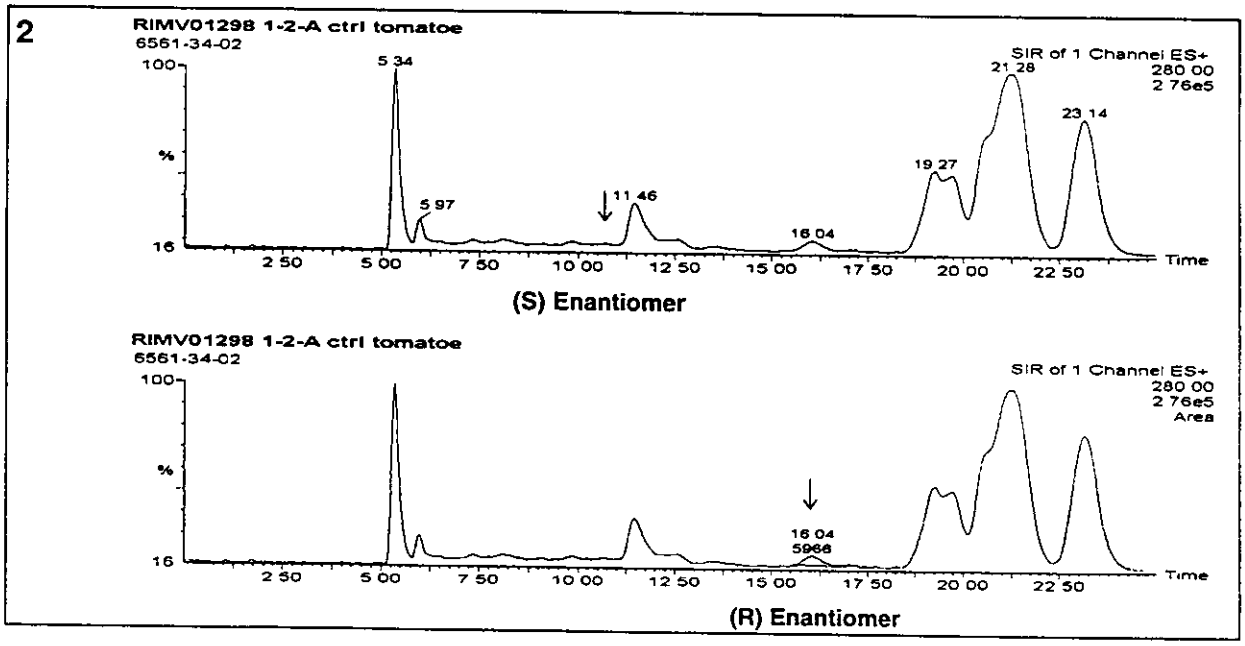


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-3-B	(R)-treated	1	4 293	2 37	0 594	12 38	2 947	3 541

FIGURE 7: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN MATURE TOMATO FRUIT

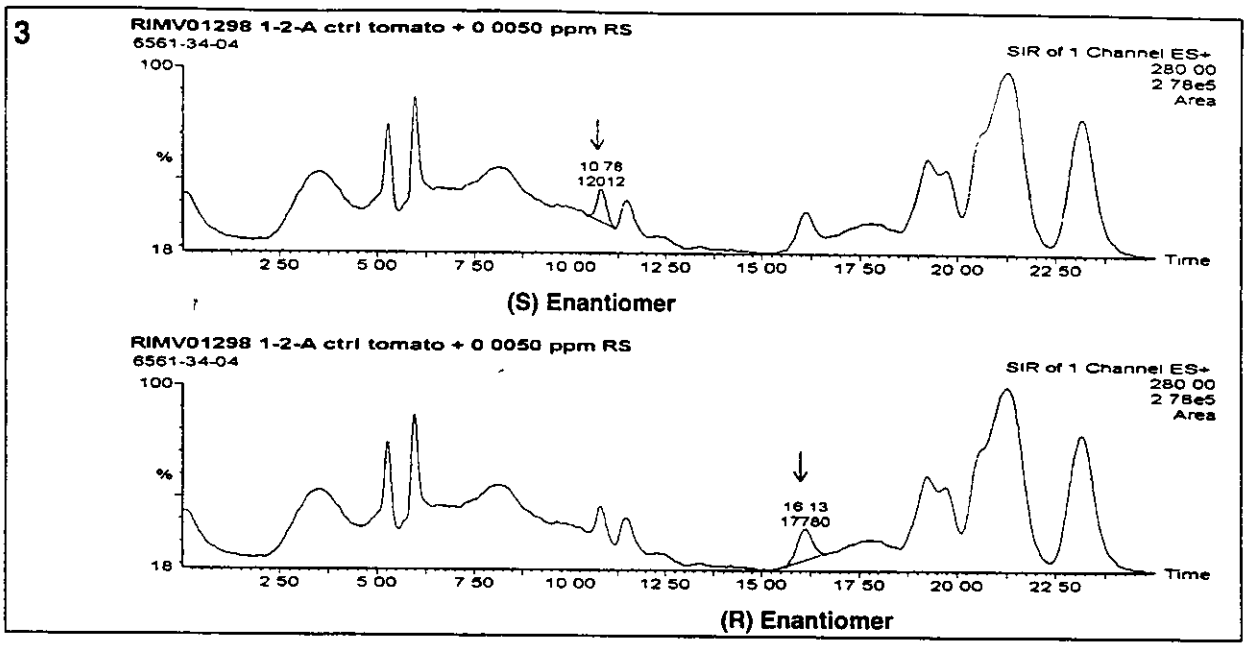


Standard	Concentration	Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0.5000	37 50	77375.1	0	0 9989	79250 7	0	0 9989

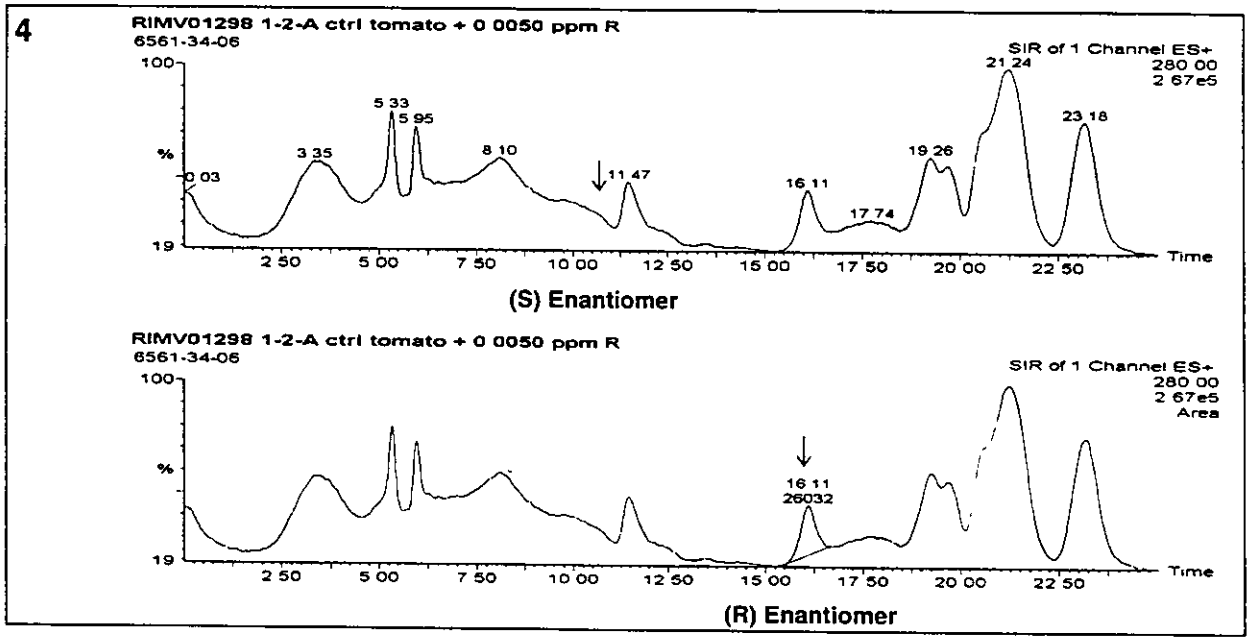


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-2-A	control	43 122	0 000	0 000	<0 005	0 080	0 002	<0 005

FIGURE 7: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN MATURE TOMATO FRUIT (Continued)

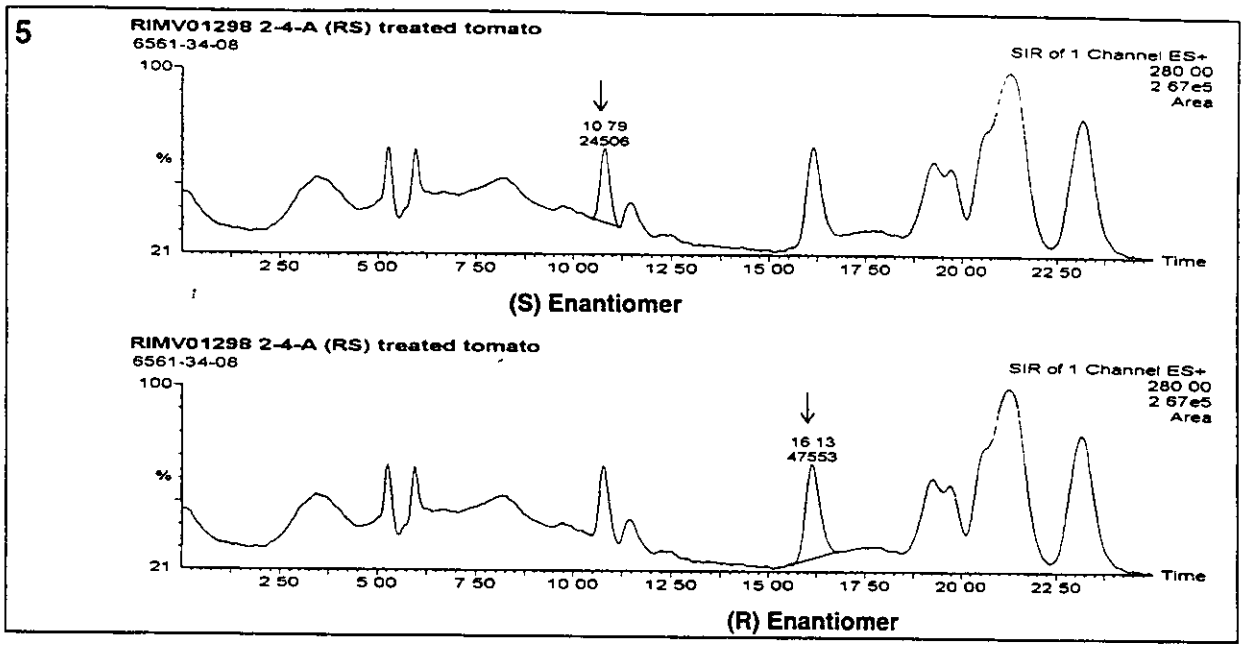


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec.	(R) Enantiomer ng	ppm uncorr	% Rec
1-2-A	recovery	0 005 ppm	43.083	0 160	0.004	74%	0.220	0.005	65%

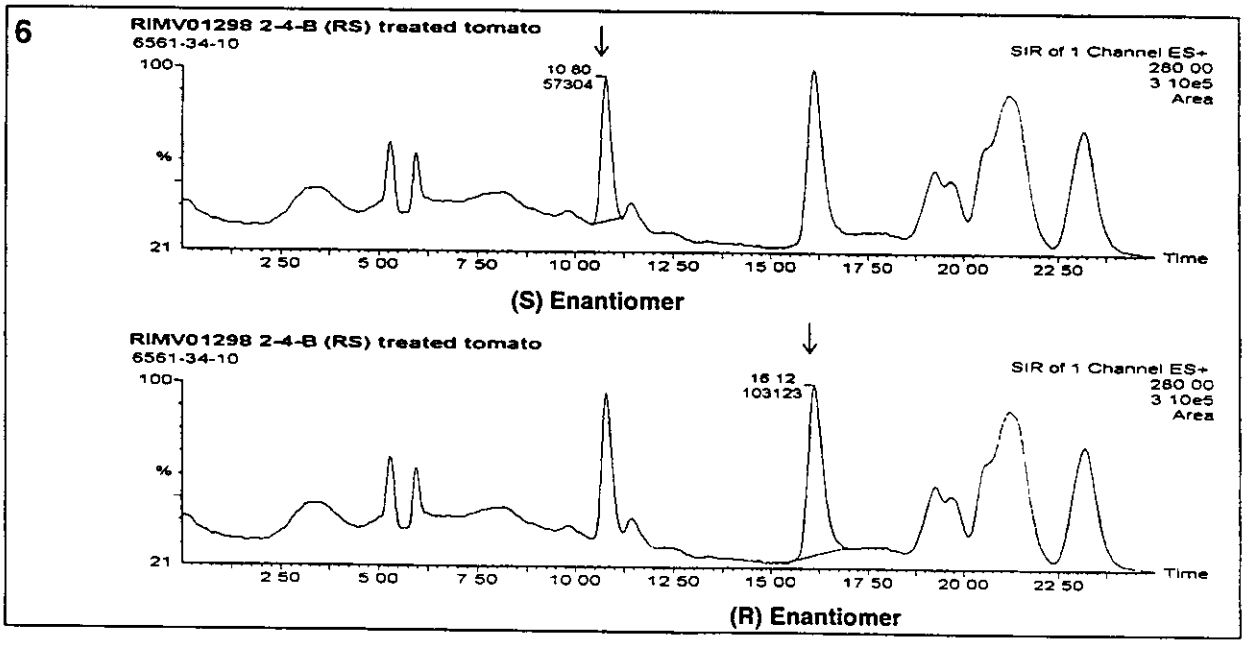


Sample Code	Desc.	Amt (S), (R) - Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec
1-2-A	recovery	0 005 ppm	42.887	0 000	0 000	-	0 330	0.008	117%

FIGURE 7: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN MATURE TOMATO FRUIT (Continued)

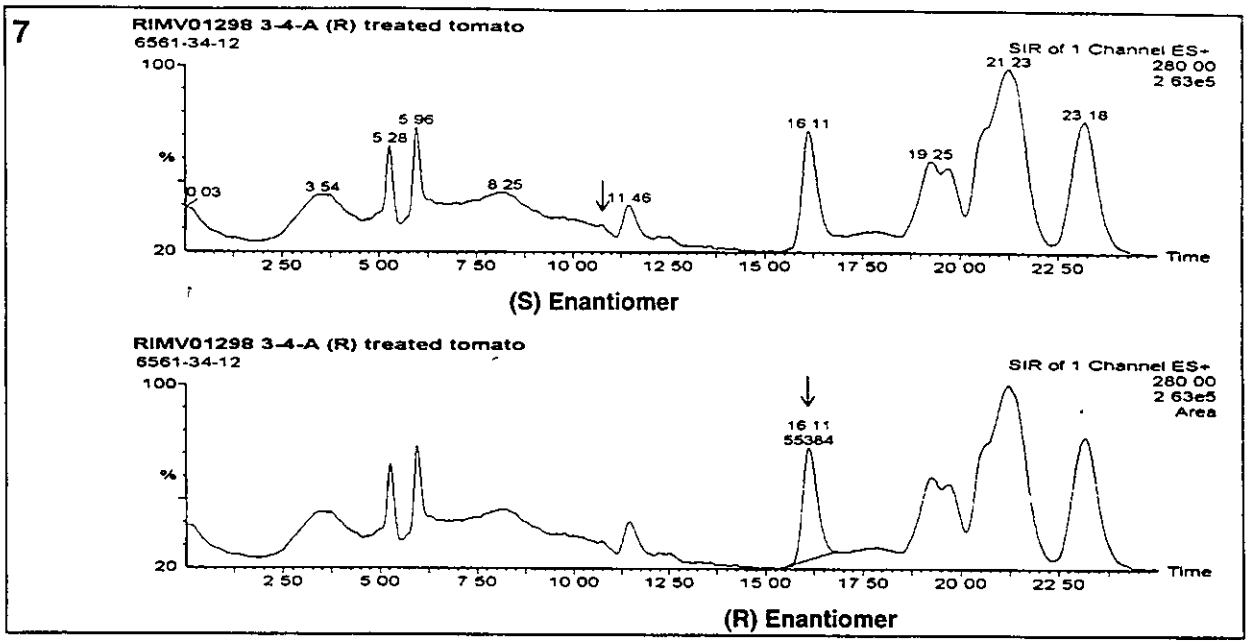


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-4-A	(RS)-treated	5	42.887	0.320	0.010	0 600	0 015	0 025

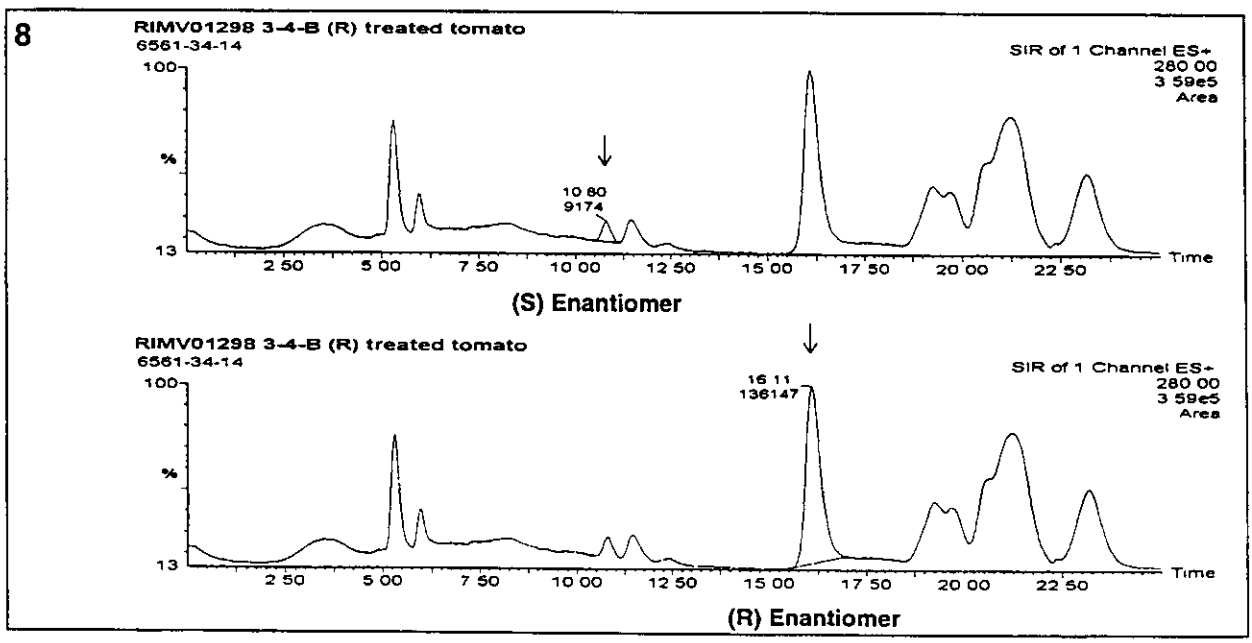


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-4-B	(RS)-treated	5	43 474	0.740	0 023	1 300	0 033	0 056

FIGURE 7: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN MATURE TOMATO FRUIT (Continued)

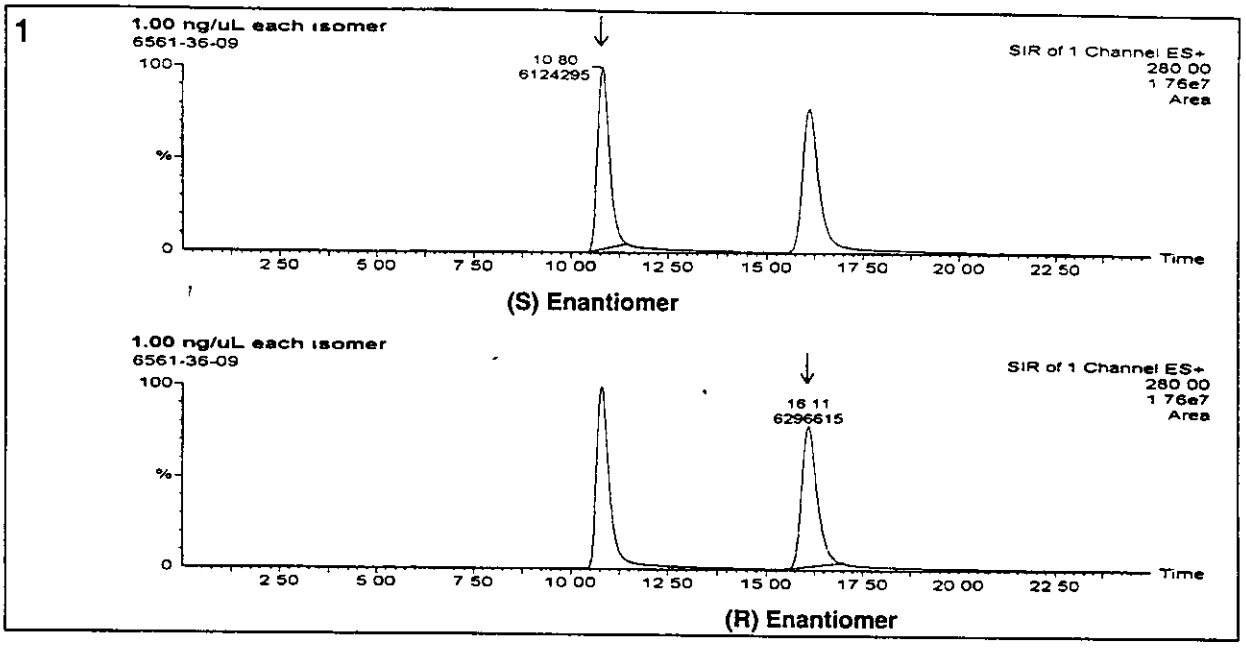


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-4-A	(R)-treated	5	42.965	0 000	<0.005	0.700	0 018	0 018

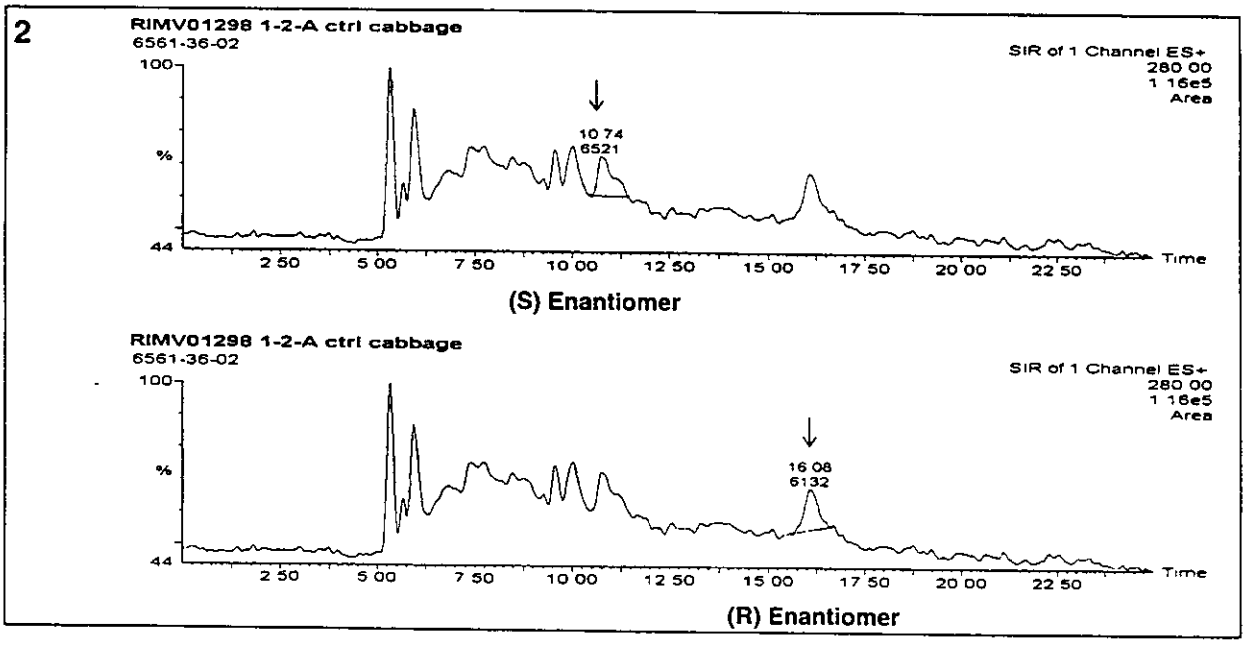


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-4-B	(R)-treated	5	43 318	0 120	<0 005	1 720	0 044	0 047

FIGURE 8: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY CABBAGE HEADS

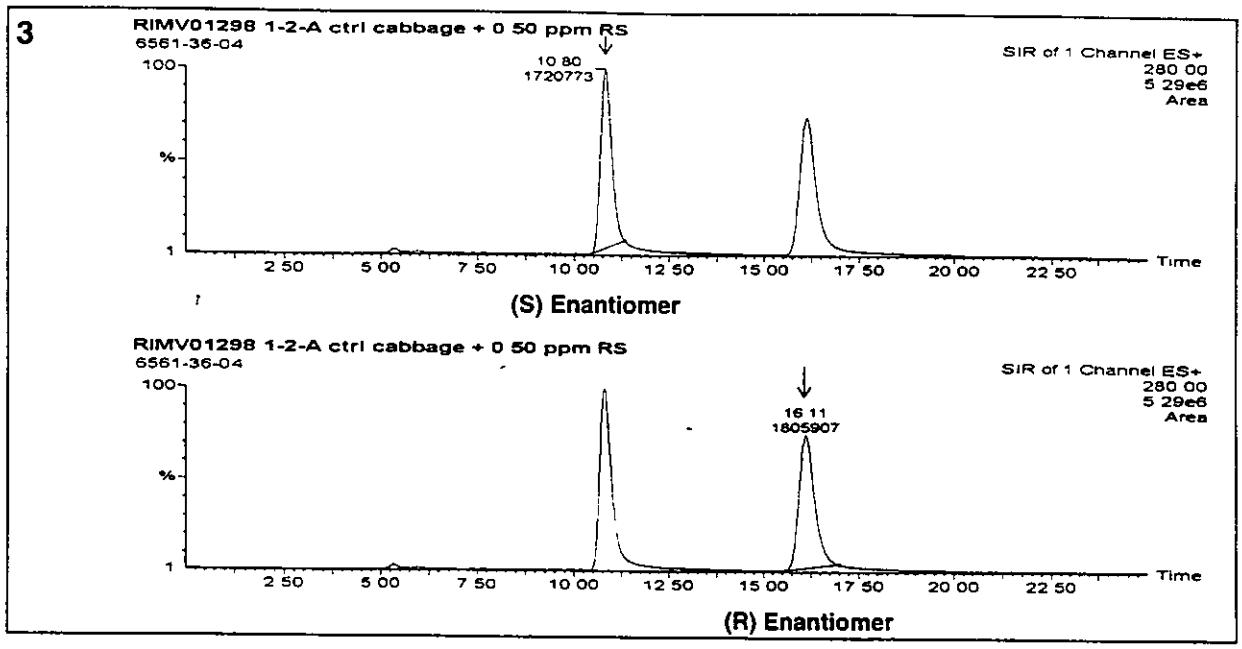


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
1 0000	75 00	81762 5	0	0 9999	84207 0	0	0 9999

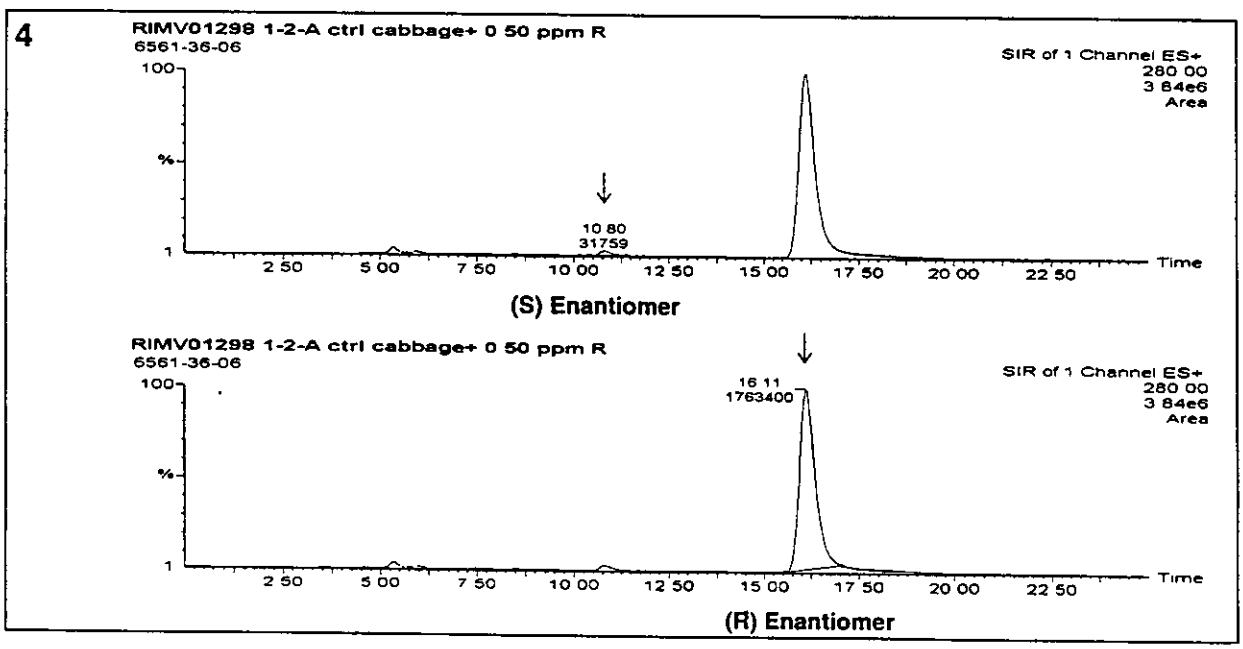


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-2-A	control	43 279	0.080	0 002	<0.005	0 070	0 002	<0 005

FIGURE 8. CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY CABBAGE HEADS (Continued)

