November 22, 2010

Melissa Turner
Michael L. Johnson, LLC
632 Cantrill Drive
Davis, CA 95618

Re: GCMS-NCI SIM / SW-846 8270 modified; Pyrethroids Analytical Method Validation Data

Dear Melissa:

Please find attached the data requested that illustrates Caltest has demonstrated the capability for performing the GCMS-NCI SIM method comparable to the Robinson/Sygenta method as submitted:

1. Caltest Standard Operating Procedure (CAL-SOP) "*PYRETHROIDS BY GCMS-NCI SIM / SW-846 8270 Modified*" which identifies the methodology was adopted from the Robinson/Syngenta method "*Analytical Method for the Determination of Synthetic Pyrethroids in Sediment by Gas Chromatography-Negative Chemical Ionization Mass Spectrometry*" (2006) and represents a modification of SW-846 8270 method "*Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS)*".

2. In response to requirements "*Guide to Method Flexibility and Approval of EPA Water Methods*" and "*Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water*", Caltest is referencing the primary Method Validation submitted by the Pyrethroid Working Group to the California Dept. of Pesticides Regulations, Nov.29, 2006 (final report), of the Robinson/Syngenta method.

3. Initial Calibration (ICAL) data demonstrating linearity (r^2 ranging 0.991-0.999) with minimum response over a dynamic range of 0.005 – 0.05 ppm (on-column), relating to reporting limits 0.33 - >300 ug/Kg and yielding an average response RSD% <15%, meeting SW-846 8270 criteria. ICAL verification (pyr 2* std @ 0.01ppm) passed all for all compounds > 70% (range 72-93%).

4. Initial Method Detection Limit (MDL) Study performed July 2009, per 40CFR Appendix B to Part 136, reflecting MDL results ranging 2.5-8 times lower than Reporting Limit (RL) objective of 0.33 ug/Kg. MDL results yielded average 77 ± 0.2 to 100 ± 0.3 (mean % recovery ± standard deviation).

5. Initial precision and recovery (IPR) as our Demonstration of Capability reflects four spiked lab matrix samples' analytical results yielding passing recoveries meeting 100 ± 50% acceptance criteria and Relative Standard Deviation (RSD%) of ≤ 30% for solids.

6. Other QC included;
   a. Method Blank demonstrating process and system are contamination-free with surrogate recovery 86%;
   b. All spiked lab matrix samples reflect passing surrogate recoveries for LCS samples ranging 82-93% and MDL samples 83-136% with a mean of 102%.

Please feel free to contact me for any questions and should more information be desired. Thank you.

Sincerely,
Caltest Analytical Laboratory

Patrick Ingram
Laboratory Director

Enclosures

Analytical Method Standard Operating Procedure

CALTEST STANDARD OPERATING PROCEDURE

PYRETHROIDS BY GCMS-NCI SIM / SW-846 8270 Modified

Reviewed by:	Richard Heines	Title: Organics Technical Lead	Date: 11/23/10
Reviewed by:	Sonya Babcock	Title: Quality Assurance Officer	Date: 11/23/10
Approved by:	Patrick Ingram	Title: Laboratory Director	Date: 11/23/10

This SOP outlines the exact procedure to be followed by all staff of Caltest Laboratory who are performing the indicated method. It is the responsibility of any individual performing the procedure to follow these instructions outlined in this document. Any significant modifications to this method require an amendment to this SOP with the approval of the department Coordinator, Laboratory Director (LD) and QAO. All amendments must be identified below, and attached to this document to be considered valid changes. Any deviations from this SOP require prior authorization from the department Coordinator, Lab. Director and QAO. In addition, all deviations from the written procedure require complete documentation in the appropriate logbook.

Review	Section	Description of Changes	Date	Initials Coord., LD, QA
#1	_____	_____	_____/_____/_____	_____
#2	_____	_____	_____/_____/_____	_____
#3	_____	_____	_____/_____/_____	_____
#4	_____	_____	_____/_____/_____	_____
#5	_____	_____	_____/_____/_____	_____

PYRETHROIDS by NCI ANALYTICAL PROCEDURE

1. Scope and Application

1.1 This method is applicable for trace level determination of pyrethroids in water samples of low to moderate complexity (surface waters, monitoring waters, wastewaters, et al) and solid matrices (soil, sediment, bio-solids, etc.).

1.2 Samples are extracted and undergo extensive cleanup measures prior to analysis by Gas Chromatography/Negative Chemical Ion-Mass Spectrometry-Selective Ion Monitoring (GCMS-NCI SIM) using the appropriate sample preparation procedures. All soils, sediments and sludges are dryed before extraction.

1.3 This method is specific to the determination of Bifenthrin, Cyfluthrin, Lambda-Cyhalothrin, Cypermethrin, Deltamethrin, Esfenvalerate, Fenpropathrin, and Permethrin as primary reportable target compounds with five additional optional pyrethroids and two organo-phosphate pesticides, but should be applicable to other pyrethroids and related pesticides as well. However, the selectivity of Chemical Ionization Mode limits the determination of compounds.

1.4 The detection limits of this method approaches instrumental limitations and may be affected by the level of sample interferences. The calibration range is targeted to be 0.5 ng/L to 5 ng/L for waters and 0.25 µg/Kg to 1 µg/Kg for solids, which typify the minimum quantity that can be detected with no interferences present.

1.5 This method is for use by or under the supervision of analysts experienced in the use and interpretation of Gas Chromatography/Negative Chemical Ion-Mass Spectrometry data.

2. Summary of Method

2.1 The analytical procedures of this method model the Robinson/Syngenta method, "*Analytical Method for the Determination of Synthetic Pyrethroids in Sediment by Gas Chromatography-Negative Chemical Ionization Mass Spectrometry*" (2006).

2.2 This method represents a modification of SW-846 method 8270 "*Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*".

2.3 This method is appropriate for extracts of all appropriate matrices prepared by EPA Methods 3510C, 3520C, 3535 3540C, 3541, 3550 (refer to appropriate SOP)..

2.3.1 Extraction of water samples are based on a nominal volume of 1000mL of sample extracted into a final volume of 1mL in Hexane, to achieve method detection limits;

2.3.2 Solids are based on a nominal sample amount of 15 grams of sample extracted into a final volume of 1mL in Hexane, to achieve method detection limits;

2.3.3 When actual sample amounts used differ from the nominal values stated, detection limits will be adjusted accordingly.

2.4 All soils/sludges and waters may necessitate extensive clean up to remove or reduce interferences before being analyzed (refer to clean up SOP DOC#: O-3-phase clean up).

2.5 The compounds are introduced into the GCMS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph. The mass spectrometer is operated in the negative chemical ionization (NCI) and selective ion monitoring (SIM) modes in order to improve sensitivity and selectivity of the pyrethroid pesticides. If needed to possibly minimize matrix enhancements of the analytes the GC can be split to two detectors, a MS and micro Electron Capture Detector (µECD) in an 5:1 split ratio. The µECD signal can also be a better aid in determining the extent of the matrix background than the mass spectrometer run in the highly

selective negative chemical ionization mode. In the dual detector mode, results could be reported from the µECD and confirmed by the MS, if all data meets method criteria.

2.6 Qualitative identification of the target analytes (Pyrethroid pesticides, Chlorpyrifos and Diazinon) on the MSD is accomplished by comparing their selective mass ion ratios to the mass ion ratios of an authentic standard. There are two techniques of quantitation that are used in this method. Either internal multi point ICAL quantitation or external two point bracketing standard quantitation.

2.6.1 The internal multi point ICAL quantitation technique is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.6.2 The external two point bracketing standard quantitation technique is accomplished by comparing the response of a major (quantitation) ion relative to an external (no internal standard used) two point bracketing standard calibration curve. The external two point bracketing quantitation technique is useful when there are known matrix enhancements or detector response drift which is fairly common with GCMS when operated in the negative chemical ionization mode.

3. Definitions

The following terms are defined for use in this document:

3.1 ACCURACY: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

3.2 BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. An analytical batch is comprised of up to 20 environmental samples plus related QC samples.

3.3 CONTROL SAMPLE: A QC sample introduced into a process to monitor the performance of the system.

3.4 FIELD DUPLICATES: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

3.5 LABORATORY CONTROL SPIKE: A known matrix spiked with compound(s) representative of sample target analytes. This is used to document laboratory performance.

3.6 MATRIX: The component or substrate (e.g., surface water, drinking water), which contains the analyte of interest.

3.7 MATRIX DUPLICATE: An intralaboratory-split sample, which is used to document the precision of a method in a given sample matrix.

3.8 MATRIX SPIKE: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

3.9 MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

3.10 METHOD BLANK: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.11 PRECISION: The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses. These samples should contain concentrations of analyte above the MDL, and may involve the use of matrix spikes. The most commonly used estimates of precision are:

3.11.1 Relative Standard Deviation (RSD) or the Coefficient of Variation (CV) measuring 2, or more, sample replicates; RSD = CV = 100 S/x;
where: x = the arithmetic mean of the measurements, and S = xi variance;

3.11.2 Relative Percent Difference (RPD) when only two sample replicates are available.
RPD = 100 $[(x^1 - x^2)/\{(x^1 + x^2)/2\}]$ where: x = sample concentration, respectively

3.12 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

3.13 REAGENT WATER: Water that has been generated by any method, which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

3.14 STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards, which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

3.15 SURROGATE: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

3.16 SIM: Selective ion monitoring

3.17 PCI: Positive Chemical Ionization

3.18 NCI: Negative Chemical Ionization

3.19 MSD: Mass selective detector.

3.20 GCMS: Gas Chromatography Mass Spectrometry

3.21 ECD: Electron Capture Detector

3.22 SOURCE: Component of MSD where ionization and ion focusing take place

3.23 AMU: Atomic mass unit

4. Interferences

4.1 Solvents, reagents, glassware and other hardware used during extraction and standards preparation can yield interferences. The lot numbers, or the Caltest reagent ID number or Caltest source number of all reagents and solvents used for standards preparation shall be recorded in the Standards Log Book to trace any sources of contamination that may occur.

4.2 Plastics should be avoided during the extract preparation procedure to eliminate sources of phthalate esters. Care should also be taken to limit contact of the latex gloves with the sample or reagents. Only Teflon, glass or metal equipment are to be used, such as Teflon squirt bottles, and all surfaces that come in contact with the sample should be solvent rinsed with methylene chloride.

4.3 Avoid cross-contamination between samples by rinsing any materials used for multiple samples between samples. Do not touch pipettes, graduated cylinders or squirt bottles to any of the glassware.

4.4 Matrix interference.

4.4.1 IS Interference - Low or high internal standard areas may be corrected by diluting the sample or by using the external standard calibration technique if target compounds are not affected by the interference.

4.4.2 Carryover - Contamination may occur from a previous injection. If this is suspected, re-analyze the sample with instrument blanks before and after the sample. Dirty extracts may require dilution and/or additional clean up to prevent interference, carryover, or overloading the column and detectors. Document any dilution or cleanup procedure in the Instrument Log Book. Footnote the report as appropriate.

4.4.3 Dirty Injection Port - Maintenance such as replacing the injection port liner, seal, and clipping the front of the column should be done routinely.

4.4.4 Column bleed - Rising baselines late in the chromatogram indicate column bleed. Utilize the column conditioning techniques outlined by the column manufacturer; trim both ends of the column, or solvent rinse the column.

4.4.5 Chemical Ionization source requires more cleaning than the electron impact source, and cannot tolerate any residual water in the extracts or any air leaks. If either air or water are introduced into the chemical ionization source, response and mass signal to noise are seriously degraded.

4.4.6 Chemical Ionization can also experience matrix enhancements that have been observed in matrix fortified spikes (Matrix Spikes). The addition of matrix modifiers such as peanut oil can minimize this affect. This method adds peanut oil to all standards and sample extracts thru the internal standard addition. The peanut oil concentration of the standards and sample extracts is at 0.1 percent.

5. Safety and Precautions

5.1 This method is used to analyze potentially hazardous samples. Prior to performing this analysis, review the MSDS for all standards and reagents to be used. Observe the recommended safety precautions. Protective clothing and safety glasses must be worn when handling samples or reagents, and all manipulations must be performed in a hood.

5.3 Maintain a clean and uncluttered workspace. Return all chemicals, reagents and resultant wastes to their designated storage area at the completion of the test.

6. Equipment and Supplies

6.1 2 ml ALS vials, inserts, and crimp top seals.

6.2 Crimper, 12mm

6.3 Analytical column: 30 meter x 0.25 mm I.D. , 0.25 μm df, capillary fused silica .

6.4 Vials - 8 mL, 4 mL and 2 mL screw-cap vials with teflon faced septum, clear and amber.

6.5 Syringes, Class A, gas-tight, or precision bore: 10 μl, 25 μl, 100 μl, 500 μl, 1000 μl. Autosampler syringes: 10 μl.

6.6 Volumetric flasks (Class A): 5.0 mL, 10.0 mL, 100.0 mL.

6.7 Data System: Agilent MS ChemStation (E.02.00.493) with ThruPut Target Data Analysis Software, Revision 4.12.

6.8 GCMS system: Agilent 7890A-GC with Agilent 5975 inert XL/EI/CI-MSD equipped with Agilent 7683B autosampler.

6.9 Misc. Chromatography supplies: ferrules- 0.4mm (85%vespel-15% graphite, and 100% graphite) Restek 4.0mm double gooseneck deactivated injection port liners with glass wool, 1.2 mm inlet seals, 10 mm diameter septa.

6.10 Balance -Mettler analytical balance capable of reading to 0.01g.

7. Reagents and Standards

7.1. Methylene chloride, Burdick & Jackson, High purity solvent.

7.2. Acetone, Mallinkrodt, Nanograde.

7.3. Hexane, Omnisolve, high priority solvent.

7.4. Internal Standards:

7.4.1. 2000 µg/ml, 4,4'-Dibromooctafluorobiphenyl; Accustandard M-625-06-10x

7.4.2. 2000µg/ml PCB-205; Accustandard C-205S
Six-month expiration date upon opening ampule.

7.4.3. Working Internal Standard Solution, 0.02 µg/mL, mix of above standards.
Six month expiration date.

7.5. Calibration Standards (Primary): custom Pyrethroid standard, 1000µg/ml, Accustandard S-12458-R4
Six month expiration date upon opening ampules.

7.6. Surrogate mix: Decachlorobiphenyl, Accustandard, neat, C-209N
One year expiration date upon opening ampule.

7.7. Working Standards with Surrogate-0.25 ppm,
Six month expiration date

7.8. Second source Standards: Custom Pyrethroid standard, 1000µg/ml. Accustandard, S-12458-R4
Prepared independently using a different lot and gravimetric weighing from the primary calibration standard
Six month expiration date upon opening ampule.

8. Sample Preservation and Storage

8.1. The containers used for sampling and storage should be glass or Teflon and have screw caps with Teflon. All samples and extracts should be stored at 4°C.

8.2. Aqueous Samples should be extracted within 3 days, but no later than 14days, of sampling, (Cyhalothrin and Permethrin potentially lacks stability after 3 days).

8.3. Soils can be frozen to extend the stability time.

9. Quality Control Procedures

9.1. A method blank (MB) is included for every 24-hour extraction batch, or not to exceed 20 environmental samples, and should be subjected to the same procedures as the samples. The blank is spiked with the same surrogate used in the samples. The blank should be free of contamination. If there are any target hits found in the analysis of the blank, a fresh extract aliquot should be prepped and run to determine if the contamination is a result of extraction or instrument contamination. If instrument contamination is suspected, run a solvent blank to confirm the problem.

9.2. At least one LCS must be prepared per analytical batch. Acceptance Criteria for Spiking Compounds in LCS can be found in Caltest internal QC. Recovery outside of these limits must be checked and noted. Consult Caltest Internal QC for corrective measures.

9.3. A set of Matrix Spike and Matrix Spike Duplicate should be prepared and analyzed per analytical batch. Acceptance Criteria for Spiking Compounds in MS/MSD can be found in Caltest internal QC. Recovery outside of these limits must be checked and noted. Consult Caltest Internal QC for corrective measures.

9.4. Internal Standard QC, shall be added to every sample in an analytical batch (including QC samples) at the same level of concentration as in the calibration standards. This is a good point to also add the 0.1% peanut oil to each sample.

An area range of plus 100% and minus 50% is calculated from the average ISTD area from the calibration curve or from the continuing calibration standard. An ISTD area outside of the acceptance range necessitates prepping and analyzing a fresh extract to verify the results. If the problem is due to matrix interference, dilute the sample, inject ISTD and analyze. Repeated ISTD failures within the sequence indicate a problem with the system or ISTD solution. Correct any problems and rerun with a fresh ISTD solution. Re-prep fresh sample extracts if new ISTD solution has been prepared.

9.5. Surrogate QC-The surrogates spike level, and the acceptance criteria are listed in Caltest internal QC.

9.6. Qualitative Data Analysis

9.6.1. The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample quantitation, secondary and tertiary ion ratio. The reference selective mass ion ratios must be generated by the laboratory using the conditions of this method. Compounds should be identified as present when the criteria below are met

9.6.2. The intensities of the characteristic ions of a compound maximize at the same scan or within one scan of each other. Selection of a peak by a data system where identification is based on the presence of chromatographic peaks containing ions specific for the target compound at a known retention time will be accepted as meeting this criterion

9.6.3. The Relative Retention Time of the sample component is within ± 0.06 RRT units of the RRT of the standard component $RRT = \frac{RT\ sample}{Rt\ Istd}$

RT sample = Retention Time of sample component
RT Istd = Retention Time of internal standard.

9.6.4. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference selective mass ion ratios.

9.6.5. Each chromatographic peak is quantified independently then the sum of isomers are reported as a total pesticide residue for each target analyte.

9.6.6. Identification is hampered when sample components (i.e. matrix interferences) are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When a peak obviously represents more than one sample component, appropriate selection of analyte spectra and any necessary background spectra subtraction is important. When analytes coelute, the identification criteria can be met, providing unique ions are present, but each analyte spectrum will contain a portion of the individual compounds coeluting.

10. Calibration and Standardization

10.1 Calibration Criteria: The GCMS system must pass the following criteria prior to the analysis of any samples. Should any criterion not be met, the problem must be corrected before proceeding. (ie: instrument maintenance performed and/or standards and samples rerun.)

10.2 The GCMS system must be tuned prior to sample analysis. Launch Chemstation MSD application and select Tune function:

10.2.1 Adjust methane reagent gas flow in PCI mode before doing an autotune. While doing the methane flow in the PCI mode also look for protonated water (amu = 19) this is a good indicator if there is an air leak (water vapor in air);

Note: When an air leak is determined, find and seal the leak, vent the system; then re-clean the source followed by pump-down of at least 4 hours (consult manufacturer procedures); and initiate another tune in PCI mode again.

10.2.2 Perform the autotune in the PCI mode first. Review the PCI autotune report for air leaks and proper methane flow.

10.2.3 Then load the NCI tune method and do a second autotune in the NCI mode. If the autotune completes, it passes.

10.2.4 There is no tune check standard (i.e. DFTPP).

10.3 Initial Calibration -An initial calibration is performed prior to the analysis of any samples using a minimum of five points containing all the compounds of interest. The laboratory analyses standards that range from 0.5ppb to 500ppb, on-column, representing 0.5 – 500 ng/L report values.

10.4 Using 4µl injections for the split mode or 3ul injections if only the MSD is being used. Each standard is acquired on the appropriate detector. Tabulate area responses against concentration for each compound of interest including the Internal Standard, and calculate the Response Factor (RF) for each compound using the following equation:

RF = (AsCis)/(AisCs) where:

As = Response for the parameter to be measured.
Ais = Response for the Internal Standard.
Cis = Concentration of the Internal Standard (µg/mL).
Cs = Concentration of the parameter to be measured (µg/mL).

The results are used to plot a calibration curve of Response Ratios, vs. RF. The linearity of the curve for each compound is checked and adjustments are made as necessary. A calibration curve is considered linear (acceptable) if the average RF is <= 15% and/or the grand mean average RF of all the target analytes is <=15%. If a quadratic curve is used you must use no less than 6 calibration points and achieve a correlation coefficient of 0.995 or higher (approaching 1.0).

10.4 A mid-level secondary standard is injected following the calibration curve to verify the validity of the primary standards. This standard is from a different Lot # and gravimetric weighing than those purchased for the initial calibration. All compounds should meet 30% acceptance criteria. Should any compound not meet the criteria, the samples will be additionally reviewed for that compound. Should the sample prove positive for that compound, the discrepancy between the calibration and the secondary standard will be resolved and the sample re run.

11. Procedure

11.1 Proper documentation is essential at all points of sample preparation.

11.1.1 Before preparing any sample extract for analysis, check the extraction sheet for any comments concerning extraction and multiplier, check the Semi-volatiles Worklist for any work notes, test requested, age, client ID, matrix, and sample description. Confirm any anomalies with the Confirmation order or the Chain of Custody. Notify the department manager or project manager if there are any discrepancies.

11.1.2 The GCMS Instrument Run Log must be created during the course of the analysis. See SOP Q-LOGBOOK for the correct procedure for logbook entries. The following information is needed to complete the GCMS Instrument Logbook (See Appendix 1 for a sample of the logbook page).

11.1.2.1 Operator - Enter the Initials of the analyst(s) responsible for loading the samples and standards.

11.1.2.2 Method - Enter acquisition and data analysis method if separate.

11.1.2.3 Column - Analytical column

11.1.2.4 Comments-Lot #s for Standards, detailed information about sample dilutions (ie: 100µl-500µl=5x), and any other comments.

11.1.3 All containers or glassware used to hold the samples or QC should be clearly marked to prevent confusions or switching of samples.

11.1.4 ALS vials-Label with sample number, date extract was prepared for analysis, Det code, and any dilution made to the sample.

11.1.5 The Semivolatiles Standards Log Book must be completed whenever standards or reagents are prepared. See SOP Q-LOGBOOK for the correct procedure for logbook entries.

11.1.6 MSDF Maintenance Log Book: Any maintenance; routine or special, column changes, additions or changes performed on the system must be recorded in the MSDF Maintenance Log Book. The result of any maintenance procedures must also be recorded.

11.2 Acquisition

11.2.1 Refer to instrument maintenance logbook for run instrument parameters. Update run instrument parameters every time they are changed.

11.3 Analysis

11.3.1 The samples are extracted according to Caltest SOP's O-3510PREP, O-3550PREP and O-GCMSPREP. ALS vials are labeled appropriately. Samples may be diluted with hexane. **The ISTD must have equilibrated at room temperature before use.** A dilution on the sample, or a new curve must be prepared that will properly bracket the concentration of that compound. All dilutions are recorded on the extract vial, in the Instrument Log Book, within the analytical sequence run log and entered into Target Analytical Software for each sample.

11.3.2 Sequence - A typical sequence is:

11.3.2.1 Priming standards (up to five injections @ 500ppb)

11.3.2.2 ICAL 0.5 to 500ppb (5 to 12 levels)

11.3.2.3 Secondary source standard, mid level

11.3.2.4 Response consistency check standards (up to five injections @ 20ppb)

11.3.2.5 Bracketing standards @ 5,20 and 50ppb, alternating 1-2 levels bracketing up to four injections until closing response check sample

11.3.2.6 QC samples-MB, LCS, LCSD

11.3.2.7 Samples, MS, MSD.

11.3.2.8 Closing response check (0.5ppb) to check system sensitivity check.

11.4 Sample extracts in the refrigerator at ≤ 6°C until prepped and analyzed. Analyzed extracts should be archived until the data is reviewed for completeness. Neat extracts will be kept for 60 days past the extraction date. All extracts must be disposed in the proper manner.

11.5 Data Reporting, and Archival

11.5.1 Generate Quant Reports using Target software for samples and QC. IS and surrogate recoveries must pass QC criteria outlined in section 9 of this document. Corrective steps

may be taken when necessary to achieve QC requirements (ie: manual integration of a peak, dilution of the sample, or other measures

11.5.2 Using the Review feature of the Target software, check any hits that are suspect. Manual integration of a peak may be necessary to accurately quantitate the sample. Regenerate the report after any changes are made.

11.5.3 Taking into account the multiplier for the sample, report concentrations of compounds that meet all of the following criteria:

11.5.3.1 The mass spectral data meets the criteria set forth in section 10.

11.5.3.2 The peak has a retention time that falls within the retention time window. See section 10.

11.5.3.3 The concentration calculated is above the reporting limit taking into account the sample extraction and dilution factors.

11.5.3.4 The experience of the analyst must weigh heavily in the interpretation of chromatograms.

11.6 Sample Data Entry - When reporting sample results on the LIMS system, enter the following data as appropriate:

11.6.1 DILUTION FACTOR: The detection limits in LIMS assume a dilution factor of 1 reflecting a 1000mL -> 1mL prep for aqueous samples and a 15g -> 2mL for soils/sludges. All dilutions entered in LIMS should be rounded to one significant figure). To calculate dilution factor multipliers, when two or more dilutions were made on a sample, enter the product dilution factor.

11.6.2 RESULTS – Post results in LIMS as raw data values, LIMS will report results in proper significant figures or “ND” for non-detected target compounds.

11.7 To report QC results on LIMS, first generate a TARGET QC report. All QC data results posted to LIMS using raw data with a minimum of three significant figures.

11.8 Completed data results are to be submitted for second-party review to a designated competent, trained analyst reviewer familiar with this analysis or the Technical Lead/Coordinator/Lab Director for data approval and filing.

11.9 Trouble shooting

11.9.1 The most common causes of loss of response are a dirty source and / or an air leak. The GC inlet septum should be replaced frequently due to multiple punctures of a septum are a source of air leakage when a detector is under vacuum such as a mass spectrometer. Other low response checks include the auto sampler syringe for breakage or partial blockage. Check ferrule nuts at injector and detector and tighten if necessary. Check gas supplies and check for leaks. Check for septum particles in the injector liner. Cut several inches of column from the injector end and/or detector end. Low responses may also be caused poorly performing filaments, or an old multiplier (voltage >2400mV)

11.9.2 No peaks-Check the autosampler syringe for breakage and replace if necessary. Check if analytical column is broken or leaking. Check the gas supplies and replace tanks as necessary. Verify flow through the column. Check injector liner for septum debris.

11.9.4 Large, misshapen peaks-Compounds in the sample may be overloading the detector. Dilute the sample and rerun.

11.9.5 Noisy baseline- remove column nuts and check for crushed ferrule and/or column. Remove a few inches of column, replace with a new ferrule and bake out system at 325° C for 30 minutes and check for baseline stability. Further problems may indicate a contaminated column requiring solvent rinsing or replacement.

12. Calculations

12.1 The internal standard technique -The Enviroquant GC software and Target Analytical software automatically calculates the concentrations of analytes using the following equation:

$$\text{Concentration, } \mu g/L = \frac{(As)\,(Is)}{(Ais)\,(RF)}$$

As = Quant ion response for the compound to be measured.
Is = Amount of internal standard.
Ais= Quant ion response for the internal standard.
RF = Response factor

12.2 The external standard technique is to be employed as in cases where there is interference with the IS or detector drift or matrix enhancements. The concentration can be determined by using the following equation:

$$\text{Concentration } \mu g/L = \frac{Auk \times Cs}{As}$$

Auk = Quant ion response of the compound being measured.
As = Quant ion response of the compound in the standard.
Cs = Concentration of the standard.

12.3 Multipliers are used to relate the results calculated in the final analysis to the reported units.

12.3.1 Liquid matrices

Analysis units = ng/mL-ppb Reporting units = ng/L-ppt

$$\text{Result: on column amount} \times \frac{\text{Final Volume}}{\text{Initial Volume}} \times \text{Dilution Factor}$$

12.3.2 Solid matrices

Analysis units = ng/mL-ppb Reporting units = µg/Kg-ppb

$$\text{Result: on column amount} \times \frac{\text{Final Volume}}{\text{Initial Volume}} \times \text{Dilution Factor}$$

13. Pollution Prevention

13.1 There are no particular pollution prevention steps taken by the laboratory for this test.

14. Data Assessment and Acceptance Criteria for Quality Control Measures.

14.1 The data is referenced in Caltest's internal QC summary manual.

15. Corrective Actions for Out of Control Data.

15.1 The data is referenced in Caltest's internal QC summary manual.

16. Contingencies for Handling Out-Of-Control or Unacceptable Data.

16.1 The contingencies for handling out-of-control or unacceptable data are addressed in the Caltest SOP: Q-CORRECT.

17. Waste Management

17.1 All solvent saturated aqueous waste is collected in a drum. The waste is shipped out for disposal using a hazard waste disposal company. It is profiled as waste corrosive liquid.

17.2 All dichloromethane is collected in a solvent drum. The waste is shipped out for disposal using a hazard waste disposal company. It is profiled as waste Dichloromethane.

17.3 All Acetone waste is collected in a drum. The waste is shipped out for disposal using a hazard waste disposal company. It is profiled as waste flammable liquid.

17.4 All waste handling in performed in accordance with Caltest waste handling procedures.

18. Method Performance

18.1 Certified Reference and Performance Evaluation materials are not currently available specific to Pyrethroids analysis.

19. References

19.1 *Analytical Method For The Determination Of Synthetic Pyrethroids in Sediment by Gas Chromatography-Negative Chemical Ionization Mass Spectometry*. Neil J. Robinson, Syngenta Ltd. (2006).

19.2 EPA SW-846 8270 *Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS)*

Appendix 1

Pyrethroid List of Analytes

Pyrethroid Analyte	***CAS Number***	***Ret Time***	***1º***	***2º***	***3º***
Bifenthrin (Biphenthrin)	82657-04-3	19.7	386	387	241
Cyfluthrin (Baythroid)	68359-37-5	24.4	207	209	171
Lambda-Cyhalothrin	91465-08-6	21.5	241	205	243
Cypermethrin	52315-07-8	24.9	207	209	171
Fenvalerate / Esfenvalerate (Pydrin)	51630-58-1	26.7	211	213	212
Tau-Fluvalinate	102851-06-9	26.7	294	296	295
Fenpropathrin *(Danitol)	39515-41-8	20.0	141		
Permethrin	52645-53-1	23.3	207	209	
Tralomethrin/ Deltamethrin	66841-25-6	27.8	81	295	297
Allethrin	584-79-2	13.6	167	168	
Tetramethrin	7696-12-0	19.8	331	332	

Additional Analyte per request:

Analyte	CAS *Number*		***1º***	***2º***	***3º***
Chlorpyrifos	2921-88-2	12.3	315	214	313
*Diazinon	333-41-5	9.6	169		

Ret Time = retention time.
1º = Quantitation Ion
2º = Qualifying Ion
3º = Monitoring Ion
* = NCI only produces one ion, the compound qualifies using 1º and retention time.

LABORATORY VALIDATION:

VALIDATION OF THE RESIDUE ANALYTICAL METHOD: "RESIDUE ANALYTICAL METHOD FOR THE DETERMINATION OF RESIDUES OF BIFENTHRIN, CYPERMETHRIN, CYFLUTHRIN, DELTAMETHRIN, ESFENVALERATE, FENPROPATHRIN, LAMBDA-CYHALOTHRIN AND PERMETHRIN IN SEDIMENT"

FINAL REPORT

DATA REQUIREMENT: EPA: Ecological Effects Test Guidelines
OPPTS 850.7100 Data Reporting for Environmental Chemistry Methods

AUTHOR: Richard L. Reed II

COMPLETION DATE: November 29, 2006

PERFORMING LABORATORY: Morse Laboratories, Inc.
1525 Fulton Avenue
Sacramento, CA 95825 USA

STUDY IDENTIFICATION: Protocol No.: MLI-06-02
Morse Labs Project No.: ML06-1286-PWG

SPONSOR: Pyrethroid Working Group
c/o Fred Pearson
Chair PWG Coordination Committee
Syngenta Crop Protection, Inc.
410 Swing Road
Greensboro, NC 27409 USA

Vol. 1 of 2

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

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).

Company: Pyrethroid Working Group (PWG) — Consortium No. 64977
Member Companies: Bayer CropScience, DuPont Crop Protection, FMC Corporation, Pytech Chemicals GmbH, Valent USA Corporation, and Syngenta Crop Protection

Company Agent: Fred Pearson
Chair PWG Coordination Committee

Dec 22 2006
Date

Claim of Ownership

This document is being submitted to the United States Environmental Protection Agency (USEPA) specifically under provisions contained in FIFRA as amended. Pyrethroid Working Group thereby consents to the use and disclosure of this material by USEPA according to FIFRA §10. In submitting this material to the USEPA according to the method and format requirements contained in PR Notice 86-5, Pyrethroid Working Group does not waive any protection or right involving this material that would have been claimed by the company if this material had not been submitted to the USEPA.

NOTE: THIS DOCUMENT (INCLUDING INFORMATION CONTAINED HEREIN) IS THE PROPRIETARY PROPERTY OF PYRETHROID WORKING GROUP.

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

All aspects of this study carried out by **MORSE LABORATORIES, INC.** were conducted in accordance with the FIFRA Good Laboratory Practice Standards (40 CFR 160), with the following exceptions:

The fresh water sediment sample, BUCGR, was collected and prepared prior to the Study Director signing the protocol.

Sediment characterization analyses were not conducted according to the Good Laboratory Practice Standards.

Richard L. Reed II
Study Director
Morse Laboratories, Inc.

November 29, 2006
Date

Gary L. Westberg
Laboratory Director
Morse Laboratories, Inc.

Nov 29, 2006
Date

Fred Pearson
Sponsor Representative
Chair PWG Coordination Committee

Dec 22 2006
Date

REPORT APPROVAL

Richard L. Reed II
Study Director
Morse Laboratories, Inc.

November 29, 2006
Date

Gary L. Westberg
Laboratory Director
Morse Laboratories, Inc.

Nov 29, 2006
Date

N. Robinson

Neil Robinson
Study Monitor
Syngenta Crop Protection, Inc.
Jealott's Hill Research Centre
Bracknell, Berkshire
RG42 6EY, UK

Dec 7, 2006
Date

Fred Pearson
Sponsor Representative
Chair PWG Coordination Committee
Representative of Submitter/Sponsor

FJP 12/22/06
Dec 22, 2006
Date

Submitter/Sponsor: Pyrethroid Working Group
c/o Fred Pearson
Chair PWG Coordination Committee
Syngenta Crop Protection, Inc.
410 Swing Road
Greensboro, NC 27409 USA

QUALITY ASSURANCE STATEMENT

Morse Laboratories, Inc. Project Number: ML06-1286-PWG

Protocol Number: MLI-06-02

Quality Assurance inspections were carried out during the execution of the Study by Quality Assurance personnel according to §40 CFR 160.35 of the EPA Good Laboratory Practice Standards to ensure the integrity of the data. The final report reflects the raw data.

Inspection Phase	Dates of Inspection	Study Director*
Protocol Review	04/17/06	05/23/06
In Process	05/16/06, 05/17/06, 05/23/06	05/23/06
Analytical Data Audit	08/03/06, 08/04/06 09/18/06, 09/19/06	08/15/06 11/29/06
Final Report	09/14/06, 09/15/06, 09/18/06, 09/19/06, 09/26/06, 11/29/06	11/29/06

*And Study Director's Management

Sherita Normington
Morse Laboratories, Inc. Quality Assurance Coordinator

November 29, 2006
Date

GENERAL INFORMATION

Study Title:	Validation of the Residue Analytical Method: "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment"
Sponsor:	Pyrethroid Working Group c/o Fred Pearson Chair PWG Coordination Committee Syngenta Crop Protection, Inc. 410 Swing Road Greensboro, NC 27409 USA
Study Monitor:	Neil Robinson Syngenta Crop Protection, Inc. Jealott's Hill Research Centre Bracknell, Berkshire RG42 6EY, UK
Test Site:	Morse Laboratories, Inc. 1525 Fulton Ave. Sacramento, CA 95825
Protocol No.:	MLI-06-02
Morse Labs Project No.:	ML06-1286-PWG
Test Items/Reference Substances:	Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin, Permethrin
Test System:	Sediment

STUDY PARTICIPANTS

Staff:

Sample Preparation:	Mike Garrett *Sample Manager/Freezer Custodian* Paul Gilbert *Laboratory Assistant*

Extractions:	Mina Bhatt *Laboratory Chemist* Seth Nasca *Laboratory Technician II*
Instrumentation:	Kevin Clark *Chief GC Chemist*
Calculations:	Tiffany Borrero *Data Specialist* Andrea Westberg *Laboratory Administrator*
Report Author:	Richard L. Reed II *Analytical Project Coordinator*
Study Director:	Richard L. Reed II *Analytical Project Coordinator*
Quality Assurance:	Sherita Normington *Quality Assurance Coordinator*
Management:	Gary L. Westberg *Laboratory Director*

SCHEDULE

Date Study Protocol was signed by the Study Director:	April 12, 2006
Start of Experiments:	April 21, 2006
Completion of Experiments (last instrumentation date):	May 18, 2006
Date of Final Report:	November 29, 2006

GUIDELINES

This study was performed in compliance with the following guideline:

- EPA: Ecological Effects Test Guidelines: OPPTS 850.7100 Data Reporting for Environmental Chemistry Methods

ARCHIVE LOCATIONS

All raw data for this study not specific to the operation of Morse Laboratories, Inc., the original final report and the study protocol, will be archived at Syngenta, GLP Archives, Jealott's Hill International Research Centre, Bracknell, Berkshire, RG42 6EY, UK, per the Sponsor's request. Exact copies of the raw data for this study will be retained at Morse Laboratories, Inc.

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1.0 SUMMARY

This report describes an analytical method used for the determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin in sediment samples. The method was developed by Syngenta Crop Protection, Inc. entitled "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment." A draft version of the method was used to generate the data. The results of this validation study, including any modifications needed to be made to the draft version are documented in the final version of the method found in Appendix 1.

Bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin residues were extracted from sediment by shaking with a methanol/water mixture and hexane for one hour. The sample was centrifuged and an aliquot of the upper hexane layer was evaporated to dryness and re-dissolved in a small volume of hexane. The hexane sample was then subjected to a silica solid phase extraction (SPE) cleanup step prior to residue determination by gas chromatography with mass selective detection using negative ion chemical ionization (GC-MS/NICI). The limit of quantitation of the method was 0.1 µg/kg for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and 1.0 µg/kg for permethrin.

The purpose of this study was to validate this method according to the EPA Ecological Effects Test Guidelines: OPPTS 850.7100 Data Reporting for Environmental Chemistry Methods. A limit of quantitation (LOQ) of 0.1 µg/kg for all targeted analytes except permethrin, which was 1.0 µg/kg, and a working range from 0.1 µg/kg to 1.0 µg/kg for all targeted analytes except permethrin, which was from 1.0 µg/kg to 10 µg/kg, was validated in sediment. The validation was performed on two different types of sediment samples (fresh water and estuarine) collected from sites in California.

The study was conducted by Morse Laboratories, Inc. (Morse Labs) of Sacramento, California according to Protocol No. MLI-06-02, entitled "Validation of the Residue Analytical Method: "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment" and Protocol Amendments 1 and 2 (Appendix 2).

For each sediment type, the validation included five replicates of control sample fortified with all targeted compounds at their respective LOQs (0.1 µg/kg for all except permethrin, which was 1.0 µg/kg) and five replicates of control sample fortified with all targeted compounds at 10 times their respective LOQs (1.0 µg/kg for all except permethrin, which was 10 µg/kg). All fortified samples, along with two control samples and one reagent blank were analyzed for residues of all targeted compounds. The results, which demonstrate that the method is applicable for the determination of the targeted pyrethroid compounds in both types of sediment, are summarized below:

Summary of Method Validation Recoveries					
Matrix	**Analyte**	**Spike level (µg/kg)**	**Sample size (n)**	**Net Recoveries[a] (%)**	**Overall Mean ± std dev (%)**
Sediment, Fresh Water	Bifenthrin	0.1	5	103, 106, 107, 107, 109	107 ± 4.5
		1.0	5	100, 111, 109, 102, 115	
	Cypermethrin	0.1	5	107, 102, 105, 104, 110	112 ± 9.4
		1.0	5	111, 130, 120, 105, 123	
	Cyfluthrin	0.1	5	107, 105, 107, 101, 108	111 ± 8.1
		1.0	5	111, 127, 119, 108, 120	
	Deltamethrin	0.1	5	89, 78, 84, 86, 102	98 ± 13
		1.0	5	104, 116, 111, 99, 109	
	Esfenvalerate	0.1	5	76, 77, 82, 73, 84	95 ± 19
		1.0	5	105, 122, 117, 99, 115	
	Fenpropathrin	0.1	5	102, 94, 107, 108, 107	108 ± 6.8
		1.0	5	110, 115, 116, 108, 116	
	Lambda-Cyhalothrin	0.1	5	84, 83, 96, 93, 108	103 ± 13
		1.0	5	106, 121, 113, 104, 118	
	Permethrin	1.0	5	97, 99, 94, 100, 108	104 ± 10
		10	5	97, 124, 107, 93, 117	
Sediment, Estuarine	Bifenthrin	0.1	5	91, 94, 90, 91, 87	95 ± 5.7
		1.0	5	93, 98, 106, 101, 95	
	Cypermethrin	0.1	5	98, 114, 108, 96, 97	106 ± 6.9
		1.0	5	102, 110, 114, 111, 105	
	Cyfluthrin	0.1	5	89, 108, 92, 107, 98	102 ± 7.8
		1.0	5	98, 107, 113, 108, 102	
	Deltamethrin	0.1	5	87, 78, 89, 84, 86	85 ± 7.7
		1.0	5	74, 81, 96, 97, 77	
	Esfenvalerate	0.1	5	97, 111, 112, 109, 118	107 ± 7.2
		1.0	5	95, 109, 112, 107, 102	
	Fenpropathrin	0.1	5	95, 109, 106, 111, 105	105 ± 5.9
		1.0	5	98, 105, 114, 107, 100	
	Lambda-Cyhalothrin	0.1	5	90, 106, 102, 104, 105	102 ± 5.9
		1.0	5	97, 105, 111, 106, 99	
	Permethrin	1.0	5	98, 111, 102, 107, 111	105 ± 5.5
		10	5	96, 106, 111, 108, 101	

[a]Calculated following correction by the average residue concentration measured in the corresponding unfortified control samples

The validation of this method was successful. For each sediment type, the mean recoveries for all analytes at each fortification level were between 70% and 120%. All corresponding

relative standard deviations (RSD) in all cases were ≤20%. Except for five instances where analyte residues found in the unfortified control samples were greater than 30% of the LOQ (in fresh water sediment: deltamethrin at an average of 0.0453 µg/kg, esfenvalerate at an average of 0.0655 µg/kg, and lambda-cyhalothrin at an average of 0.0414 µg/kg; and in estuarine sediment: bifenthrin at an average of 0.0469 µg/kg and deltamethrin at an average of 0.0720 µg/kg), all remaining residues were less than 30% of the LOQ.

Although some residues greater than 30% of the LOQ (but still less than the LOQ) were found in untreated control samples and some individual recoveries fell outside the 70-120% acceptance range, based on the overall precision and accuracy of the recovery data, this method, in general, meets the guideline requirements (1).

2.0 INTRODUCTION

Syngenta Crop Protection, Inc. method entitled "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment" was validated in this study. A draft version of the method was used to generate the data. The results of this validation study, including any modifications needed to be made to the draft version are documented in the final version of the method found in Appendix 1.

This study was conducted to satisfy guideline requirements described in the EPA Ecological Effects Test Guidelines: OPPTS 850.7100 Data Reporting for Environmental Chemistry Methods.

The method was validated on two types of sediment (fresh water and estarine) using unfortified and fortified samples of each. Fortifications were conducted at both the LOQ and 10 × LOQ of each targeted compound (5 replicates each). The validation results are reported herein.

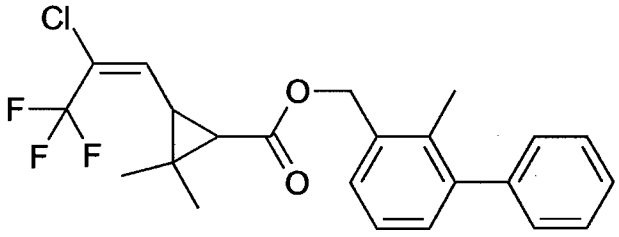
The study was conducted by Morse Laboratories, Inc. (Morse Labs) of Sacramento, California according to Protocol No. MLI-06-02, entitled "Validation of the Residue Analytical Method: "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment" and Protocol Amendments 1 and 2 (Appendix 2).

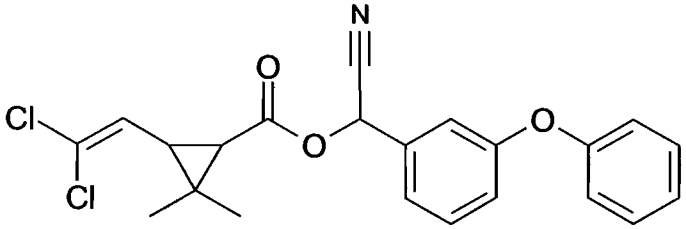
This report contains the following: test item and test system information, experimental details, method summary, calculations, results and discussion, example chromatography, and results generated from the analyses performed by Morse Laboratories, Inc.

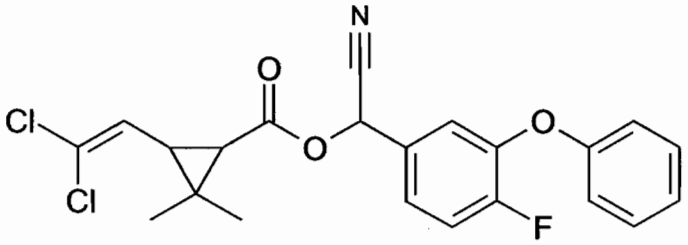
3.0 MATERIALS and METHODS

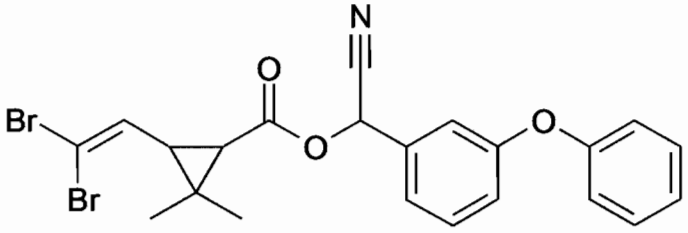
3.1 Test Items/Reference Substances

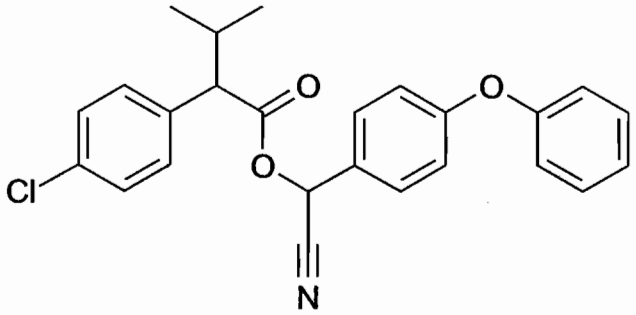
The analytical (reference) standards (test items) used in this study were:

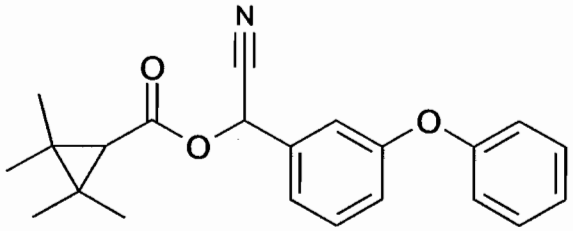
Compound	Bifenthrin
IUPAC Name:	2-methylbiphenyl-3-ylmethyl (*Z*)-(1*RS*,3*RS*)-3-(2-chloro-3-3-3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	82657-04-3
CAS Name:	(2-methyl[1,1'-biphenyl]-3-(2-chloro-3-3-3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	97.8
Lot No.:	BI-29
Source:	FMC Agricultural Products
Expiration Date:	8/2007
Storage:	Typically -8 °C to -22 °C

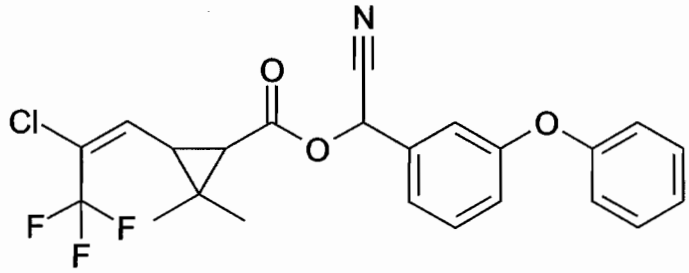
Compound	Cypermethrin
IUPAC Name:	(*RS*)-α-cyano-3-phenoxybenxyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	52315-07-8
CAS Name:	Cyano(3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	99.4
Lot No.:	AMS 202/102
Source:	Syngenta Crop Protection
Expiration Date:	3/2009
Storage:	Ambient

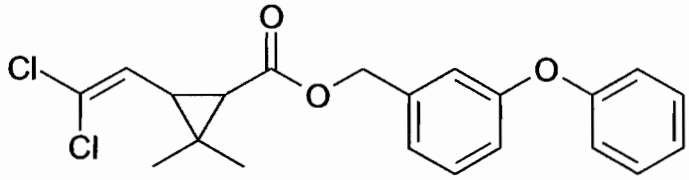
Compound	Cyfluthrin
IUPAC Name:	*(RS)*-α-cyano-4-fluoro-3-phenoxybenzyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	68359-37-5
CAS Name:	Cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	50.2
Lot No.:	K-618
Source:	Bayer CropScience
Expiration Date:	12/31/13
Storage:	Typically -8 °C to -22 °C

Compound	Deltamethrin
IUPAC Name:	(*S*)-α-cyano-3-phenoxybenzyl (1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	52918-63-5
CAS Name:	1-[*R*-[1-α(*S**),3α]]-cyano(3-phenoxyphenyl)methyl 3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	99.4
Lot No.:	K-1375
Source:	Bayer CropScience
Expiration Date:	9/8/10
Storage:	Typically -8 °C to -22 °C

Compound	Esfenvalerate
IUPAC Name:	(*S*)-α-cyano-3-phenoxybenzyl (*S*)-2-(4-chlorophenyl)-3-methylbutyrate
CAS Number:	66230-04-4
CAS Name:	[*S*-(*R**,*R**)]-cyano(3-phenoxyphenyl)methyl 4-chloro-2-(1-methylethyl)benzeneacetate
Structure:	
% Purity:	98.7
Lot No.:	YB656-058
Source:	DuPont Crop Protection
Expiration Date:	11/26/07
Storage:	Ambient

Compound	Fenpropathrin
IUPAC Name:	(*RS*)-α-cyano-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate
CAS Number:	64257-84-7
CAS Name:	Cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate
Structure:	
% Purity:	99.7
Lot No.:	AS 459h
Source:	Valent
Expiration Date:	8/5/06
Storage:	Typically -8 °C to -22 °C

Compound	Lambda-cyhalothrin
IUPAC Name:	A reaction product containing equal quantities of (*S*)-α-cyano-3-phenoxybenzyl (*Z*)-(1*R*,3*R*)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate and (*R*)-α-cyano-3-phenoxybenzyl (*Z*)-(1*R*,3*R*)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	91465-08-6
CAS Name:	[1α(*S**),3α(*Z*)]-(±)-cyano(3-phenoxyphenyl)methyl 3-(2-chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	98.7
Lot No.:	ASJ10012-04
Source:	Syngenta Crop Protection
Expiration Date:	8/2006
Storage:	Typically 1 °C to 8 °C

Compound	Permethrin
IUPAC Name:	3-phenoxybenzyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	52645-53-1
CAS Name:	(3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	99.7
Lot No.:	S01-2613
Source:	Syngenta Crop Protection
Expiration Date:	7/2006
Storage:	Typically -8 °C to -22 °C

Characterization data for the reference substances are maintained by the Pyrethroid Working Group.

The test and reference substances used in this study were procured and stored as directed on "Analytical Standard Certificate" or by the Study Monitor. All solutions made from the reference substances were stored according to the method.

3.2 Test Systems

Two types of sediment were evaluated in this study: fresh water and estuarine. Both control sediment samples were submitted to Morse Labs by Pacific Ecorisk of Matrinez, CA. The fresh water sediment (BUCGR) was received at the lab on March 30, 2006. The estuarine sediment (Paradise Cove) was received at the lab on May 12, 2006

Upon receipt of the samples at the laboratory, they were immediately placed in refrigerated storage (typically 1-8 °C), where they remained pending subsampling. The samples were transferred to freezer storage (typically -20 ± 5 °C) after subsampling.

3.3 Equipment and Reagents

The equipment and reagents used for the method validation were as outlined in the method (Section 2. Materials and Appendices 1 and 2). Identical or equivalent apparatus and materials were used, as permitted by the method.

3.4 Standard Solution Preparation

3.4.1 Stock Standard Solutions

The following concentrations of stock standard solutions were prepared. These solutions were prepared to contain each targeted analyte individually.

All targeted analytes except cyfluthrin:

Ten (10.0) mg (corrected for purity) of each applicable analytical standard were accurately weighed and quantitatively transferred to individual 50-mL volumetric flasks and brought to volume with acetone. The resulting concentration was 200 µg/mL.

Cyfluthrin:

Five (5.0) mg (corrected for purity) of cyfluthrin analytical standard were accurately weighed and quantitatively transferred to a 25-mL volumetric flask and brought to volume with acetone. The resulting concentration was 200 µg/mL.

3.4.2 Laboratory (Procedural) Fortification Standard Solutions

The following concentrations of fortification standard solutions were prepared. These solutions were prepared as mixtures containing all targeted analytes. The first concentration listed is for all analytes except permethrin, which is represented by the second concentration listed:

1.0 µg/mL/ 10 µg/mL:	125 µL of each of the 200 µg/mL stock standard solutions prepared for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, and lambda-cyhalothrin and 1.25 mL of the 200 µg/mL stock standard solution prepared for permethrin were transferred to a 25-mL volumetric flask. The solution was brought to a final volume of 25 mL with acetone. The solution was mixed well.
0.1 µg/mL/ 1.0 µg/mL:	2.5 mL of the 1.0 µg/mL/10 µg/mL standard solution mixture prepared above were transferred to a 25-mL volumetric flask. The solution was brought to a final volume of 25 mL with acetone. The solution was mixed well.

3.4.3 Instrumentation (Calibration) Standard Solutions

The following concentrations of calibration standard solutions were prepared. These solutions were prepared as mixtures containing all targeted analytes. The first concentration listed is for all analytes except permethrin, which is represented by the second concentration listed:

20 ng/mL/ 200 ng/mL:	5.0 mL of a 0.1 µg/mL/1.0 µg/mL mixed fortification standard solution were transferred to a 25-mL volumetric flask. The contents were brought to volume with 0.1% (v/v) peanut oil in acetone. The solution was mixed well.
10 ng/mL/ 100 ng/mL:	2.5 mL of a 0.1 µg/mL/1.0 µg/mL mixed fortification standard solution were transferred to a 25-mL volumetric flask. The contents were brought to volume with 0.1% (v/v) peanut oil in acetone. The solution was mixed well.
5.0 ng/mL/ 50 ng/mL:	1.25 mL of a 0.1 µg/mL/1.0 µg/mL mixed fortification standard solution were transferred to a 25-mL volumetric flask. The contents were brought to volume with 0.1% (v/v) peanut oil in acetone. The solution was mixed well.

2.5 ng/mL/ 25 ng/mL:	625 µL of a 0.1 µg/mL/1.0 µg/mL mixed fortification standard solution were transferred to a 25-mL volumetric flask. The contents were brought to volume with 0.1% (v/v) peanut oil in acetone. The solution was mixed well.
1.0 ng/mL/ 10 ng/mL:	250 µL of a 0.1 µg/mL/1.0 µg/mL mixed fortification standard solution were transferred to a 25-mL volumetric flask. The contents were brought to volume with 0.1% (v/v) peanut oil in acetone. The solution was mixed well.
0.5 ng/mL/ 5.0 ng/mL:	125 µL of a 0.1 µg/mL/1.0 µg/mL mixed fortification standard solution were transferred to a 25-mL volumetric flask. The contents were brought to volume with 0.1% (v/v) peanut oil in acetone. The solution was mixed well.

3.5 Sample Preparation

Each untreated control sample was processed for analysis by transferring the entire sample received into a large metal bowl. Any rocks or plant debris present was removed by hand. The samples were then mixed well using a large metal spoon. Following processing, the samples were divided into six equal portions and each portion was placed into a properly labeled plastic bag. The bagged portions were placed into frozen storage (typically at less than or equal to -15 °C) where they remained until removal for subsampling and analysis.

3.6 Fortification Procedures

Each validation set per sediment type consisted of one reagent blank, two control samples, five control samples fortified at the LOQ (0.1 µg/kg for all analytes except permethrin which was 1.0 µg/kg) for each analyte and five control samples fortified at 10 × LOQ for each analyte as shown below:

Matrix	Sample Type	Fortifying Compounds	Fortification Level	# of Samples
None	Reagent blank	None	0.0 µg/kg	1
Sediment	Control	None	0.0 µg/kg	2
Sediment	Spike	All targeted analytes (except Permethrin) and Permethrin	0.1 µg/kg (LOQ) 1.0 µg/kg (LOQ)	5
Sediment	Spike	All targeted analytes (except Permethrin) and Permethrin	1.0 µg/kg (10 x LOQ) 10 µg/kg (10 x LOQ)	5

Twelve 50.0-g portions of a specific sediment type control sample were used as samples for each validation set. Samples were designated as controls or fortified controls. Fortified controls were each fortified with all eight analytes at either the LOQ (50 µL of a mixed standard solution containing 0.1 µg/mL of all analytes except permethrin which was at 1.0 µg/mL) or 10 × LOQ (50 µL of a mixed standard solution containing 1.0 µg/mL of all analytes except permethrin which was at 10 µg/mL). Once fortified, the fortified control samples along with appropriate controls and a reagent blank, were analyzed as per method.

3.7 Calibration Procedures

Determination and quantitation of pyrethroid residues were conducted using gas chromatography employing negative chemical ionization mass selective detection (GC-MS/NICI). The instrument calibration standards for this study were prepared by making appropriate dilutions of stock standard solutions of the test item. The standard solutions bracketed the working range. The lowest standard of the working range was no less than half the concentration of the lowest sample concentration expected.

Calibration standards were injected concurrently with the sample injections during the course of the analytical run. For preparation of the calibration curve, the standards were injected sequentially during the run. Because calculations were based on bracketing standard response factors, appropriate standards were injected so that no more than four sample injections were bracketed between standards. Each analytical run began and ended with a standard injection.

3.8 Analytical Method

The method developed by Syngenta Crop Protection, Inc. entitled "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment" was used for the analyses in this study. A draft version of the method was used to generate the data. The results of this validation study, including any modifications needed to be made to the draft version are documented in the final version of the method found in Appendix 1.

Bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin residues were extracted from sediment by shaking with a methanol/water mixture and hexane for one hour. The sample was centrifuged and an aliquot of the upper hexane layer was evaporated to dryness and re-dissolved in a small volume of hexane. The hexane sample was then subjected to a silica solid phase extraction (SPE) cleanup step prior to residue determination by gas chromatography with mass selective detection using negative ion chemical ionization (GC-MS/NICI). The limit of quantitation of the method was 0.1 µg/kg for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and 1.0 µg/kg for permethrin.

3.9 Instrumentation

The GC conditions employed for the analyses in this study were as follows:

Operating Conditions

Instrument:	An Agilent 6890 gas chromatograph equipped with an Agilent 5973N mass selective detector operated in negative chemical ionization mode, a HP 7683 autosampler, and a HP G1701CA MS ChemStation.
Column:	30 m × 0.25 mm i.d. fused silica column crossbonded with 0.25 µm film thickness Varian CP-Sil 8CB-MS
Inlet liner:	4 mm i.d. double gooseneck splitless liner (unpacked)
Injection volume:	2 µL
Injection mode:	pulsed splitless, 30 psi for 1 minute, purge flow to split vent 50 mL/min @ 2 minutes
Carrier gas:	Helium
Column flow:	0.9 mL/min, constant flow

Detector reagent gas: Methane @ 30%

Dwell time: 50 msec

Tuning: Prior to analysis, the instrument is autotuned for ions m/z 185, 351 and 449

Temperatures: Injector: 275 °C

GC/MSD Transfer line: 280 °C

Column:
Initial: 80 °C, hold 1.00 minute
Rate 1: 40 °C/minute to 180 °C
Rate 2: 5 °C/minute
Final: 305 °C

Ions monitored:

Analyte	Target Ion (*m/z*)	Qualifier Ion 1 (*m/z*)	Qualifier Ion 2 (*m/z*)
Bifenthrin	386	387	241
Fenpropathrin	141	-	-
Lambda-Cyhalothrin	205	241	243
Permethrin	207	209	-
Cyfluthrin	207	209	171
Cypermethrin	207	209	171
Esfenvalerate	211	213	-
Deltamethrin	297	81	296

Retention times:

Analyte	Peak #	Retention time (min)
Bifenthrin	1	18.1
Fenpropathrin	1	18.5
Lambda-Cyhalothrin	1	19.6
	2	19.9
Permethrin	1	21.5
	2	21.8
Cyfluthrin	1	22.5
	2	22.7
	3	22.8
	4	22.9
Cypermethrin	1	23.2
	2	23.4
	3	23.5
	4	23.6
Esfenvalerate	1	24.9
	2	25.3
Deltamethrin	1	25.9
	2	26.3

3.10 Calculations

A validated software application was used to generate a standard curve based on linear regression. The regression functions were used to calculate a best-fit line and to demonstrate linearity of the GC/MSD detector system.

Calculations for instrumental analysis were conducted using an Excel spreadsheet. Average response factors for bracketing standards were used to determine sample residues. Where compounds produced multiple peaks (isomers), the total area under all peaks was used in the calculations.

The equations used for calculation purposes are as follows:

1. $$Response\ factor = \frac{analyte\ response\ (area)\ for\ standard}{concentration}$$

2. $$Average\ response\ factor = \frac{Sum\ of\ response\ factors\ of\ bracketing\ standards}{2}$$

3. The amount of analyte (in µg/kg) found in the sample was calculated according to the following equation:

$$\mu g/kg = \frac{total\ peak\ resp.}{avg.\ resp.\ fact.} \times \frac{FV\ (mL)}{Samp.\ wt.\ (g)} \times \frac{solv.\ (mL)}{aliq.\ (mL)} \times GC\ dil.\ factor$$

where:

total peak resp.	=	total of all peak responses (isomers) where applicable for sample.
avg. resp. fact.	=	average response factor for the bracking standards
FV (mL)	=	volume of extract submitted to instrumentation (1.0 mL)
Samp. wt. (g)	=	amount of sample taken through the extraction process (50.0 g)
solv. (mL)	=	volume of extraction solvent added (50 mL)
aliq. (mL)	=	volume of extraction solvent taken through the method (10.0 mL)
GC dil. factor	=	dilution of sample extract required to produce analyte responses bracketed by standards

4. The percent recovery for fortified control samples was calculated as follows:

$$\%\ Recovery = \frac{\mu g/kg\ found\ in\ fortified\ control - \mu g/kg\ found\ in\ control}{\mu g/kg\ added} \times 100$$

Example Calculations

All targeted analyte residues were calculated in an identical manner for both sediment types. Only examples of bifenthrin residue calculations in estuarine sediment will be provided and thus serve to illustrate the calculations for all other analytes in all sediment types.

1. ML ticket #83942, Bifenthrin, Estuarine Sediment, Set #3, Paradise Cove, **Control** 5 (Figure 10):

Peak response (area) = 3071

1. $Response\ factor\ std.1 = \frac{6282}{1.0} = 6282$

   $Response\ factor\ std.2 = \frac{121315}{20} = 6066$

2. $Average\ response\ factor = \frac{6282 + 6066}{2} = 6174$

3. $\mu g/kg = \frac{3071}{6174} \times \frac{1.0\,mL}{50.0\,g} \times \frac{50\,mL}{10.0\,mL} \times 1$

   $\mu g/kg = 0.049740849$

   $Reported = <0.1\ \mu g/kg$

2. ML ticket #83942, Bifenthrin, Estuarine Sediment, Set #3, Paradise Cove, **Fortified Control** 21 @ 0.1 µg/kg (Figure 15):

   Peak response (area) = 8465

1. $Response\ factor\ std.1 = \frac{6312}{1.0} = 6312$

   $Response\ factor\ std.2 = \frac{5926}{1.0} = 5926$

2. $Average\ response\ factor = \frac{6312 + 5926}{2} = 6119$

3. $\mu g/kg = \frac{8465}{6119} \times \frac{1.0\,mL}{50.0\,g} \times \frac{50\,mL}{10.0\,mL} \times 1$

   $\mu g/kg = 0.138339598$

   $Reported = 0.138\ \mu g/kg$

4. $\%\ Recovery = \frac{0.138\,\mu g/kg - 0.0469\,\mu g/kg}{0.1\,\mu g/kg\ added} \times 100$

   $Recovery = 91\%$

4.0 RESULTS

4.1 Method Validation Results

A summary of the validation results are presented below:

Summary of Method Validation Recoveries					
Matrix	**Analyte**	**Spike level (µg/kg)**	**Sample size (n)**	**Net Recoveries[a] (%)**	**Overall Mean ± std dev (%)**
Sediment, Fresh Water	Bifenthrin	0.1	5	103, 106, 107, 107, 109	107 ± 4.5
		1.0	5	100, 111, 109, 102, 115	
	Cypermethrin	0.1	5	107, 102, 105, 104, 110	112 ± 9.4
		1.0	5	111, 130, 120, 105, 123	
	Cyfluthrin	0.1	5	107, 105, 107, 101, 108	111 ± 8.1
		1.0	5	111, 127, 119, 108, 120	
	Deltamethrin	0.1	5	89, 78, 84, 86, 102	98 ± 13
		1.0	5	104, 116, 111, 99, 109	
	Esfenvalerate	0.1	5	76, 77, 82, 73, 84	95 ± 19
		1.0	5	105, 122, 117, 99, 115	
	Fenpropathrin	0.1	5	102, 94, 107, 108, 107	108 ± 6.8
		1.0	5	110, 115, 116, 108, 116	
	Lambda-Cyhalothrin	0.1	5	84, 83, 96, 93, 108	103 ± 13
		1.0	5	106, 121, 113, 104, 118	
	Permethrin	1.0	5	97, 99, 94, 100, 108	104 ± 10
		10	5	97, 124, 107, 93, 117	
Sediment, Estuarine	Bifenthrin	0.1	5	91, 94, 90, 91, 87	95 ± 5.7
		1.0	5	93, 98, 106, 101, 95	
	Cypermethrin	0.1	5	98, 114, 108, 96, 97	106 ± 6.9
		1.0	5	102, 110, 114, 111, 105	
	Cyfluthrin	0.1	5	89, 108, 92, 107, 98	102 ± 7.8
		1.0	5	98, 107, 113, 108, 102	
	Deltamethrin	0.1	5	87, 78, 89, 84, 86	85 ± 7.7
		1.0	5	74, 81, 96, 97, 77	
	Esfenvalerate	0.1	5	97, 111, 112, 109, 118	107 ± 7.2
		1.0	5	95, 109, 112, 107, 102	
	Fenpropathrin	0.1	5	95, 109, 106, 111, 105	105 ± 5.9
		1.0	5	98, 105, 114, 107, 100	
	Lambda-Cyhalothrin	0.1	5	90, 106, 102, 104, 105	102 ± 5.9
		1.0	5	97, 105, 111, 106, 99	
	Permethrin	1.0	5	98, 111, 102, 107, 111	105 ± 5.5
		10	5	96, 106, 111, 108, 101	

[a]Calculated following correction by the average residue concentration measured in the corresponding unfortified control samples

For each sediment type, the mean recoveries for all analytes at each fortification level were between 70% and 120%. All corresponding relative standard deviations (RSD) in all cases were ≤20%. Except for five instances where analyte residues found in the unfortified control samples were greater than 30% of the LOQ (in fresh water sediment: deltamethrin at an average of 0.0453 µg/kg, esfenvalerate at an average of 0.0655 µg/kg, and lambda-cyhalothrin at an average of 0.0414 µg/kg; and in estuarine sediment: bifenthrin at an average of 0.0469 µg/kg and deltamethrin at an average of 0.0720 µg/kg), all remaining residues were less than 30% of the LOQ.

4.2 Confirmation of Residues

Peak validation (confirmation and demonstration of specificity) was not performed due to the highly specific mode of determination (e.g GC-MS/NICI).

4.3 Interferences

Detailed interference studies were not performed. No interferences due to solvents or labware were observed.

4.4 Characterization Data

Percent organic matter (Total Organic Carbon) was determined for on each bulk sediment sample used as control samples in this study. The results are as follows:

Sample Identification	Sediment Type	Total Organic Carbon (%)
BUCGR	Fresh Water	1.31
Paradise Cove	Estuarine	0.86

See Appendix 5 for Analytical Report from the performing laboratory (Columbia Analytical Services, Kelso, Washington). With regard to this study, these analyses were not performed under GLP.

4.5 Ruggedness Testing

Formal ruggedness testing was not conducted for this method.

4.6 Time Required for Analysis

A set of 12 samples, from initial extraction to setting the samples up for GC analysis, should require approximately 8 hours. Set up of the GC analysis should require approximately 1.0 hour. The automated GC analysis should take approximately 13-14 hours for the chromatographic run. Data processing and review should take approximately 6 hours.

4.7 Problems Encountered/Modifications/Recommendations

Several of the targeted analytes determined by this method produce distinct GC responses related to their isomeric composition. The method should more throughly discuss how to handle these isomer responses in the calculation process and provide examples of how to sum the peaks for quantitation.

4.8 Limits of Quantitation (LOQ) and Limits of Detection (LOD)

The limits of quantitation (LOQ) and detection (LOD) were not statistically determined for this method. However, for evaluation of this method, the LOQ was defined as the lower limit of method validation (the lowest level of fortification demonstrated to have acceptable recovery and precision), which was 0.1 µg/kg for all targeted analytes except permethrin, which was 1.0 µg/kg. The LOD of the method, as discussed in the method, is estimated to be 0.02 µg/kg for all targeted analytes except permethrin, which is estimated at 0.2 µg/kg.

4.9 Representative Chromatography

For fresh water sediment, example chromatograms of typical GC standards (bracketing standards) are presented in this report as Figures 1, 3, 5, 6, and 8. Example chromatograms from one control sample and two fortified control samples (one at each fortification level) are presented in Figures 2, 4, and 7. For estuarine sediment, example chromatograms of typical GC standards (bracketing standards) are presented in this report as Figures 9, 11, 13, 14, and 16. Example chromatograms from one control sample and two fortified control samples (one at each fortification level) are presented in Figures 10, 12, and 15. Example chromatograms for the calibration standards and related standard curves are presented in Figures 17-22.

4.10 Protocol/SOP/Method Deviations

Three protocol deviations, dated May 23, 2006, August 4, 2006, and September 25, 2006, respectively, were generated for this study. The first two deviations documented control samples used in the study that contained targeted analyte residues greater than 30% of the LOQ: fresh water control sample (BUCGR) contained mean residues of 0.0414 µg/kg, 0.0655 µg/kg, and 0.0453 µg/kg for lambda-cyhalothrin, esfenvalerate, and deltamethrin, respectively and estuarine sediment control sample (Paradise Cove) contained mean residues of 0.0469 µg/kg and 0.0720 µg/kg for bifenthrin and deltamethrin, respectively. The third deviation documented the omission of partlce size determination with regard to characterization of the sediment samples used in the study. See Appendix 3.

No SOP deviations were generated for this study.

One method deviation, dated August 4, 2006, was generated for this study. It documented the actual equation used in the calculation of sample residues.

5.0 CONCLUSION

The validation of this method was successful. For each sediment type, the mean recoveries for all analytes at each fortification level were between 70% and 120%. All corresponding relative standard deviations (RSD) in all cases were ≤20%. Except for five instances where analyte residues found in the unfortified control samples were greater than 30% of the LOQ (in fresh water sediment: deltamethrin at an average of 0.0453 µg/kg, esfenvalerate at an average of 0.0655 µg/kg, and lambda-cyhalothrin at an average of 0.0414 µg/kg; and in estuarine sediment: bifenthrin at an average of 0.0469 µg/kg and deltamethrin at an average of 0.0720 µg/kg), all remaining residues were less than 30% of the LOQ.

Although some residues greater than 30% of the LOQ (but still less than the LOQ) were found in untreated control samples and some individual recoveries fell outside the 70-120% acceptance range, based on the overall precision and accuracy of the recovery data, this method, in general, meets the guideline requirements (1).

The analytical method described in this report is determined to be suitable for the analysis of the targeted analytes (bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin) in sediment samples.

6.0 REFERENCE

1. U.S. Environmental Protection Agency,.Office of Pollution Prevention and Toxic Substances (OPPTS), "Public Draft - Data Reporting for Environmental Chemistry Methods," OPPTS 850.7100, EPA-712-C-96-348, 1996.

TABLES SECTION

Abbreviations used in tables:

C.I.	=	confidence interval
Fort.	=	fortification
fort. cont.	=	fortified control
ID	=	identification
LOQ	=	limit of quantitation
n	=	number
N/A	=	not applicable
ND	=	none detected, no observable chromatographic response at retention time of analyte
µg/kg	=	microgram per kilogram
Rec.	=	recovery
RSD	=	relative standard deviation
Std. Dev.	=	standard deviation

TABLE 1. Method Validation Recoveries from Fresh Water Sediment

Sample ID	Set #	Fort. Level[a] µg/kg	Bifenthrin µg/kg found	Bifenthrin % Rec.	Cypermethrin µg/kg found	Cypermethrin % Rec.	Cyfluthrin µg/kg found	Cyfluthrin % Rec.	Deltamethrin µg/kg found[b]	Deltamethrin % Rec.[c]
Reagent Blank 2	2	N/A	ND	N/A	ND	N/A	ND	N/A	ND	N/A
BUCGR control 3	2	N/A	ND	N/A	ND	N/A	ND	N/A	<0.1 (0.0664)	N/A
BUCGR control 4	2	N/A	ND	N/A	ND	N/A	ND	N/A	<0.1 (0.0243)	N/A
BUCGR fort. cont. 11	2	LOQ	0.103	103	0.107	107	0.107	107	0.134	89
BUCGR fort. cont. 12	2	LOQ	0.106	106	0.102	102	0.105	105	0.123	78
BUCGR fort. cont. 13	2	LOQ	0.107	107	0.105	105	0.107	107	0.129	84
BUCGR fort. cont. 14	2	LOQ	0.107	107	0.104	104	0.101	101	0.131	86
BUCGR fort. cont. 15	2	LOQ	0.109	109	0.110	110	0.108	108	0.147	102
LOQ Summary		**Mean:**	N/A	**106**	N/A	**106**	N/A	**106**	N/A	**88**
		Std. Dev. (+/-):	N/A	**2.2**	N/A	**3.0**	N/A	**2.8**	N/A	**8.9**
		%RSD:	N/A	**2.1**	N/A	**2.9**	N/A	**2.6**	N/A	**10**
		95% C.I.(+/-):	N/A	**2.7**	N/A	**3.8**	N/A	**3.5**	N/A	**11**
BUCGR fort. cont. 16	2	10 x LOQ	0.997	100	1.11	111	1.11	111	1.09	104
BUCGR fort. cont. 17	2	10 x LOQ	1.11	111	1.30	130	1.27	127	1.21	116
BUCGR fort. cont. 18	2	10 x LOQ	1.09	109	1.20	120	1.19	119	1.16	111
BUCGR fort. cont. 19	2	10 x LOQ	1.02	102	1.05	105	1.08	108	1.04	99
BUCGR fort. cont. 20	2	10 x LOQ	1.15	115	1.23	123	1.20	120	1.14	109
10 × LOQ Summary		**Mean:**	N/A	**107**	N/A	**118**	N/A	**117**	N/A	**108**
		Std. Dev. (+/-):	N/A	**6.3**	N/A	**9.9**	N/A	**7.6**	N/A	**6.5**
		%RSD:	N/A	**5.8**	N/A	**8.4**	N/A	**6.5**	N/A	**6.1**
		95% C.I.(+/-):	N/A	**7.8**	N/A	**12**	N/A	**9.4**	N/A	**8.1**
Overall Recovery Summary (n = 10)		**Mean:**	N/A	**107**	N/A	**112**	N/A	**111**	N/A	**98**
		Std. Dev. (+/-):	N/A	**4.5**	N/A	**9.4**	N/A	**8.1**	N/A	**13**
		%RSD:	N/A	**4.2**	N/A	**8.4**	N/A	**7.3**	N/A	**13**
		95% C.I.(+/-):	N/A	**3.2**	N/A	**6.7**	N/A	**5.8**	N/A	**9.2**

[a]LOQ = 0.1 µg/kg for all analytes. 10 × LOQ = 1.0 µg/kg for all analytes.
[b]actual amount found in parentheses
[c]net recovery, corrected for average control contribution

TABLE 1. Method Validation Recoveries from Fresh Water Sediment (continued)

Sample ID	Set #	Fort. Level[a] µg/kg	Esfenvalerate		Fenpropathrin		Lambda-cyhalothrin		Permethrin	
			µg/kg found[b]	% Rec.[c]	µg/kg found	% Rec.	µg/kg found[b]	% Rec.[c]	µg/kg found	% Rec.
Reagent Blank 2	2	N/A	ND	N/A	ND	N/A	ND	N/A	ND	N/A
BUCGR control 3	2	N/A	<0.1 (0.0935)	N/A	ND	N/A	<0.1 (0.0420)	N/A	ND	N/A
BUCGR control 4	2	N/A	<0.1 (0.0376)	N/A	ND	N/A	<0.1 (0.0409)	N/A	ND	N/A
BUCGR fort. cont. 11	2	LOQ	0.141	76	0.102	102	0.125	84	0.973	97
BUCGR fort. cont. 12	2	LOQ	0.142	77	0.0938	94	0.124	83	0.988	99
BUCGR fort. cont. 13	2	LOQ	0.147	82	0.107	107	0.137	96	0.939	94
BUCGR fort. cont. 14	2	LOQ	0.138	73	0.108	108	0.134	93	0.995	100
BUCGR fort.cont. 15	2	LOQ	0.149	84	0.107	107	0.149	108	1.08	108
LOQ Summary		**Mean:**	N/A	**78**	N/A	**104**	N/A	**93**	N/A	**100**
		Std. Dev. (+/-):	N/A	**4.5**	N/A	**5.9**	N/A	**10**	N/A	**5.2**
		%RSD:	N/A	**5.7**	N/A	**5.7**	N/A	**11**	N/A	**5.2**
		95% C.I.(+/-):	N/A	**5.6**	N/A	**7.3**	N/A	**13**	N/A	**6.5**
BUCGR fort. cont. 16	2	10 x LOQ	1.12	105	1.10	110	1.10	106	9.72	97
BUCGR fort. cont. 17	2	10 x LOQ	1.29	122	1.15	115	1.25	121	12.4	124
BUCGR fort. cont. 18	2	10 x LOQ	1.24	117	1.16	116	1.17	113	10.7	107
BUCGR fort. cont. 19	2	10 x LOQ	1.06	99	1.08	108	1.08	104	9.32	93
BUCGR fort. cont. 20	2	10 x LOQ	1.22	115	1.16	116	1.22	118	11.7	117
10 × LOQ Summary		**Mean:**	N/A	**112**	N/A	**113**	N/A	**112**	N/A	**108**
		Std. Dev. (+/-):	N/A	**9.4**	N/A	**3.7**	N/A	**7.4**	N/A	**13**
		%RSD:	N/A	**8.4**	N/A	**3.3**	N/A	**6.6**	N/A	**12**
		95% C.I.(+/-):	N/A	**12**	N/A	**4.6**	N/A	**9.2**	N/A	**16**
Overall Recovery Summary (n = 10)		**Mean:**	N/A	**95**	N/A	**108**	N/A	**103**	N/A	**104**
		Std. Dev. (+/-):	N/A	**19**	N/A	**6.8**	N/A	**13**	N/A	**10**
		%RSD:	N/A	**20**	N/A	**6.3**	N/A	**13**	N/A	**9.9**
		95% C.I.(+/-):	N/A	**13**	N/A	**4.9**	N/A	**9.5**	N/A	**7.4**

[a]LOQ = 0.1 µg/kg for all analytes except permethrin, which is 1.0 µg/kg. 10 × LOQ = 1.0 µg/kg for all analytes except permethrin, which is 10 µg/kg.
[b]actual amount found in parentheses
[c]net recovery, corrected for average control contribution

TABLE 2. Method Validation Recoveries from Estuarine Sediment

Sample ID	Set #	Fort. Level[a] µg/kg	Bifenthrin µg/kg found	Bifenthrin % Rec.[c]	Cypermethrin µg/kg found	Cypermethrin % Rec.	Cyfluthrin µg/kg found	Cyfluthrin % Rec.[c]	Deltamethrin µg/kg found[b]	Deltamethrin % Rec.[c]
Reagent Blank 3	2	N/A	ND	N/A	ND	N/A	ND	N/A	ND	N/A
Paradise Cove control 5	2	N/A	<0.1 (0.0497)	N/A	ND	N/A	<0.1 (0.0234)	N/A	<0.1 (0.0641)	N/A
Paradise Cove control 6	2	N/A	<0.1 (0.0441)	N/A	ND	N/A	ND	N/A	<0.1 (0.0798)	N/A
Paradise Cove fort. cont. 21	2	LOQ	0.138	91	0.0979	98	0.101	89	0.159	87
Paradise Cove fort. cont. 22	2	LOQ	0.141	94	0.114	114	0.120	108	0.150	78
Paradise Cove fort. cont. 23	2	LOQ	0.137	90	0.108	108	0.104	92	0.161	89
Paradise Cove fort. cont. 24	2	LOQ	0.138	91	0.0955	96	0.119	107	0.156	84
Paradise Cove fort. cont. 25	2	LOQ	0.134	87	0.0970	97	0.110	98	0.158	86
LOQ Summary		**Mean:**	N/A	**91**	N/A	**103**	N/A	**99**	N/A	**85**
		Std. Dev. (+/-):	N/A	**2.5**	N/A	**8.0**	N/A	**8.6**	N/A	**4.2**
		%RSD:	N/A	**2.8**	N/A	**7.8**	N/A	**8.7**	N/A	**5.0**
		95% C.I.(+/-):	N/A	**3.1**	N/A	**9.9**	N/A	**11**	N/A	**5.2**
Paradise Cove fort. cont. 26	2	10 x LOQ	0.979	93	1.02	102	0.992	98	0.812	74
Paradise Cove fort. cont. 27	2	10 x LOQ	1.03	98	1.10	110	1.08	107	0.879	81
Paradise Cove fort. cont. 28	2	10 x LOQ	1.11	106	1.14	114	1.14	113	1.03	96
Paradise Cove fort. cont. 29	2	10 x LOQ	1.06	101	1.11	111	1.09	108	1.04	97
Paradise Cove fort. cont. 30	2	10 x LOQ	1.00	95	1.05	105	1.03	102	0.844	77
10 × LOQ Summary		**Mean:**	N/A	**99**	N/A	**108**	N/A	**106**	N/A	**85**
		Std. Dev. (+/-):	N/A	**5.1**	N/A	**4.8**	N/A	**5.8**	N/A	**11**
		%RSD:	N/A	**5.2**	N/A	**4.5**	N/A	**5.5**	N/A	**13**
		95% C.I.(+/-):	N/A	**6.4**	N/A	**6.0**	N/A	**7.2**	N/A	**13**
Overall Recovery Summary (n = 10)		**Mean:**	N/A	**95**	N/A	**106**	N/A	**102**	N/A	**85**
		Std. Dev. (+/-):	N/A	**5.7**	N/A	**6.9**	N/A	**7.8**	N/A	**7.7**
		%RSD:	N/A	**6.0**	N/A	**6.6**	N/A	**7.6**	N/A	**9.1**
		95% C.I.(+/-):	N/A	**4.1**	N/A	**5.0**	N/A	**5.6**	N/A	**5.5**

[a]LOQ = 0.1 µg/kg for all analytes. 10 × LOQ = 1.0 µg/kg for all analytes.
[b]actual amount found in parentheses
[c]net recovery, corrected for average control contribution

TABLE 2. Method Validation Recoveries from Estuarine Sediment (continued)

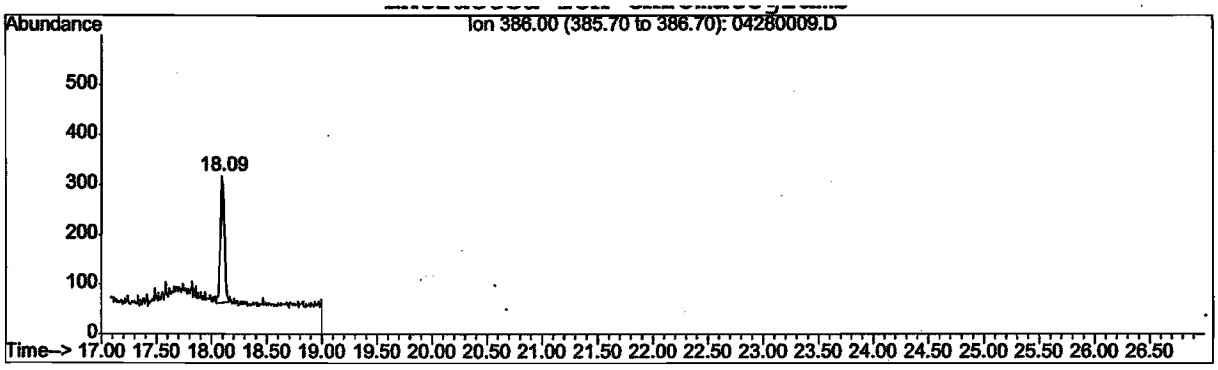
Sample ID	Set #	Fort. Level[a] µg/kg	Esfenvalerate µg/kg found[b]	Esfenvalerate % Rec.[c]	Fenpropathrin µg/kg found	Fenpropathrin % Rec.	Lambda-cyhalothrin µg/kg found[b]	Lambda-cyhalothrin % Rec.[c]	Permethrin µg/kg found	Permethrin % Rec.
Reagent Blank 3	2	N/A	ND	N/A	ND	N/A	ND	N/A	ND	N/A
Paradise Cove control 5	2	N/A	<0.1 (0.0155)	N/A	ND	N/A	<0.1 (0.0117)	N/A	ND	N/A
Paradise Cove control 6	2	N/A	<0.1 (0.0141)	N/A	ND	N/A	<0.1 (0.0104)	N/A	ND	N/A
Paradise Cove fort. cont. 21	2	LOQ	0.112	97	0.0945	95	0.101	90	0.984	98
Paradise Cove fort. cont. 22	2	LOQ	0.126	111	0.109	109	0.117	106	1.11	111
Paradise Cove fort. cont. 23	2	LOQ	0.127	112	0.106	106	0.113	102	1.02	102
Paradise Cove fort. cont. 24	2	LOQ	0.124	109	0.111	111	0.115	104	1.07	107
Paradise Cove fort. cont. 25	2	LOQ	0.133	118	0.105	105	0.116	105	1.11	111
LOQ Summary		**Mean:**	N/A	**109**	N/A	**105**	N/A	**101**	N/A	**106**
		Std. Dev. (+/-):	N/A	**7.7**	N/A	**6.2**	N/A	**6.5**	N/A	**5.7**
		%RSD:	N/A	**7.0**	N/A	**5.9**	N/A	**6.5**	N/A	**5.4**
		95% C.I.(+/-):	N/A	**9.6**	N/A	**7.7**	N/A	**8.1**	N/A	**7.1**
Paradise Cove fort. cont. 26	2	10 x LOQ	0.967	95	0.984	98	0.978	97	9.58	96
Paradise Cove fort. cont. 27	2	10 x LOQ	1.10	109	1.05	105	1.06	105	10.6	106
Paradise Cove fort. cont. 28	2	10 x LOQ	1.13	112	1.14	114	1.12	111	11.1	111
Paradise Cove fort. cont. 29	2	10 x LOQ	1.08	107	1.07	107	1.07	106	10.8	108
Paradise Cove fort. cont. 30	2	10 x LOQ	1.03	102	0.999	100	1.00	99	10.1	101
10 × LOQ Summary		**Mean:**	N/A	**105**	N/A	**105**	N/A	**104**	N/A	**104**
		Std. Dev. (+/-):	N/A	**6.7**	N/A	**6.3**	N/A	**5.6**	N/A	**5.9**
		%RSD:	N/A	**6.4**	N/A	**6.0**	N/A	**5.4**	N/A	**5.7**
		95% C.I.(+/-):	N/A	**8.3**	N/A	**7.8**	N/A	**7.0**	N/A	**7.4**
Overall Recovery Summary (n = 10)		**Mean:**	N/A	**107**	N/A	**105**	N/A	**102**	N/A	**105**
		Std. Dev. (+/-):	N/A	**7.2**	N/A	**5.9**	N/A	**5.9**	N/A	**5.5**
		%RSD:	N/A	**6.7**	N/A	**5.6**	N/A	**5.7**	N/A	**5.3**
		95% C.I.(+/-):	N/A	**5.1**	N/A	**4.2**	N/A	**4.2**	N/A	**4.0**

[a]LOQ = 0.1 µg/kg for all analytes except permethrin, which is 1.0 µg/kg. 10 × LOQ = 1.0 µg/kg for all analytes except permethrin, which is 10 µg/kg.
[b]actual amount found in parentheses
[c]net recovery, corrected for average control contribution

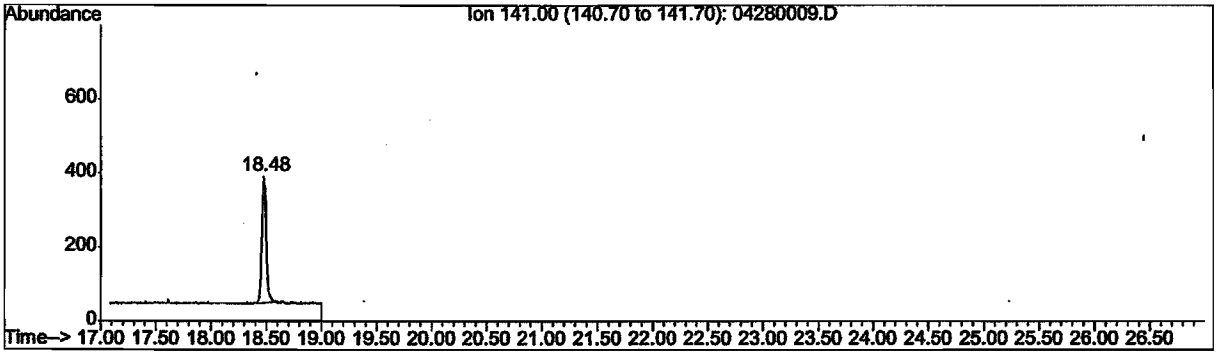
FIGURES SECTION

FIGURE 1. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



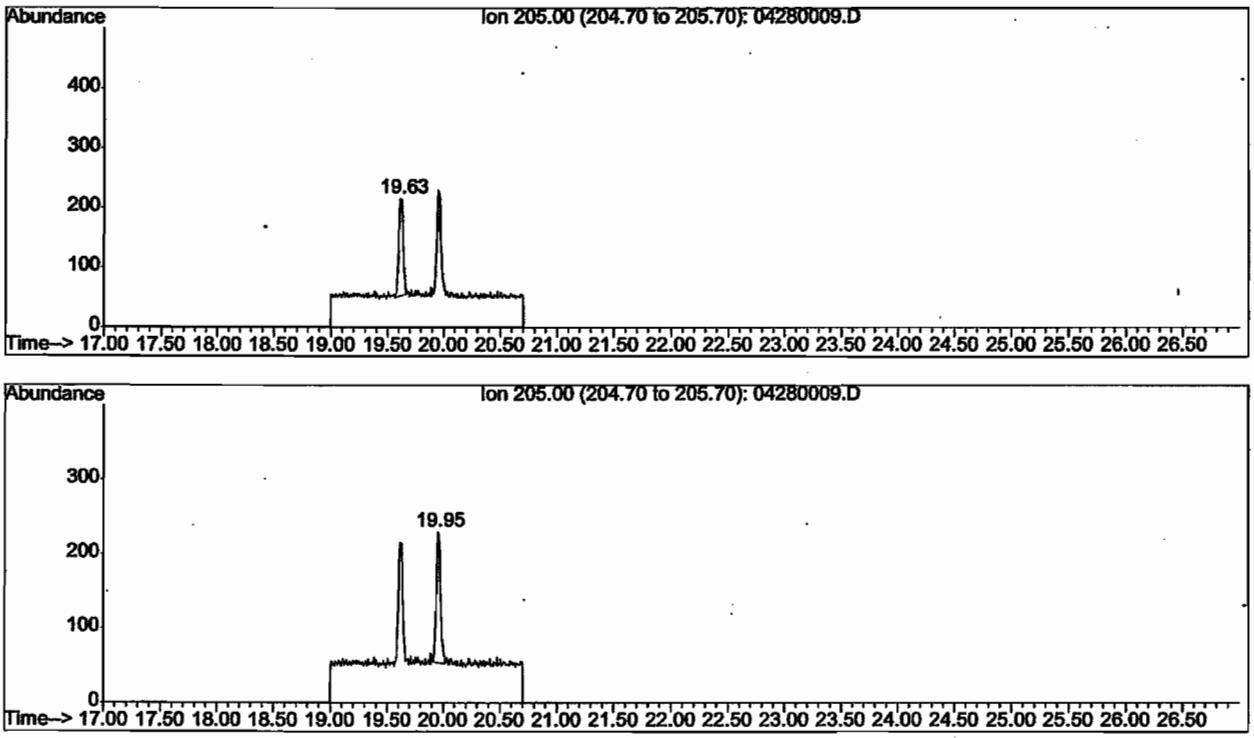
Bifenthrin
1.0 ng/mL
Peak Response (area): 6326



Fenpropathrin
1.0 ng/mL
Peak Response (area): 9369

FIGURE 1. (con't) Representative Chromatograms – Bracketing Standards

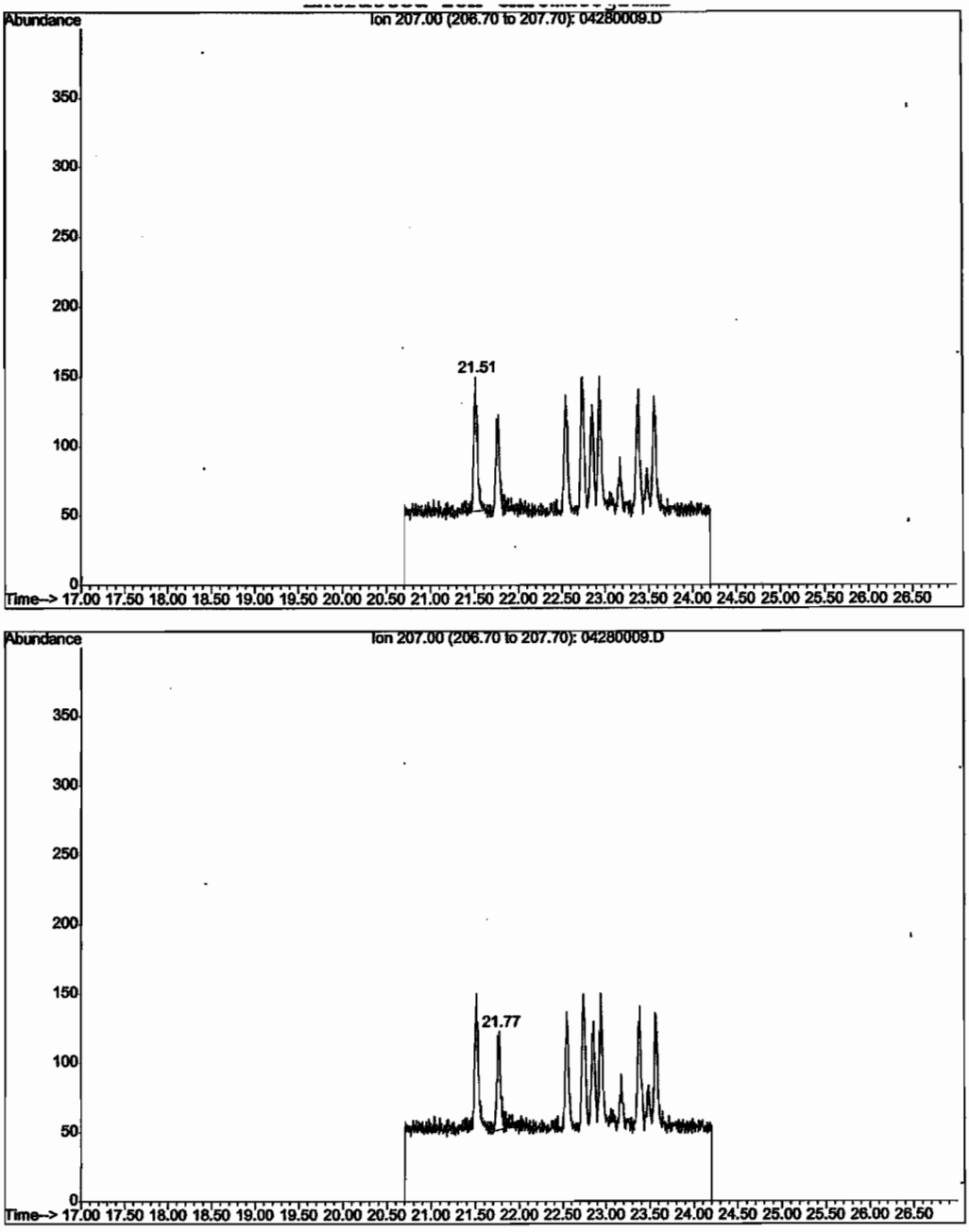
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 8917

FIGURE 1. (con't) Representative Chromatograms – Bracketing Standards

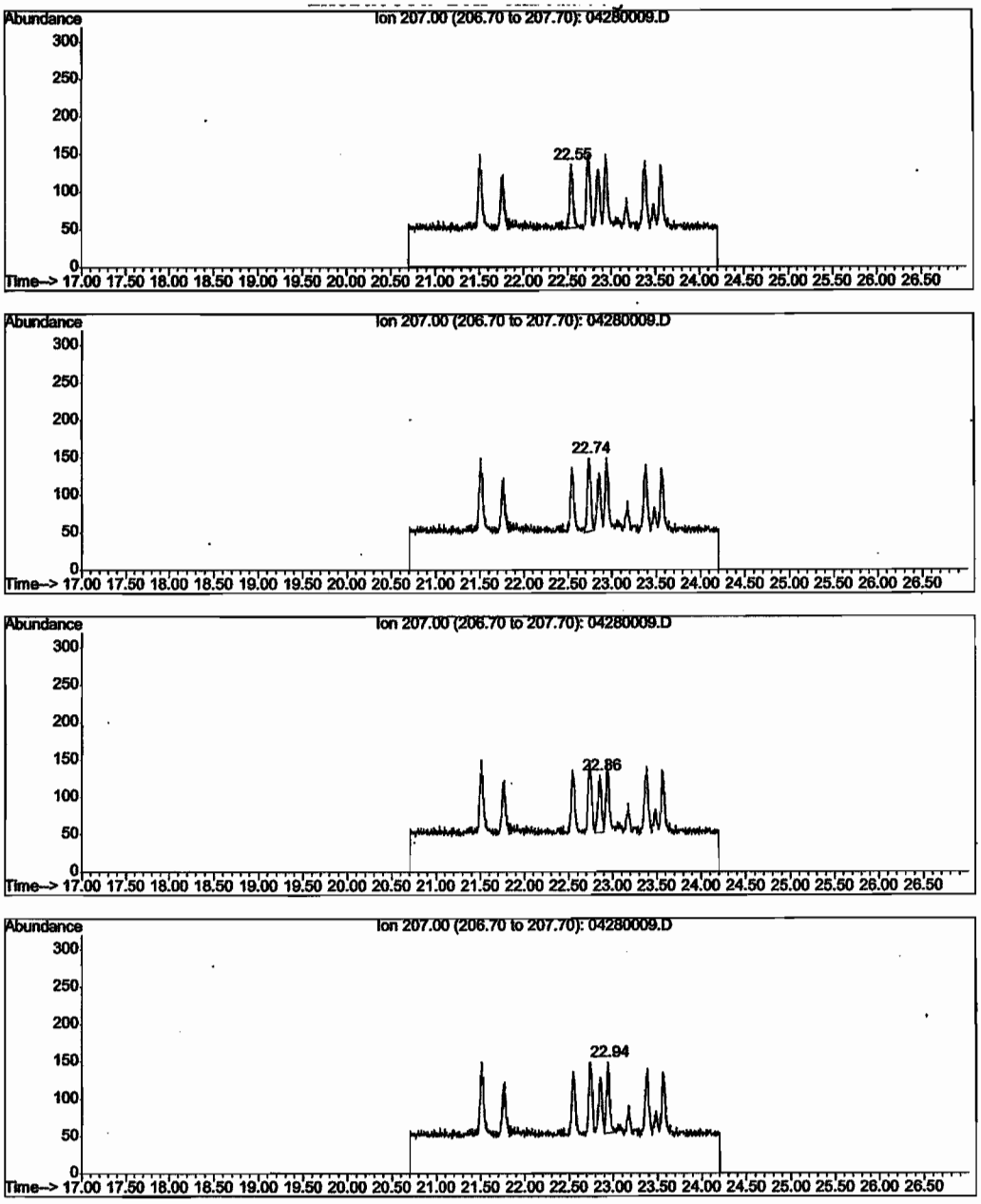
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Permethrin
10 ng/mL
Peak Response (area): 4519

FIGURE 1. (con't) Representative Chromatograms – Bracketing Standards

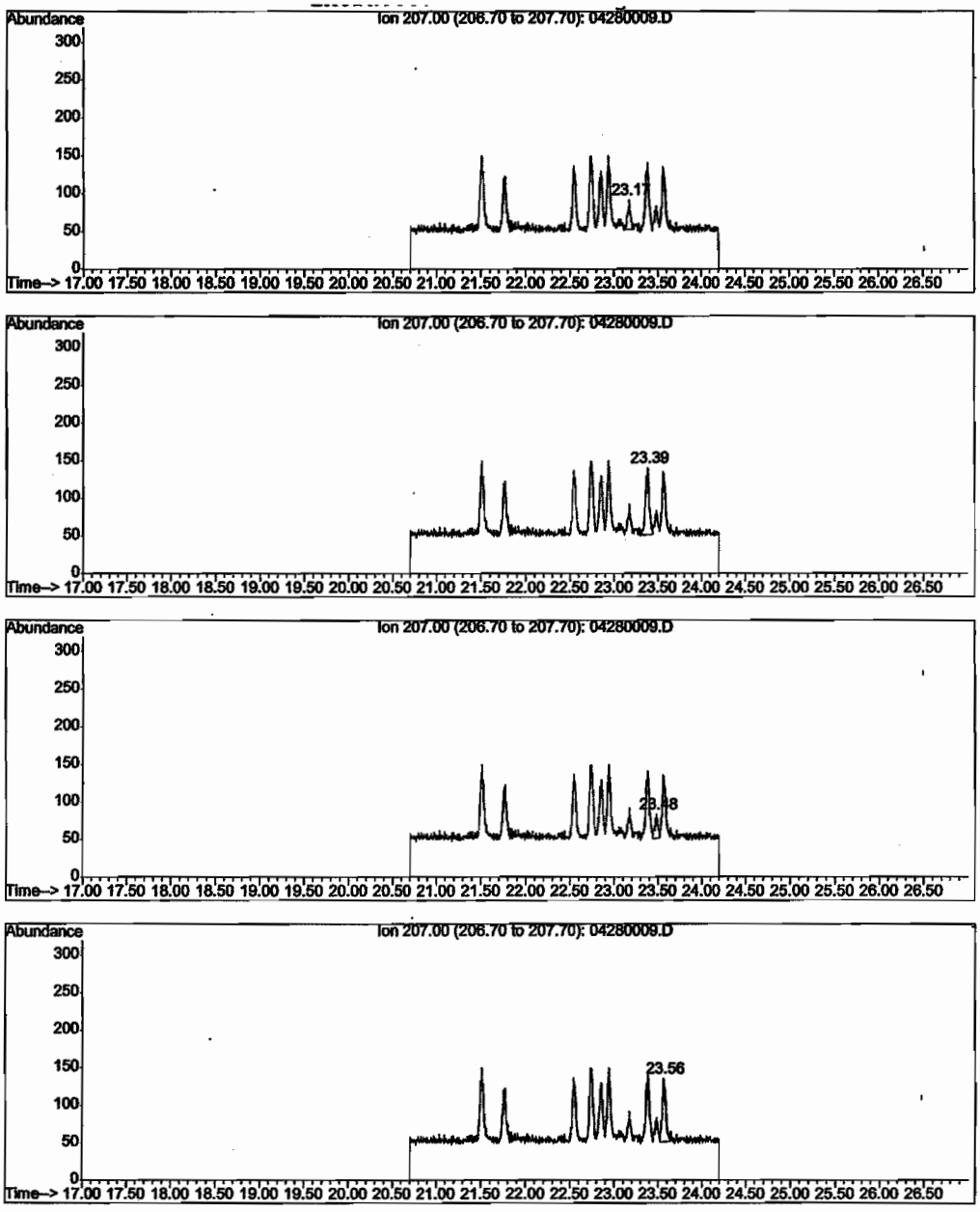
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Cyfluthrin
1.0 ng/mL
Peak Response (area): 9501

FIGURE 1. (con't) Representative Chromatograms – Bracketing Standards

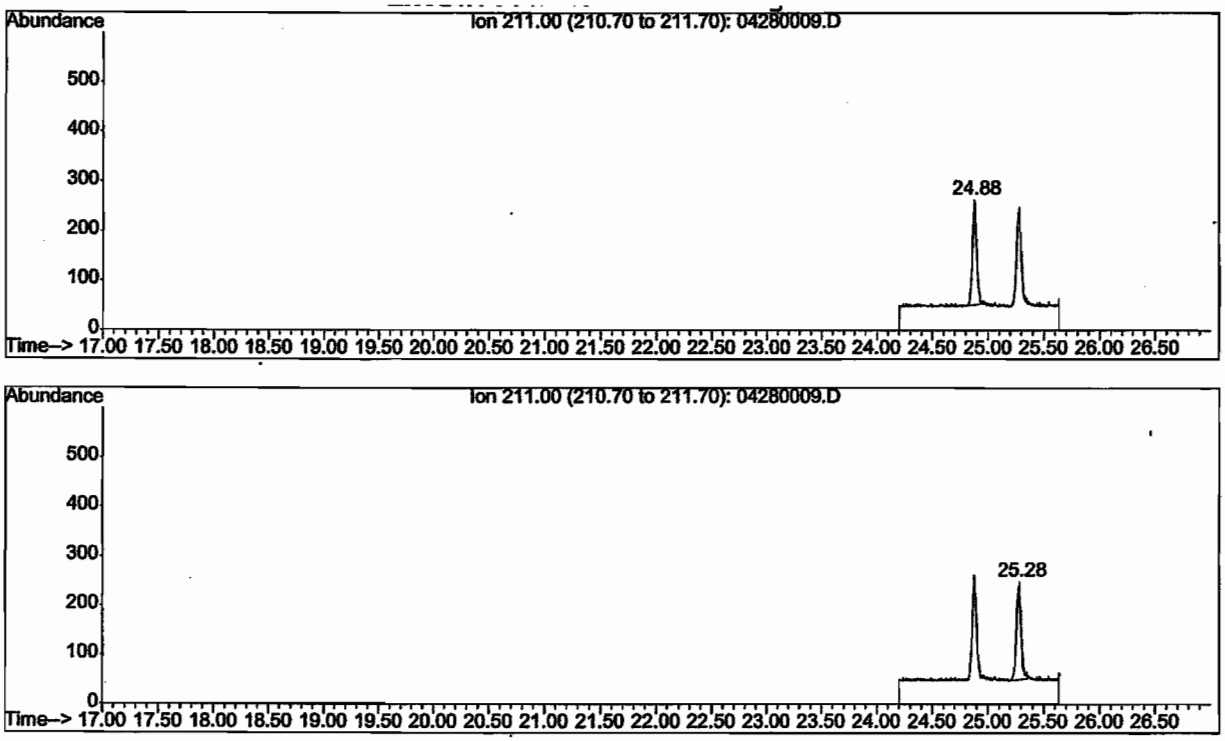
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Cypermethrin
1.0 ng/mL
Peak Response (area): 6464

FIGURE 1. (con't) Representative Chromatograms – Bracketing Standards

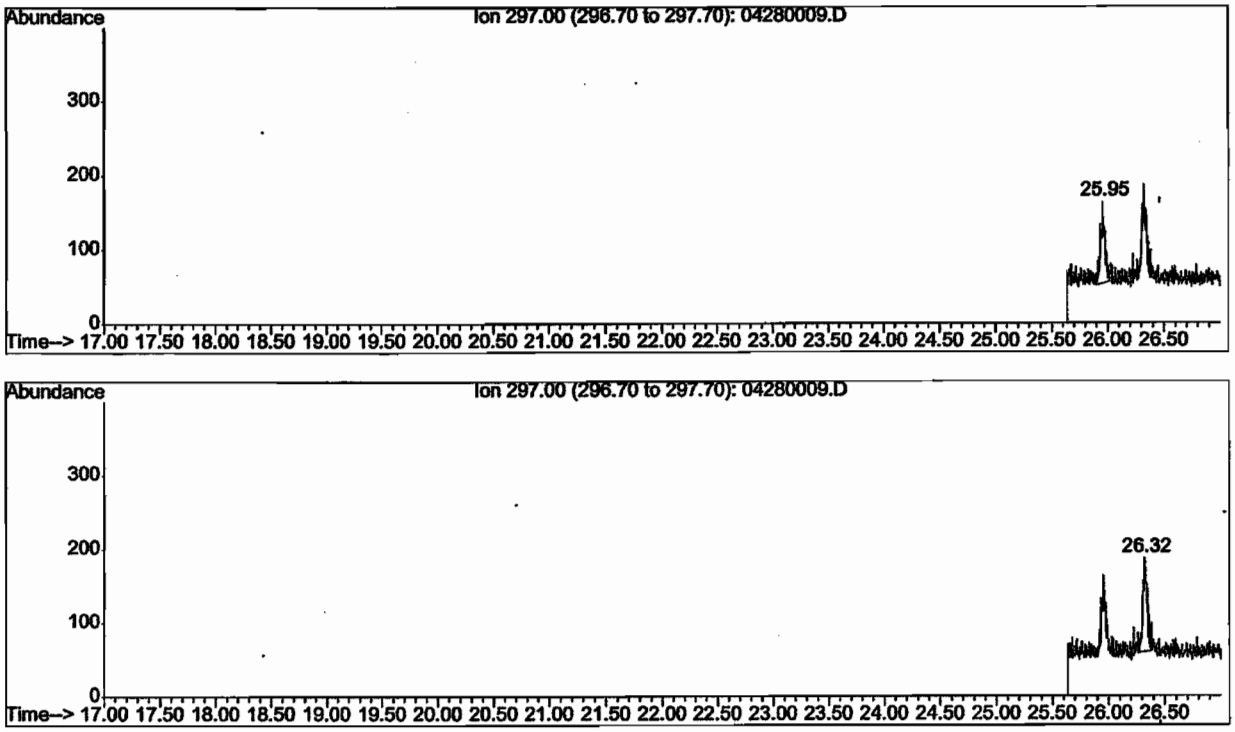
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Esfenvalerate
1.0 ng/mL
Peak Response (area): 11274

FIGURE 1. (con't) Representative Chromatograms – Bracketing Standards

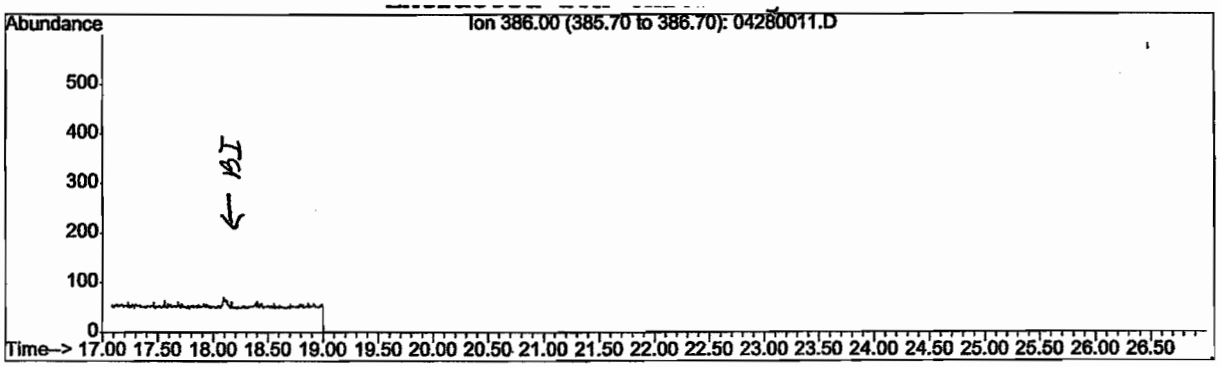
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



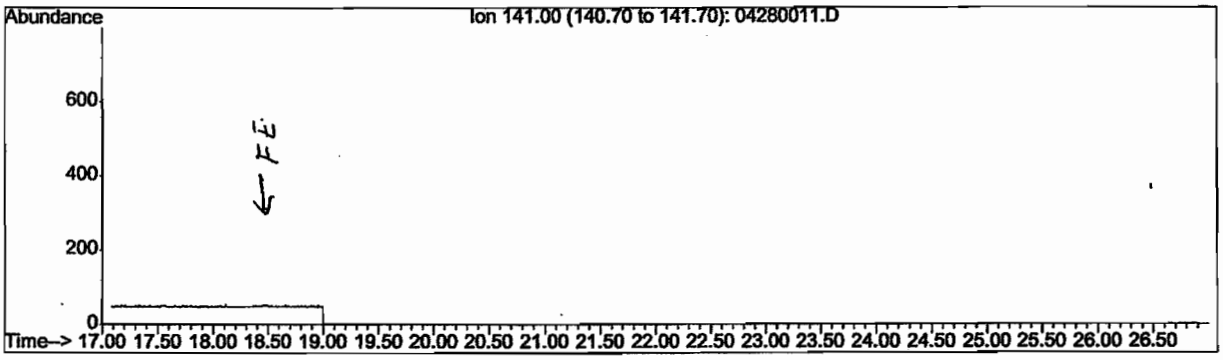
Deltamethrin
1.0 ng/mL
Peak Response (area): 5996

FIGURE 2. Representative Chromatograms - Fresh Water Sediment, Untreated Control

Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



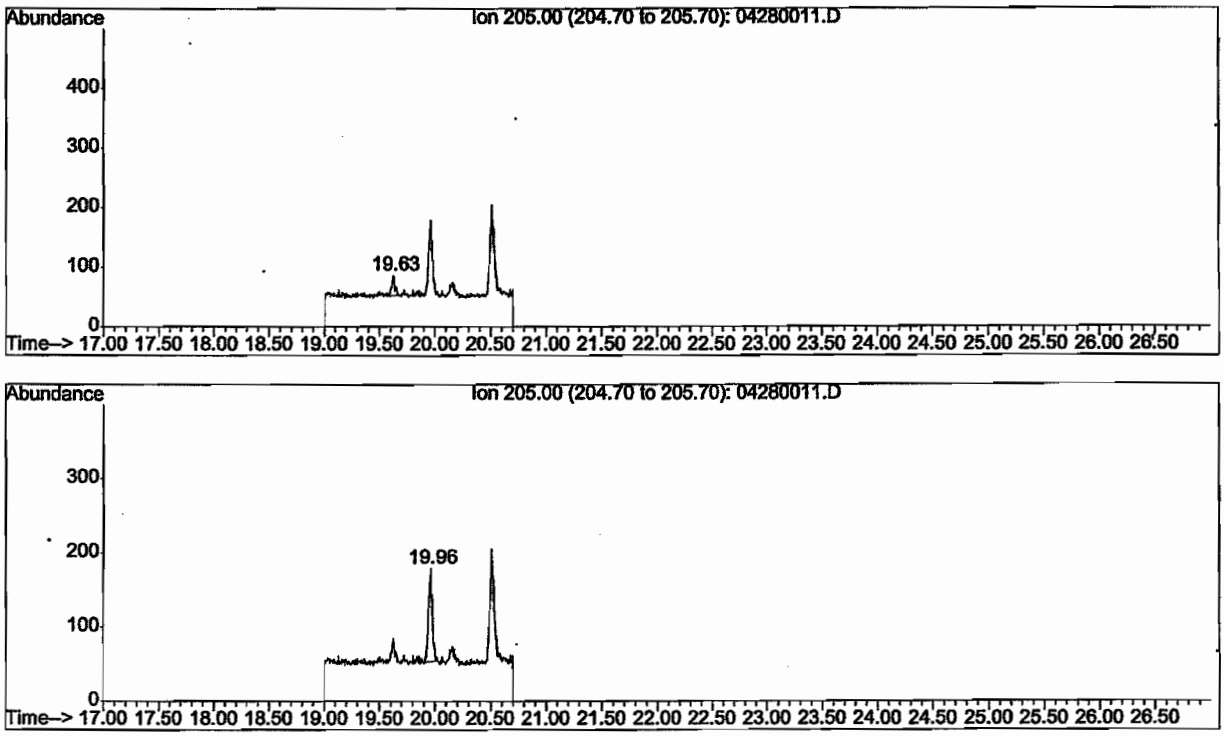
Bifenthrin
Peak Response (area): 0
Bifenthrin Reported: None Detected



Fenpropathrin
Peak Response (area): 0
Fenpropathrin Reported: None Detected

FIGURE 2. (con't) Representative Chromatograms – Fresh Water Sediment, Untreated Control

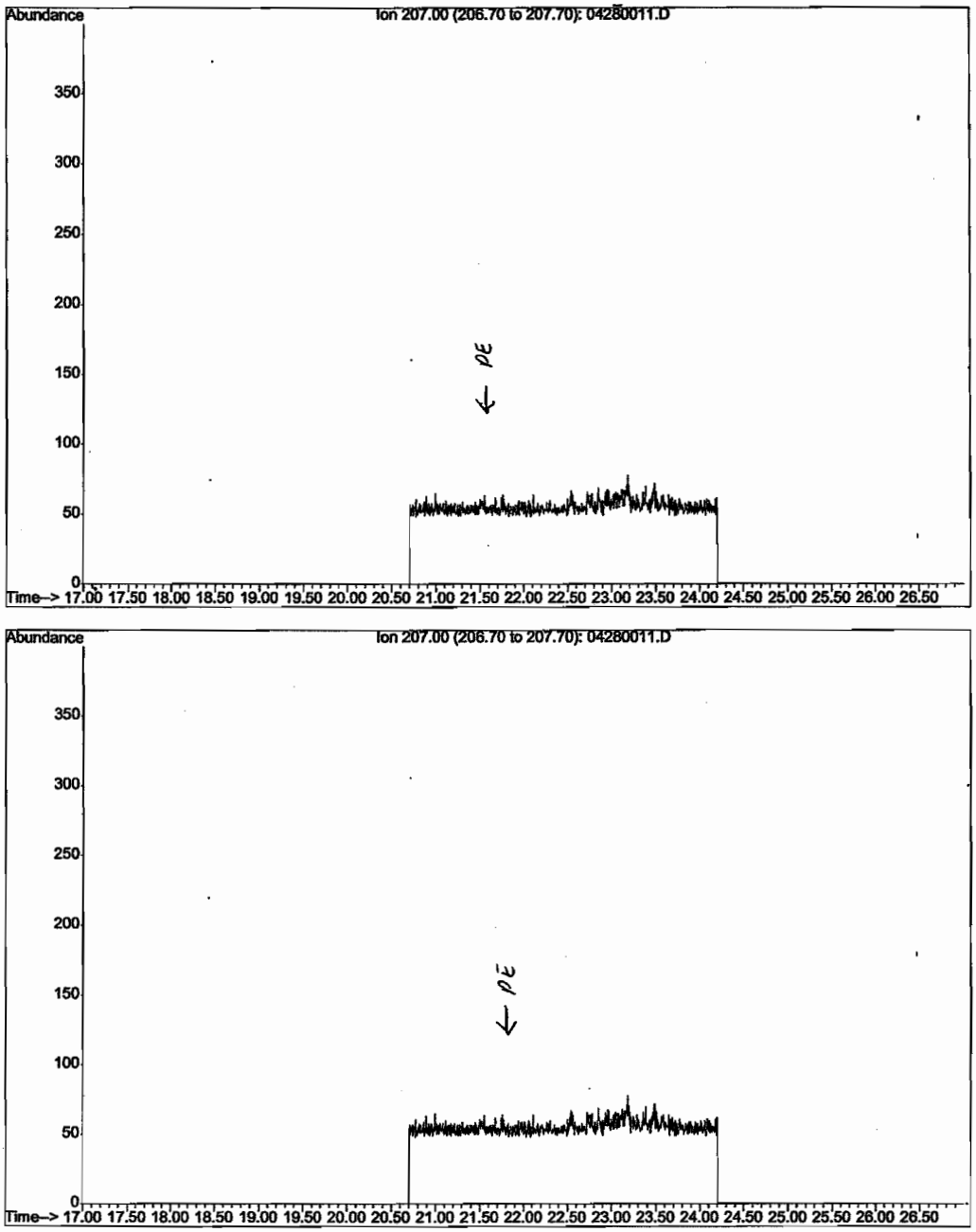
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



Lambda-cyhalothrin
Peak Response (area): 3484
Lambda-cyhalothrin Reported: <0.1 µg/kg

FIGURE 2. (con't) Representative Chromatograms – Fresh Water Sediment, Untreated Control

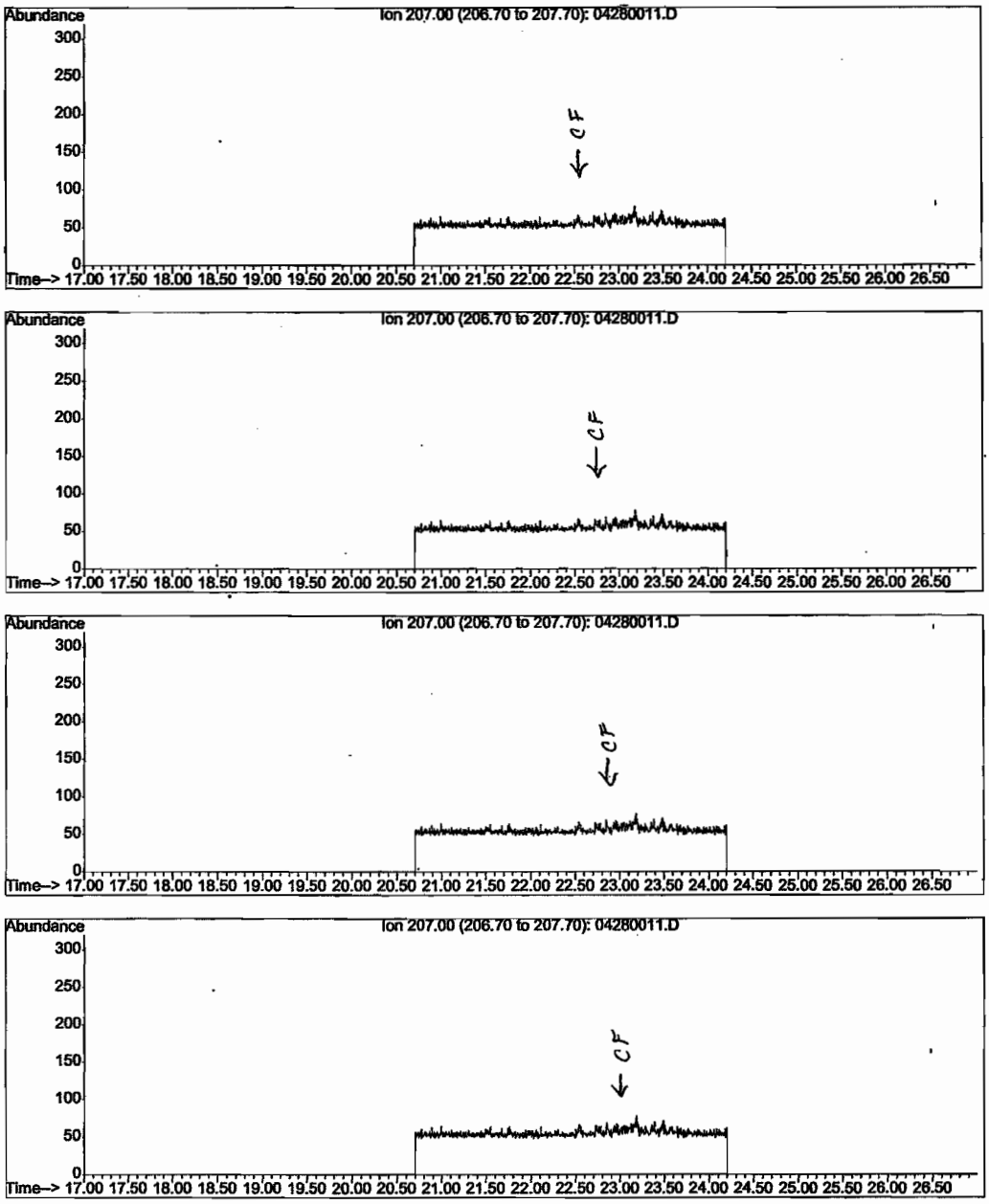
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



Permethrin
Peak Response (area): 0
Permethrin Reported: None Detected

FIGURE 2. (con't) Representative Chromatograms – Fresh Water Sediment, Untreated Control

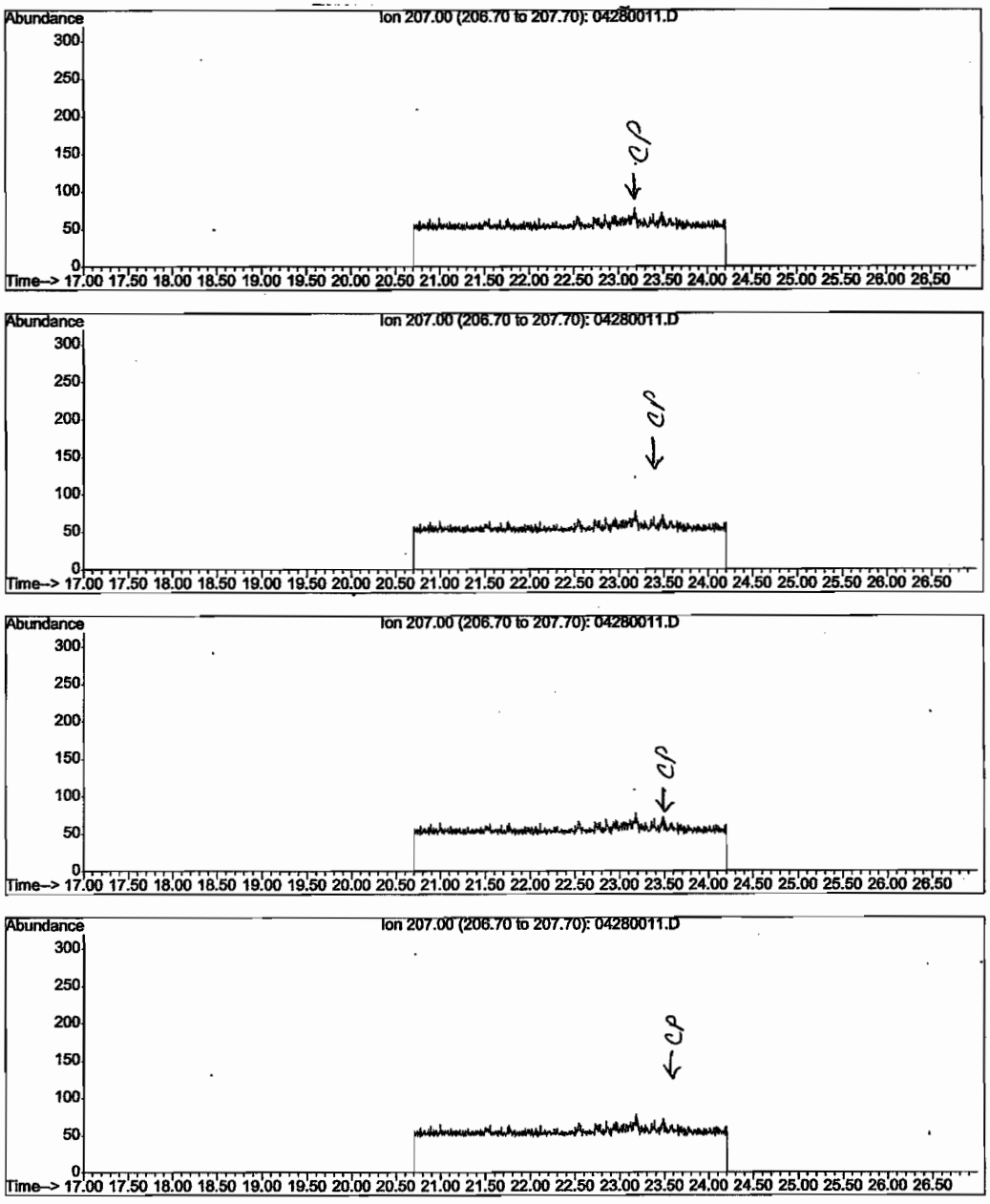
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



Cyfluthrin
Peak Response (area): 0
Cyfluthrin Reported: None Detected

FIGURE 2. (con't) Representative Chromatograms – Fresh Water Sediment, Untreated Control

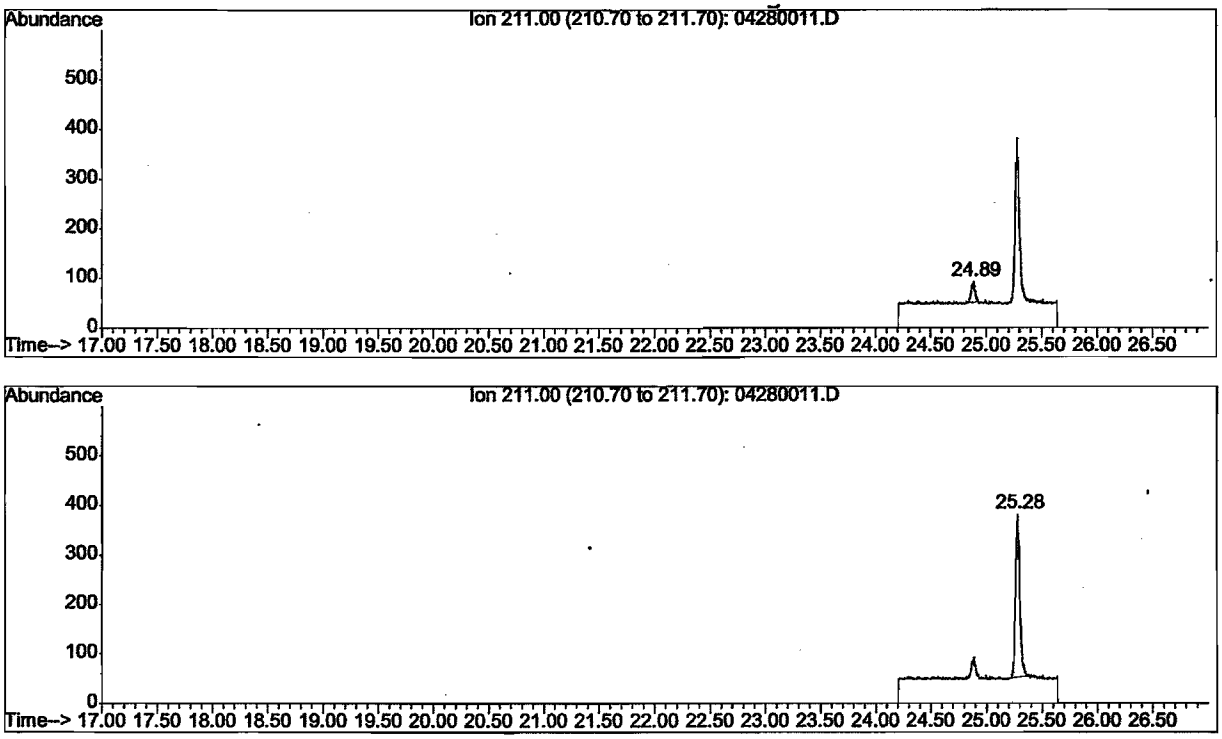
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



Cypermethrin
Peak Response (area): 0
Cypermethrin Reported: None Detected

FIGURE 2. (con't) **Representative Chromatograms – Fresh Water Sediment, Untreated Control**

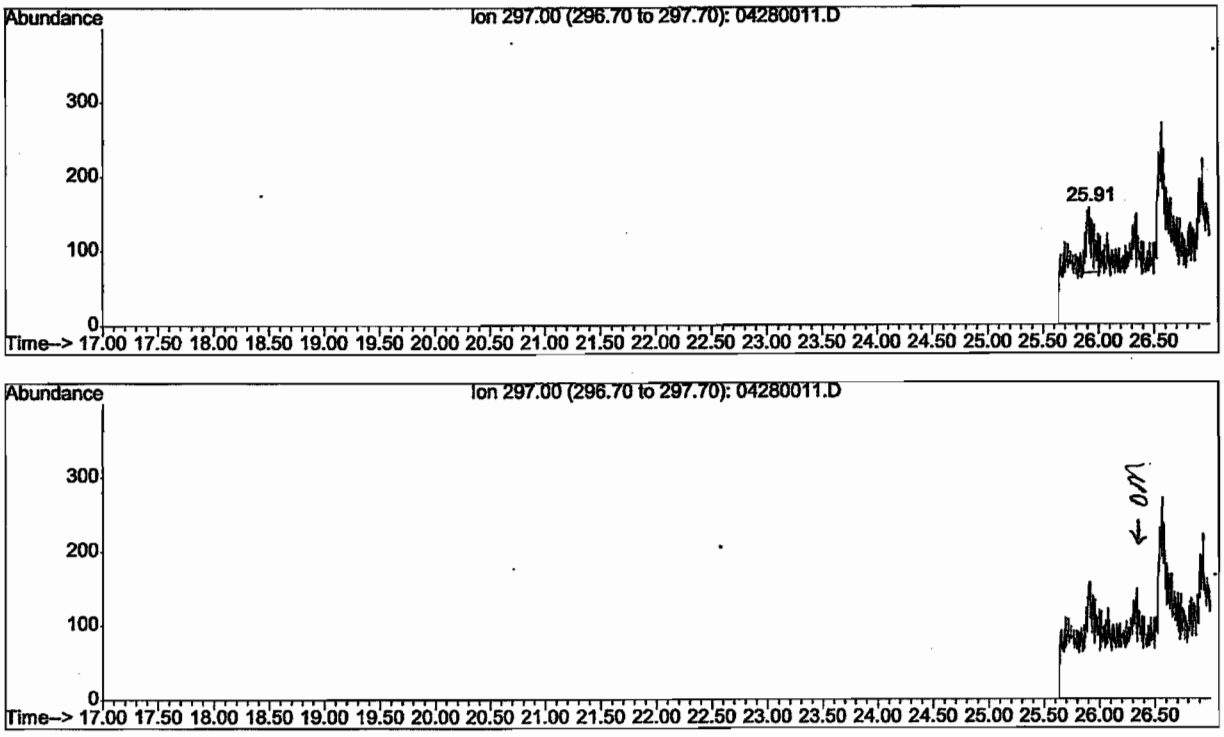
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



Esfenvalerate
Peak Response (area): 9601
Esfenvalerate Reported: <0.1 µg/kg

FIGURE 2. (con't) Representative Chromatograms – Fresh Water Sediment Untreated Control

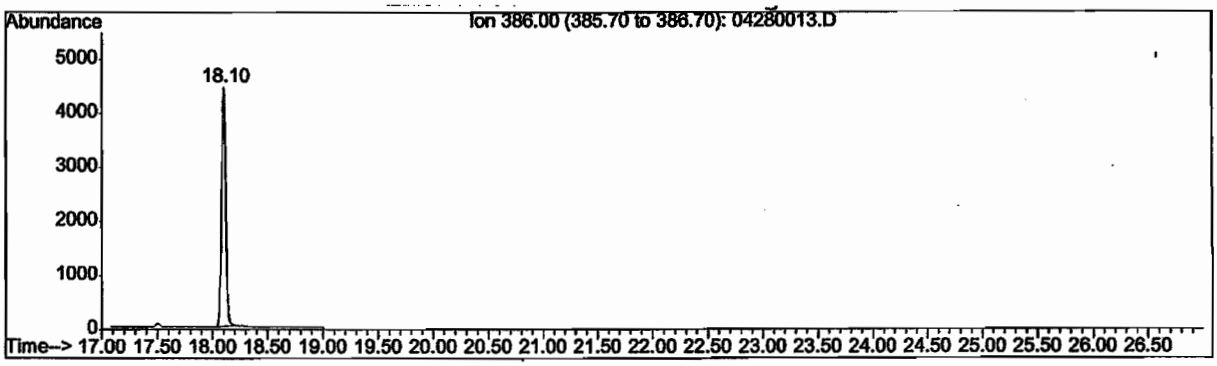
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



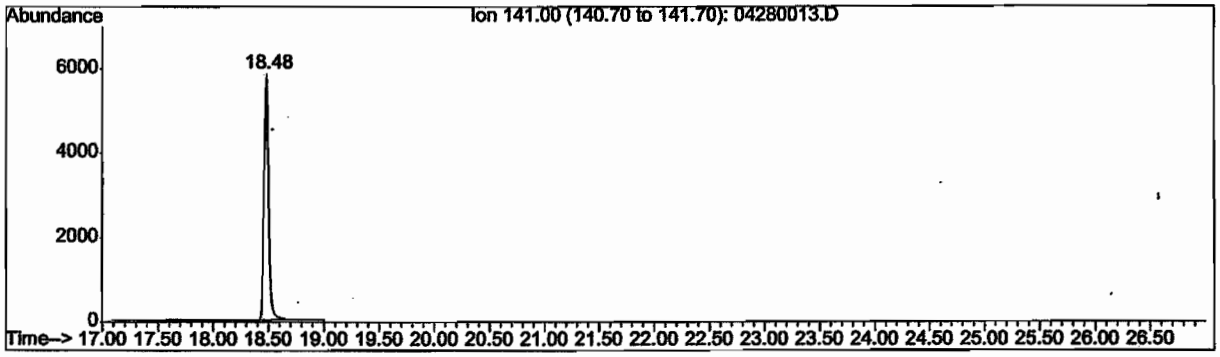
Deltamethrin
Peak Response (area): 3496
Deltamethrin Reported: <0.1 µg/kg

FIGURE 3. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



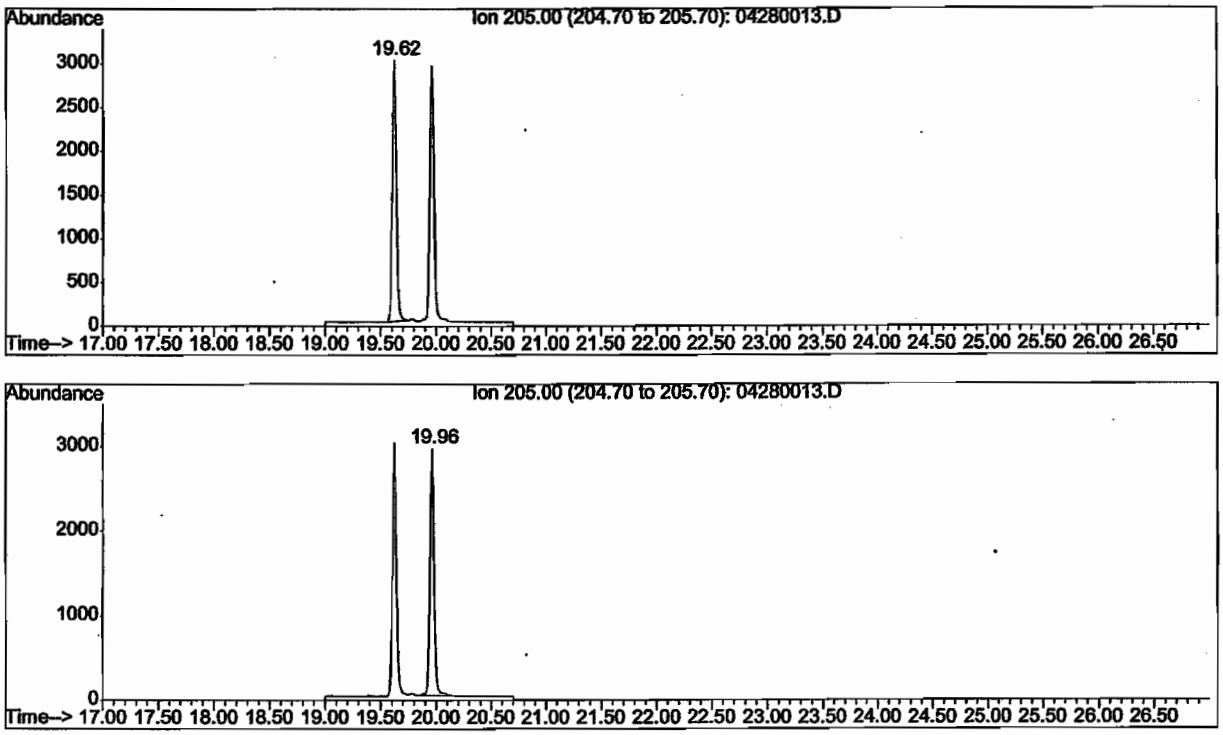
Bifenthrin
20 ng/mL
Peak Response (area): 115423



Fenpropathrin
20 ng/mL
Peak Response (area): 170037

FIGURE 3. (con't) Representative Chromatograms – Bracketing Standards

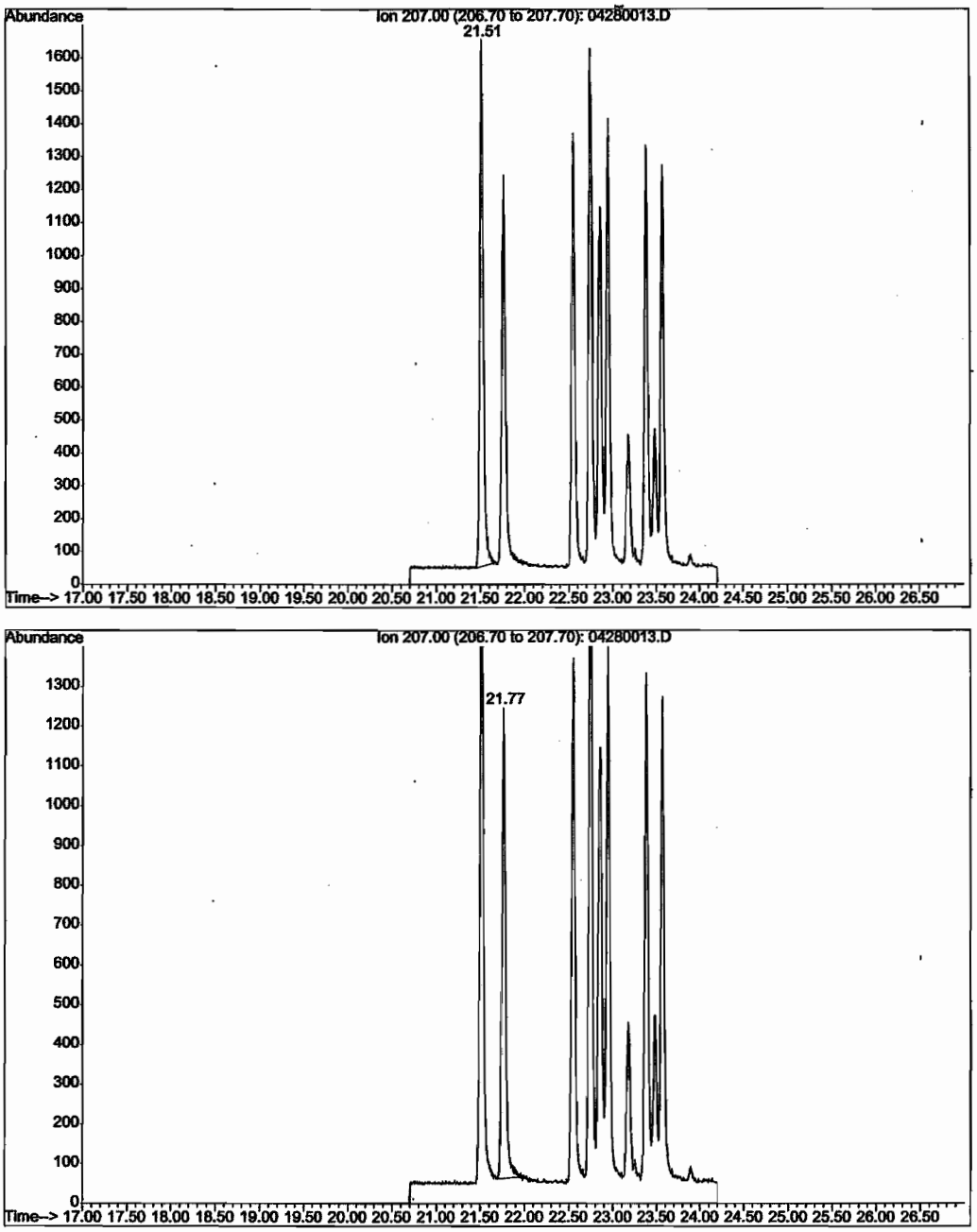
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 153812

FIGURE 3. (con't) Representative Chromatograms – Bracketing Standards

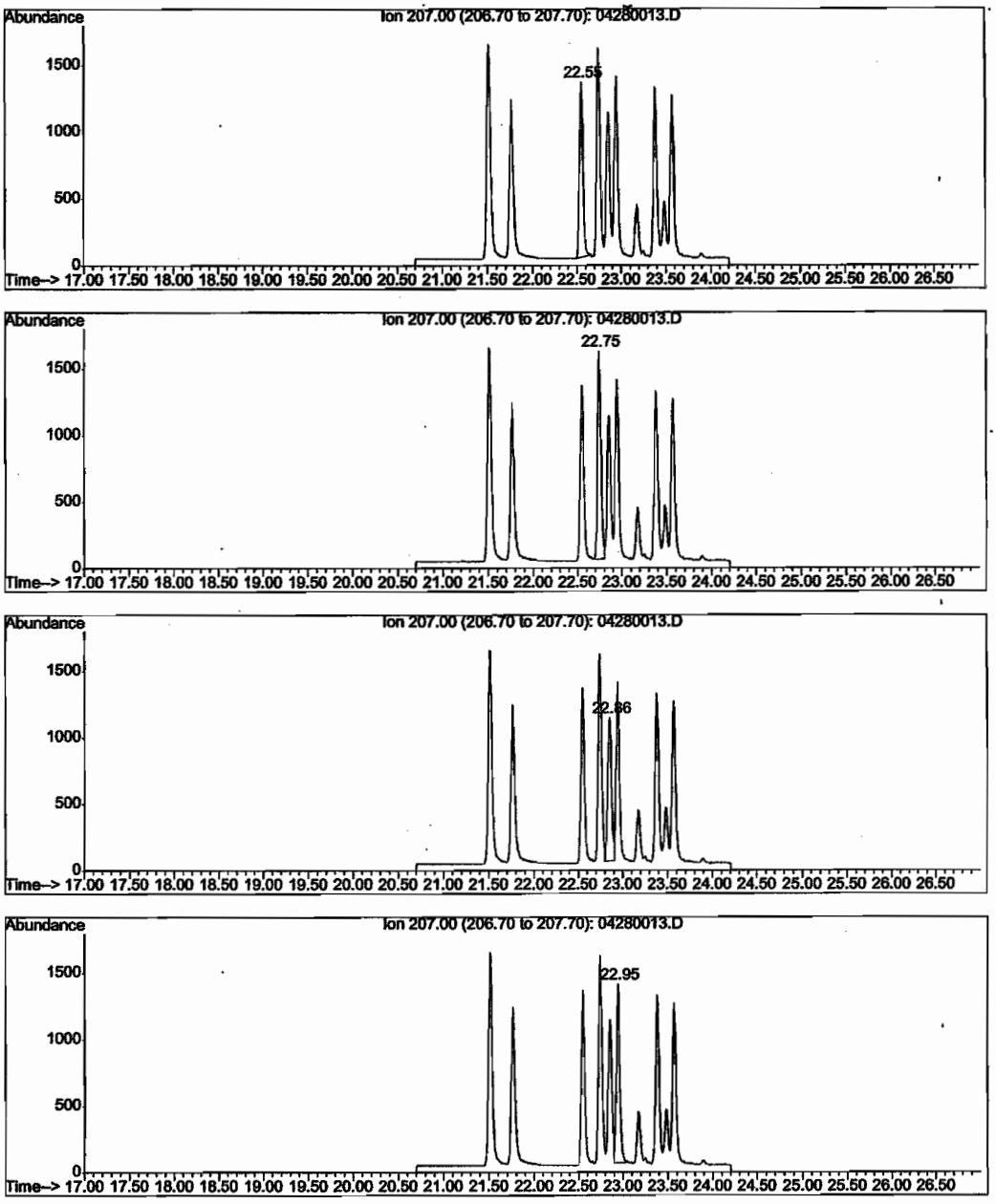
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Permethrin
200 ng/mL
Peak Response (area): 80094