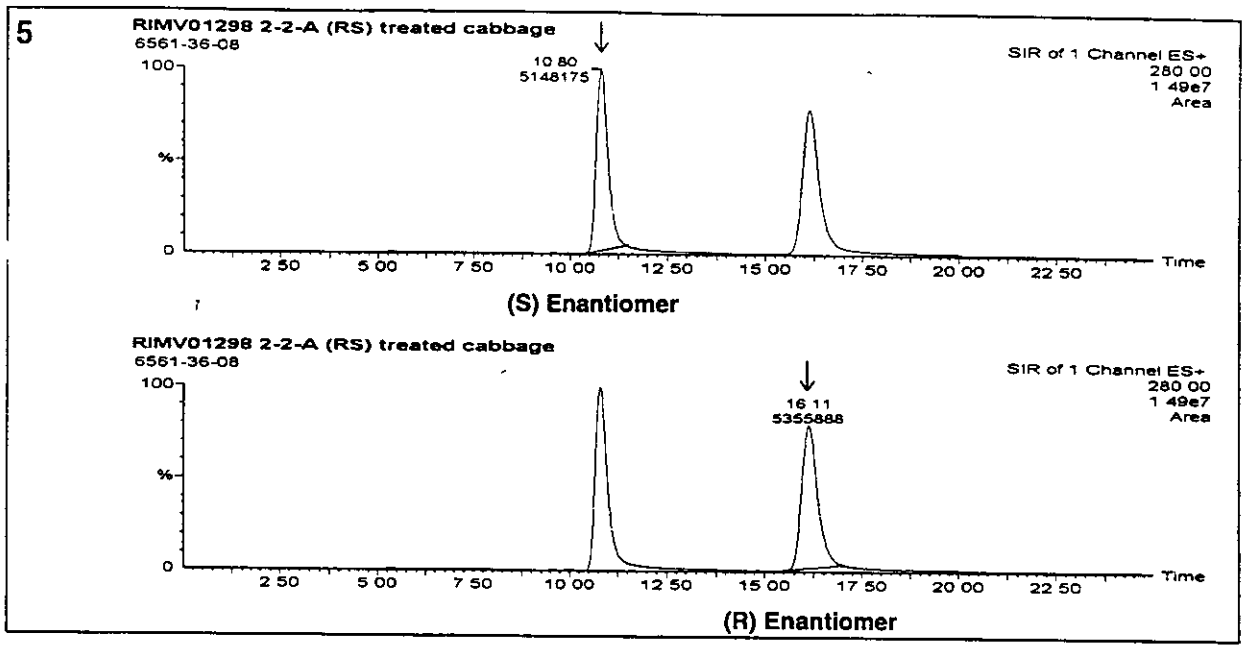


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec.	(R) Enantiomer ng	ppm uncorr	% Rec.
1-2-A	recovery	0 50 ppm	43 004	21.050	0.489	98%	21.450	0.499	99%

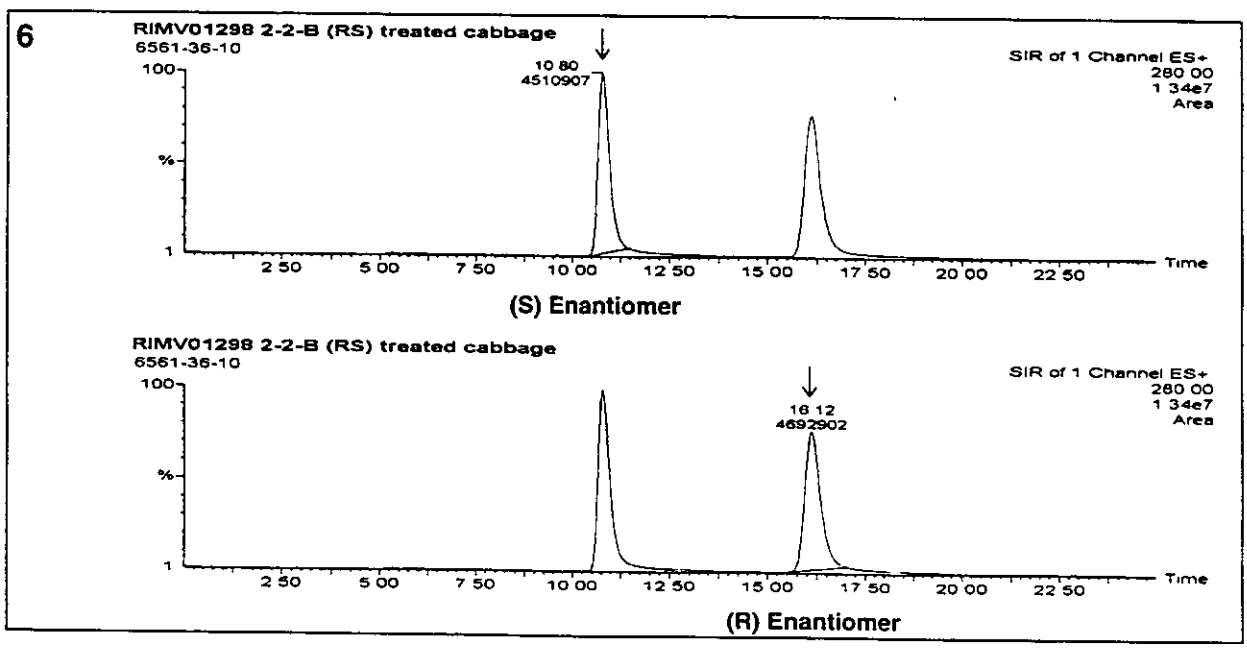


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec
1-2-A	recovery	0.50 ppm	43 279	0 390	0 009	-	20 940	0 484	96%

FIGURE 8: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY CABBAGE HEADS (Continued)

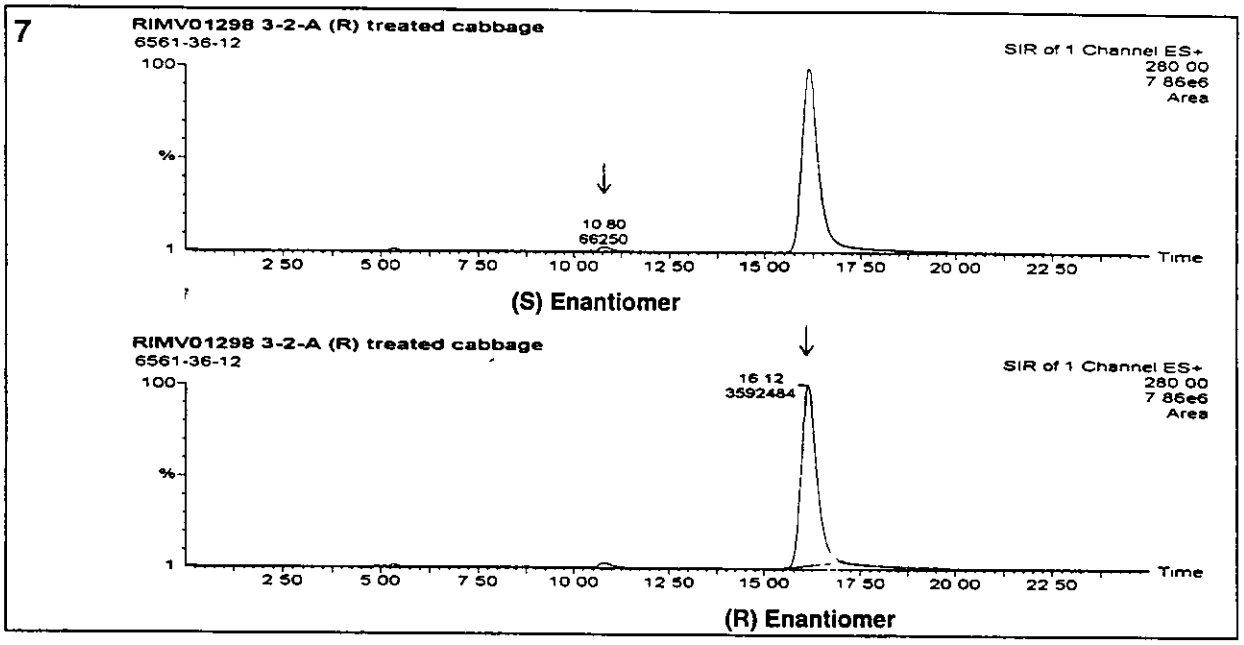


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-2-A	(RS)-treated	0	43.239	62 960	1.493	63 600	1.502	2 995

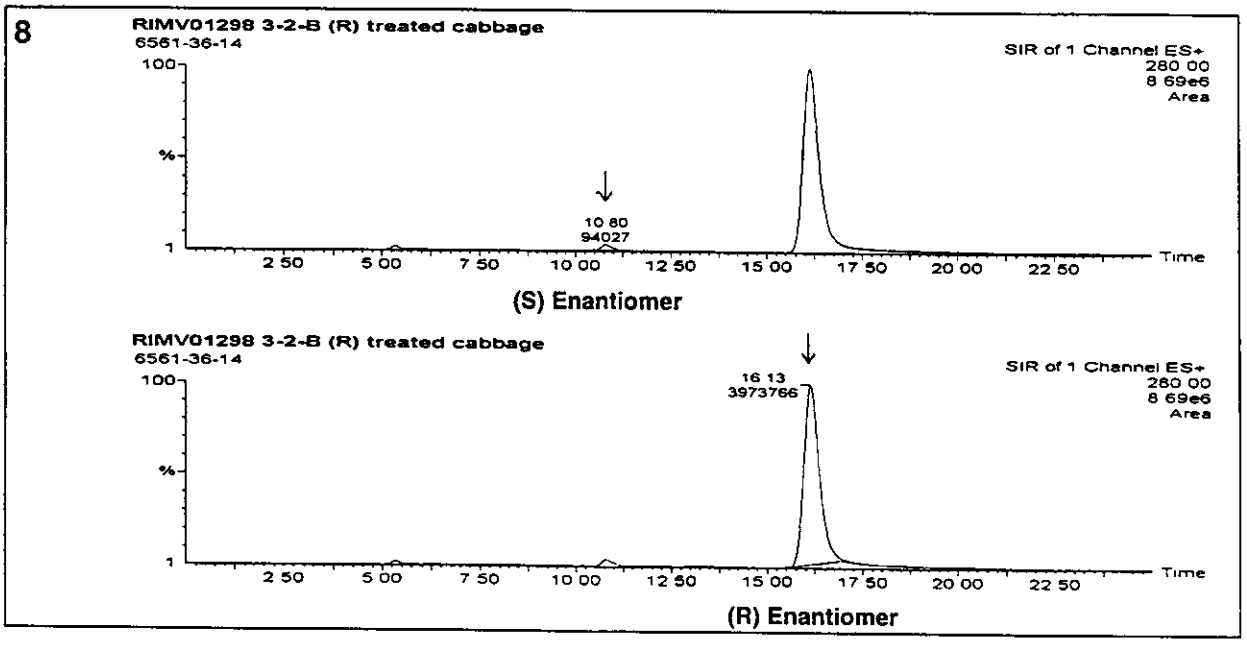


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-2-B	(RS)-treated	0	42.965	55.170	1 317	55 730	1 324	2 641

FIGURE 8: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY CABBAGE HEADS (Continued)

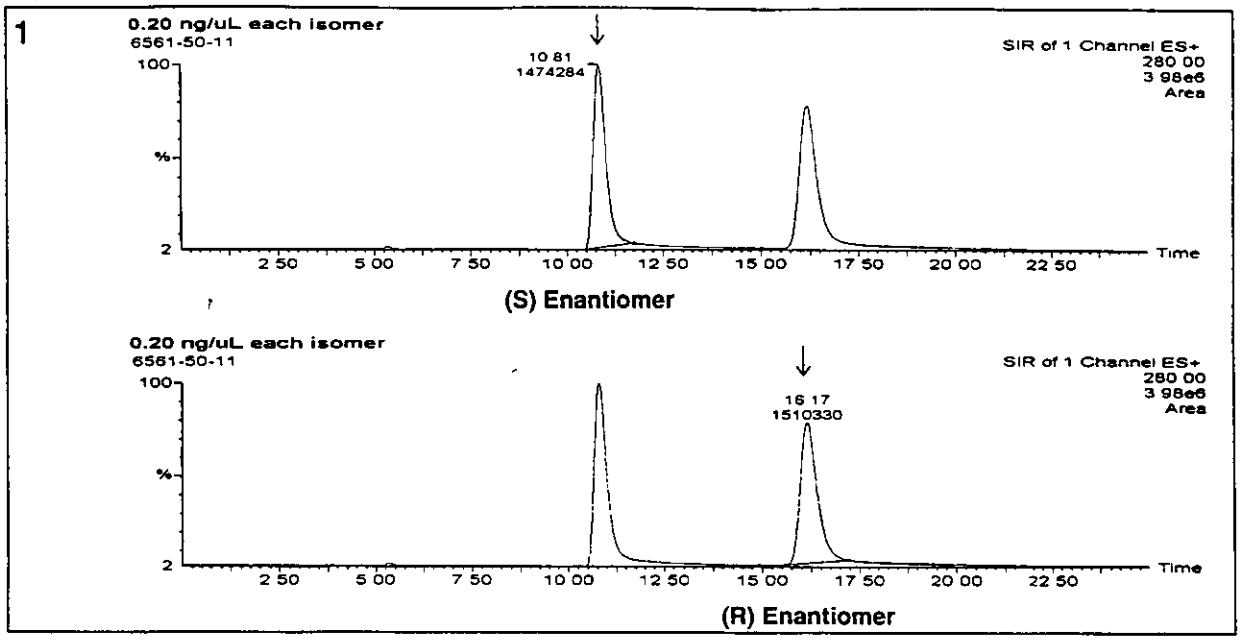


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-2-A	(R)-treated	0	42 239	0 810	0.019	42 660	1 007	1.026

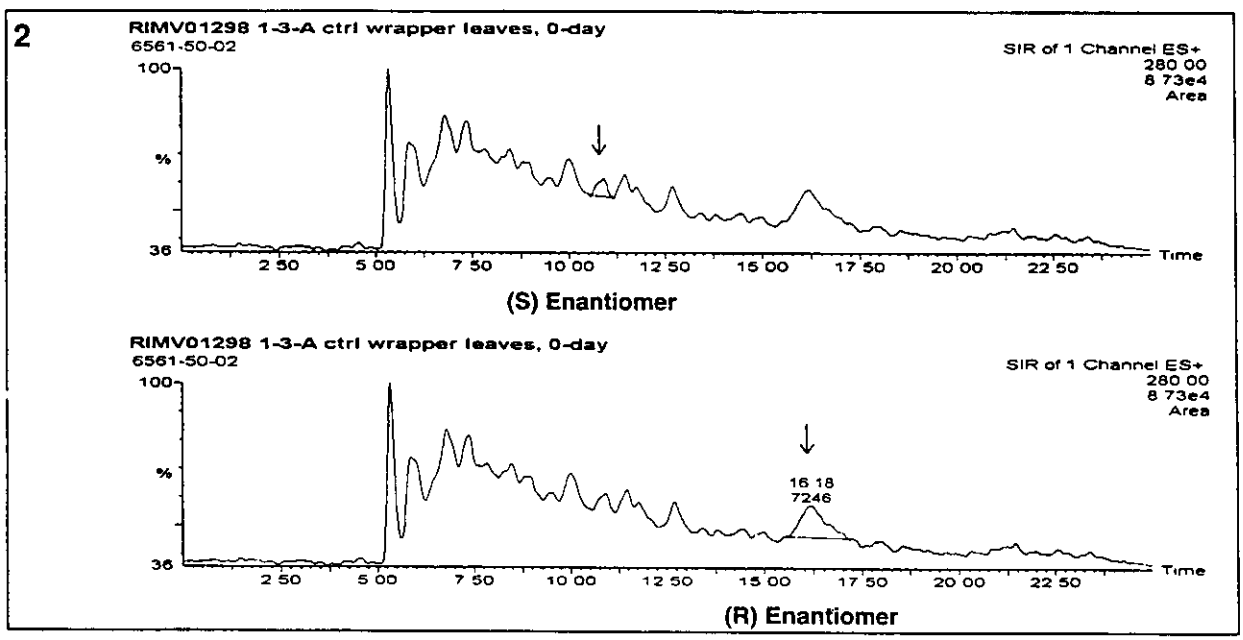


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-2-B	(R)-treated	0	43 043	1 150	0 027	47.190	1 119	1 146

FIGURE 9: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY CABBAGE WRAPPER LEAVES

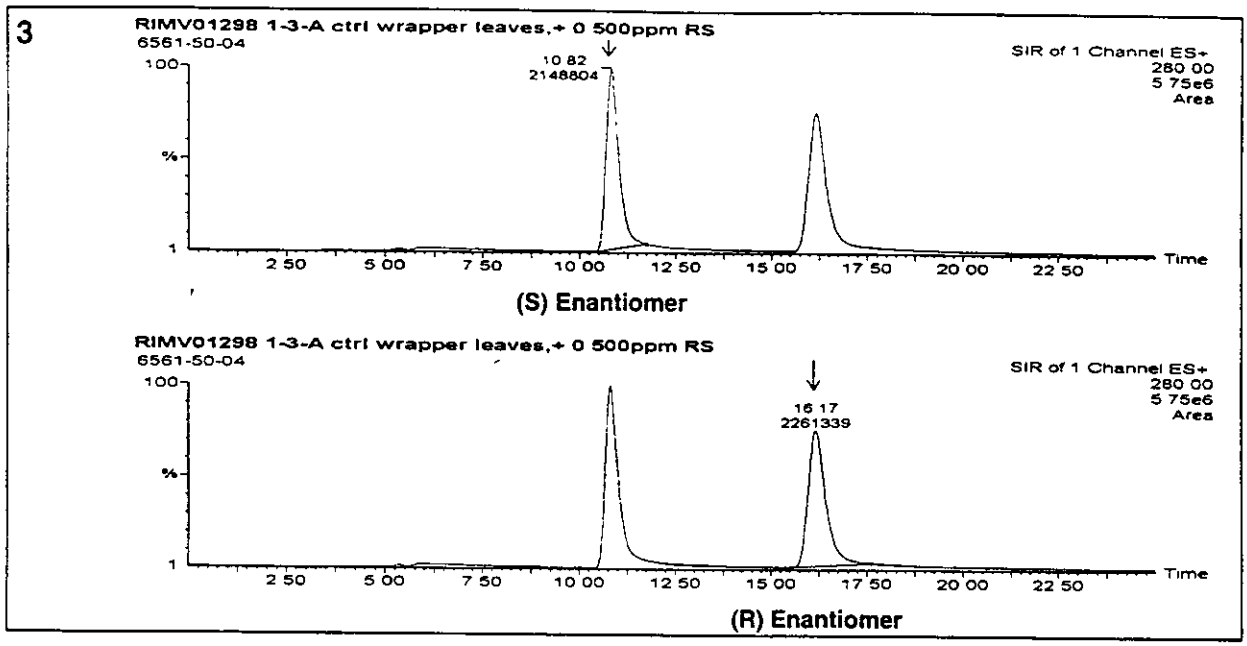


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 2000	15.00	88994 4	0	0 9984	94143 1	0	0 9970

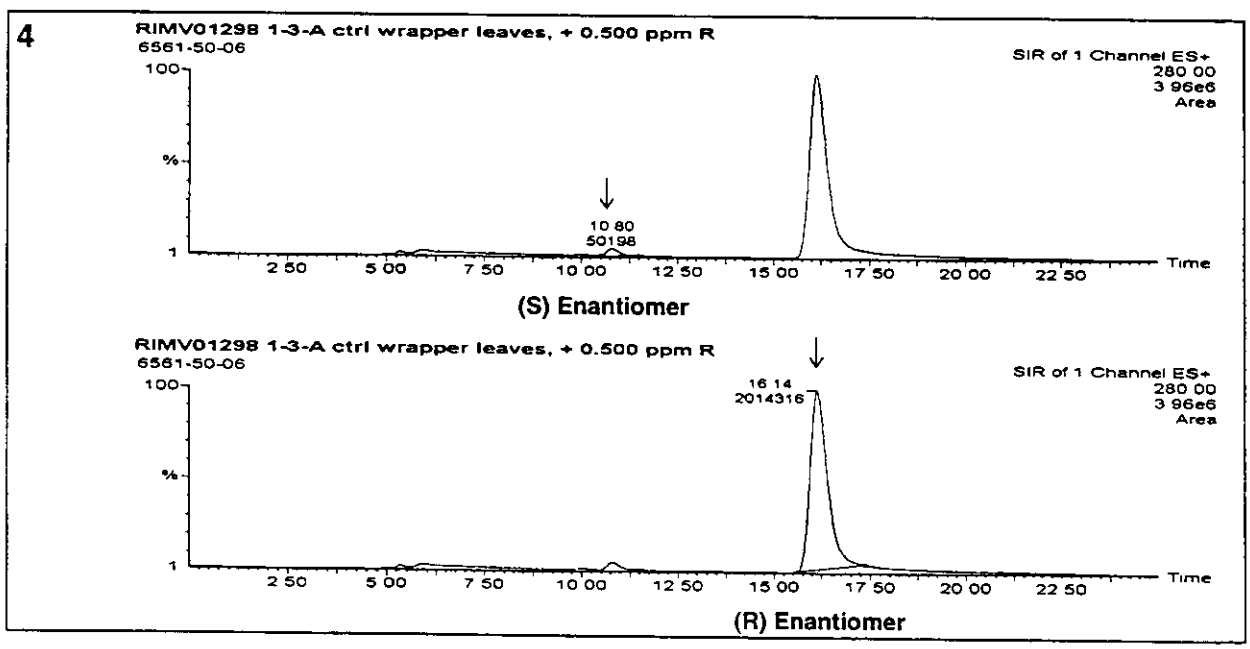


Sample		mg	(S) Enantiomer			(R) Enantiomer		
Code	Desc.	injected	ng	ppm uncorr	ppm	ng	ppm uncorr	ppm
1-3-A	control	42 926	0 020	0 000	<0 005	0 080	0 002	<0 005

FIGURE 9: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY CABBAGE WRAPPER LEAVES (Continued)

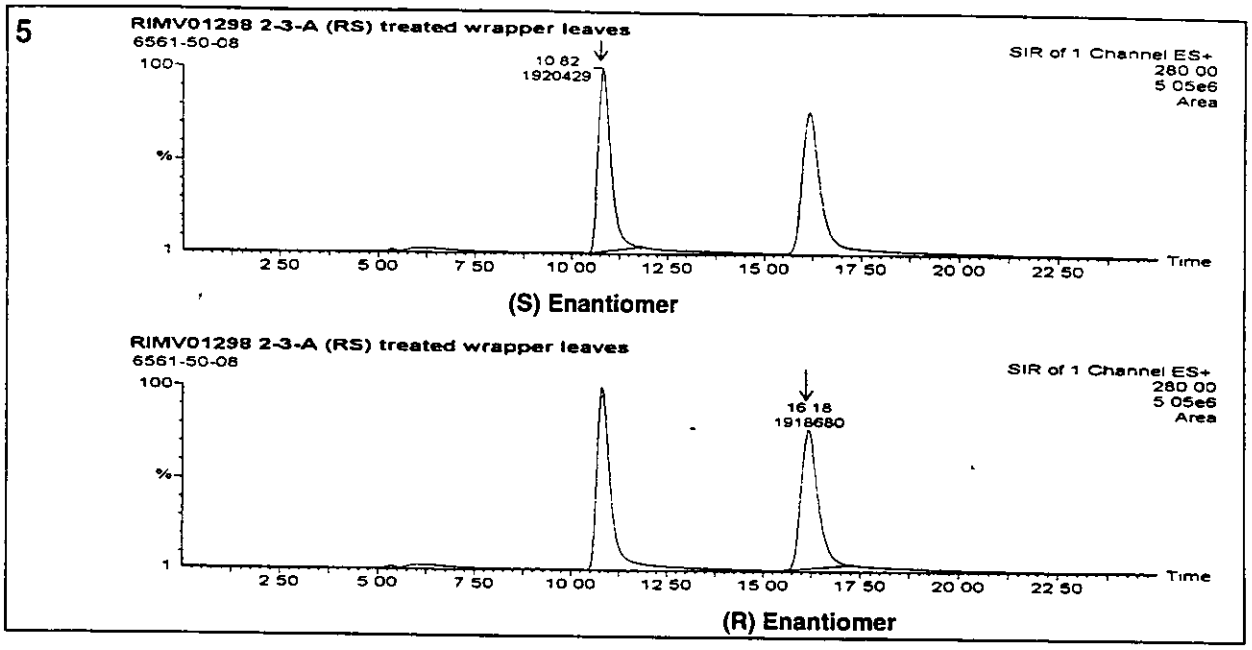


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec
1-3-A	recovery	0 50 ppm	42 887	24 150	0.563	113%	24.020	0 560	112%

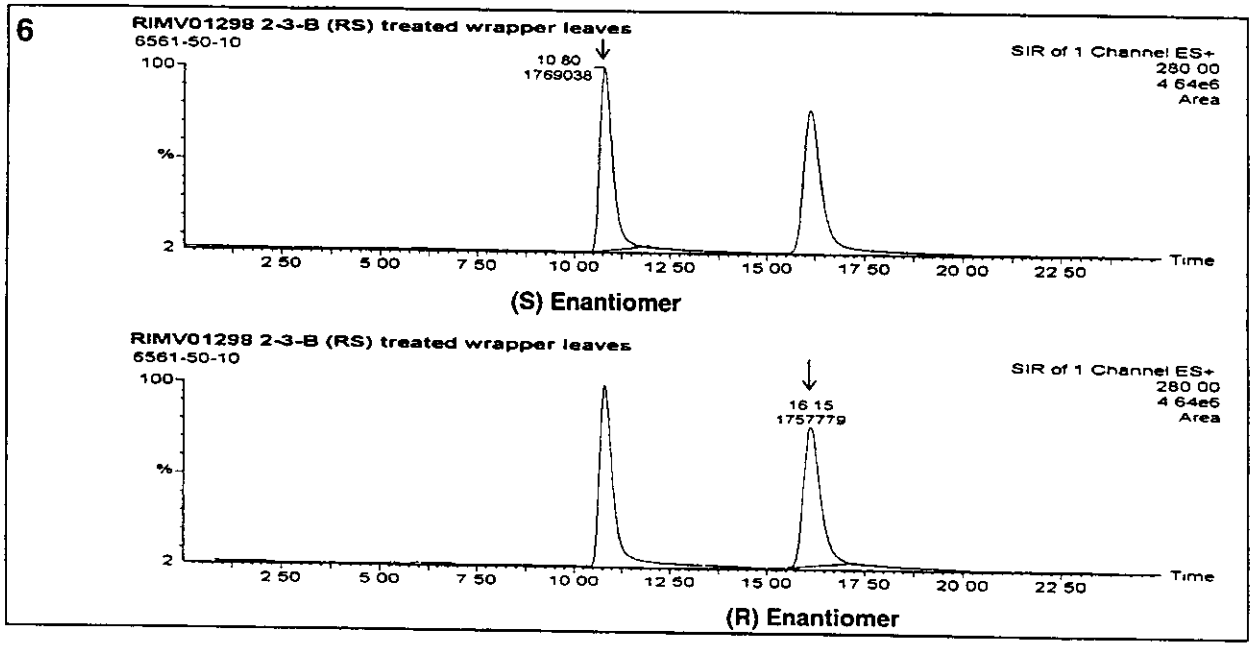


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec
1-3-A	recovery	0 50 ppm	42 926	0 560	0 013	-	21 400	0 499	99%

FIGURE 9: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY CABBAGE WRAPPER LEAVES (Continued)

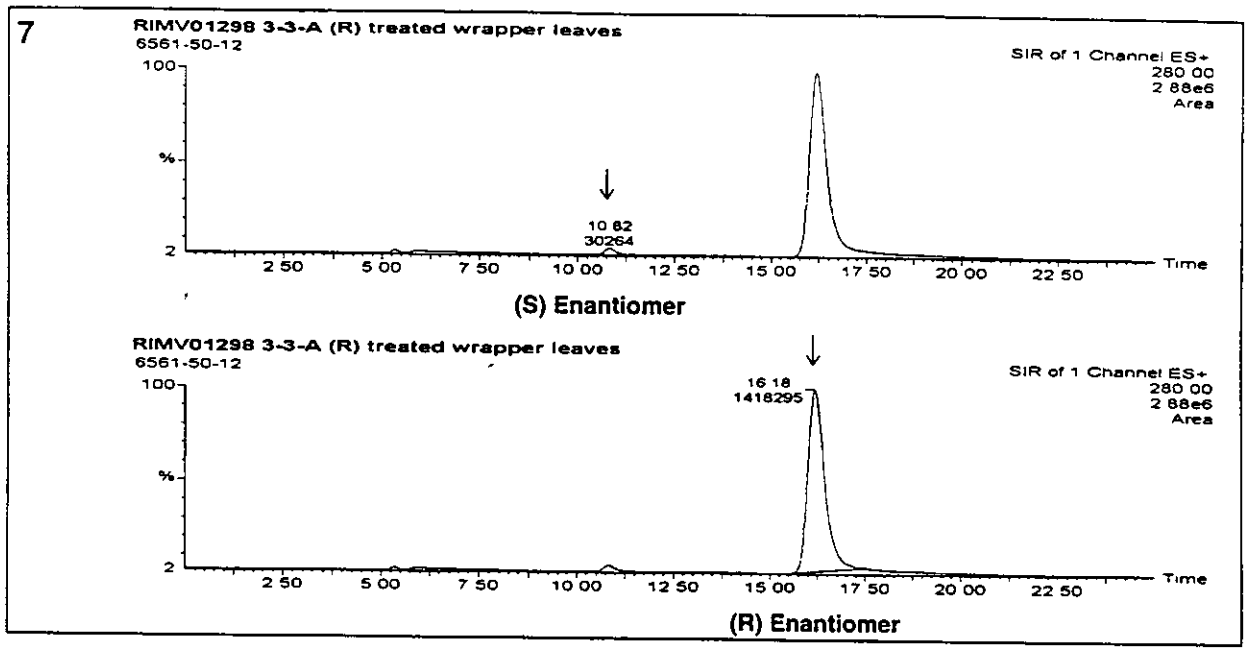


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr.	(RS) Racemate ppm
2-3-A	(RS)-treated	0	6.868	21.580	3 142	20 380	2.967	6 109

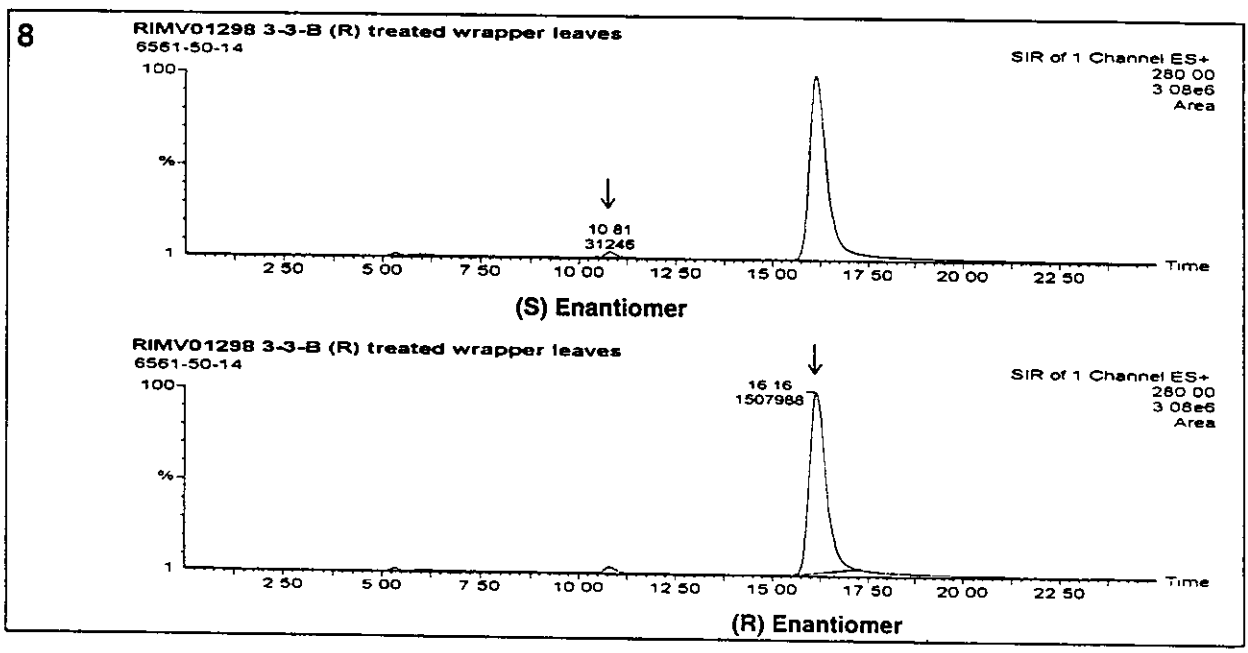


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-3-B	(RS)-treated	0	6.862	19.880	2 897	18.670	2.721	5.618

FIGURE 9: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY CABBAGE WRAPPER LEAVES (Continued)

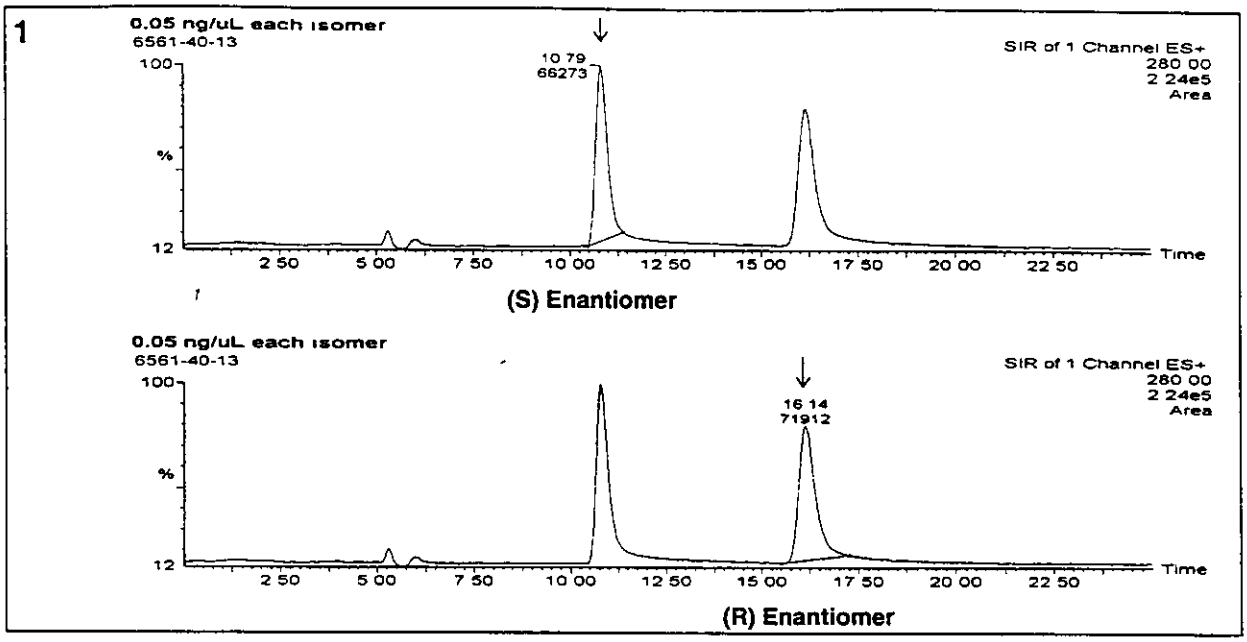


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-3-A	(R)-treated	0	6 856	0 340	0.050	15.070	2.198	2 248

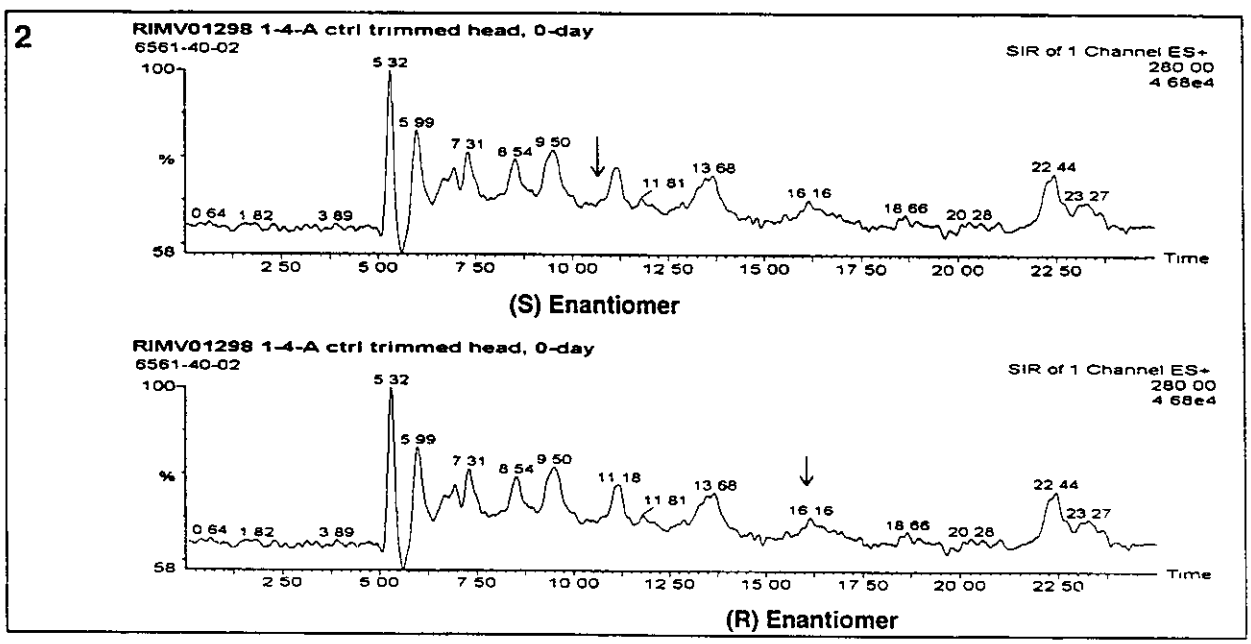


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-3-B	(R)-treated	0	6 874	0 350	0 051	16.020	2 330	2 381

FIGURE 10: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY TRIMMED CABBAGE HEADS

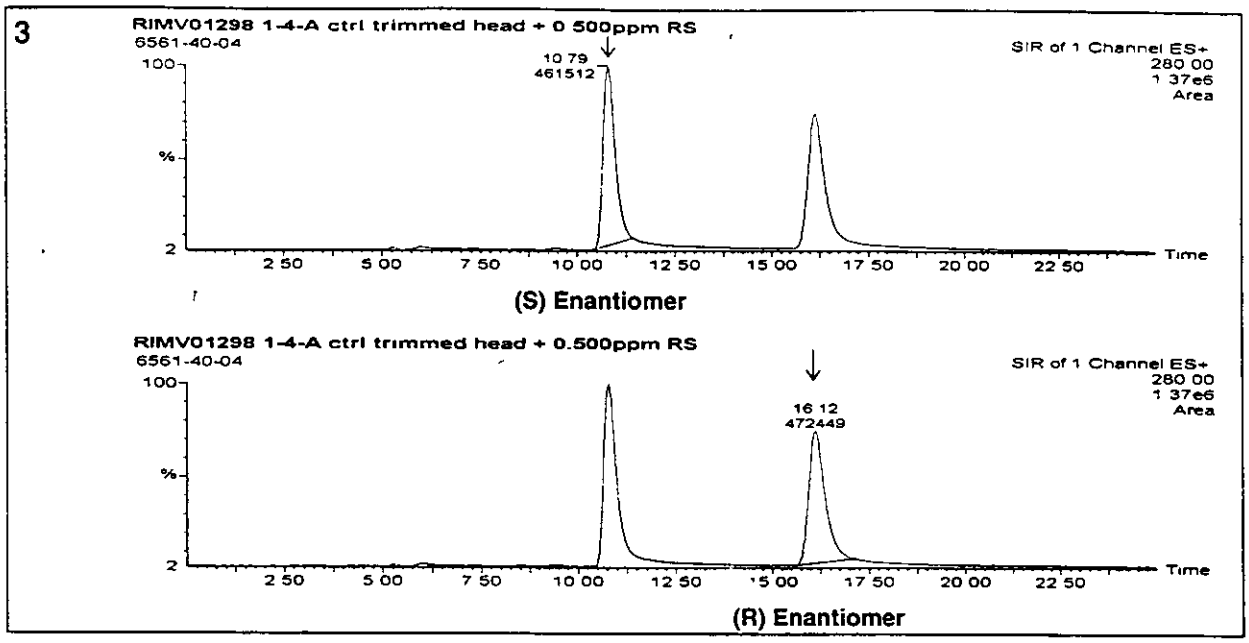


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 0500	3 750	20762 9	0	0 9991	21453.8	0	0 9987

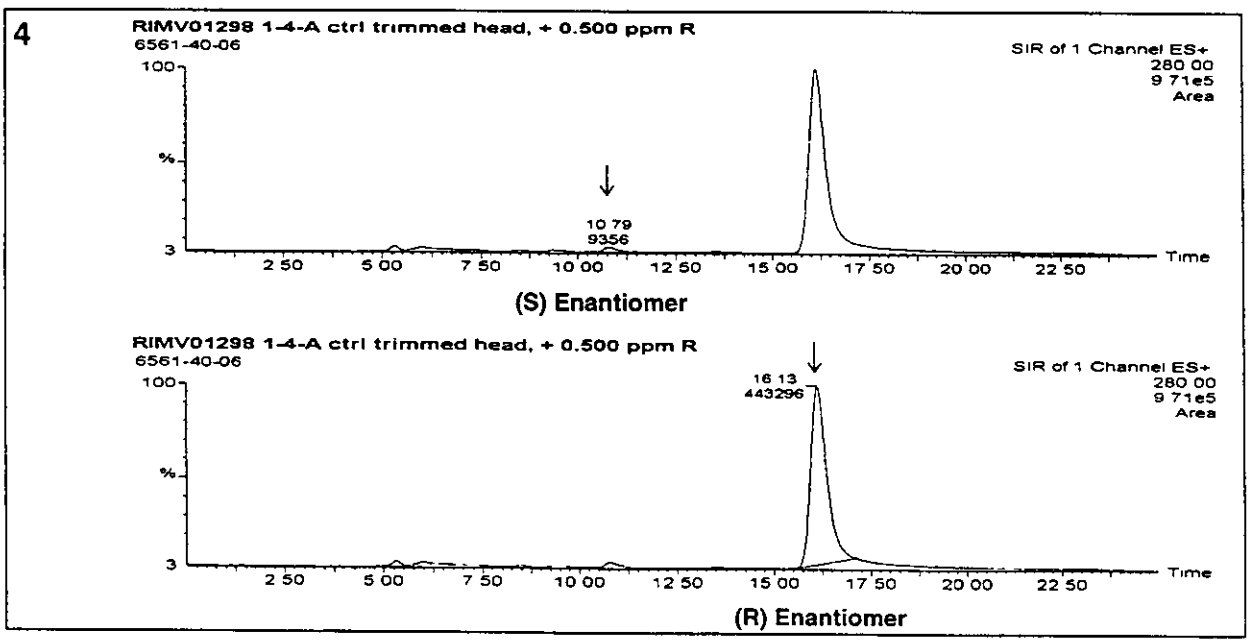


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-4-A	control	42 965	0 000	0 000	<0 005	0 000	0 000	<0 005

FIGURE 10: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY TRIMMED CABBAGE HEADS (Continued)

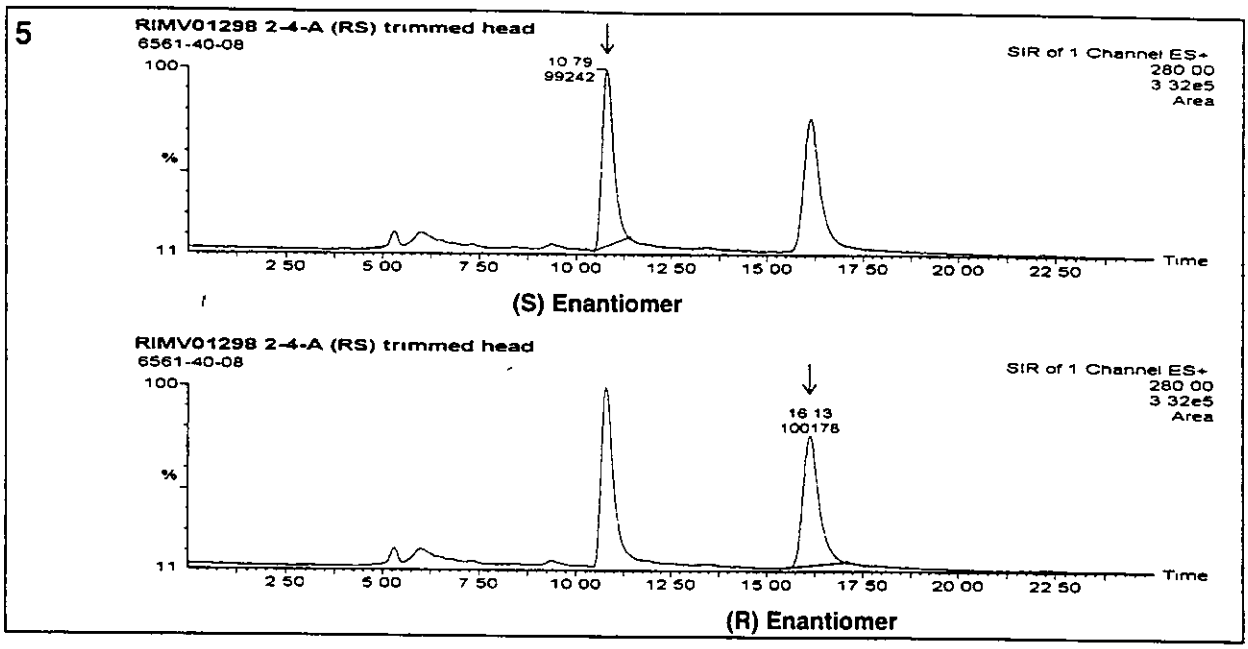


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm uncorr	(S) Enantiomer % Rec.	(R) Enantiomer ng	(R) Enantiomer ppm uncorr	(R) Enantiomer % Rec
1-4-A	recovery	0.50 ppm	42 926	22 230	0 518	103%	22 020	0 513	103%

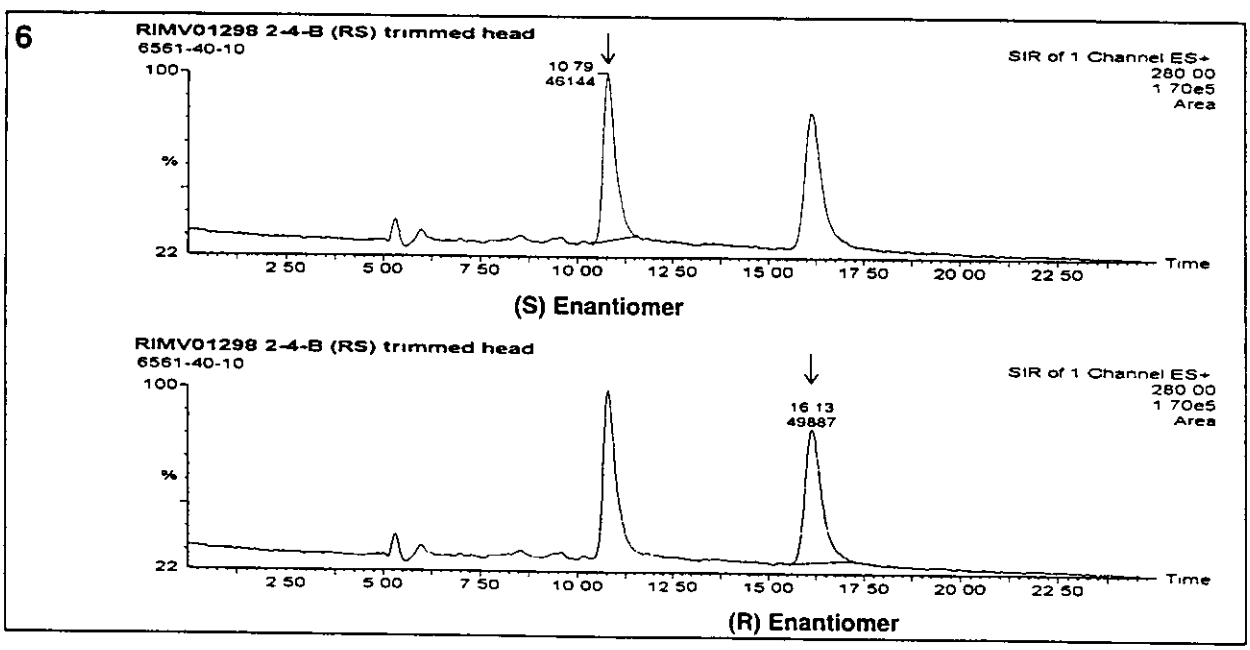


Sample Code	Desc	Amt (S), (R)⁻ Added	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm uncorr	(S) Enantiomer % Rec	(R) Enantiomer ng	(R) Enantiomer ppm uncorr	(R) Enantiomer % Rec
1-4-A	recovery	0 50 ppm	42 965	0 450	0 010	-	20 660	0 481	96%

FIGURE 10: CHROMATOGRAMS.
(S) AND (R) ENANTIOMERS IN 0-DAY TRIMMED CABBAGE HEADS (Continued)

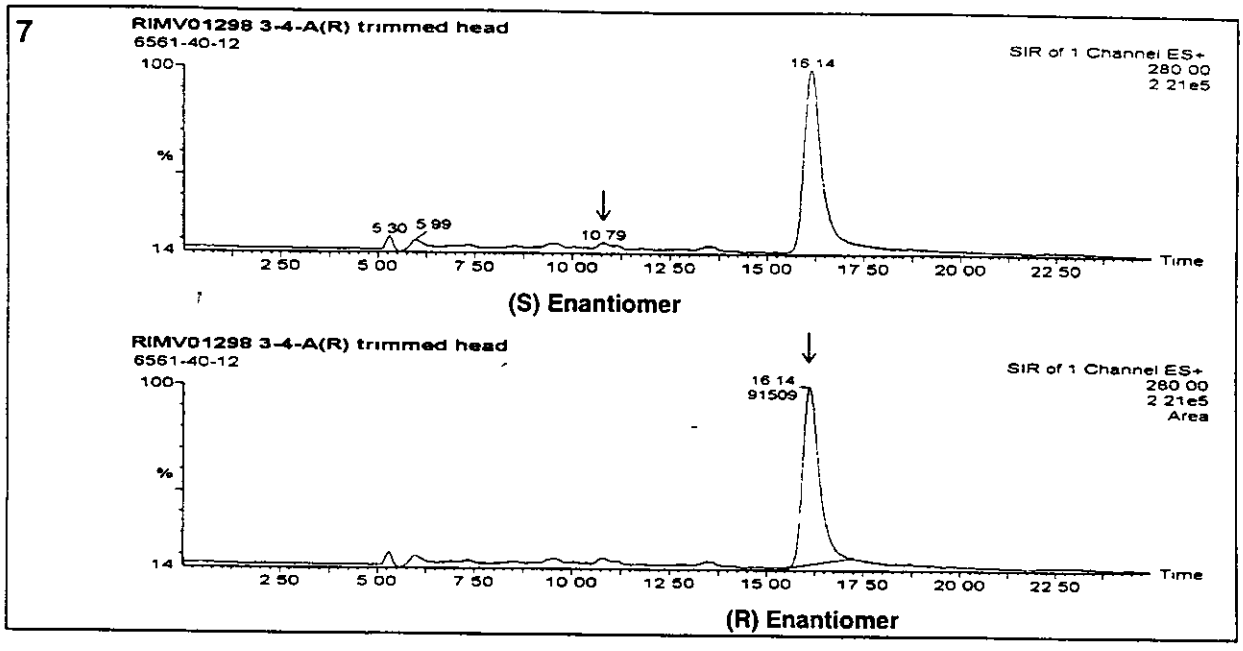


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-4-A	(RS)-treated	0	42 965	4 780	0 111	4.670	0 109	0.220

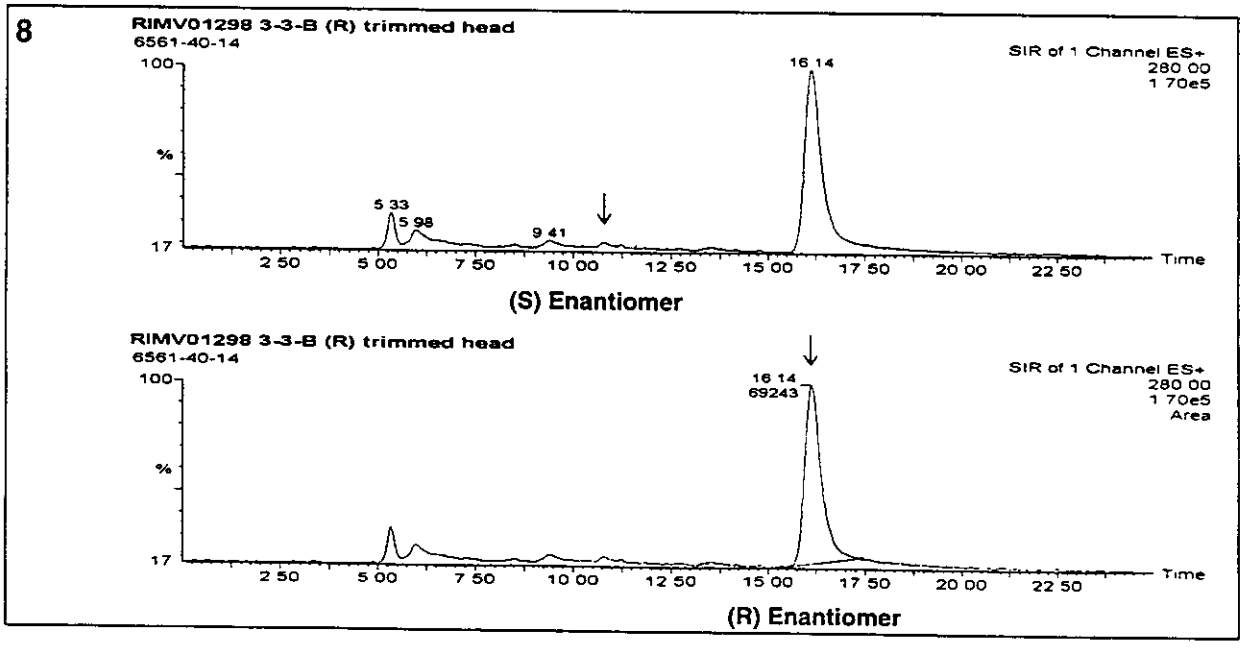


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-4-B	(RS)-treated	0	42 808	2 220	0 052	2 330	0 055	0 107

FIGURE 10: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN 0-DAY TRIMMED CABBAGE HEADS (Continued)

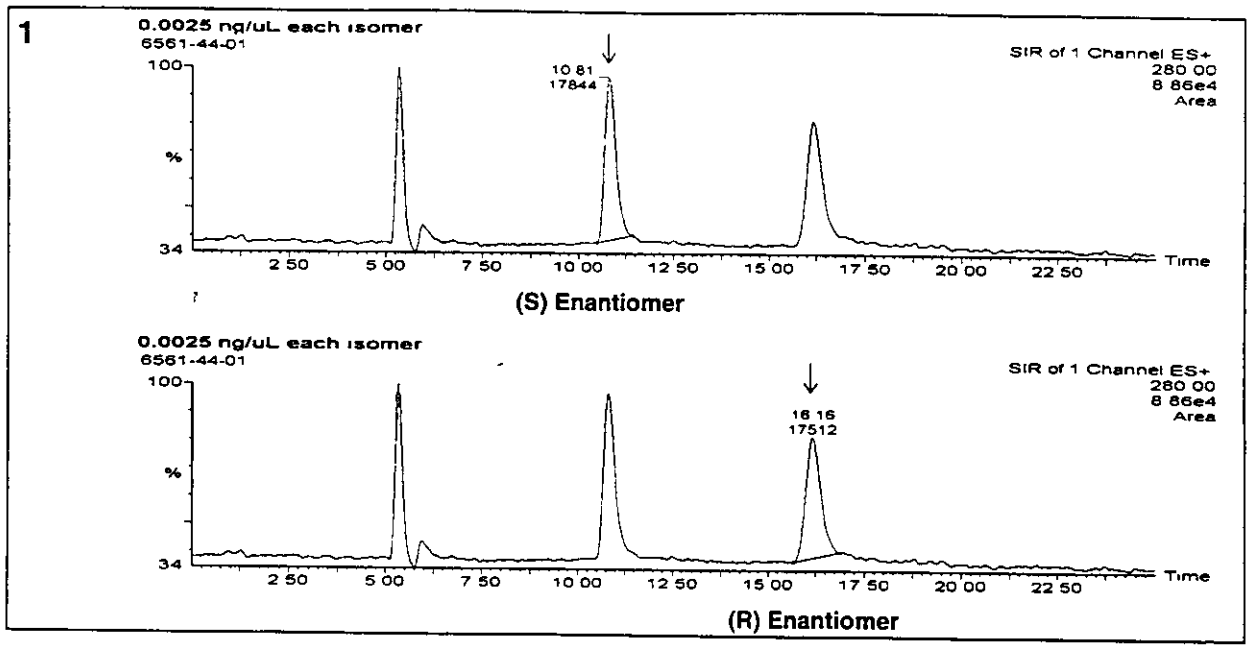


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-4-A	(R)-treated	0	42 887	0.000	<0 005	4 270	0 100	0.100

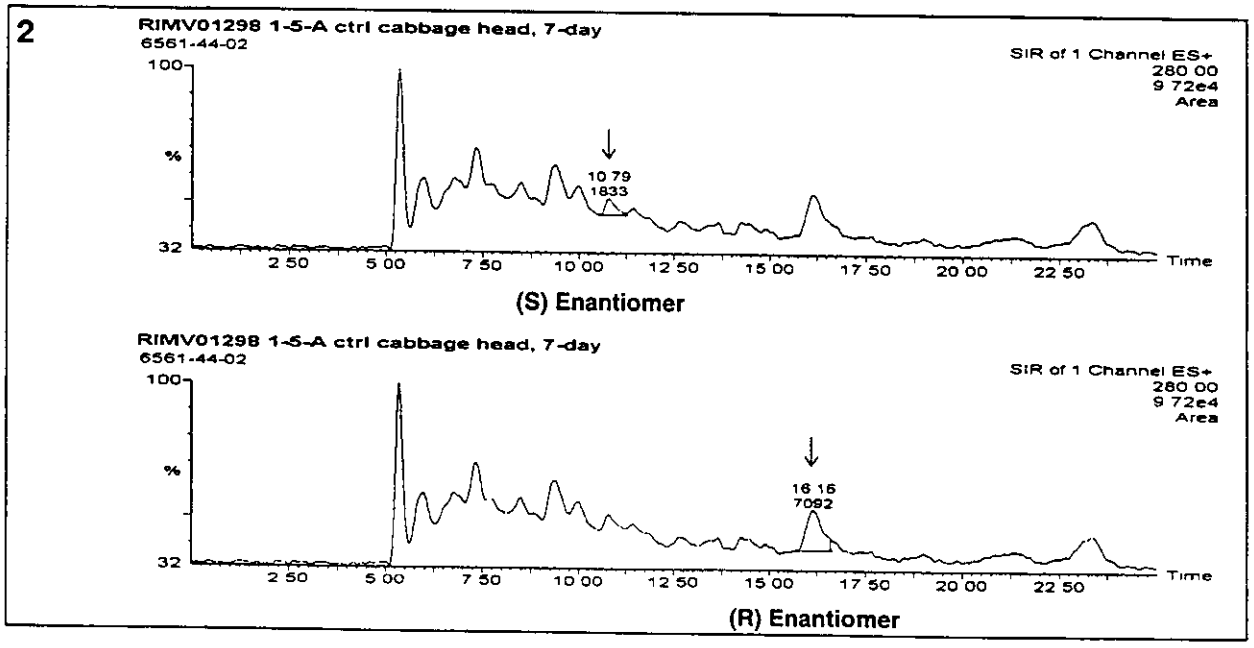


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-4-B	(R)-treated	0	43 357	0 000	<0 005	3 230	0.075	0 075

FIGURE 11. CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN CABBAGE HEADS

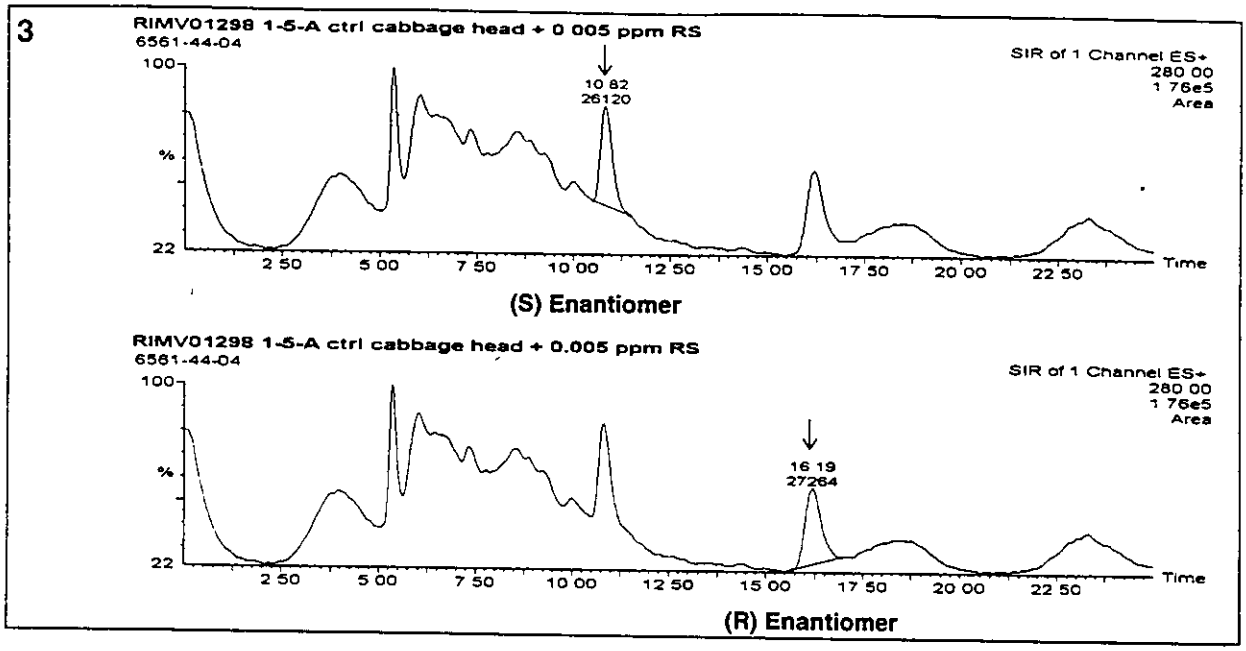


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 0025	0 1875	105664	0	0 9989	111032	0	0 9998