FIGURE 3. (con't) Representative Chromatograms – Bracketing Standards

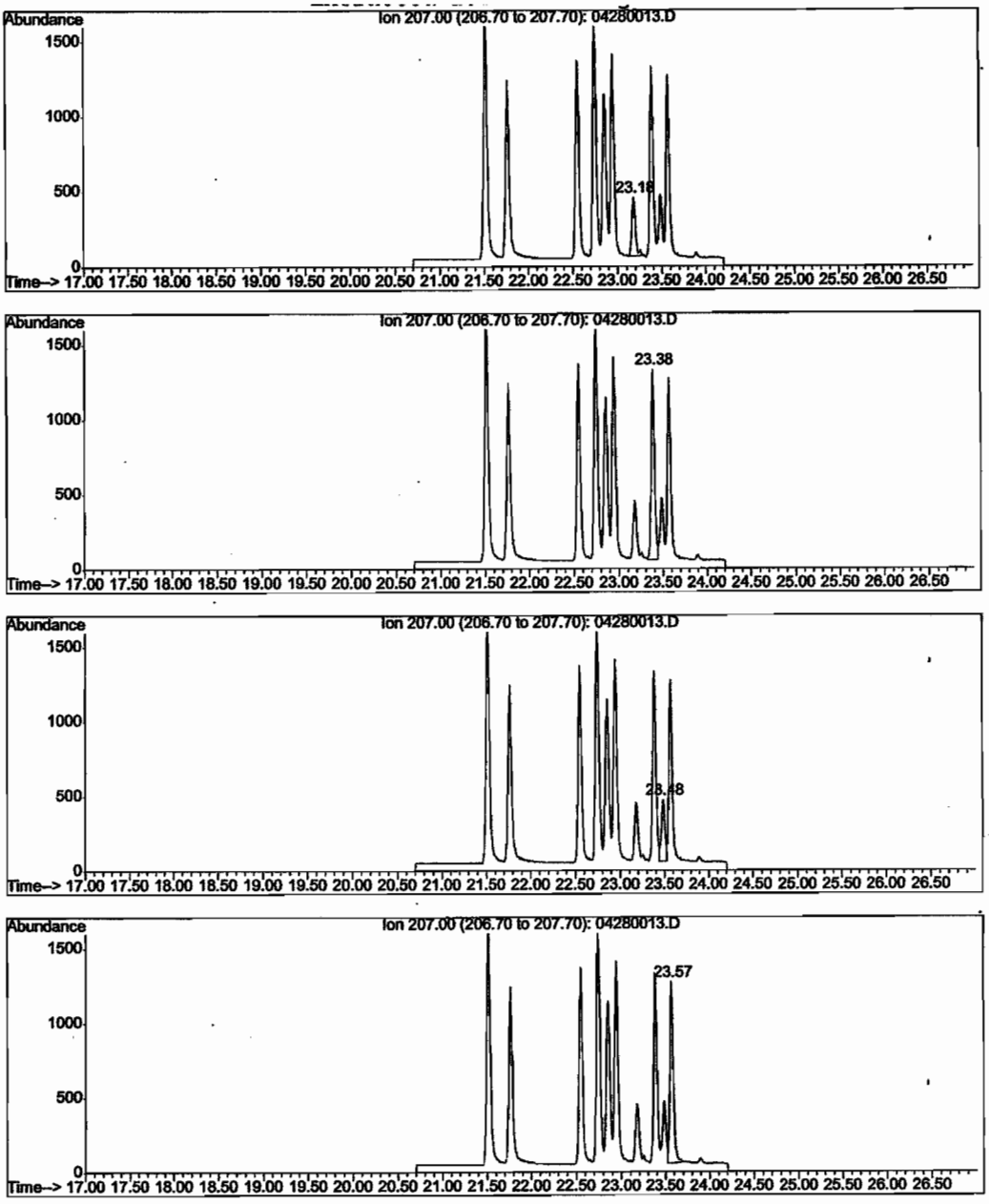
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Cyfluthrin
20 ng/mL
Peak Response (area): 146068

FIGURE 3. (con't) Representative Chromatograms – Bracketing Standards

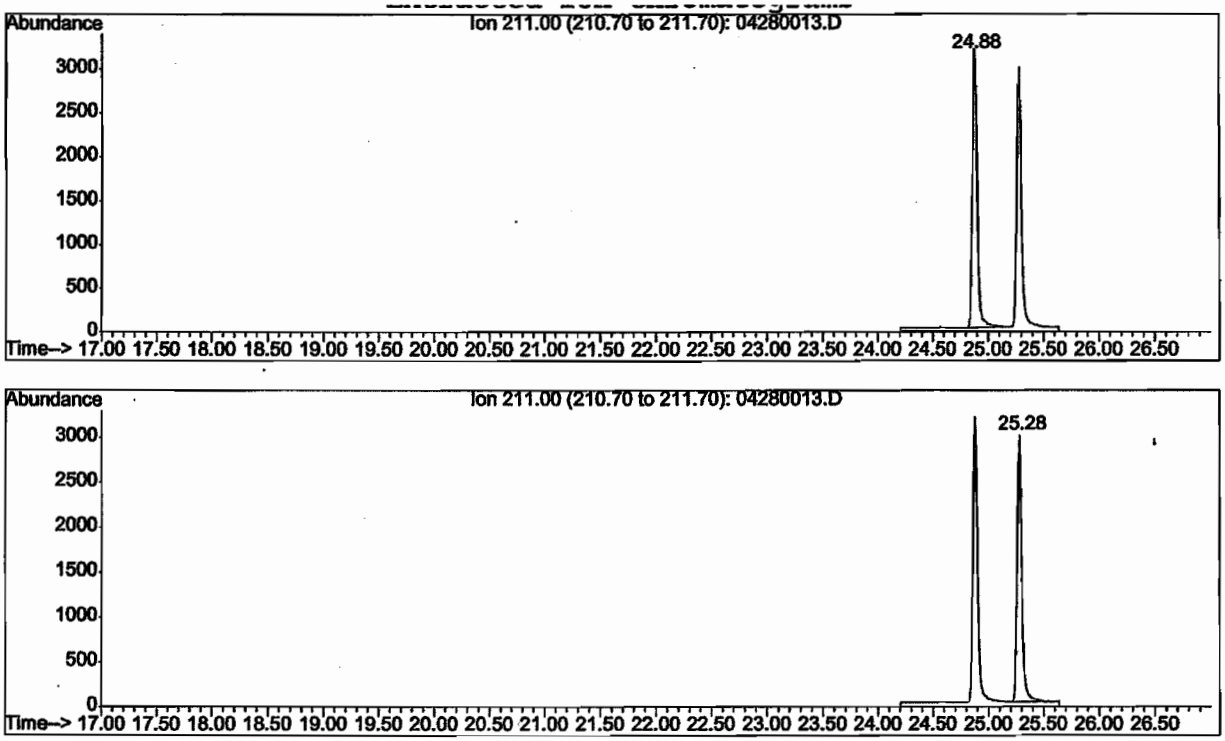
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Cypermethrin
20 ng/mL
Peak Response (area): 90994

FIGURE 3. (con't) Representative Chromatograms – Bracketing Standards

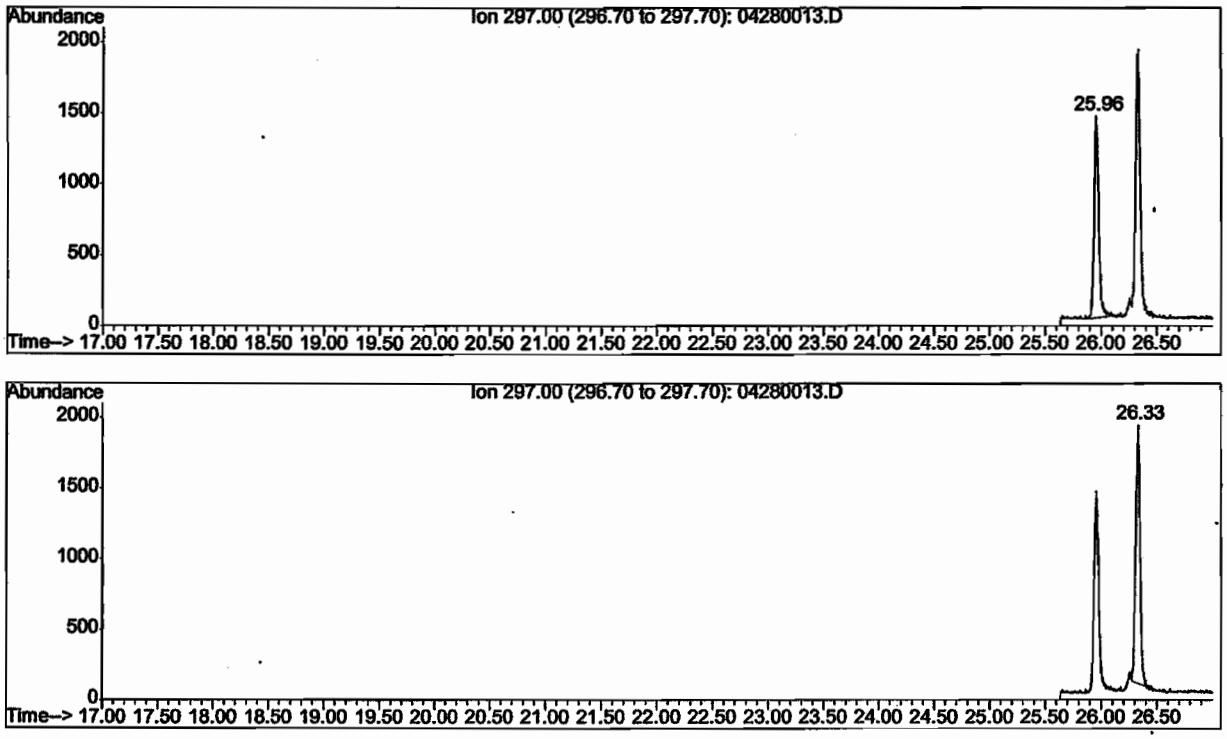
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Esfenvalerate
20 ng/mL
Peak Response (area): 185356

FIGURE 3. (con't) Representative Chromatograms – Bracketing Standards

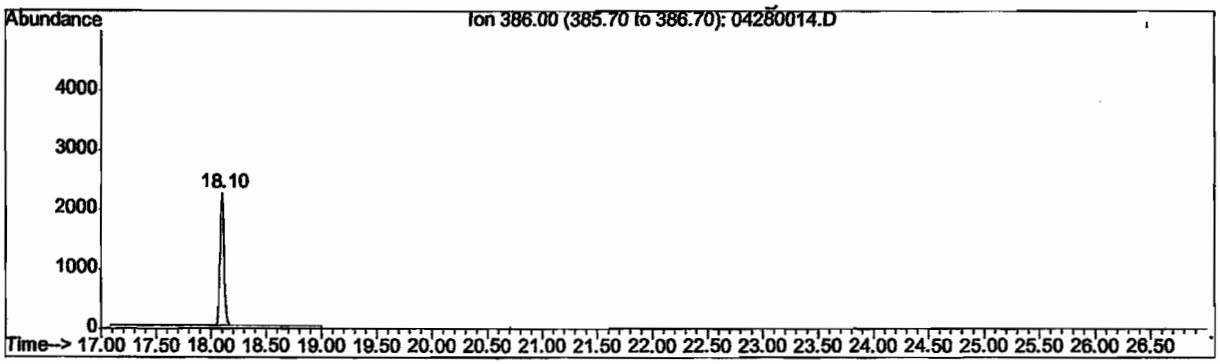
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



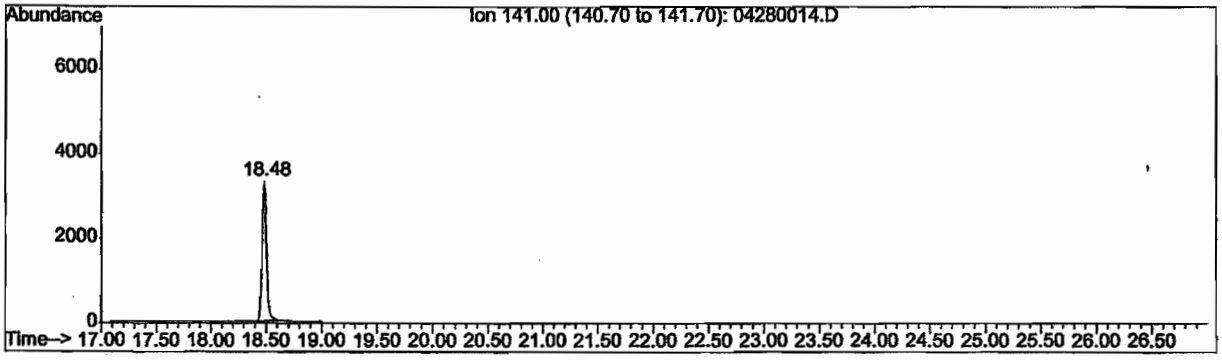
Deltamethrin
20 ng/mL
Peak Response (area): 90699

FIGURE 4. Representative Chromatograms - Fresh Water Sediment, Fortified Control (10×LOQ)

Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #2



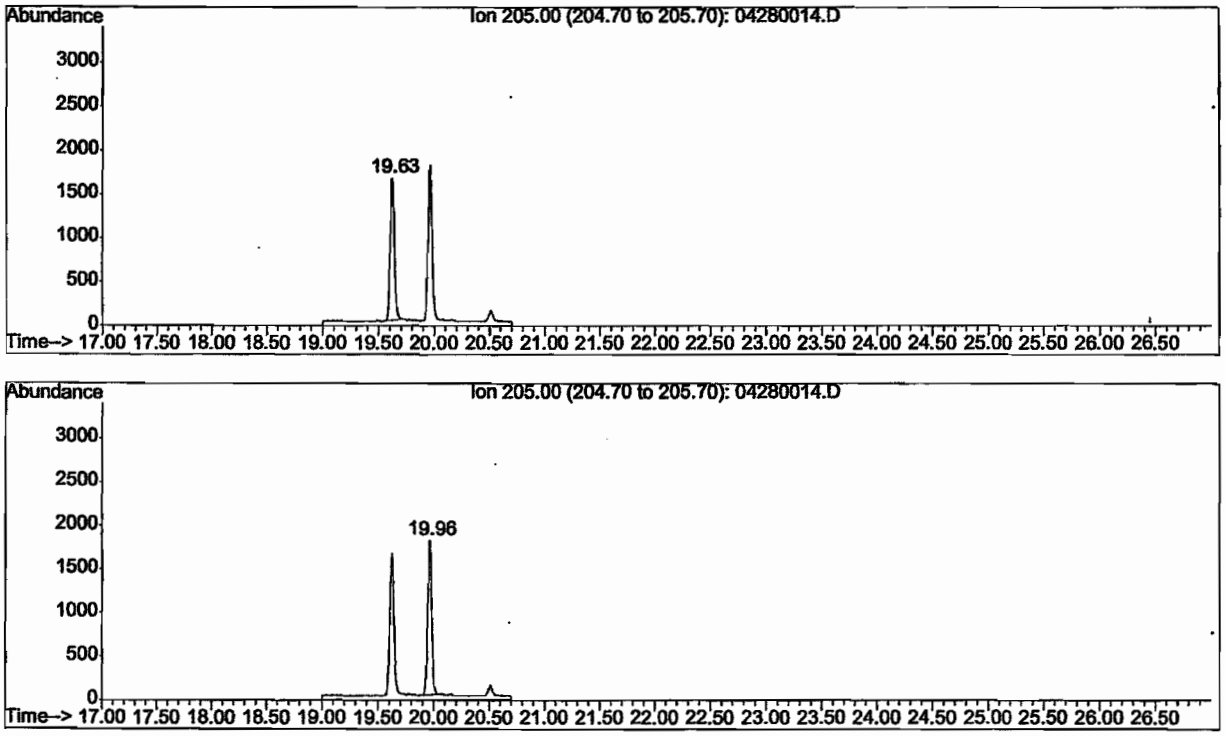
Bifenthrin
Peak Response (area): 57760
Bifenthrin Reported Recovery: 100%



Fenpropathrin
Peak Response (area): 93570
Fenpropathrin Reported Recovery: 110%

FIGURE 4. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (10×LOQ)

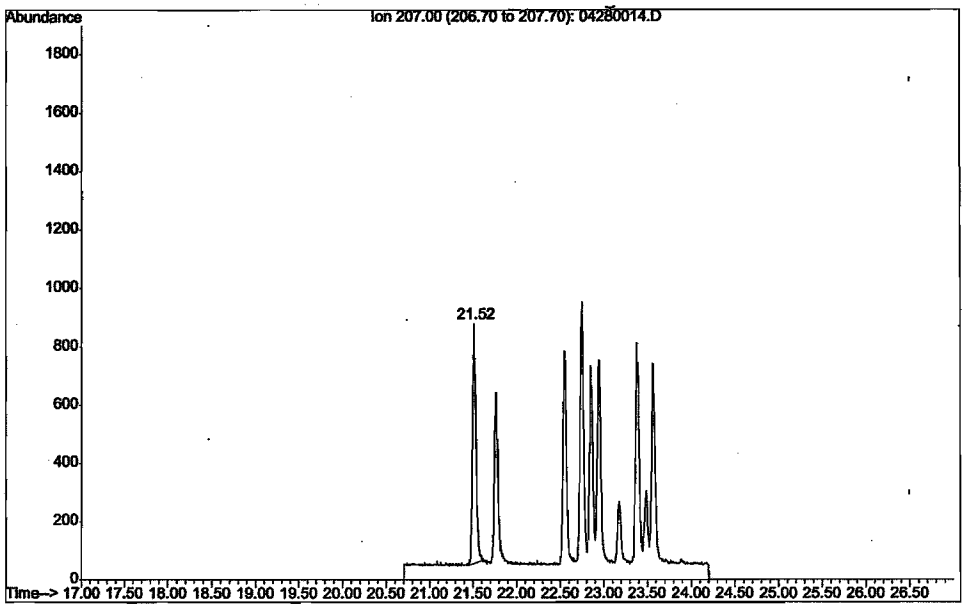
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #2

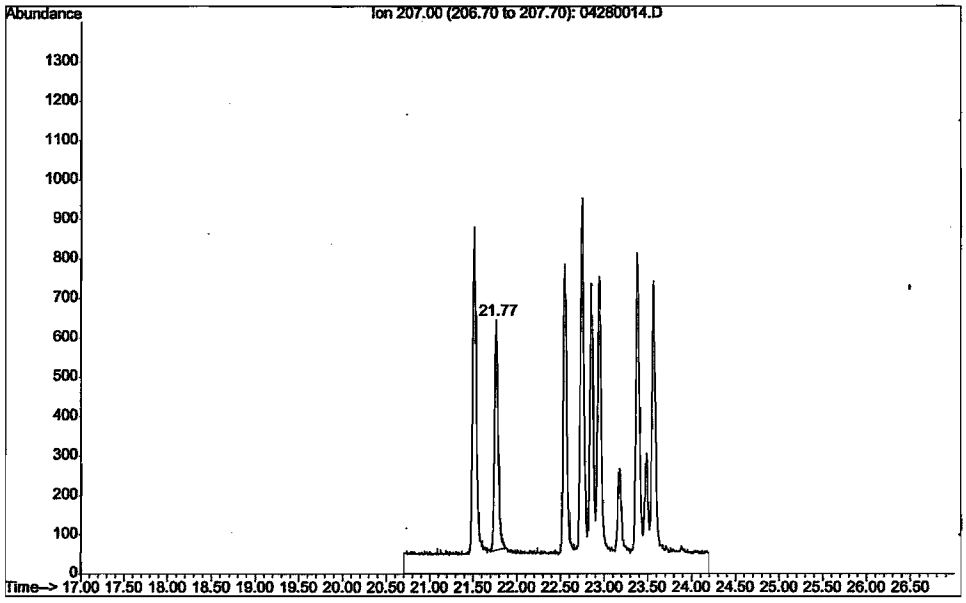


Lambda-cyhalothrin
Peak Response (area): 85102
Lambda-cyhalothrin Reported Recovery: 106%

FIGURE 4. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (10×LOQ)

Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #2

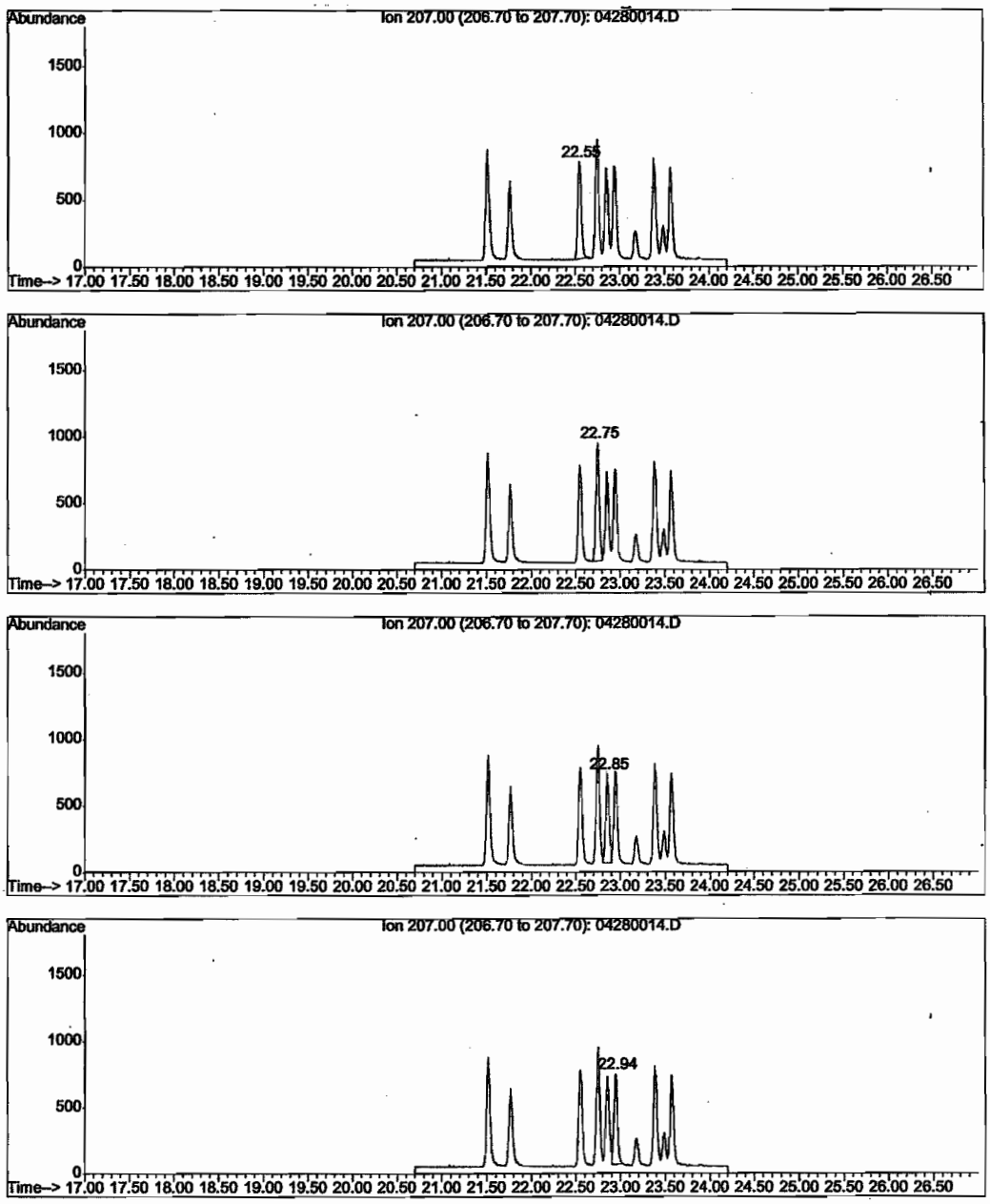




Permethrin
Peak Response (area): 39075
Permethrin Reported Recovery: 97%

FIGURE 4. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (10×LOQ)

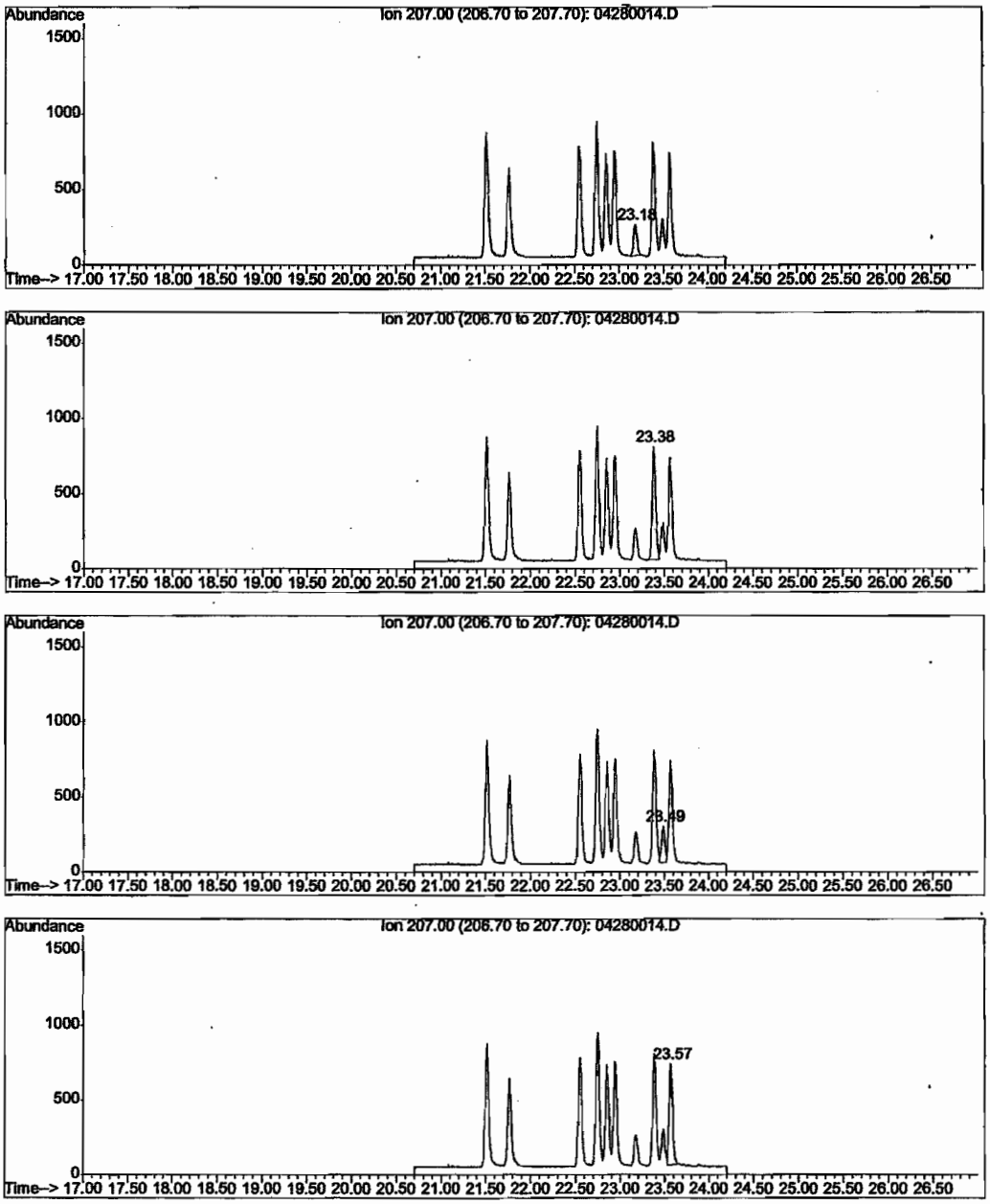
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #2



Cyfluthrin
Peak Response (area): 80574
Cyfluthrin Reported Recovery: 111%

FIGURE 4. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (10×LOQ)

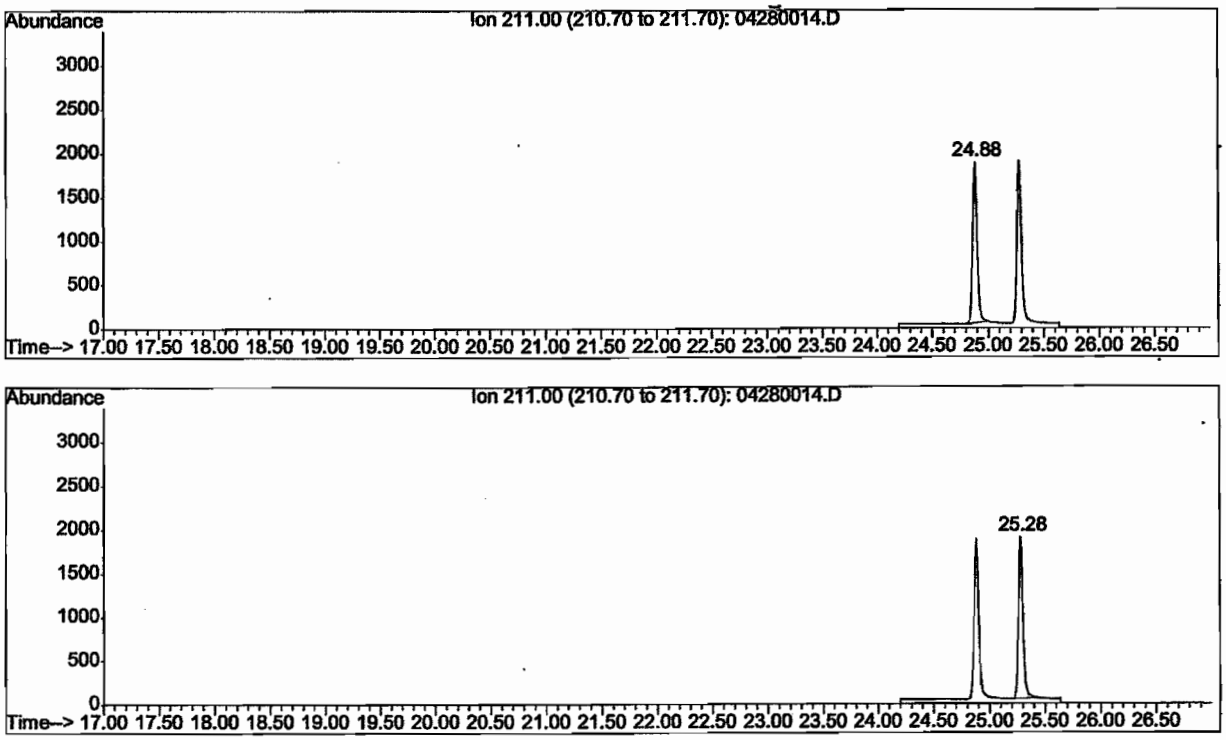
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #2



Cypermethrin
Peak Response (area): 50676
Cypermethrin Reported Recovery: 111%

FIGURE 4. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (10×LOQ)

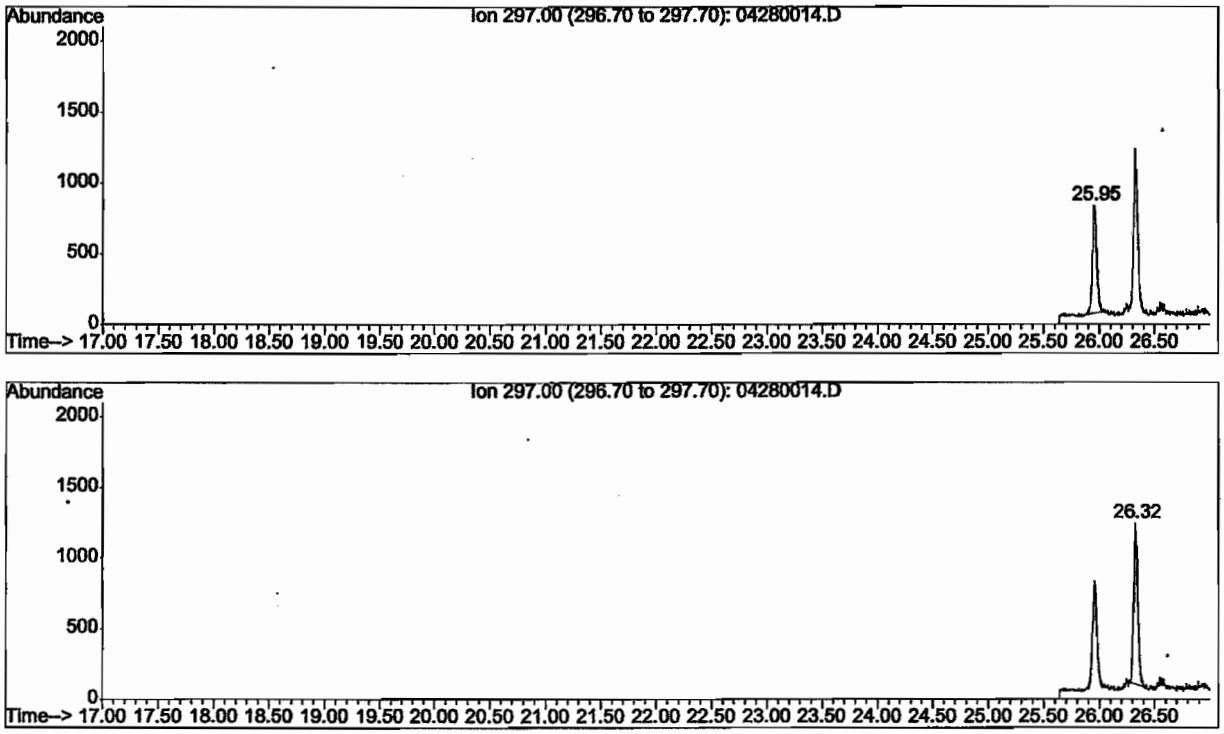
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #2



Esfenvalerate
Peak Response (area): 102955
Esfenvalerate Reported Recovery: 105%

FIGURE 4. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (10×LOQ)

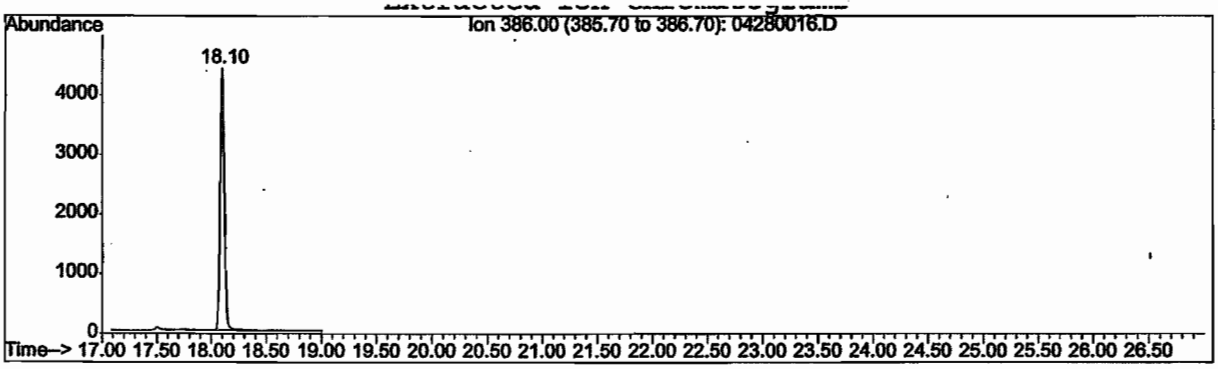
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #2



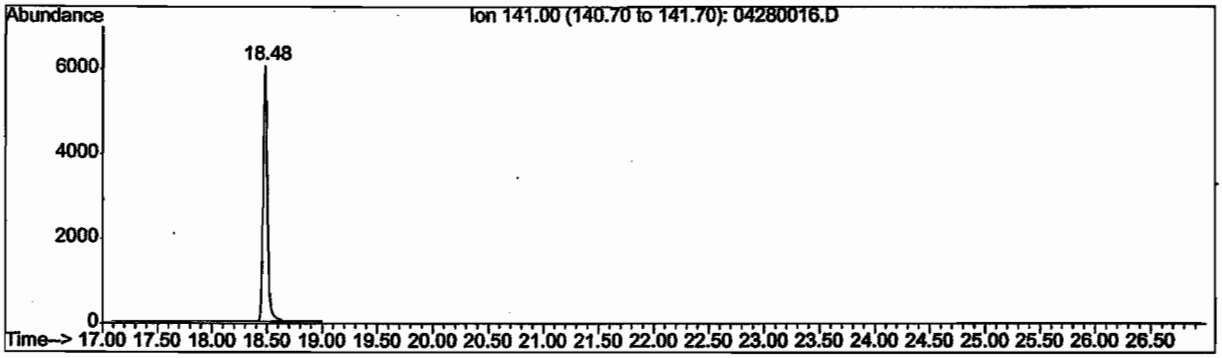
Deltamethrin
Peak Response (area): 48891
Deltamethrin Reported Recovery: 104%

FIGURE 5. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



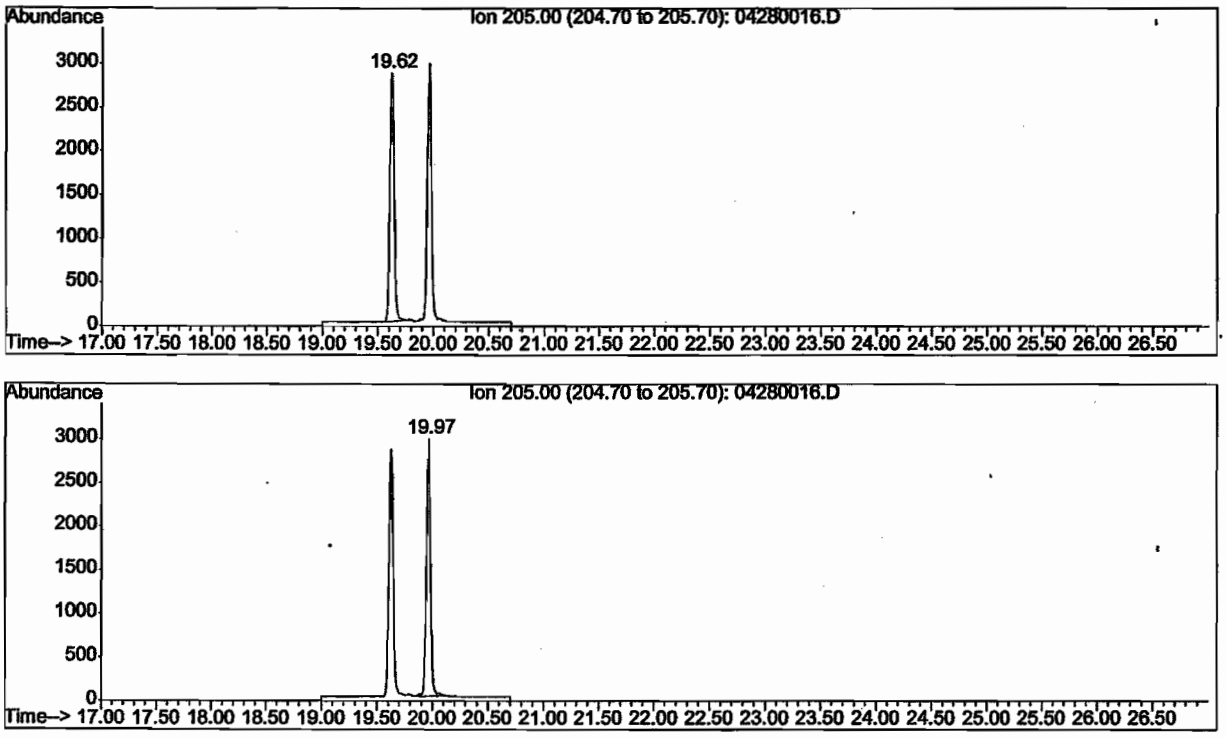
Bifenthrin
20 ng/mL
Peak Response (area): 116412



Fenpropathrin
20 ng/mL
Peak Response (area): 169884

FIGURE 5. (con't) Representative Chromatograms – Bracketing Standards

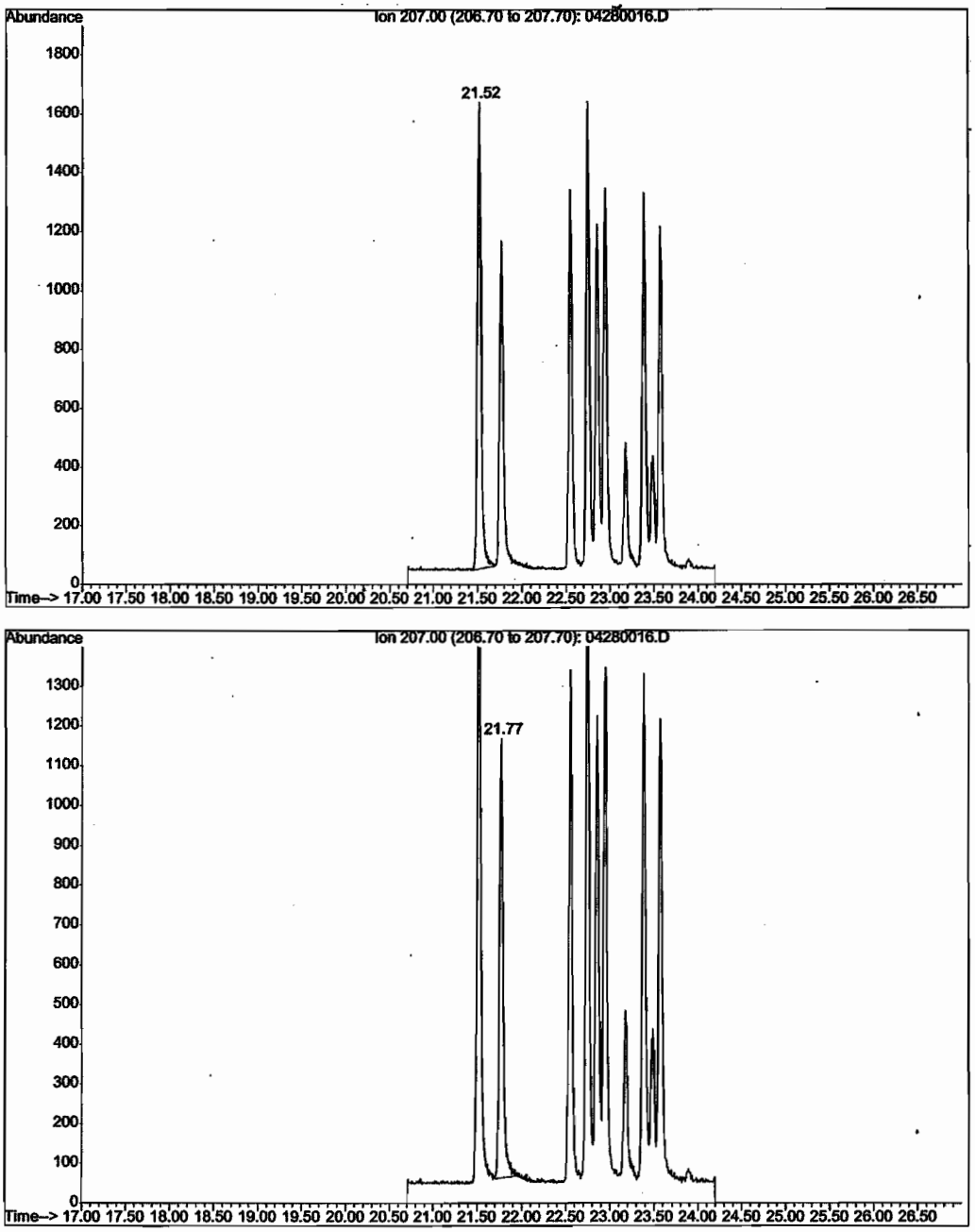
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 154546

FIGURE 5. (con't) Representative Chromatograms – Bracketing Standards

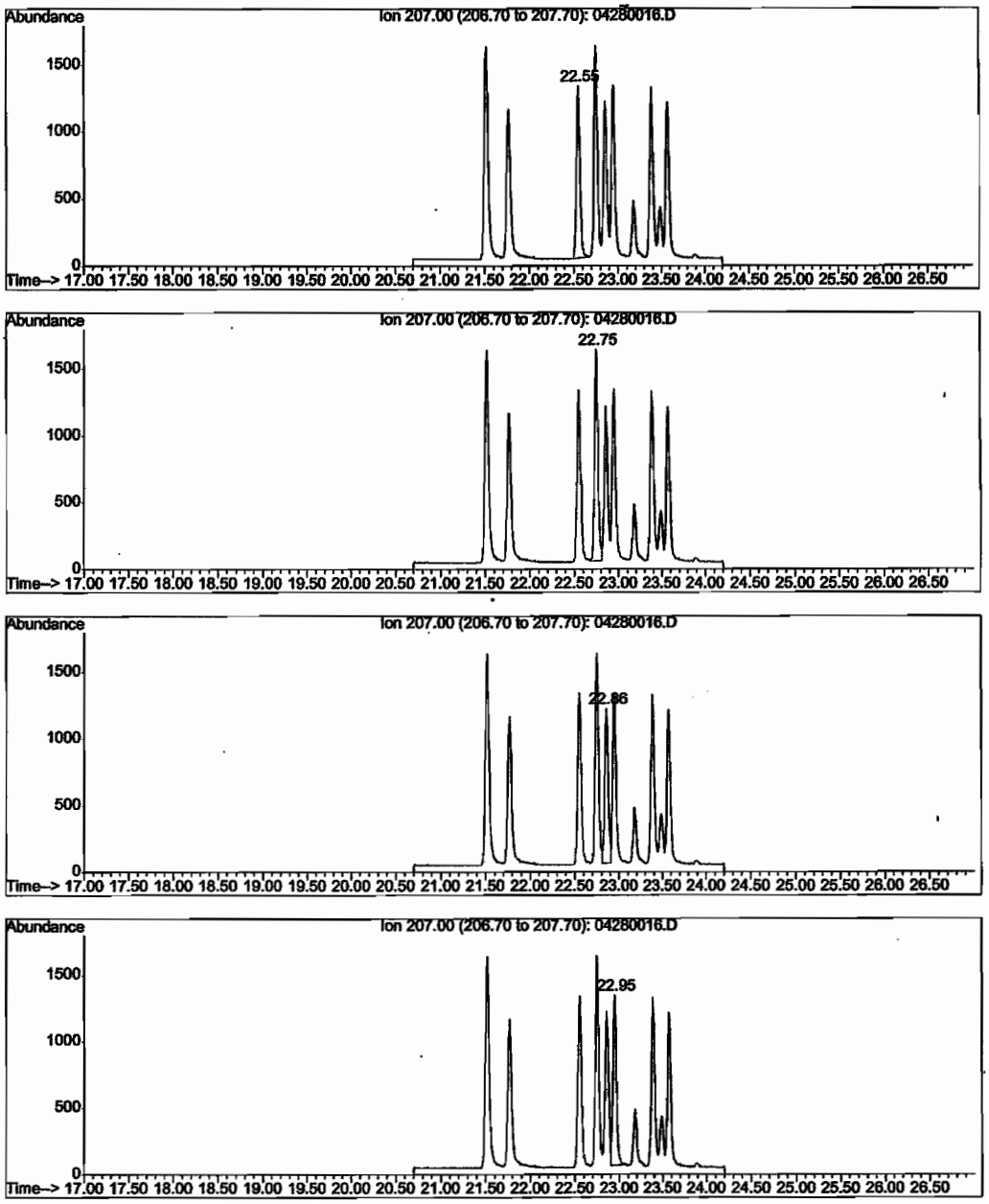
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Permethrin
200 ng/mL
Peak Response (area): 80924

FIGURE 5. (con't) Representative Chromatograms – Bracketing Standards

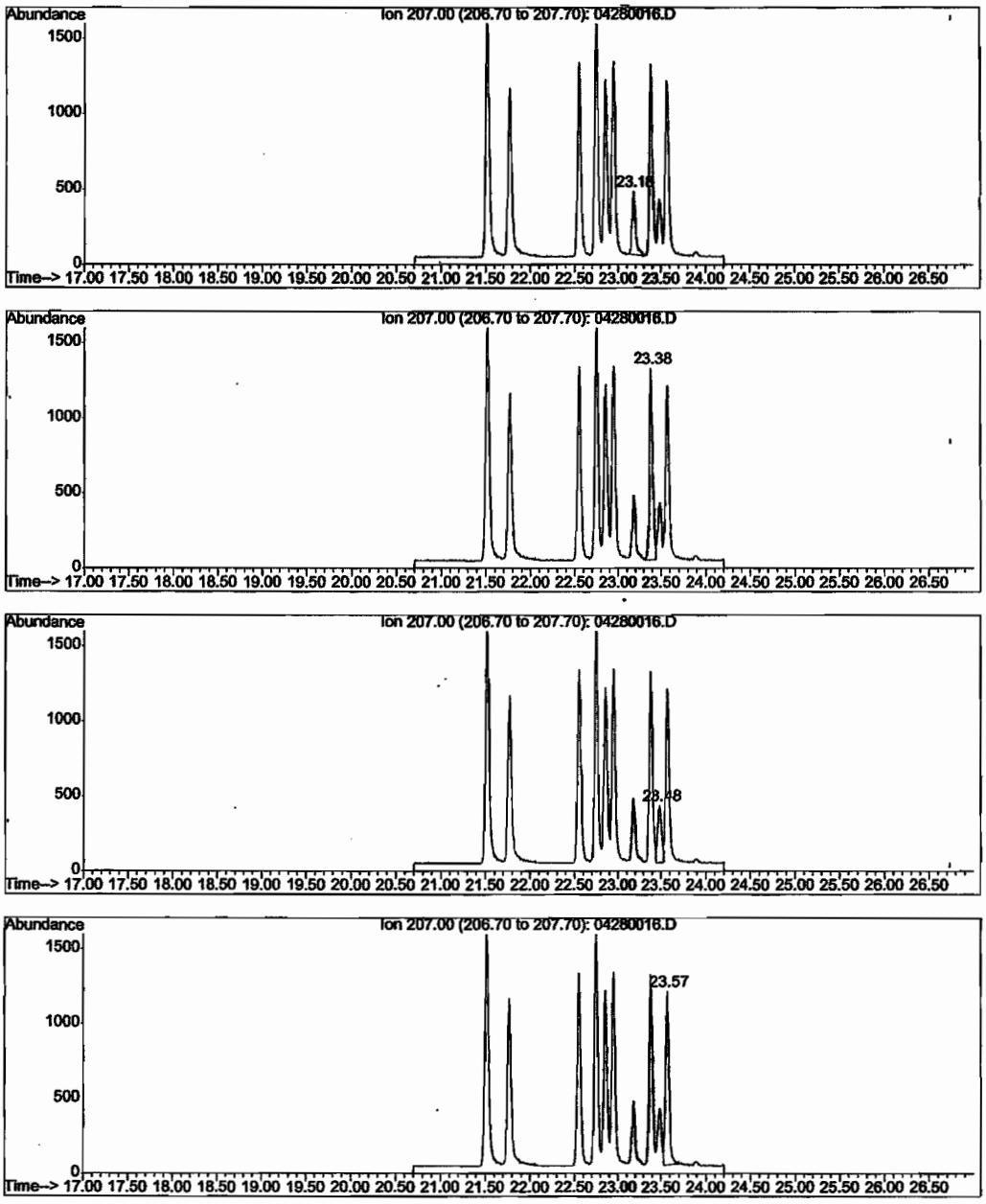
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Cyfluthrin
20 ng/mL
Peak Response (area): 144415

FIGURE 5. (con't) Representative Chromatograms – Bracketing Standards

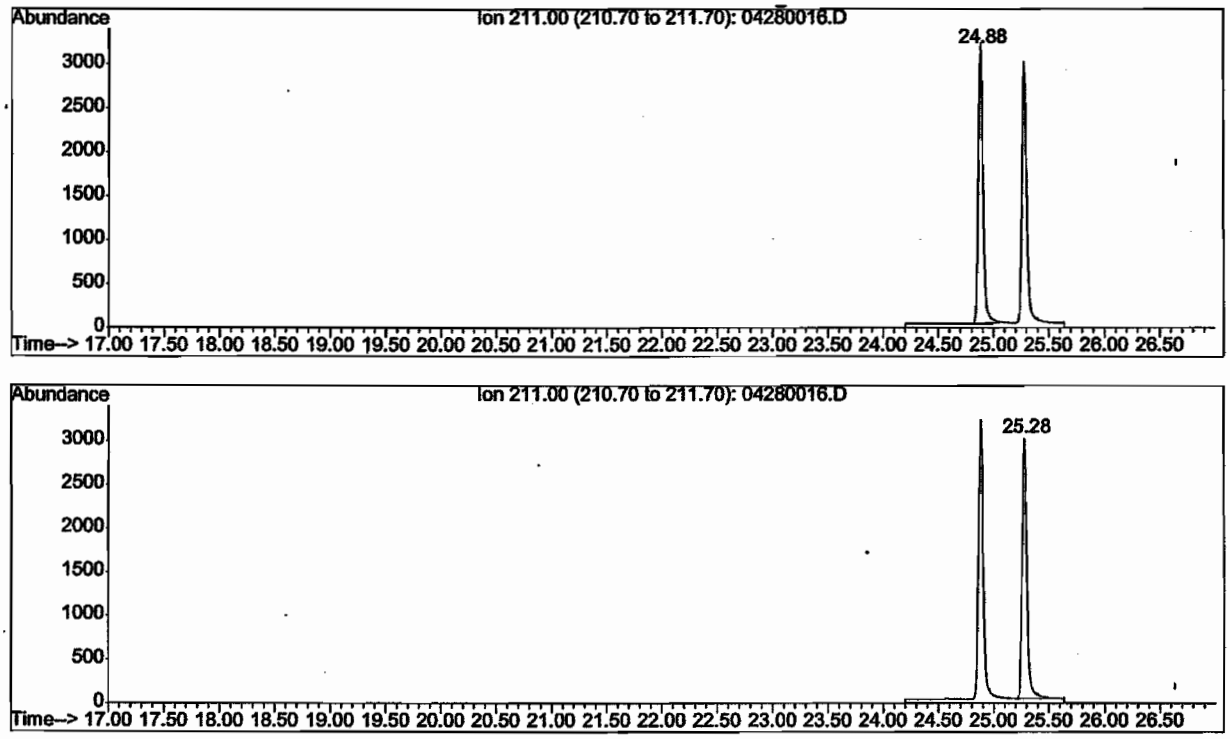
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Cypermethrin
20 ng/mL
Peak Response (area): 91267

FIGURE 5. (con't) Representative Chromatograms – Bracketing Standards

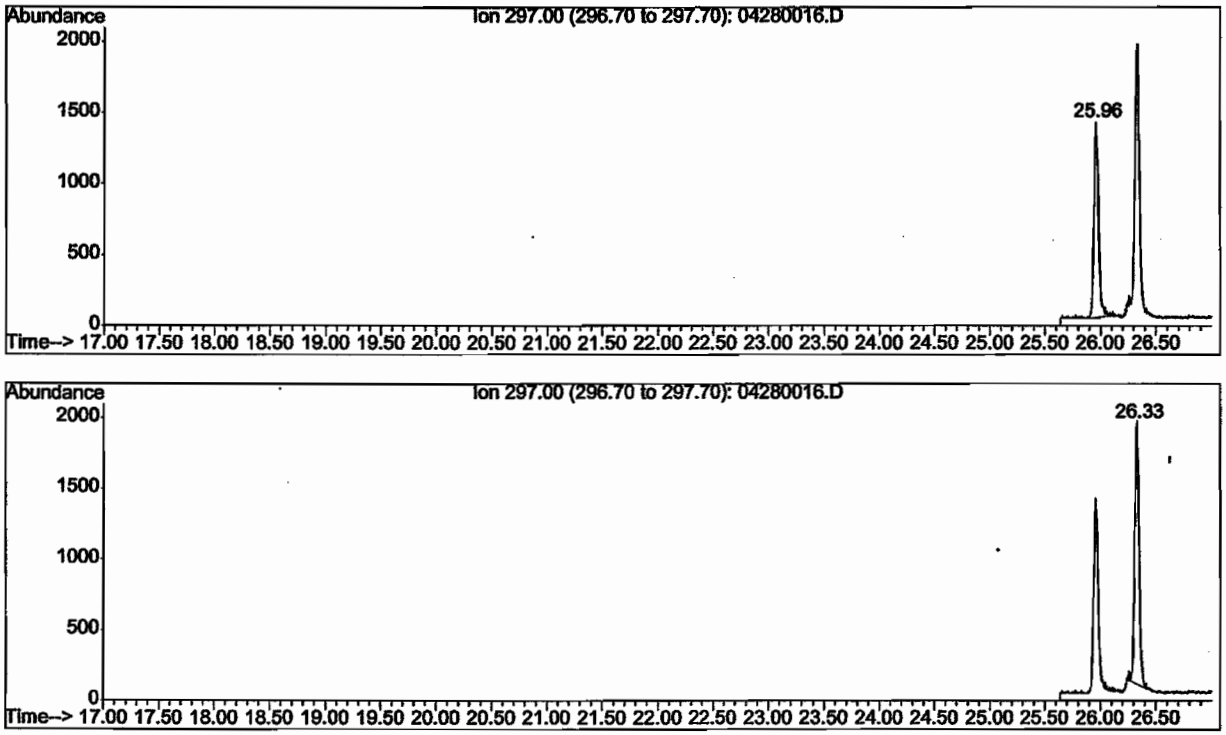
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Esfenvalerate
20 ng/mL
Peak Response (area): 181974

FIGURE 5. (con't) Representative Chromatograms – Bracketing Standards

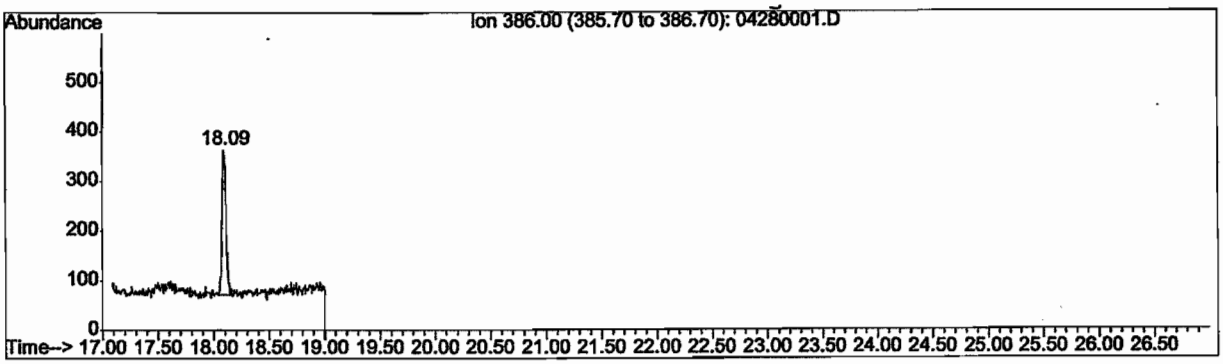
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



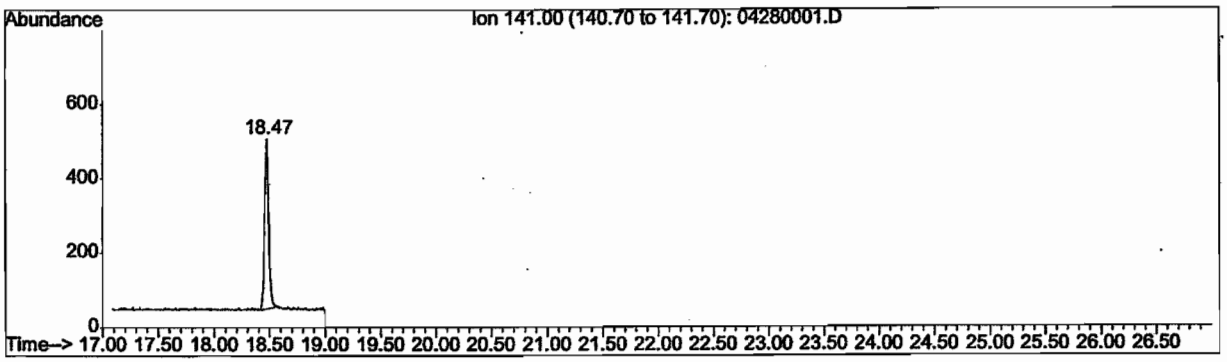
Deltamethrin
20 ng/mL
Peak Response (area): 88712

FIGURE 6. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



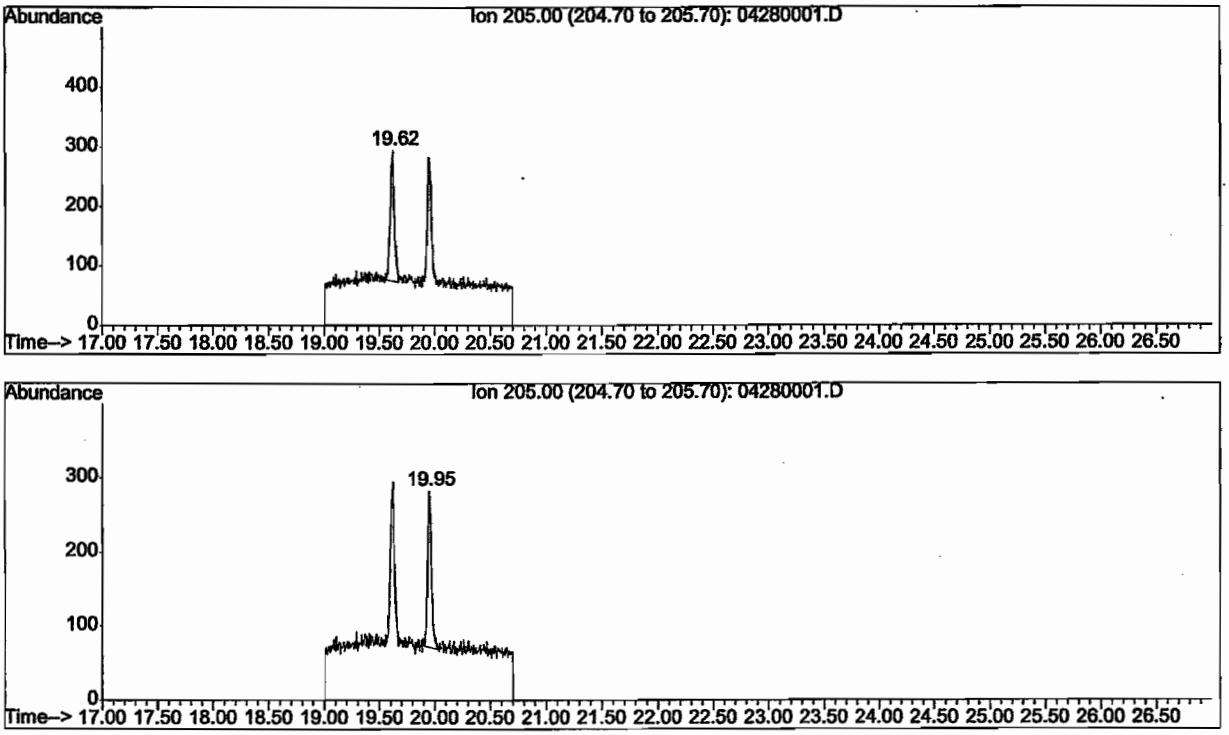
Bifenthrin
1.0 ng/mL
Peak Response (area): 7454



Fenpropathrin
1.0 ng/mL
Peak Response (area): 11706

FIGURE 6. (con't) Representative Chromatograms – Bracketing Standards

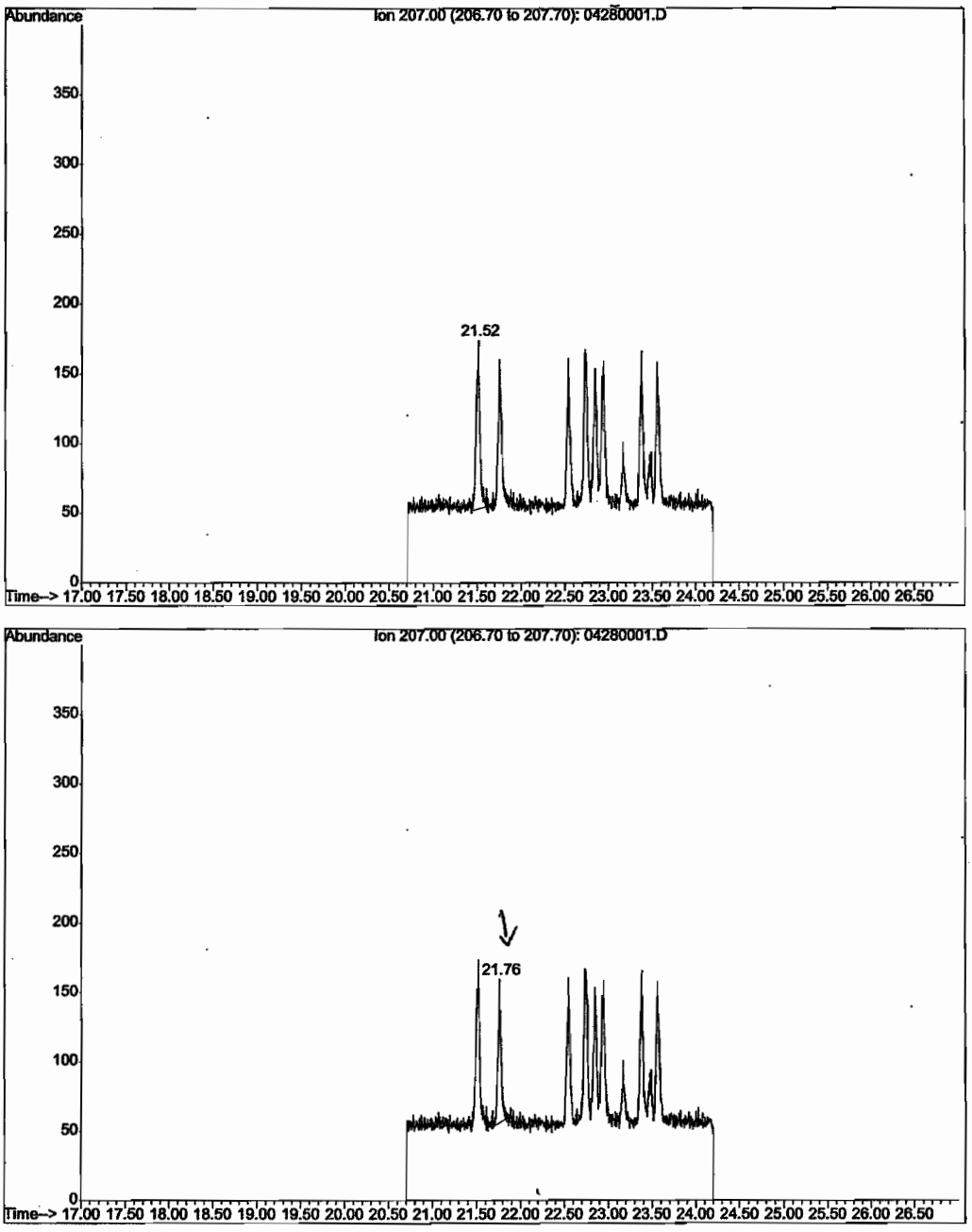
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 10784

FIGURE 6. (con't) Representative Chromatograms – Bracketing Standards

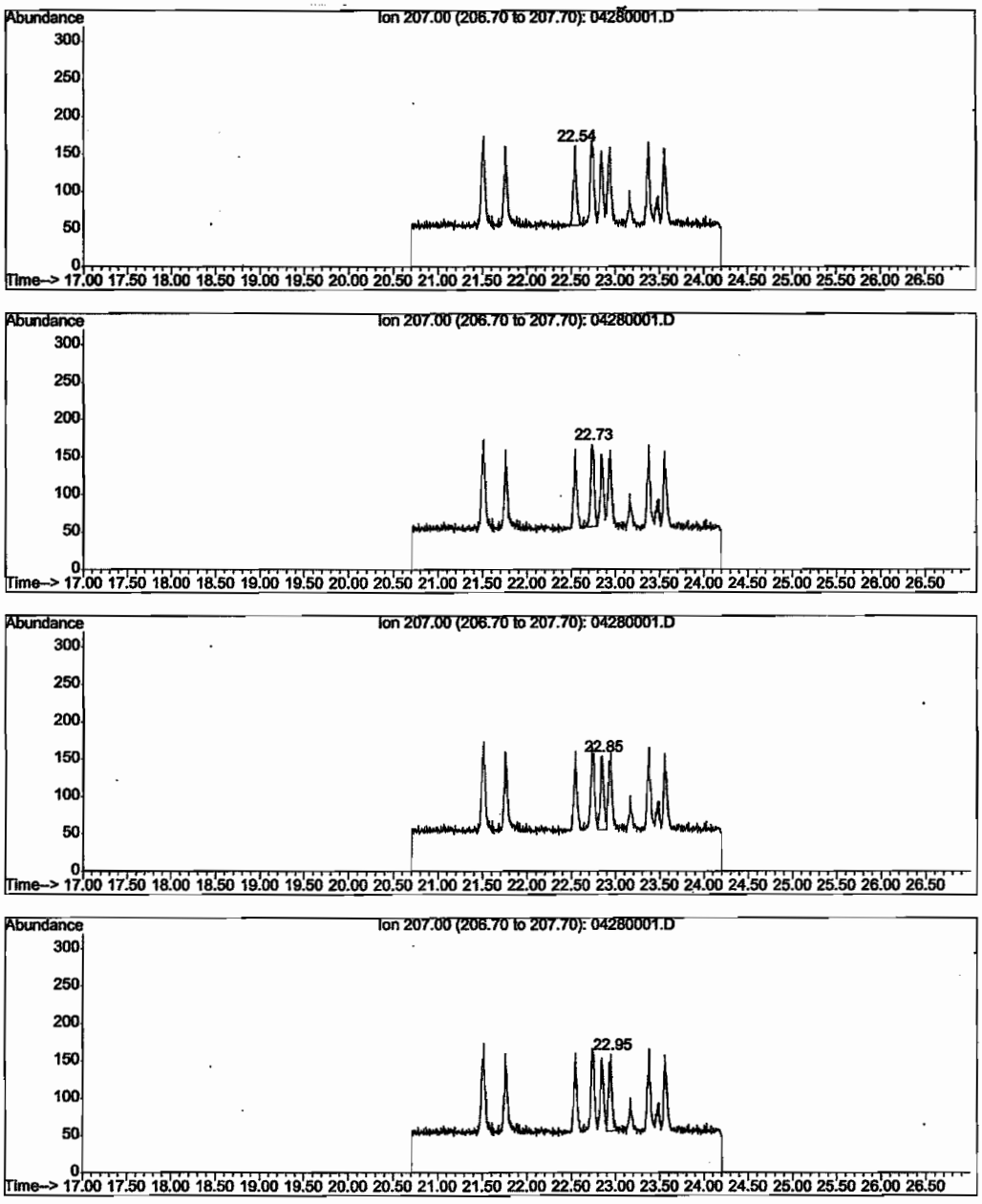
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Permethrin
10 ng/mL
Peak Response (area): 5857

FIGURE 6. (con't) Representative Chromatograms – Bracketing Standards

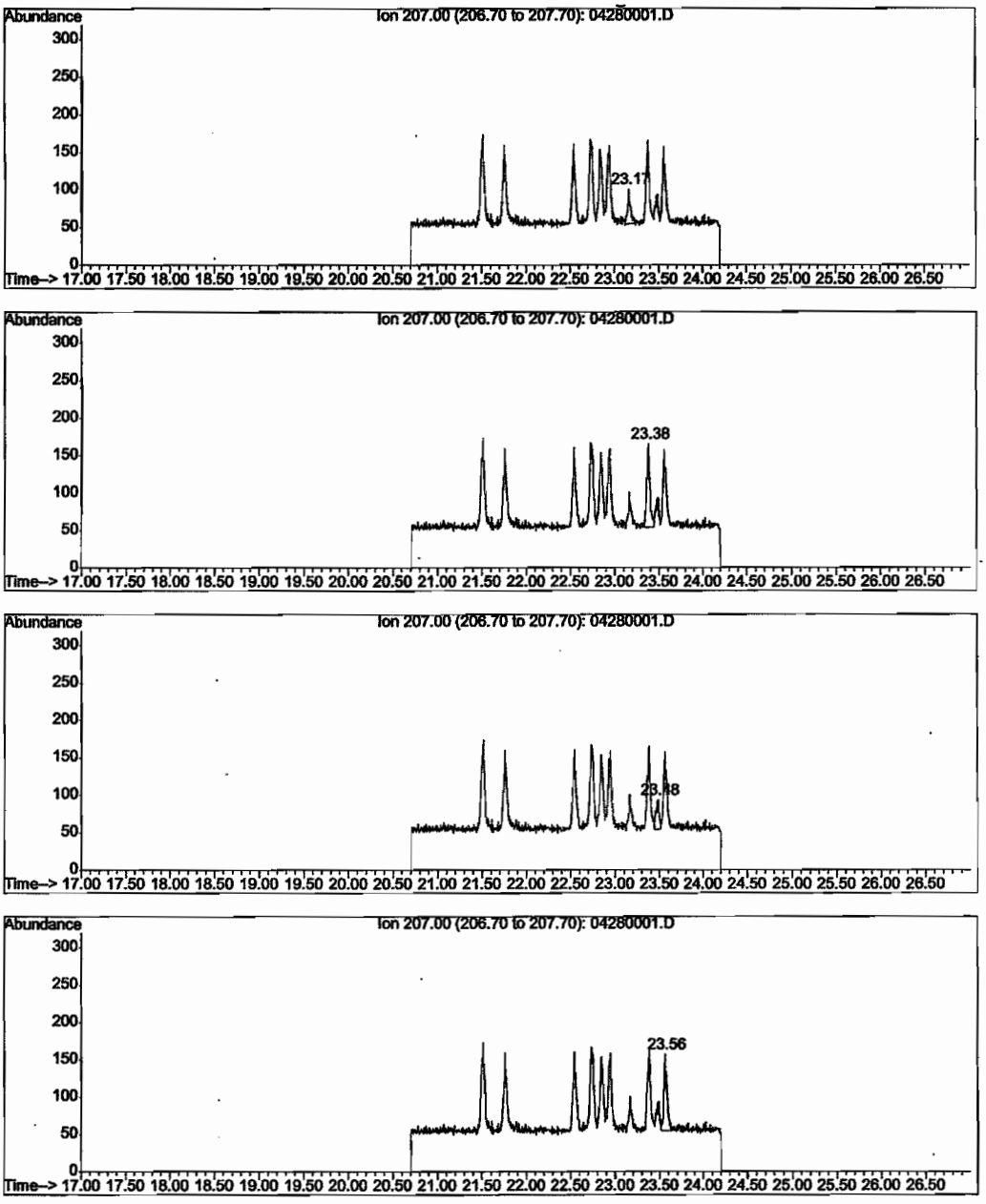
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Cyfluthrin
1.0 ng/mL
Peak Response (area): 10870

FIGURE 6. (con't) Representative Chromatograms – Bracketing Standards

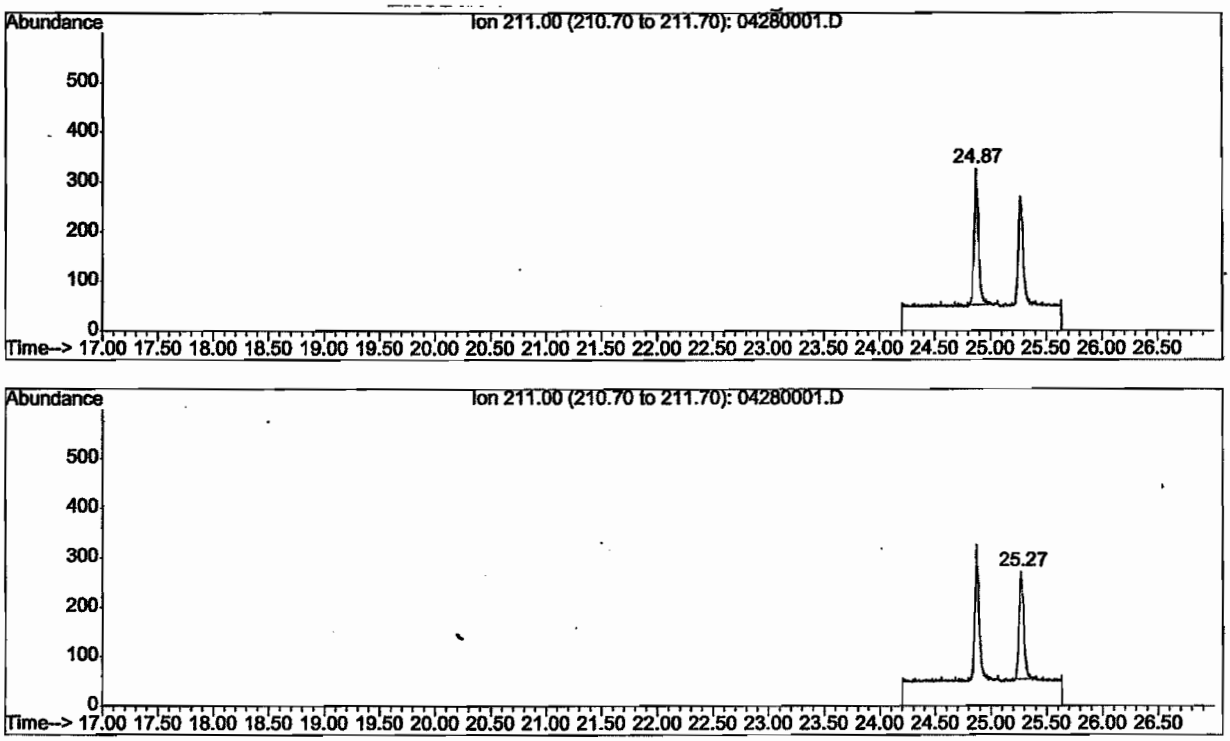
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Cypermethrin
1.0 ng/mL
Peak Response (area): 7257