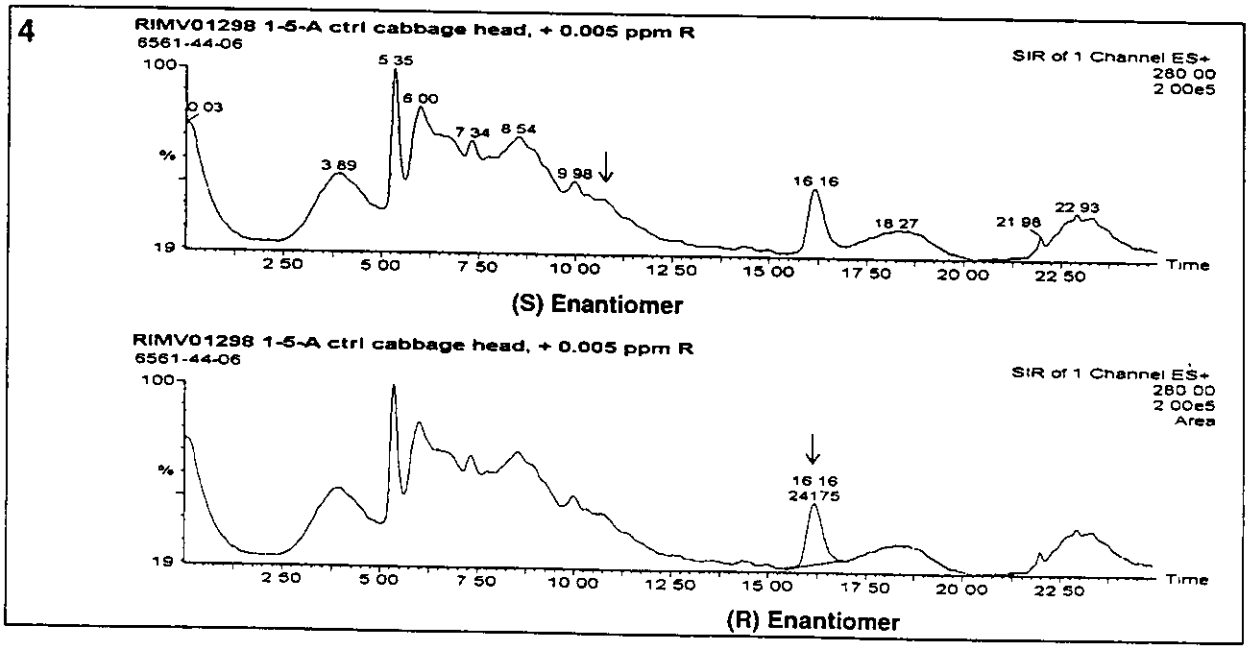


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-5-A	control	43.004	0 020	0 000	<0 005	0 060	0 001	<0 005

FIGURE 11: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN CABBAGE HEADS (Continued)

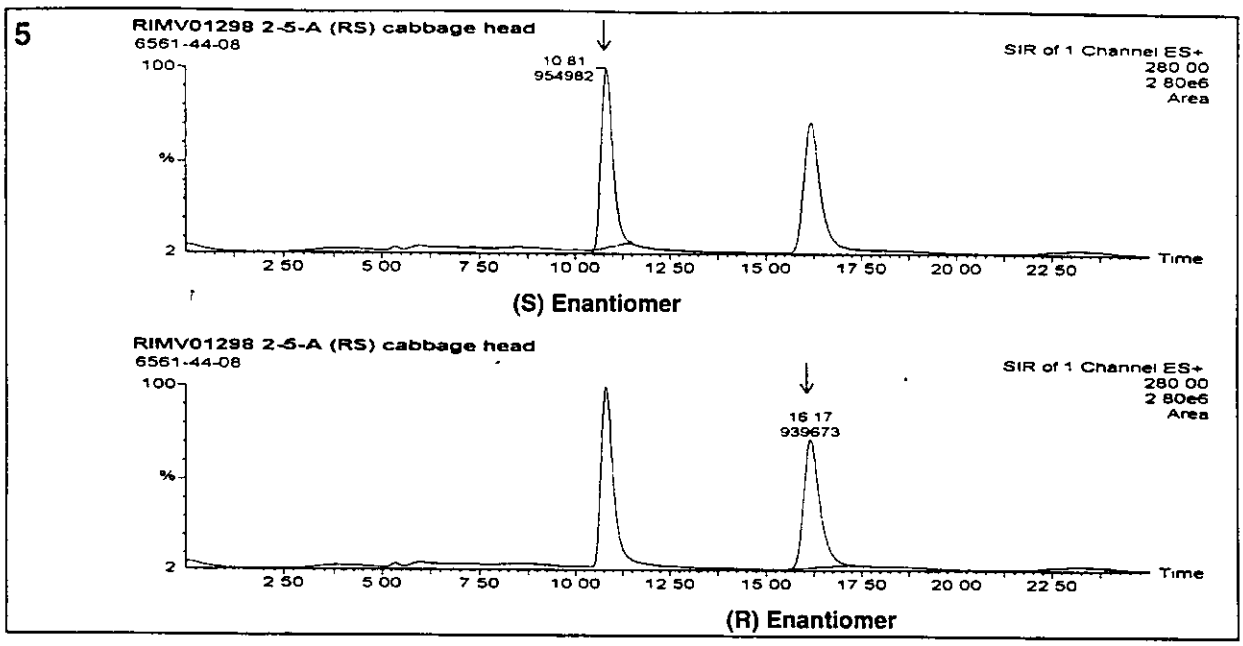


Sample Code	Desc	Amt. (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec
1-5-A	recovery	0 005 ppm	42.965	0 250	0 006	107%	0.250	0 006	88%

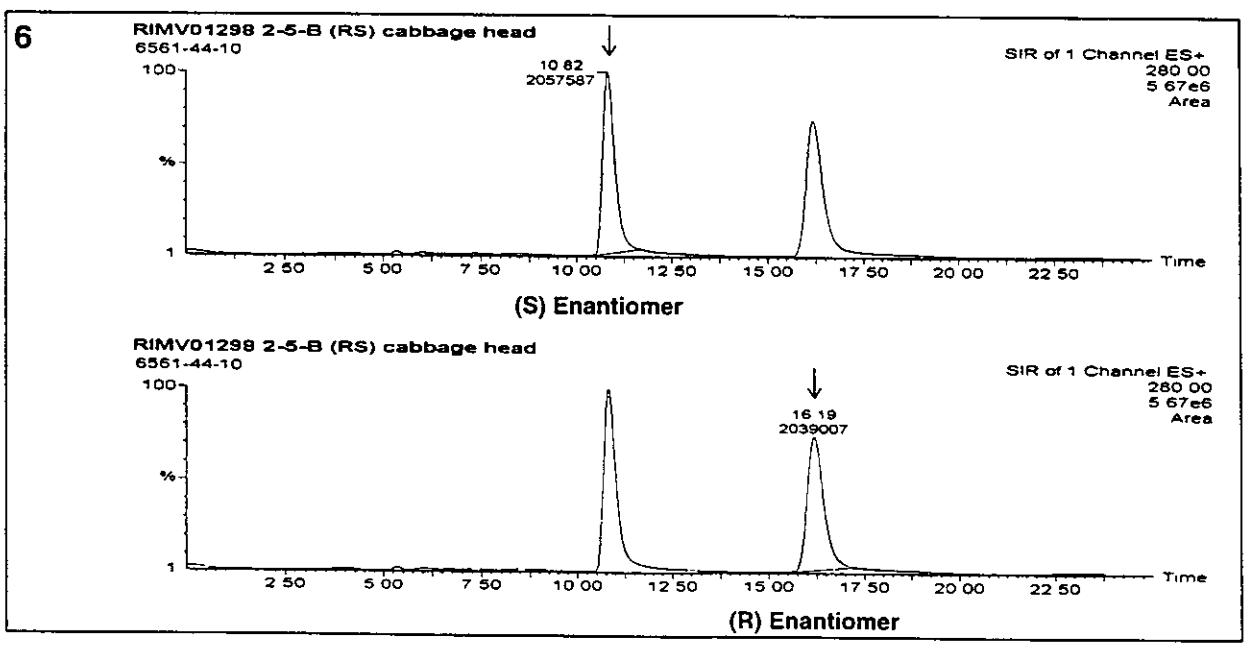


Sample Code	Desc	Amt. (S), (R)⁻ Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec.	(R) Enantiomer ng	ppm uncorr	% Rec
1-5-A	recovery	0 005 ppm	42.926	0.000	0 000	-	0 220	0 005	75%

FIGURE 11: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN CABBAGE HEADS (Continued)

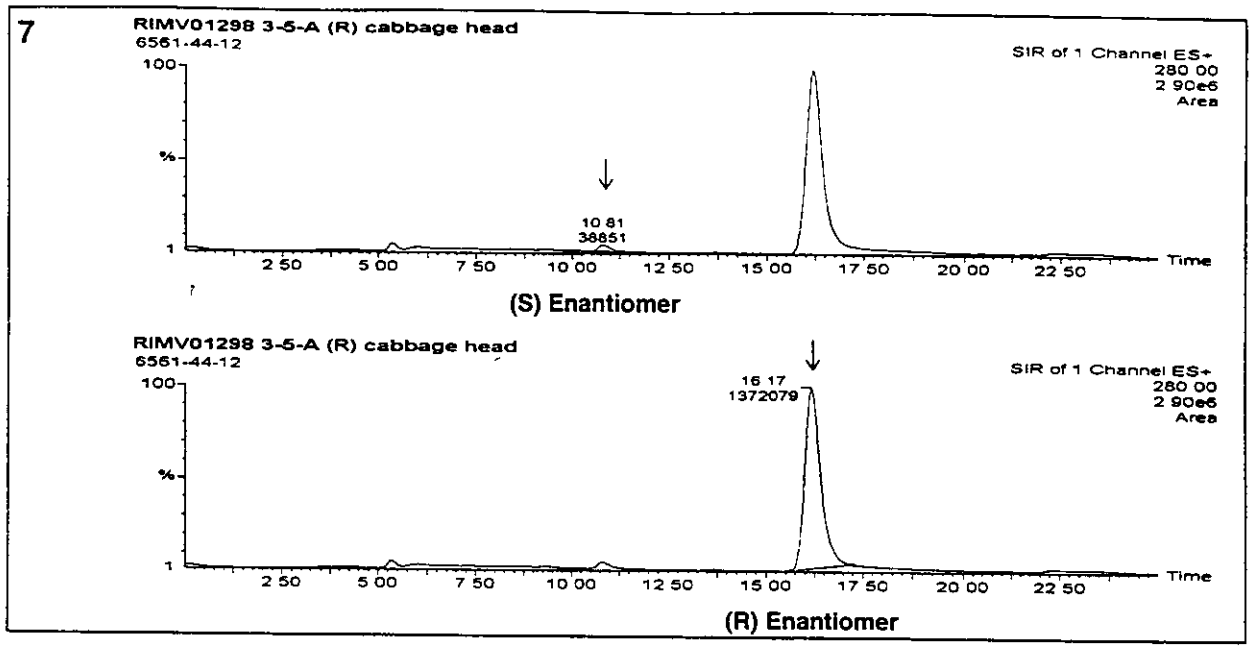


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-5-A	(RS)-treated	7	42.926	9 040	0 211	8 460	0.242	0.453

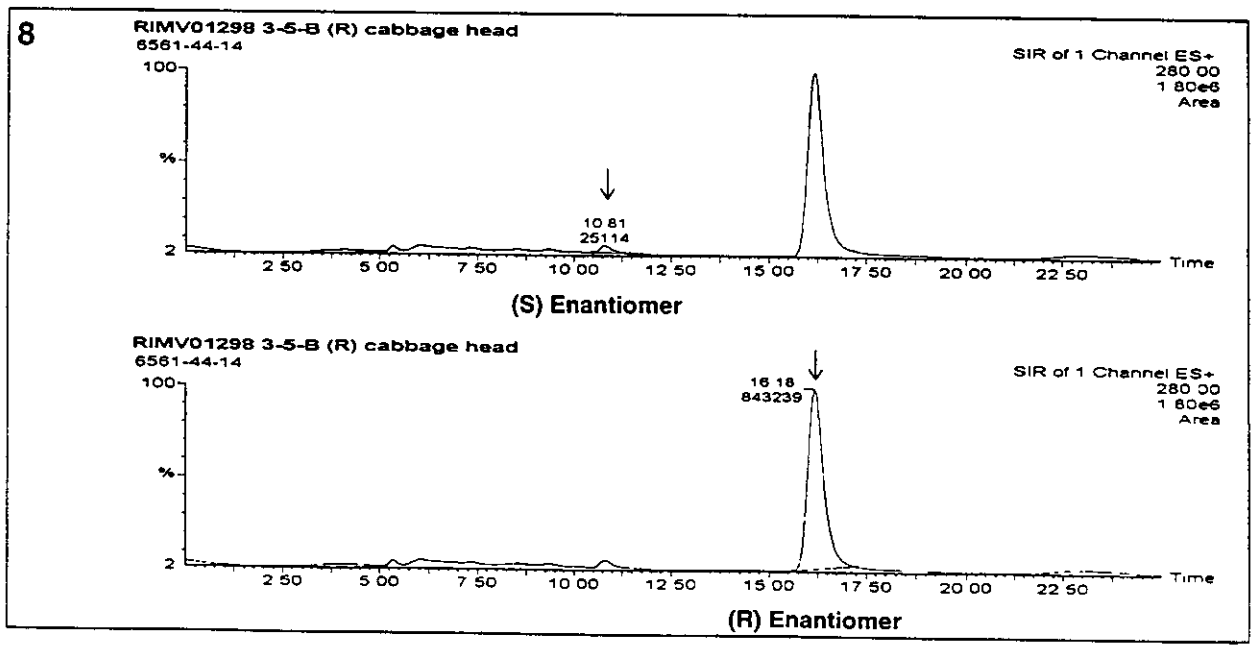


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-5-B	(RS)-treated	7	42.926	19 470	0.454	18 360	0 525	0 979

FIGURE 11: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN CABBAGE HEADS (Continued)

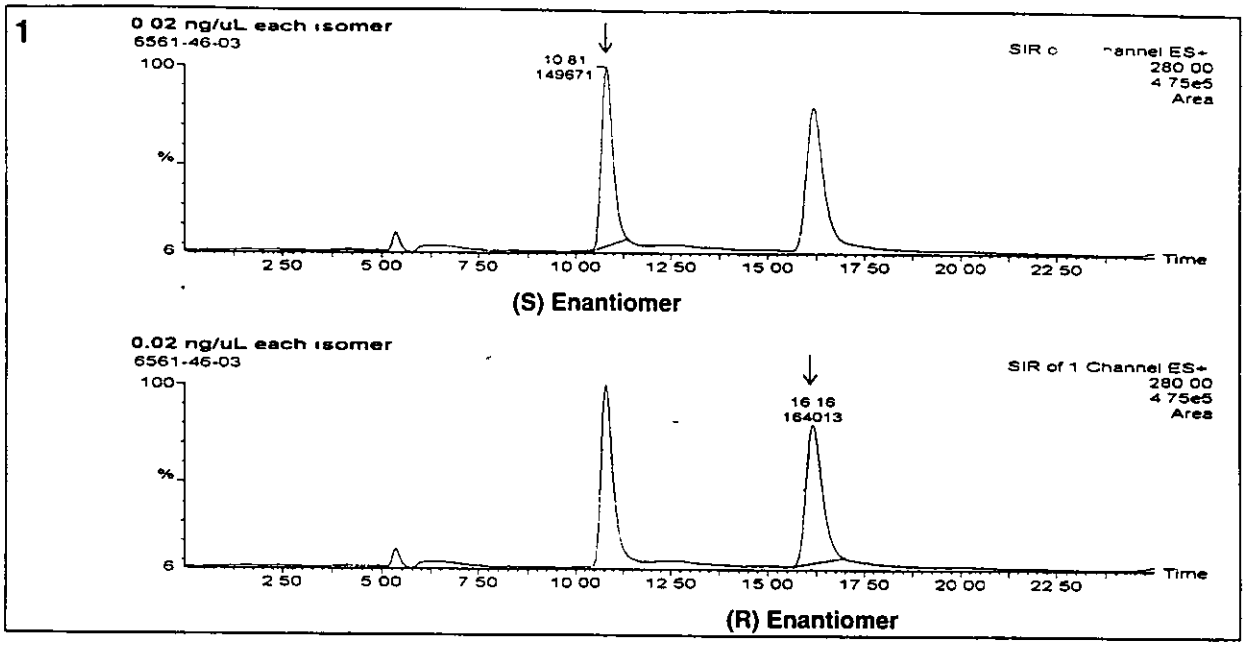


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-5-A	(R)-treated	7	43.083	0.370	0.009	12 360	0.352	0.361

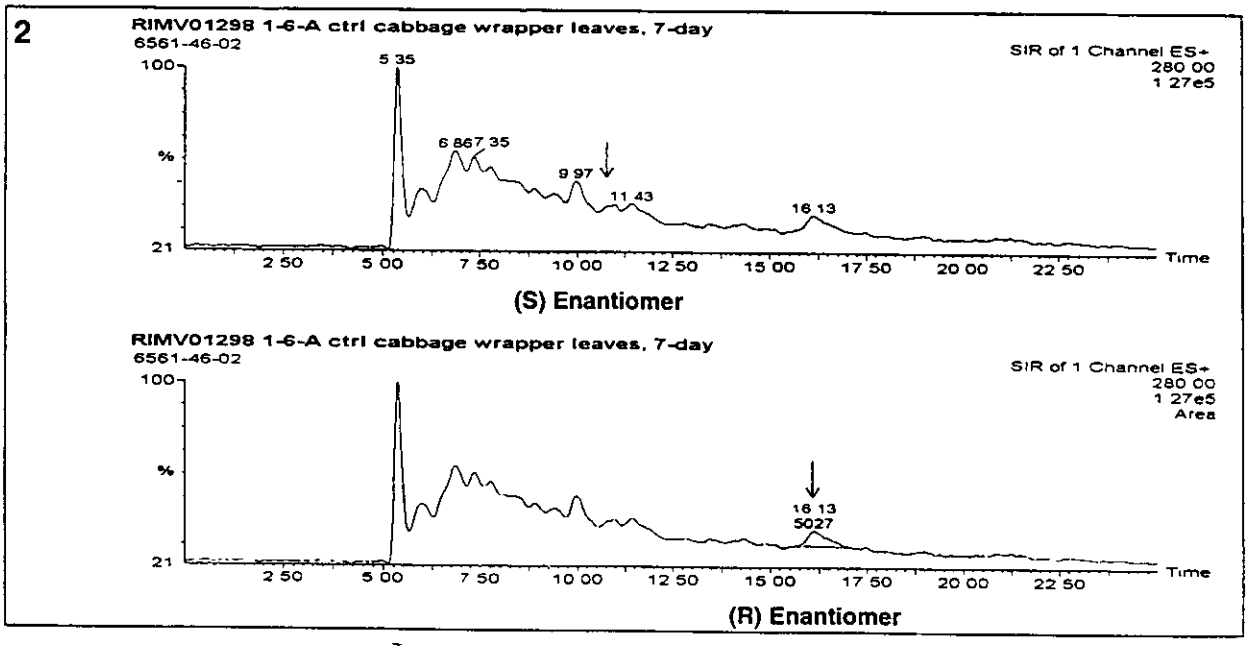


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-5-B	(R)-treated	7	44 516	0 240	0 005	7 590	0 209	0.214

FIGURE 12: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN CABBAGE WRAPPER LEAVES

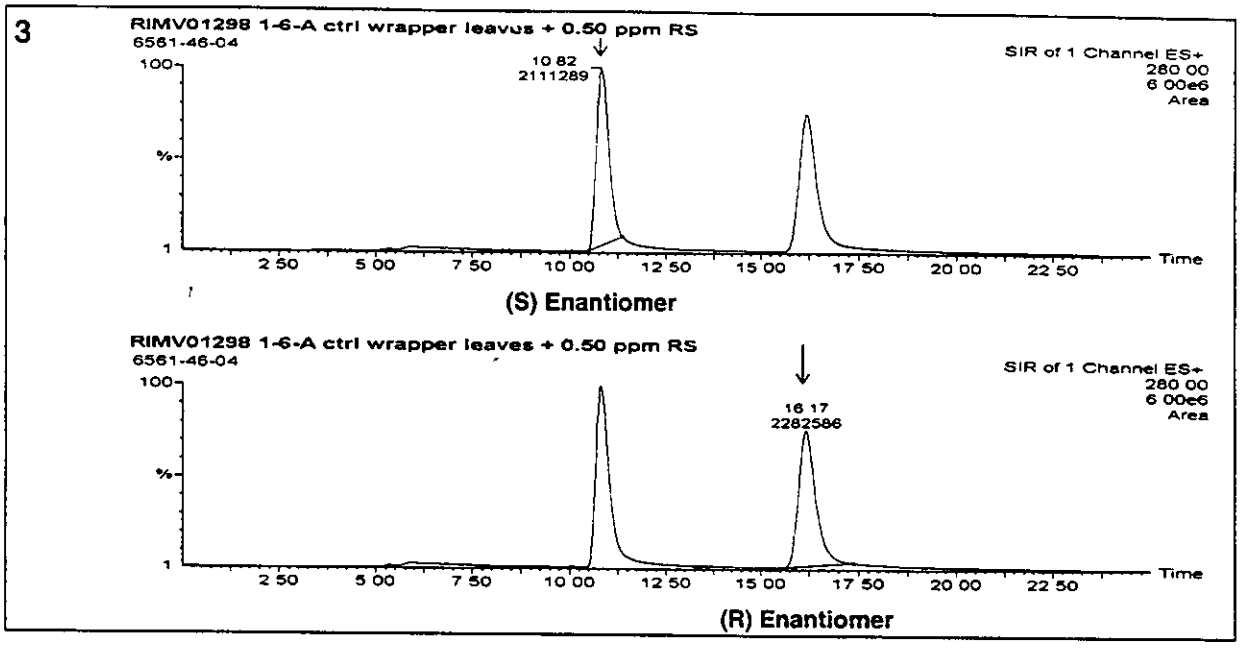


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0.0200	1 500	94933.2	0	0 9993	101448	0	0 9994

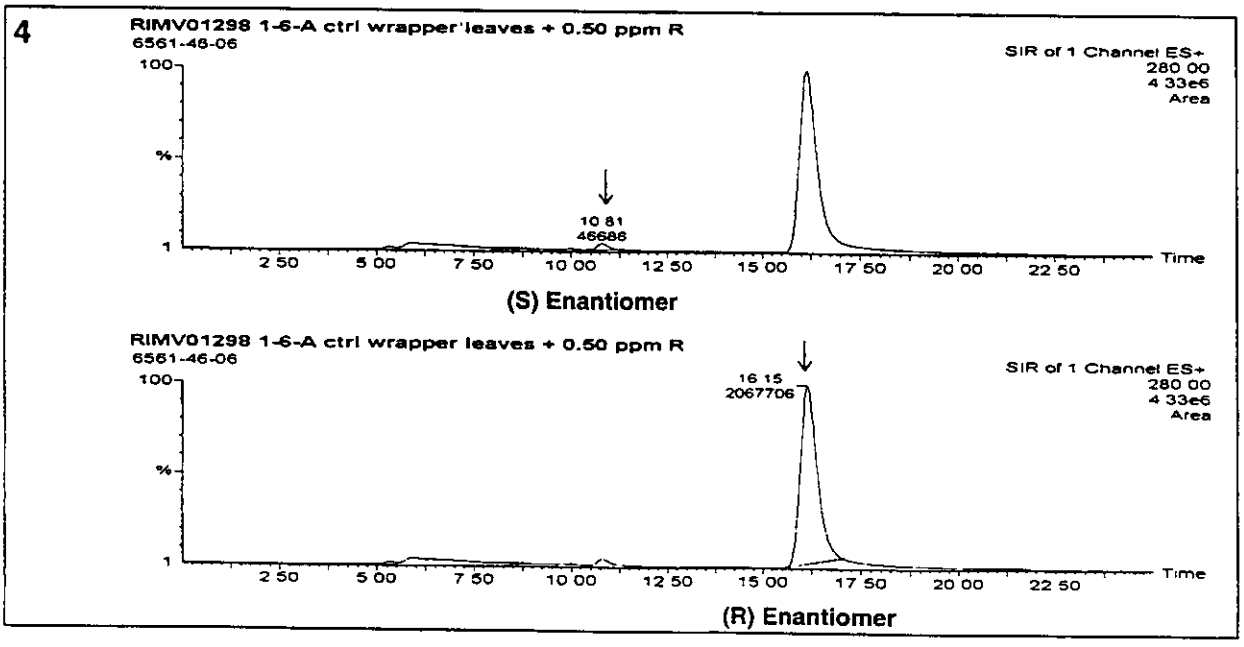


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-6-A	control	42 887	0 000	0 000	<0 005	0 050	0 001	<0 005

FIGURE 12: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN CABBAGE WRAPPER LEAVES (Continued)

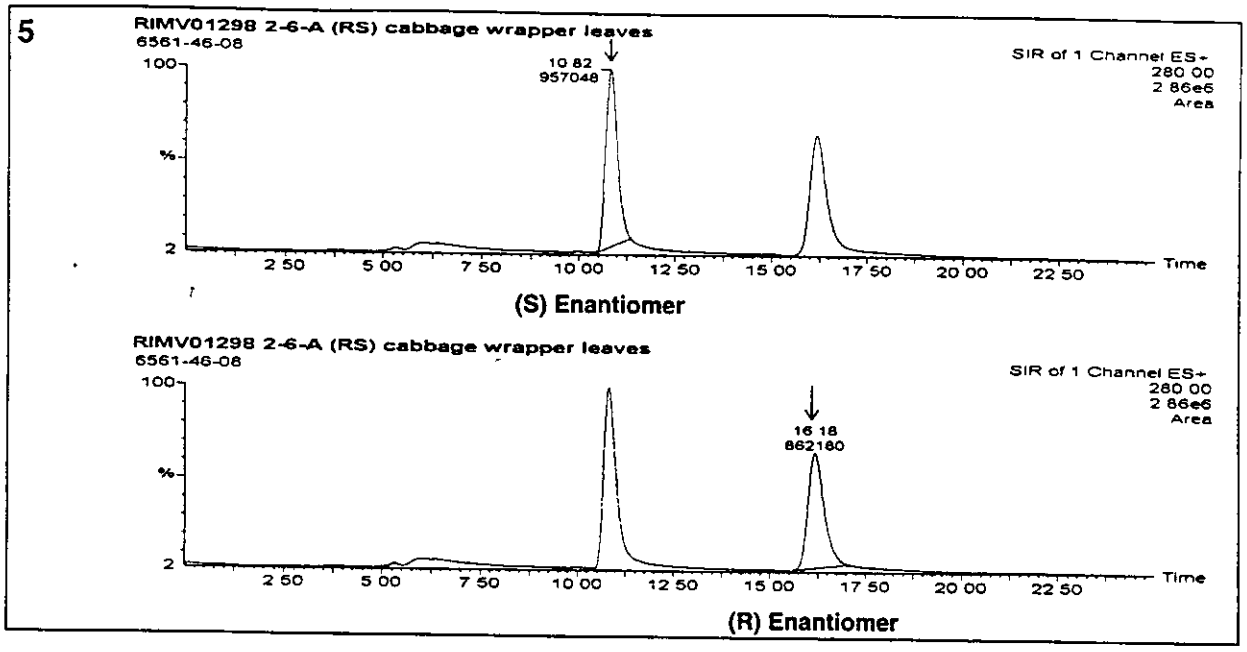


Sample Code	Desc	Amt. (S), (R) Added	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm uncorr.	(S) Enantiomer % Rec	(R) Enantiomer ng	(R) Enantiomer ppm uncorr	(R) Enantiomer % Rec.
1-6-A	recovery	0 50 ppm	42.965	22.240	0 518	104%	22 500	0 524	105%

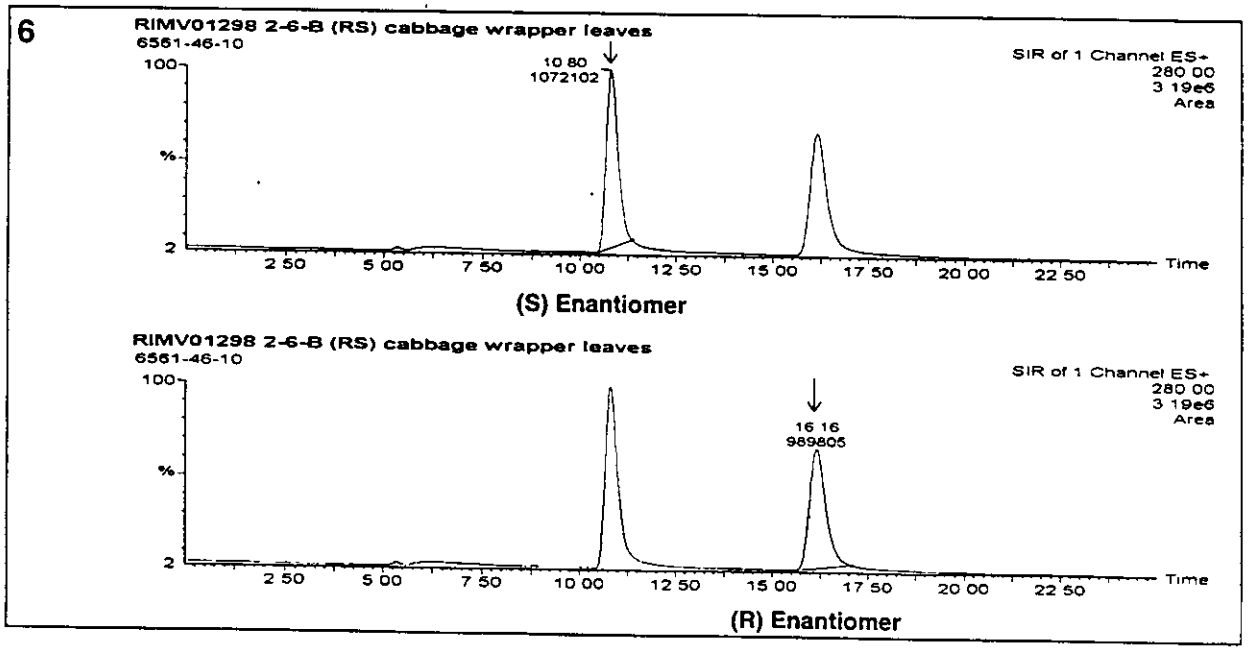


Sample Code	Desc	Amt (S), (R)⁻ Added	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm uncorr	(S) Enantiomer % Rec	(R) Enantiomer ng	(R) Enantiomer ppm uncorr	(R) Enantiomer % Rec
1-6-A	recovery	0 50 ppm	42 926	0 490	0 011	-	20 380	0 475	95%

FIGURE 12: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN CABBAGE WRAPPER LEAVES (Continued)

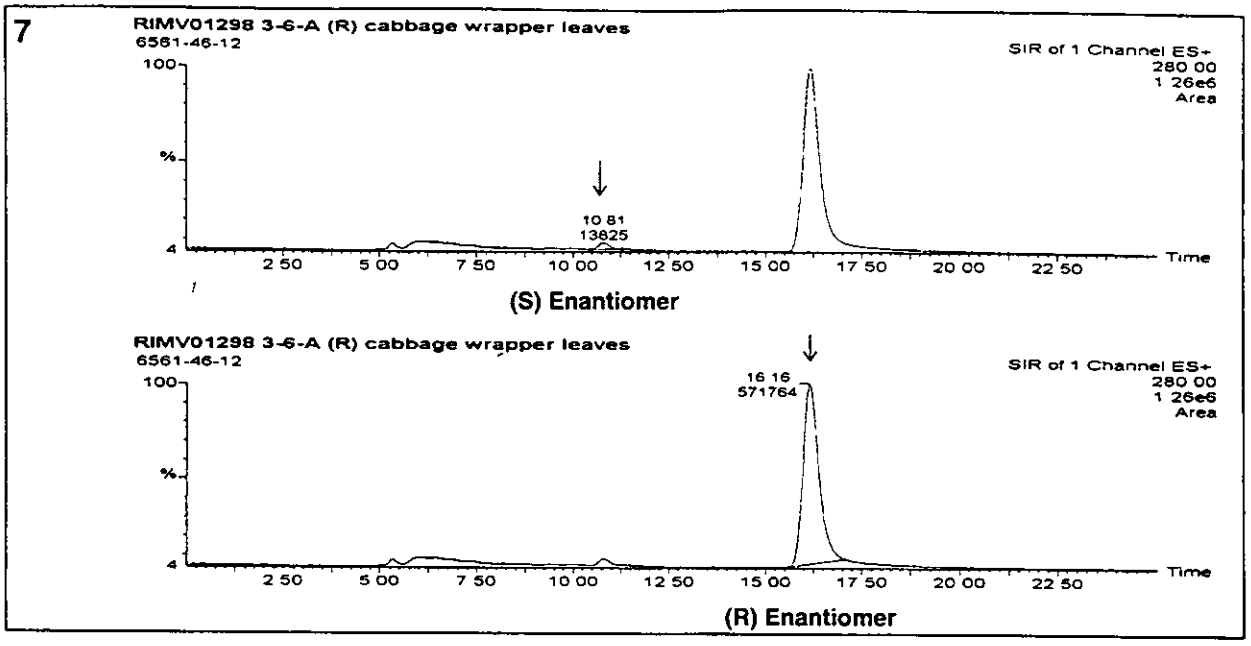


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-6-A	(RS)-treated	7	17 170	10 080	0 587	8 500	0 497	1 084

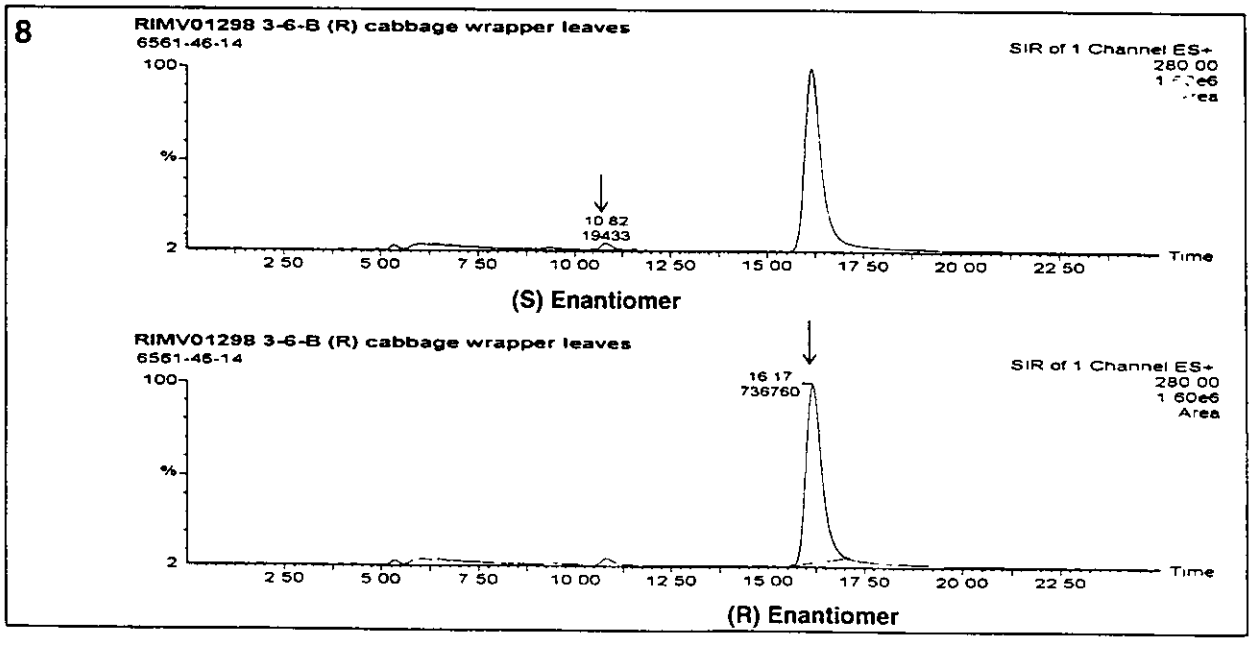


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-6-B	(RS)-treated	7	17.186	11 290	0 657	9 760	0 570	1 227

FIGURE 12: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN CABBAGE WRAPPER LEAVES (Continued)

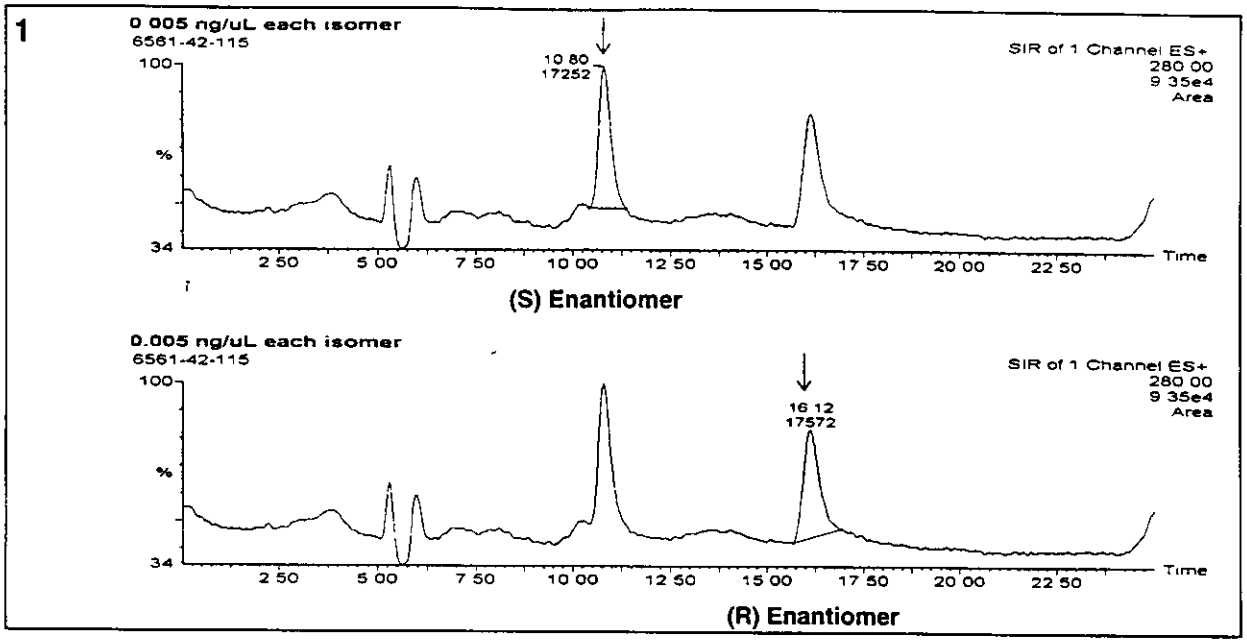


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-6-A	(R)-treated	7	17 217	0.150	0 009	5.640	0 329	0 338

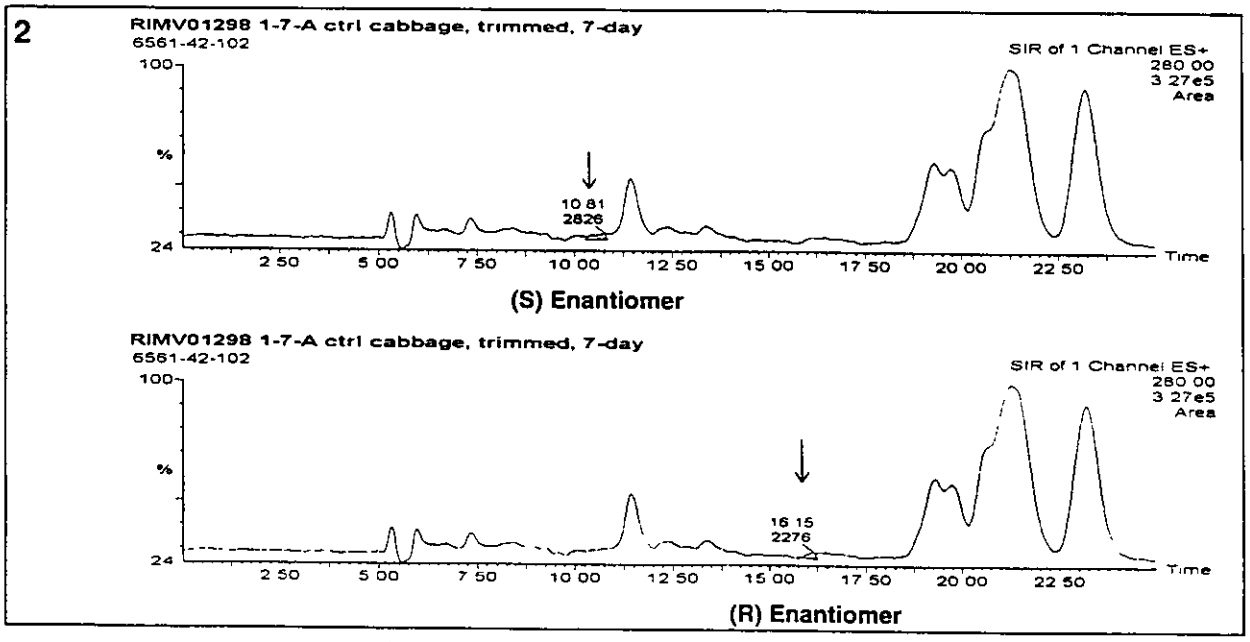


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-6-B	(R)-treated	7	17 186	0 200	0 012	7 260	0 424	0 436

FIGURE 13: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TRIMMED CABBAGE HEADS

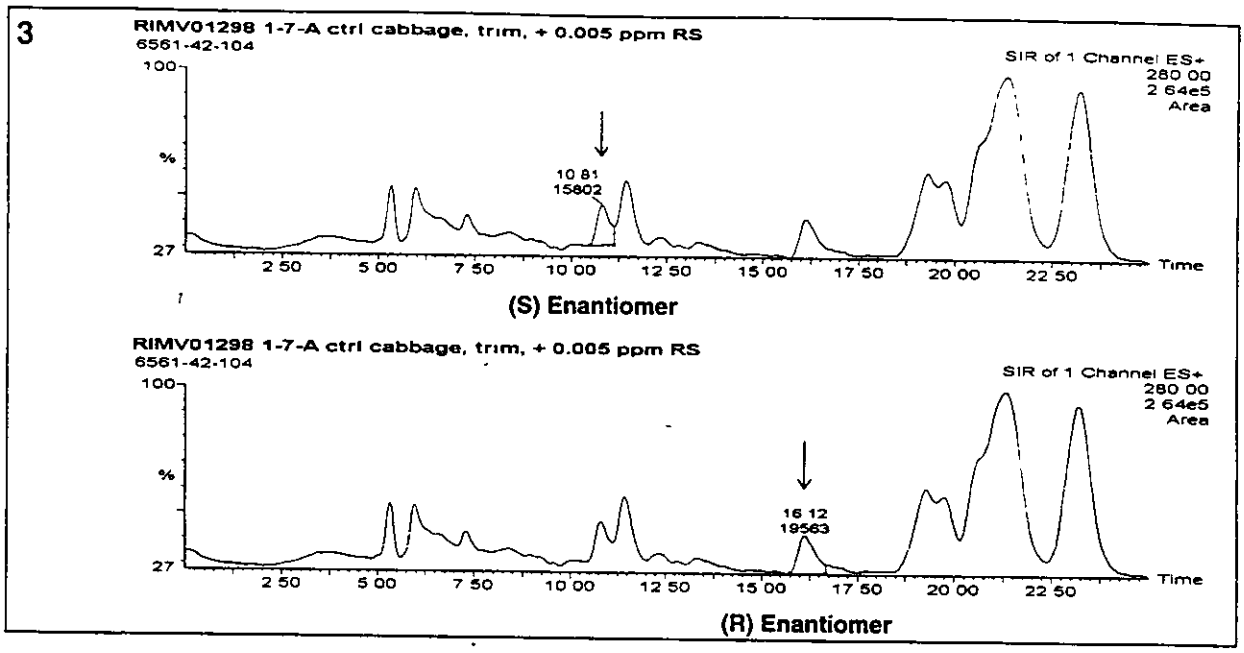


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 0050	0 3750	65131.1	0	0 9998	70736 8	0	0 9999

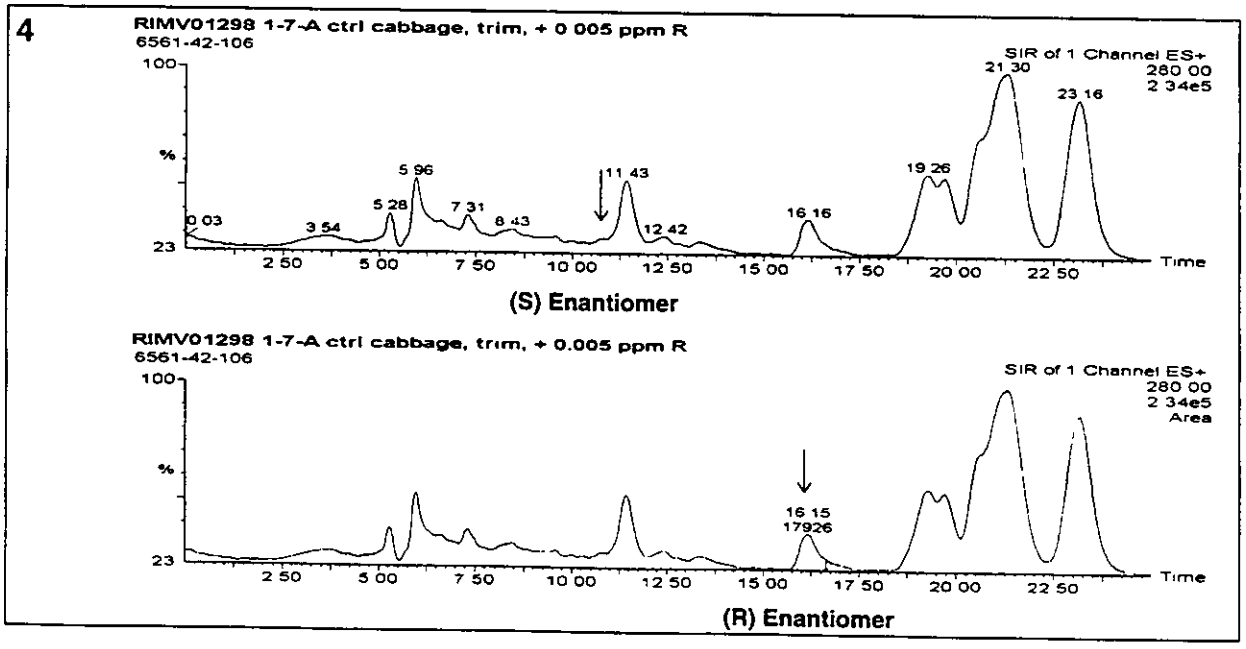


Sample		mg	(S) Enantiomer			(R) Enantiomer		
Code	Desc	injected	ng	ppm uncorr	ppm	ng	ppm uncorr	ppm
1-7-A	control	43 083	0 040	0 001	<0 005	0 030	0 001	<0 005

FIGURE 13: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TRIMMED CABBAGE HEADS (Continued)

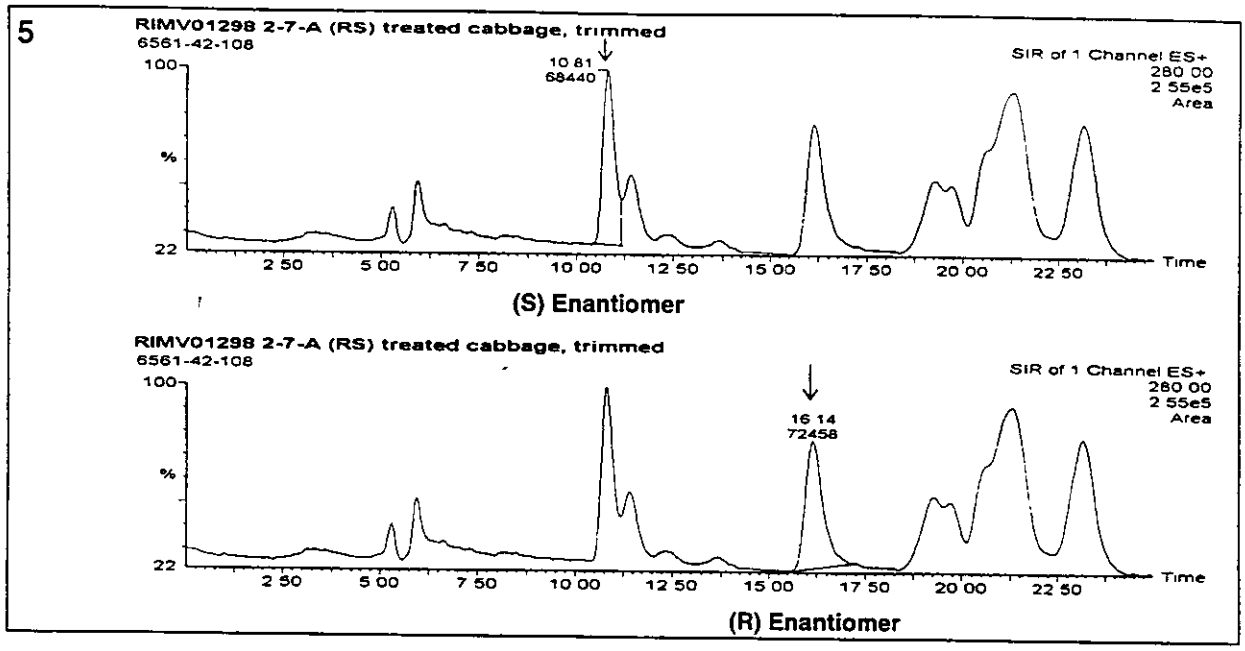


Sample Code	Desc	Amt. (S), (R) Added	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm uncorr	(S) Enantiomer % Rec.	(R) Enantiomer ng	(R) Enantiomer ppm uncorr	(R) Enantiomer % Rec.
1-7-A	recovery	0 005 ppm	42.965	0 240	0 006	93%	0.280	0 007	116%

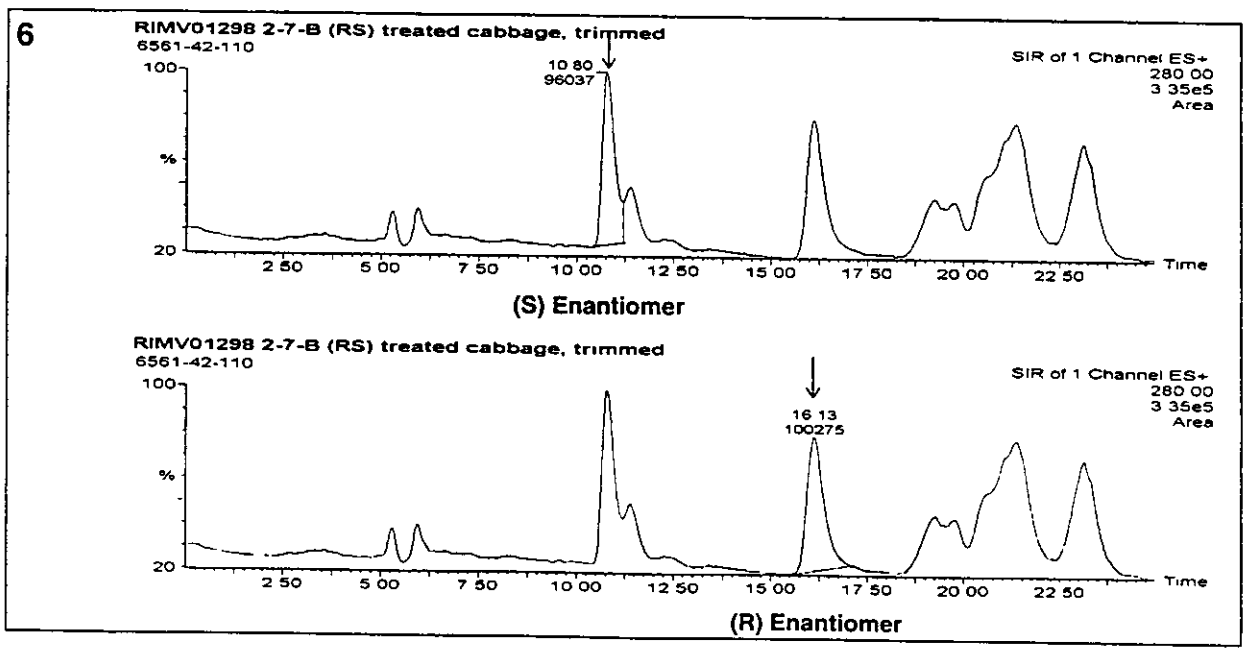


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm uncorr	(S) Enantiomer % Rec	(R) Enantiomer ng	(R) Enantiomer ppm uncorr	(R) Enantiomer % Rec
1-7-A	recovery	0 005 ppm	42 965	0.000	0 000	-	0 250	0 006	102%

FIGURE 13: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TRIMMED CABBAGE HEADS (Continued)

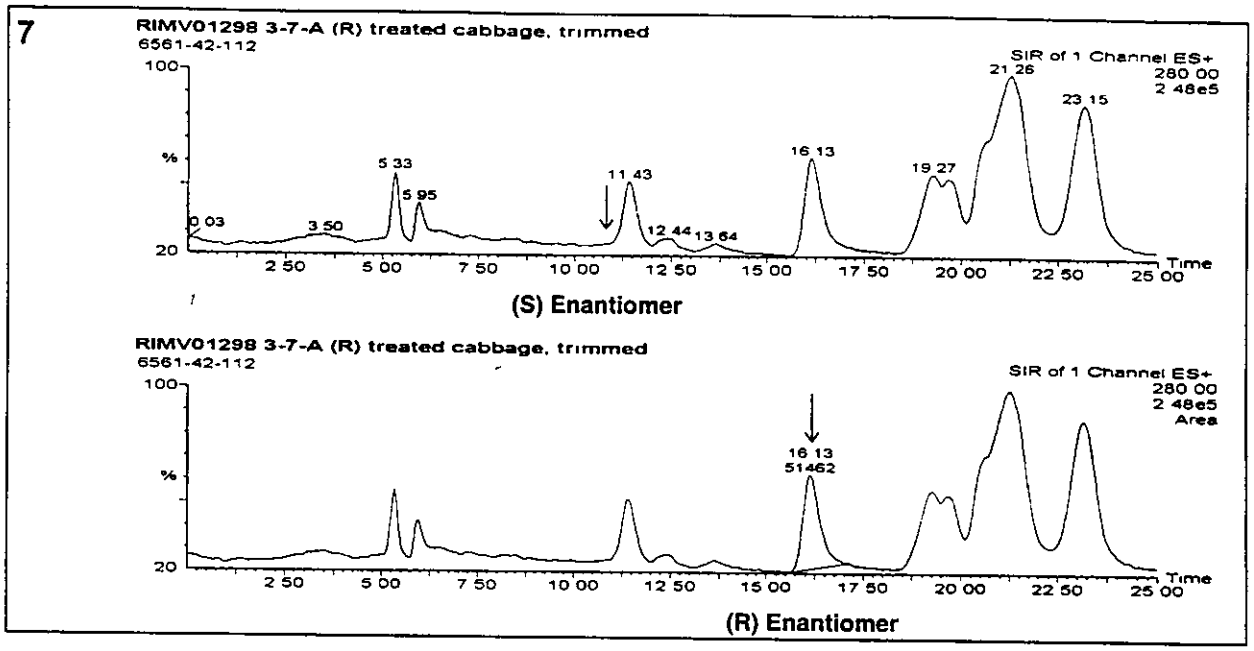


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-7-A	(RS)-treated	7	43 043	1 050	0.026	1.020	0 024	0 050

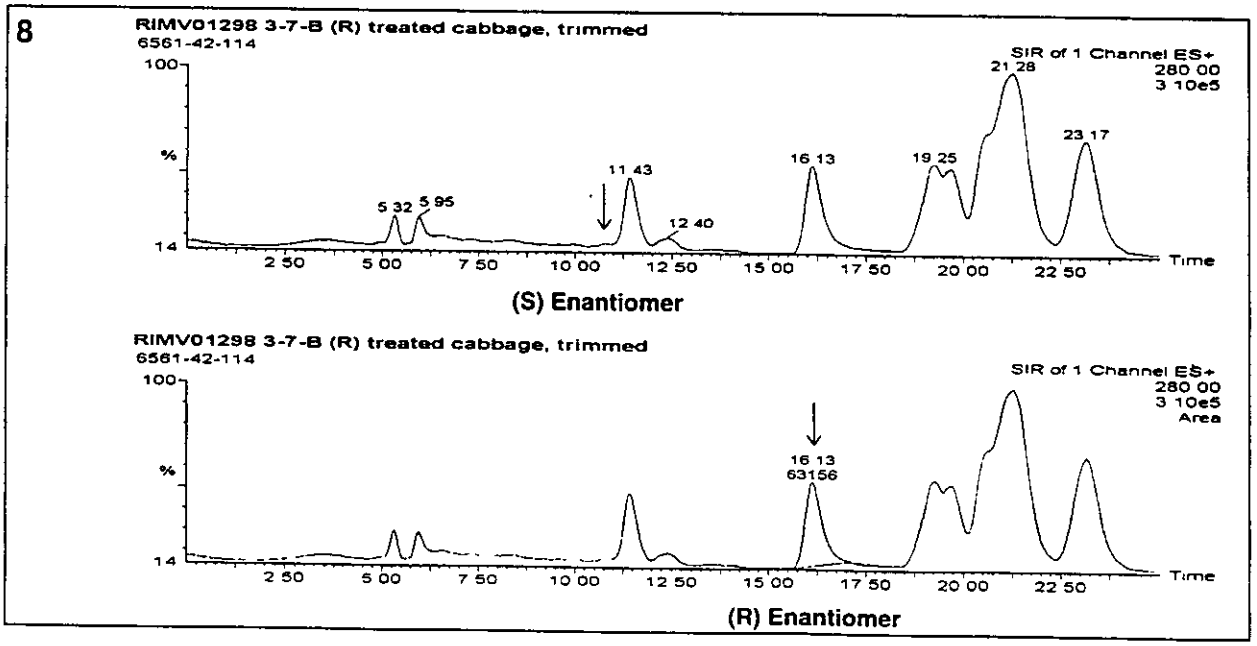


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-7-B	(RS)-treated	7	43 043	1 470	0 037	1 420	0 033	0 070

FIGURE 13: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN TRIMMED CABBAGE HEADS (Continued)

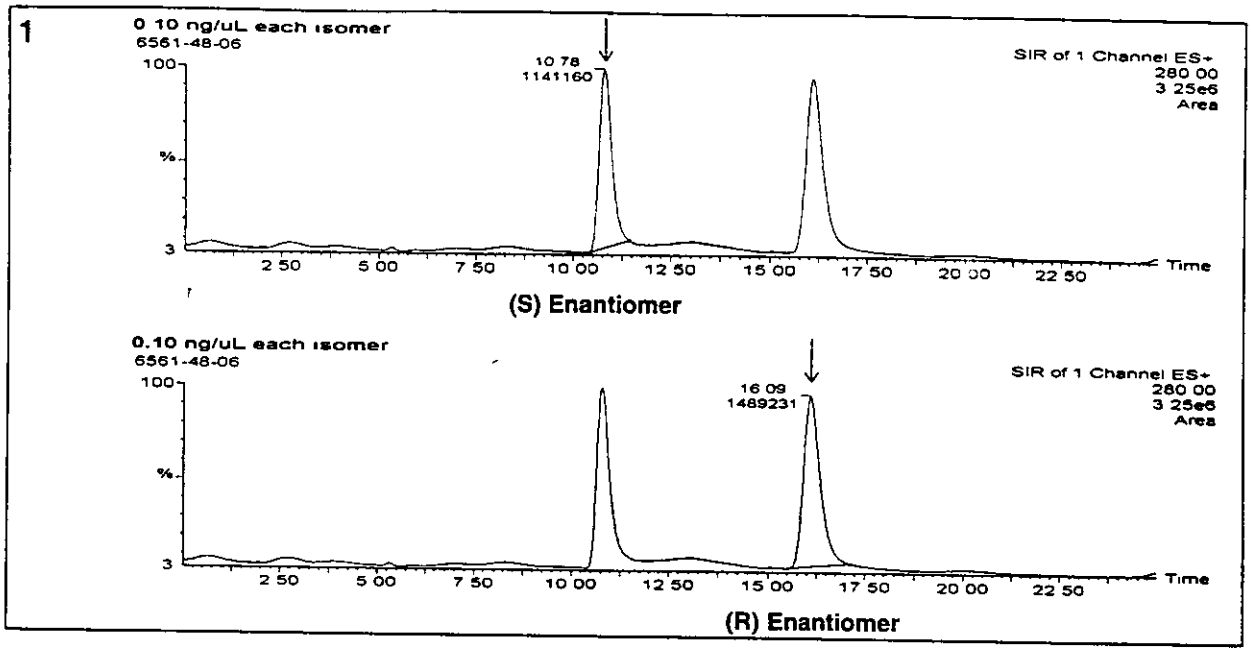


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-7-A	(R)-treated	7	43 043	0.000	<0 005	0.730	0 017	0 017