FIGURE 6. (con't) Representative Chromatograms – Bracketing Standards

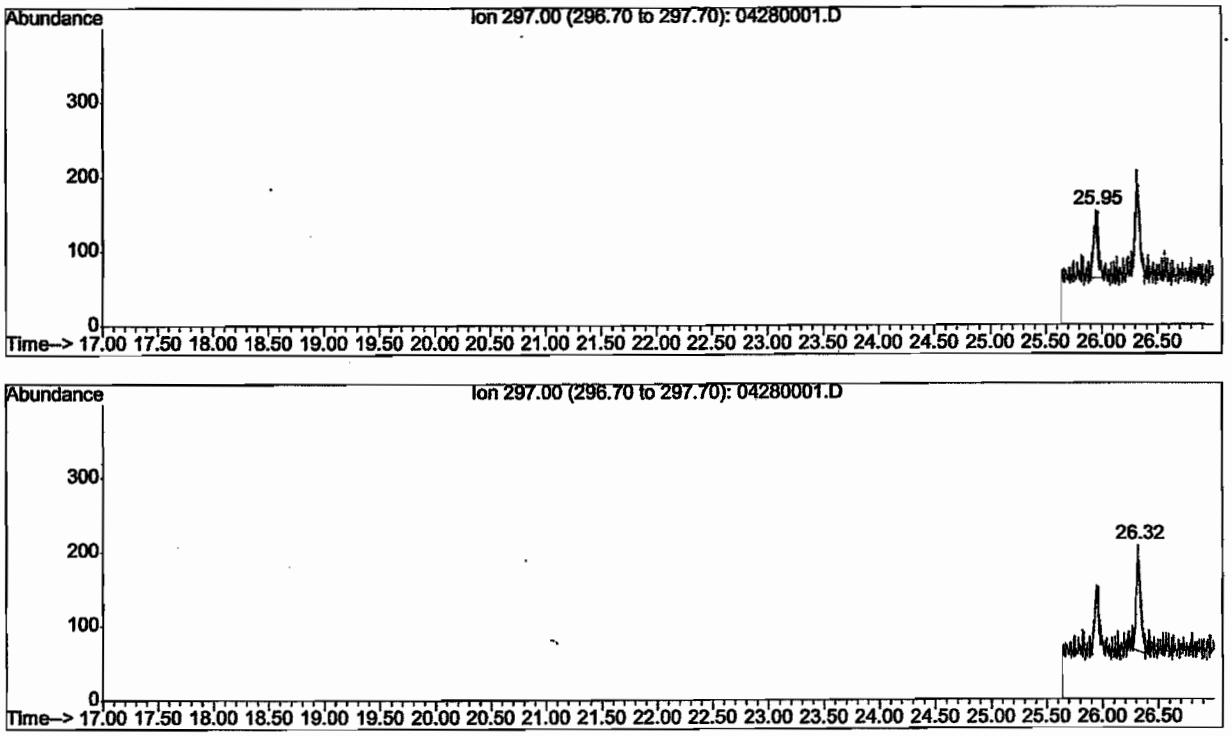
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Esfenvalerate
1.0 ng/mL
Peak Response (area): 13421

FIGURE 6. (con't) Representative Chromatograms – Bracketing Standards

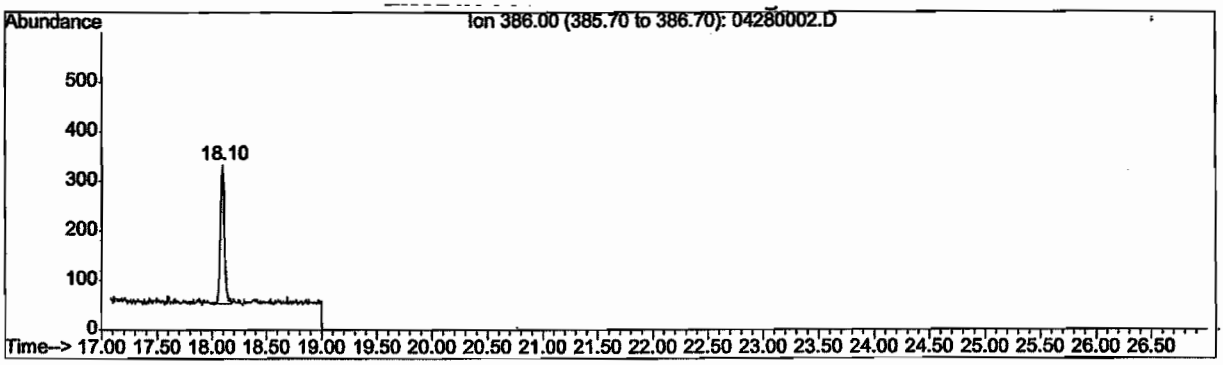
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



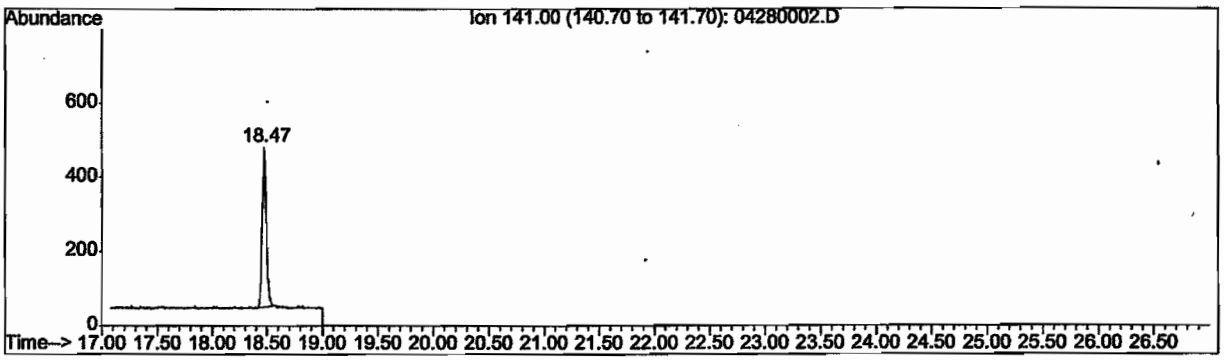
Deltamethrin
1.0 ng/mL
Peak Response (area): 6022

FIGURE 7. Representative Chromatograms - Fresh Water Sediment, Fortified Control (LOQ)

Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #2



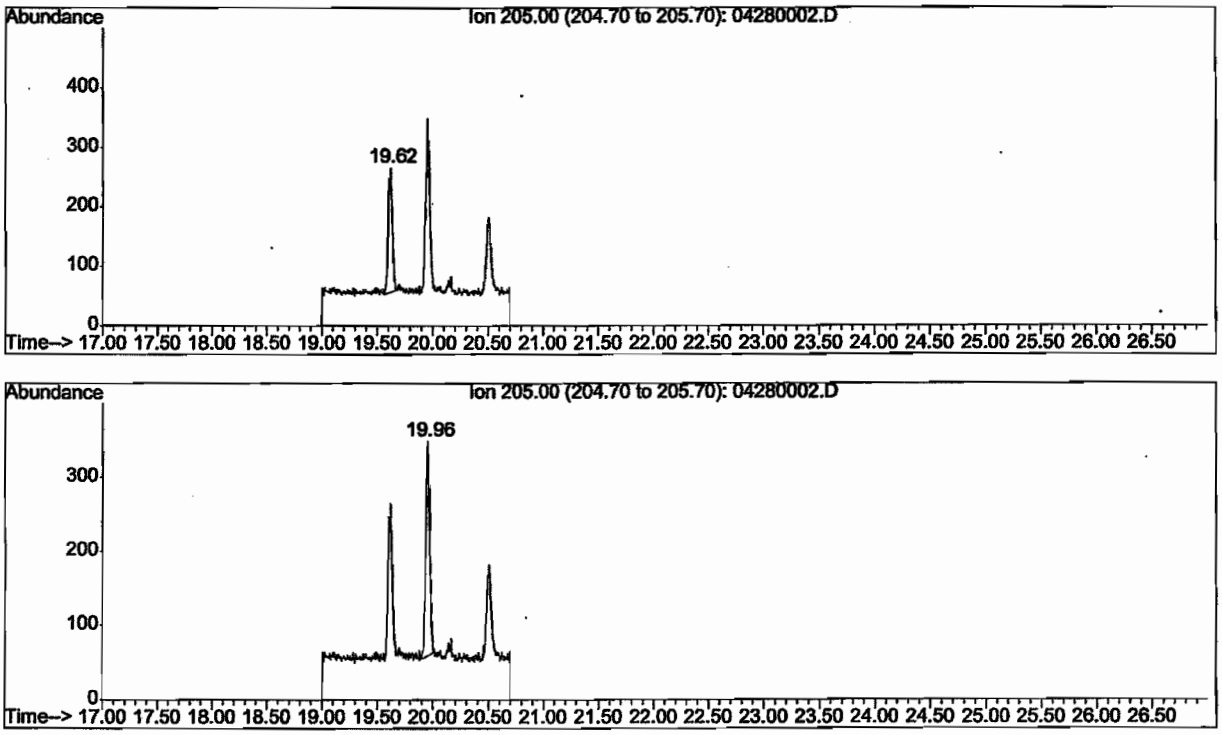
Bifenthrin
Peak Response (area): 7355
Bifenthrin Reported Recovery: 103%



Fenpropathrin
Peak Response (area): 11040
Fenpropathrin Reported Recovery: 102%

FIGURE 7. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (LOQ)

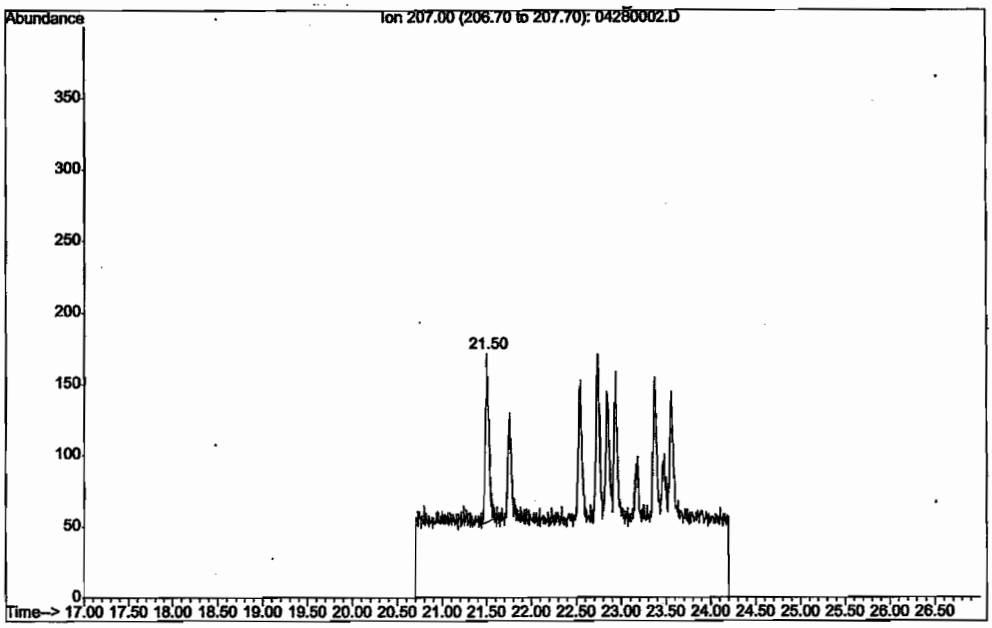
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #2

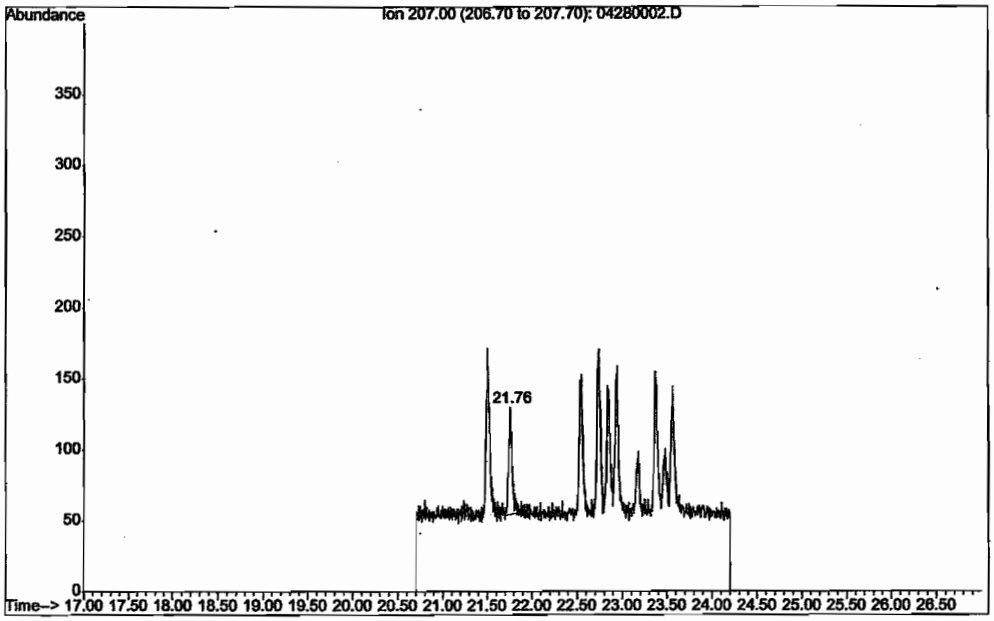


Lambda-cyhalothrin
Peak Response (area): 12470
Lambda-cyhalothrin Reported Recovery: 84%

FIGURE 7. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (LOQ)

Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #2

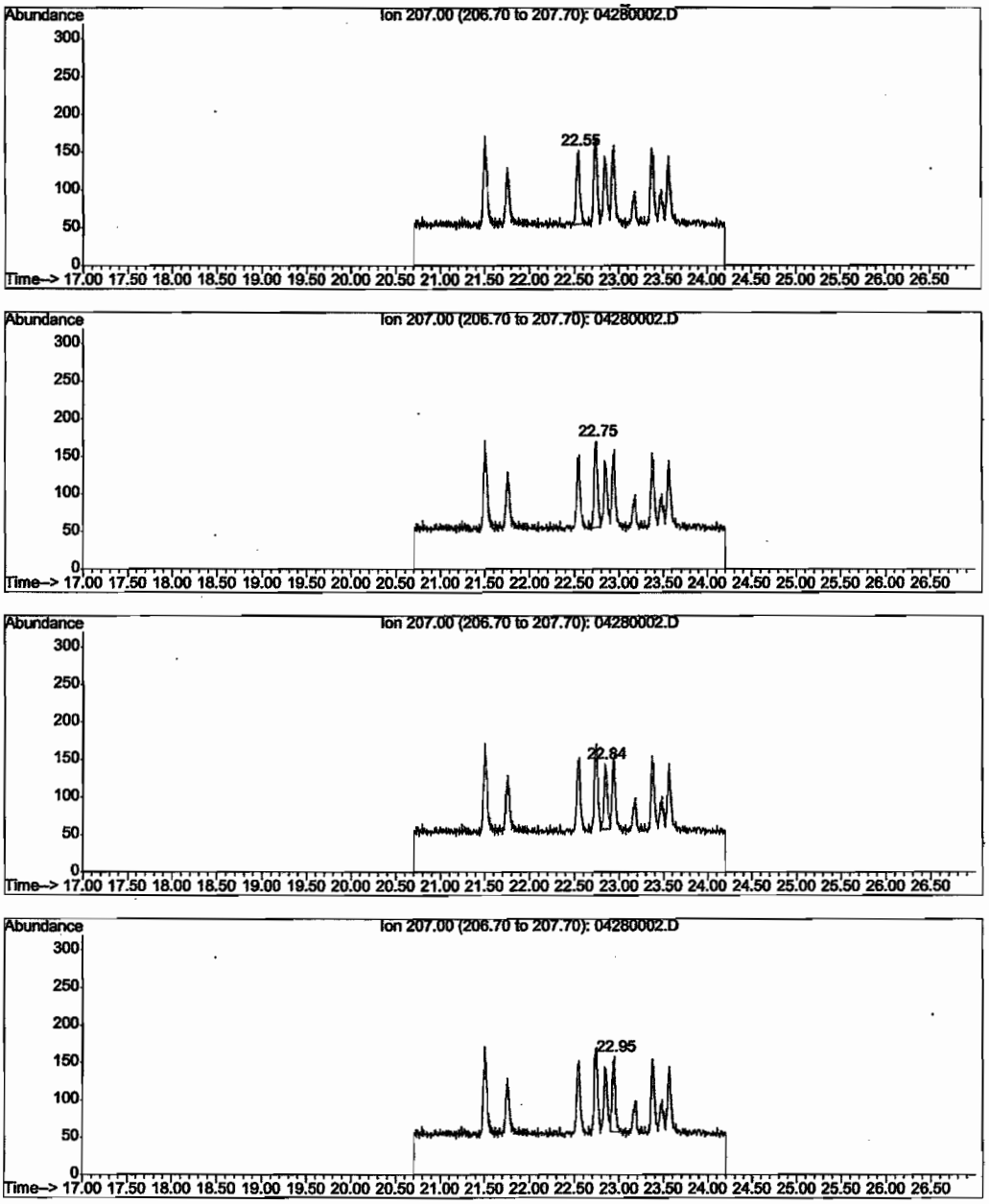




Permethrin
Peak Response (area): 5246
Permethrin Reported Recovery: 97%

FIGURE 7. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (LOQ)

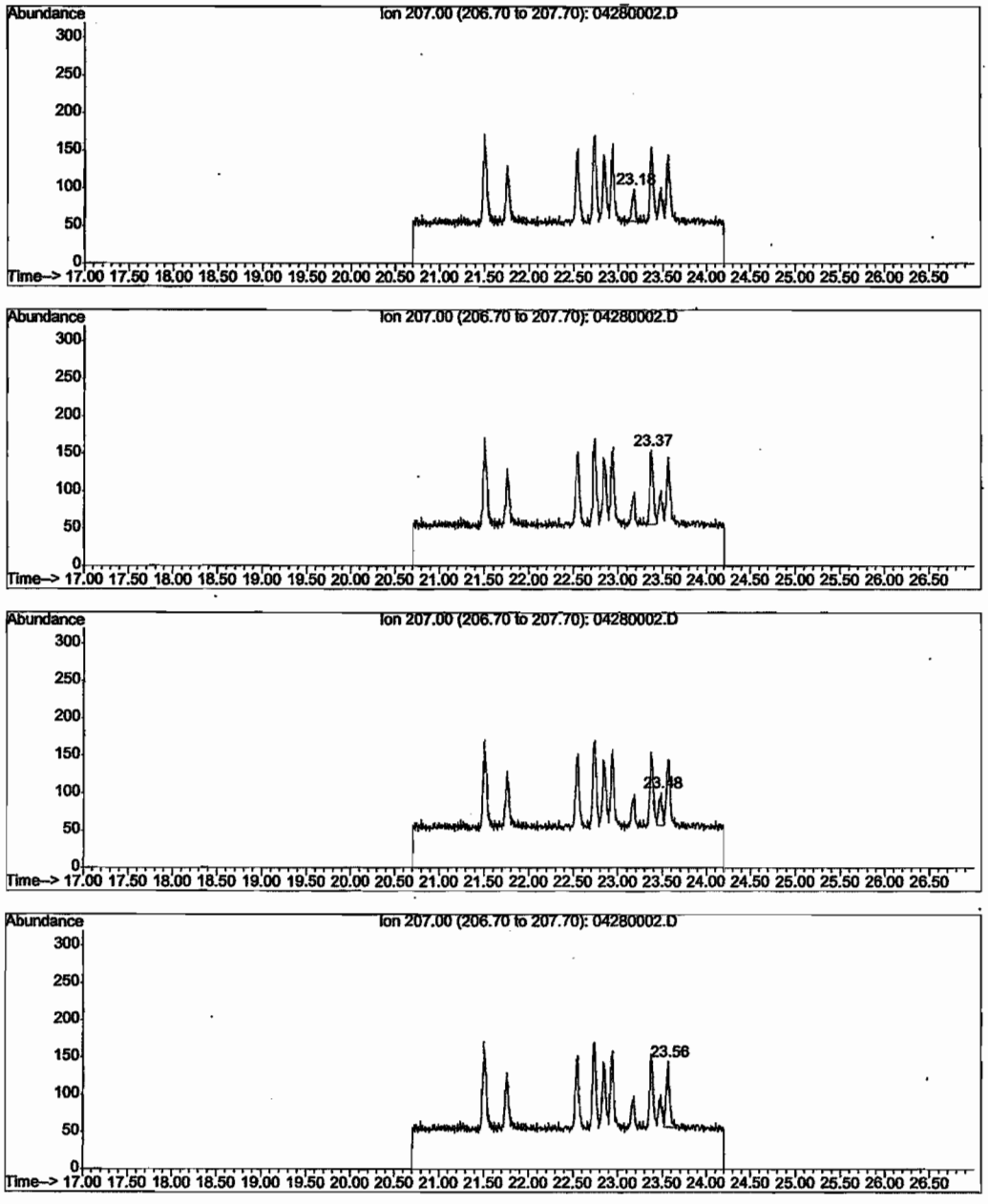
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #2



Cyfluthrin
Peak Response (area): 10542
Cyfluthrin Reported Recovery: 107%

FIGURE 7. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (LOQ)

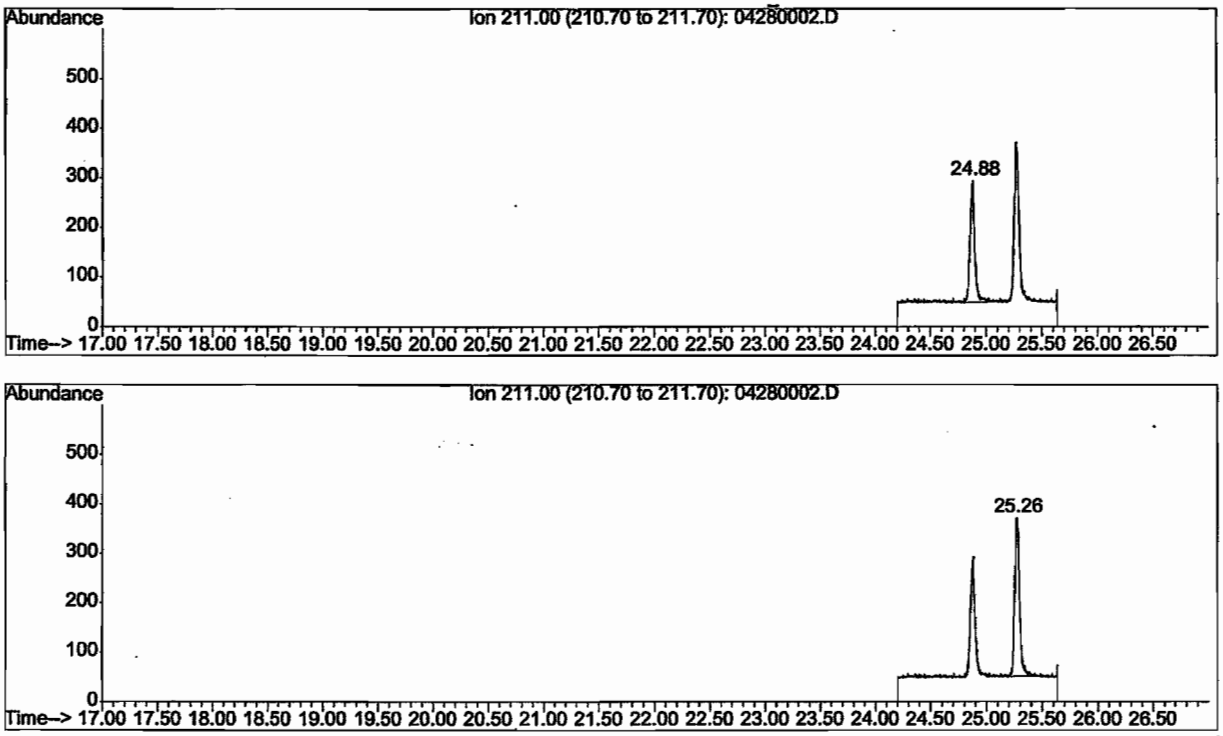
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #2



Cypermethrin
Peak Response (area): 7080
Cypermethrin Reported Recovery: 107%

FIGURE 7. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (LOQ)

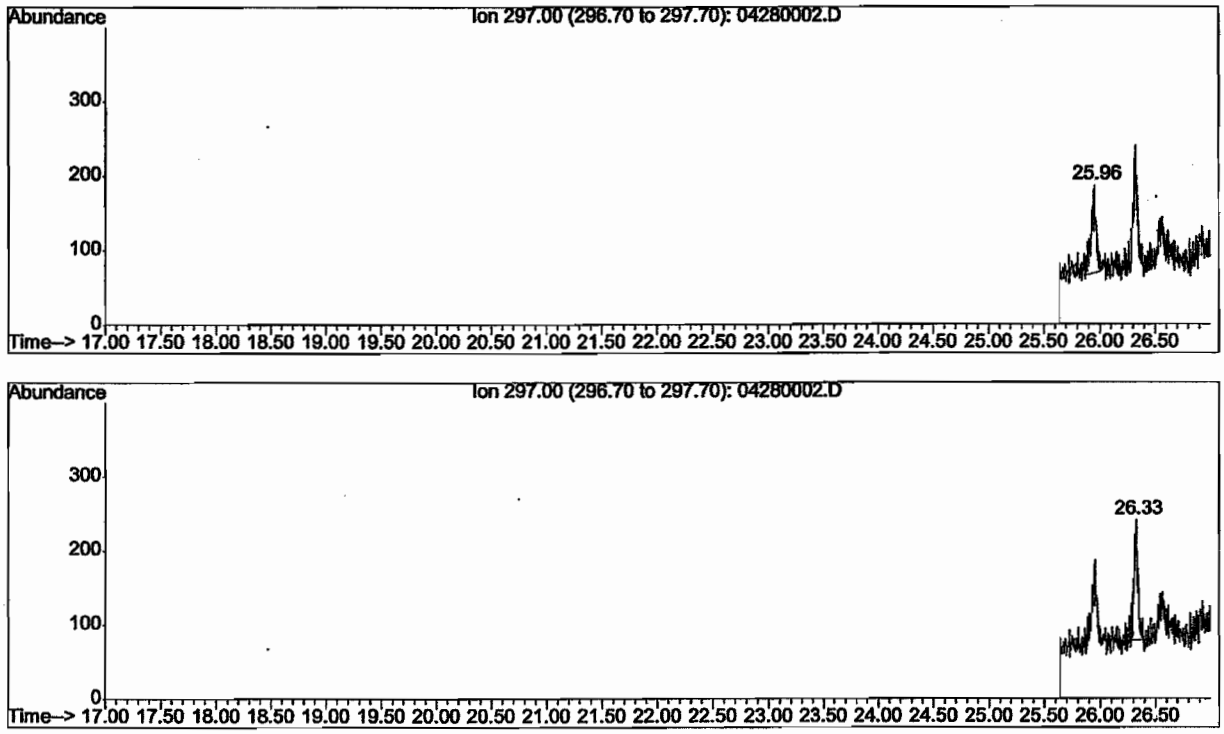
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #2



Esfenvalerate
Peak Response (area): 16887
Esfenvalerate Reported Recovery: 76%

FIGURE 7. (con't) Representative Chromatograms - Fresh Water Sediment, Fortified Control (LOQ)

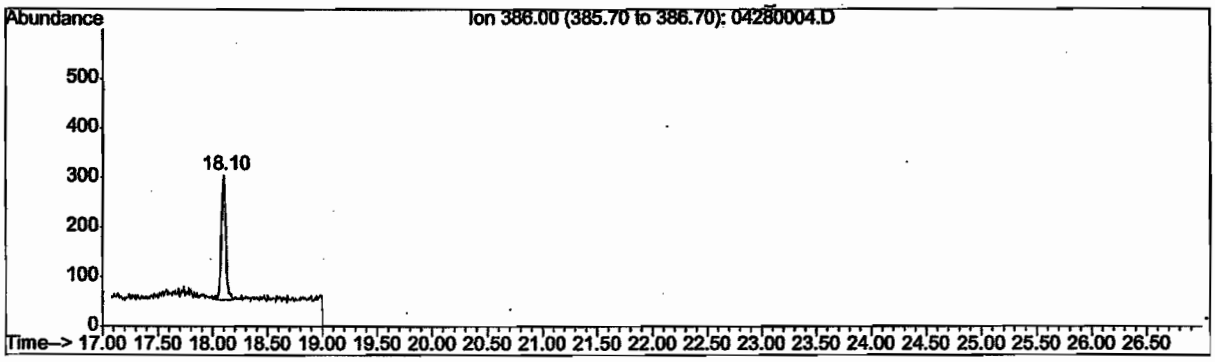
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #2



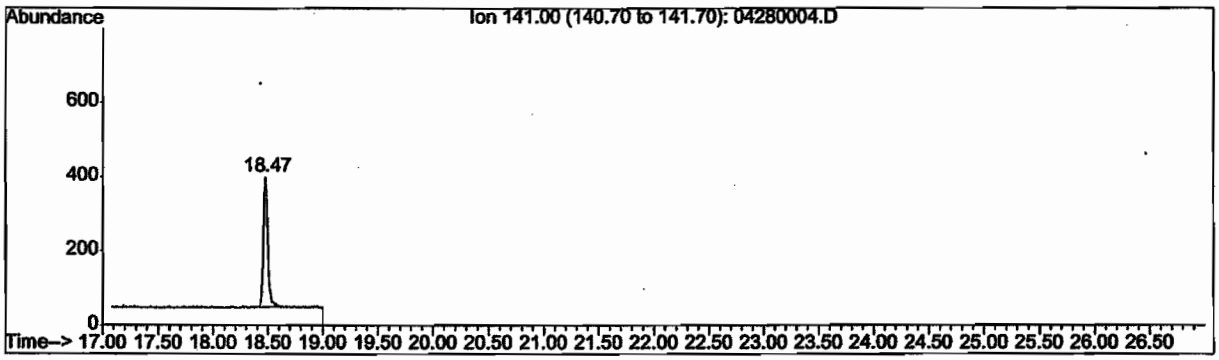
Deltamethrin
Peak Response (area): 7633
Deltamethrin Reported Recovery: 89%

FIGURE 8. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



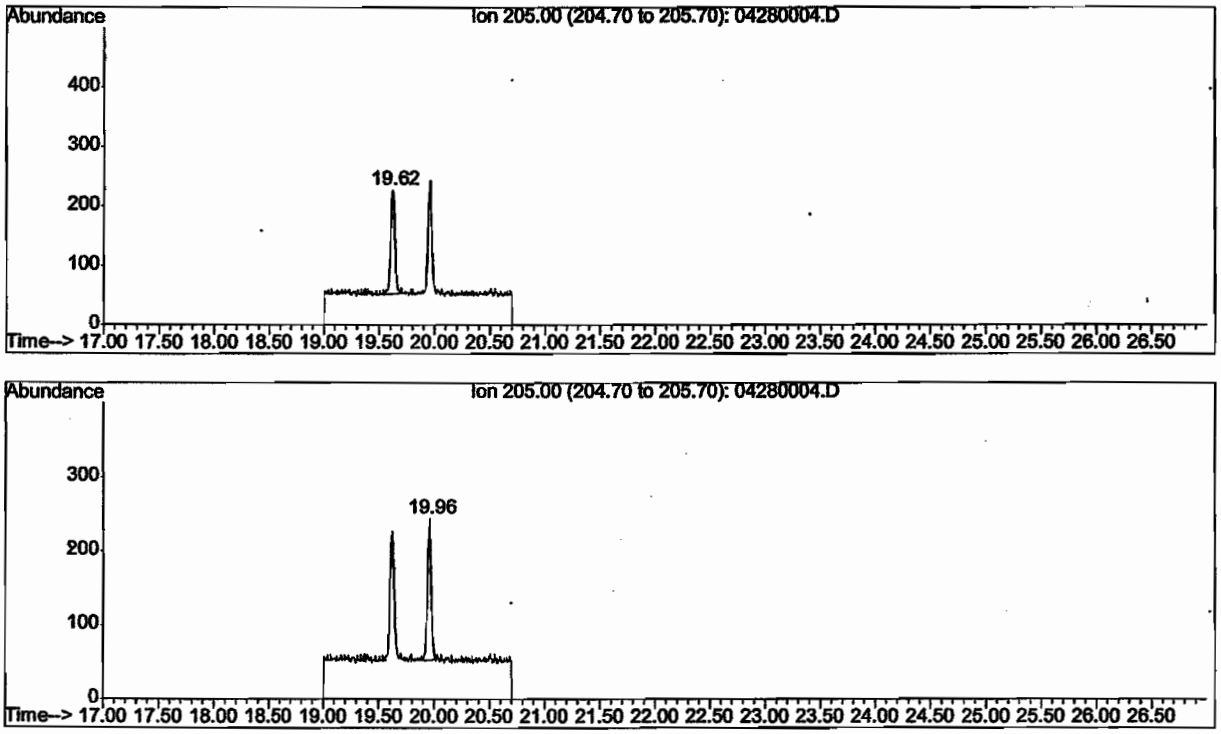
Bifenthrin
1.0 ng/mL
Peak Response (area): 6804



Fenpropathrin
1.0 ng/mL
Peak Response (area): 9881

FIGURE 8. (con't) Representative Chromatograms – Bracketing Standards

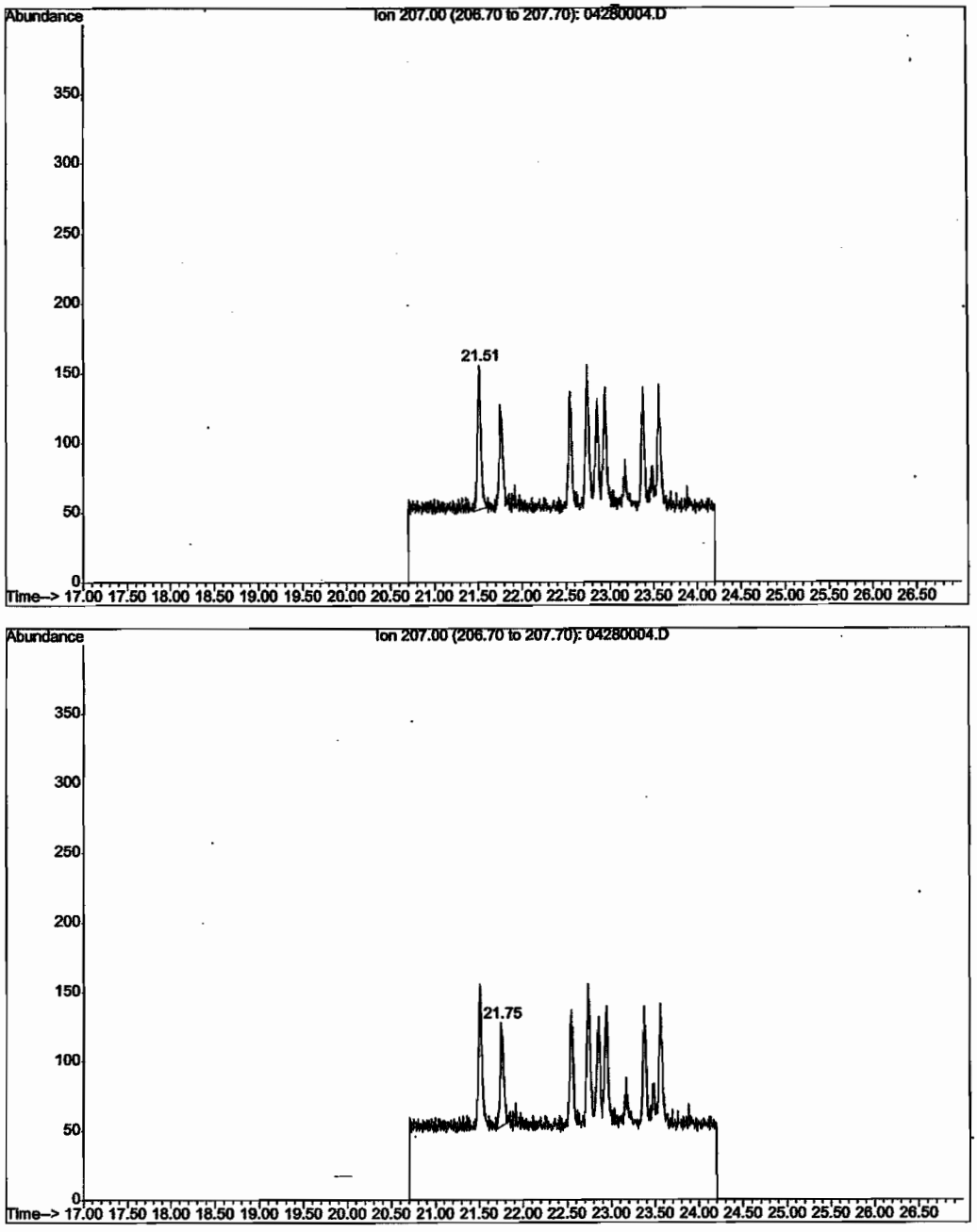
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 9119

FIGURE 8. (con't) Representative Chromatograms – Bracketing Standards

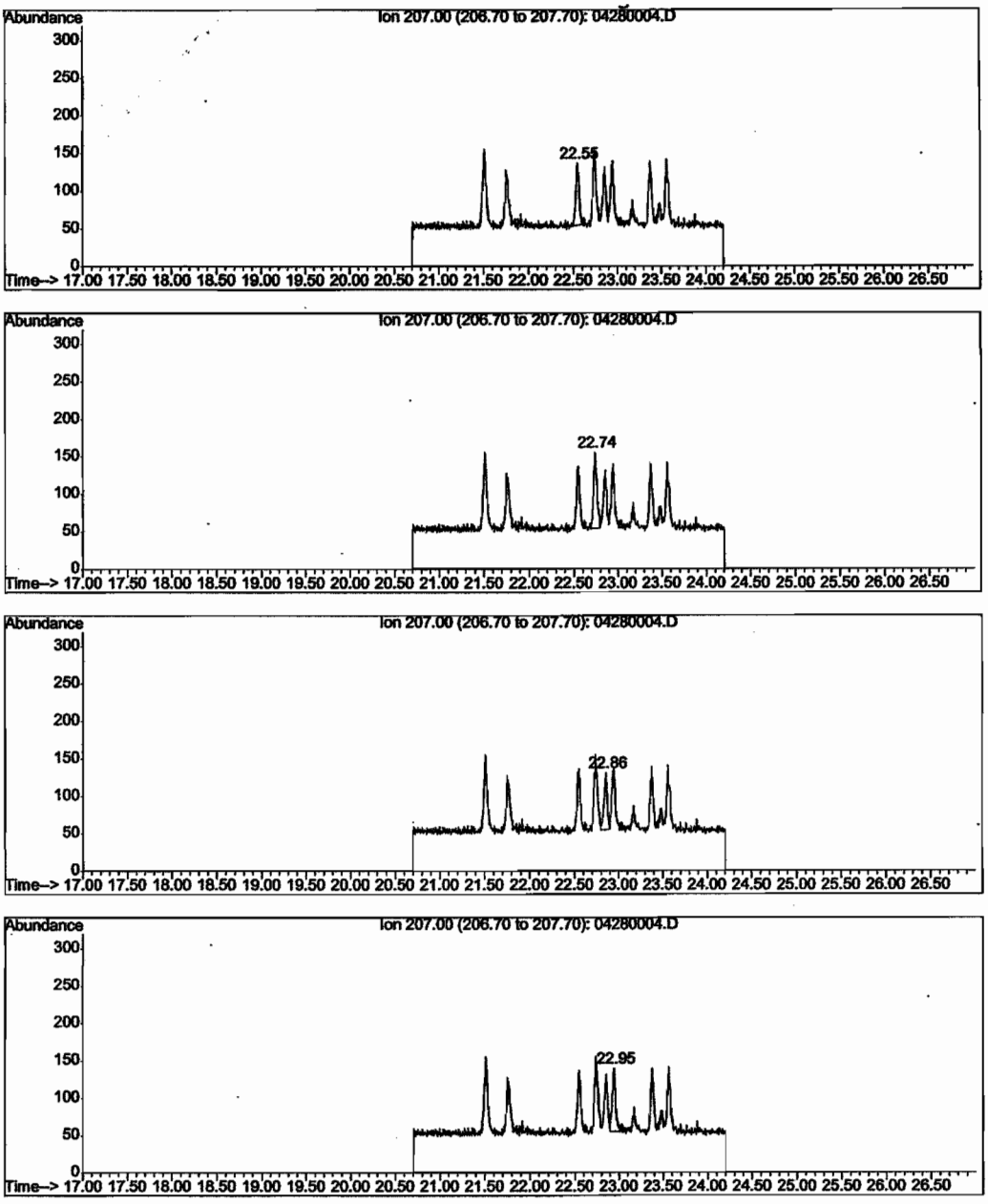
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Permethrin
10 ng/mL
Peak Response (area): 4918

FIGURE 8. (con't) Representative Chromatograms – Bracketing Standards

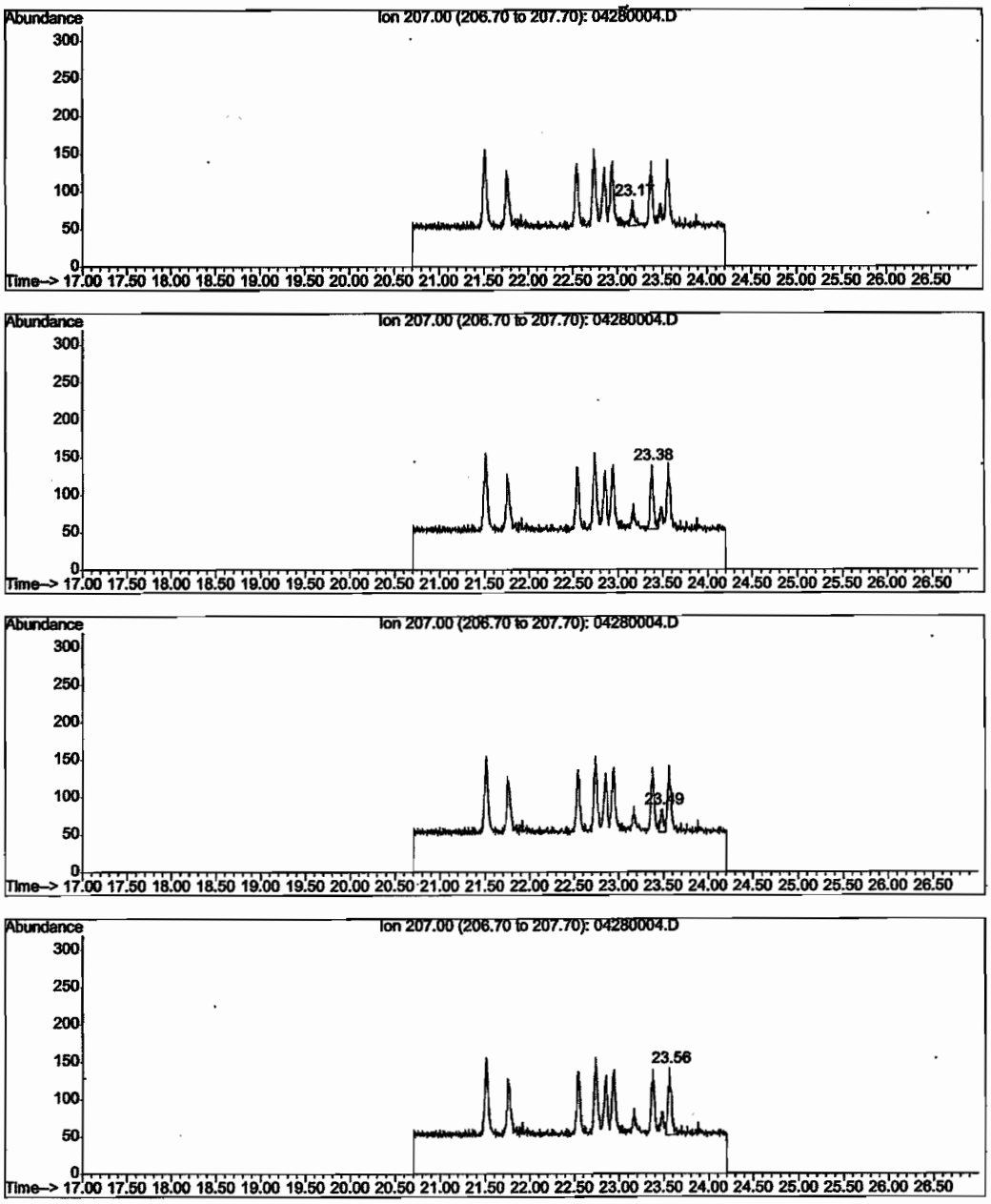
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Cyfluthrin
1.0 ng/mL
Peak Response (area): 8872

FIGURE 8. (con't) Representative Chromatograms – Bracketing Standards

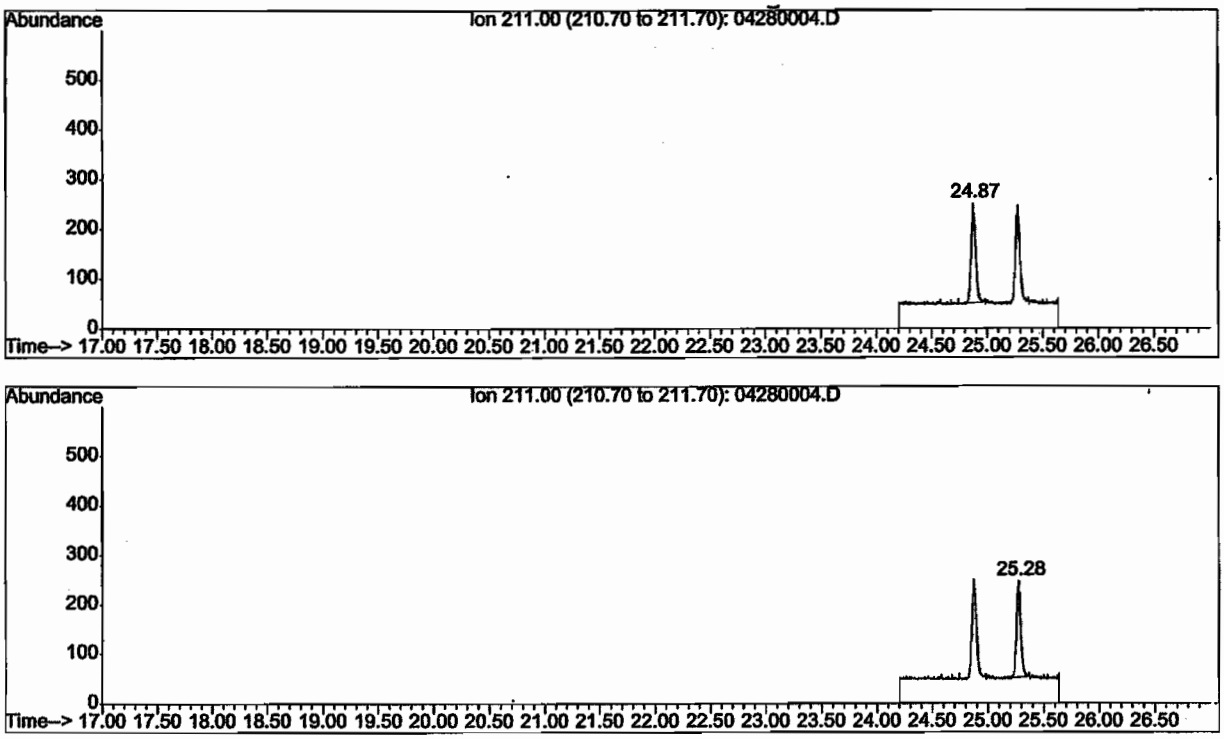
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Cypermethrin
1.0 ng/mL
Peak Response (area): 6027

FIGURE 8. (con't) Representative Chromatograms – Bracketing Standards

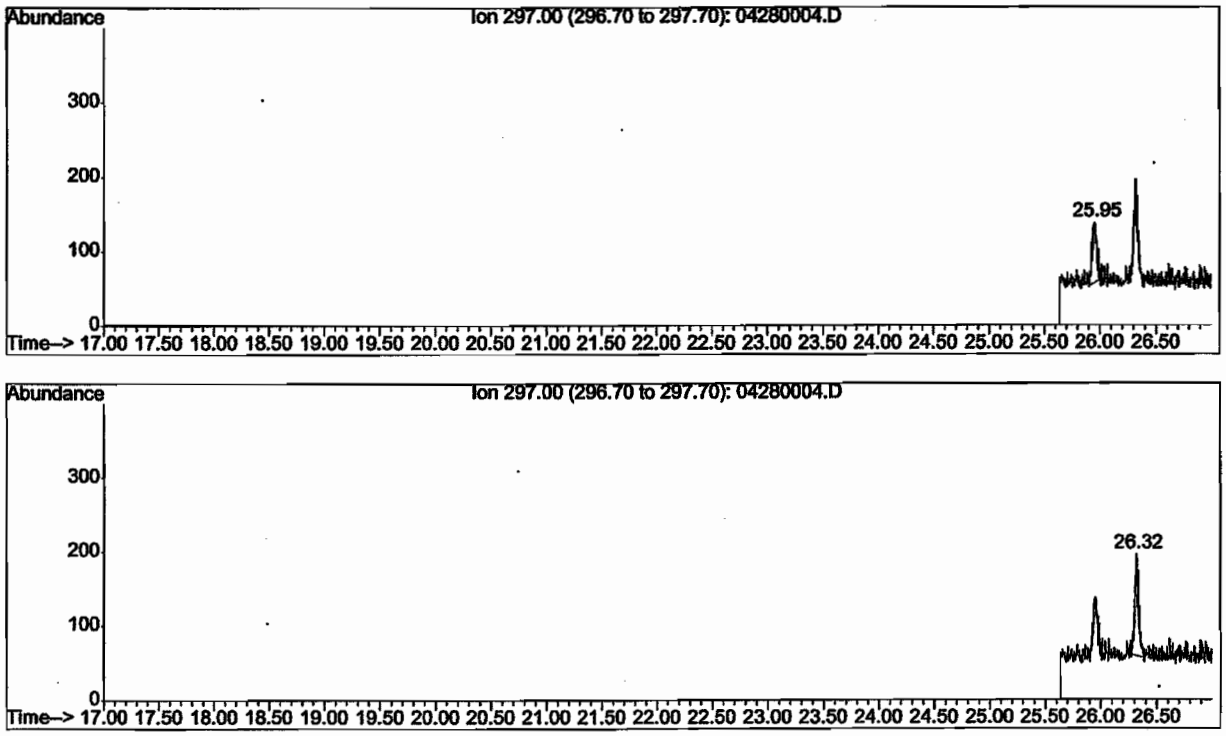
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Esfenvalerate
1.0 ng/mL
Peak Response (area): 10554

FIGURE 8. (con't) Representative Chromatograms – Bracketing Standards

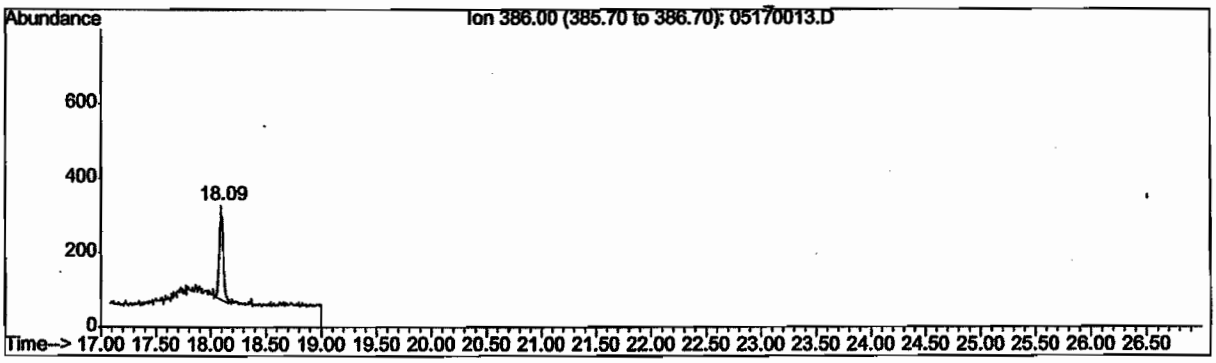
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



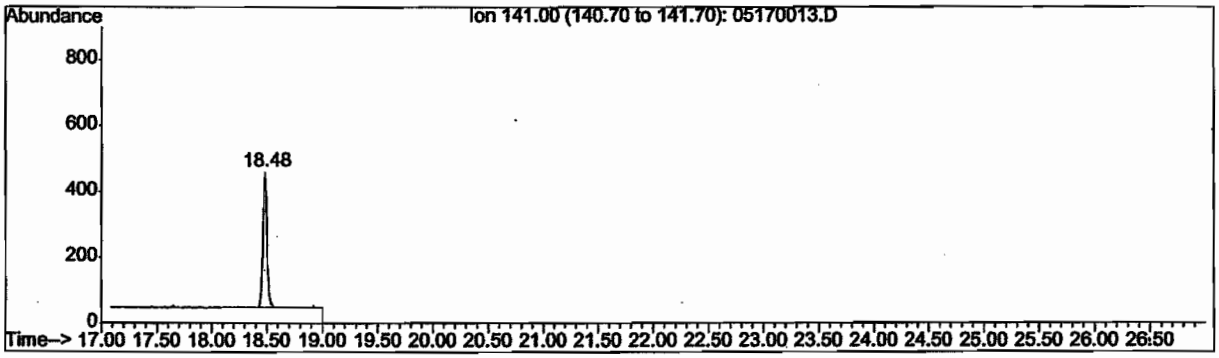
Deltamethrin
1.0 ng/mL
Peak Response (area): 5406

FIGURE 9. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



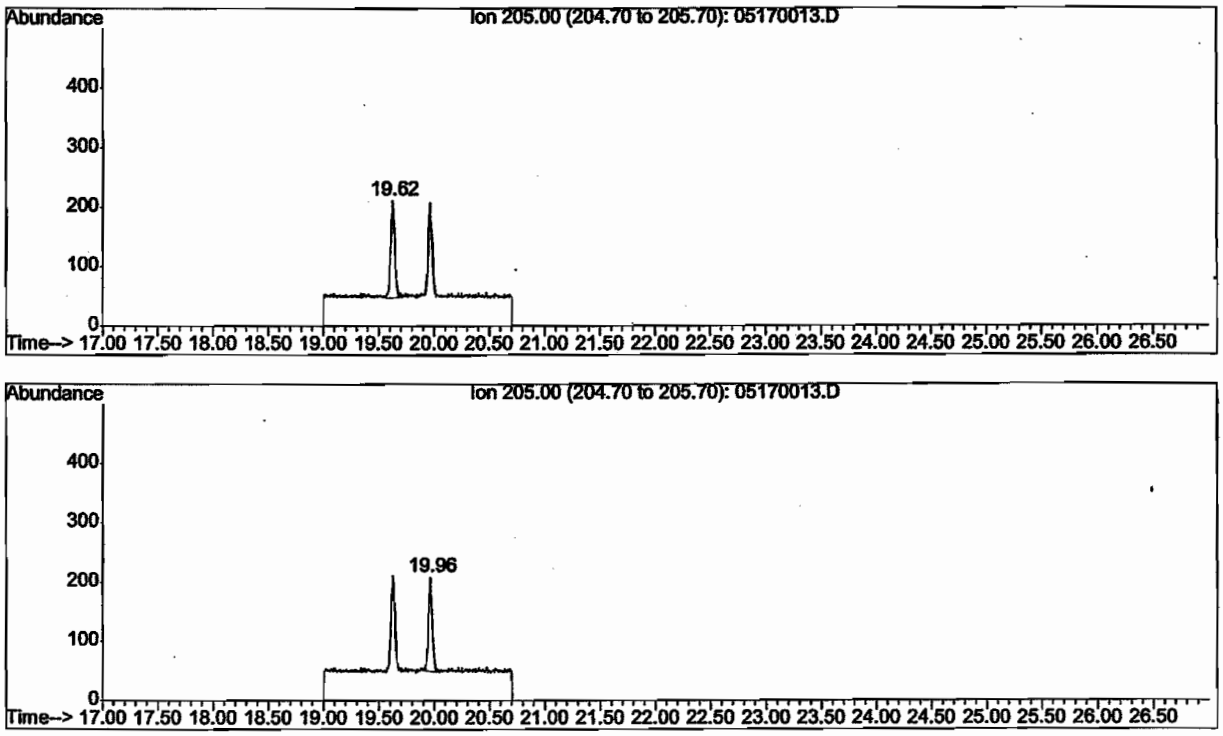
Bifenthrin
1.0 ng/mL
Peak Response (area): 6282



Fenpropathrin
1.0 ng/mL
Peak Response (area): 10454

FIGURE 9. (con't) Representative Chromatograms – Bracketing Standards

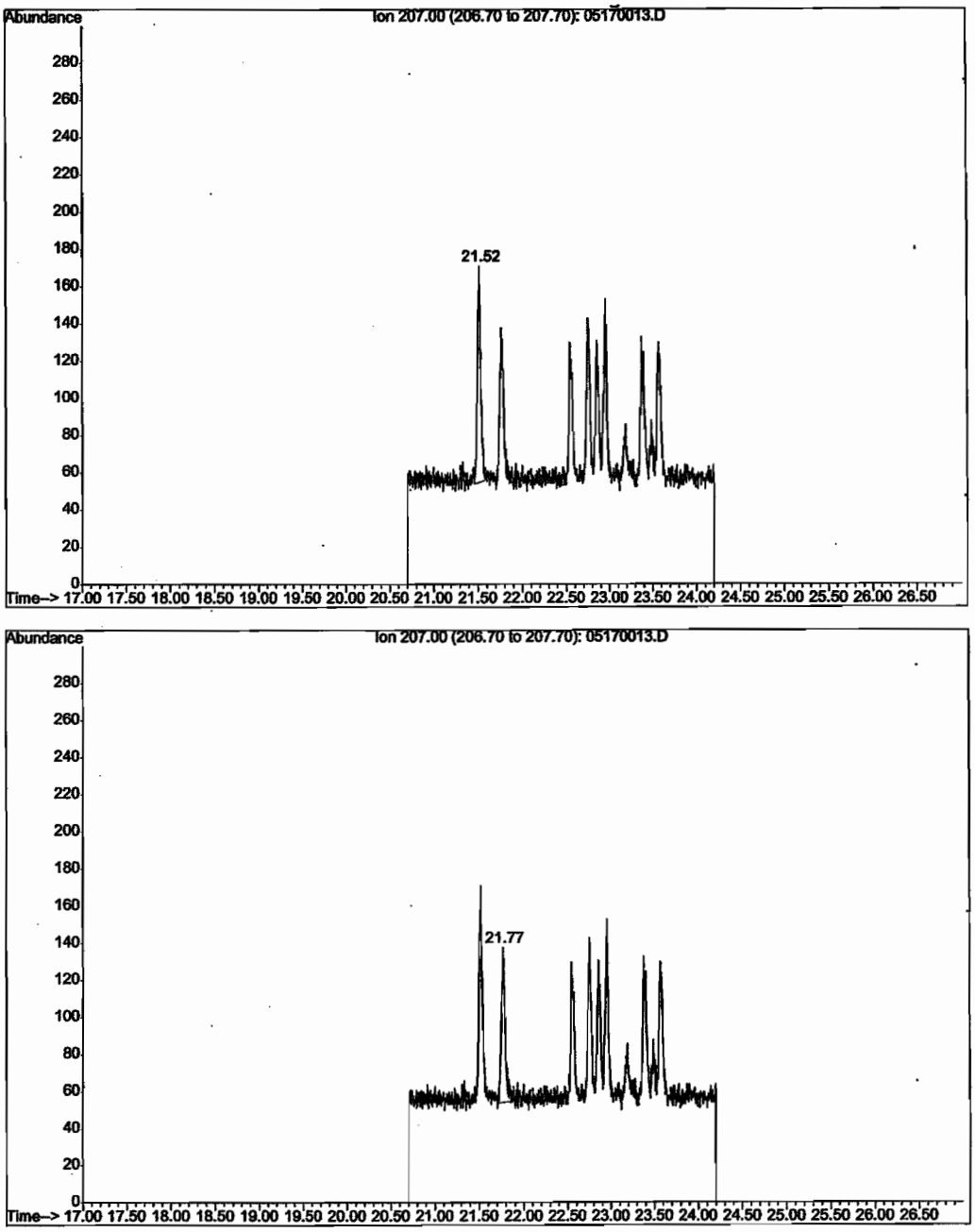
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 7973

FIGURE 9. (con't) Representative Chromatograms – Bracketing Standards

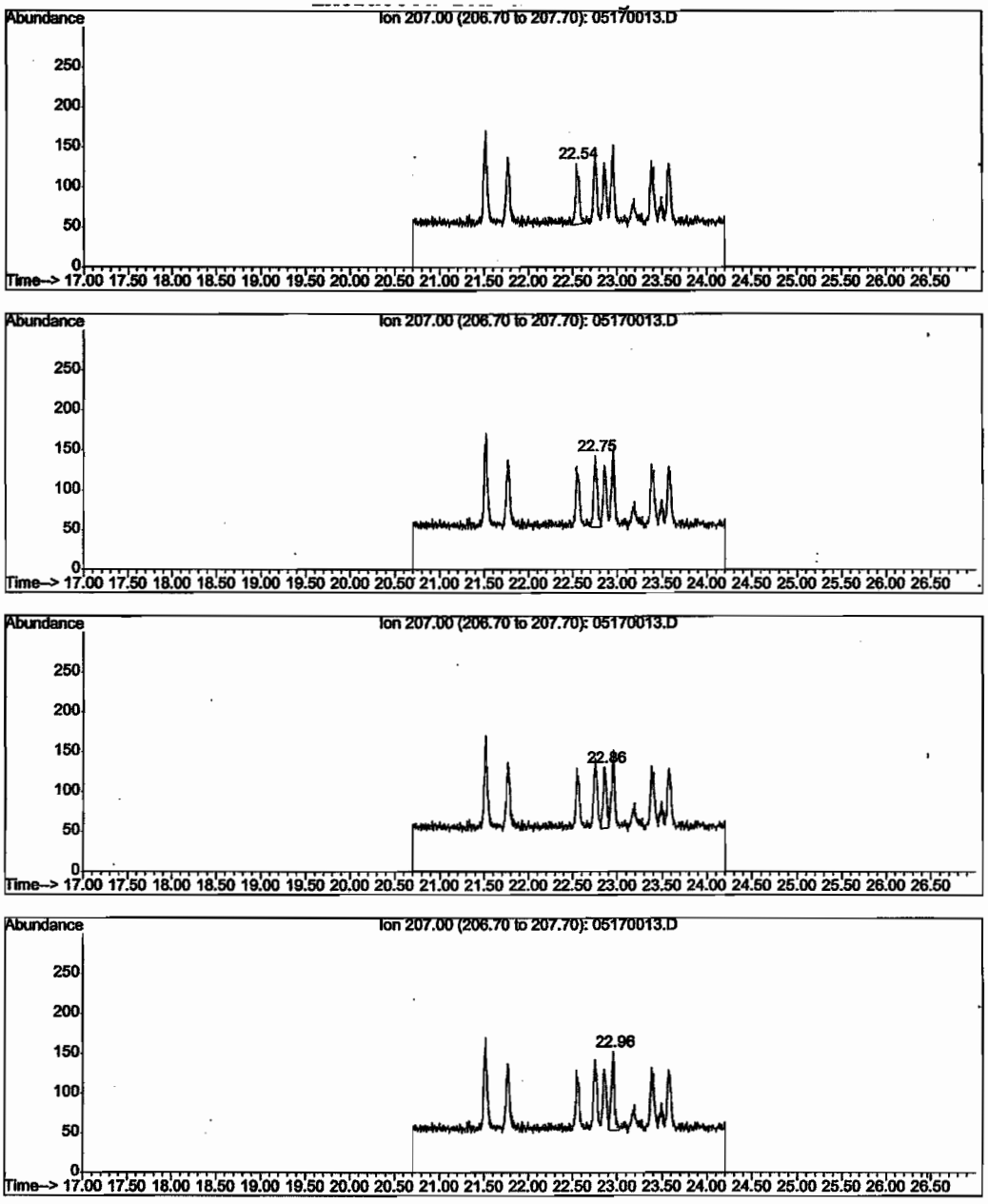
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Permethrin
10 ng/mL
Peak Response (area): 5389

FIGURE 9. (con't) Representative Chromatograms – Bracketing Standards

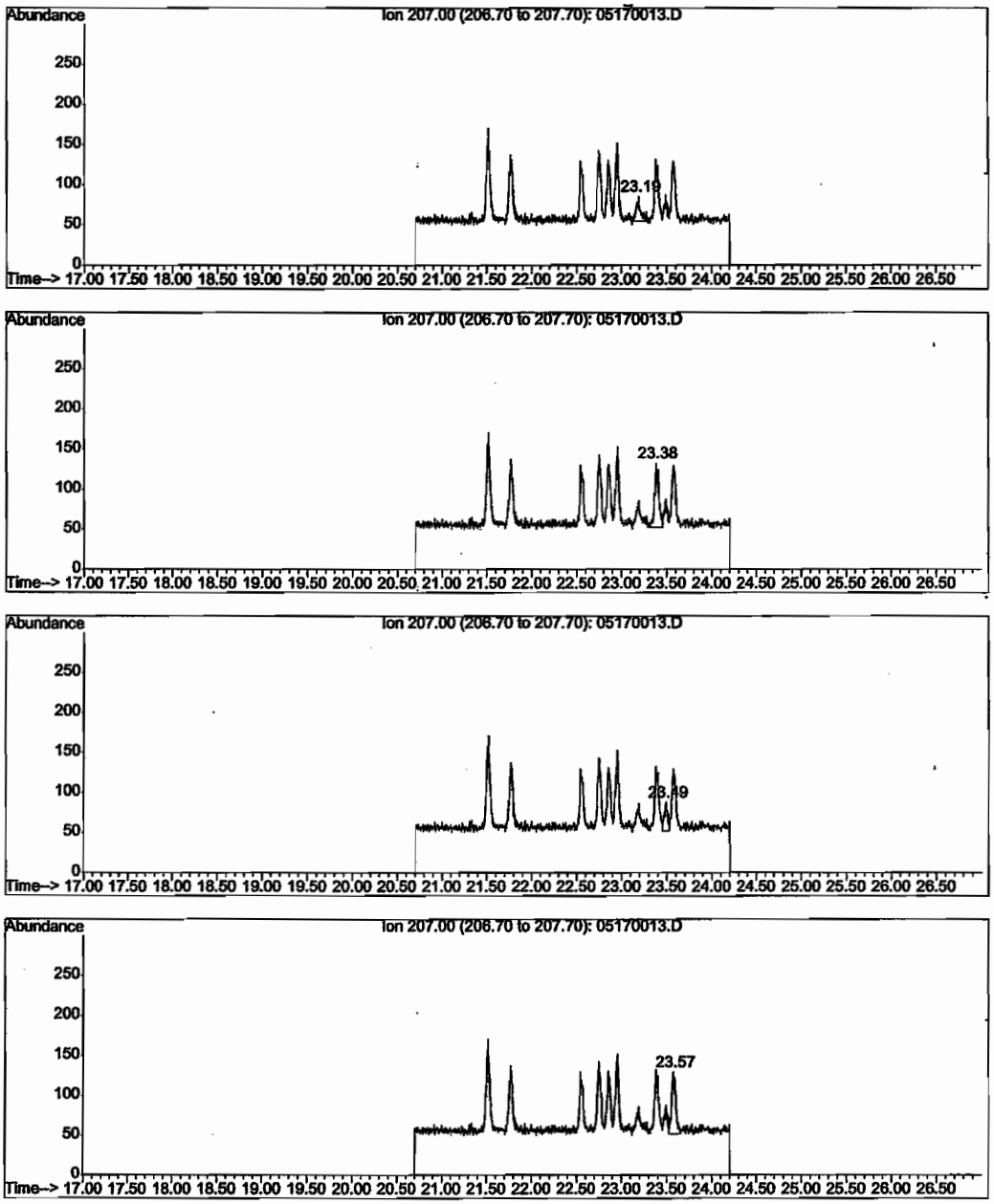
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Cyfluthrin
1.0 ng/mL
Peak Response (area): 8943

FIGURE 9. (con't) Representative Chromatograms – Bracketing Standards

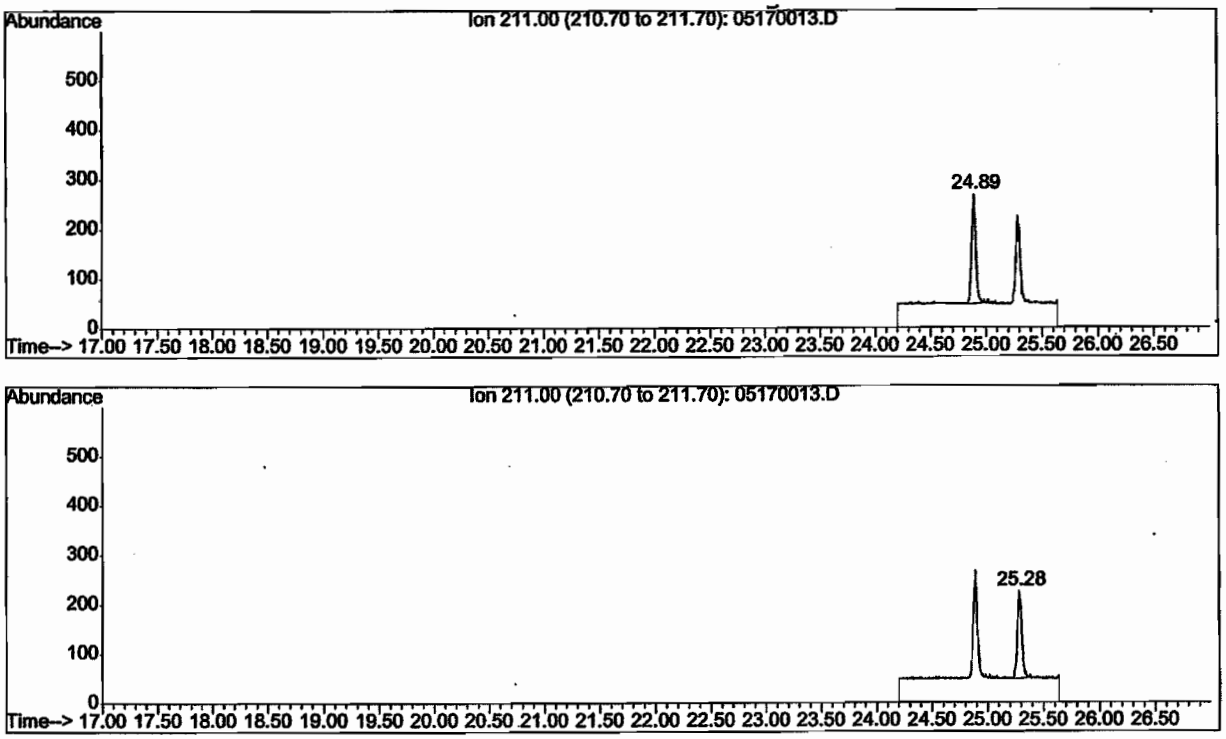
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Cypermethrin
1.0 ng/mL
Peak Response (area): 6684

FIGURE 9. (con't) Representative Chromatograms – Bracketing Standards

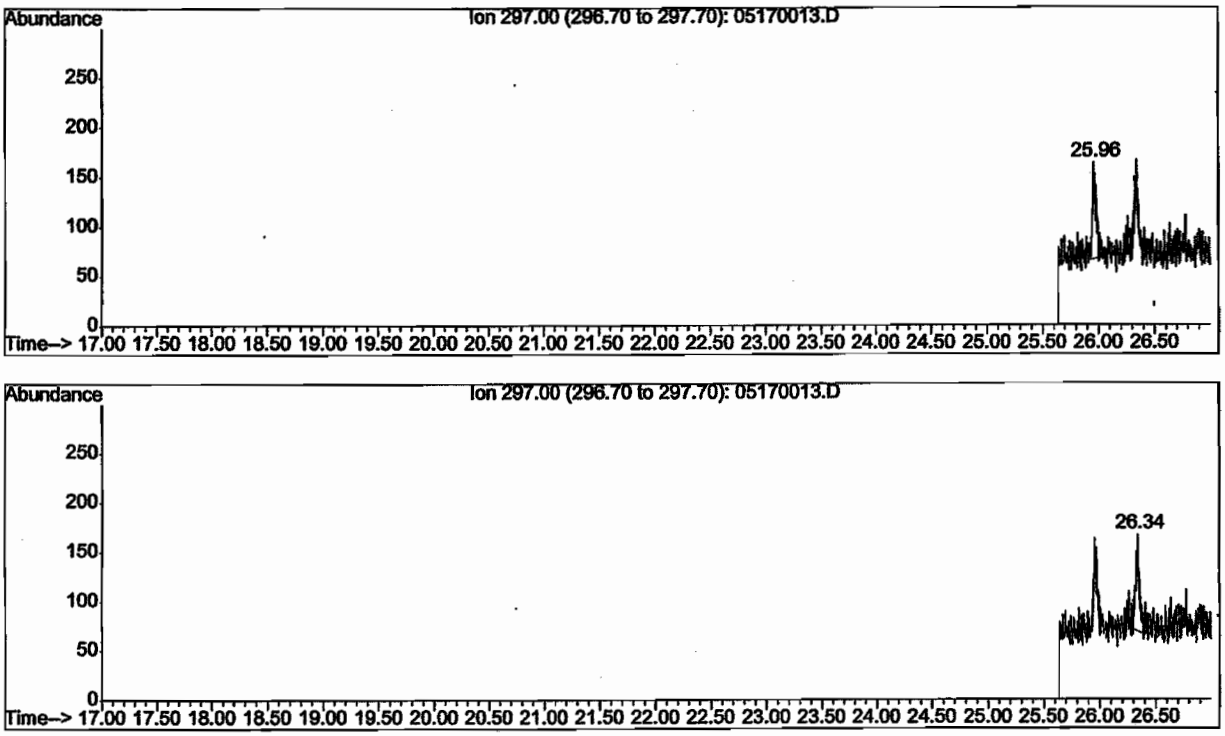
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Esfenvalerate
1.0 ng/mL
Peak Response (area): 10532