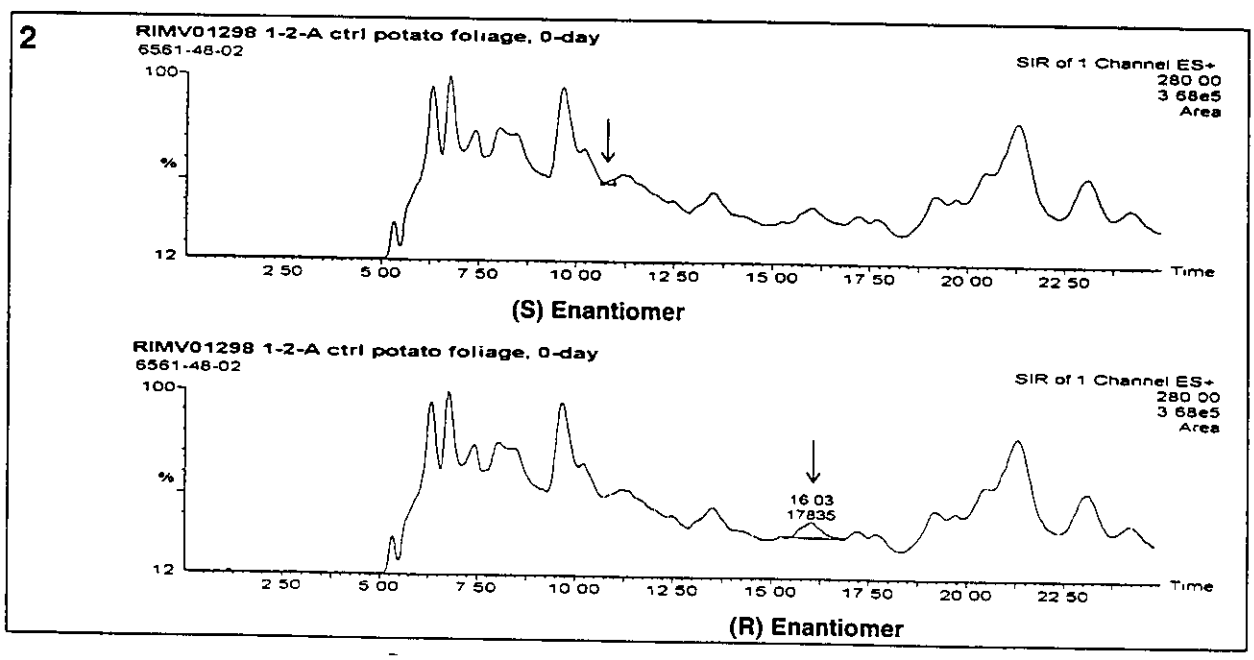


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-7-B	(R)-treated	7	42 926	0.000	<0.005	0.890	0 021	0 021

FIGURE 14: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN POTATO FOLIAGE

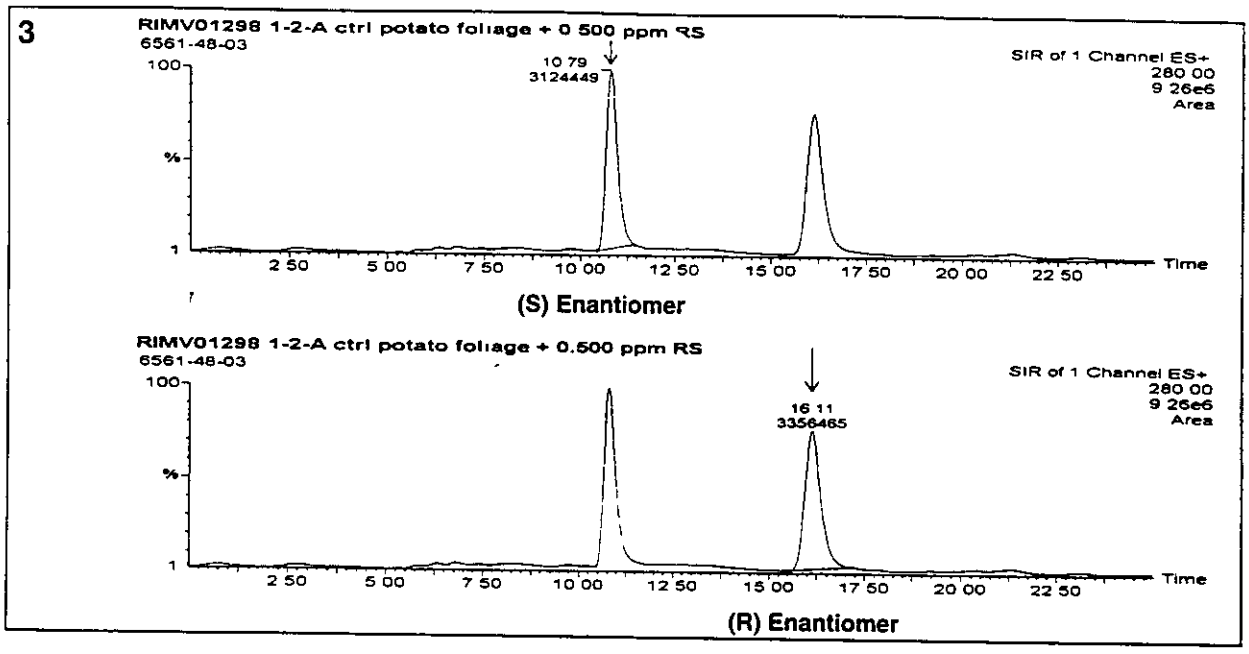


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 1000	7 500	142468	0	0 9982	152665	0	0 9982

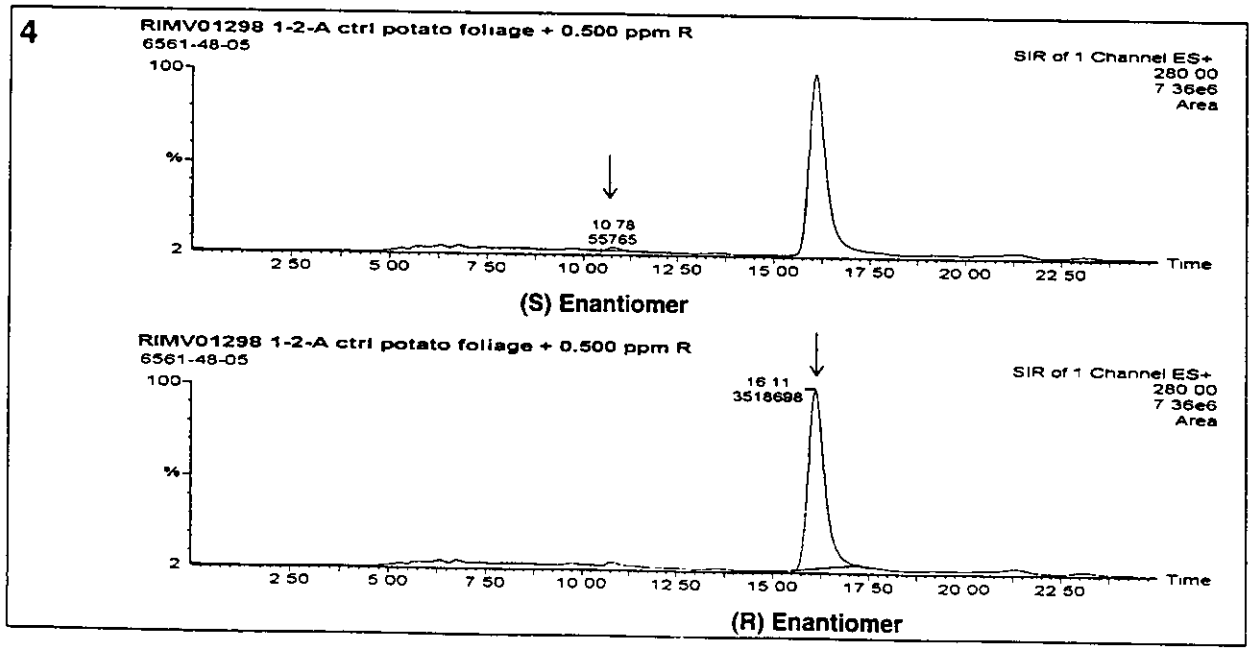


Sample Code	Desc	mg injected	(S) Enantiomer ng	ppm uncorr	ppm	(R) Enantiomer ng	ppm uncorr	ppm
1-2-A	control	46 273	0 010	0 000	<0.005	0 120	0 003	<0 005

FIGURE 14: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN POTATO FOLIAGE (Continued)

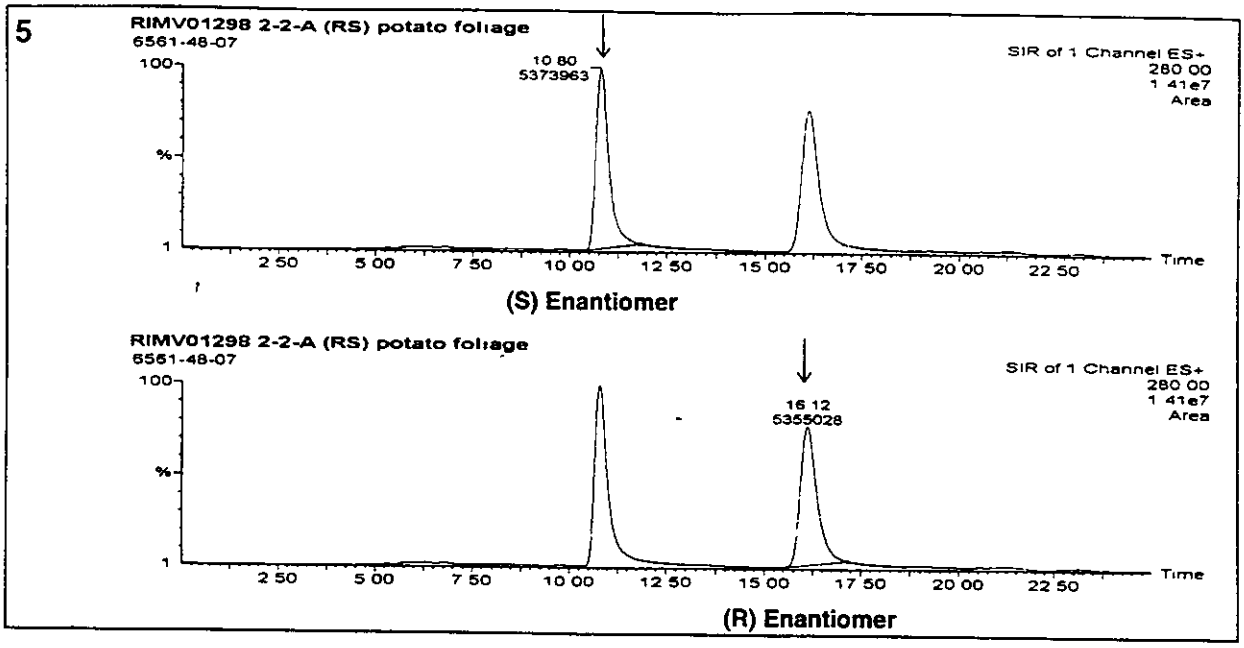


Sample Code	Desc.	Amt. (S), (R) Added	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm uncorr	(S) Enantiomer % Rec.	(R) Enantiomer ng	(R) Enantiomer ppm uncorr	(R) Enantiomer % Rec.
1-2-A	recovery	0.50 ppm	46.273	21.930	0 474	95%	21 990	0 475	95%

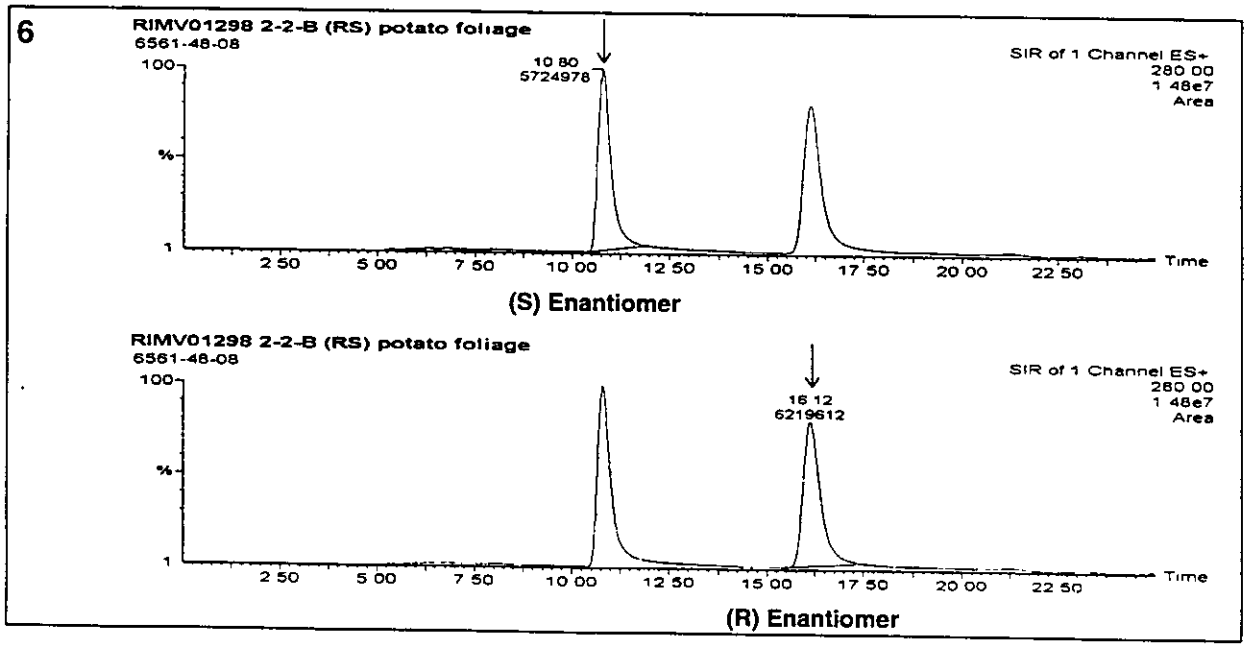


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm uncorr	(S) Enantiomer % Rec	(R) Enantiomer ng	(R) Enantiomer ppm uncorr	(R) Enantiomer % Rec
1-2-A	recovery	0 50 ppm	46 228	0 390	0 008	-	23 050	0 499	99%

FIGURE 14: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN POTATO FOLIAGE (Continued)

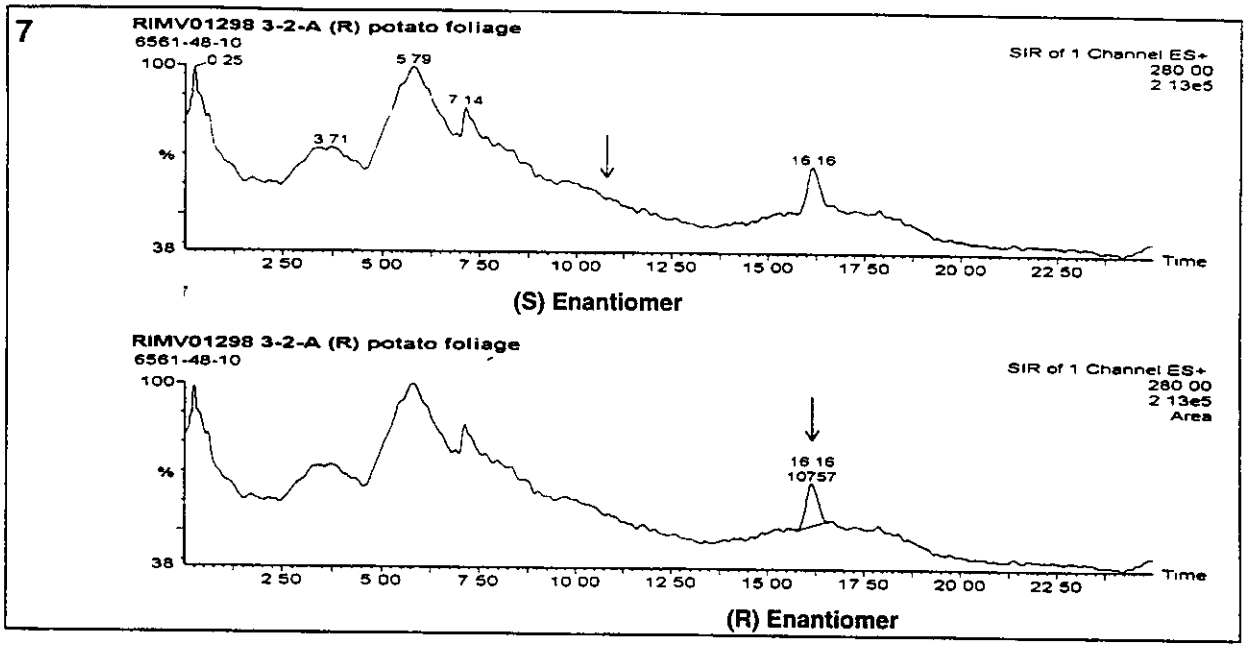


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-2-A	(RS)-treated	0	46.228	37 720	0 861	35 080	0 783	1 644

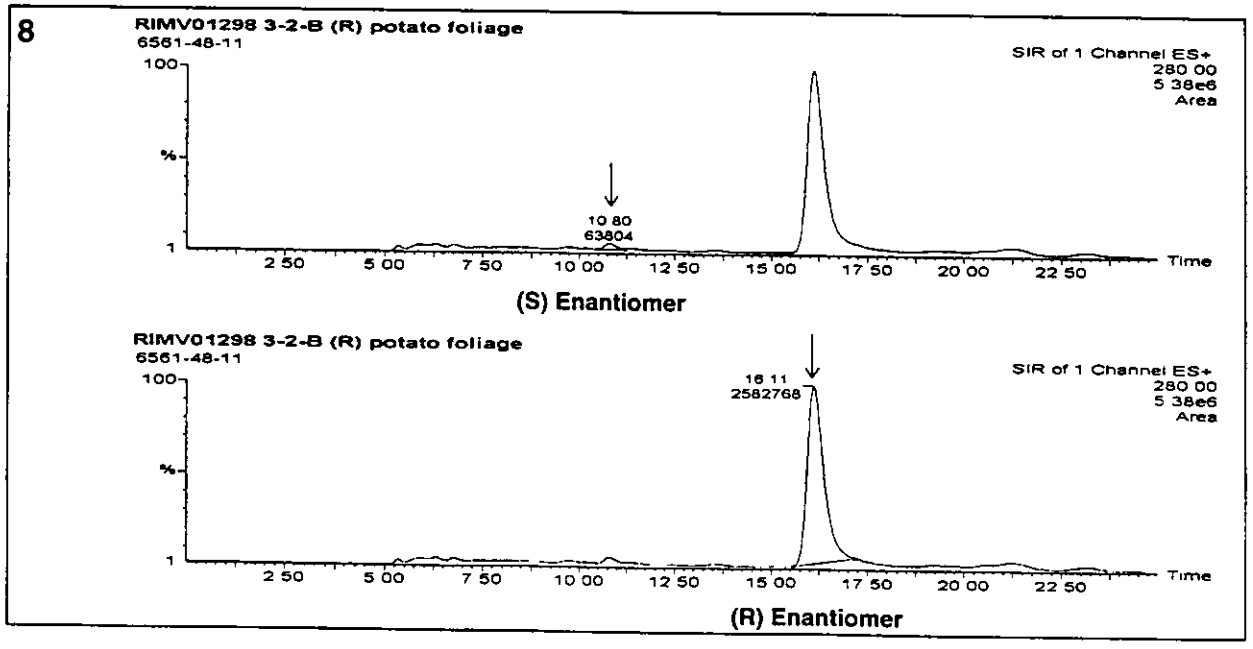


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
2-2-B	(RS)-treated	0	46 137	40.180	0 919	40 740	0 912	1 831

FIGURE 14: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN POTATO FOLIAGE (Continued)

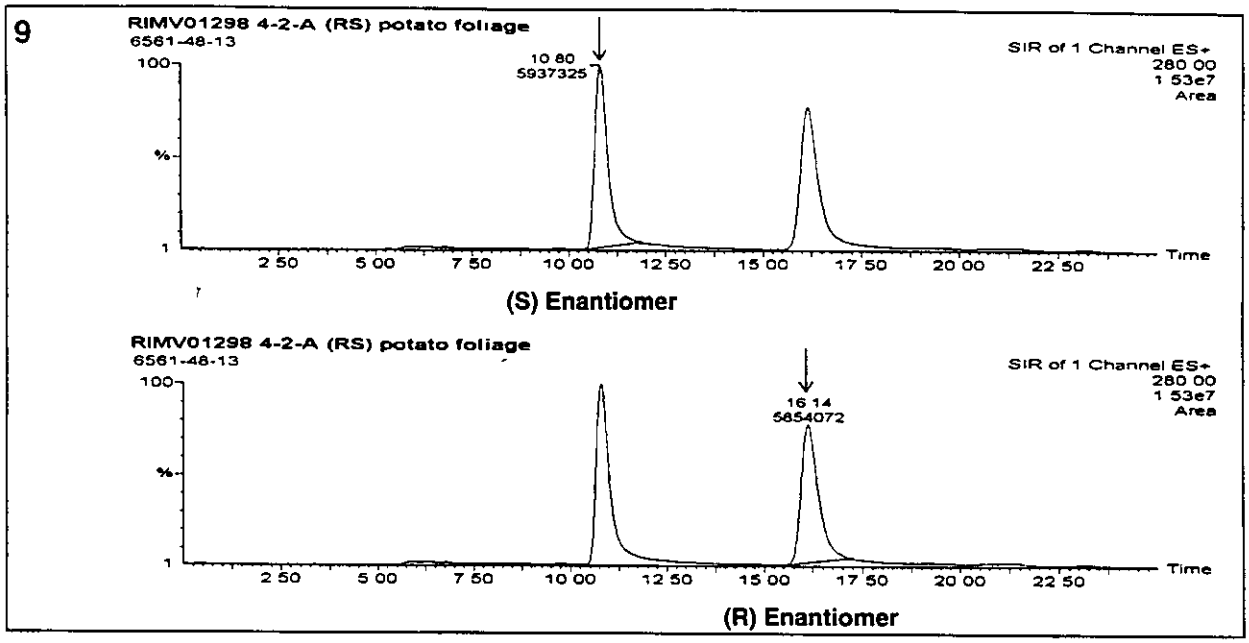


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-2-A	(R)-treated	0	46 228	0 000	<0 005	0 070	<0.005	0 002

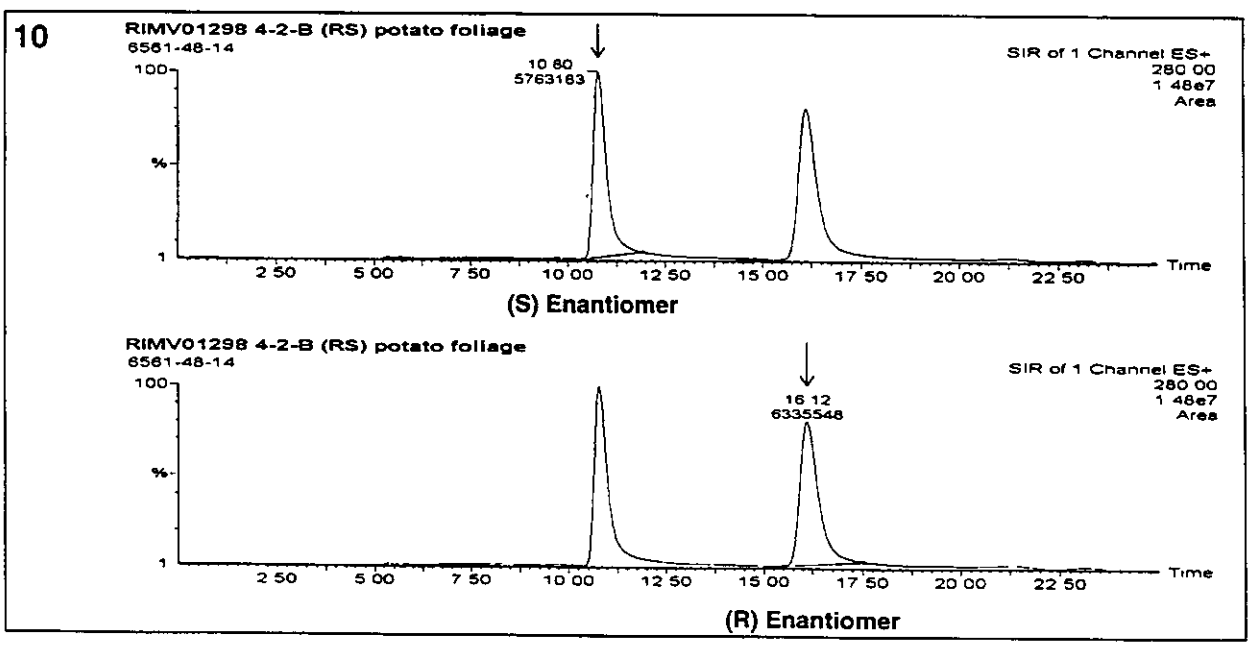


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
3-2-B	(R)-treated	0	46 228	0 450	0 010	16 920	0 378	0 388

FIGURE 14: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN POTATO FOLIAGE (Continued)

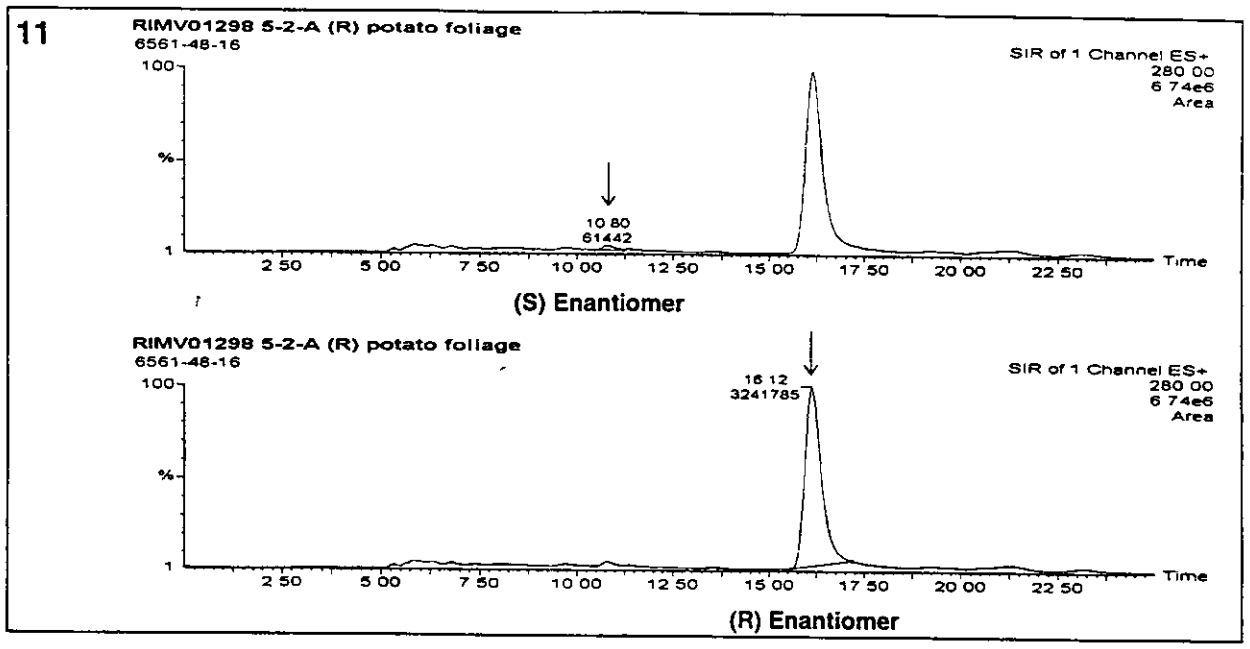


Sample Code	Desc	PHI (days)	injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
4-2-A	(RS)-treated	0	46.410	41 670	0.948	38 350	0 853	1.801

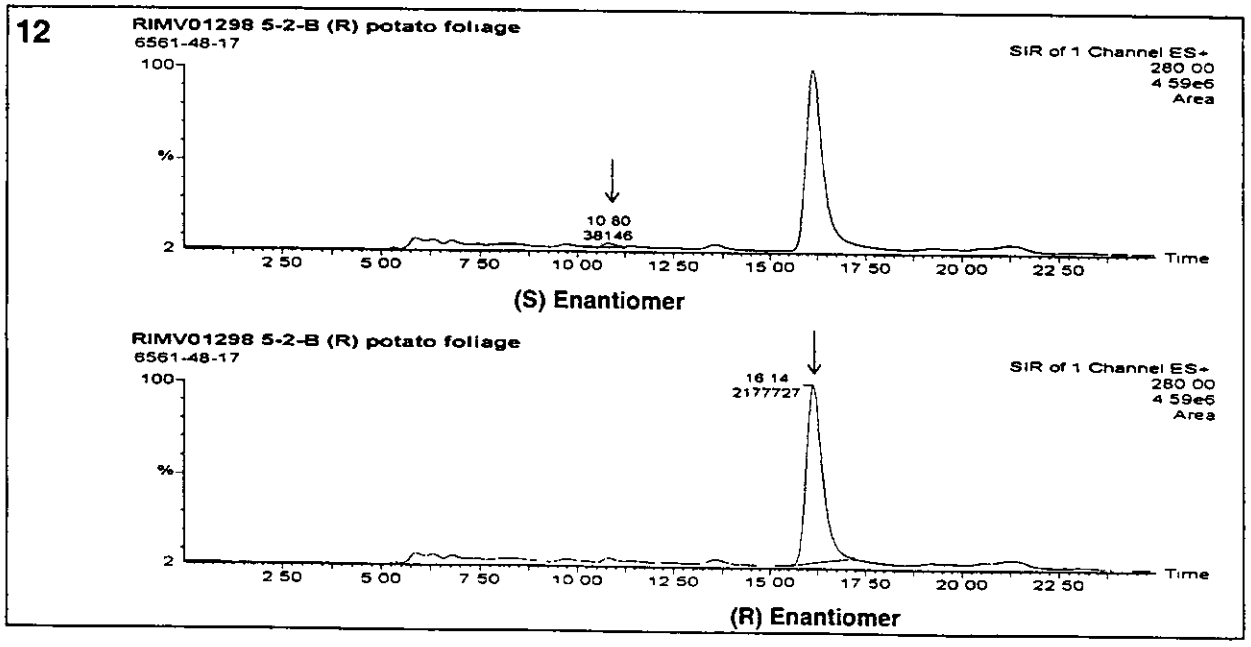


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
4-2-B	(RS)-treated	0	46 410	40 450	0 920	41 500	0 923	1 843

FIGURE 14: CHROMATOGRAMS.
(S) AND (R) ENANTIOMERS IN POTATO FOLIAGE (Continued)

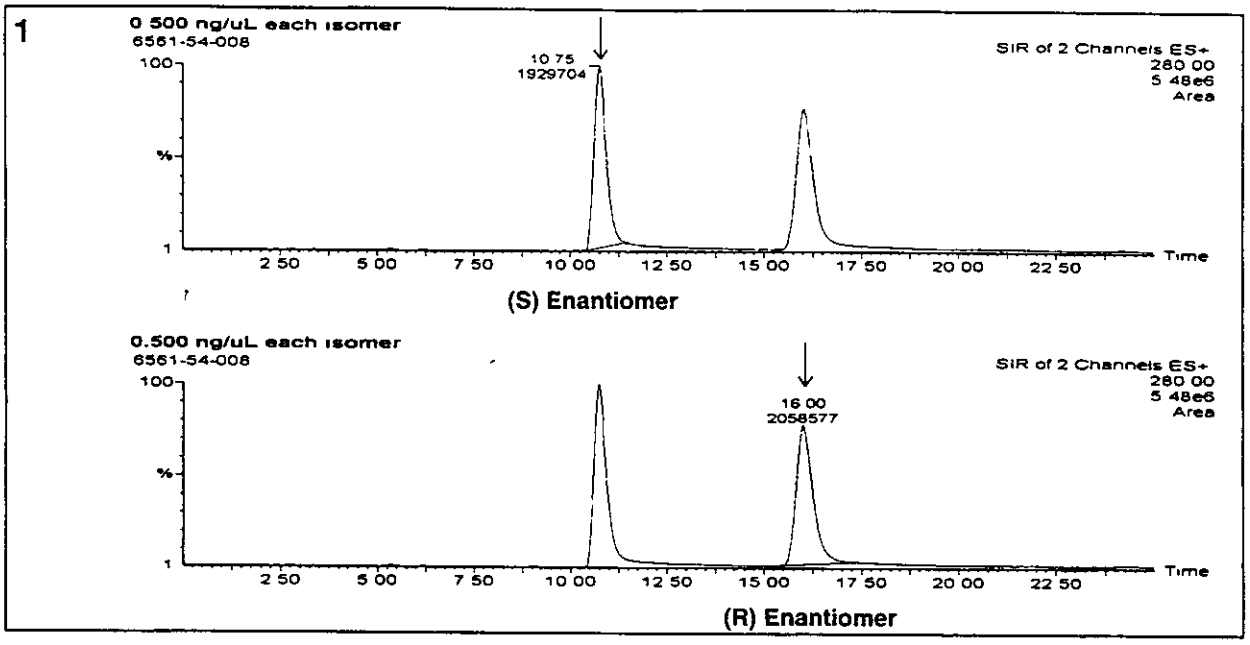


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
5-2-A	(R)-treated	0	46 137	0 430	0 010	21.230	0.475	0 485