FIGURE 9. (con't) Representative Chromatograms – Bracketing Standards

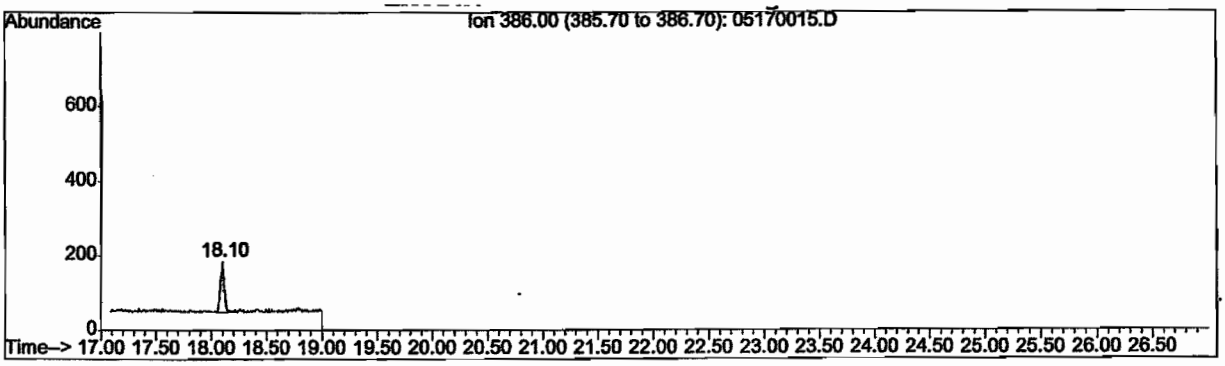
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



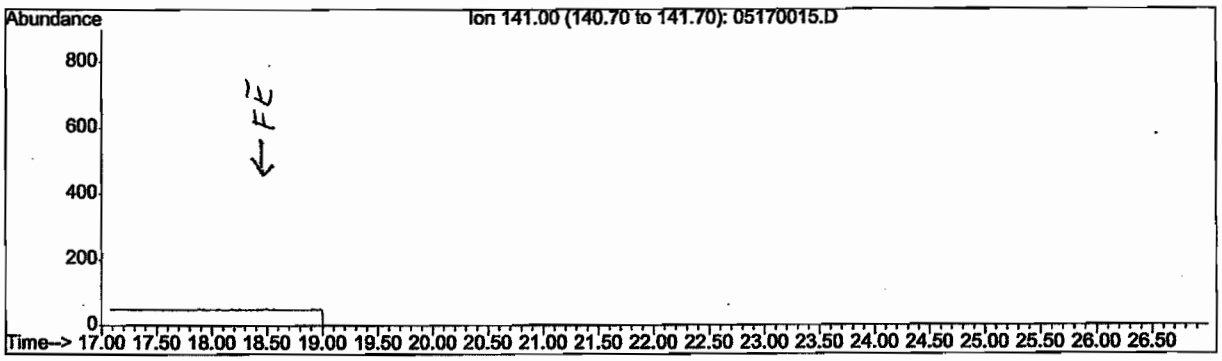
Deltamethrin
1.0 ng/mL
Peak Response (area): 4210

FIGURE 10. Representative Chromatograms - Estuarine Sediment, Untreated Control

Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL, Set #3



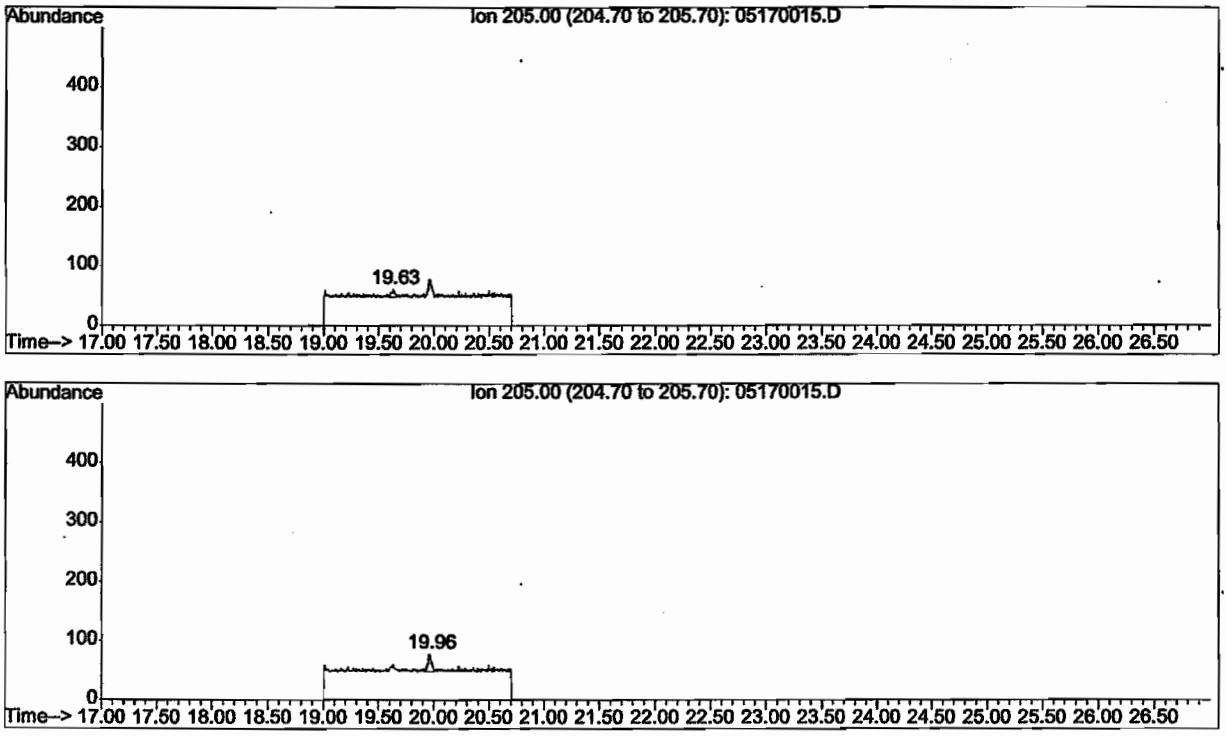
Bifenthrin
Peak Response (area): 3071
Bifenthrin Reported: <0.1 µg/kg



Fenpropathrin
Peak Response (area): 0
Fenpropathrin Reported: None Detected

FIGURE 10. (con't) Representative Chromatograms - Estuarine Sediment, Untreated Control

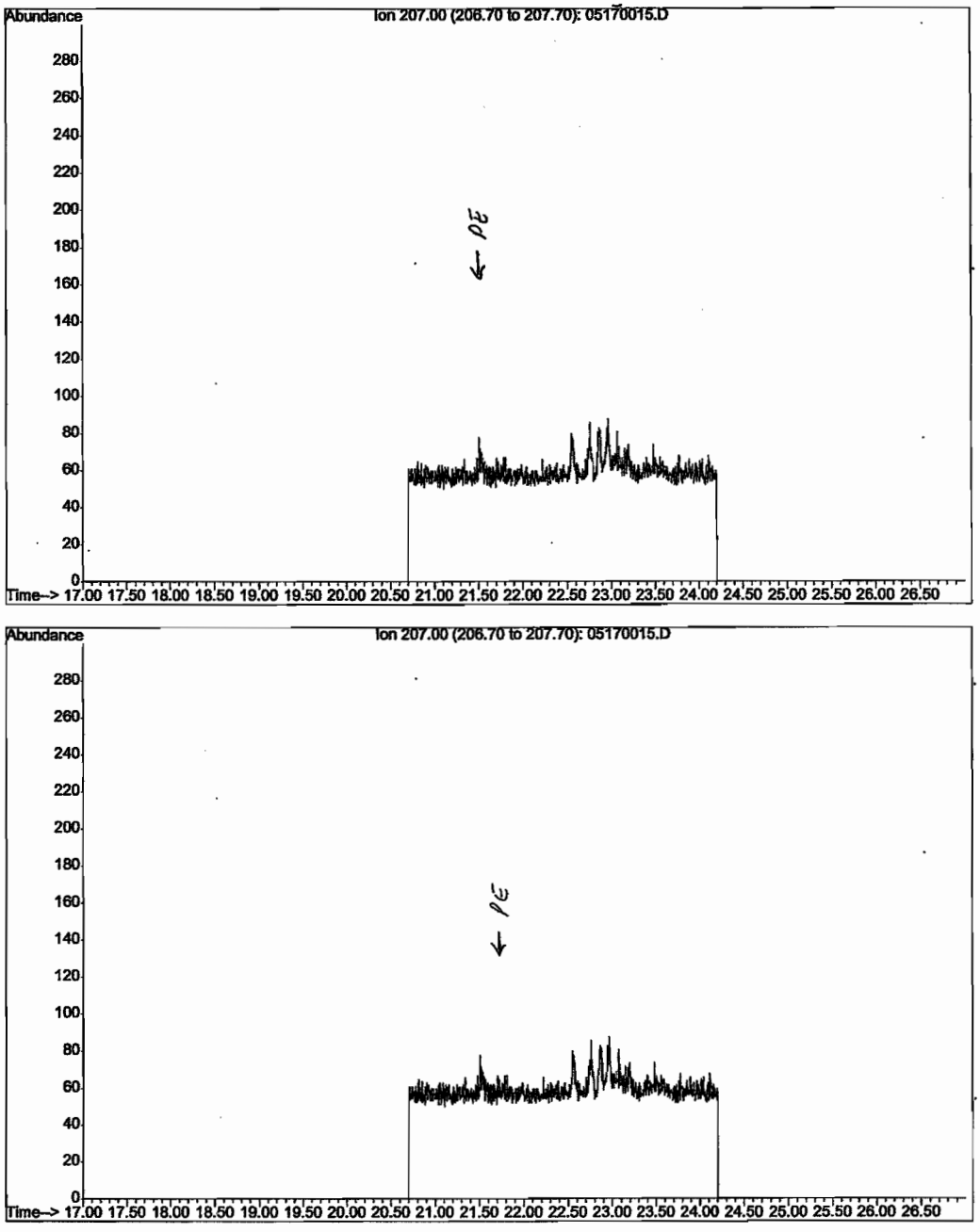
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL, Set #3



Lambda-cyhalothrin
Peak Response (area): 920
Lambda-cyhalothrin Reported: <0.1 µg/kg

FIGURE 10. (con't) Representative Chromatograms - Estuarine Sediment, Untreated Control

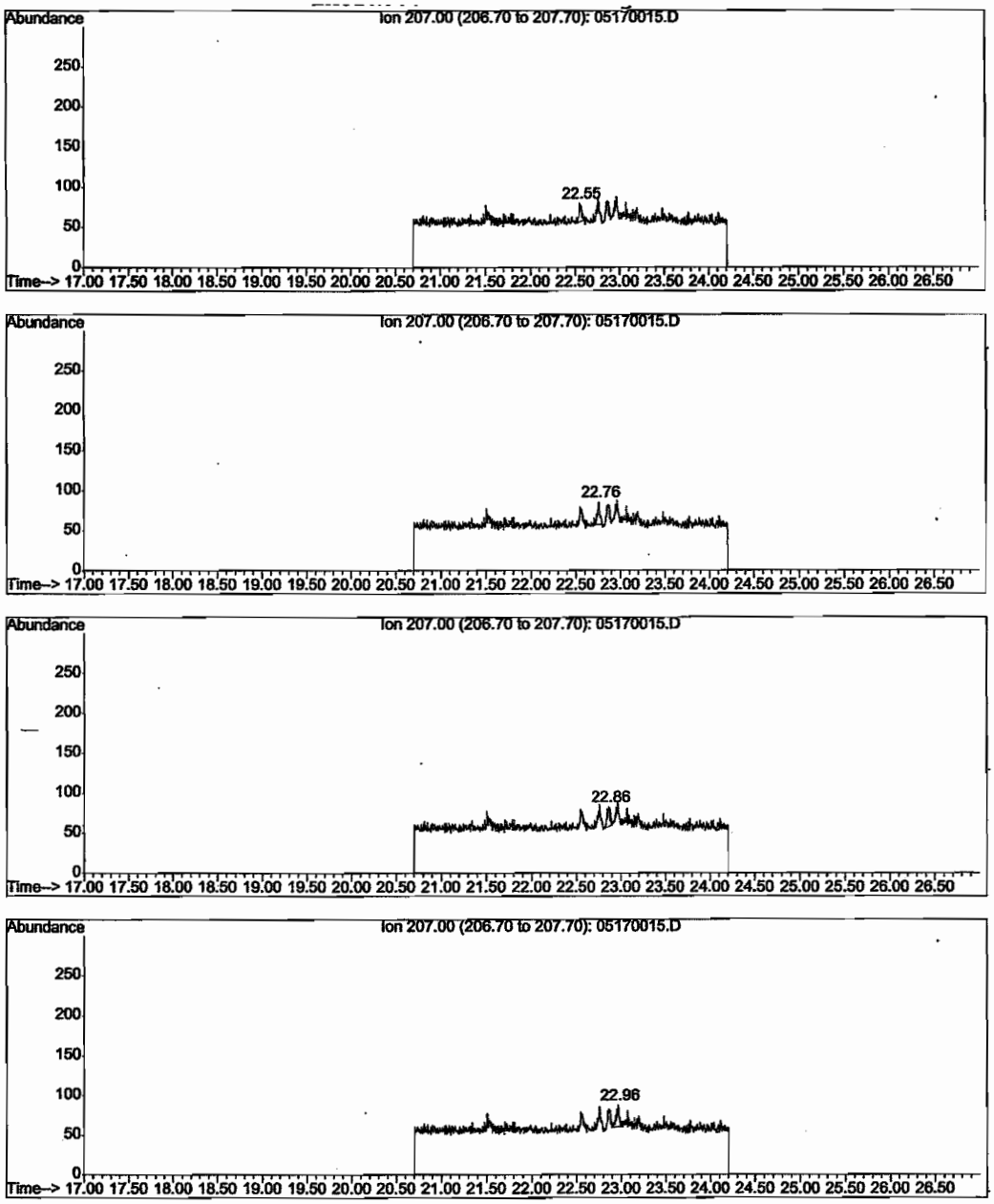
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL, Set #3



Permethrin
Peak Response (area): 0
Permethrin Reported: None Detected

FIGURE 10. (con't) Representative Chromatograms - Estuarine Sediment, Untreated Control

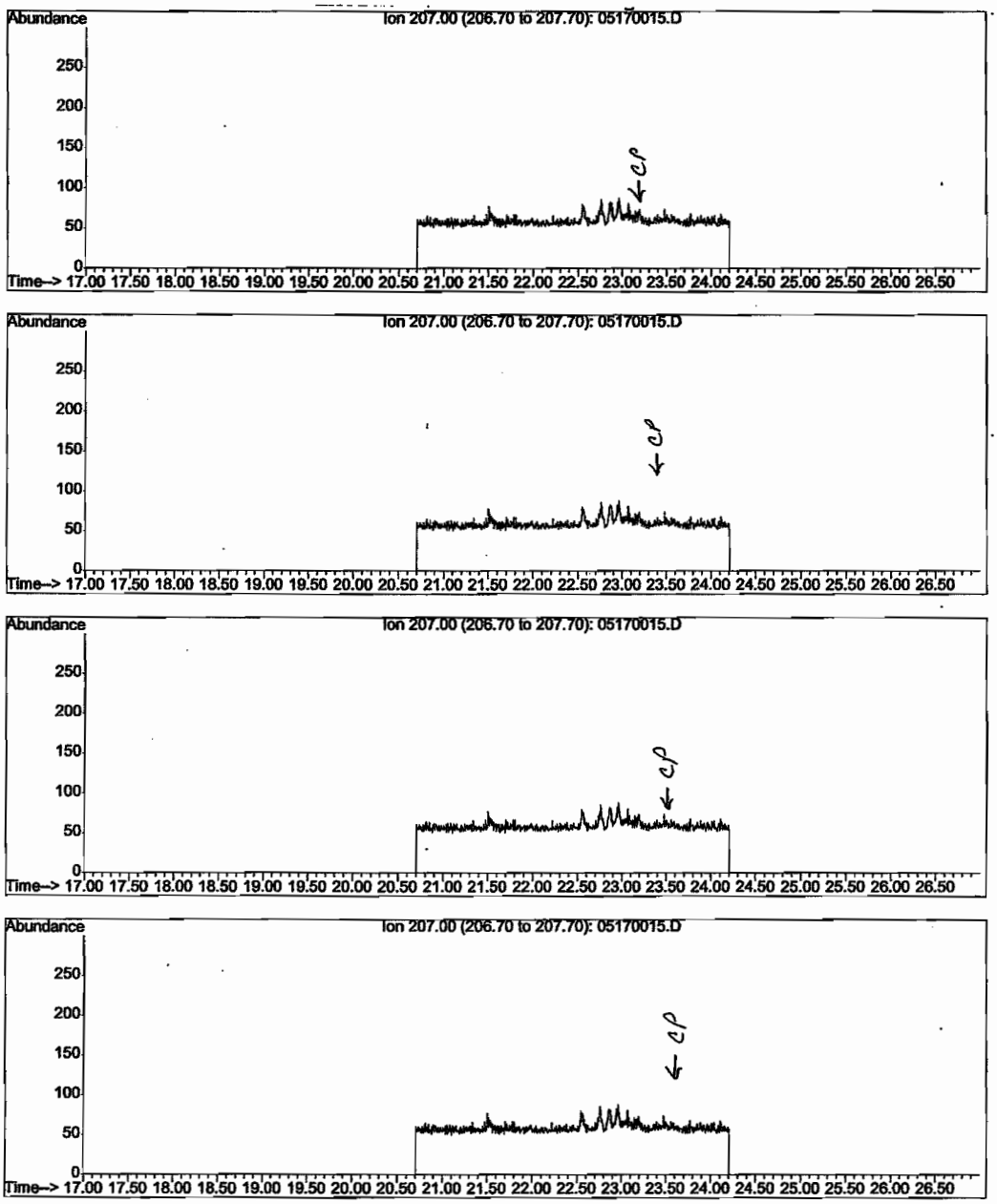
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL, Set #3



Cyfluthrin
Peak Response (area): 2029
Cyfluthrin Reported: <0.1 µg/kg

FIGURE 10. (con't) Representative Chromatograms - Estuarine Sediment, Untreated Control

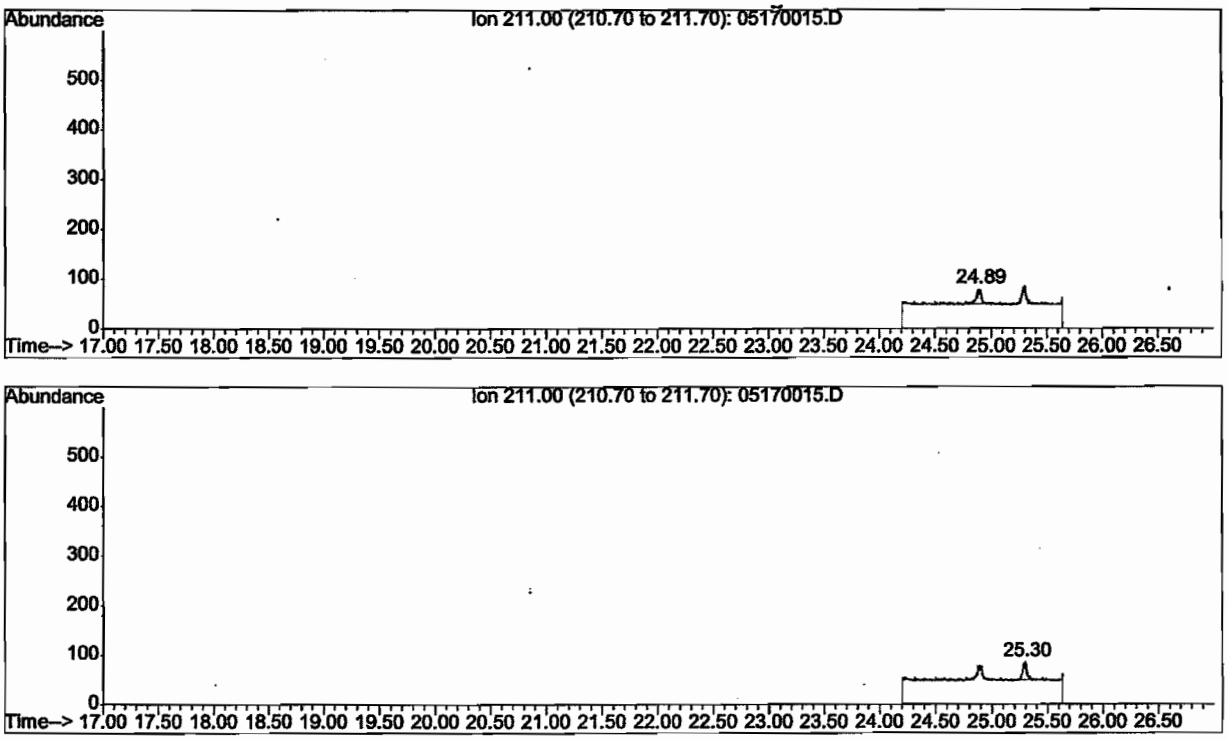
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL, Set #3



Cypermethrin
Peak Response (area): 0
Cypermethrin Reported: None Detected

FIGURE 10. (con't) Representative Chromatograms - Estuarine Sediment, Untreated Control

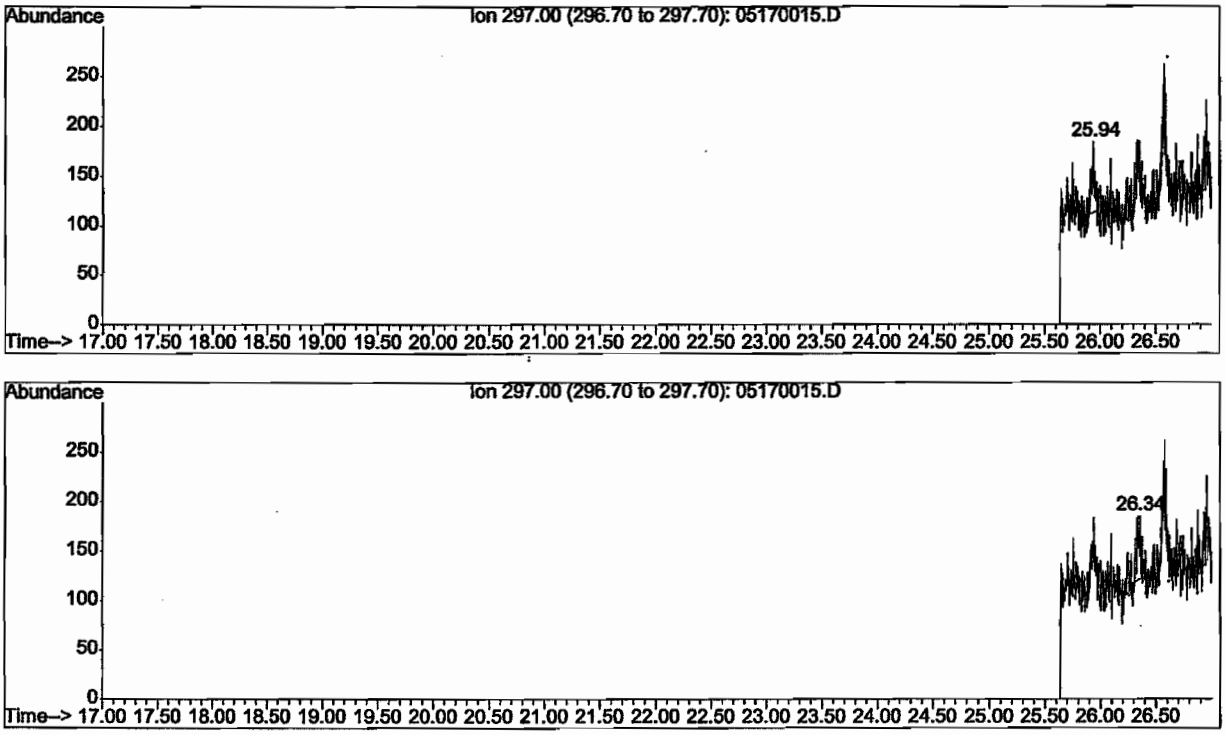
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL, Set #3



Esfenvalerate
Peak Response (area): 1671
Esfenvalerate Reported: <0.1 µg/kg

FIGURE 10. (con't) Representative Chromatograms - Estuarine Sediment, Untreated Control

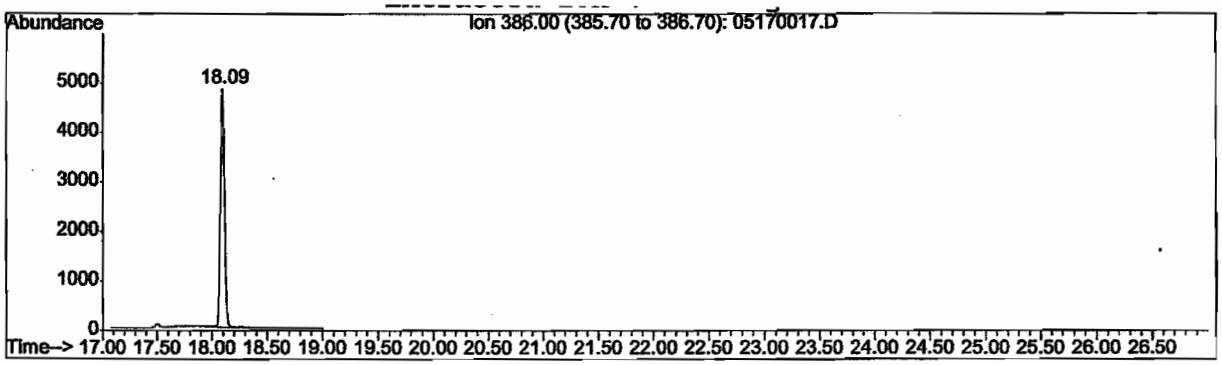
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL, Set #3



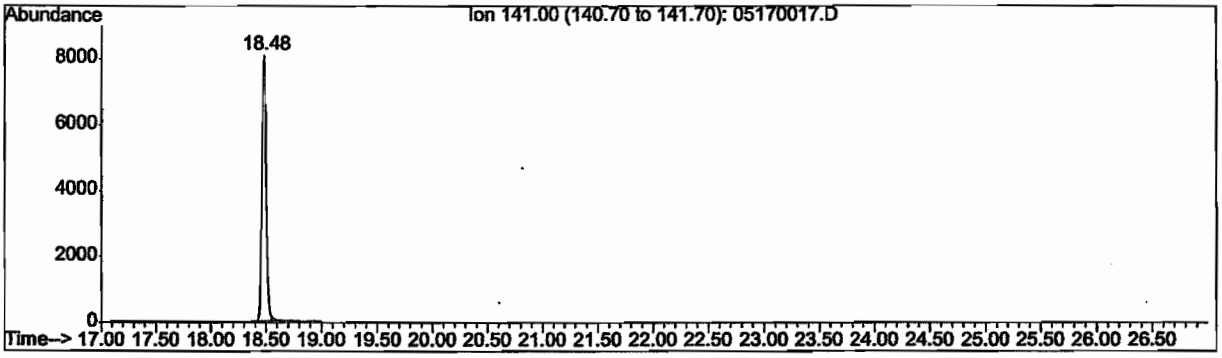
Deltamethrin
Peak Response (area): 2717
Deltamethrin Reported: <0.1 µg/kg

FIGURE 11. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



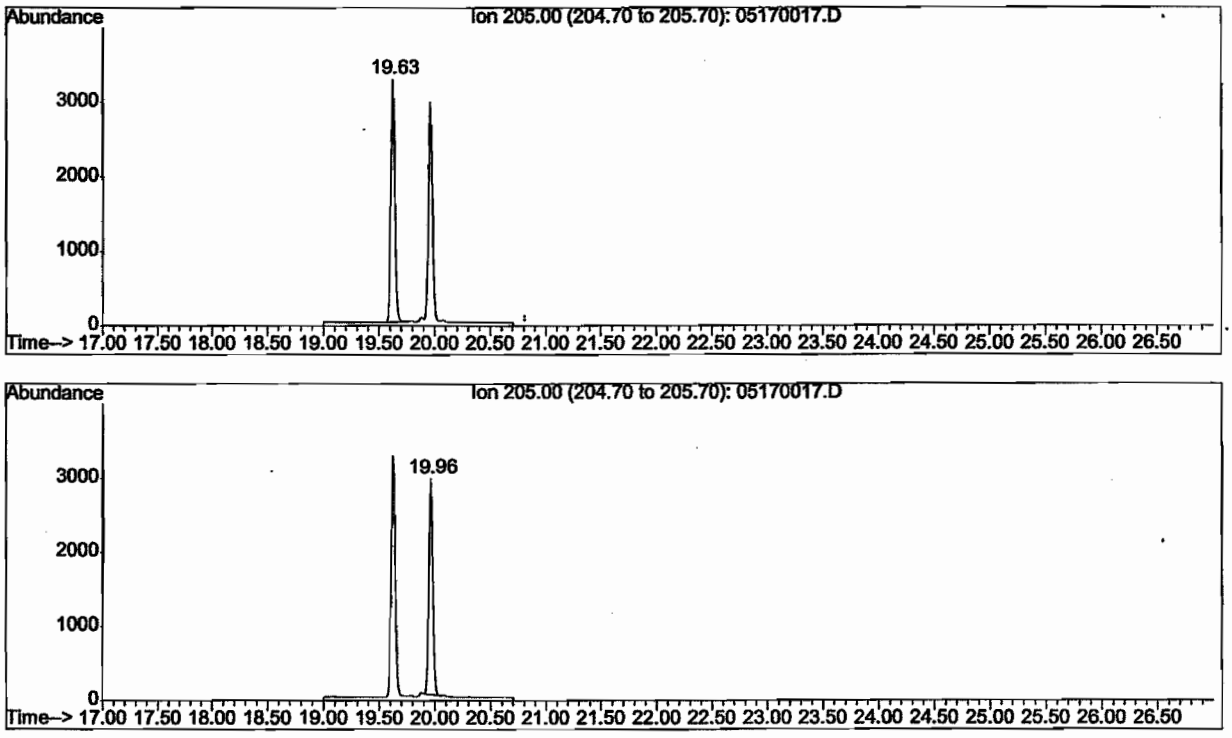
Bifenthrin
20 ng/mL
Peak Response (area): 121315



Fenpropathrin
20 ng/mL
Peak Response (area): 216355

FIGURE 11. (con't) Representative Chromatograms – Bracketing Standards

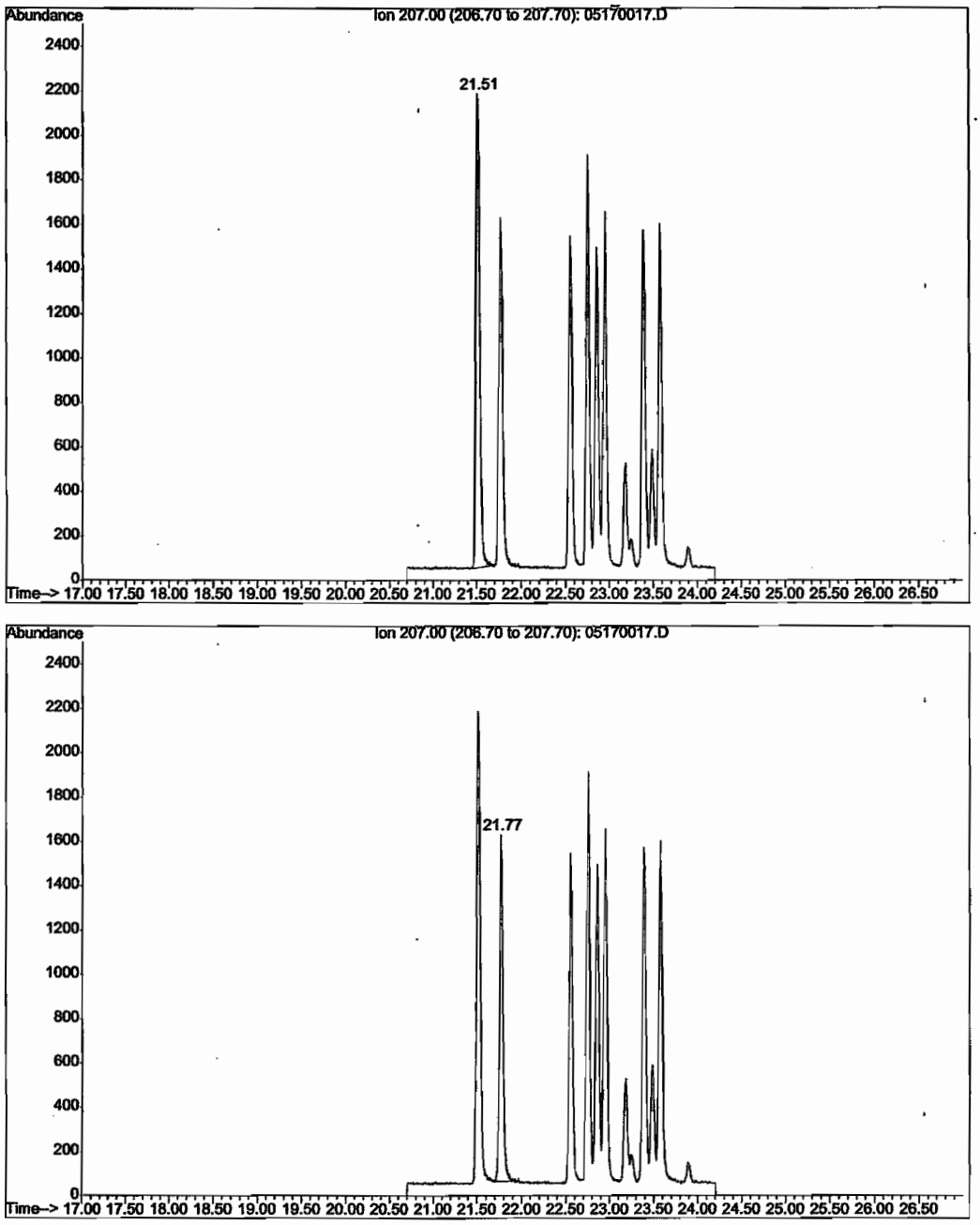
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 154964

FIGURE 11. (con't) Representative Chromatograms – Bracketing Standards

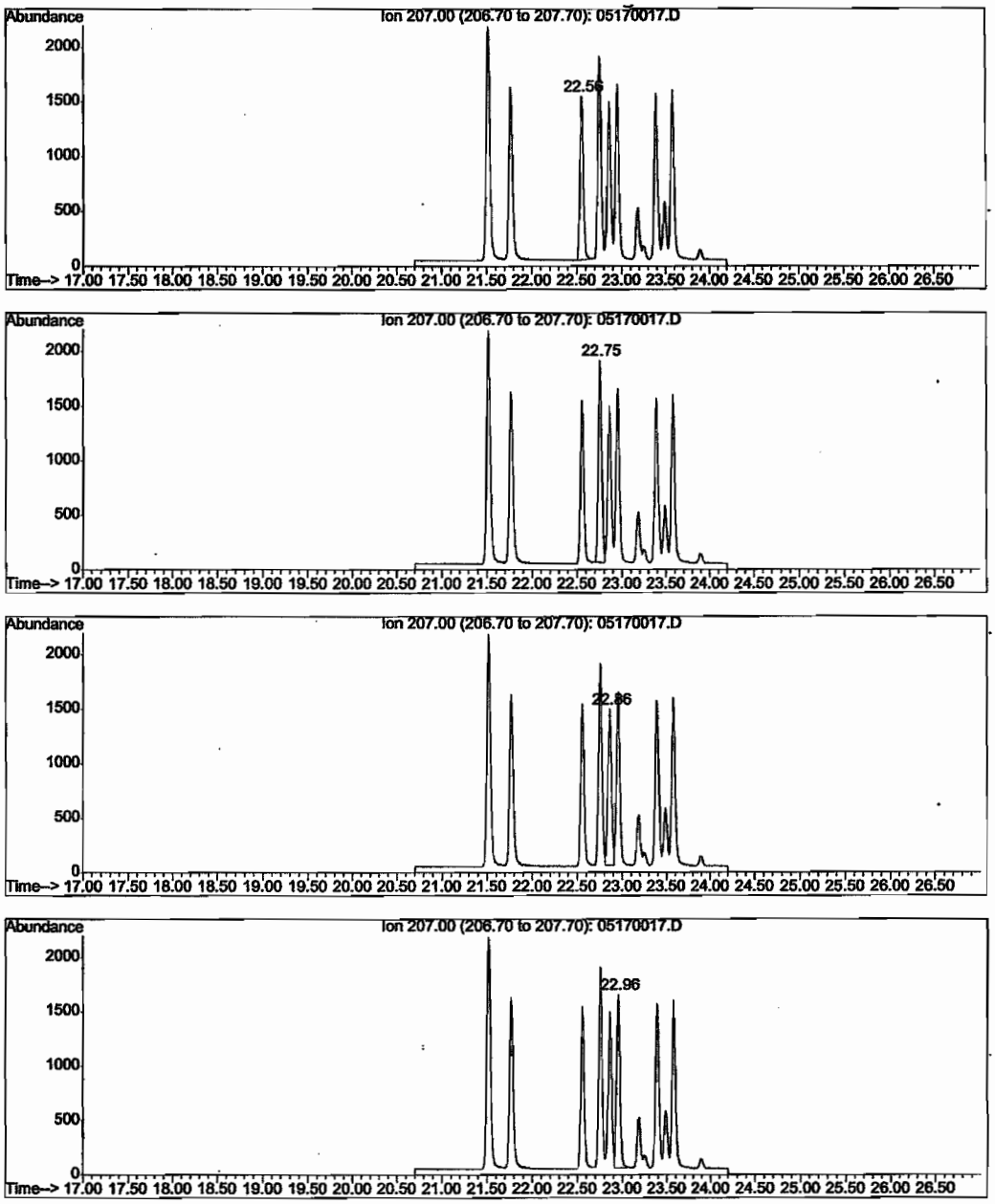
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



Permethrin
200 ng/mL
Peak Response (area): 105832

FIGURE 11. (con't) Representative Chromatograms – Bracketing Standards

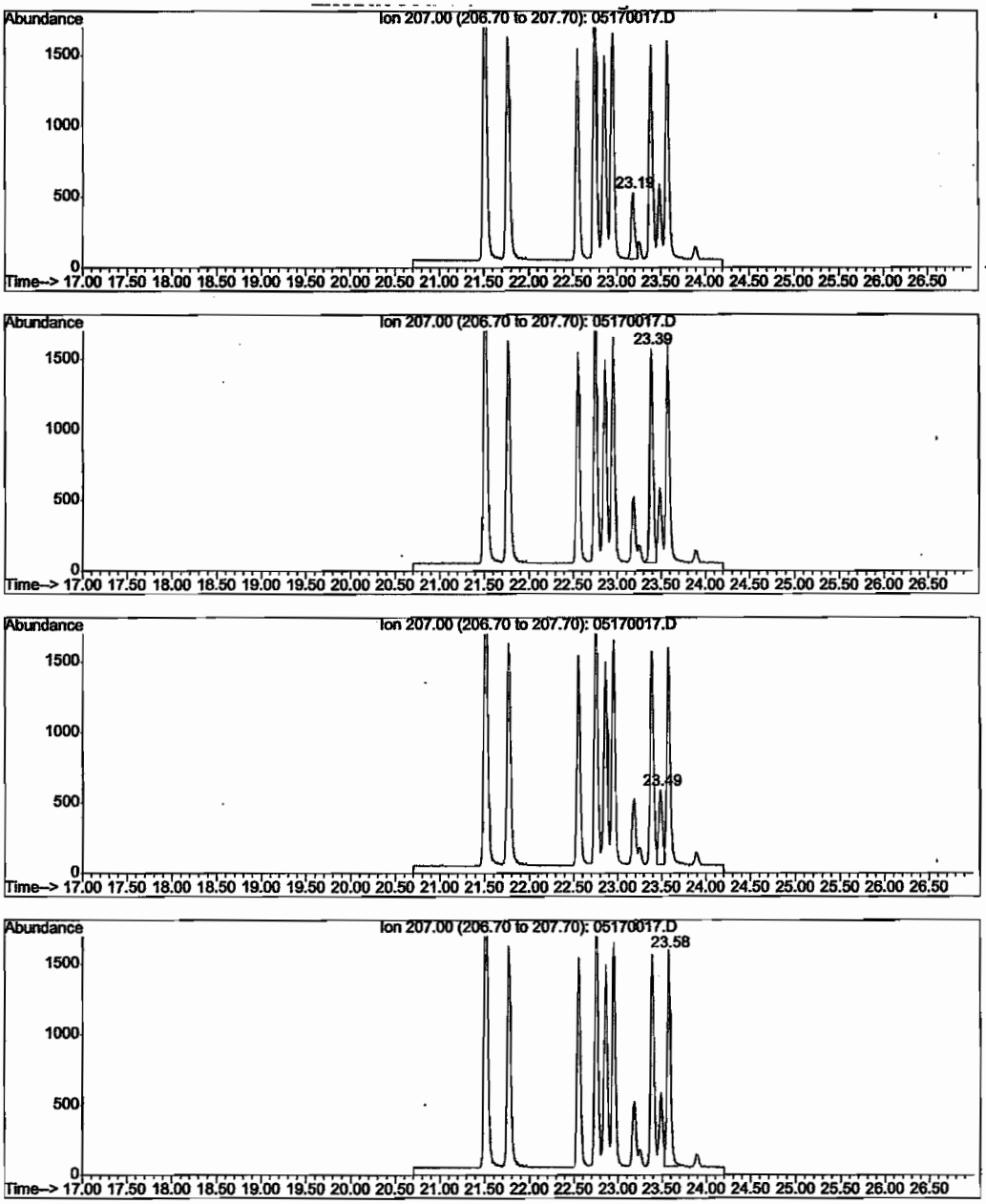
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



Cyfluthrin
20 ng/mL
Peak Response (area): 168645

FIGURE 11. (con't) Representative Chromatograms – Bracketing Standards

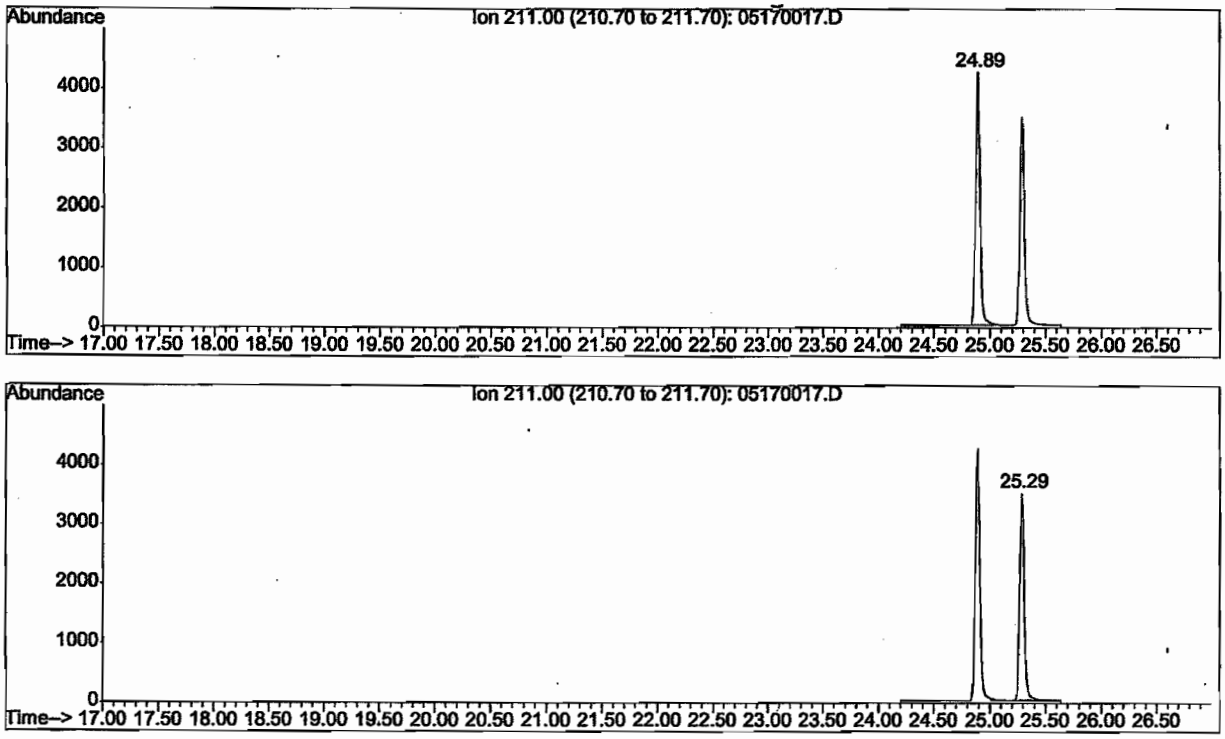
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



Cypermethrin
20 ng/mL
Peak Response (area): 111862

FIGURE 11. (con't) Representative Chromatograms – Bracketing Standards

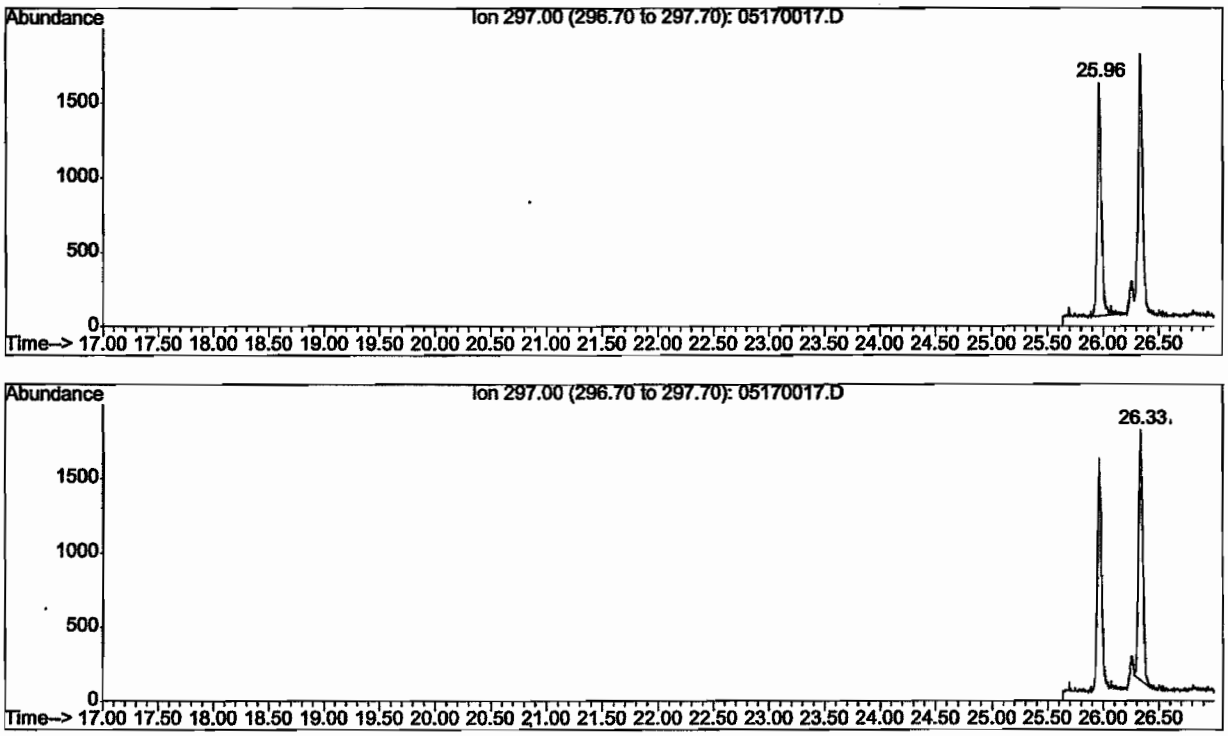
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



Esfenvalerate
20 ng/mL
Peak Response (area): 221658

FIGURE 11. (con't) Representative Chromatograms – Bracketing Standards

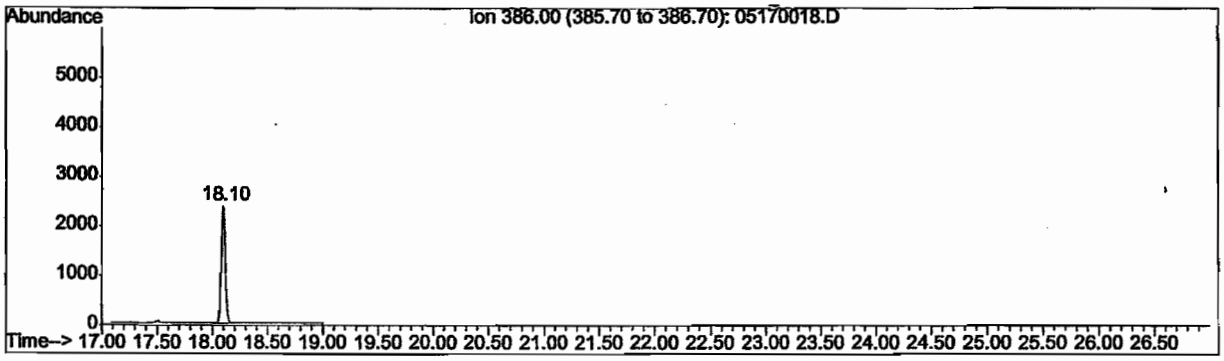
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



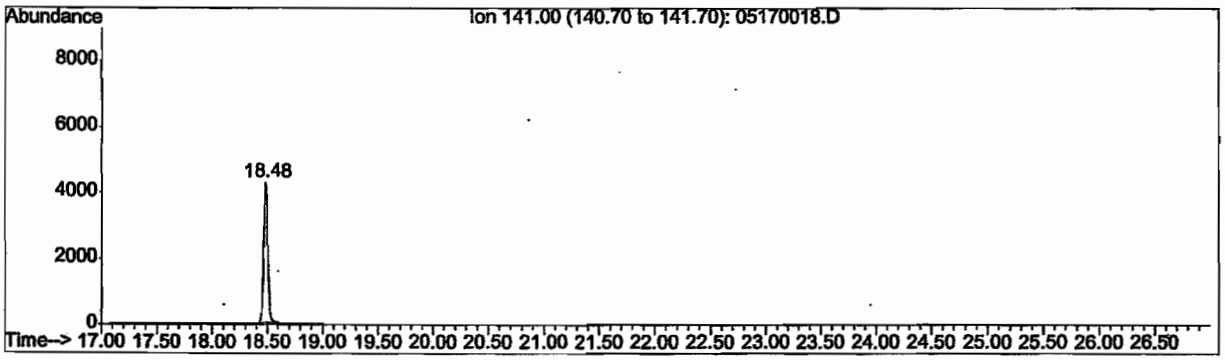
Deltamethrin
20 ng/mL
Peak Response (area): 85238

FIGURE 12. Representative Chromatograms - Estuarine Sediment, Fortified Control (10×LOQ)

Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #3



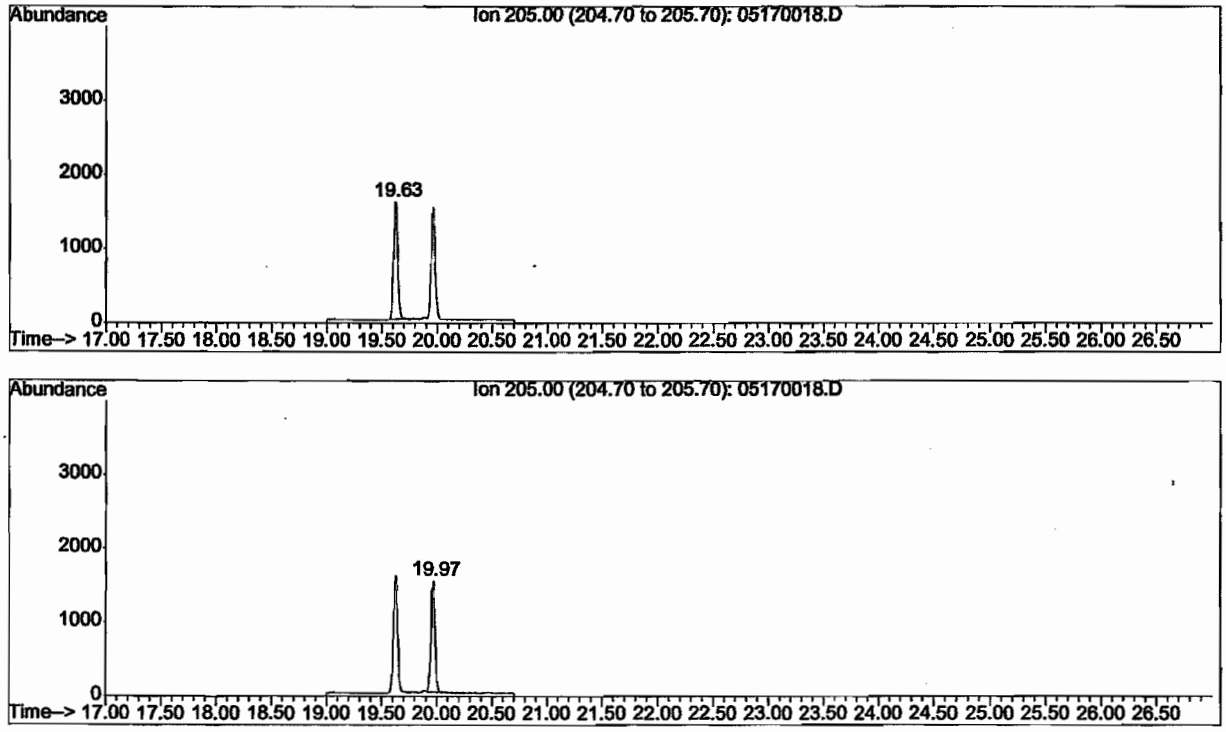
Bifenthrin
Peak Response (area): 60105
Bifenthrin Reported Recovery: 93%



Fenpropathrin
Peak Response (area): 108530
Fenpropathrin Reported Recovery: 98%

FIGURE 12. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (10×LOQ)

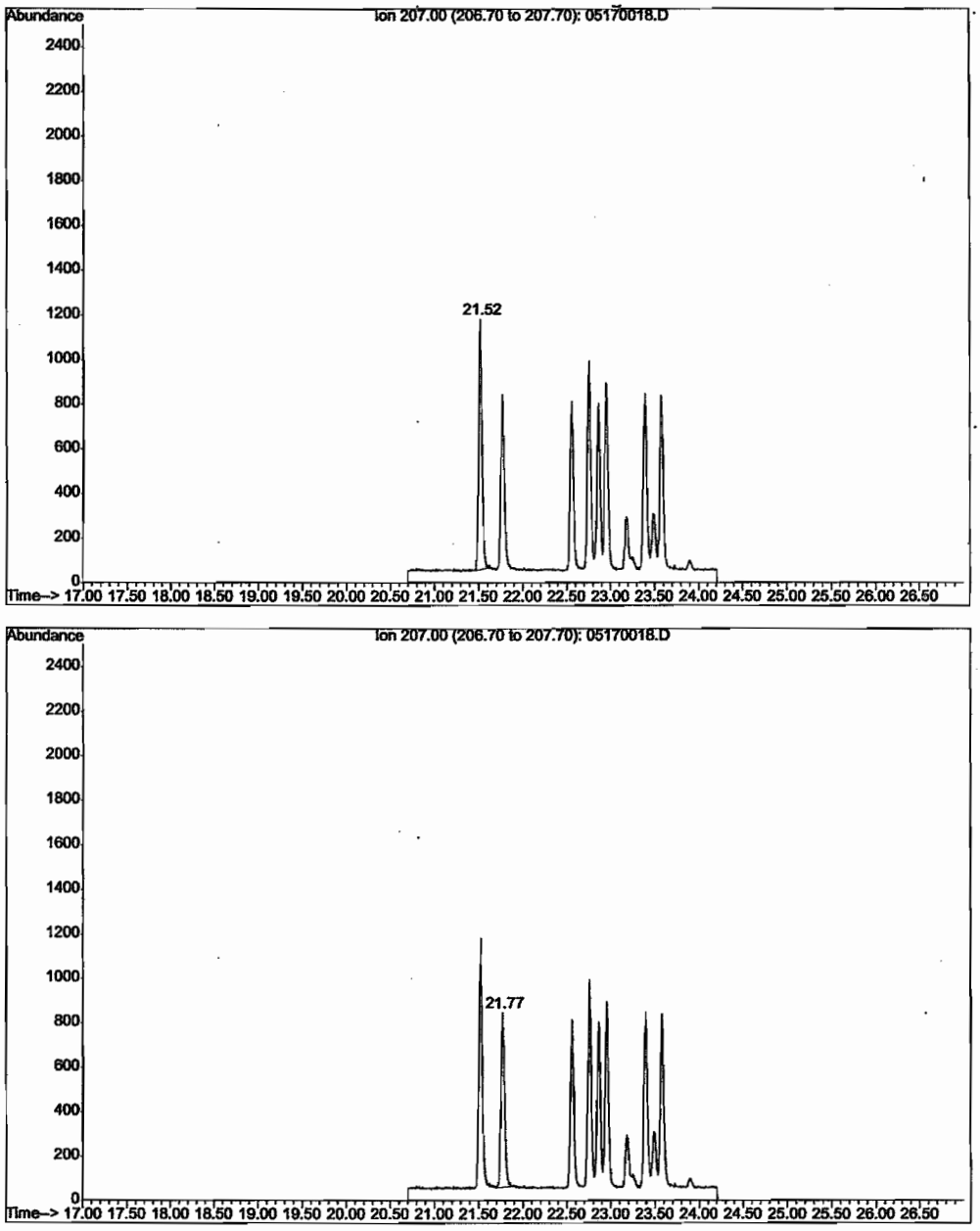
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #3



Lambda-cyhalothrin
Peak Response (area): 77659
Lambda-cyhalothrin Reported Recovery: 97%

FIGURE 12. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (10×LOQ)

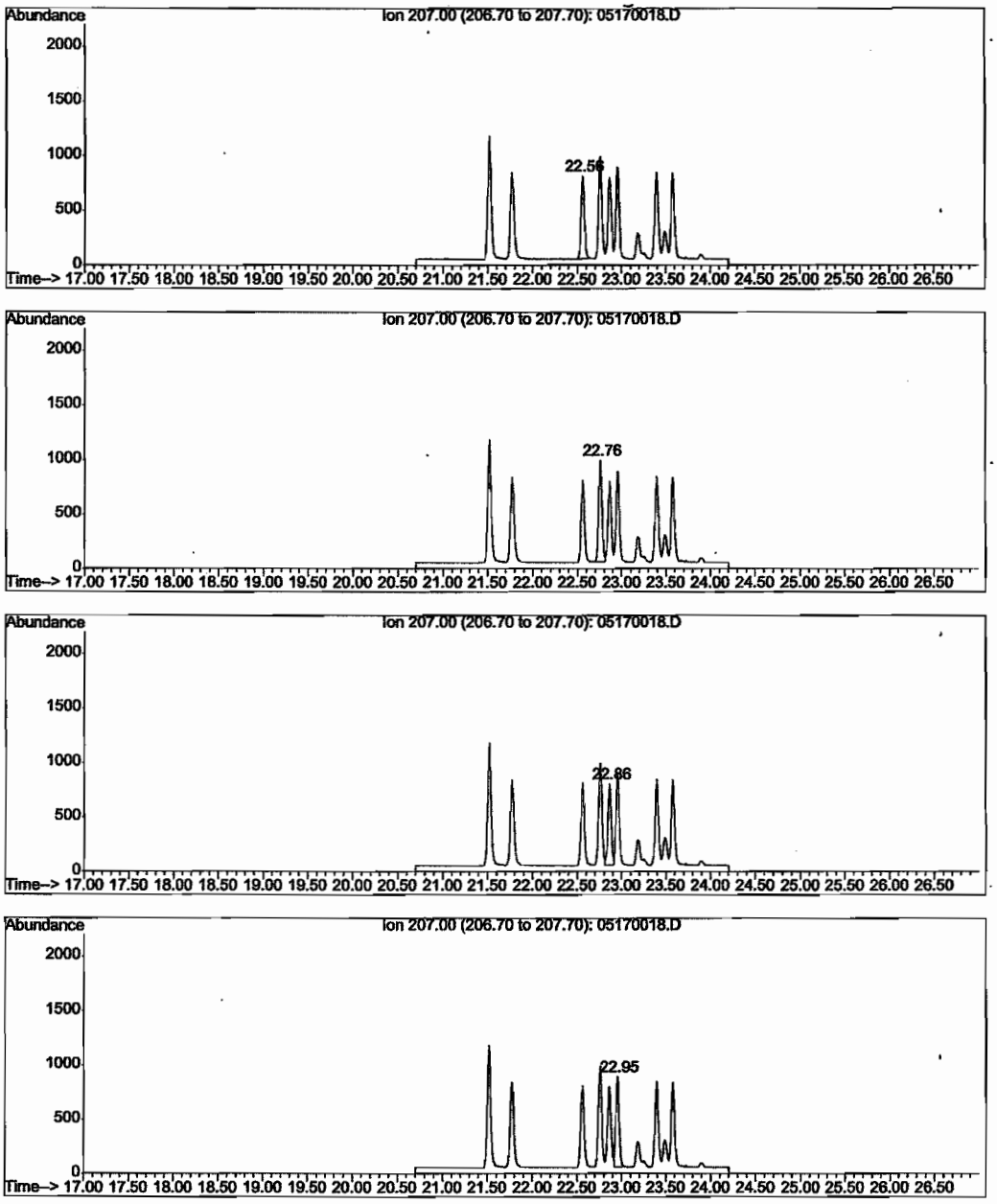
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #3



Permethrin
Peak Response (area): 51714
Permethrin Reported Recovery: 96%

FIGURE 12. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (10×LOQ)

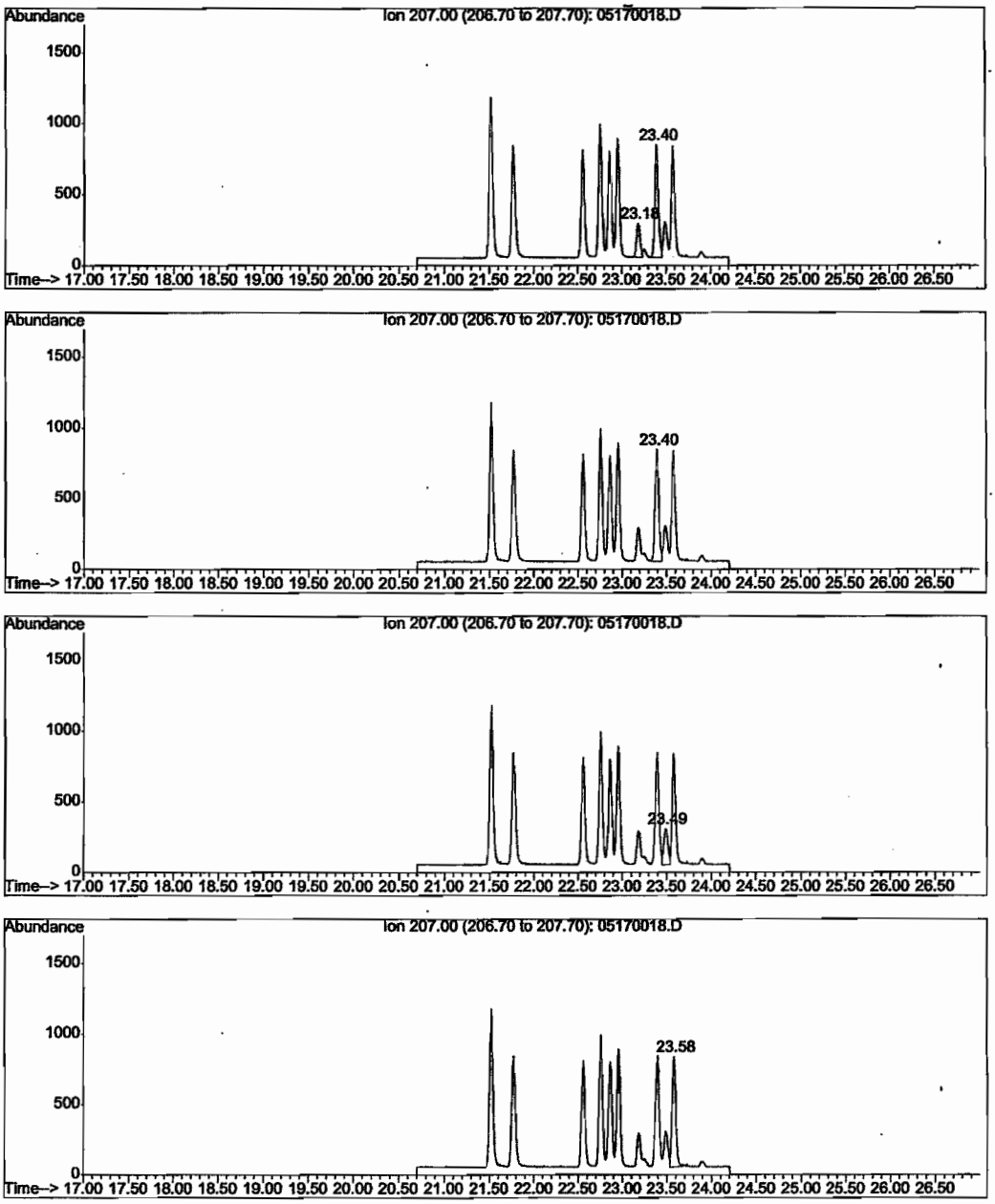
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #3



Cyfluthrin
Peak Response (area): 85979
Cyfluthrin Reported Recovery: 98%

FIGURE 12. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (10×LOQ)

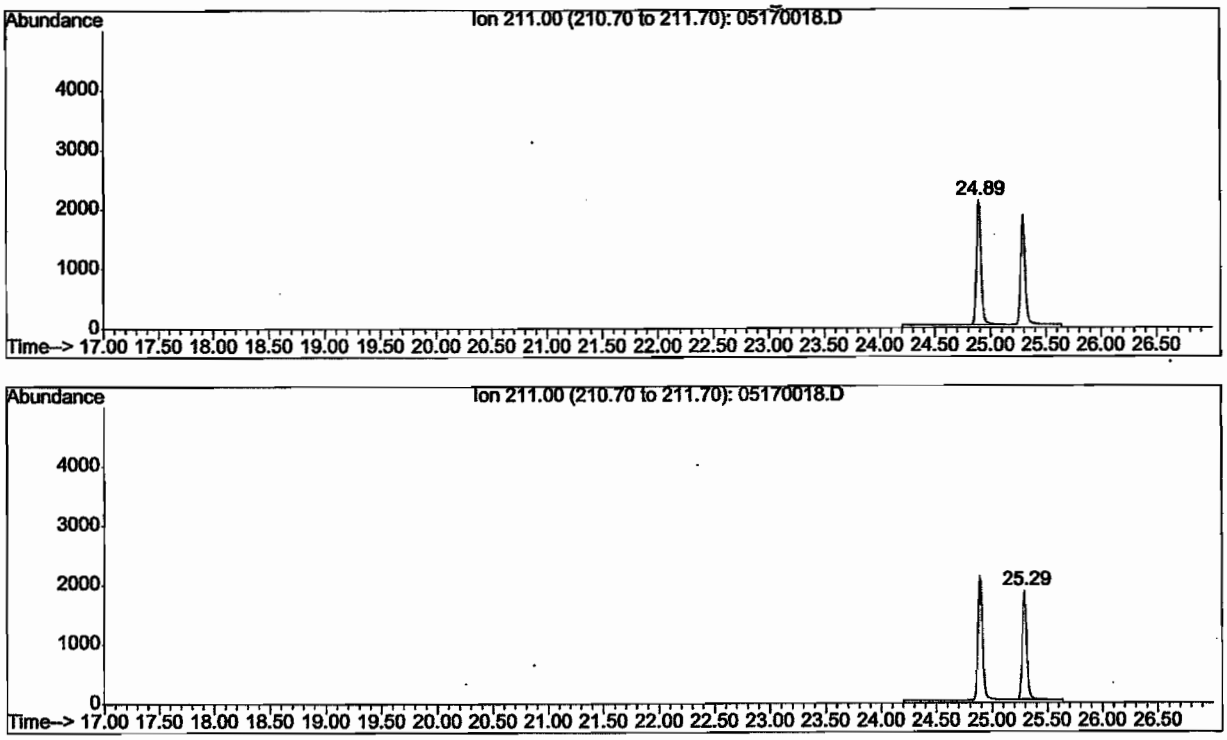
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #3



Cypermethrin
Peak Response (area): 58299
Cypermethrin Reported Recovery: 102%

FIGURE 12. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (10×LOQ)

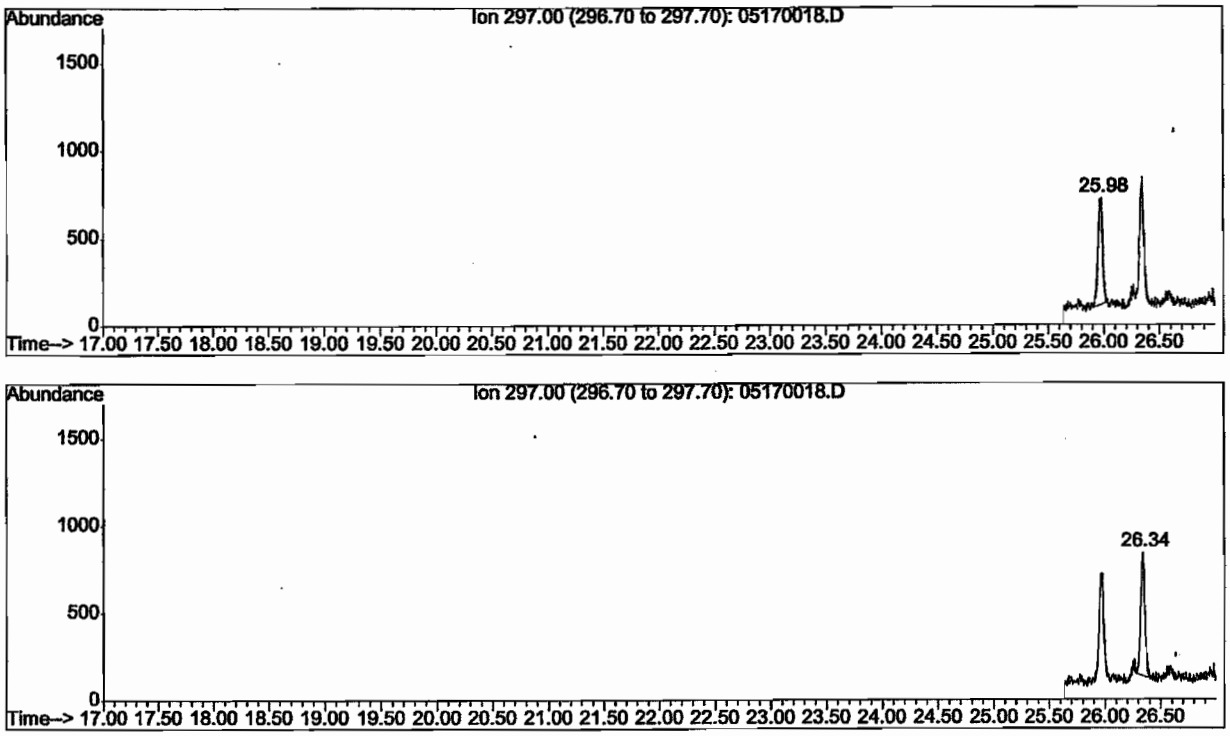
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #3



Esfenvalerate
Peak Response (area): 110281
Esfenvalerate Reported Recovery: 95%

FIGURE 12. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (10×LOQ)

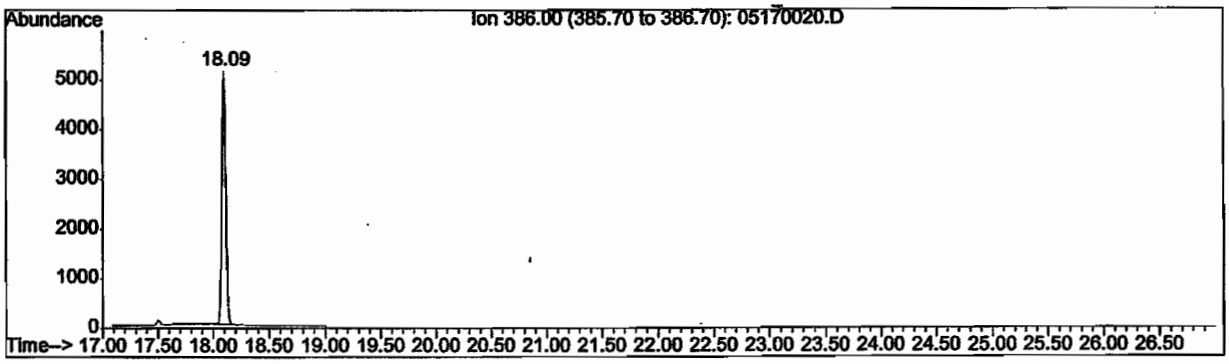
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL, Set #3



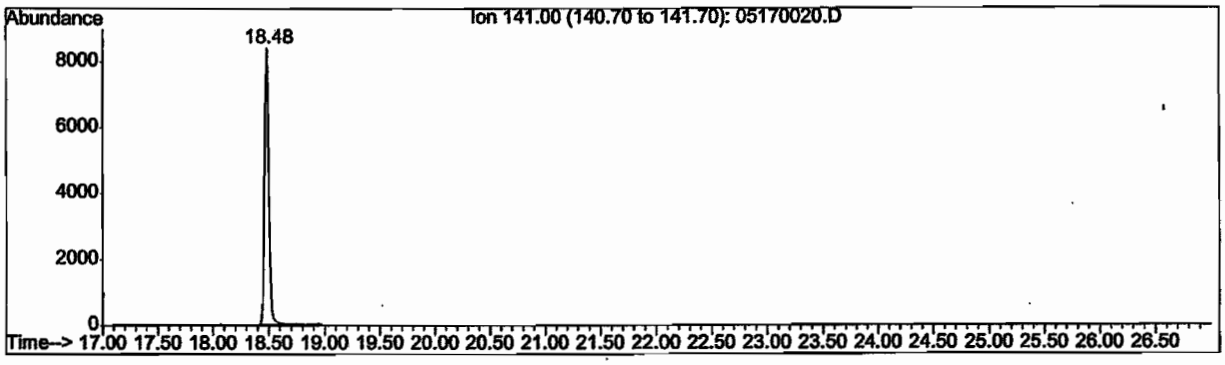
Deltamethrin
Peak Response (area): 35683
Deltamethrin Reported Recovery: 74 %

FIGURE 13. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



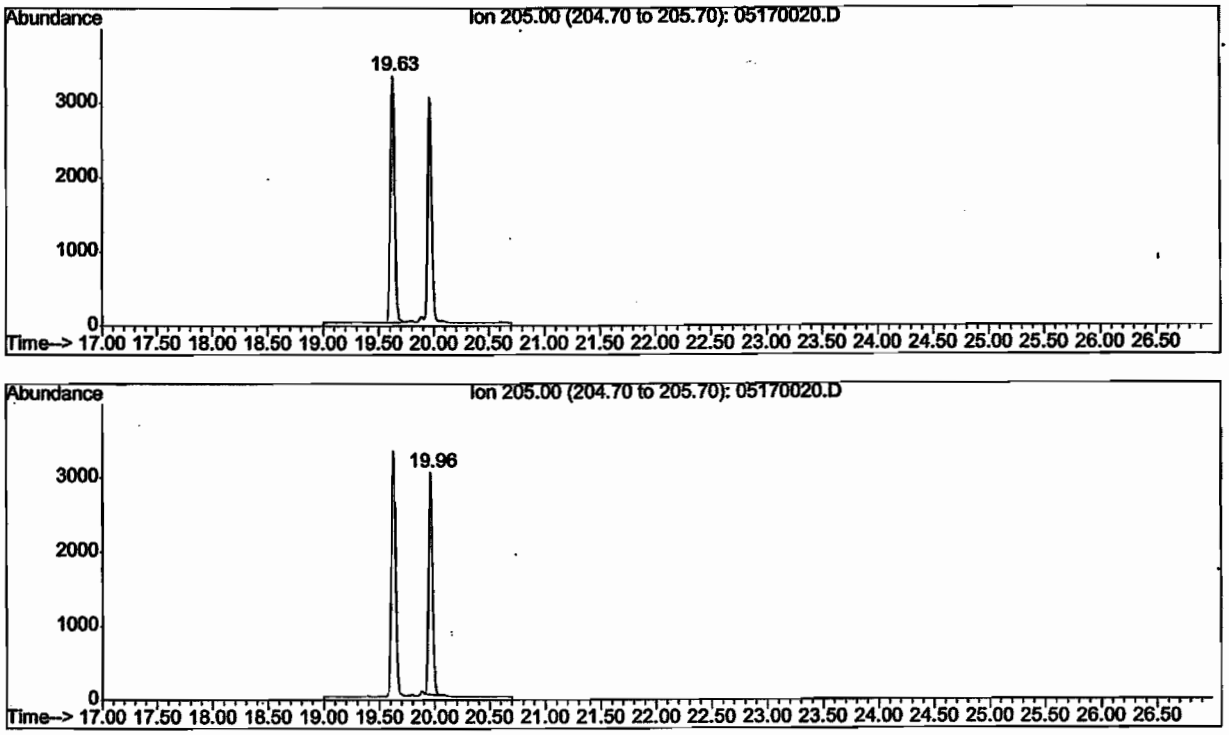
Bifenthrin
20 ng/mL
Peak Response (area): 124239