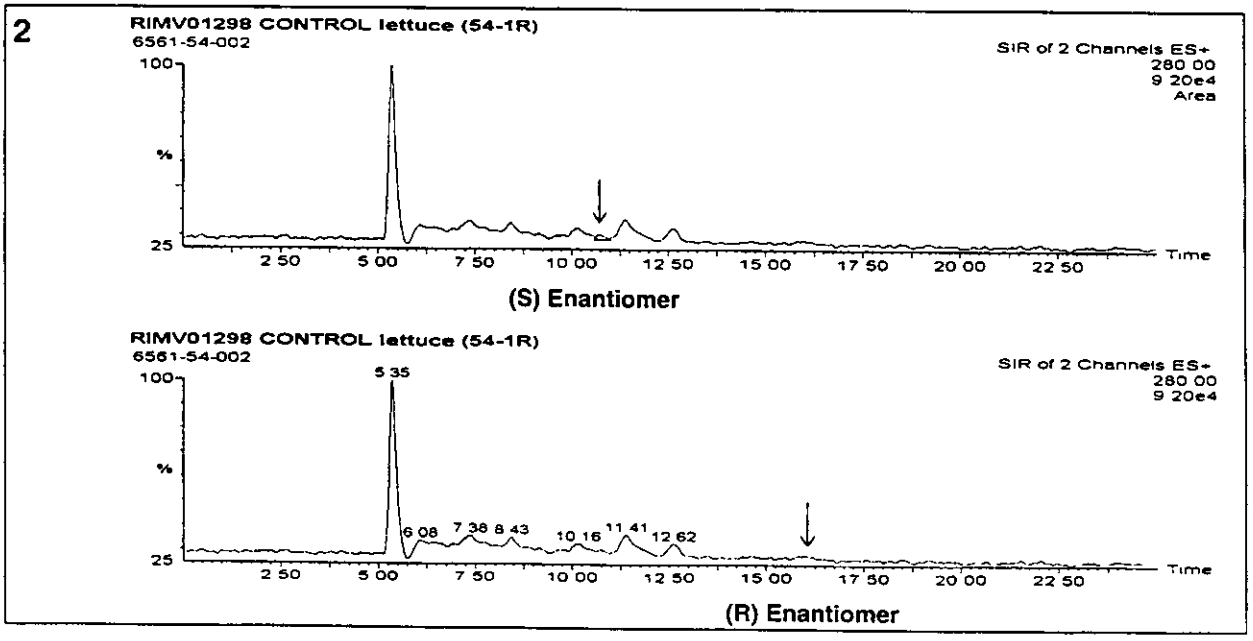


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
5-2-B	(R)-treated	0	46 182	0 270	0.006	14 260	0 319	0 325

FIGURE 15: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN ^{14}C-TREATED LETTUCE HEADS

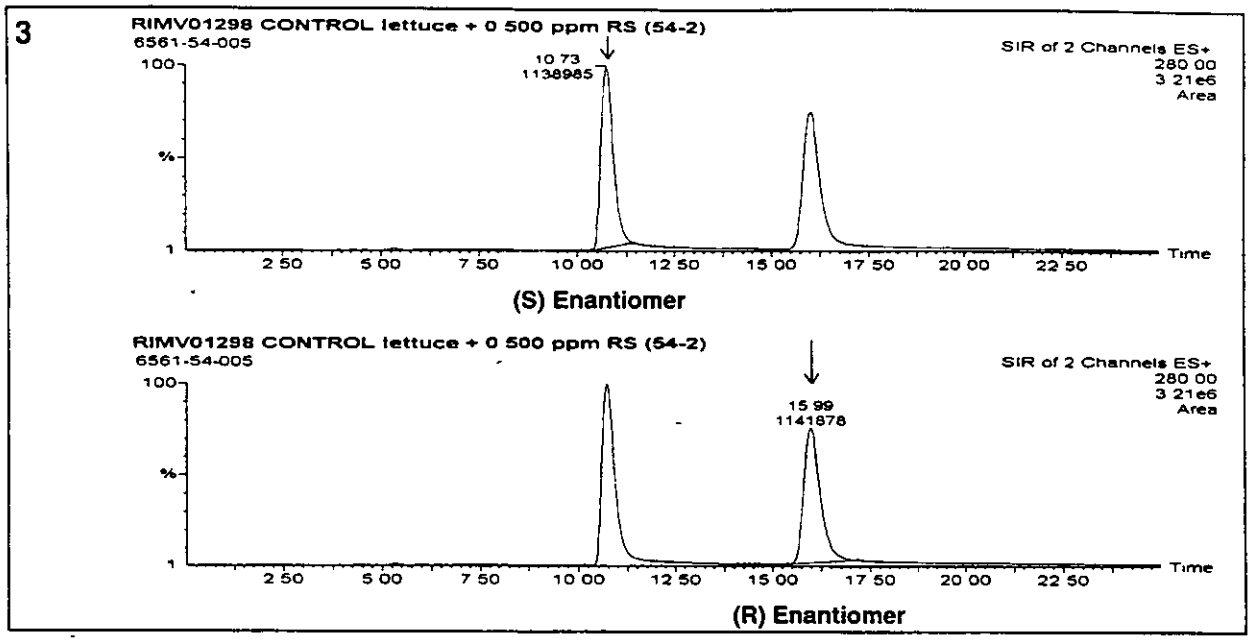


Standard Concentration		Output from Linear Regression (y=mx+b)					
		(S) Enantiomer			(R) Enantiomer		
ng/uL	ng injected	Slope (m)	Intercept (b)	Correlation	Slope (m)	Intercept (b)	Correlation
0 5000	37 500	48399 5	0	0 9960	51574 8	0	0 9965

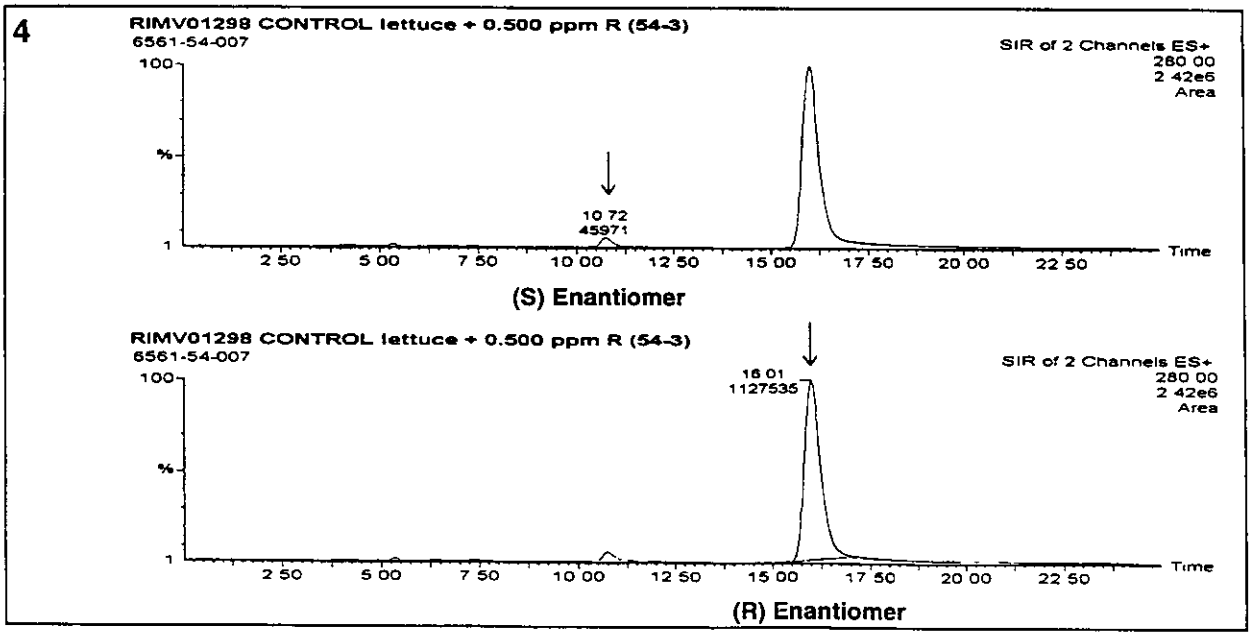


Sample		mg	(S) Enantiomer			(R) Enantiomer		
Code	Desc	injected	ng	ppm uncorr	ppm	ng	ppm uncorr	ppm
CONTROL	control	42 769	0 010	0 000	<0 005	0 000	0 000	<0 005

FIGURE 15: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN ^{14}C-TREATED LETTUCE HEADS (Continued)

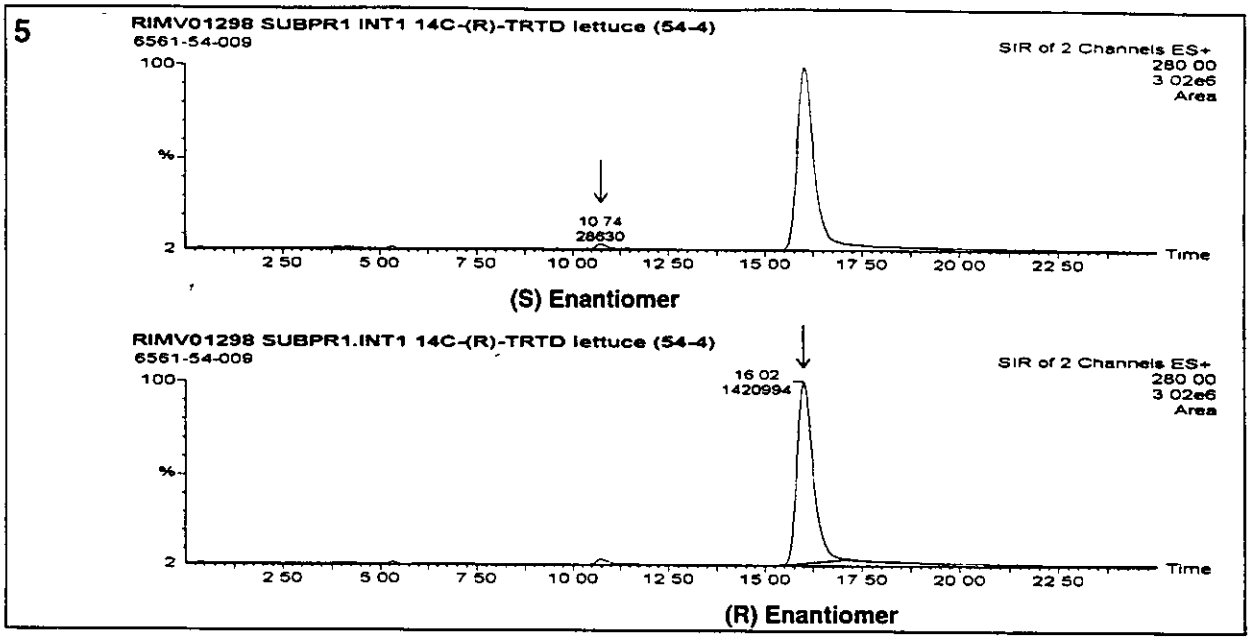


Sample Code	Desc	Amt. (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr.	% Rec.	(R) Enantiomer ng	ppm uncorr	% Rec.
CONTROL	recovery	0.50 ppm	42 925	23.530	0.548	110%	22 140	0 516	103%

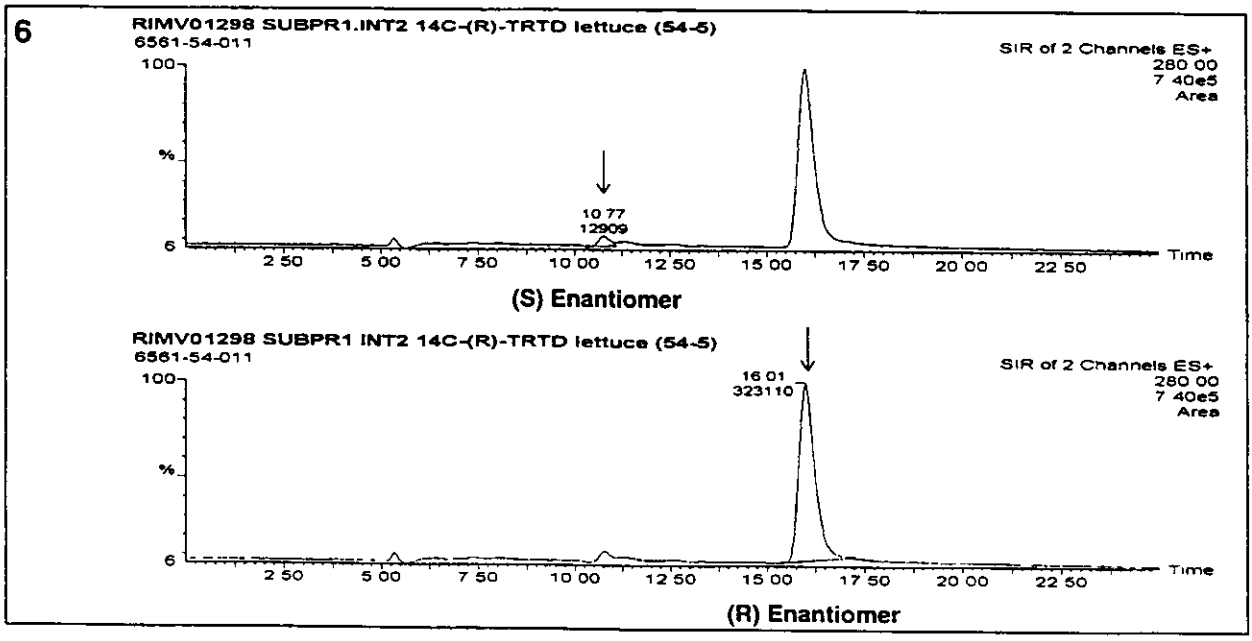


Sample Code	Desc	Amt (S), (R) Added	mg injected	(S) Enantiomer ng	ppm uncorr	% Rec	(R) Enantiomer ng	ppm uncorr	% Rec
CONTROL	recovery	0 50 ppm	42 847	0.950	0.022	-	21 860	0 510	102%

FIGURE 15: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN ^{14}C-TREATED LETTUCE HEADS (Continued)

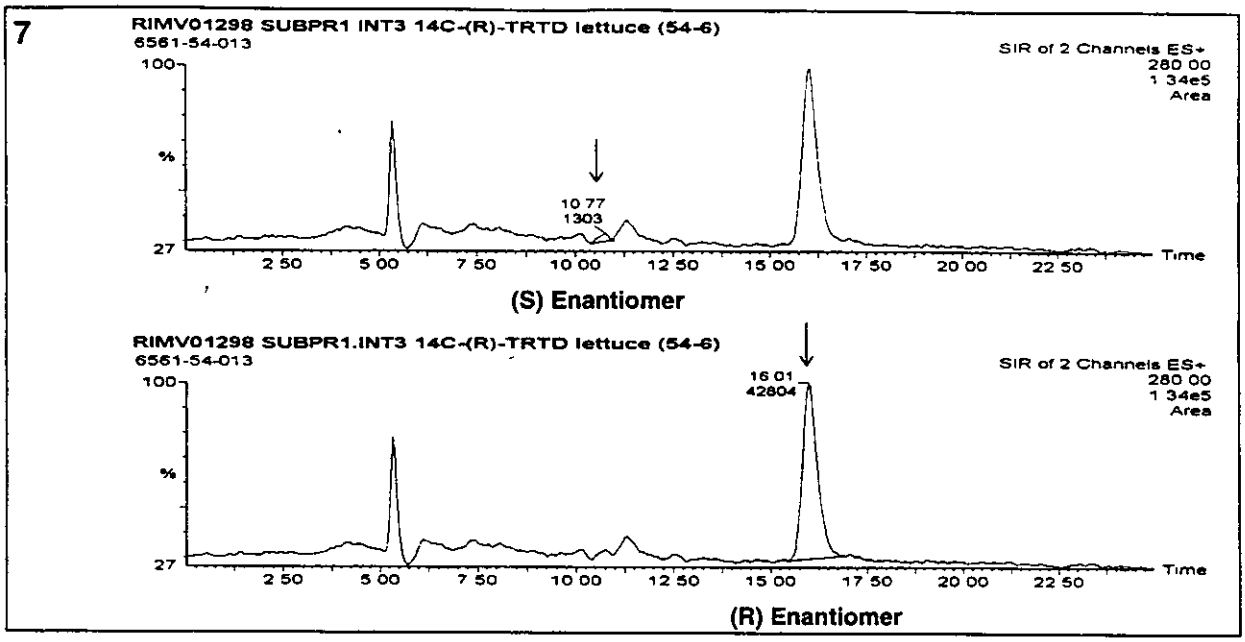


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
SUBPR1INT1	(R)-treated	0	4 312	0.590	0 137	27.550	6.389	6 526

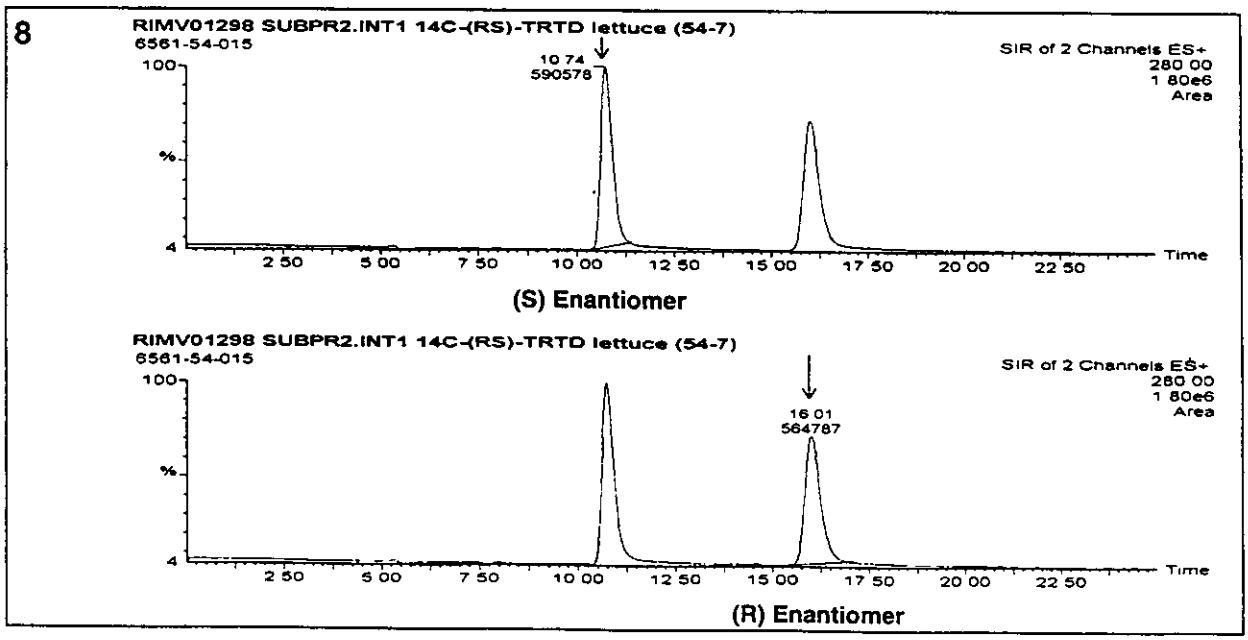


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
SUBPR1INT2	(R)-treated	14	43.393	0 270	0.006	6 260	0 144	0 150

FIGURE 15: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN ^{14}C-TREATED LETTUCE HEADS (Continued)

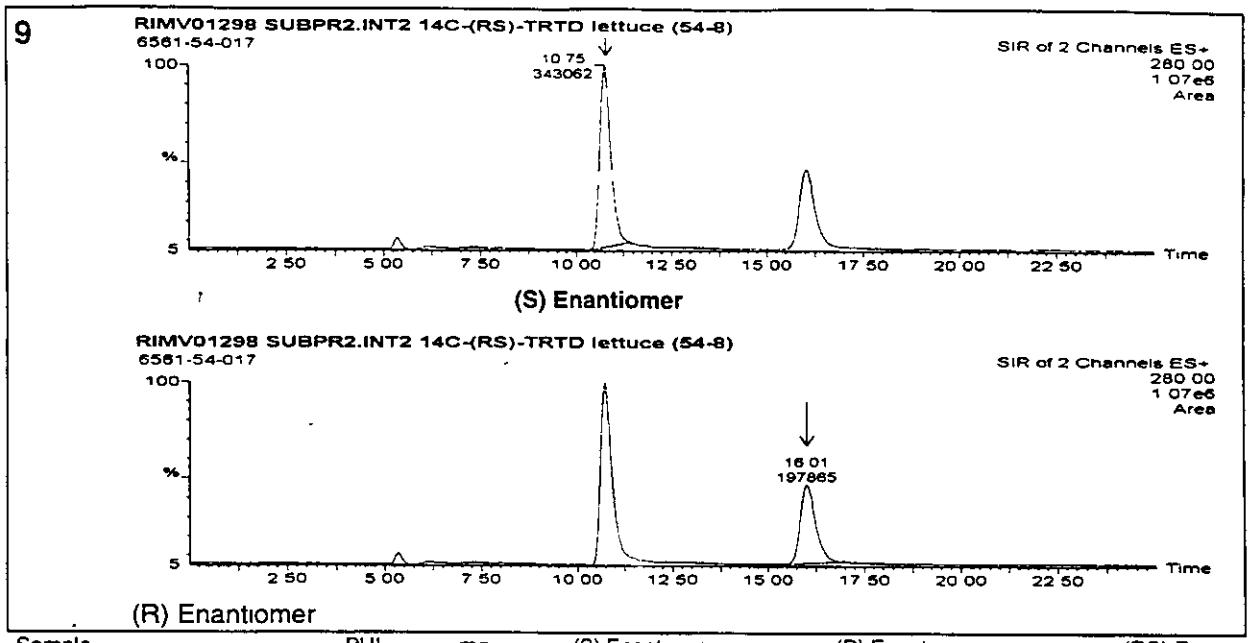


Sample Code	Desc.	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
SUBPR1INT3	(R)-treated	21	42 769	0.030	<0 005	0 830	0 019	0 020

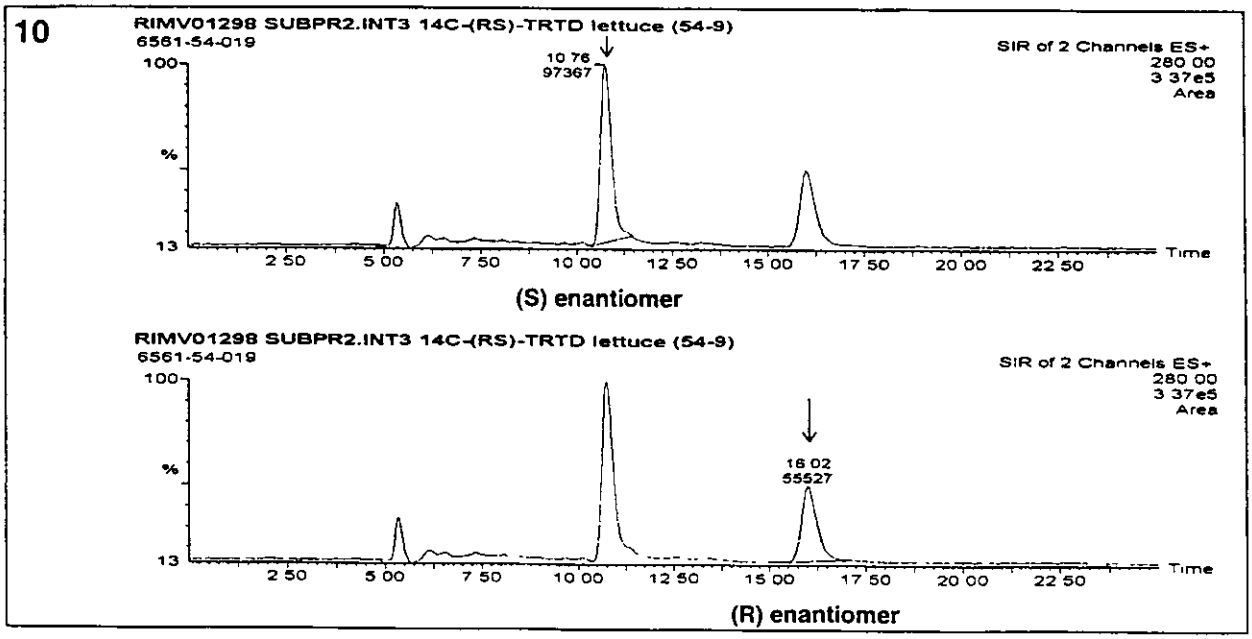


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
SUBPR2INT1	(RS)-treated	0	4 253	12.200	2 868	10 950	2 574	5 442

FIGURE 15: CHROMATOGRAMS:
(S) AND (R) ENANTIOMERS IN ^{14}C-TREATED LETTUCE HEADS (Continued)

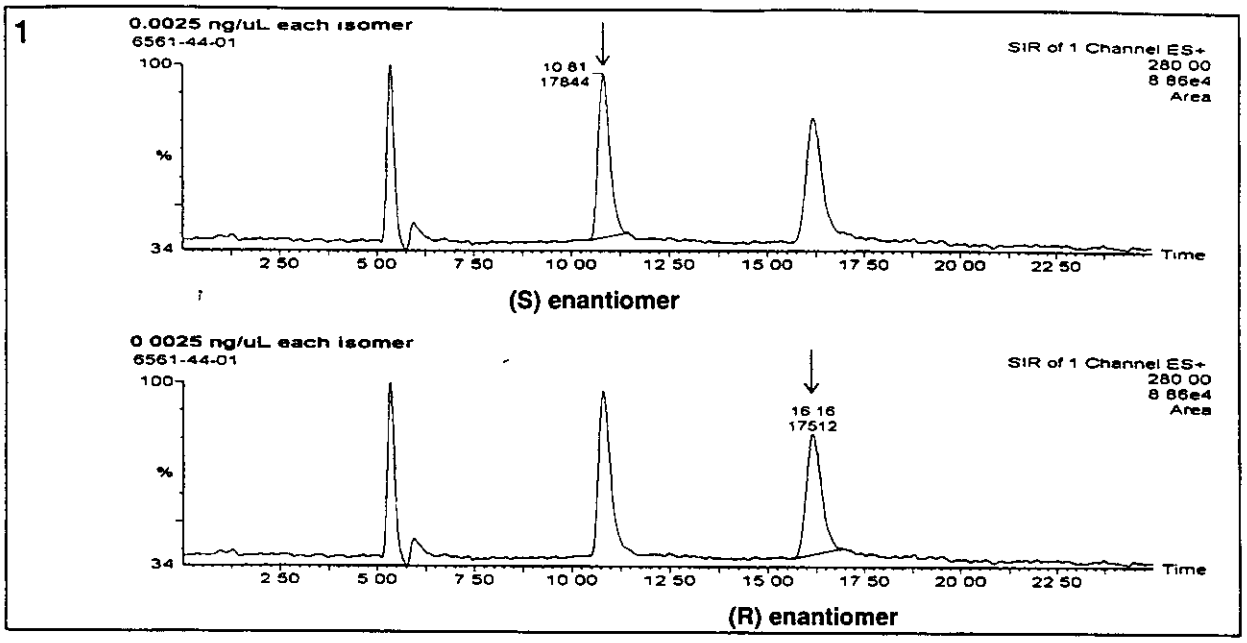


Sample Code	Desc	PHI (days)	mg injected	(S) Enantiomer ng	(S) Enantiomer ppm corr	(R) Enantiomer ng	(R) Enantiomer ppm corr	(RS) Racemate ppm
SUBPR2INT2	(RS)-treated	14	42.847	7 090	0.165	3 840	0.090	0.255

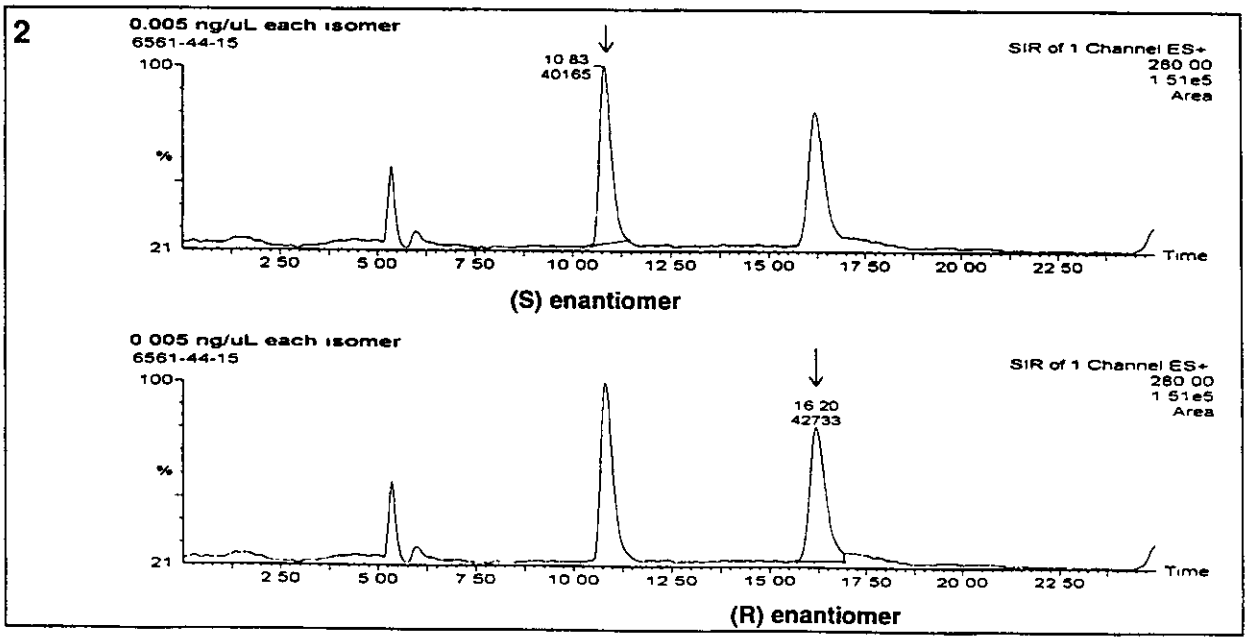


Sample Code	Desc	PHI (days)	mg injected	(S) enantiomer ng	(S) enantiomer ppm corr	(R) enantiomer ng	(R) enantiomer ppm corr	(RS) racemate ppm
SUBPR2INT3	(RS)-treated	21	42 535	2 010	0 047	1 080	0 025	0 072