Fenpropathrin
20 ng/mL
Peak Response (area): 224573

FIGURE 13. (con't) Representative Chromatograms – Bracketing Standards

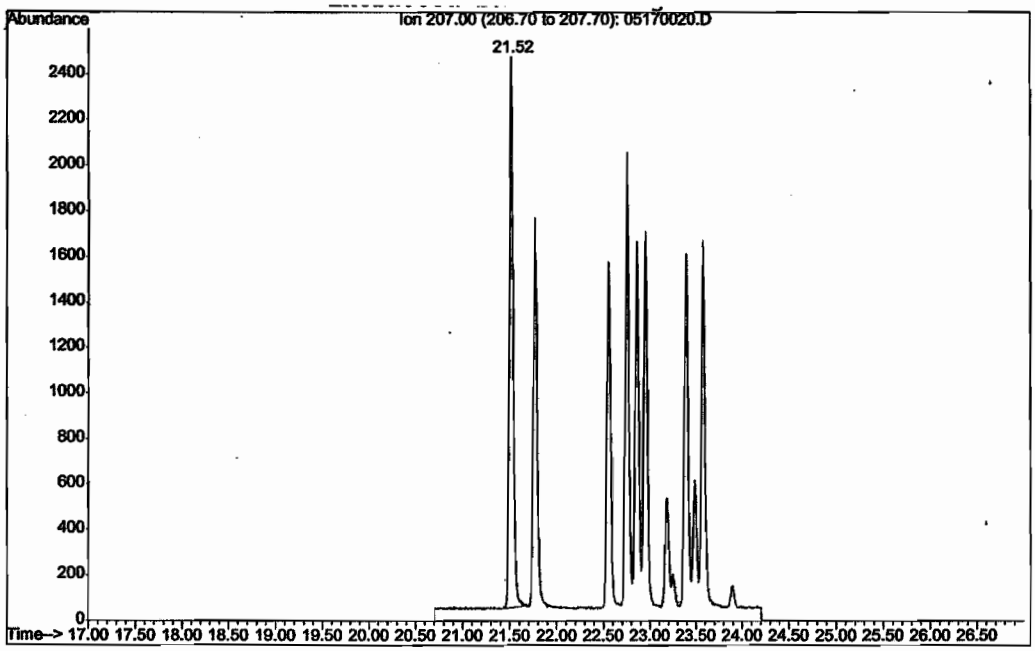
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3

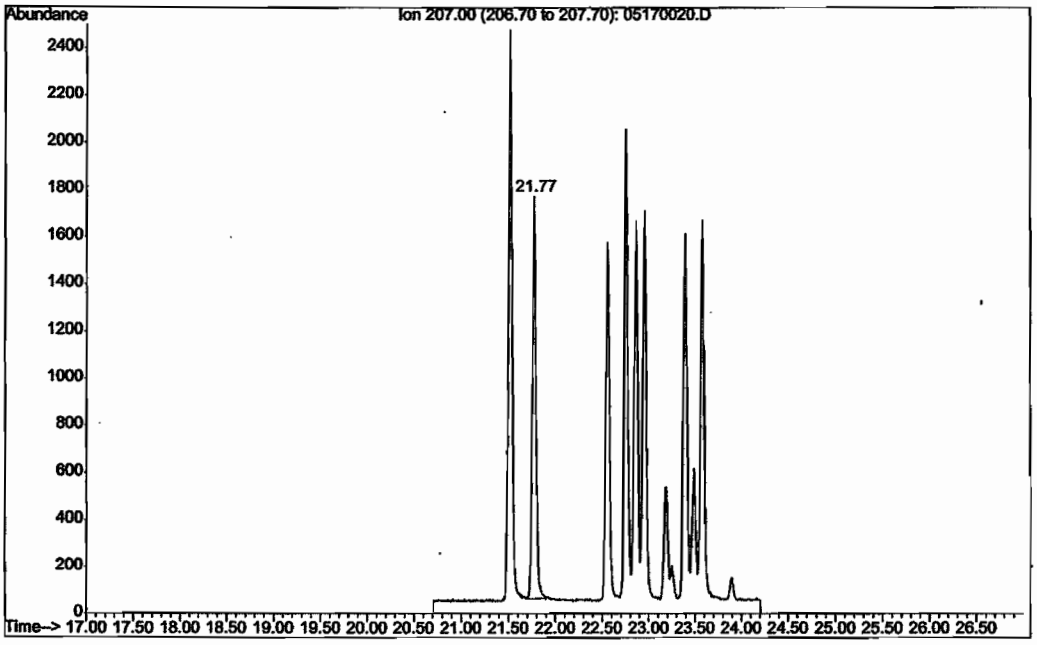


Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 162527

FIGURE 13. (con't) Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3

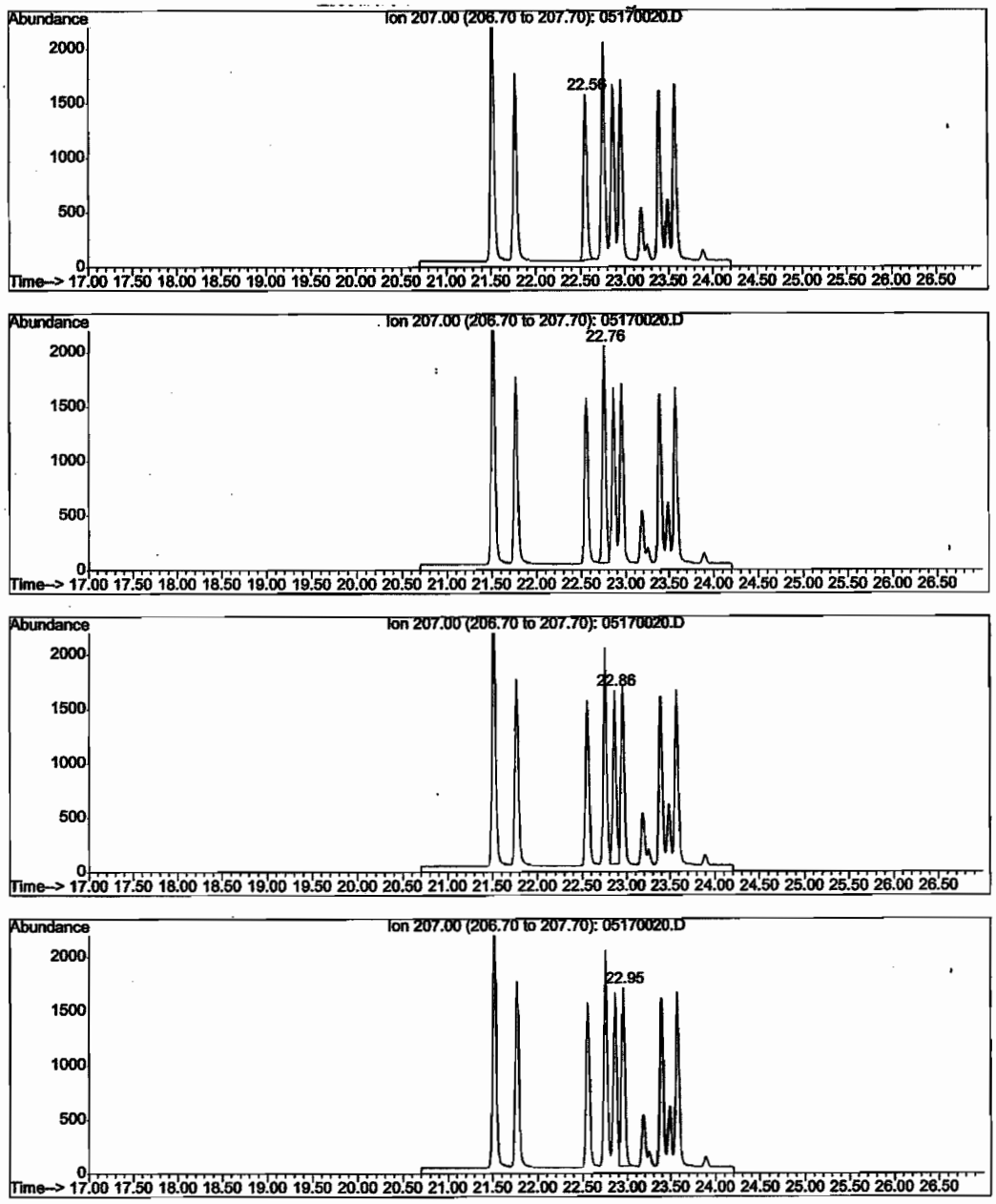




Permethrin
200 ng/mL
Peak Response (area): 110099

FIGURE 13. (con't) Representative Chromatograms – Bracketing Standards

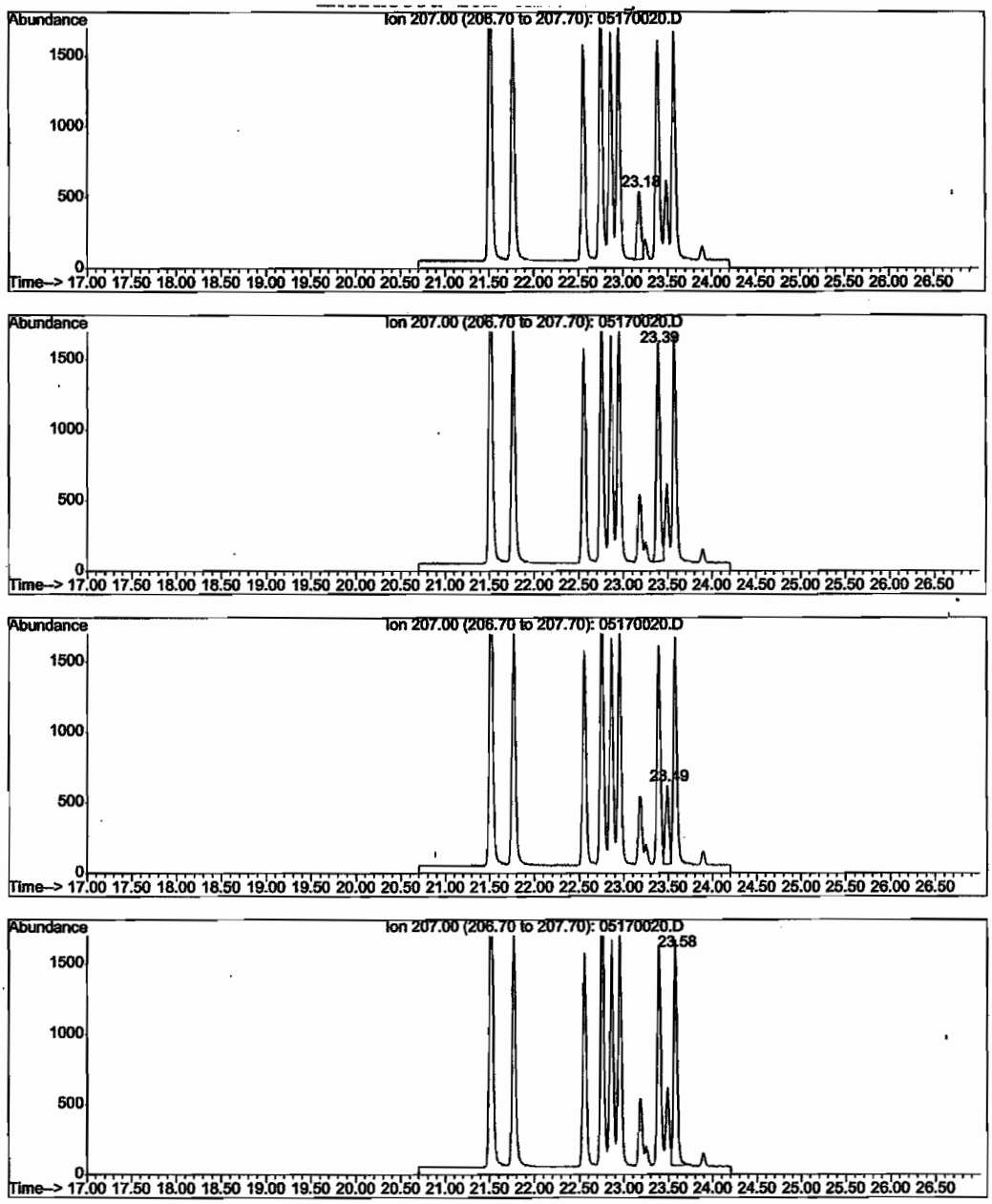
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



Cyfluthrin
20 ng/mL
Peak Response (area): 178012

FIGURE 13. (con't) Representative Chromatograms – Bracketing Standards

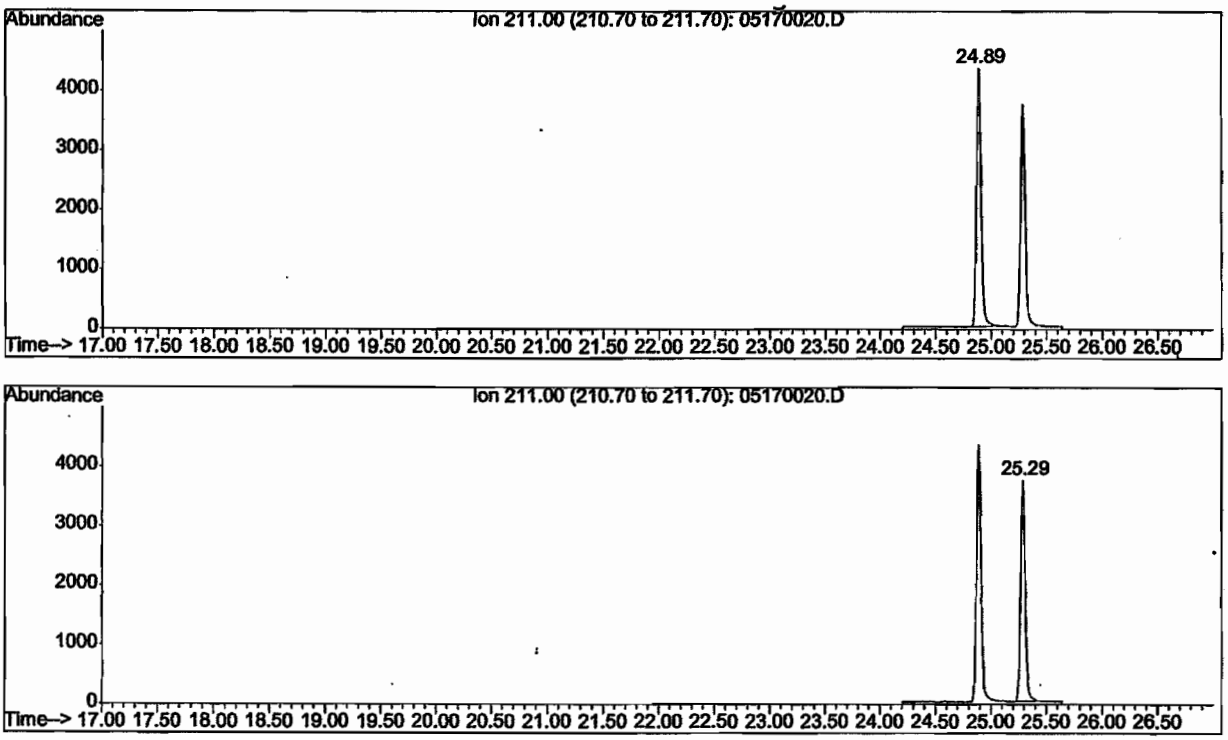
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



Cypermethrin
20 ng/mL
Peak Response (area): 117395

FIGURE 13. (con't) Representative Chromatograms – Bracketing Standards

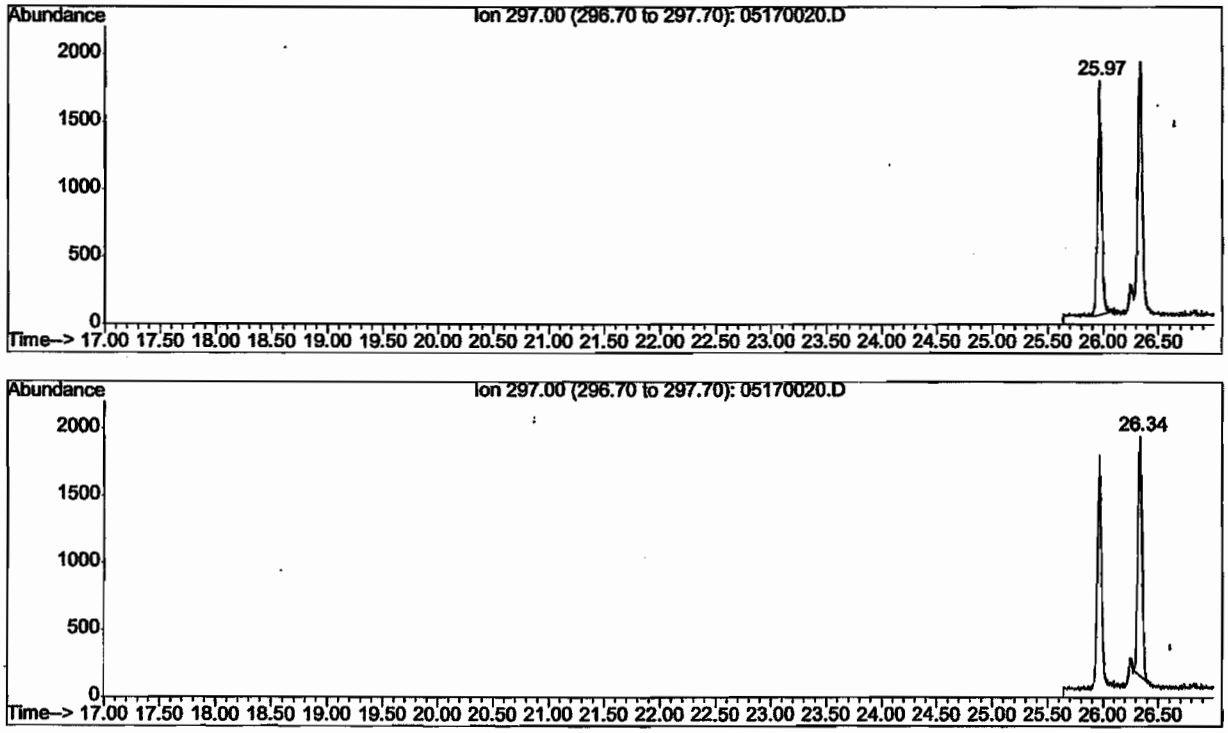
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



Esfenvalerate
20 ng/mL
Peak Response (area): 234713

FIGURE 13. (con't) Representative Chromatograms – Bracketing Standards

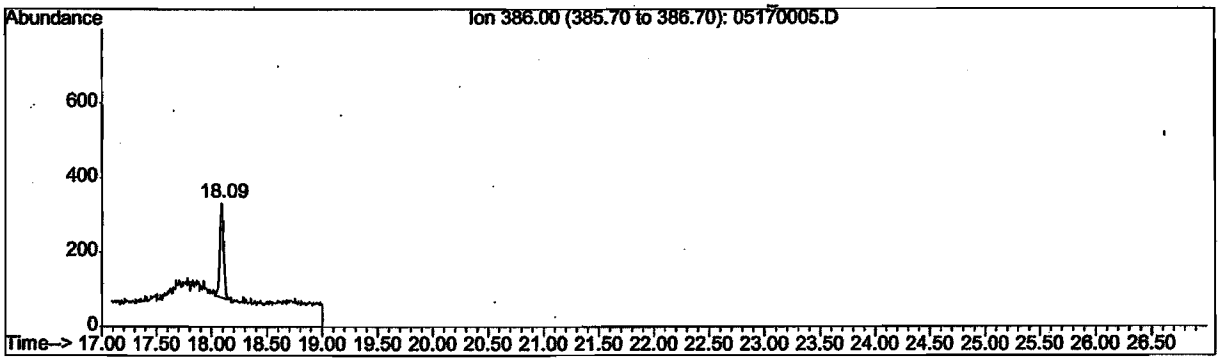
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #3



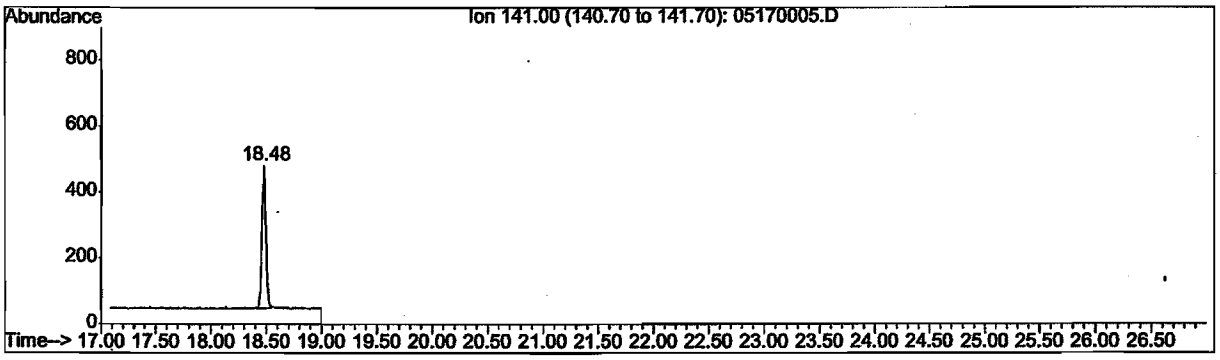
Deltamethrin
20 ng/mL
Peak Response (area): 90585

FIGURE 14. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



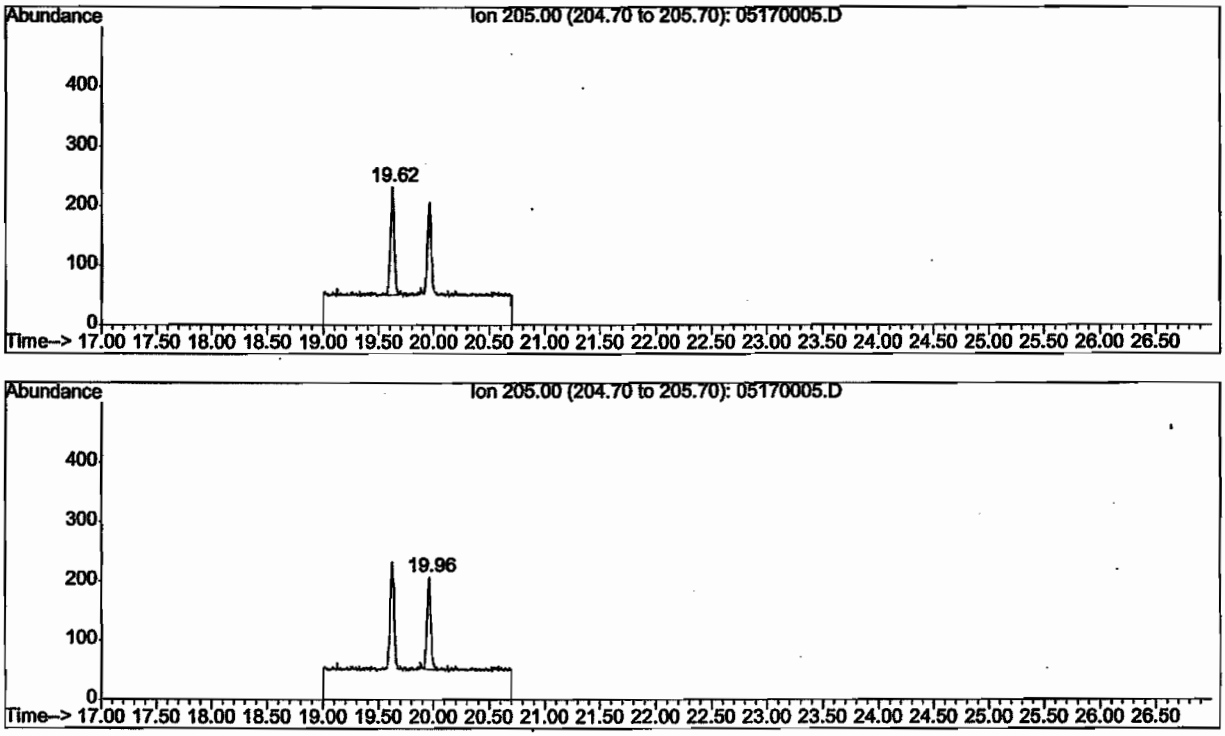
Bifenthrin
1.0 ng/mL
Peak Response (area): 6312



Fenpropathrin
1.0 ng/mL
Peak Response (area): 10783

FIGURE 14. (con't) Representative Chromatograms – Bracketing Standards

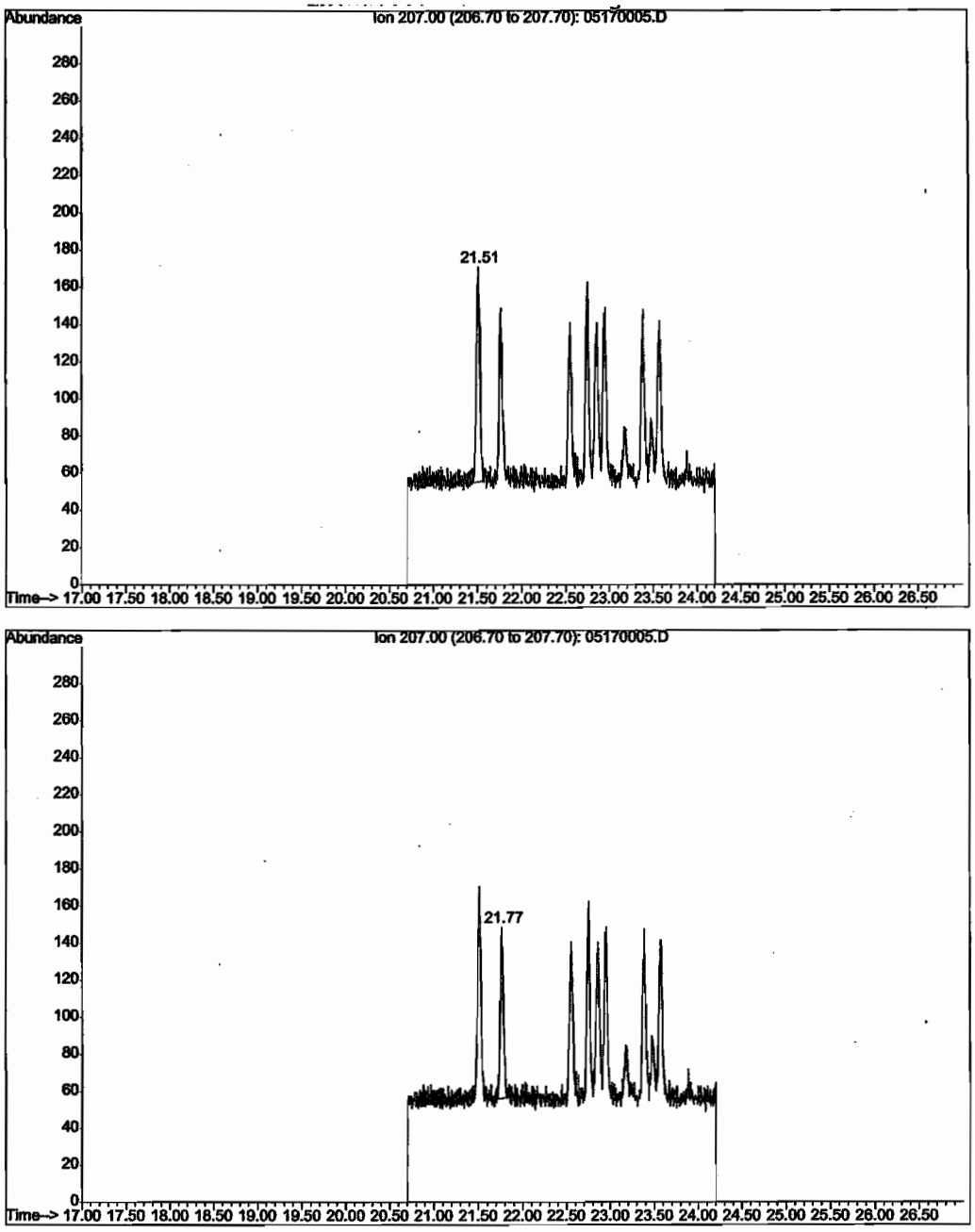
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 8164

FIGURE 14. (con't) Representative Chromatograms – Bracketing Standards

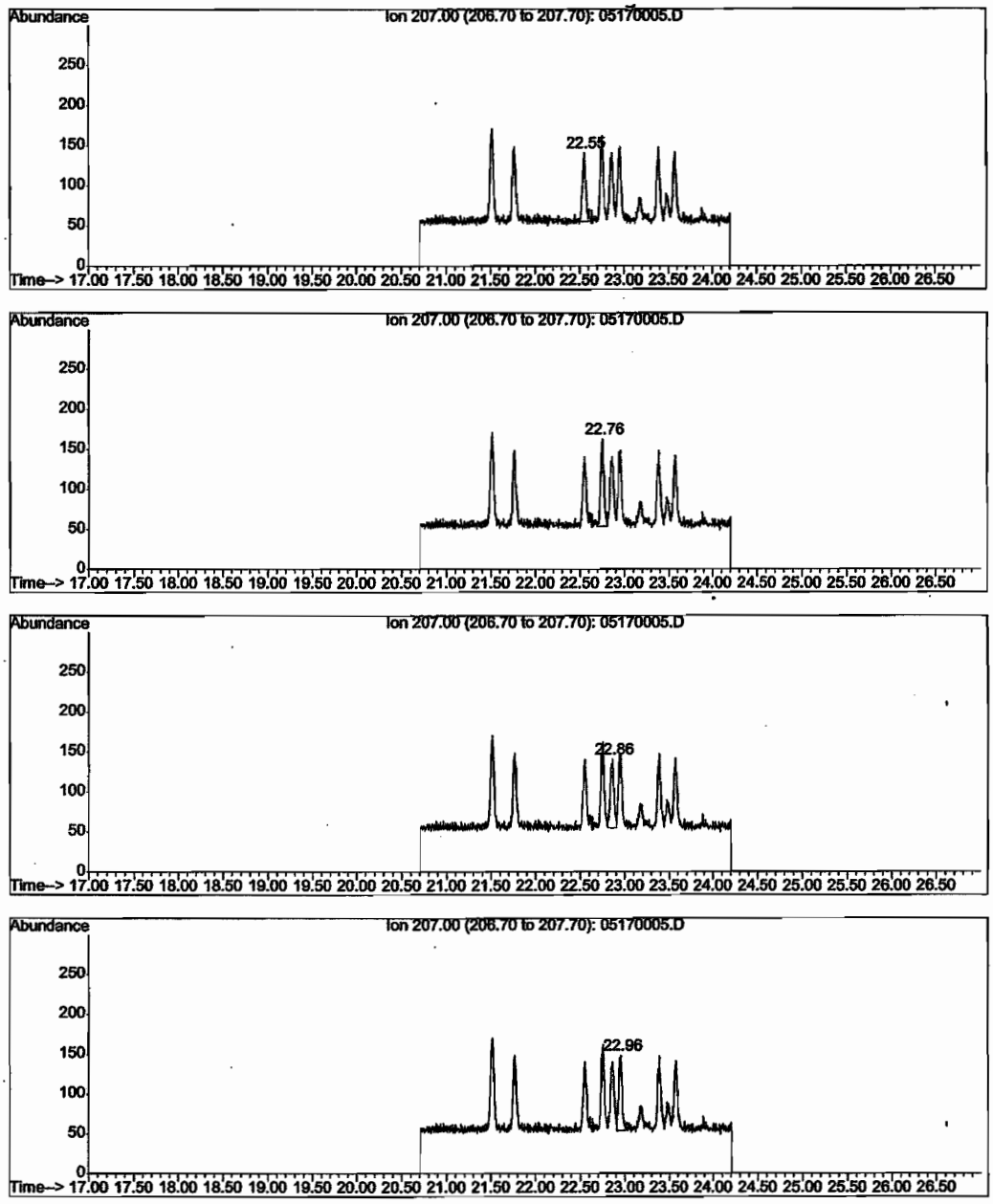
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Permethrin
10 ng/mL
Peak Response (area): 5435

FIGURE 14. (con't) Representative Chromatograms – Bracketing Standards

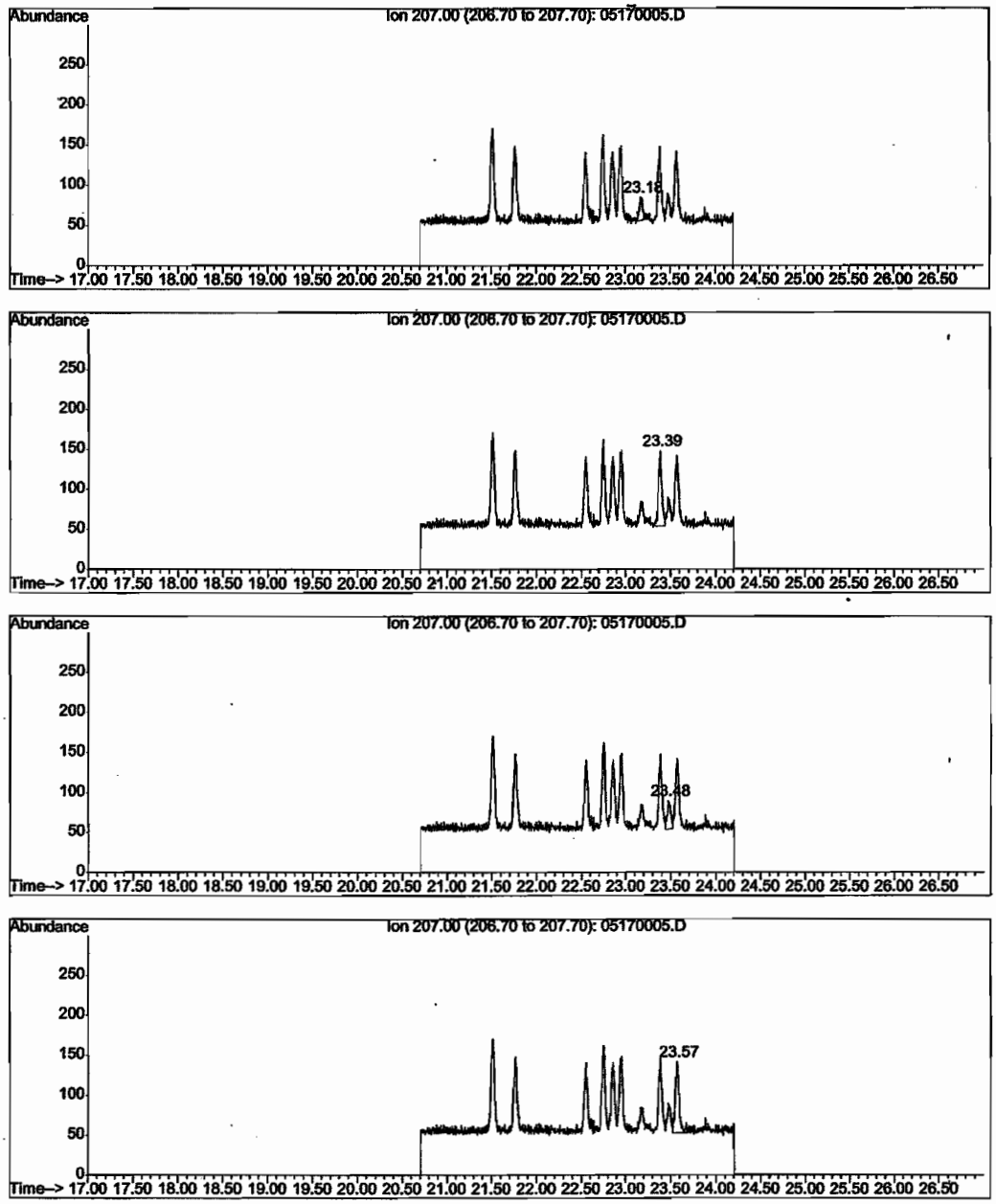
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Cyfluthrin
1.0 ng/mL
Peak Response (area): 10000

FIGURE 14. (con't) Representative Chromatograms – Bracketing Standards

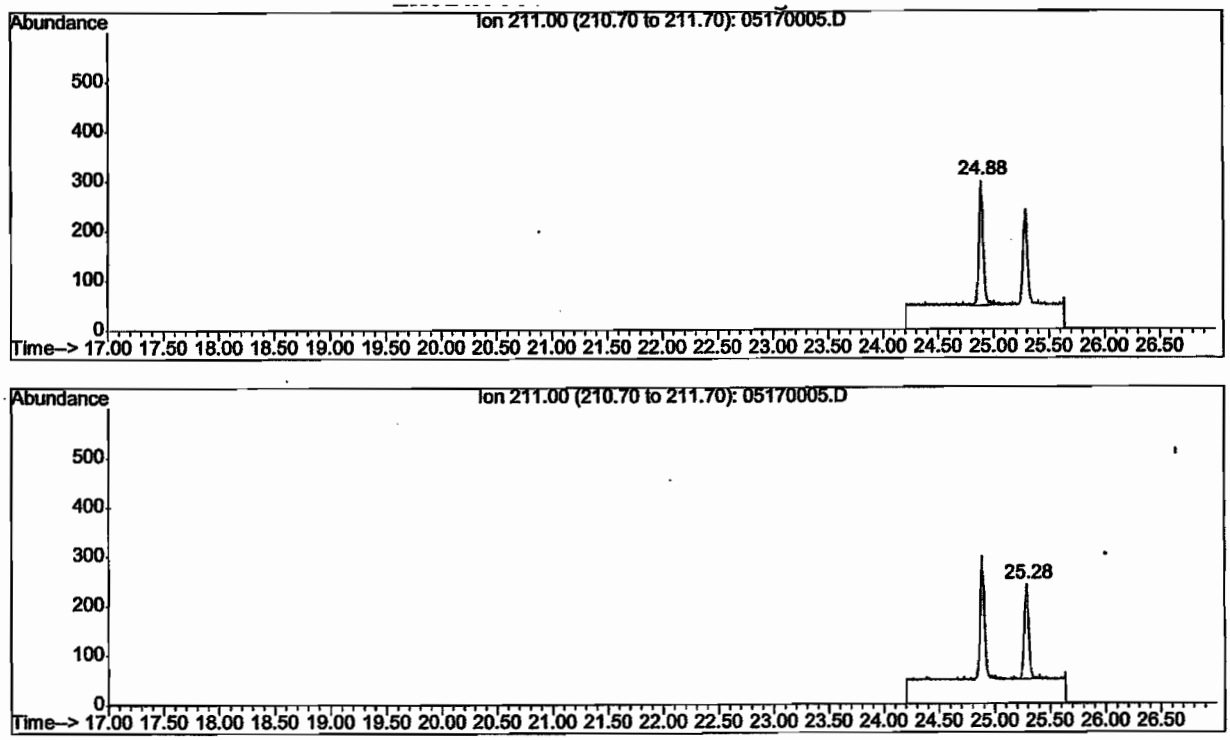
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Cypermethrin
1.0 ng/mL
Peak Response (area): 6869

FIGURE 14. (con't) Representative Chromatograms – Bracketing Standards

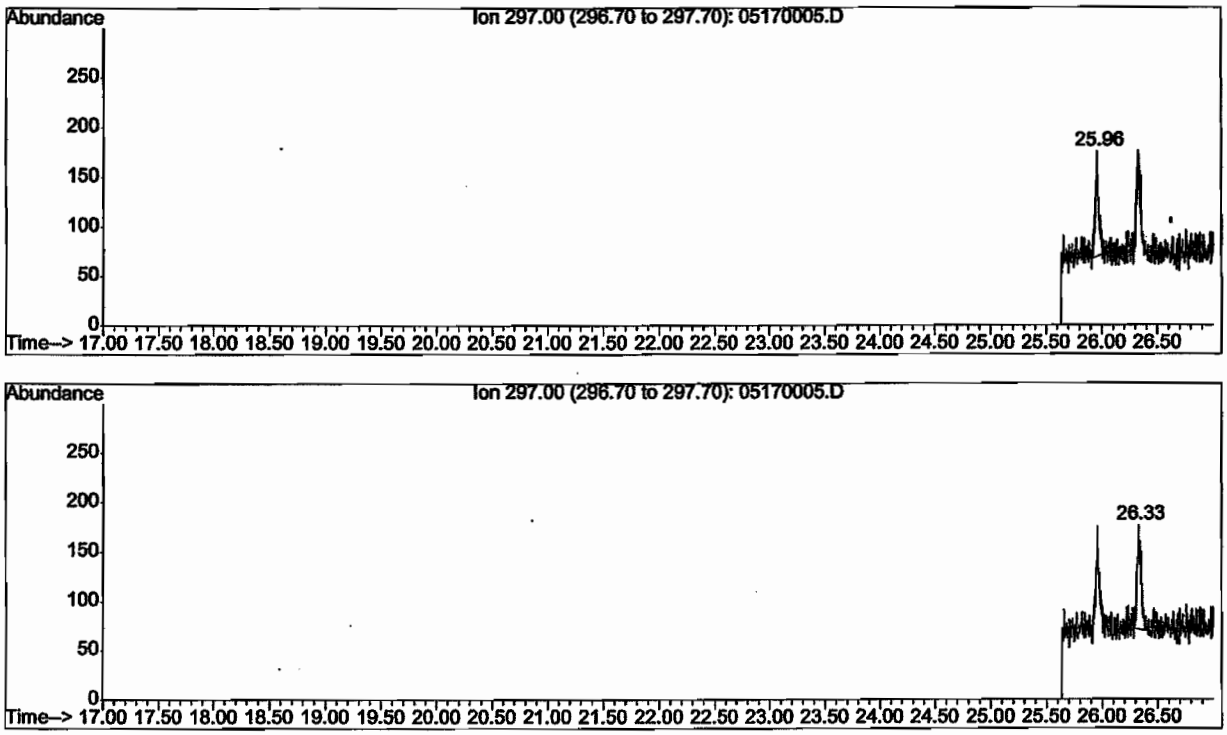
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Esfenvalerate
1.0 ng/mL
Peak Response (area): 12026

FIGURE 14. (con't) Representative Chromatograms – Bracketing Standards

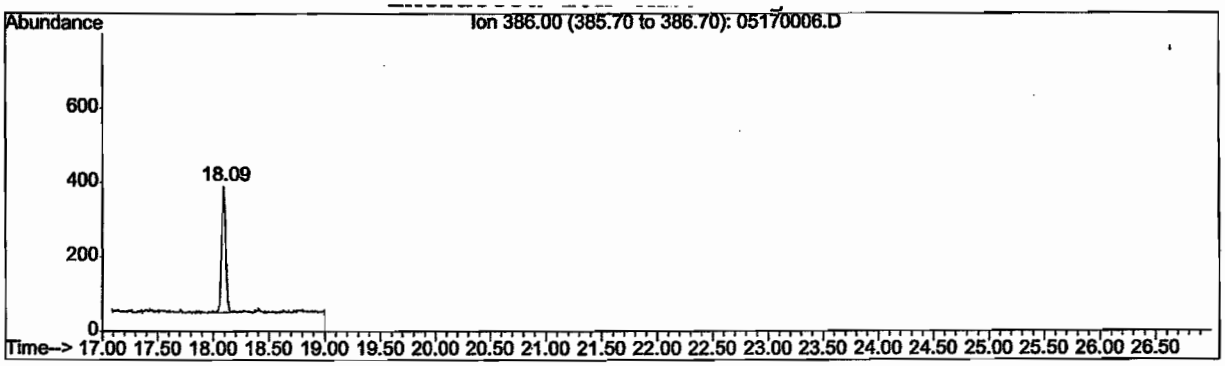
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



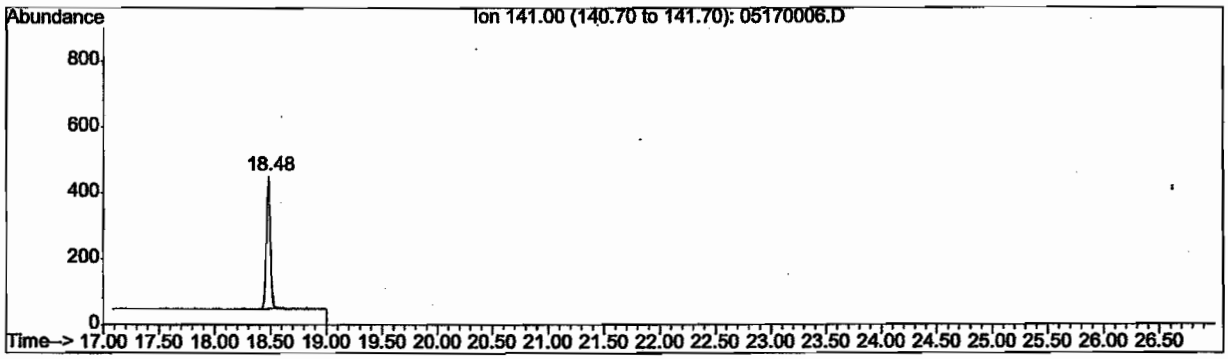
Deltamethrin
1.0 ng/mL
Peak Response (area): 4855

FIGURE 15. Representative Chromatograms - Estuarine Sediment, Fortified Control (LOQ)

Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #3



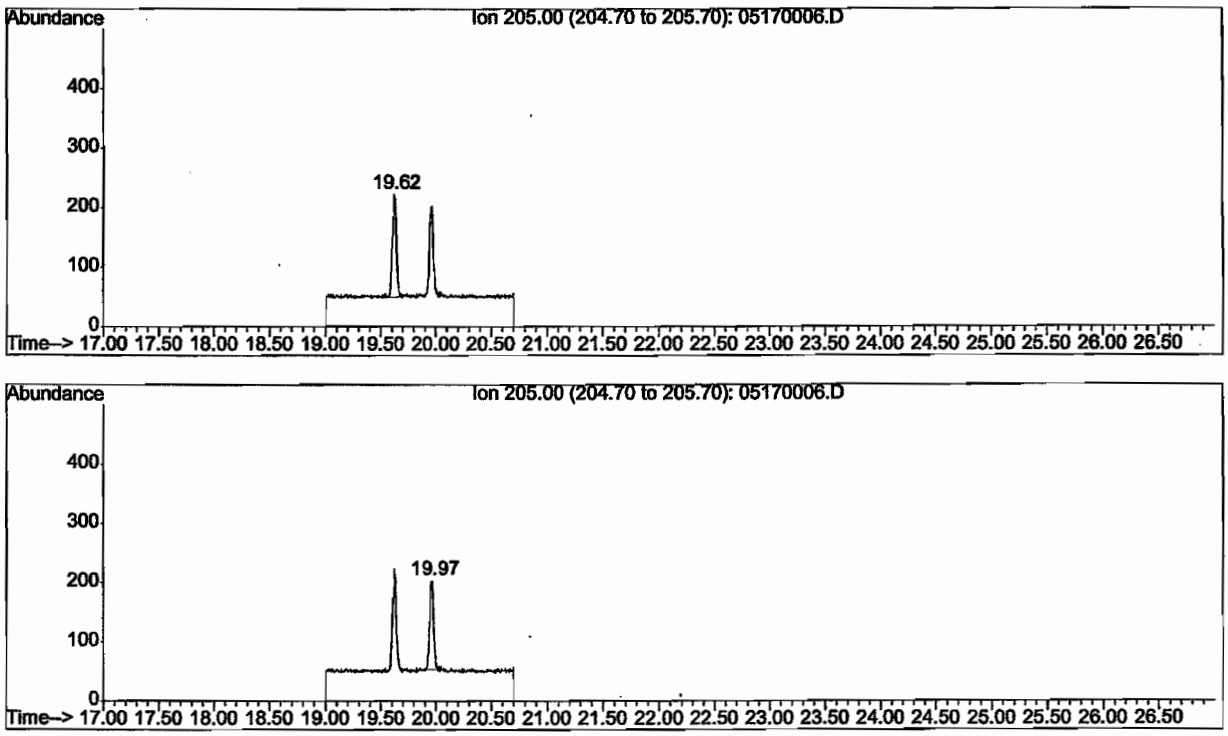
Bifenthrin
Peak Response (area): 8465
Bifenthrin Reported Recovery: 91%



Fenpropathrin
Peak Response (area): 10014
Fenpropathrin Reported Recovery: 95%

FIGURE 15. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (LOQ)

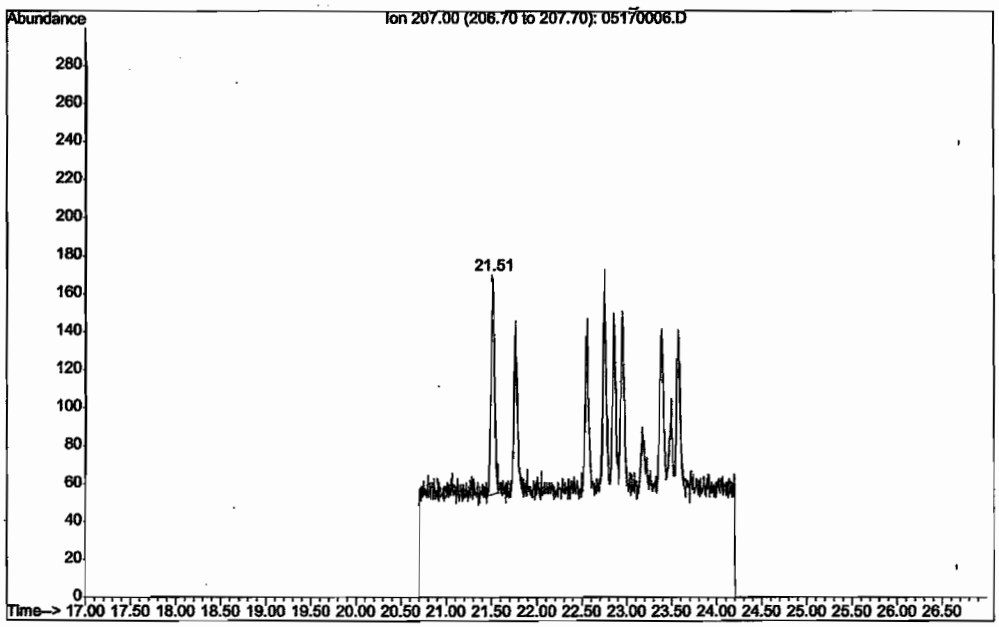
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #3

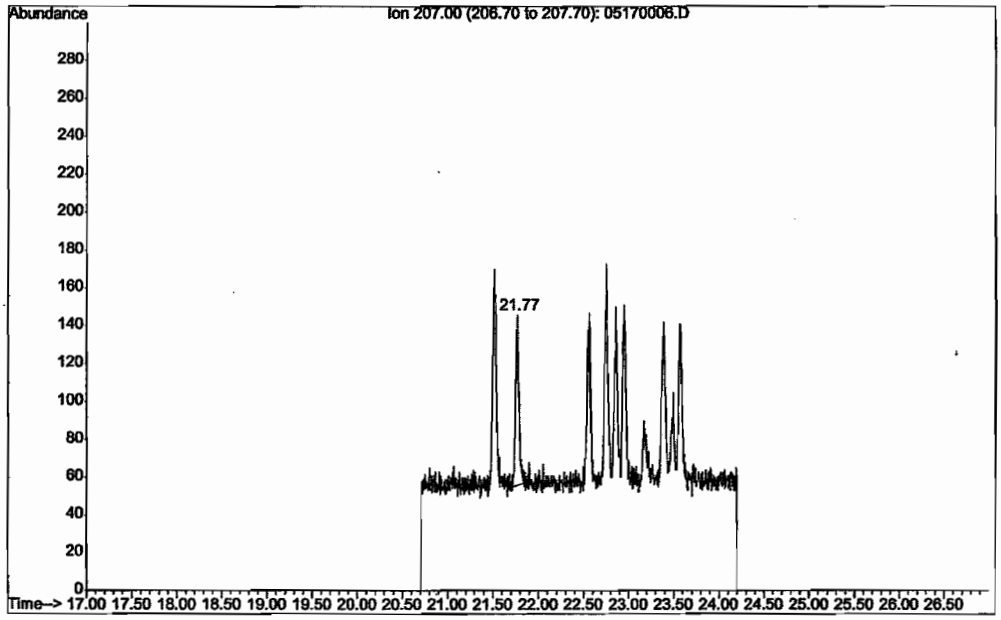


Lambda-cyhalothrin
Peak Response (area): 7959
Lambda-cyhalothrin Reported Recovery: 90%

FIGURE 15. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (LOQ)

Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #3

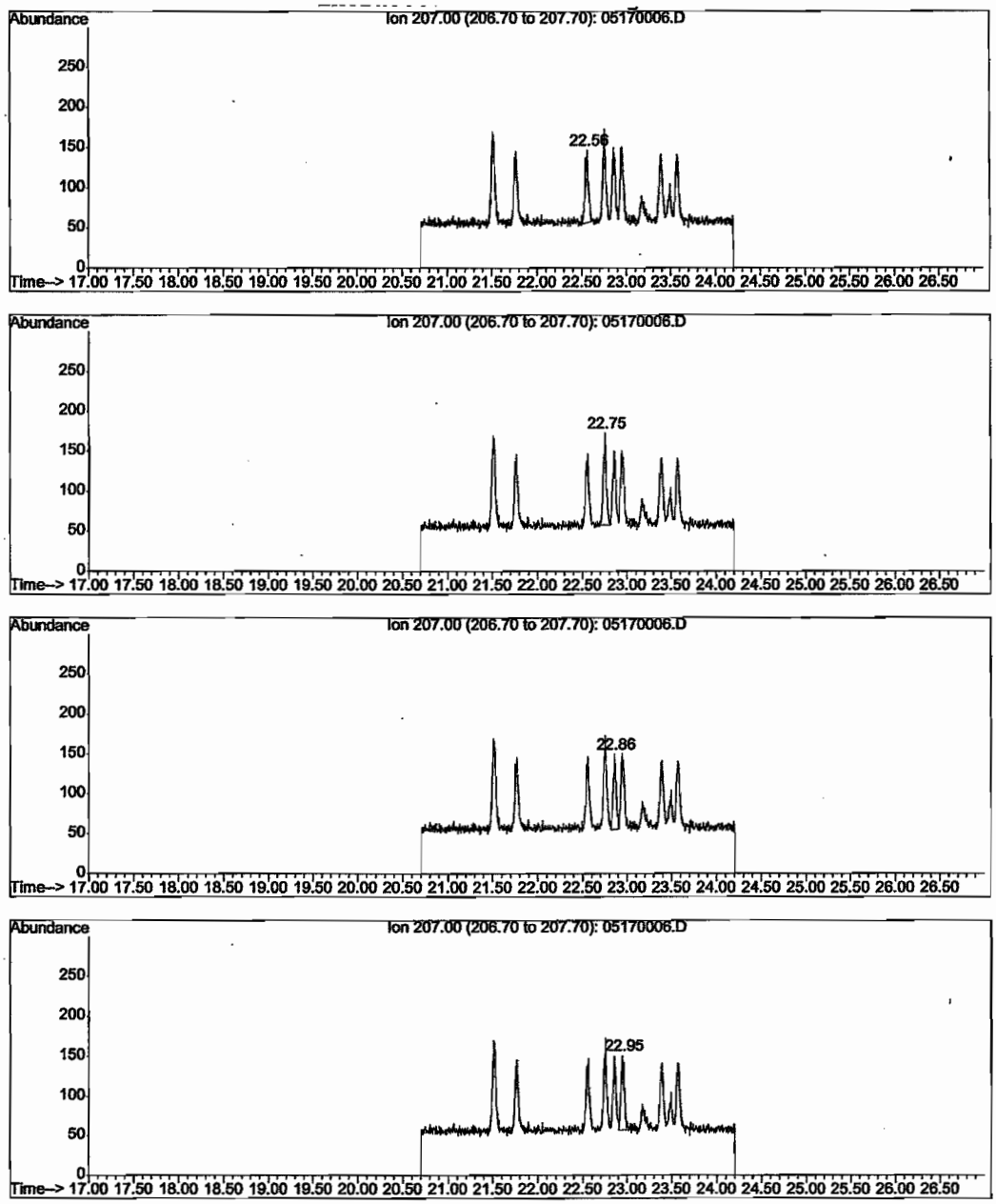




Permethrin
Peak Response (area): 5433
Permethrin Reported Recovery: 98%

FIGURE 15. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (LOQ)

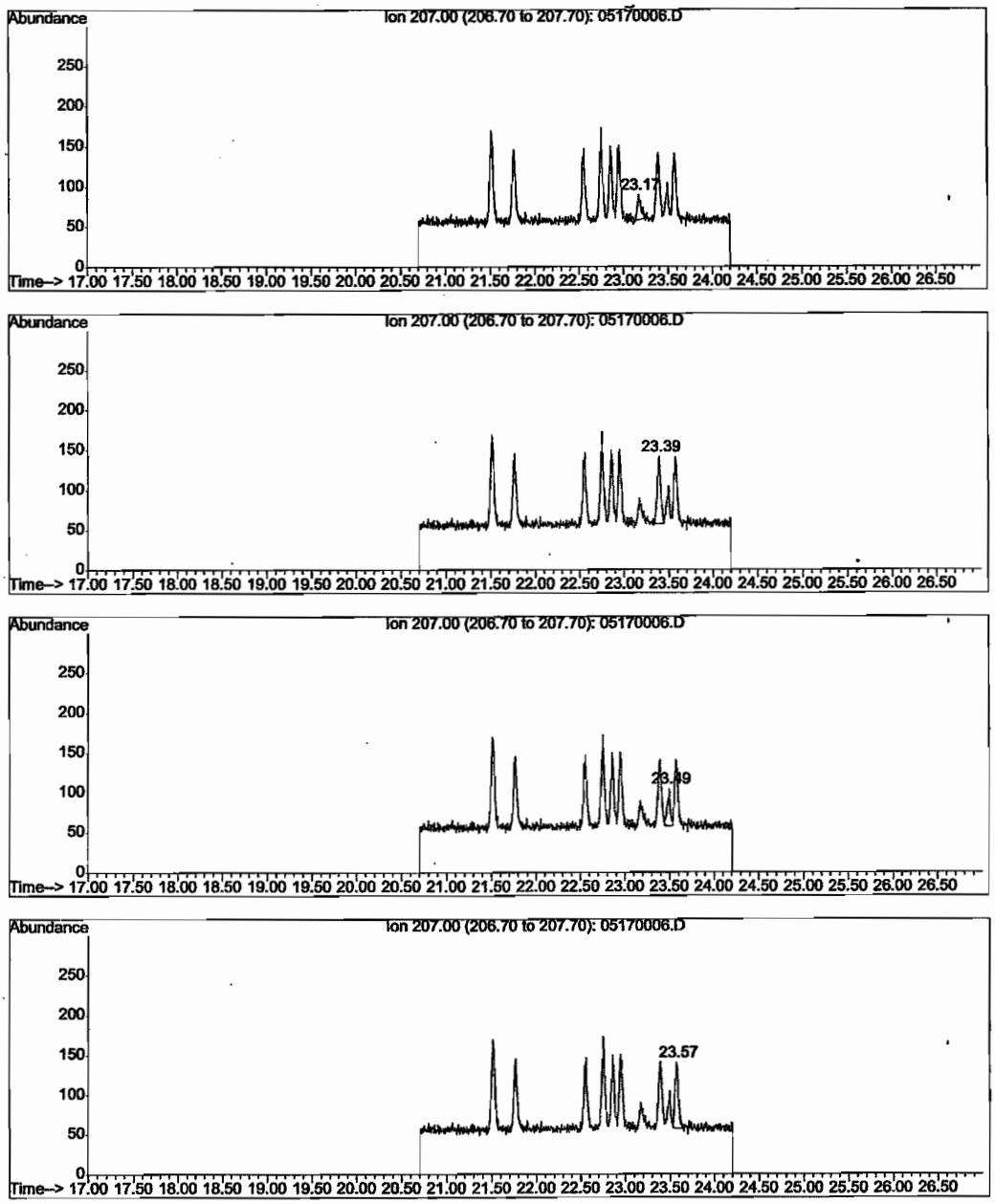
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #3



Cyfluthrin
Peak Response (area): 9613
Cyfluthrin Reported Recovery: 89%

FIGURE 15. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (LOQ)

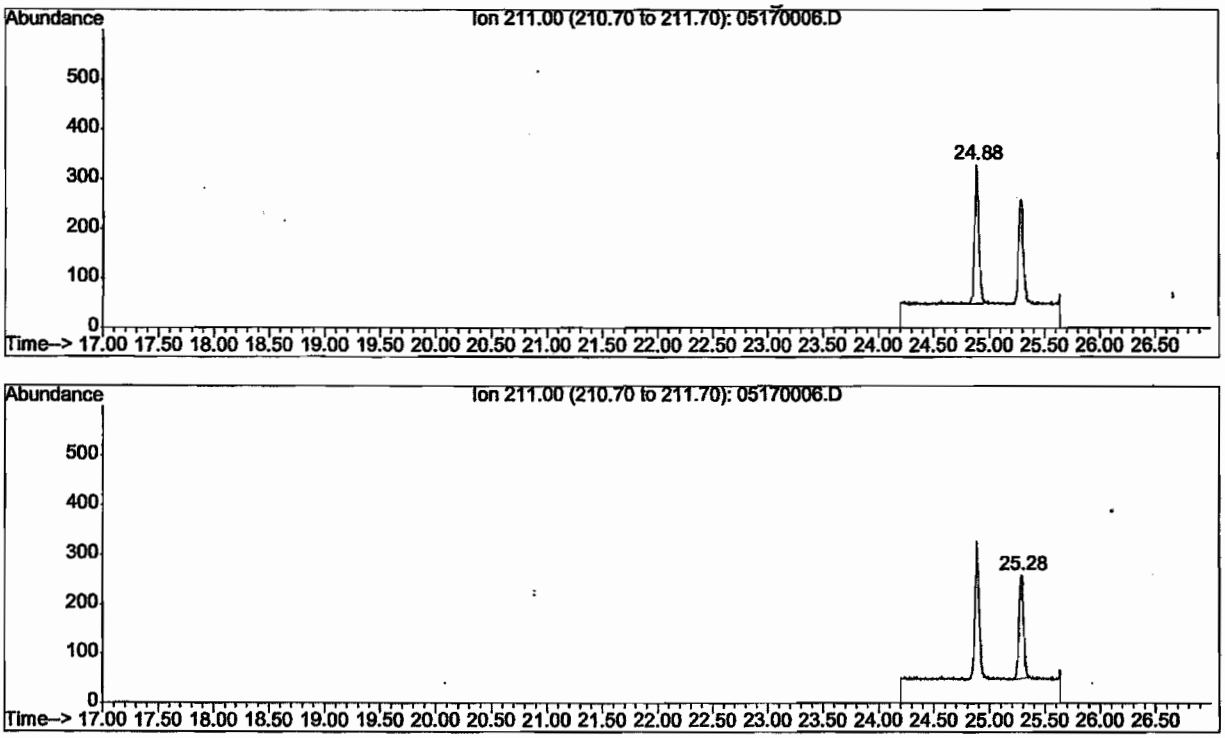
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #3



Cypermethrin
Peak Response (area): 6423
Cypermethrin Reported Recovery: 98%

FIGURE 15. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (LOQ)

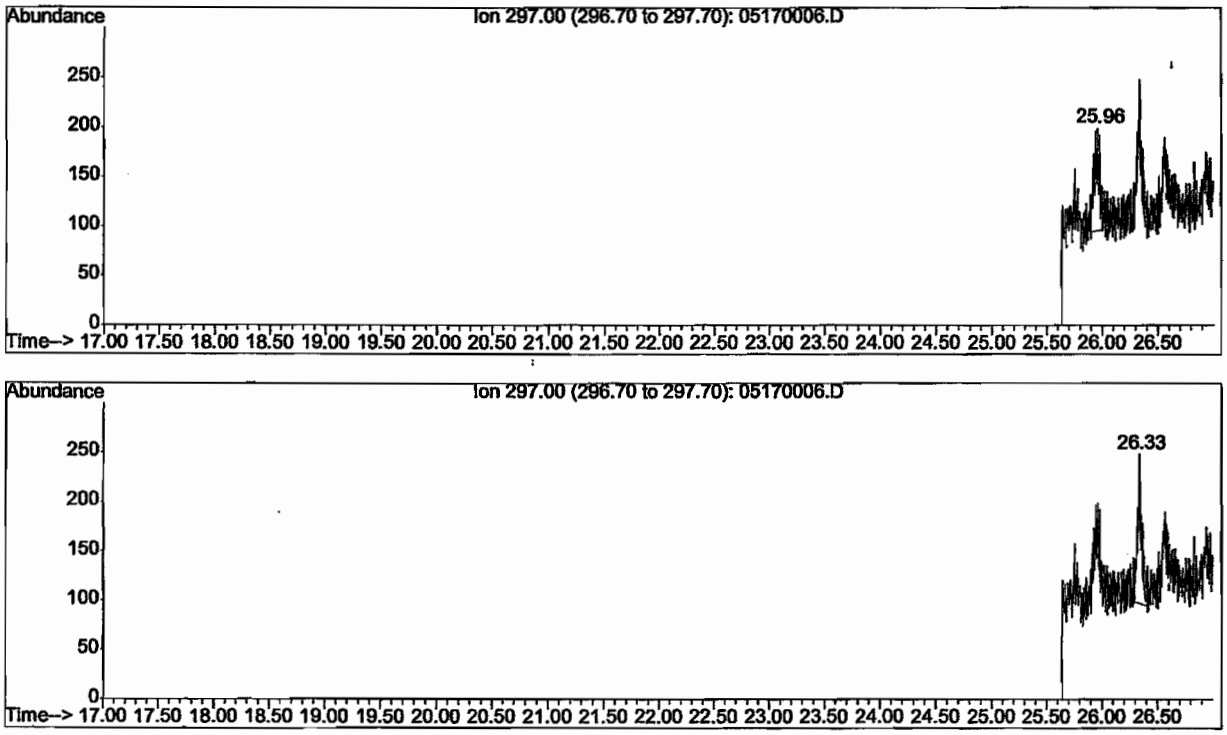
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #3



Esfenvalerate
Peak Response (area): 12911
Esfenvalerate Reported Recovery: 97%

FIGURE 15. (con't) Representative Chromatograms - Estuarine Sediment, Fortified Control (LOQ)

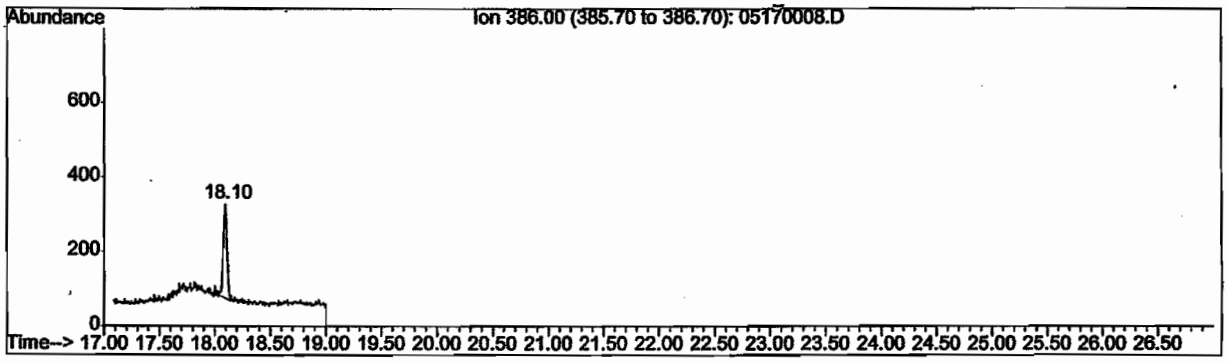
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL, Set #3



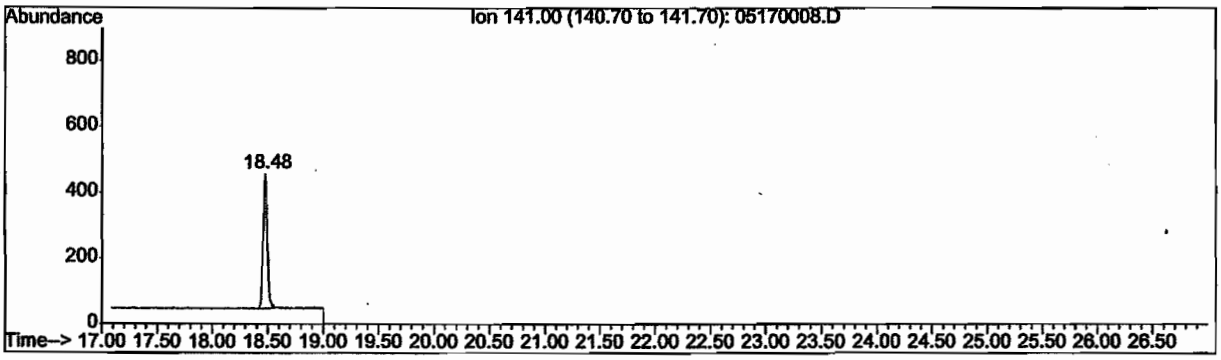
Deltamethrin
Peak Response (area): 7418
Deltamethrin Reported Recovery: 87%

FIGURE 16. Representative Chromatograms – Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



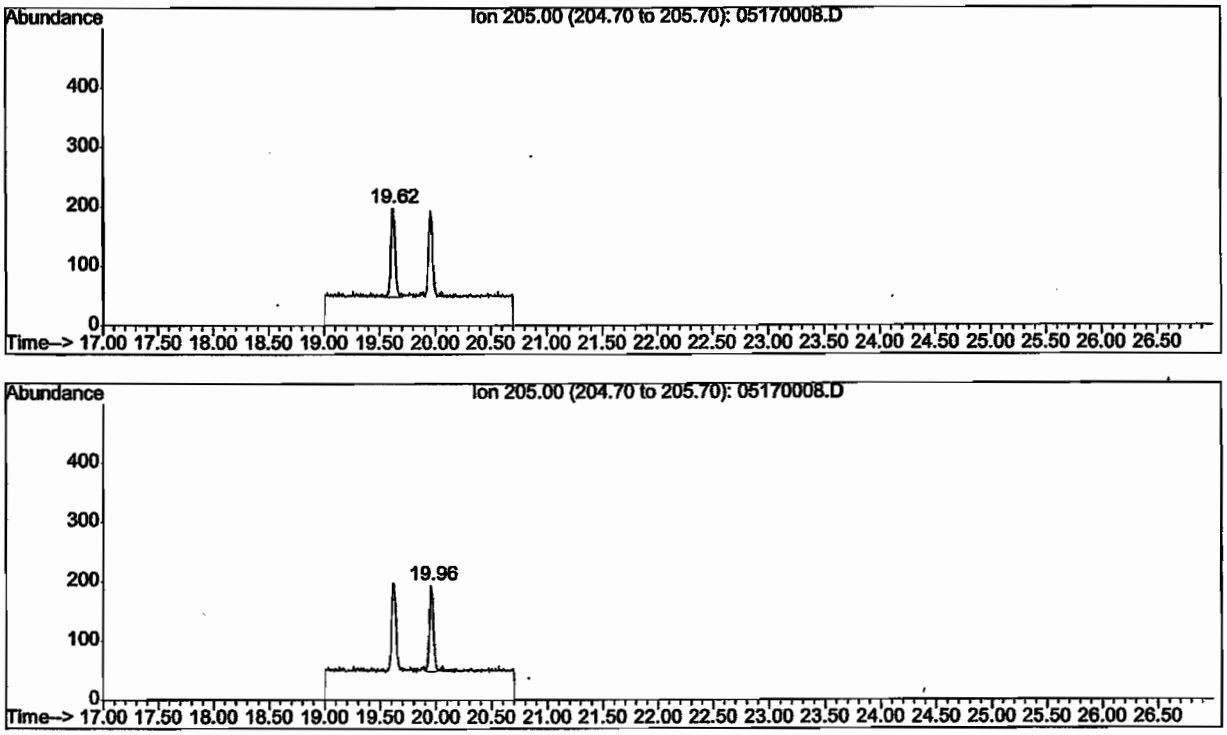
Bifenthrin
1.0 ng/mL
Peak Response (area): 5926



Fenpropathrin
1.0 ng/mL
Peak Response (area): 10414

FIGURE 16. (con't) Representative Chromatograms – Bracketing Standards

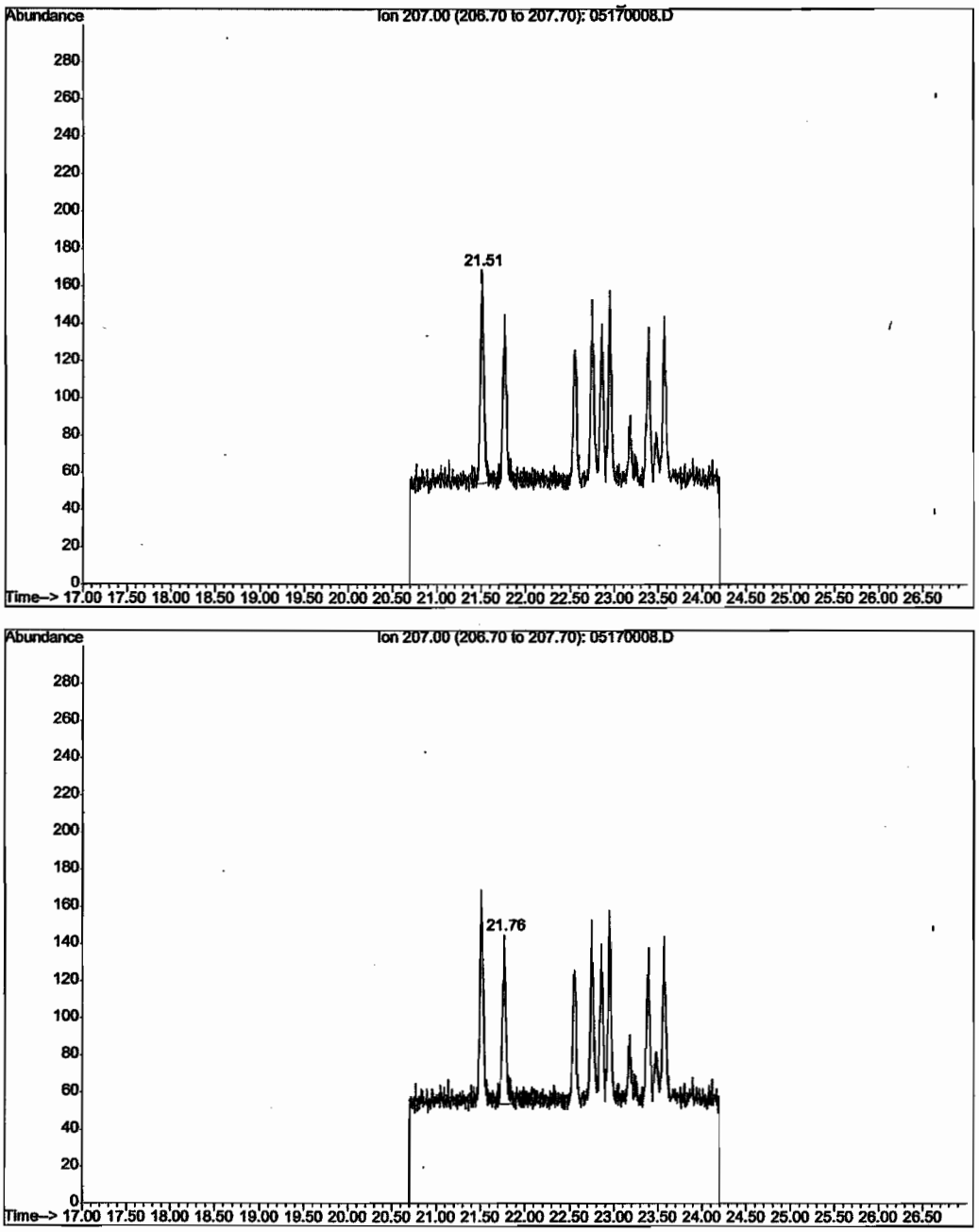
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 7569

FIGURE 16. (con't) Representative Chromatograms – Bracketing Standards

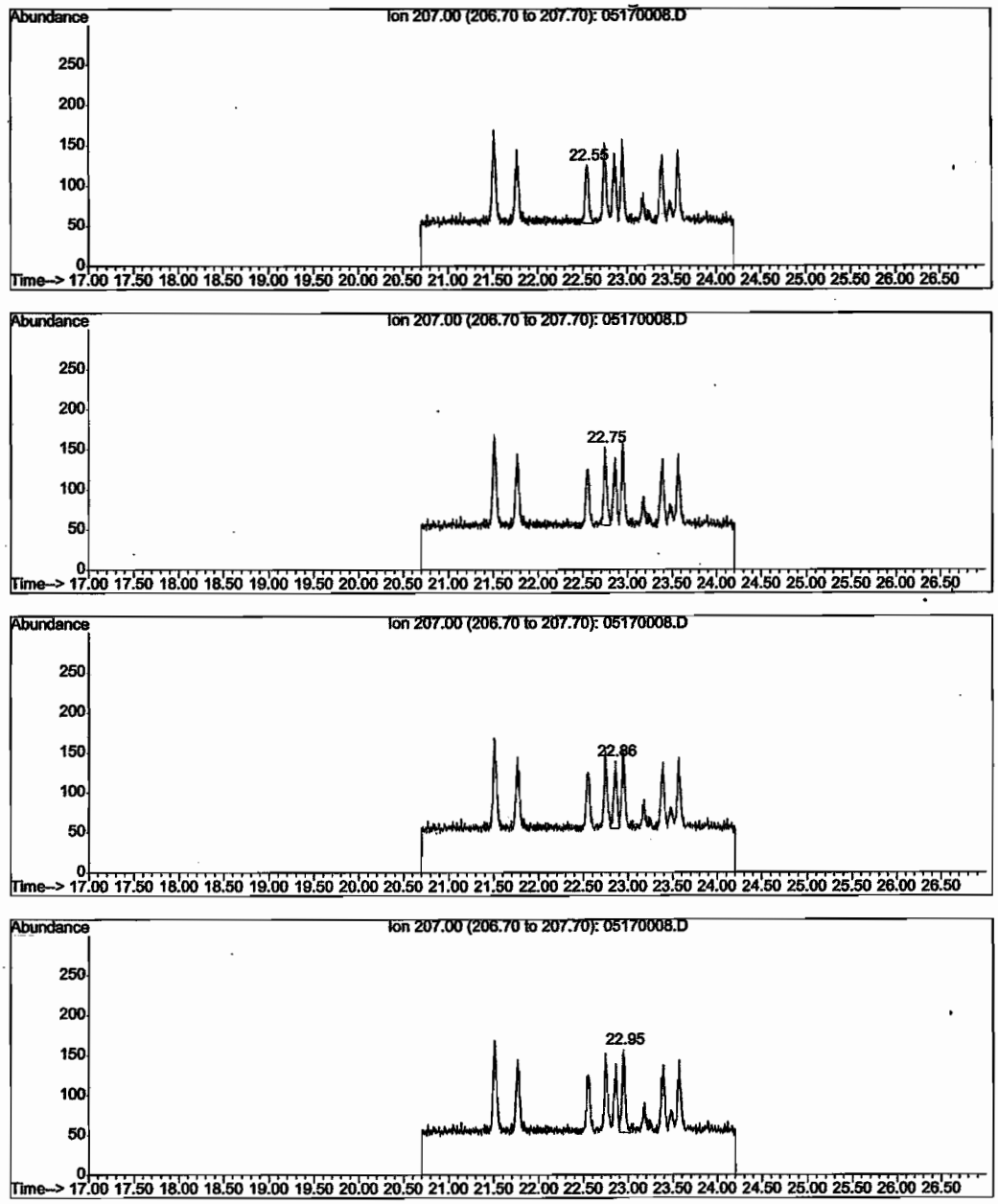
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Permethrin
10 ng/mL
Peak Response (area): 5606

FIGURE 16. (con't)Representative Chromatograms – Bracketing Standards

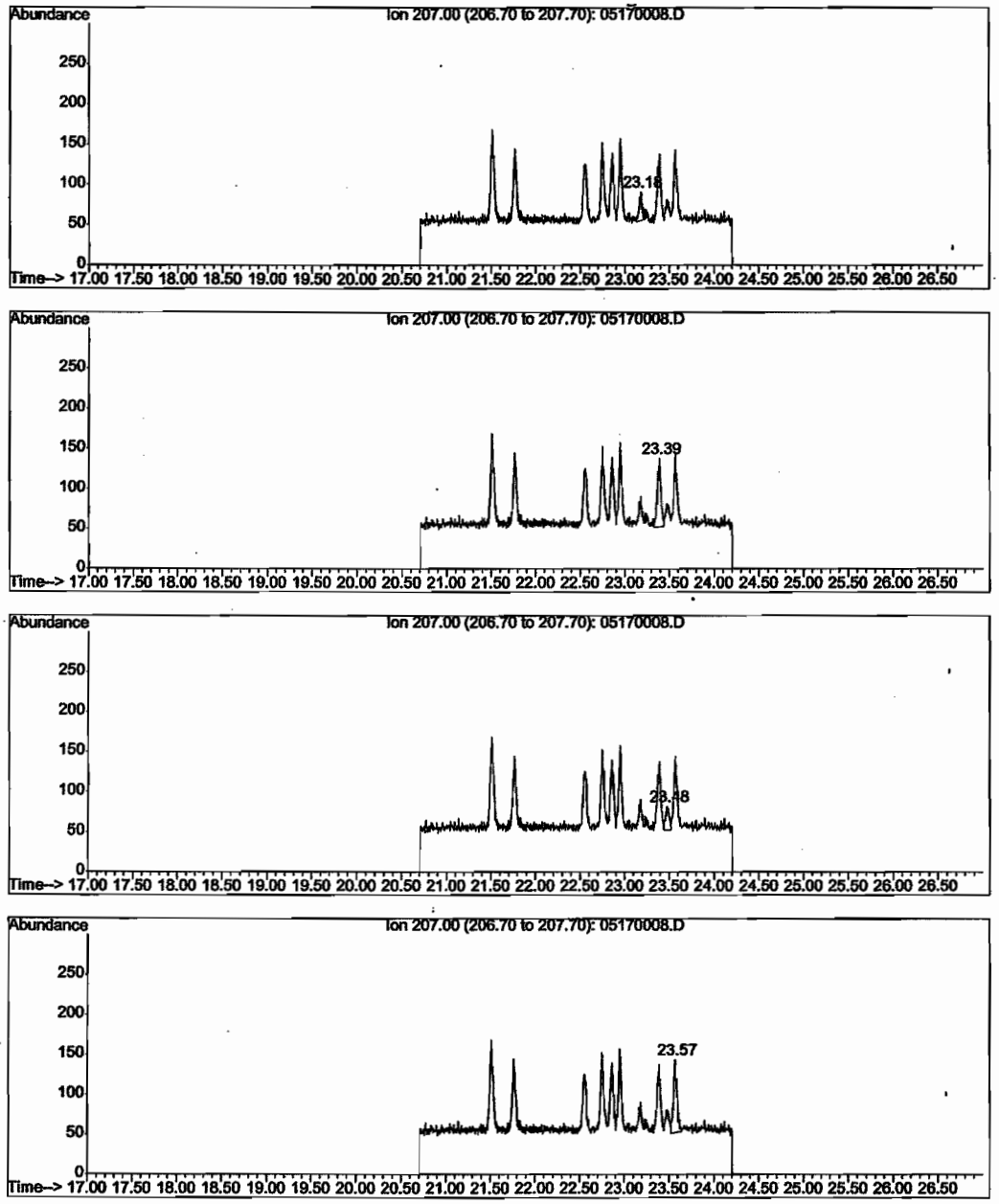
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Cyfluthrin
1.0 ng/mL
Peak Response (area): 9051

FIGURE 16. (con't) Representative Chromatograms – Bracketing Standards

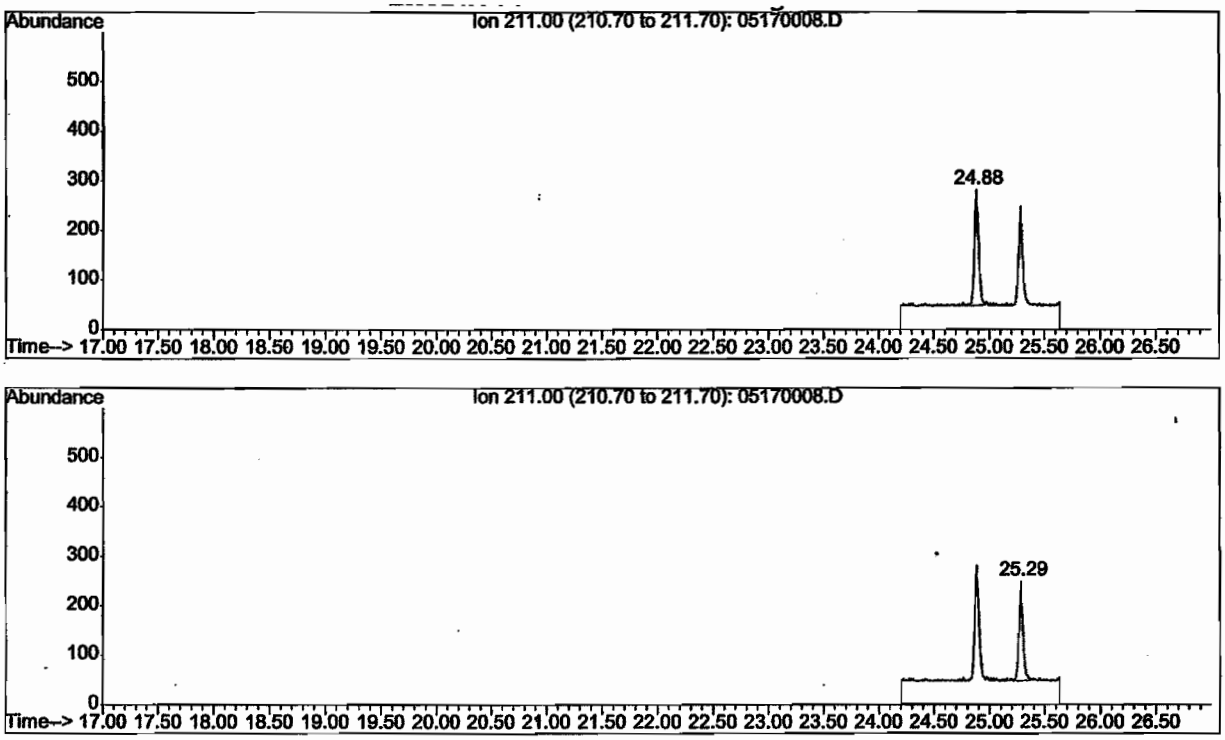
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Cypermethrin
1.0 ng/mL
Peak Response (area): 6254

FIGURE 16. (con't) Representative Chromatograms – Bracketing Standards

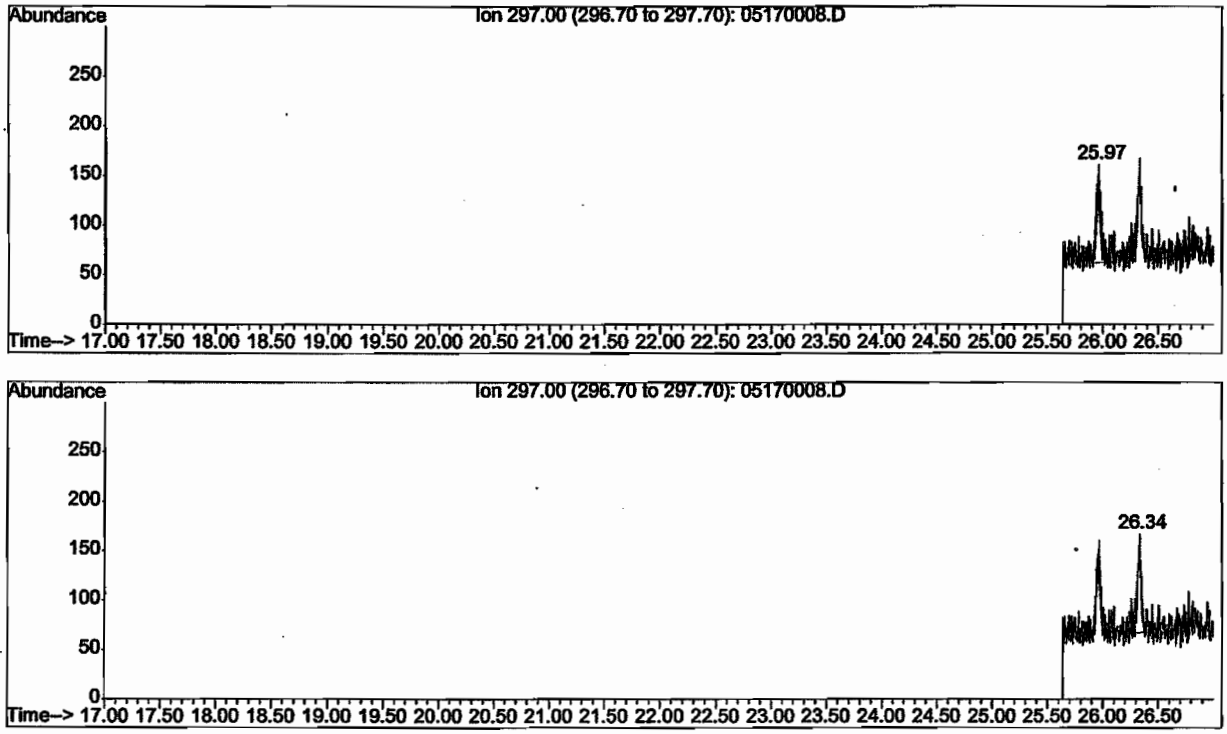
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



Esfenvalerate
1.0 ng/mL
Peak Response (area): 10940

FIGURE 16. (con't) Representative Chromatograms – Bracketing Standards

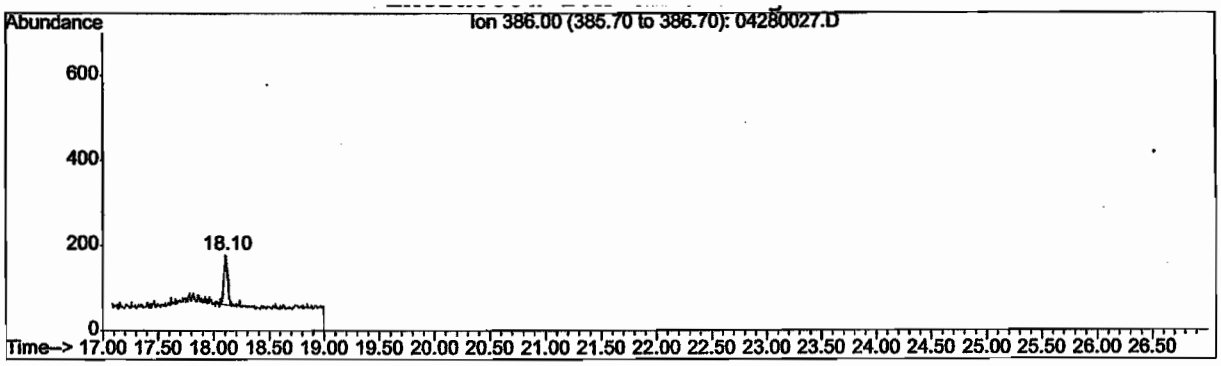
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #3



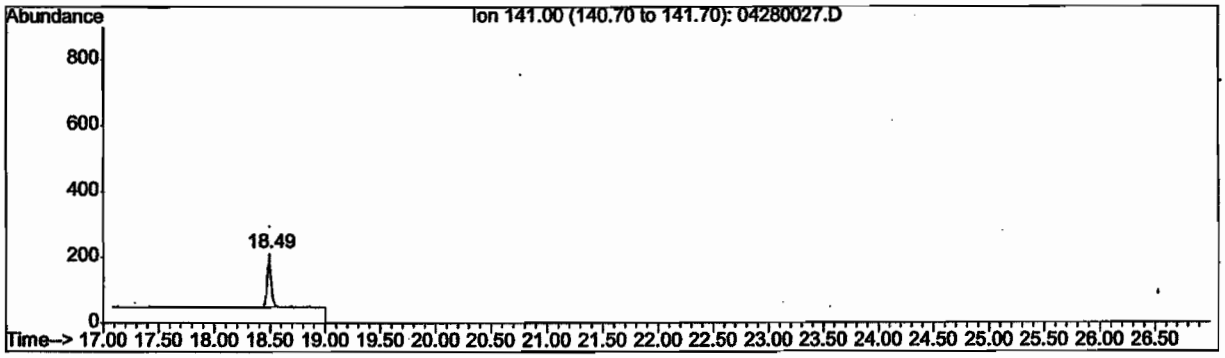
Deltamethrin
1.0 ng/mL
Peak Response (area): 4457

FIGURE 17. Representative Chromatograms - Calibration Standards @ 0.5/5 ng/mL

Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin, Set #2



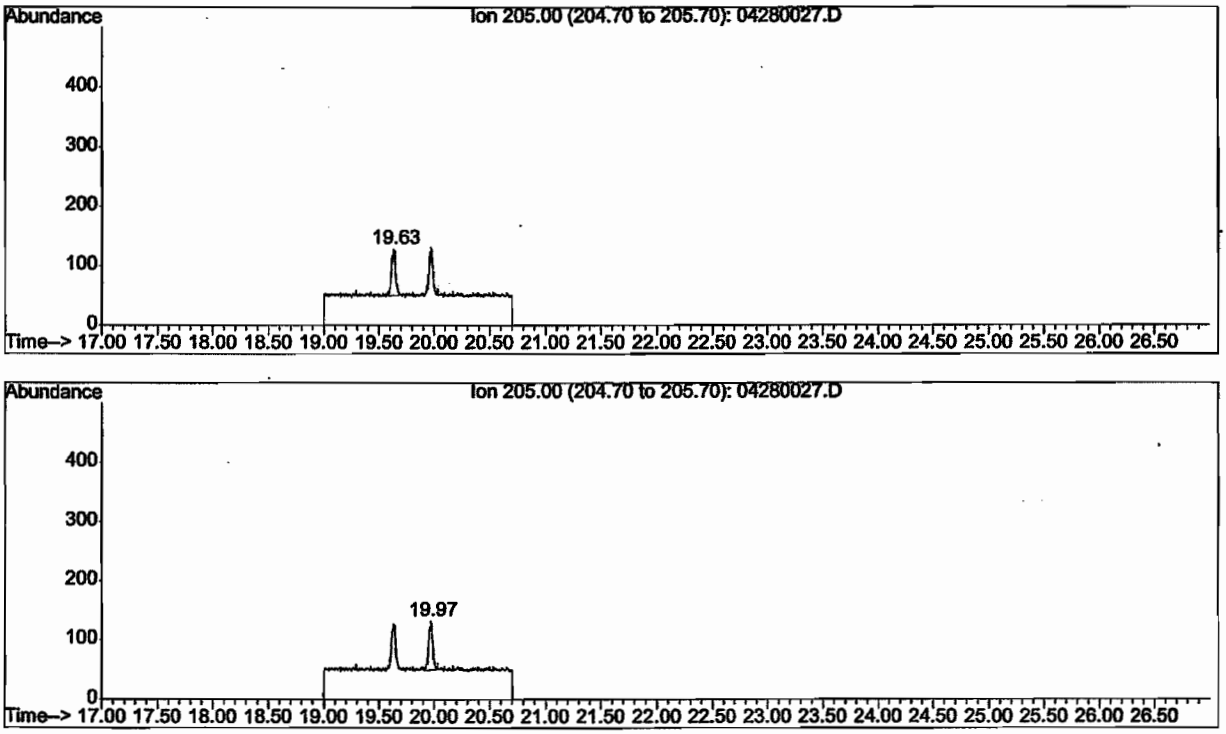
Bifenthrin
0.5 ng/mL
Peak Response (area): 2681



Fenpropathrin
0.5 ng/mL
Peak Response (area): 3802

FIGURE 17.(con't) Representative Chromatograms - Calibration Standards @ 0.5/5 ng/mL

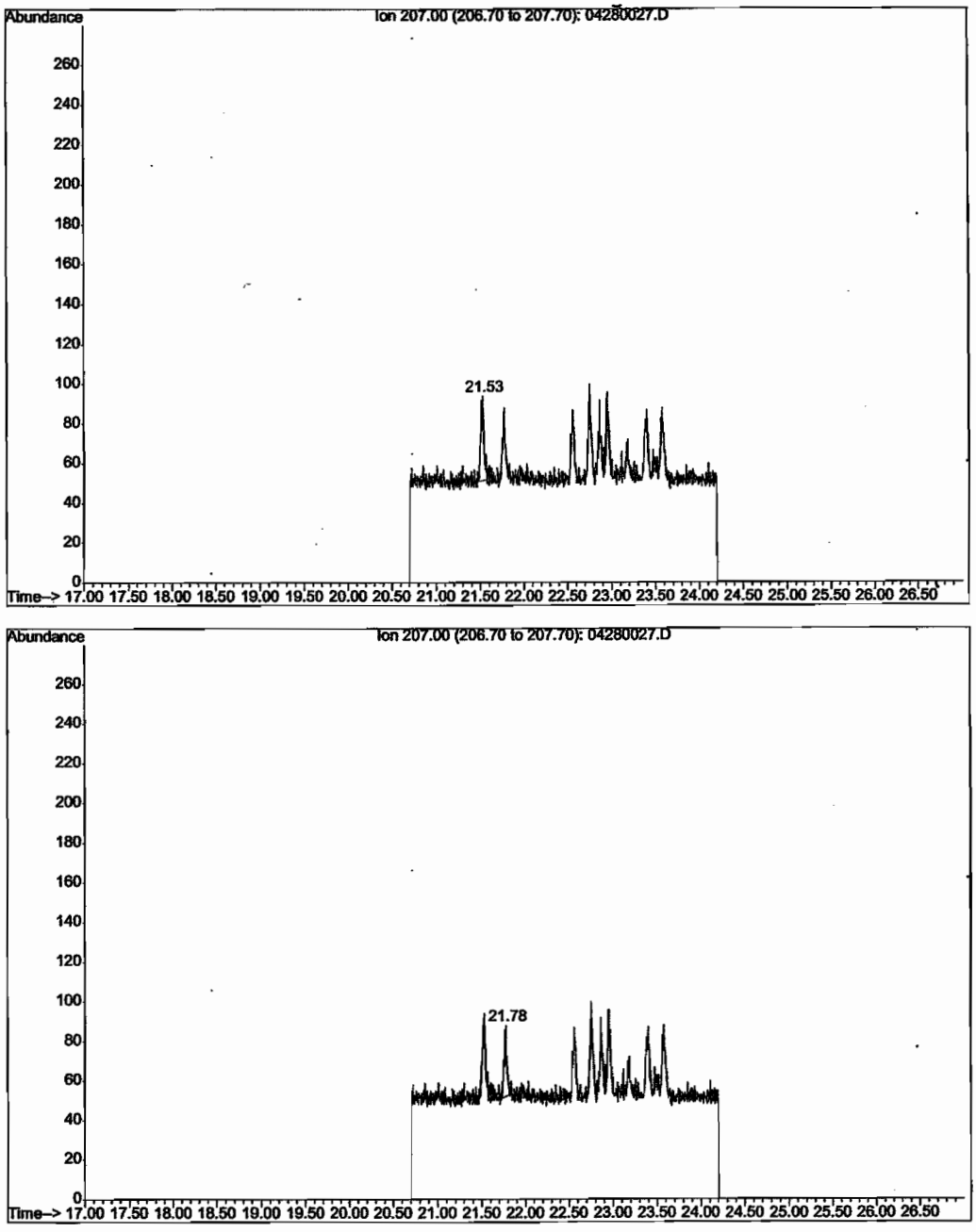
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
0.5 ng/mL
Peak Response (area): 4057

FIGURE 17.(con't) Representative Chromatograms - Calibration Standards @ 0.5/5 ng/mL

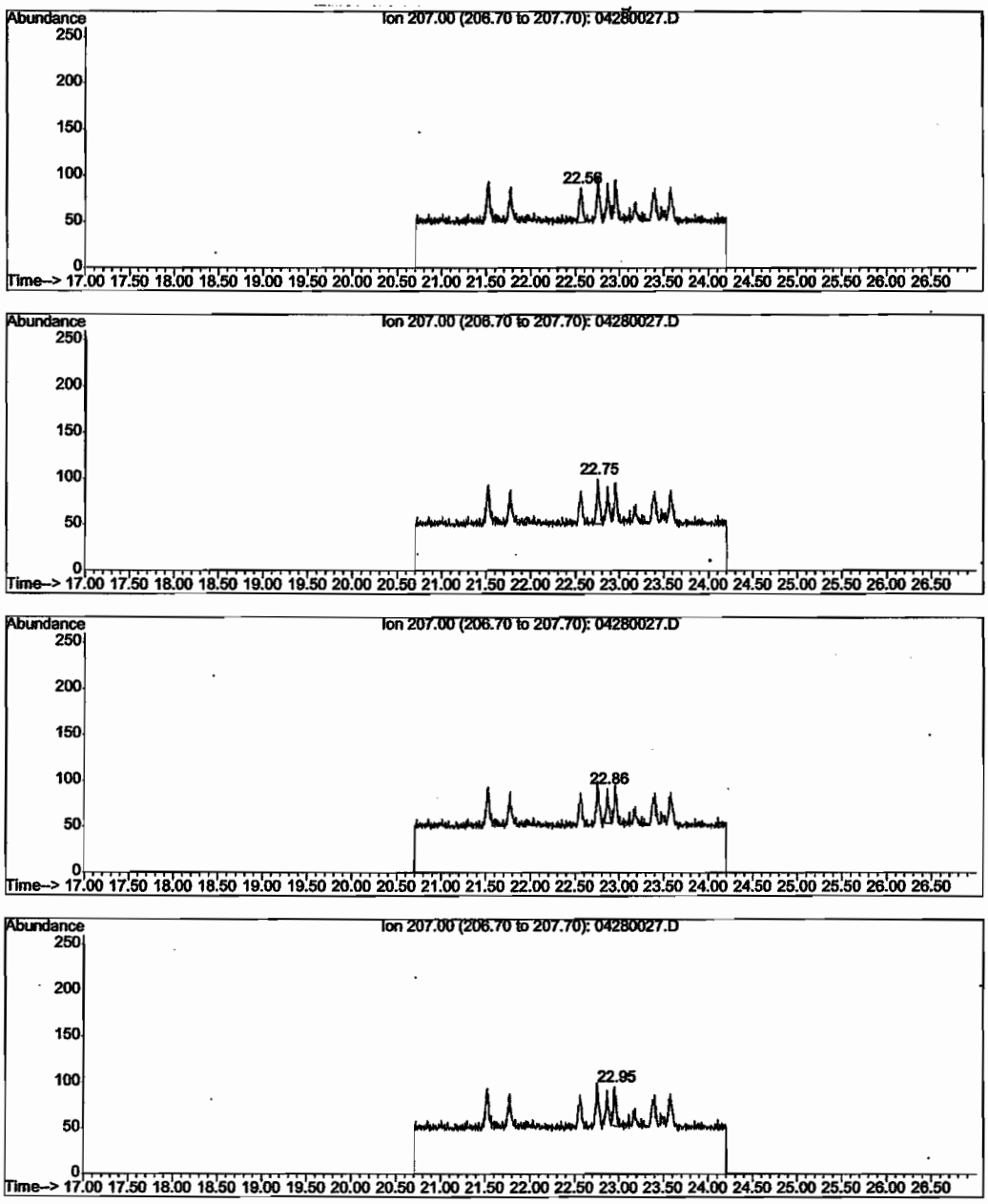
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin, Set #2



Permethrin
5.0 ng/mL
Peak Response (area): 1788

FIGURE 17.(con't) Representative Chromatograms - Calibration Standards @ 0.5/5 ng/mL

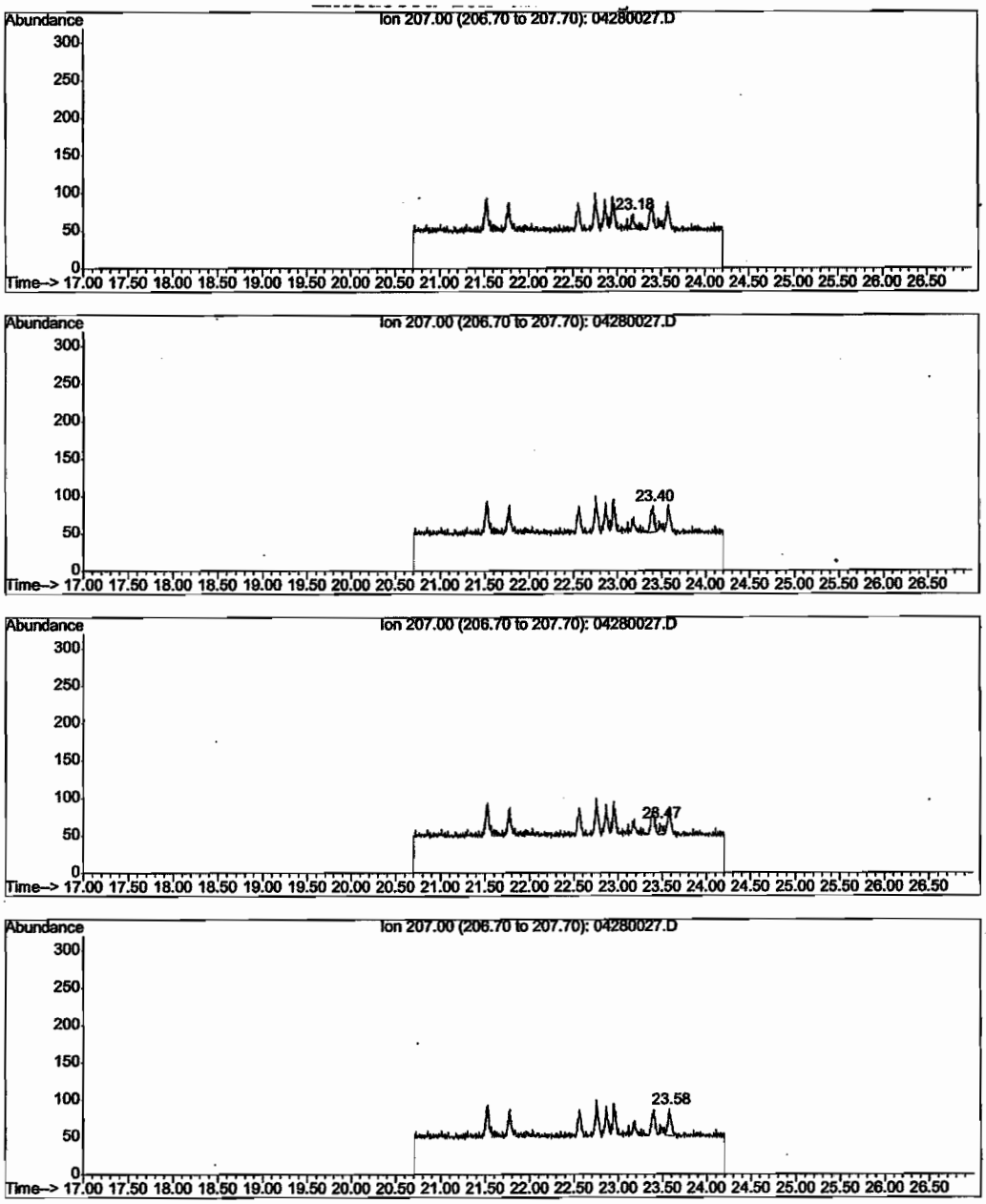
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin, Set #2



Cyfluthrin
0.5 ng/mL
Peak Response (area): 3564

FIGURE 17.(con't) Representative Chromatograms - Calibration Standards @ 0.5/5 ng/mL

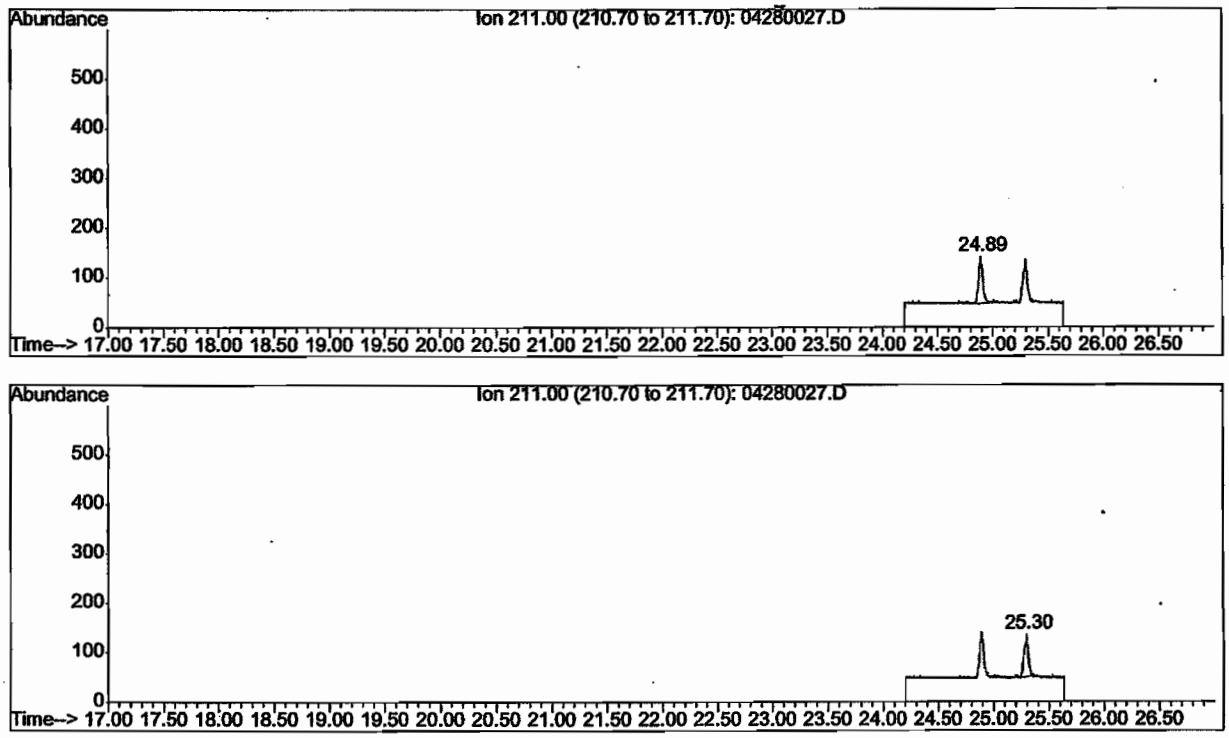
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin, Set #2



Cypermethrin
0.5 ng/mL
Peak Response (area): 2452

FIGURE 17.(con't) Representative Chromatograms - Calibration Standards @ 0.5/5 ng/mL

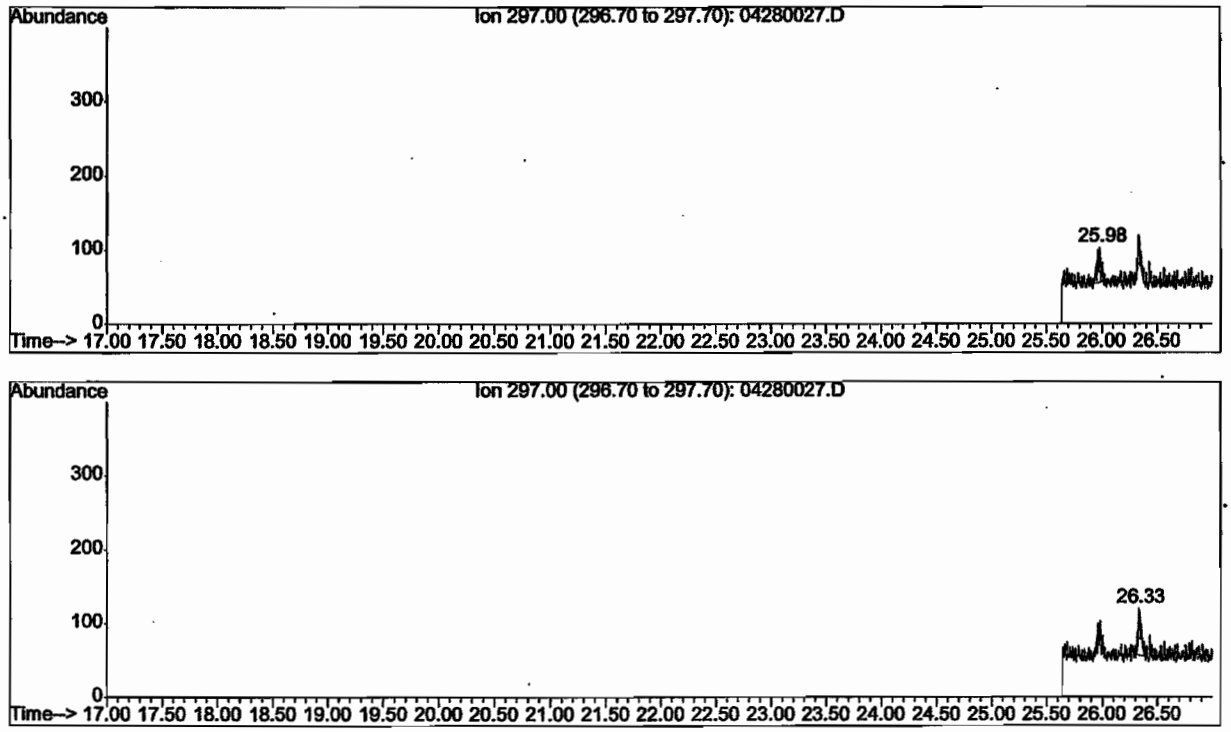
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin, Set #2



Esfenvalerate
0.5 ng/mL
Peak Response (area): 4521

FIGURE 17.(con't) Representative Chromatograms - Calibration Standards @ 0.5/5 ng/mL

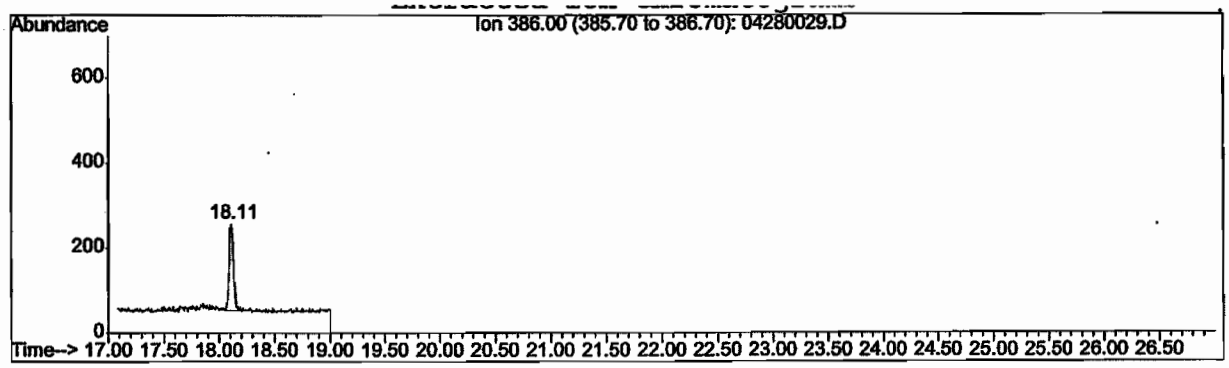
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin, Set #2



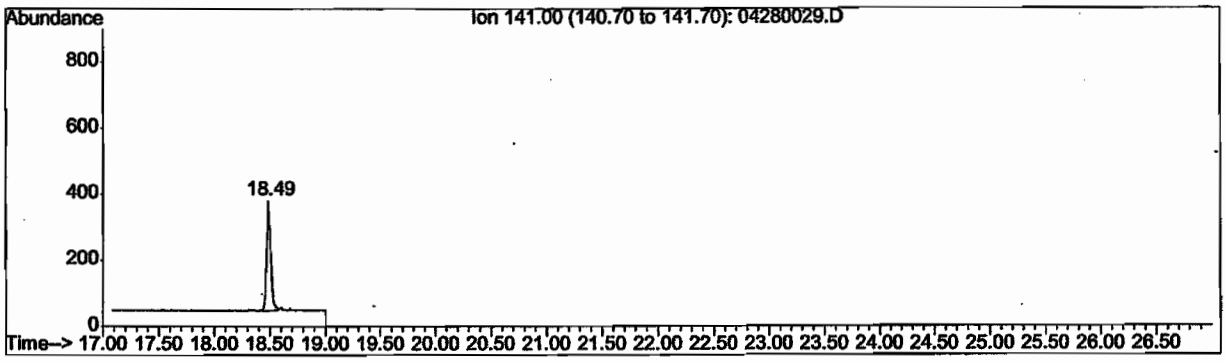
Deltamethrin
0.5 ng/mL
Peak Response (area): 2141

FIGURE 18. Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



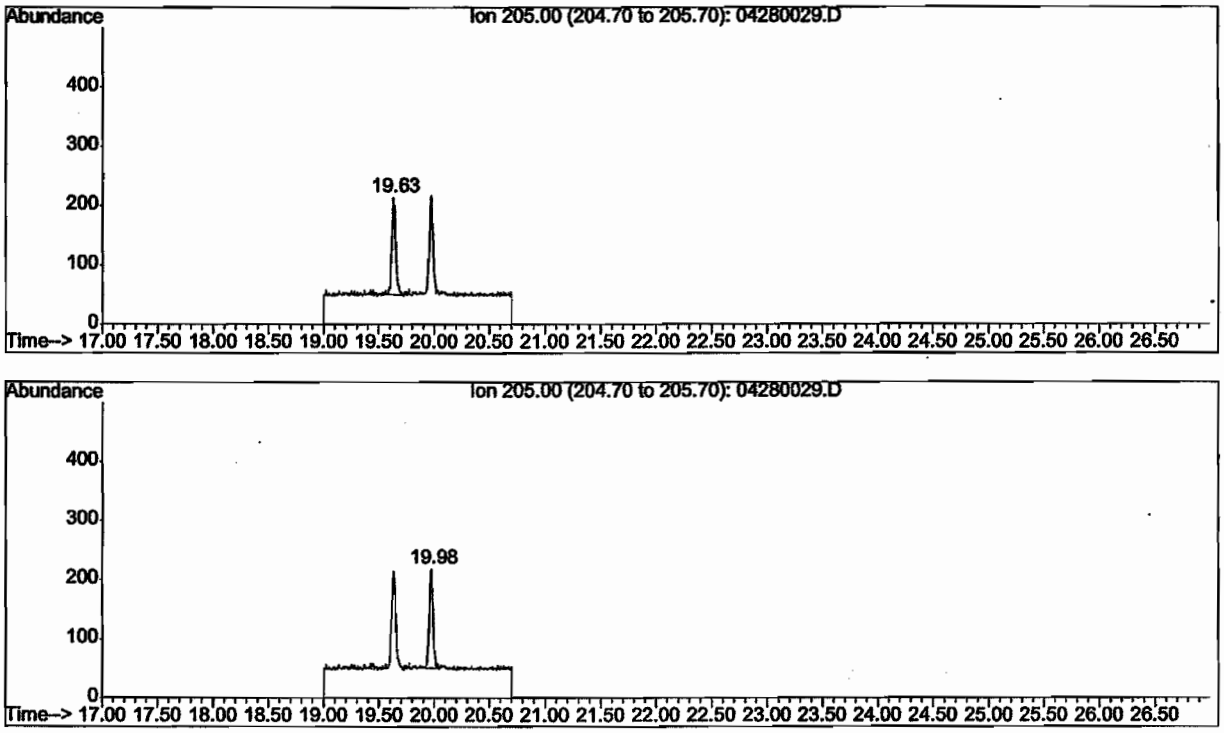
Bifenthrin
1.0 ng/mL
Peak Response (area): 5493



Fenpropathrin
1.0 ng/mL
Peak Response (area): 8251

FIGURE 18.(con't) Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL

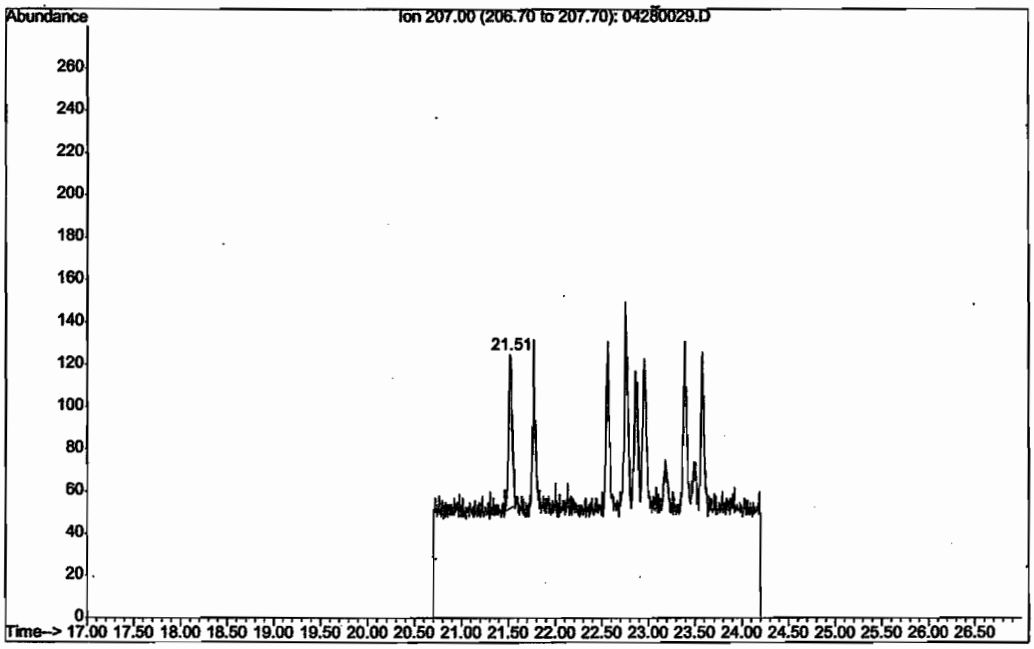
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2

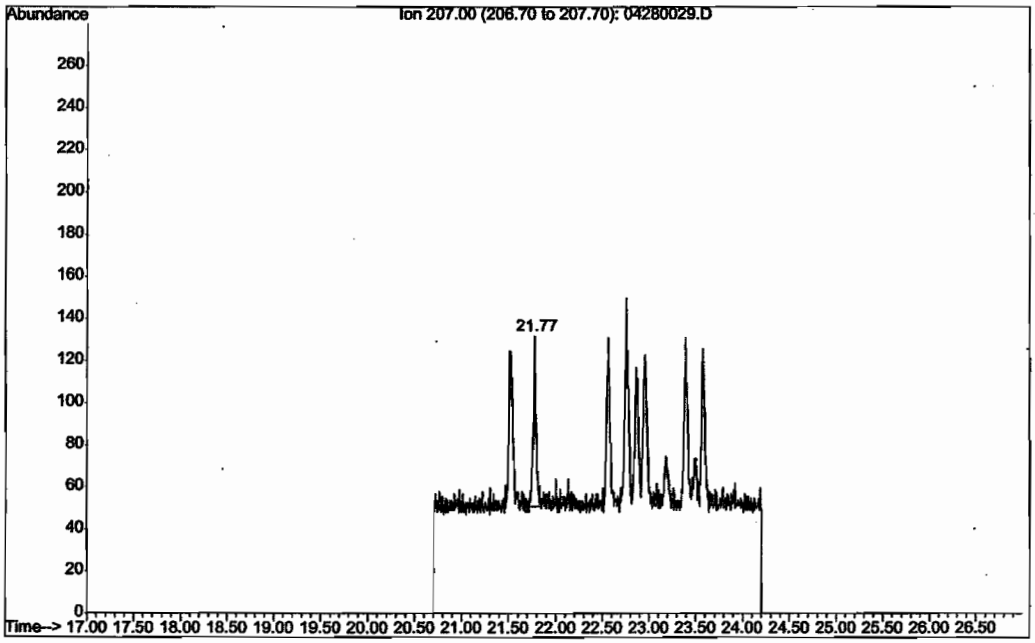


Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 8036

FIGURE 18.(con't) Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2

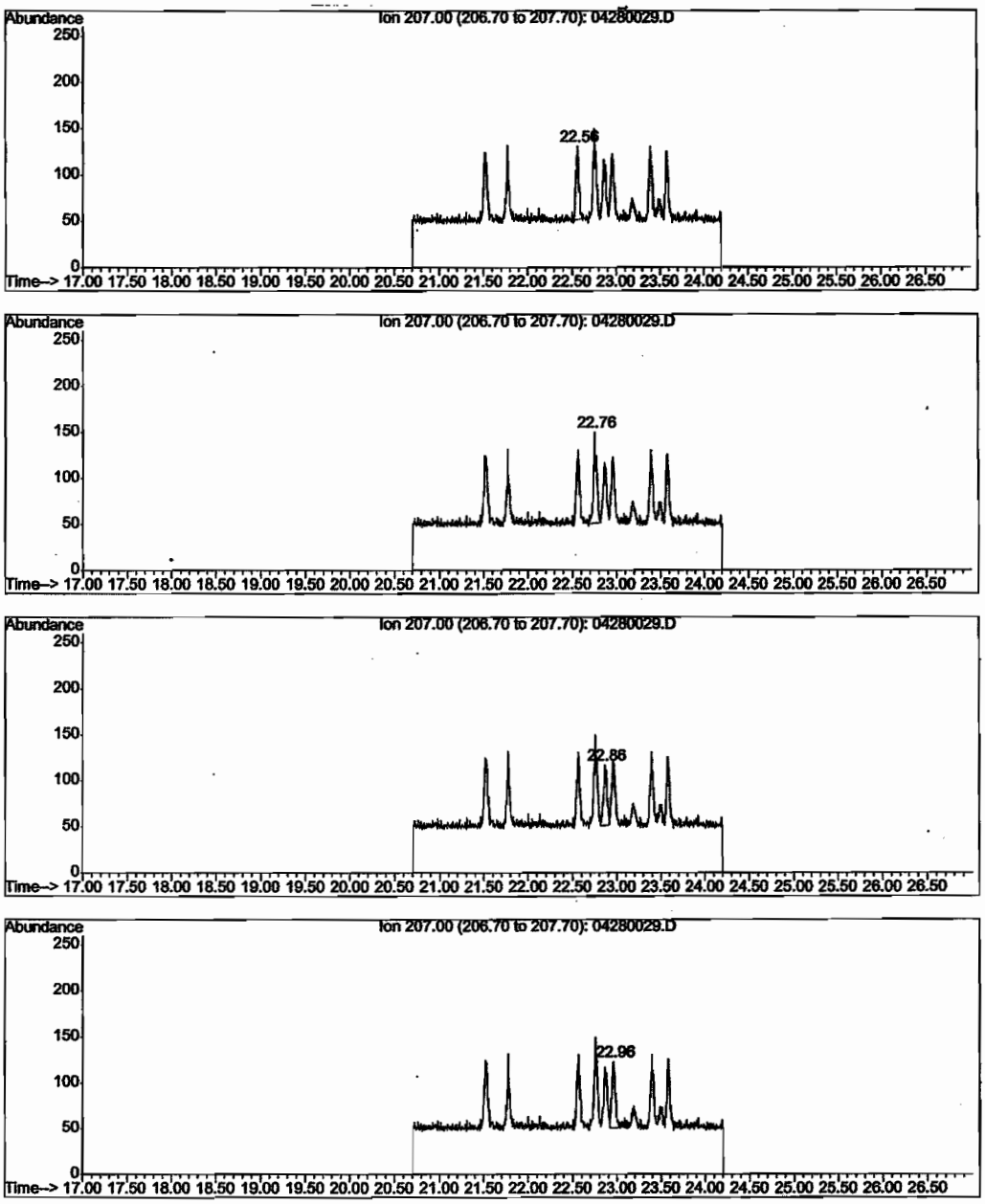




Permethrin
10 ng/mL
Peak Response (area): 3434

FIGURE 18.(con't) Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL

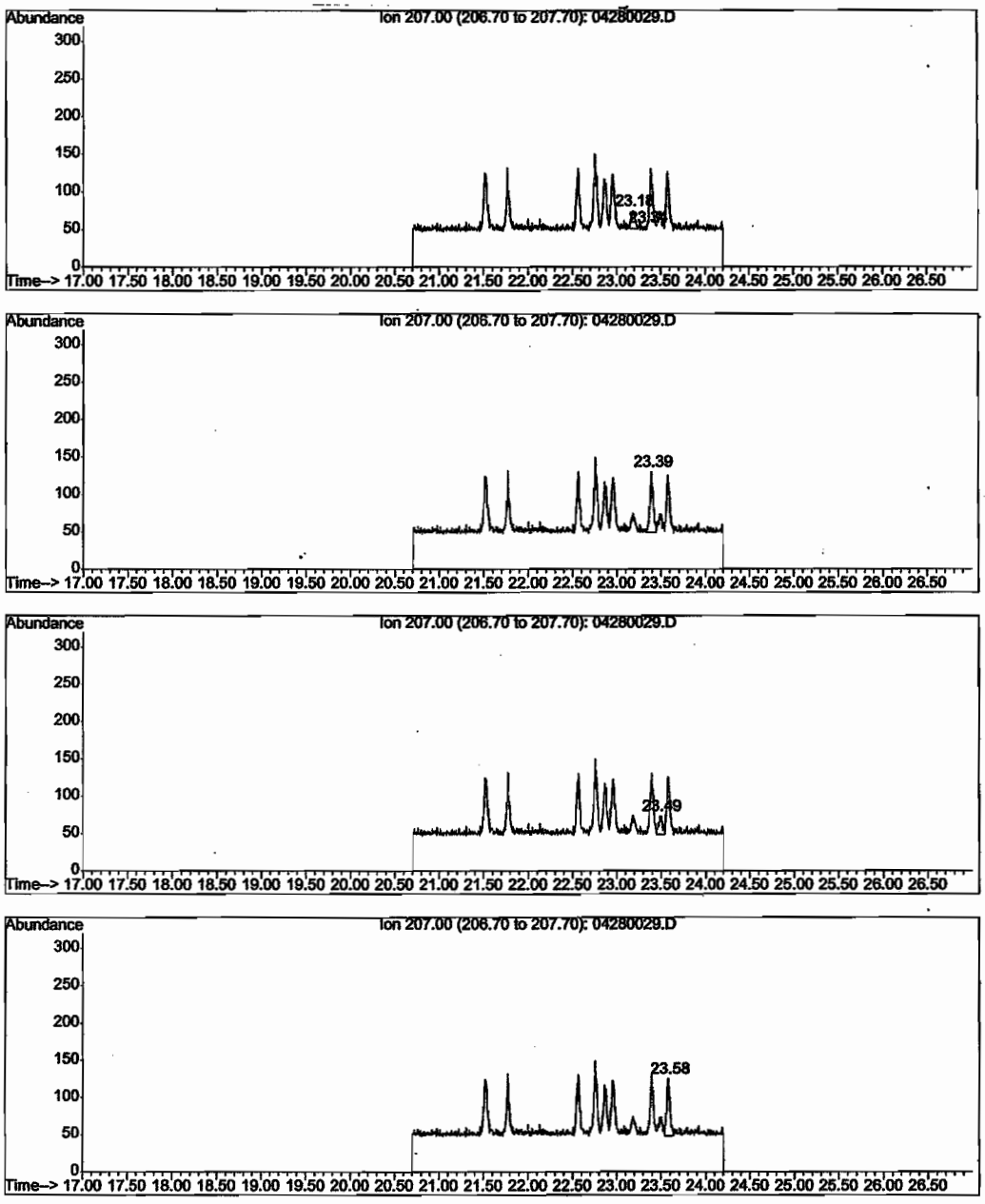
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Cyfluthrin
1.0 ng/mL
Peak Response (area): 8056

FIGURE 18.(con't) Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL

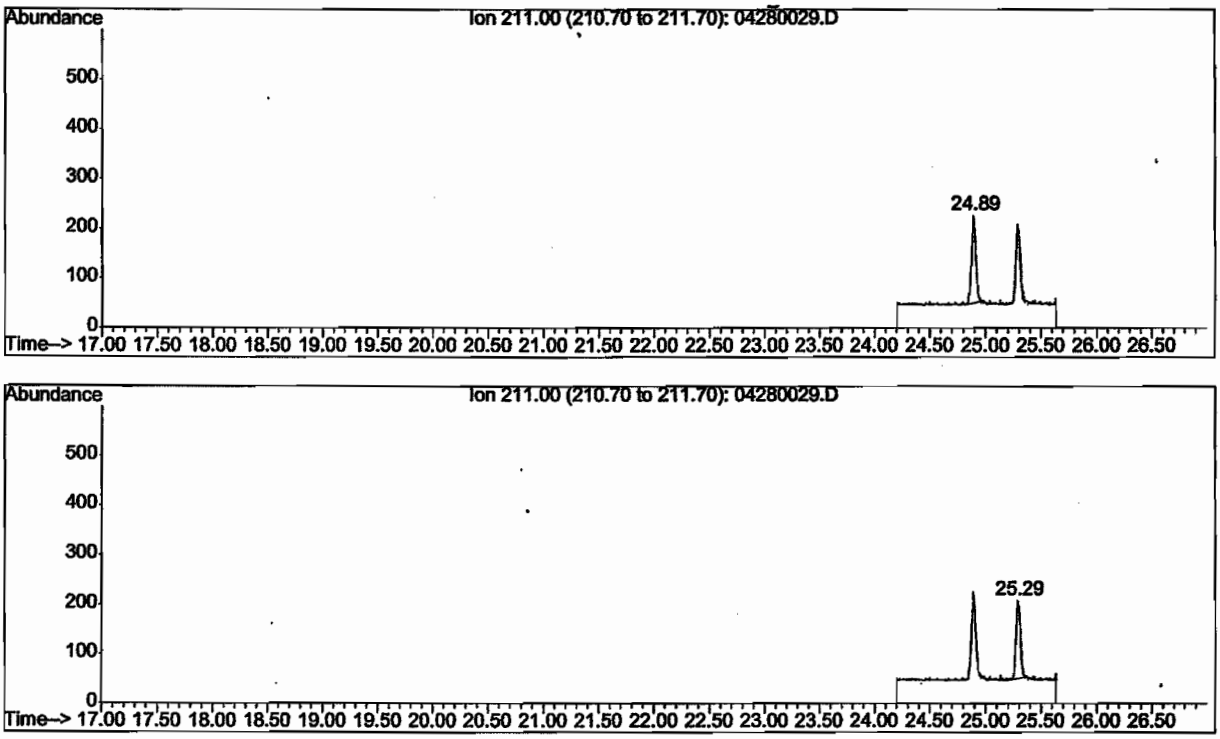
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Cypermethrin
1.0 ng/mL
Peak Response (area): 5474

FIGURE 18.(con't) Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL

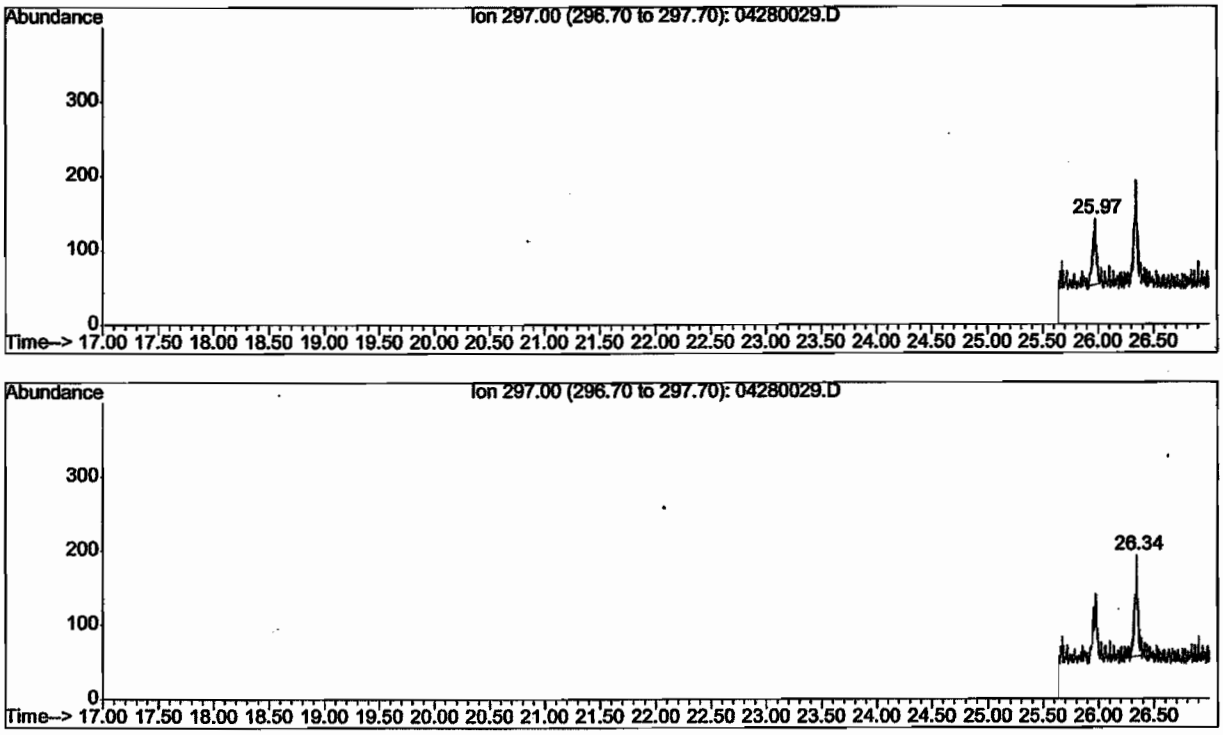
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



Esfenvalerate
1.0 ng/mL
Peak Response (area): 8960

FIGURE 18.(con't) Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL

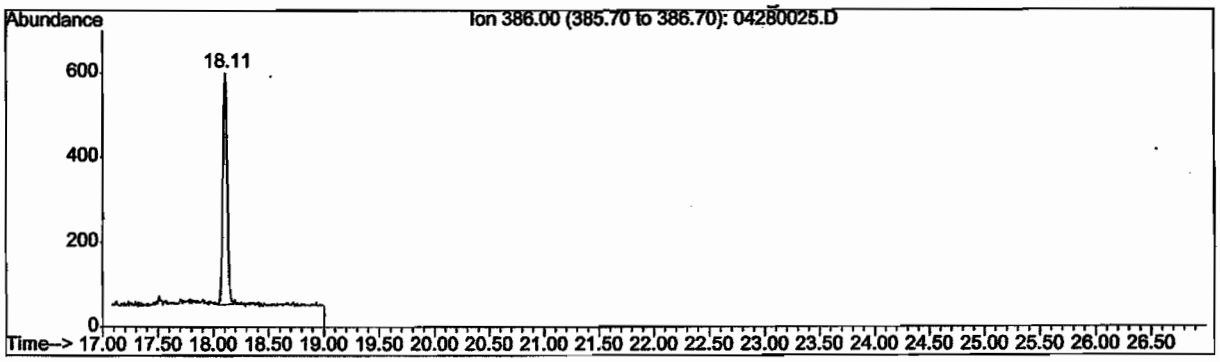
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



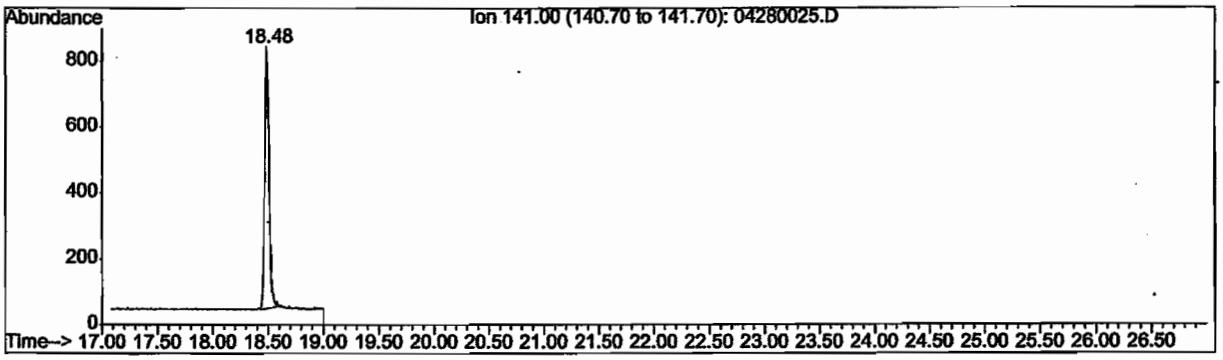
Deltamethrin
1.0 ng/mL
Peak Response (area): 4572

FIGURE 19. Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL

Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin, Set #2



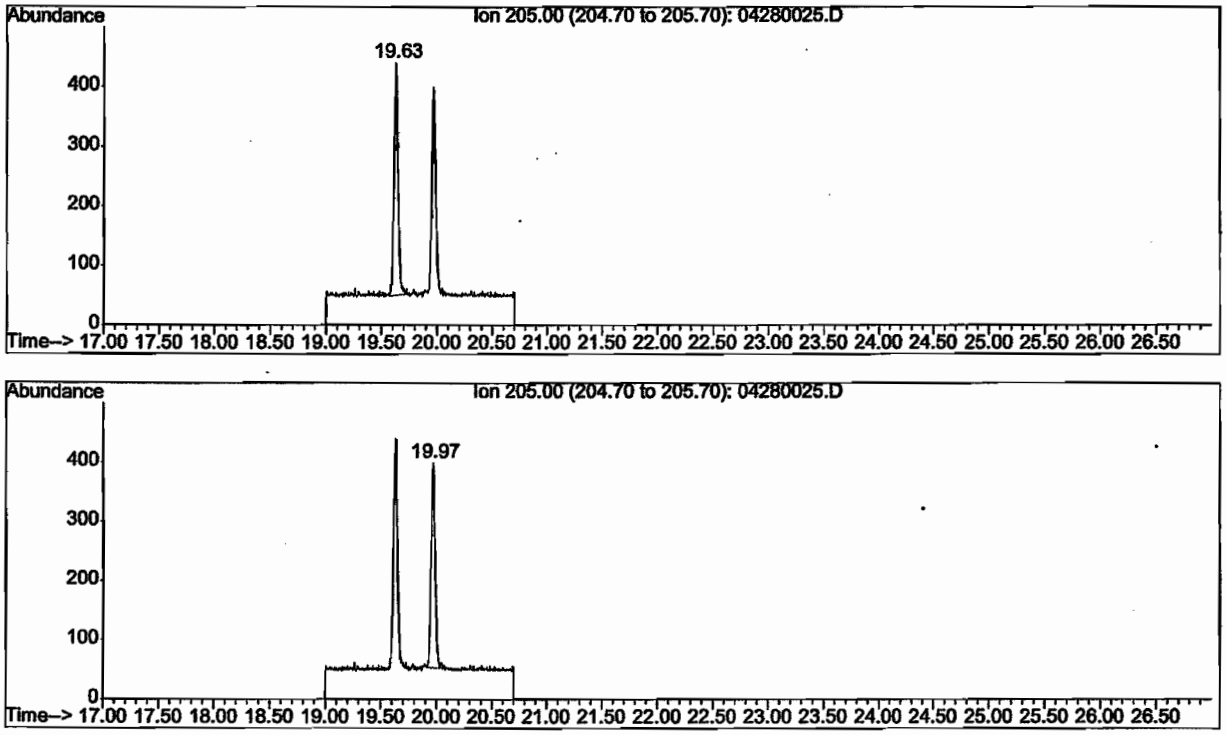
Bifenthrin
2.5 ng/mL
Peak Response (area): 14349



Fenpropathrin
2.5 ng/mL
Peak Response (area): 21056

FIGURE 19. (con't) Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL

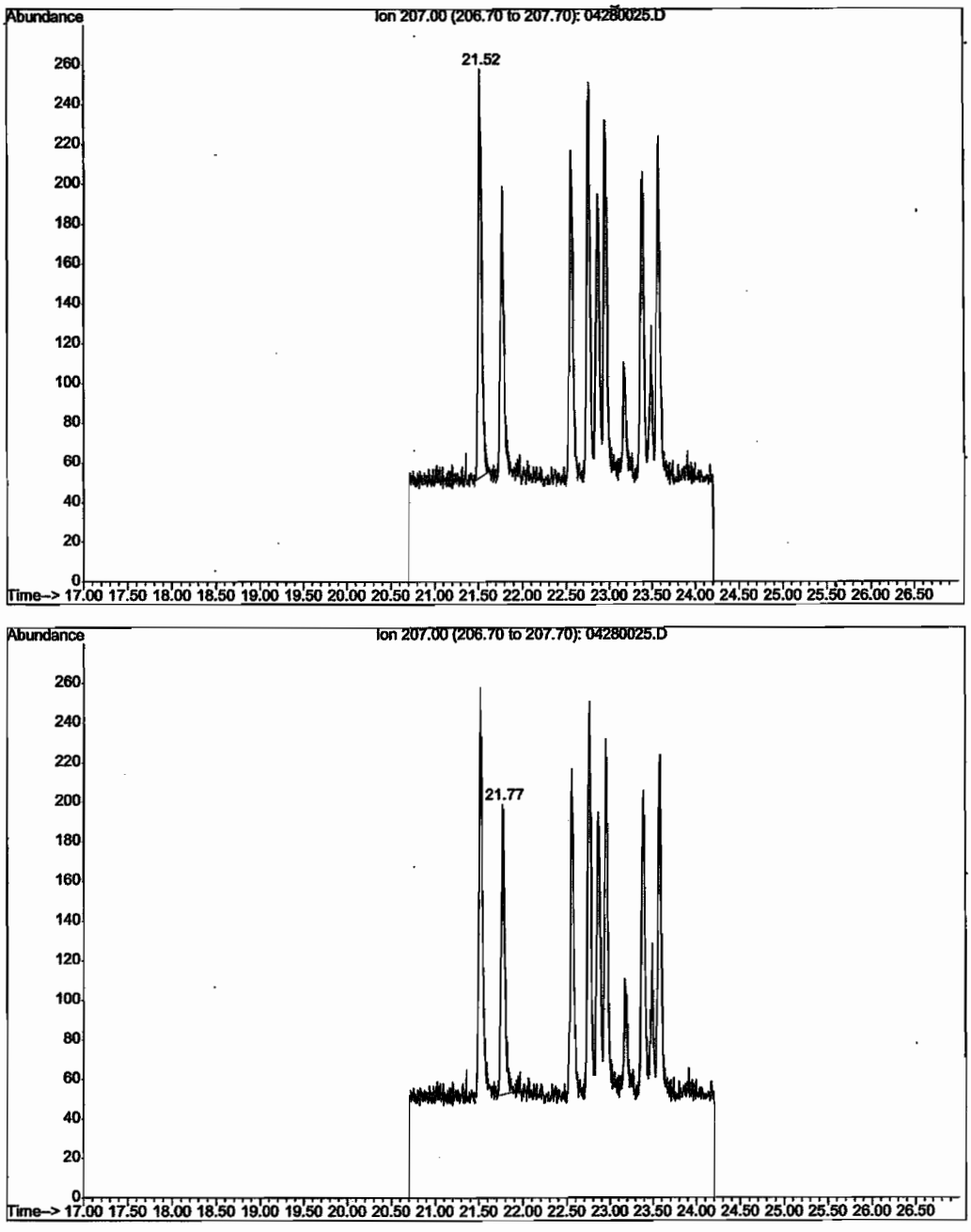
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
2.5 ng/mL
Peak Response (area): 18359

FIGURE 19. (con't) Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL

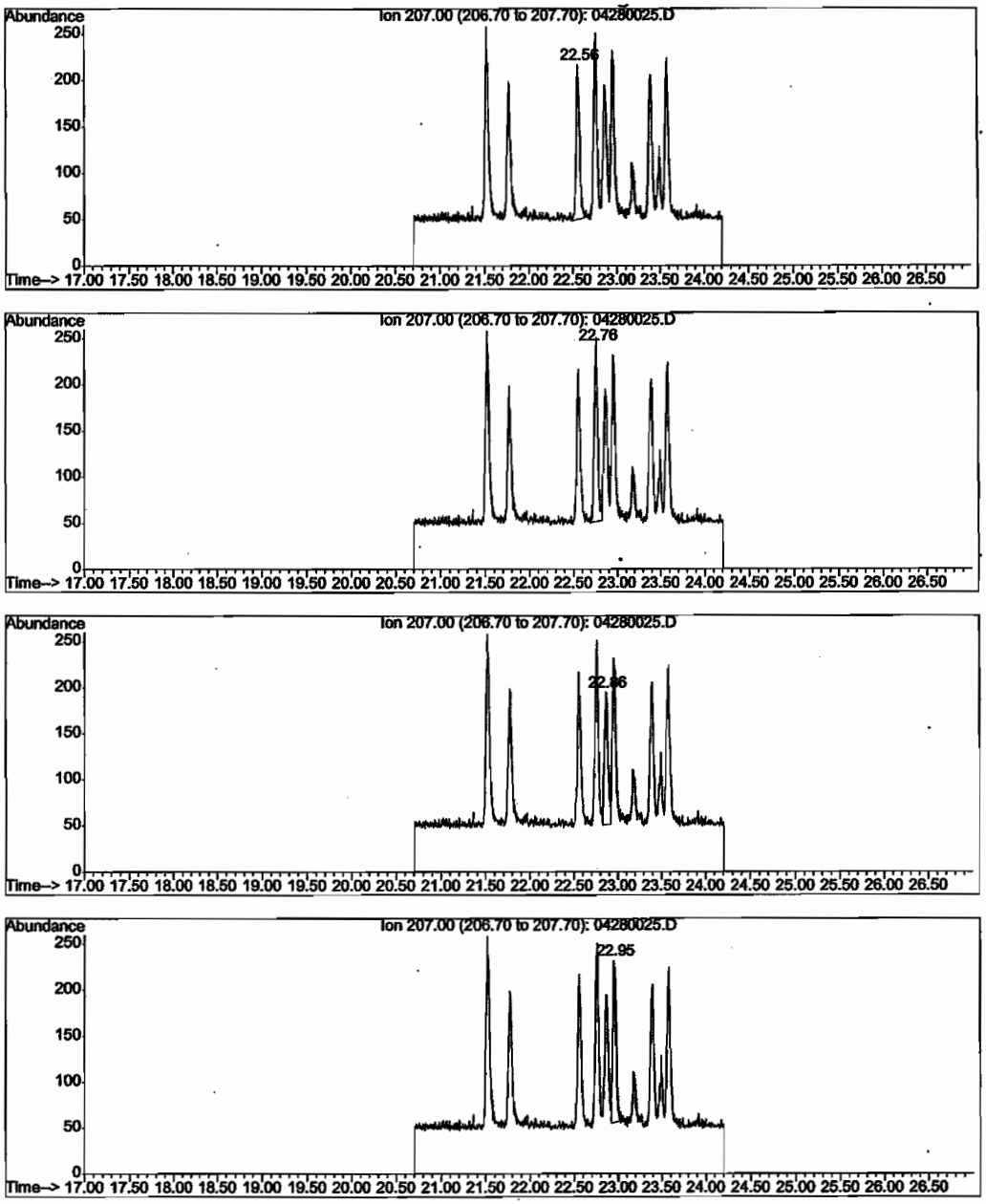
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin, Set #2