FIGURE 16: CHROMATOGRAMS:
CALIBRATION STANDARDS FROM FIGURE 11, CABBAGE HEADS

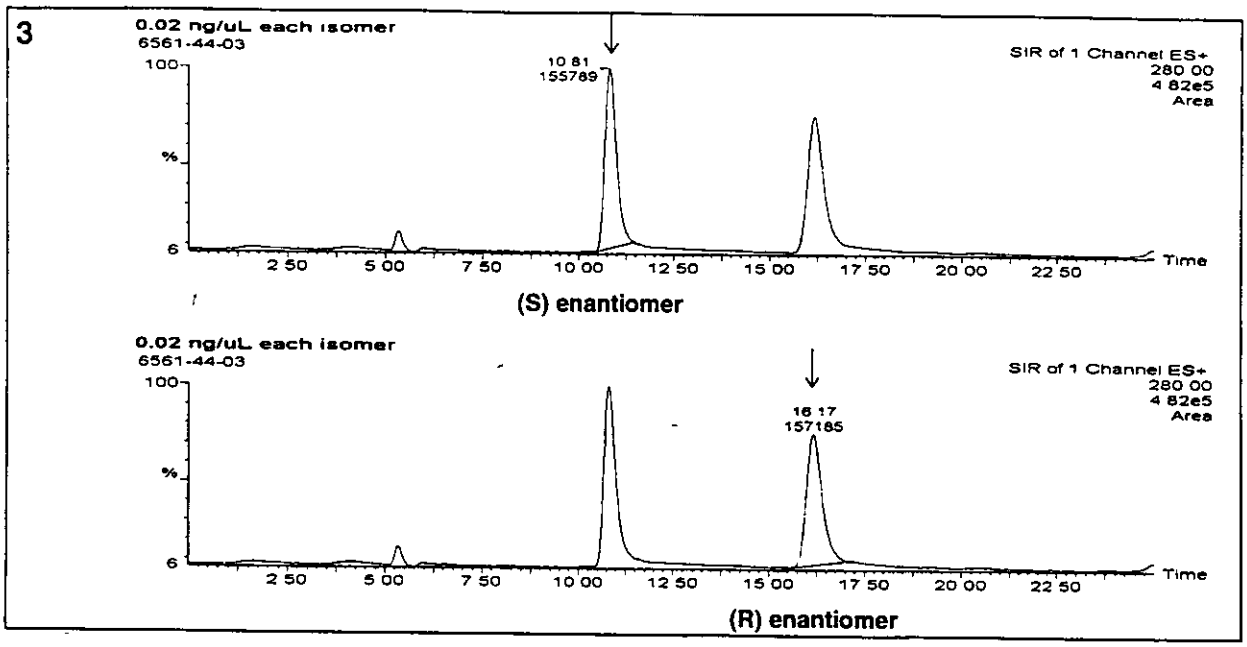


Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer	(R) enantiomer
0.1875	17844	17512

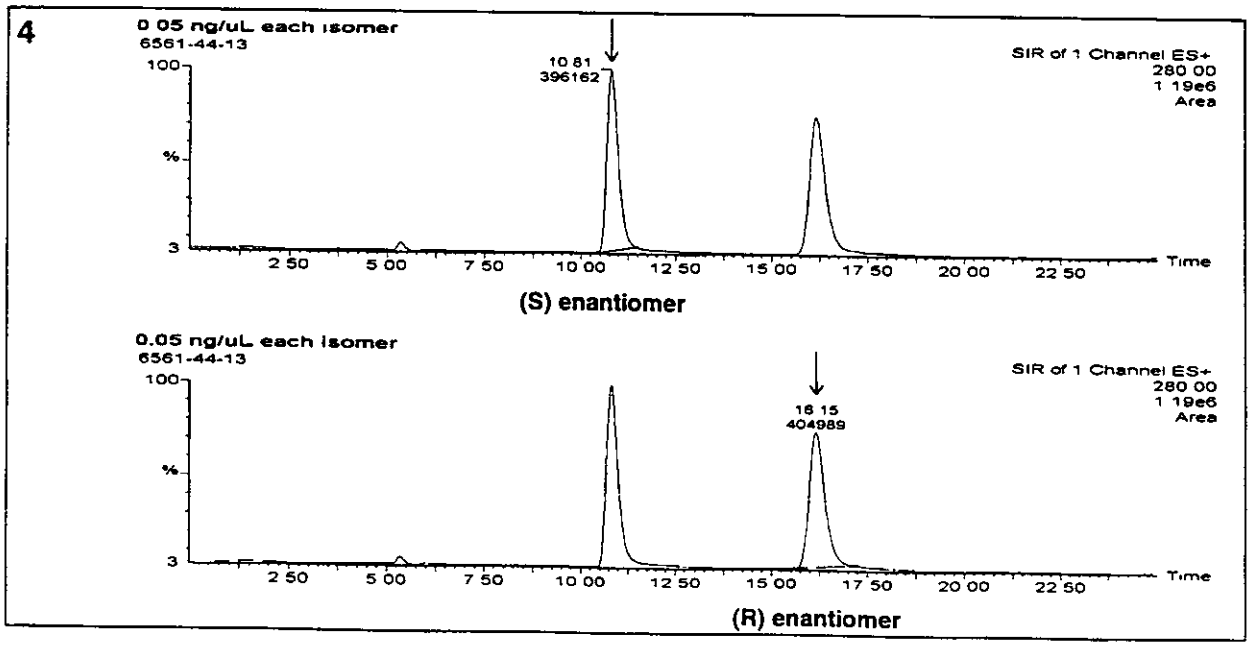


Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer	(R) enantiomer
0 3750	40165	42733

FIGURE 16: CHROMATOGRAMS:
CALIBRATION STANDARDS FROM FIGURE 11, CABBAGE HEADS (Continued)

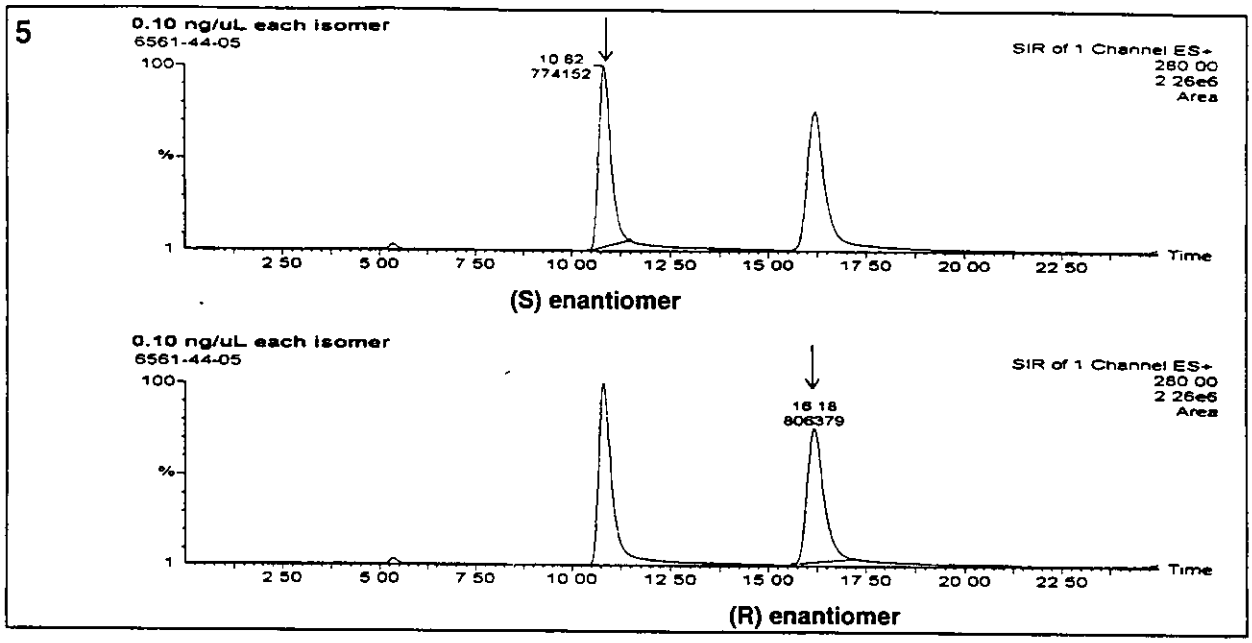


Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer	(R) enantiomer
1 500	155789	157185

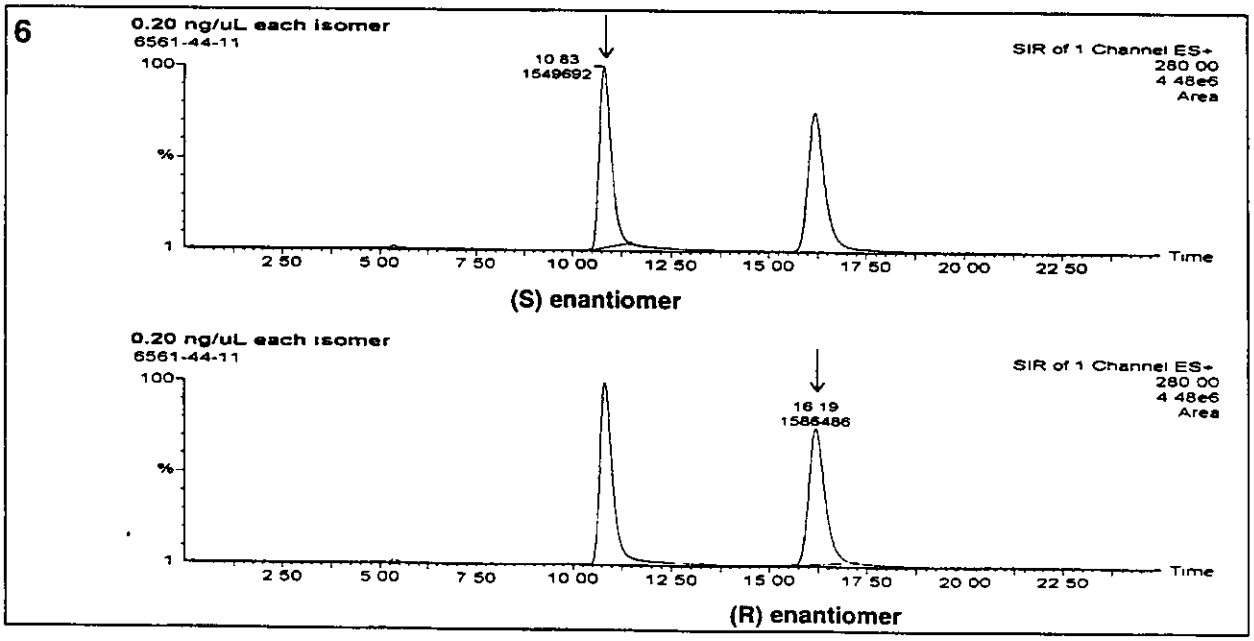


Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer	(R) enantiomer
3 750	396162	404989

FIGURE 16: CHROMATOGRAMS:
CALIBRATION STANDARDS FROM FIGURE 11, CABBAGE HEADS (Continued)

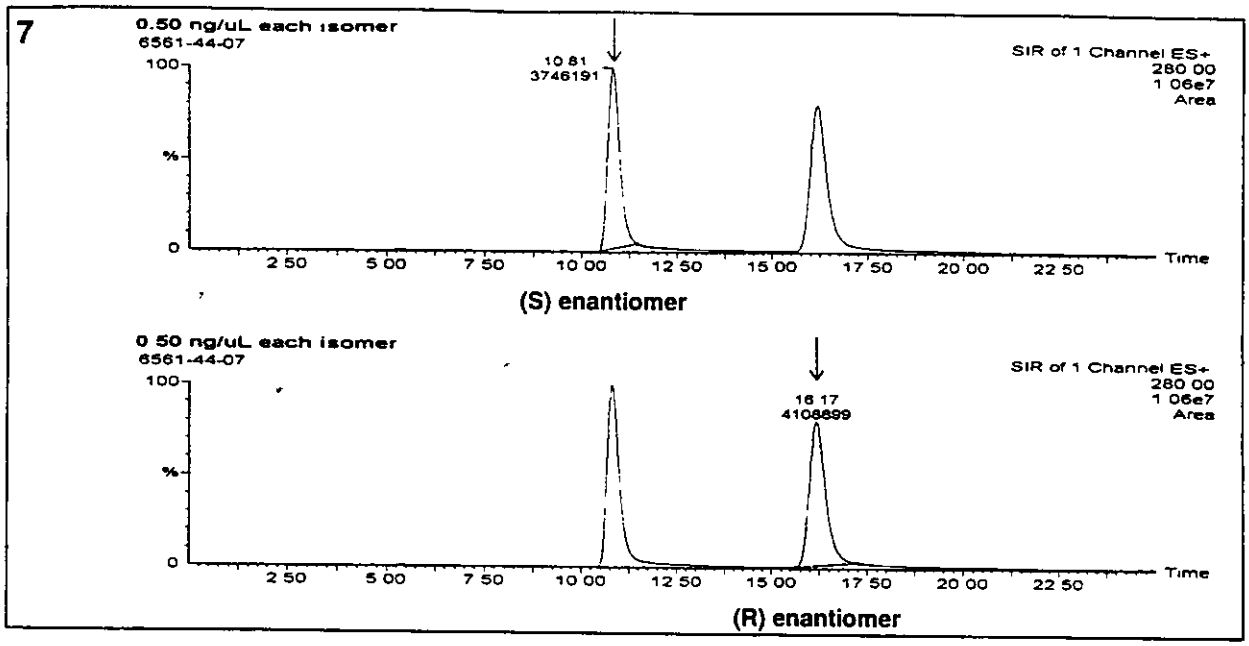


Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer	(R) enantiomer
7 500	774152	806379

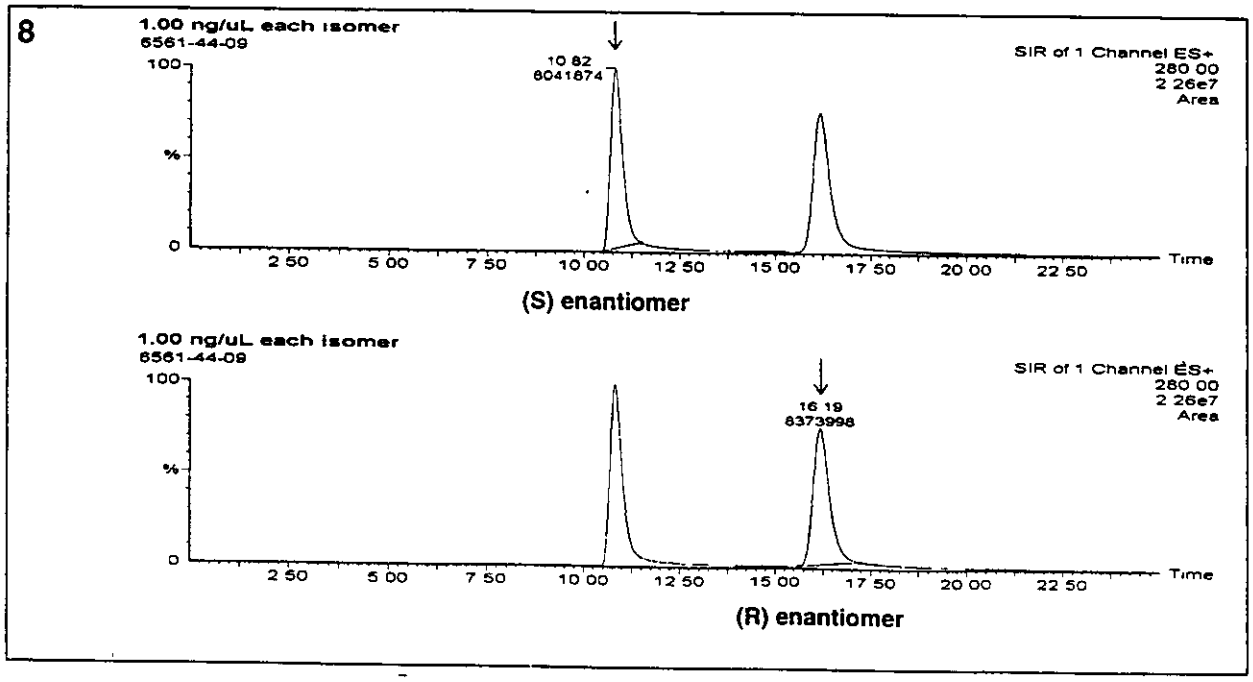


Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer	(R) enantiomer
15 00	1549692	1586486

FIGURE 16: CHROMATOGRAMS:
CALIBRATION STANDARDS FROM FIGURE 11, CABBAGE HEADS (Continued)



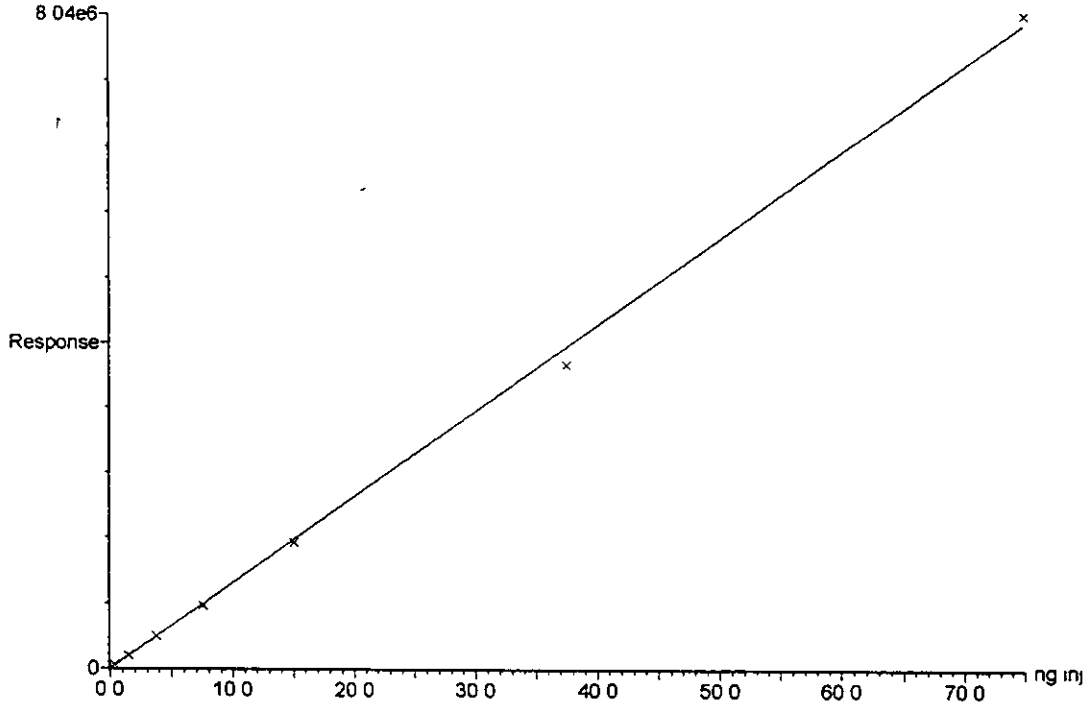
Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer	(R) enantiomer
37.50	3746191	4108899



Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer	(R) enantiomer
75.00	8041874	8373998

FIGURE 17: EXAMPLE CALIBRATION CURVES: (S) ENANTIOMER
CALIBRATION STANDARDS FROM FIGURE 16, CABBAGE HEADS

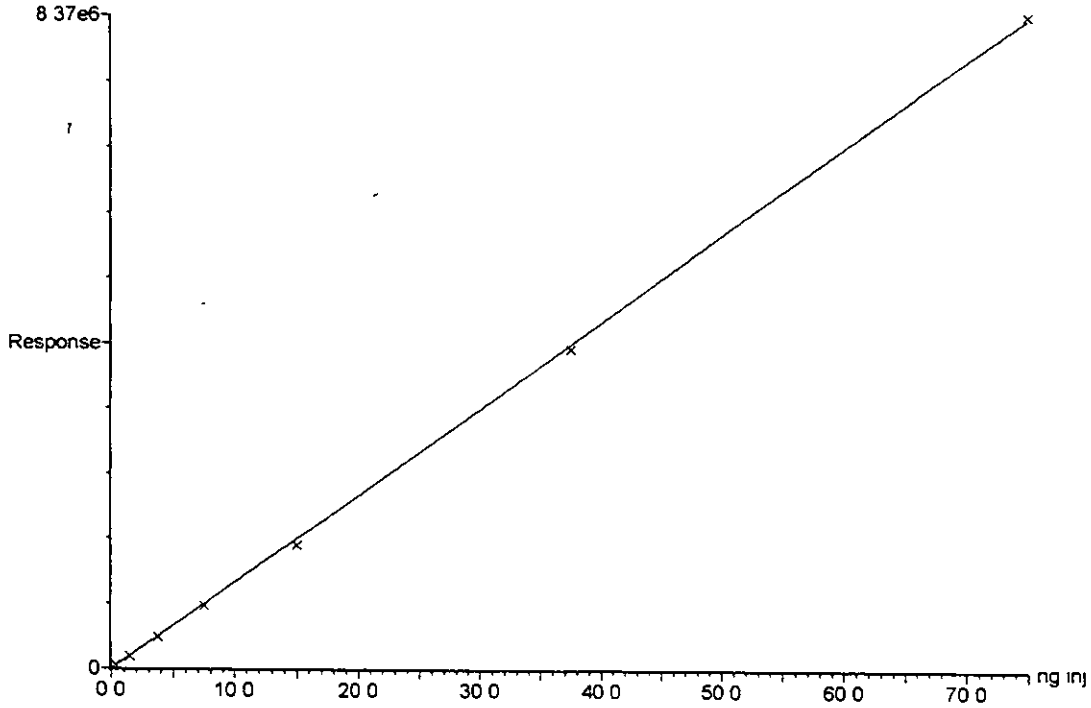
Compound 1 name (s)CGA-48988
Coefficient of Determination 0 998868
Calibration curve 105664 * x + 0
Response type External Std, Area
Curve type Linear, Origin Force, Weighting Null, Axis trans None



Standard Concentration, ng	Response (peak area, m/z 280) (S) enantiomer
0.1875	17844
0 3750	40165
1.500	155789
3 750	396162
7.500	774152
15.00	1549692
37.50	3746191
75.00	8041874

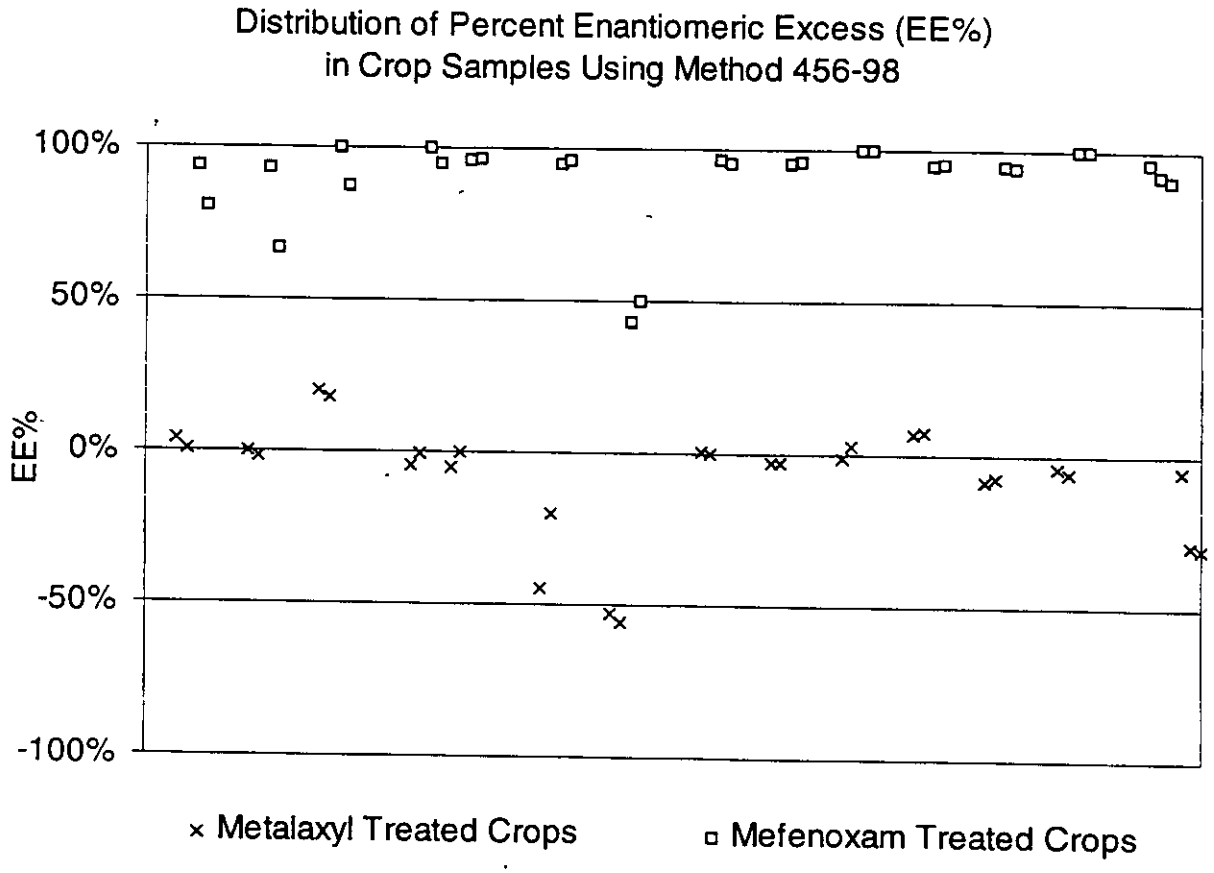
FIGURE 17: EXAMPLE CALIBRATION CURVES: (R) ENANTIOMER
CALIBRATION STANDARDS FROM FIGURE 16, CABBAGE HEADS (Continued)

Compound 2 name (r)CGA-48988
Coefficient of Determination 0 999796
Calibration curve 111032 * x + 0
Response type External Std, Area
Curve type Linear, Origin Force, Weighting Null, Axis trans None



Standard Concentration, ng	Response (peak area, m/z 280) (R) enantiomer
0 1875	17512
0.3750	42733
1 500	157185
3 750	404989
7.500	806379
15 00	1586486
37 50	4108899
75 00	8373998

FIGURE 18: GRAPHIC REPRESENTATION OF PERCENT ENANTIOMERIC EXCESS (EE%) IN SAMPLES CONTAINING FIELD-INCURRED RESIDUES OF EITHER METALAXYL OR MEFENOXAM



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3. Residue Test Report 02-FR-039-95, Report Number 1, Novartis Crop Protection. (MRID No. 43896902).

4. Residue Test Report 0S-FR-305-95, Report Number 1, Novartis Crop Protection. (MRID No. 43896905).

5. Residue Test Report 0W-FR-615-95, Report Number 1, Novartis Crop Protection. (MRID No. 43896903).

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7. Balasubramanian K., Analytical Method AG-395, "Improved Method for the Determination of Total Residues of Metalaxyl in Crop as 2,6-Dimethylaniline", December 7, 1982, Ciba-Geigy Corporation. (MRID No. 40106601).

8. Eudy, L. W., ABR-95109, "CGA-329351 - Magnitude of the Residues In or On Tobacco", January 11, 1996. (MRID No. 43896904).

9. Eudy, L. W., ABR-95118, "CGA-329351 - Magnitude of the Residues In or On Tomatoes", January 17, 1996. (MRID No. 43896902).

10. Eudy, L. W., ABR-95110, "CGA-329351 - Magnitude of the Residues In or On Cabbage", January 12, 1996. (MRID No. 43896905).

11. Eudy, L. W., ABR-95119, "CGA-329351 - Magnitude of the Residues In or On Potatoes", January 19, 1996. (MRID No. 43896903).

12. Moore, M. E., "Preparation of Crop Samples For Residue Analysis", Novartis Product Safety Group SOP Number 7.21, Revision 2, March 5, 1997.

APPENDIX I

1. Balasubramanian K., AG-395, "Improved Method for the Determination of Total Residues of Metalaxyl in Crop as 2,6-Dimethylaniline", December 7, 1982, Ciba-Geigy Corporation. (MRID No. 40106601).

2. Residue Test Report 0S-FR-108-95, Report Number 1, Novartis Crop Protection. (MRID No. 43896904).

3. Residue Test Report 02-FR-039-95, Report Number 1, Novartis Crop Protection. (MRID No. 43896902).

4. Residue Test Report 0S-FR-305-95, Report Number 1, Novartis Crop Protection. (MRID No. 43896905).

5. Residue Test Report 0W-FR-615-95, Report Number 1, Novartis Crop Protection. (MRID No. 43896903).

6. Eudy, L. W., ABR-95109, "CGA-329351 - Magnitude of the Residues In or On Tobacco", January 11, 1996. (MRID No. 43896904).

7. Eudy, L. W., ABR-95118, "CGA-329351 - Magnitude of the Residues In or On Tomatoes", January 17, 1996. (MRID No. 43896902).

8. Eudy, L. W., ABR-95110, "CGA-329351 - Magnitude of the Residues In or On Cabbage", January 12, 1996. (MRID No. 43896905).

9. Eudy, L. W., ABR-95119, "CGA-329351 - Magnitude of the Residues In or On Potatoes", January 19, 1996. (MRID No. 43896903).

EPA ADDENDUM For Residue Analytical Method
PP# 9F05044, 9E06005, and 9E06057

1) ACB made slight modifications to the HPLC/MS instrument parameters. The parameters for the HPLC/MS were:
HP Model 1100 LC/MSD MSD equipped with an electrospray ionization (ESI) source.

LC/MSD ESI Parameters were:

VCap: -4000 V
Fragmentor Voltage: 40 V
Electron Multiplier Gain: 3.0
Polarity Positive: $[M+H]^+$ ion monitored: m/z 280 for both metalaxyl and mefenoxam.
Drying Gas Temperature: 350°C.
Drying Gas Flow Rate: 10.0 L/min.
Nebulizer pressure: 40 psi.

HPLC Column: Chiralcel OD-R; 0.46cm i.d. x 25 cm.

Mobile Phase B: Water/10% Acetonitrile/0.05% Trifluoroectic Acid (TFA)
Mobile Phase A: Acetonitrile/10% Water/0.05% TFA
Column Temperature: 30° C
Injection Volume: 75 uL
Flow Rate: 0.50 mL/min (no column flow splitter was necessary)

Gradient

Time (min)	% B	% A
0.0	50	50
21.0	50	50
22.0	10	90
25.0	10	90
26.0	50	50
30.0	50	50

2. In the silica gel solid phase extraction (SPE) portionof the procedure, ACB used some vacuum assistance in order to elute the sample through the silica gel SPE cartridge. Vacuum was needed because the elution flow was far less than the recommended one drop per second, when using gravity flow.

3. Final Dilution of Sample: Although the procedure calls for diluting the sample, prior to LC/MS analysis, in the mobile phase (50/50 Acetonitrile/Water/0.05% TFA), ACB found that the sample should first be diluted to half of the intended final volume with acetonitrile and sonicated for ten seconds, followed by diluting the sample to the final volume with HPLC grade water/0.05% TFA.