Permethrin
25.0 ng/mL
Peak Response (area): 9434

FIGURE 19. (con't) Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL

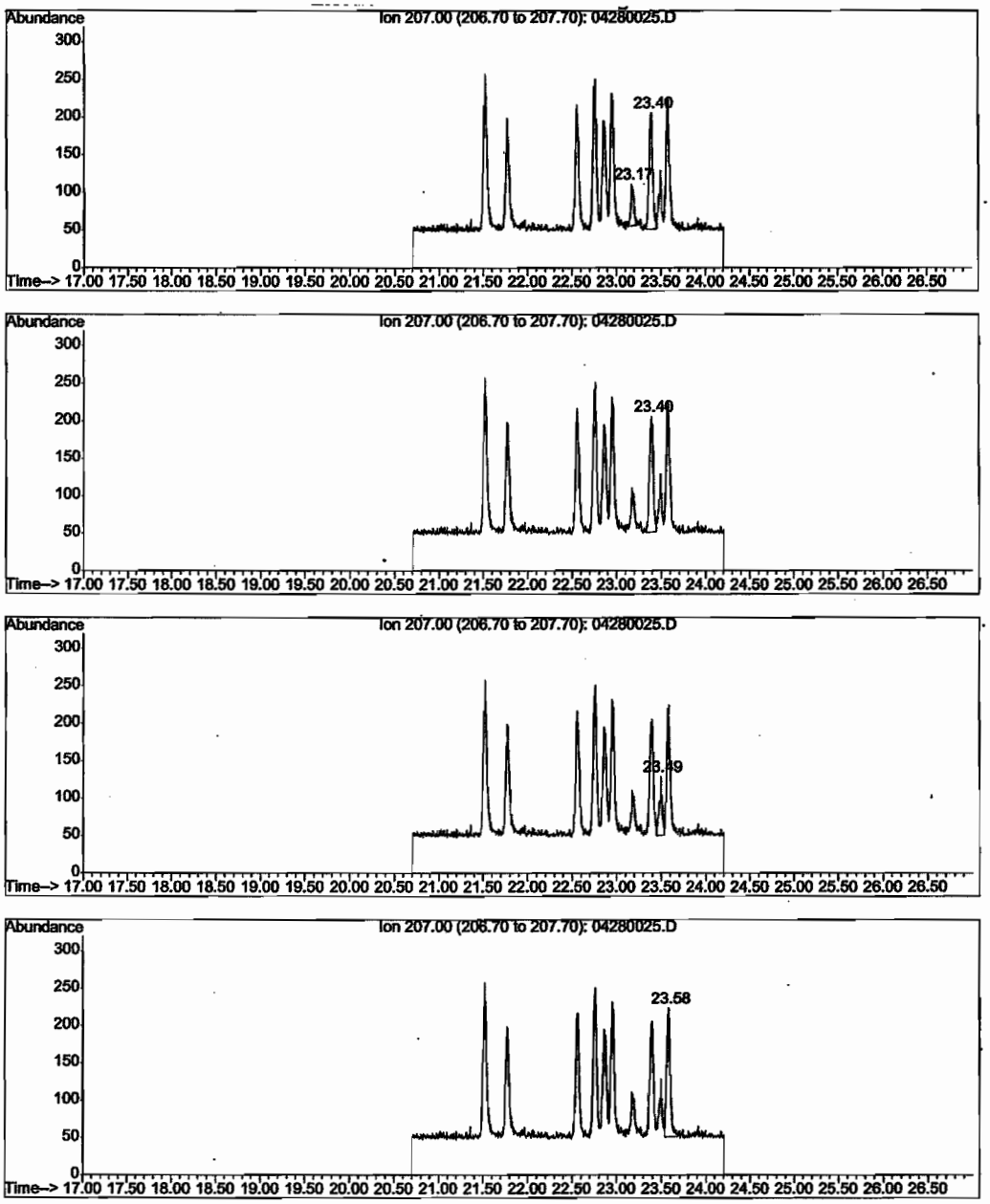
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin, Set #2



Cyfluthrin
2.5 ng/mL
Peak Response (area): 18640

FIGURE 19. (con't) Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL

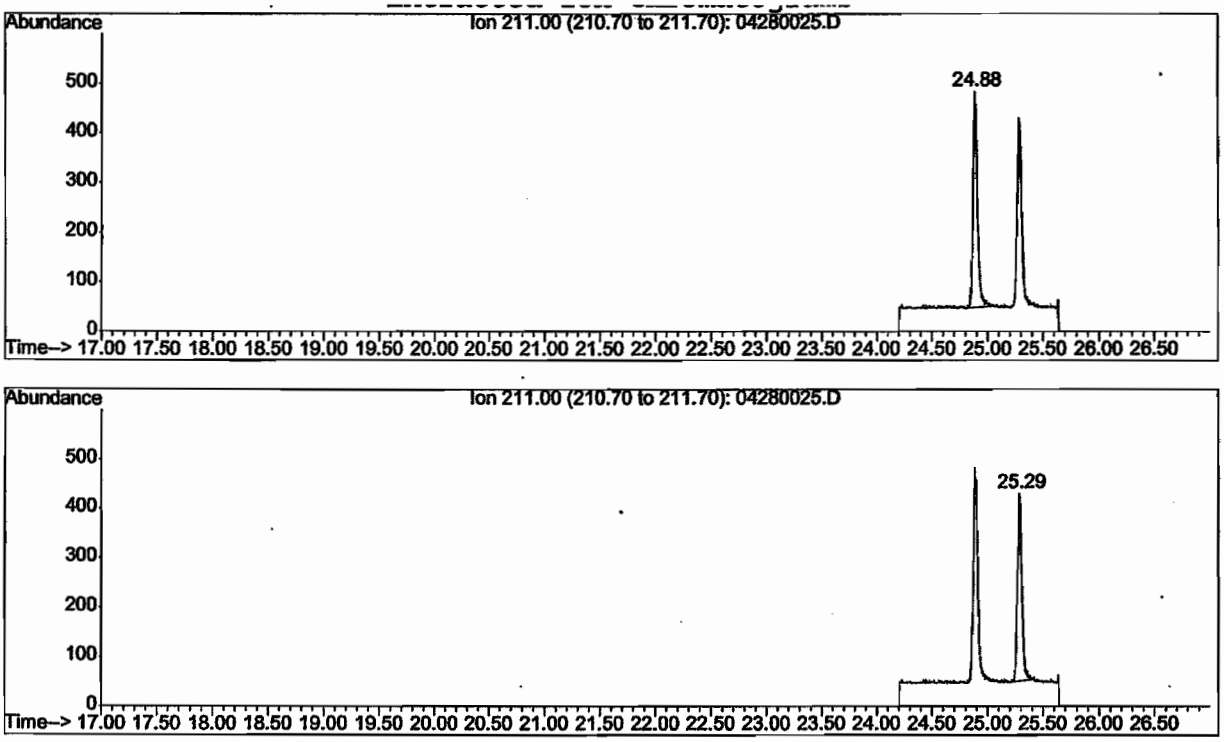
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin, Set #2



Cypermethrin
2.5 ng/mL
Peak Response (area): 12184

FIGURE 19. (con't) Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL

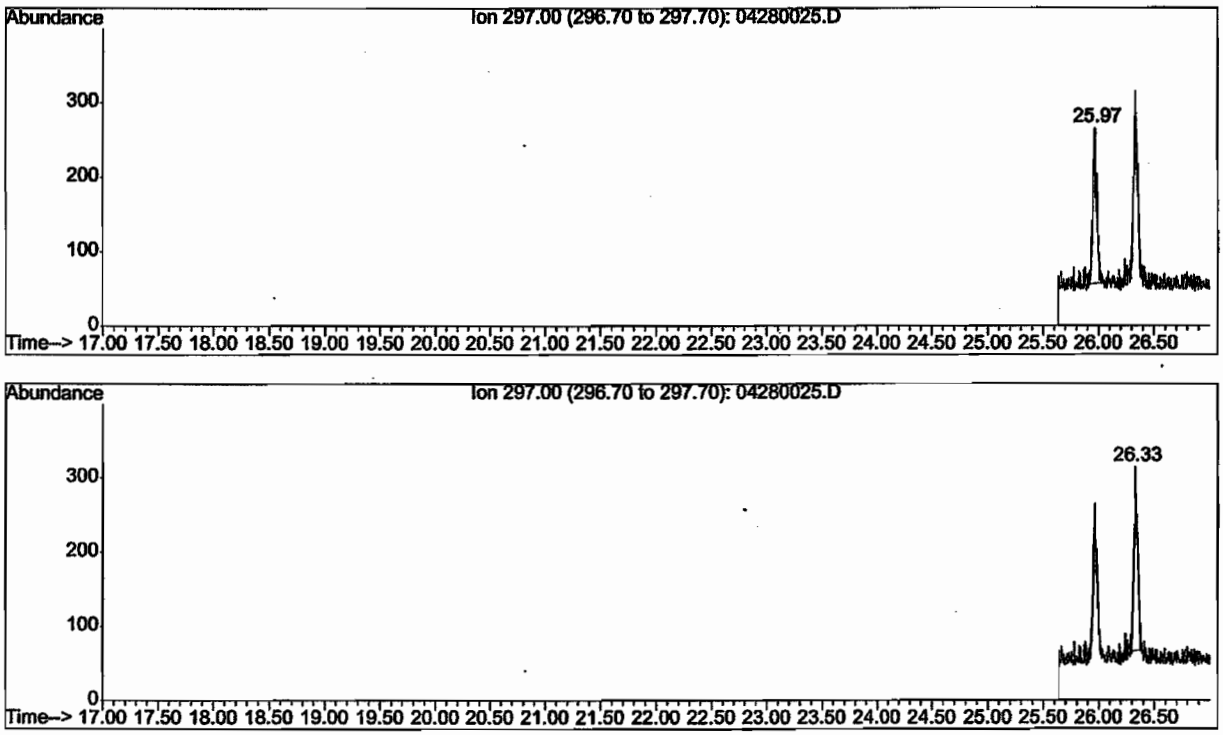
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin, Set #2



Esfenvalerate
2.5 ng/mL
Peak Response (area): 22472

FIGURE 19. (con't) Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL

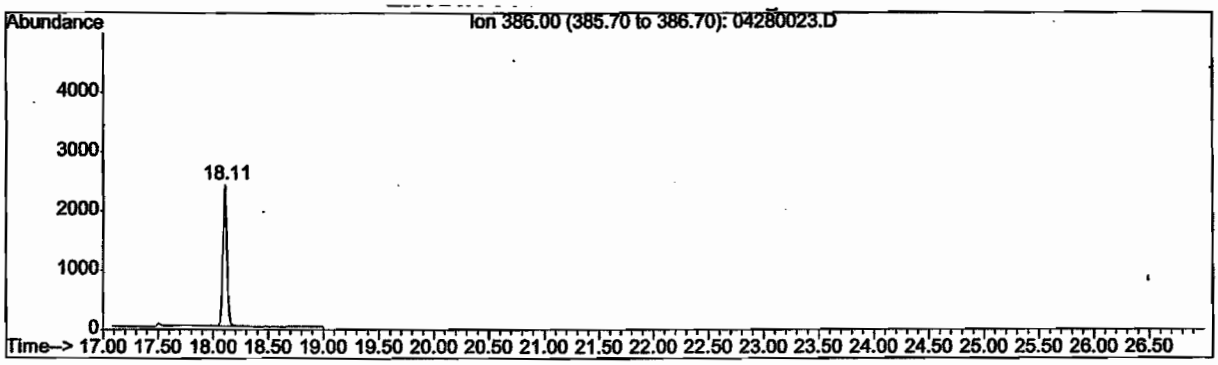
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin, Set #2



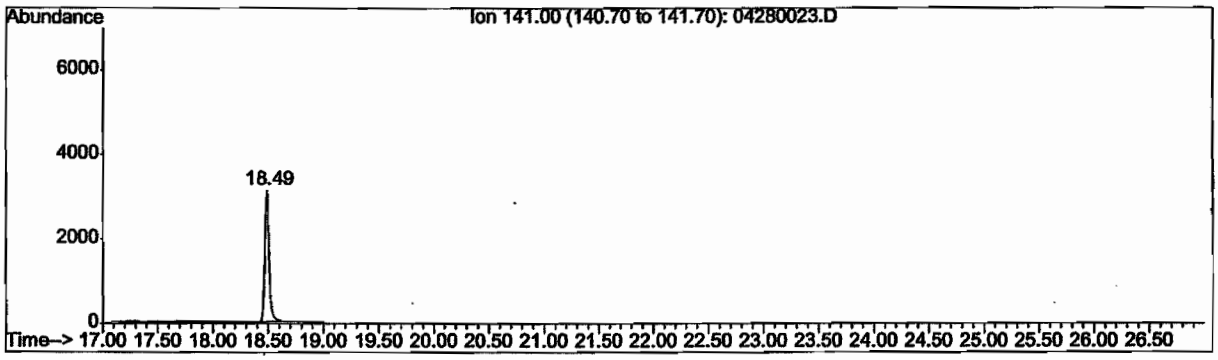
Deltamethrin
2.5 ng/mL
Peak Response (area): 10511

FIGURE 20. Representative Chromatograms - Calibration Standards @ 10/100 ng/mL

Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin, Set #2



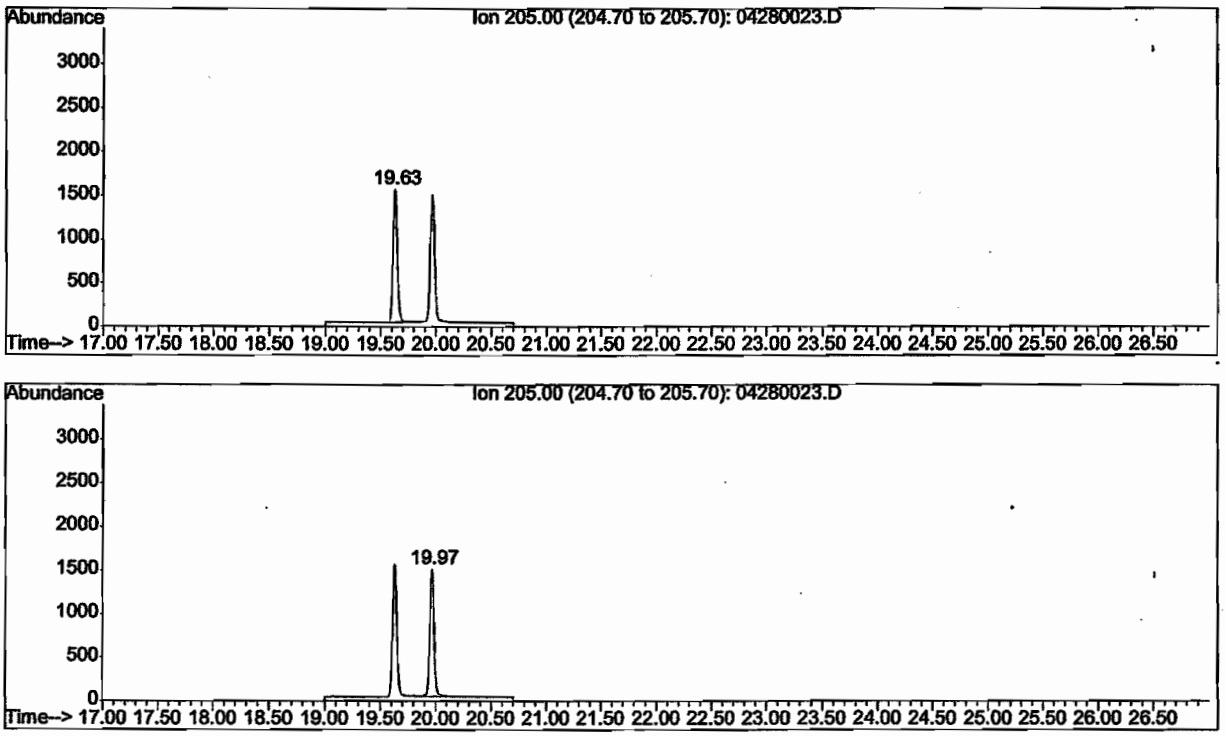
Bifenthrin
10 ng/mL
Peak Response (area): 58285



Fenpropathrin
10 ng/mL
Peak Response (area): 86322

FIGURE 20. (con't) Representative Chromatograms - Calibration Standards @ 10/100 ng/mL

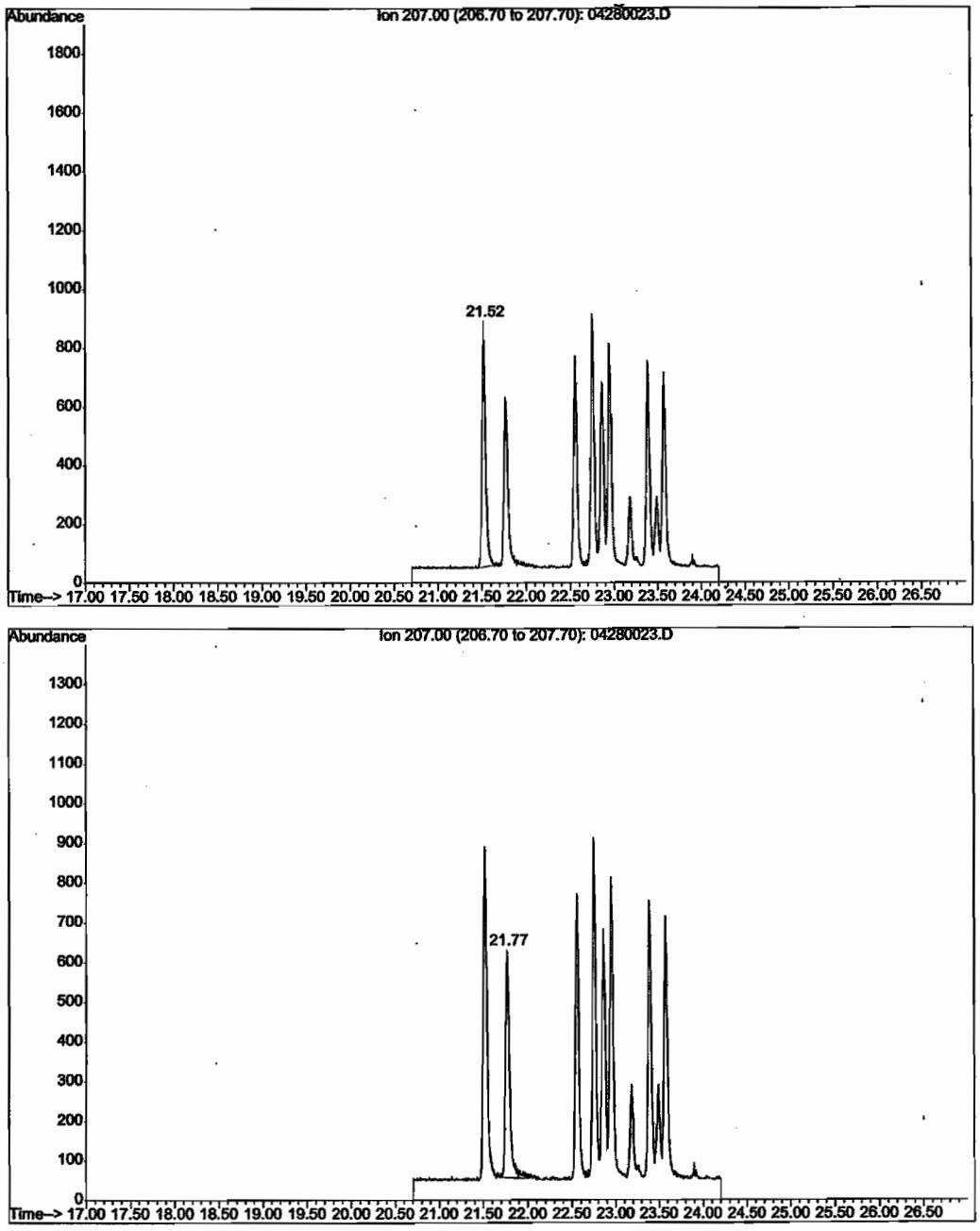
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
10 ng/mL
Peak Response (area): 81386

FIGURE 20. (con't) Representative Chromatograms - Calibration Standards @ 10/100 ng/mL

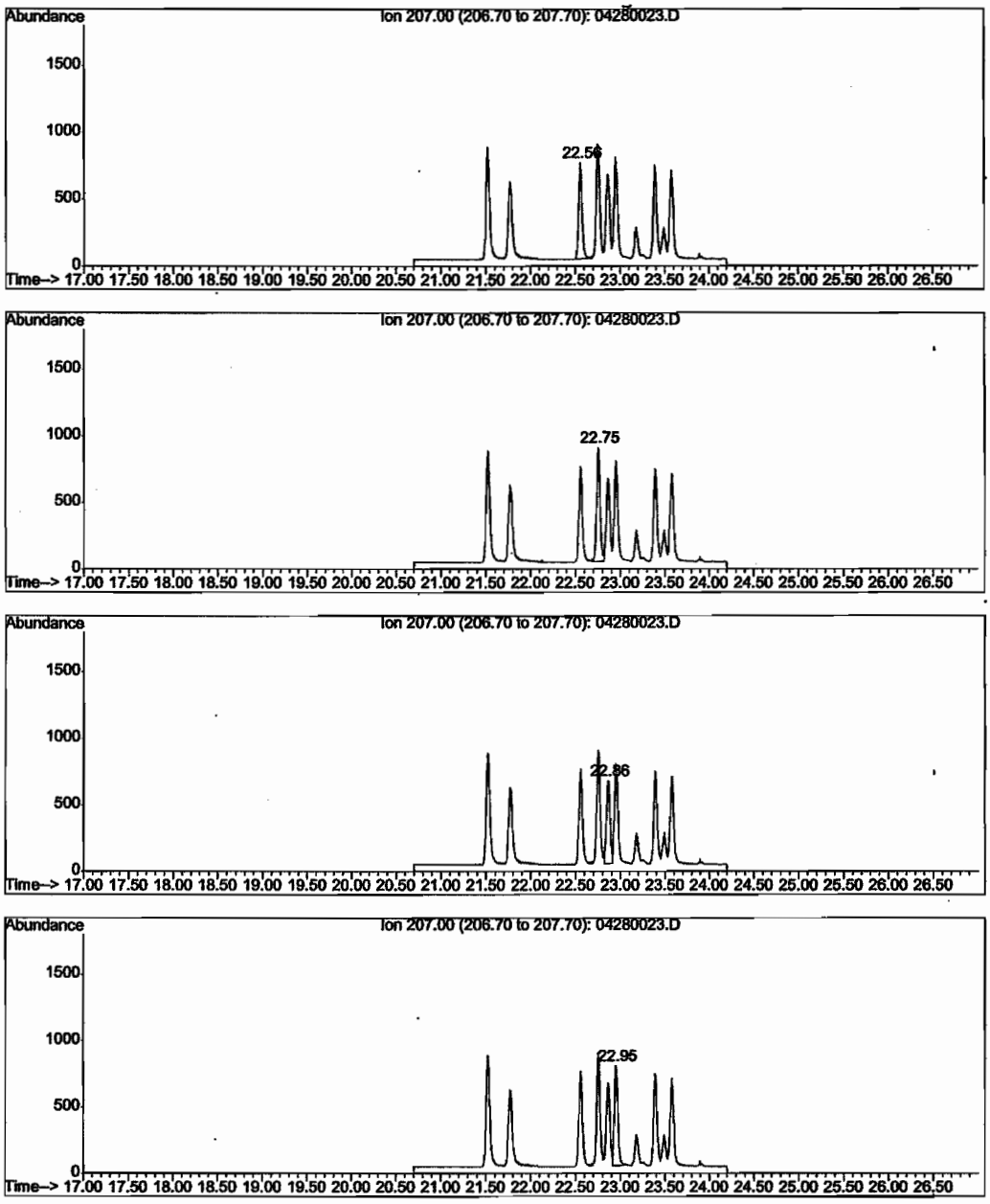
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin, Set #2



Permethrin
100 ng/mL
Peak Response (area): 42339

FIGURE 20. (con't) Representative Chromatograms - Calibration Standards @ 10/100 ng/mL

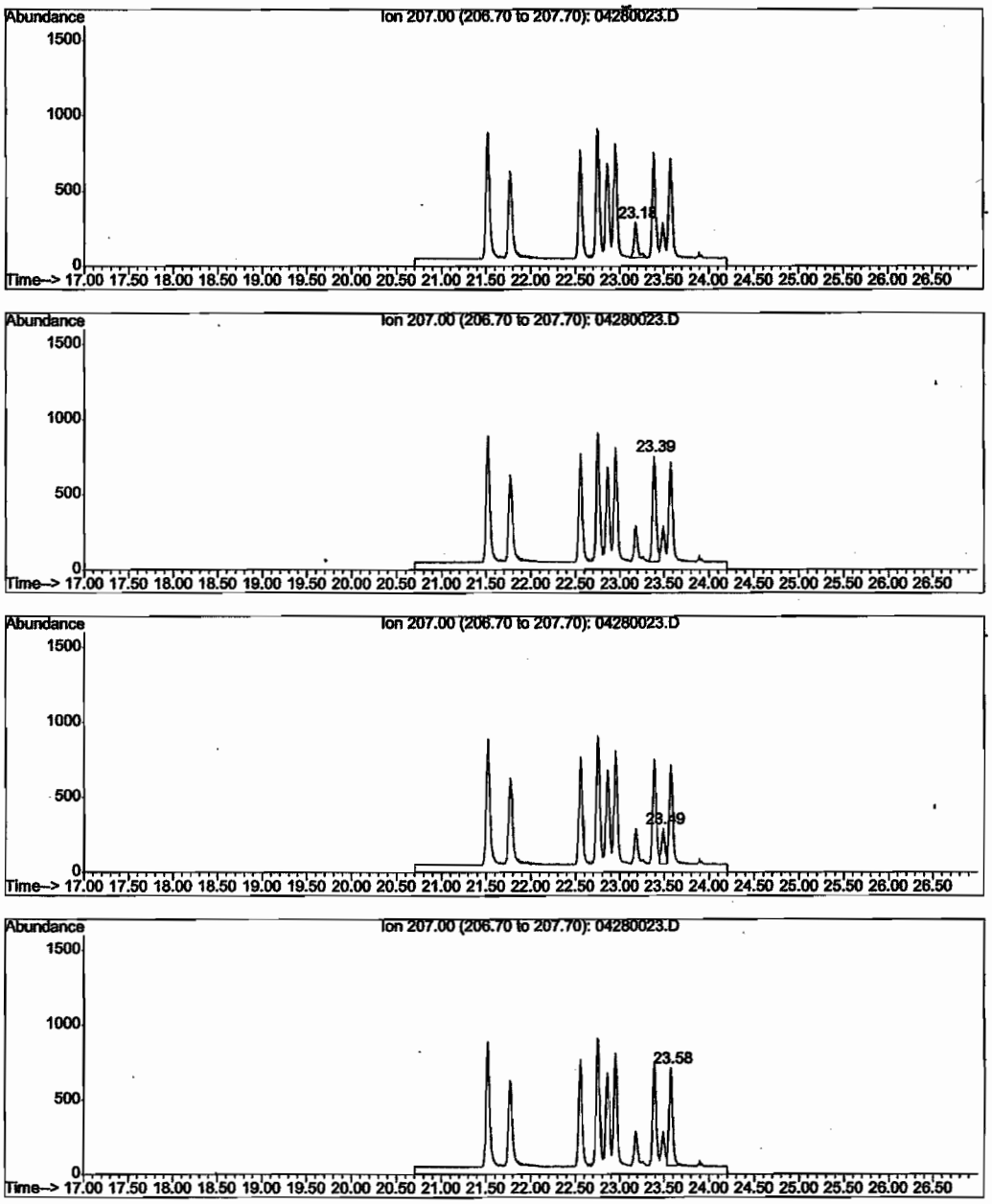
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin, Set #2



Cyfluthrin
10 ng/mL
Peak Response (area): 79513

FIGURE 20. (con't) Representative Chromatograms - Calibration Standards @ 10/100 ng/mL

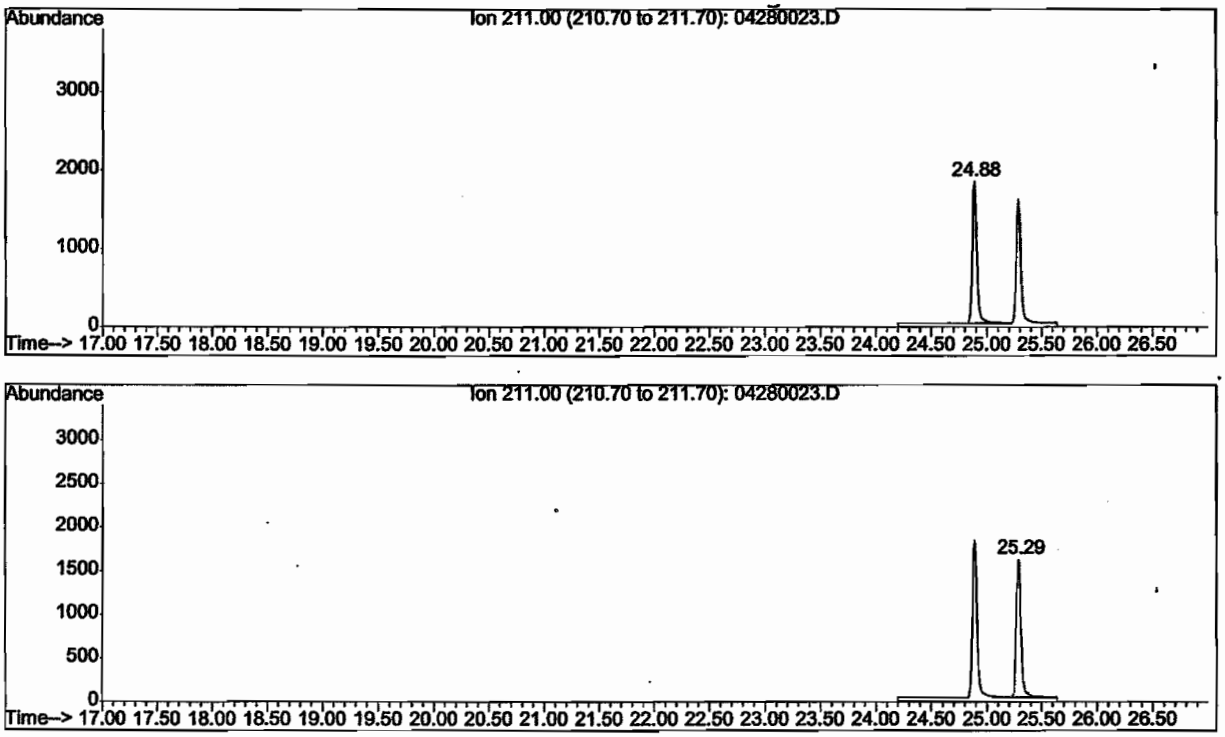
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin, Set #2



Cypermethrin
10 ng/mL
Peak Response (area): 50030

FIGURE 20. (con't) Representative Chromatograms - Calibration Standards @ 10/100 ng/mL

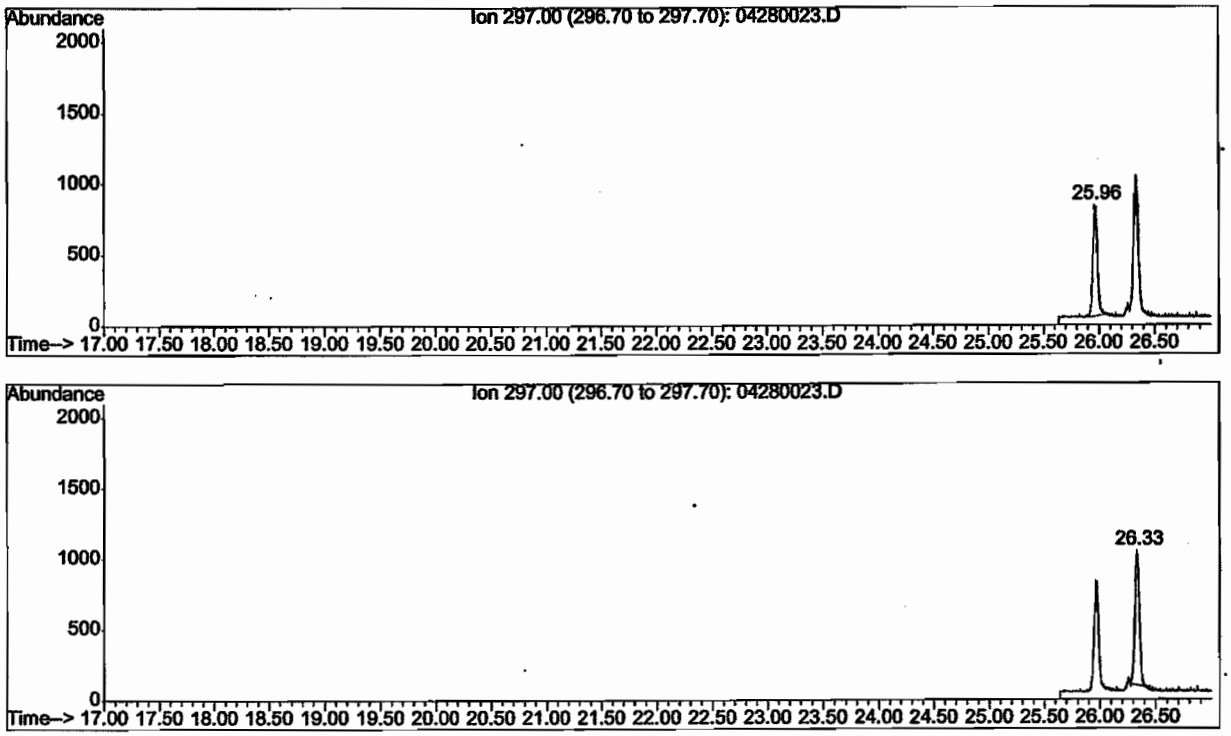
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin, Set #2



Esfenvalerate
10 ng/mL
Peak Response (area): 99991

FIGURE 20. (con't) Representative Chromatograms - Calibration Standards @ 10/100 ng/mL

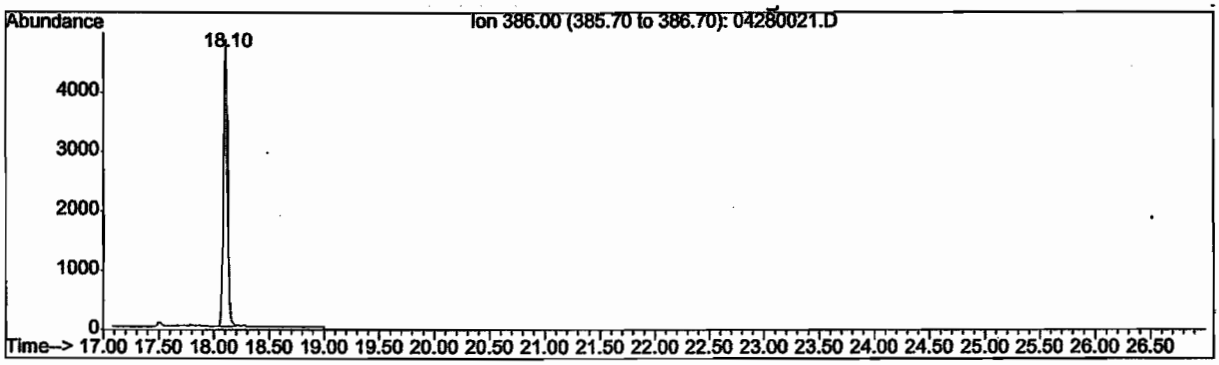
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin, Set #2



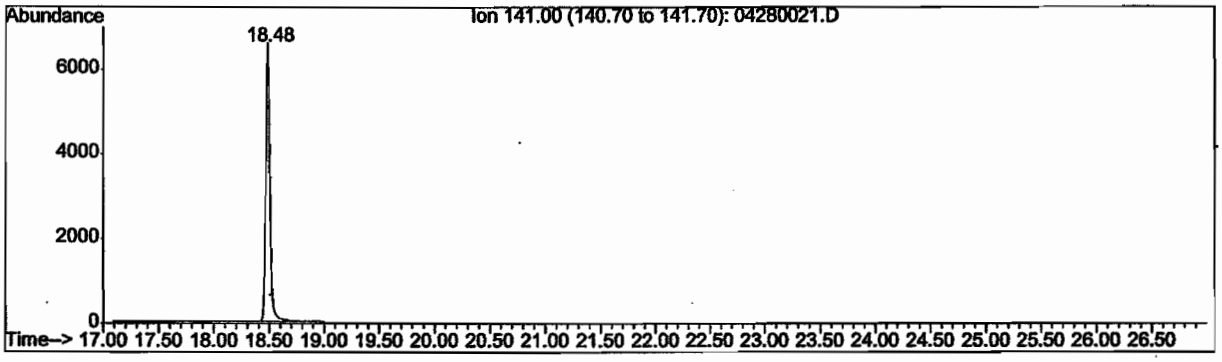
Deltamethrin
10 ng/mL
Peak Response (area): 47234

FIGURE 21. Representative Chromatograms - Calibration Standards @ 20/200 ng/mL

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



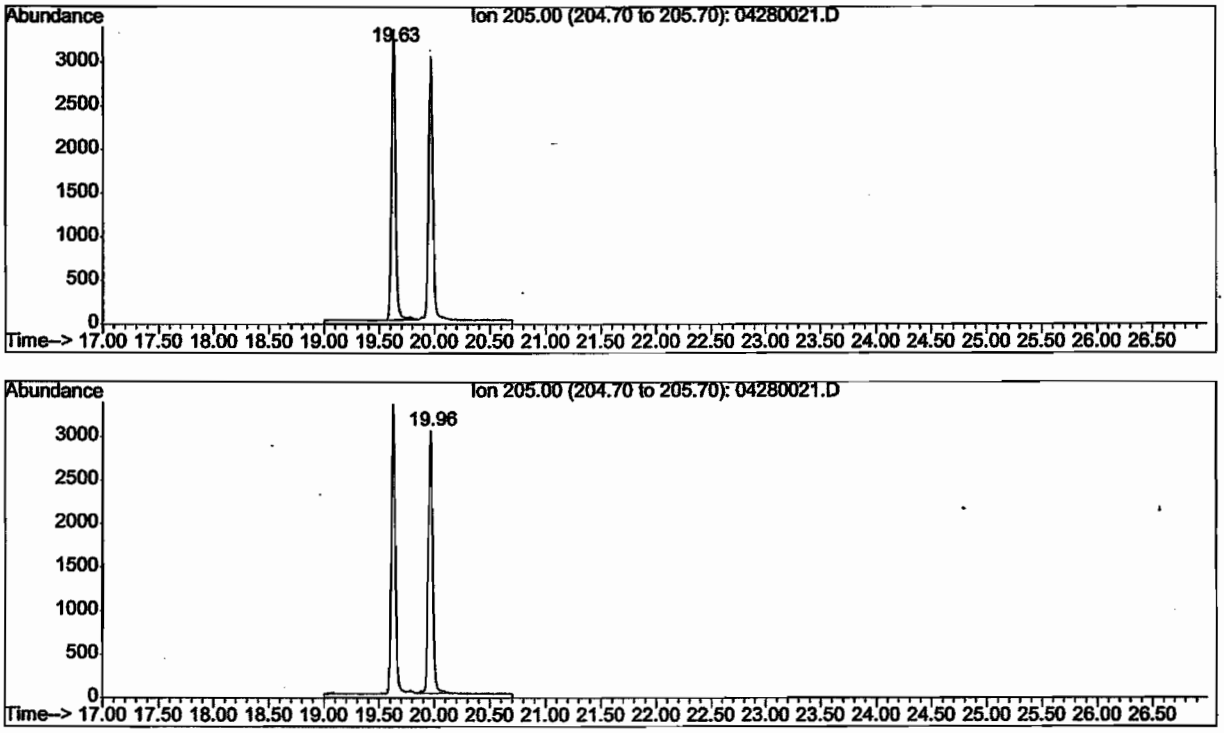
Bifenthrin
20 ng/mL
Peak Response (area): 119698



Fenpropathrin
20 ng/mL
Peak Response (area): 180408

FIGURE 21.(con't) Representative Chromatograms - Calibration Standards @ 20/200 ng/mL

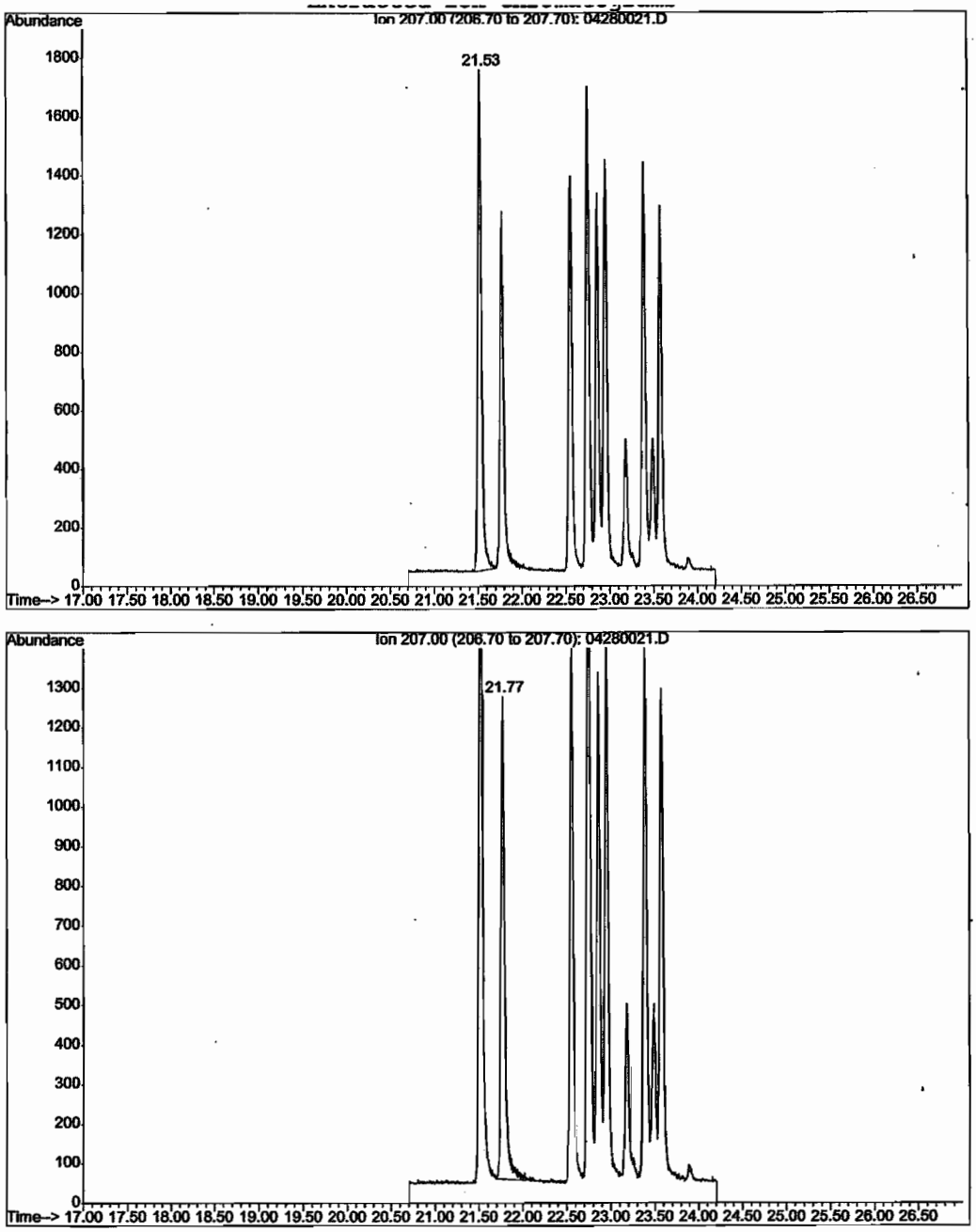
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 165637

FIGURE 21.(con't) Representative Chromatograms - Calibration Standards @ 20/200 ng/mL

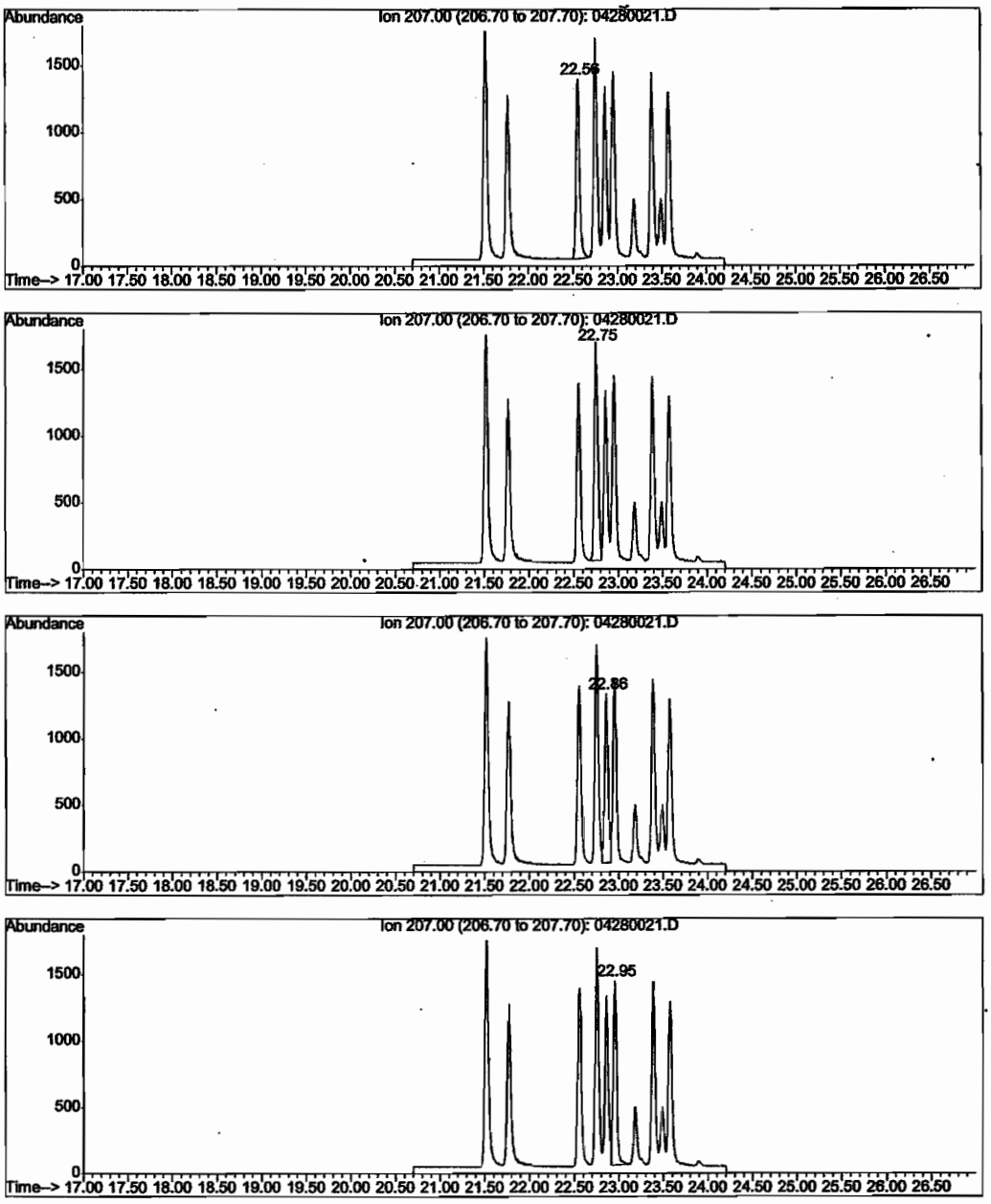
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Permethrin
200 ng/mL
Peak Response (area): 88355

FIGURE 21.(con't) Representative Chromatograms - Calibration Standards @ 20/200 ng/mL

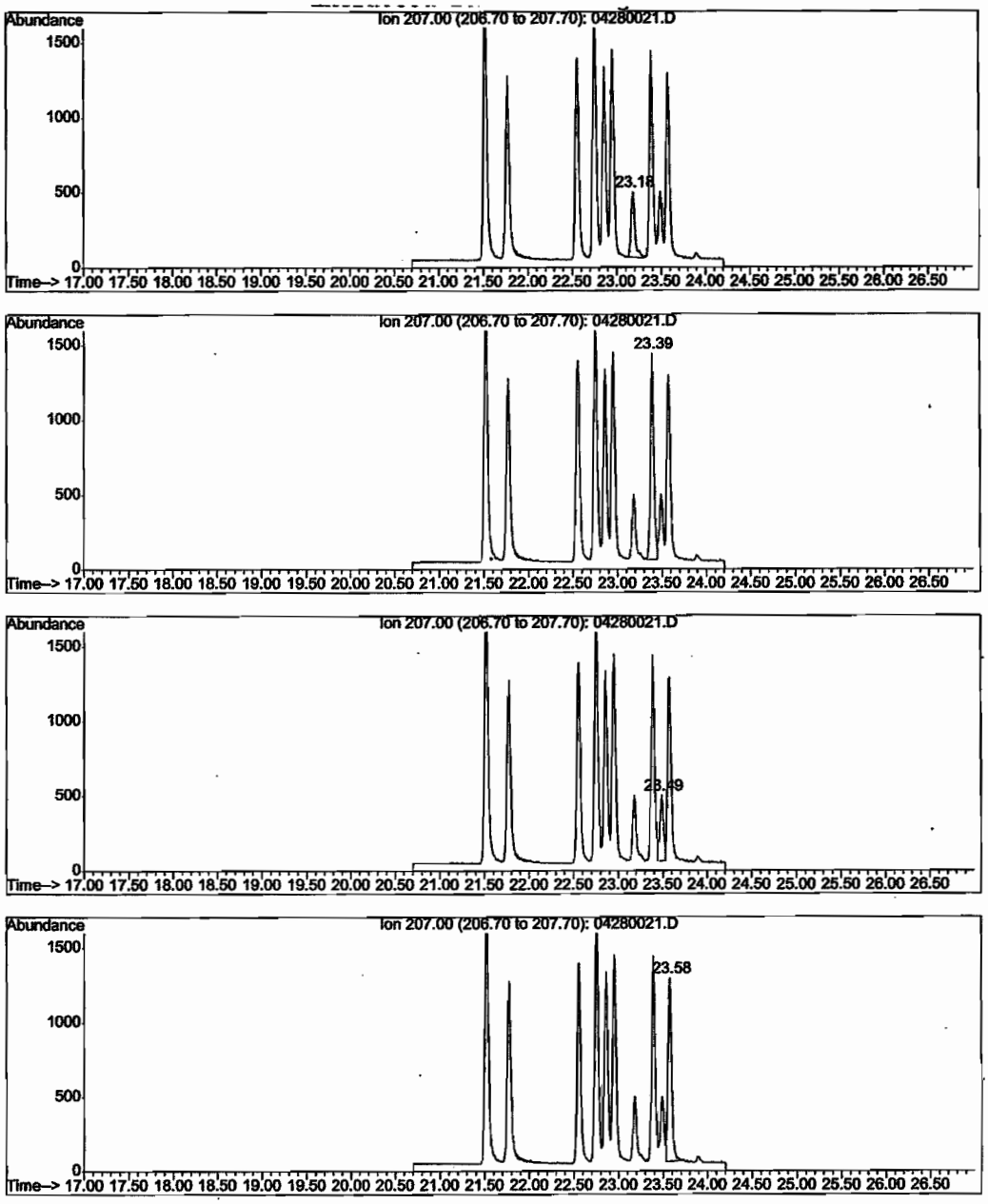
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Cyfluthrin
20 ng/mL
Peak Response (area): 156003

FIGURE 21.(con't) Representative Chromatograms - Calibration Standards @ 20/200 ng/mL

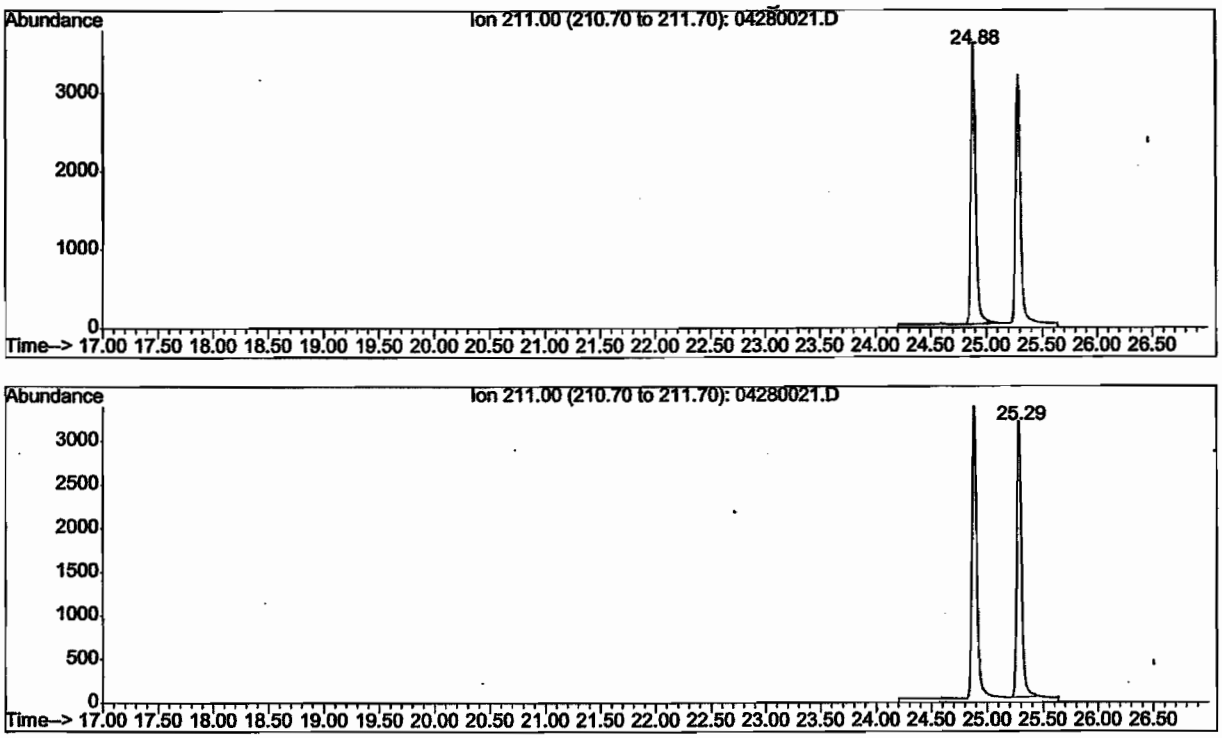
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Cypermethrin
20 ng/mL
Peak Response (area): 97707

FIGURE 21.(con't) Representative Chromatograms - Calibration Standards @ 20/200 ng/mL

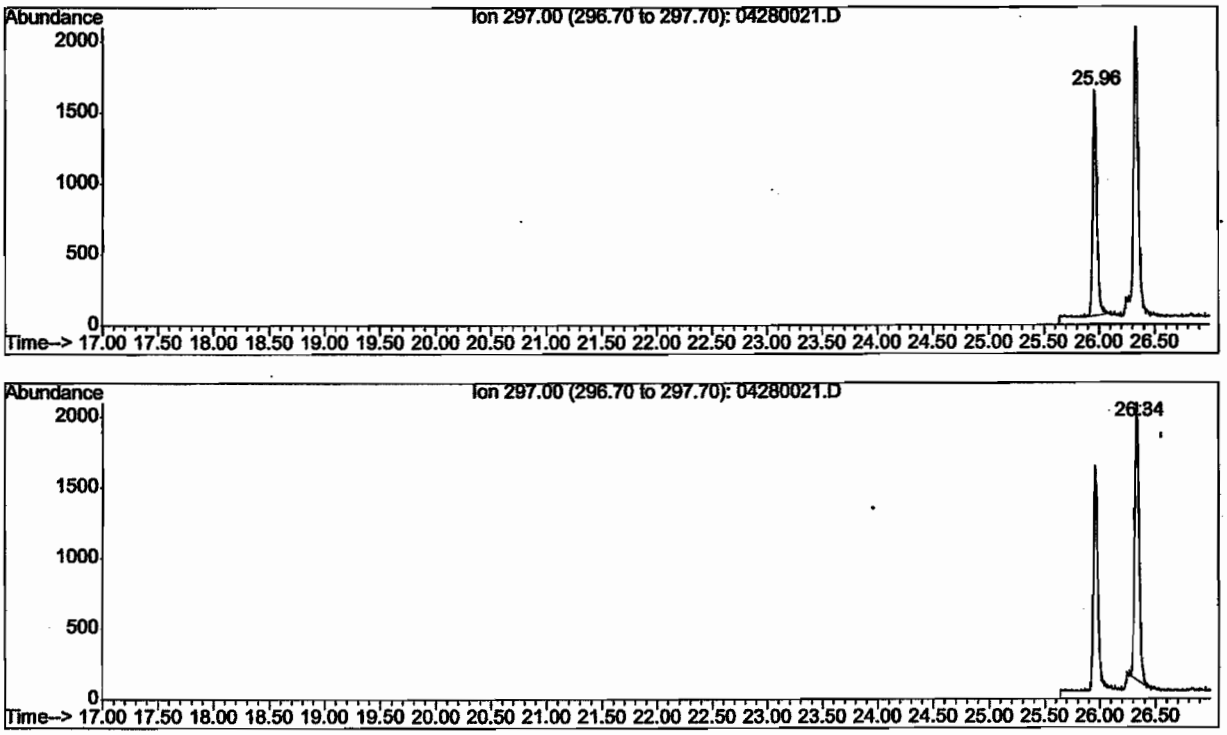
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Esfenvalerate
20 ng/mL
Peak Response (area): 197023

FIGURE 21.(con't) Representative Chromatograms - Calibration Standards @ 20/200 ng/mL

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin, Set #2



Deltamethrin
20 ng/mL
Peak Response (area): 96652

FIGURE 22. Representative Calibration Curves

Representative Bifenthrin Calibration Curve, Set #2

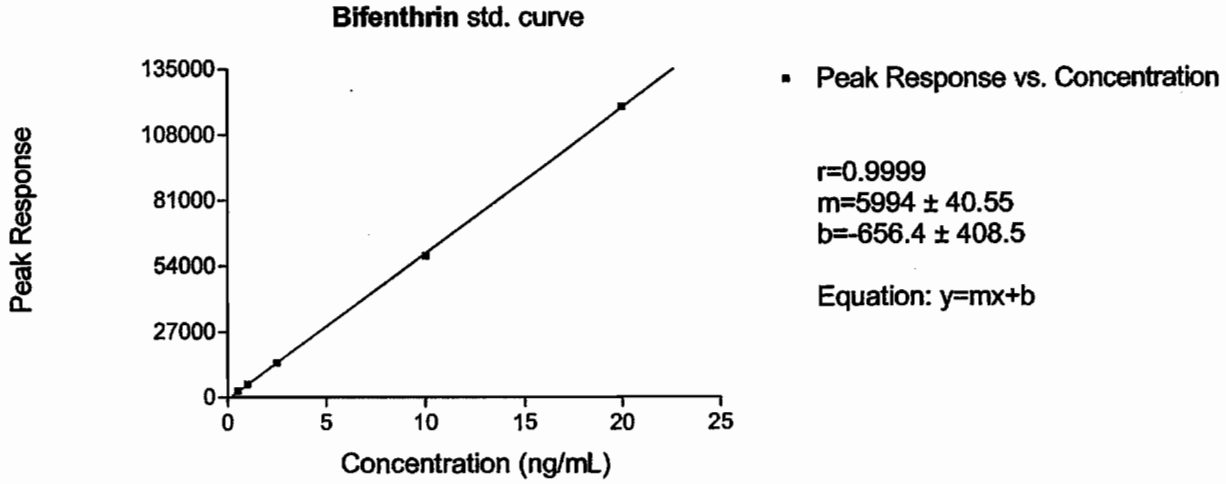
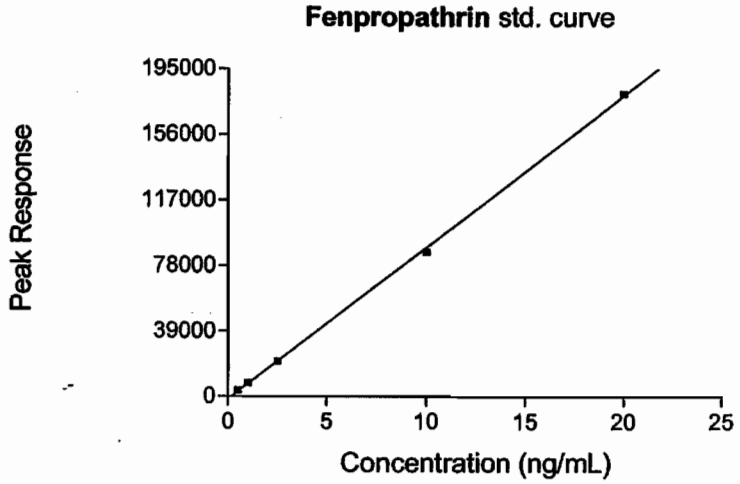


FIGURE 22. (con't) Representative Calibration Curves

Representative Fenpropathrin Calibration Curve, Set #2



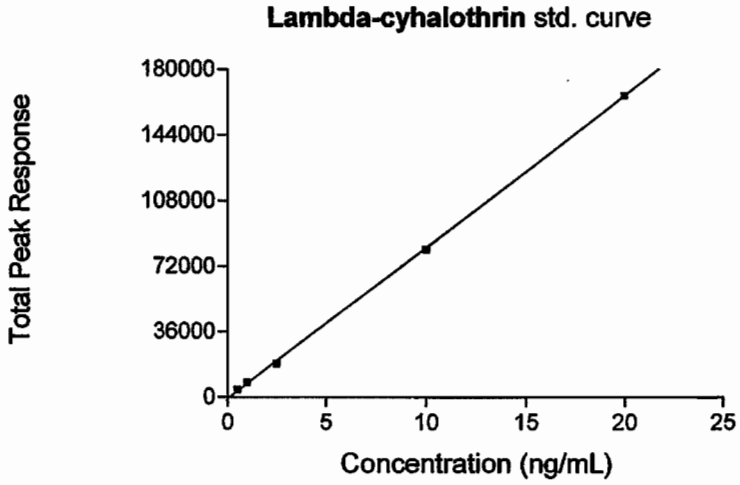
- Peak Response vs. Concentration

r=0.9998
m=9031 ± 104.0
b=-1443 ± 1048

Equation: y=mx+b

FIGURE 22. (con't) Representative Calibration Curves

Representative Lambda-cyhalothrin Calibration Curve, Set #2



- Peak Response vs. Concentration

r=0.9999
m=8309 ± 69.86
b=-1005 ± 703.8

Equation: y=mx+b

FIGURE 22. (con't) Representative Calibration Curves

Representative Permethrin Calibration Curve, Set #2

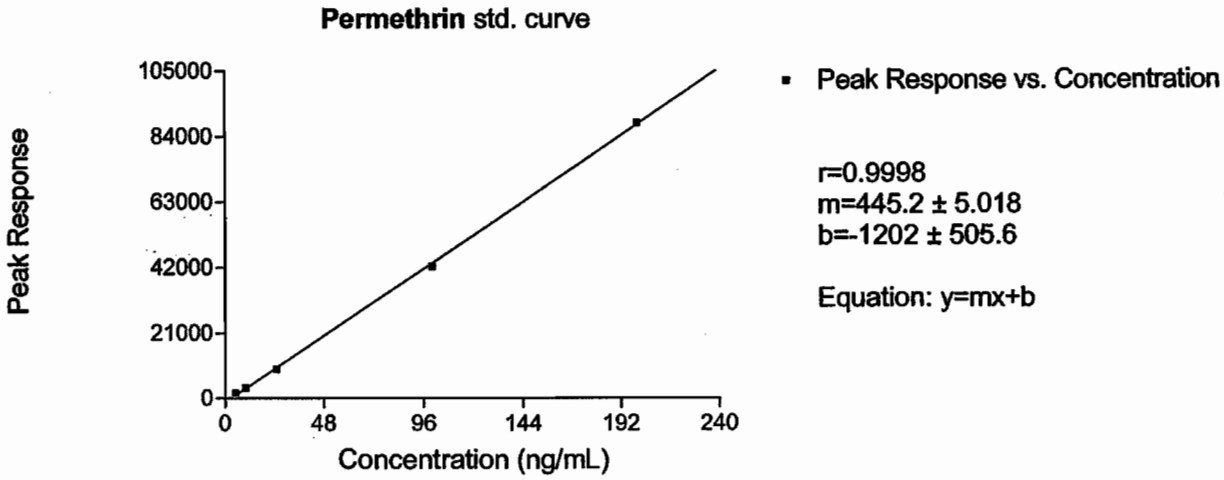


FIGURE 22. (con't) Representative Calibration Curves

Representative Cyfluthrin Calibration Curve, Set #2

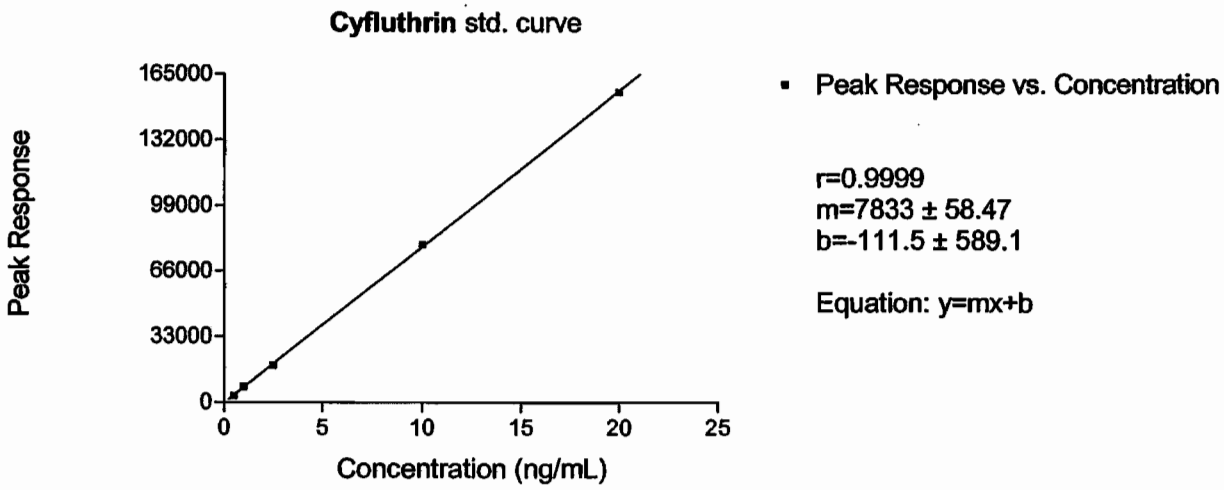


FIGURE 22. (con't) Representative Calibration Curves

Representative Cypermethrin Calibration Curve, Set #2

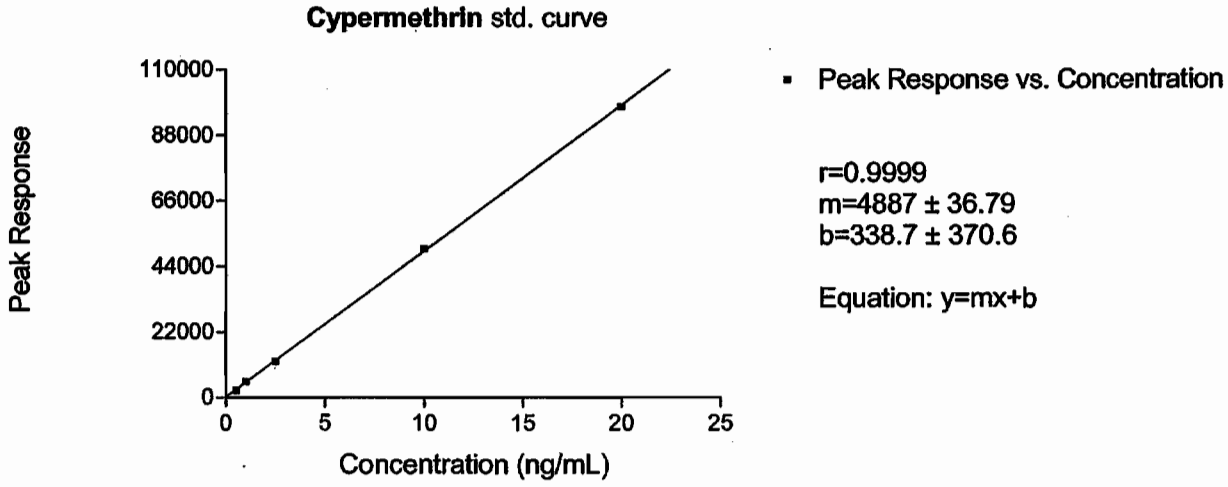
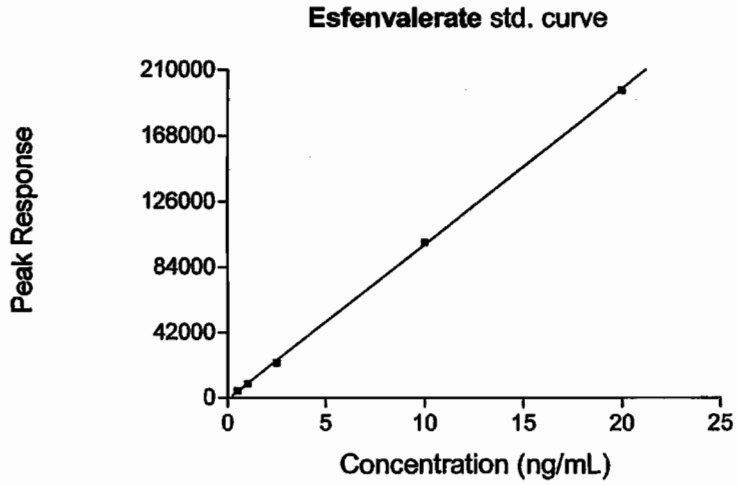


FIGURE 22. (con't) Representative Calibration Curves

Representative Esfenvalerate Calibration Curve, Set #2



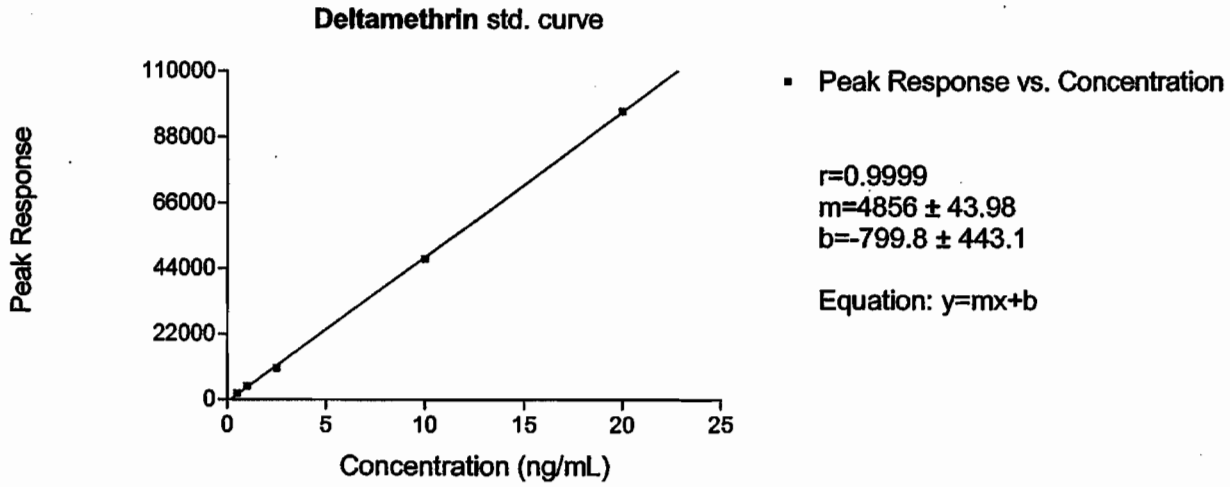
- Peak Response vs. Concentration

r=0.9999
m=9930 ± 80.03
b=-929.0 ± 806.3

Equation: y=mx+b

FIGURE 22. (con't) Representative Calibration Curves

Representative Deltamethrin Calibration Curve, Set #2



LABORATORY VALIDATION:

VALIDATION OF THE RESIDUE ANALYTICAL METHOD: "RESIDUE ANALYTICAL METHOD FOR THE DETERMINATION OF RESIDUES OF BIFENTHRIN, CYPERMETHRIN, CYFLUTHRIN, DELTAMETHRIN, ESFENVALERATE, FENPROPATHRIN, LAMBDA-CYHALOTHRIN AND PERMETHRIN IN SEDIMENT"

FINAL REPORT

DATA REQUIREMENT: EPA: Ecological Effects Test Guidelines
OPPTS 850.7100 Data Reporting for Environmental Chemistry Methods

AUTHOR: Richard L. Reed II

COMPLETION DATE: November 29, 2006

PERFORMING LABORATORY: Morse Laboratories, Inc.
1525 Fulton Avenue
Sacramento, CA 95825 USA

STUDY IDENTIFICATION: Protocol No.: MLI-06-02
Morse Labs Project No.: ML06-1286-PWG

SPONSOR: Pyrethroid Working Group
c/o Fred Pearson
Chair PWG Coordination Committee
Syngenta Crop Protection, Inc.
410 Swing Road
Greensboro, NC 27409 USA

Vol. 2 of 2

APPENDICES SECTION

APPENDIX 1. Analytical Method

Residue Analytical Method For The Determination Of Residues Of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin And Permethrin In Sediment

AUTHOR: N J Robinson on behalf of Pyrethroid Working Group

DATA REQUIREMENT: EPA: Ecological Effects Test Guidelines
OPPTS 850.7100 Data Reporting for Environmental Chemistry Methods

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1. Introduction and Summary

1.1 Scope

The analytical procedure described is suitable for the determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin (Figures 1-8) in sediment using an external standardisation procedure. The limit of quantitation of the method is 0.1 µg kg^{-1} for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and 1.0 µg kg^{-1} for permethrin.

Figure 1

Compound : Bifenthrin

IUPAC Name : 2-methylbiphenyl-3-ylmethyl (*Z*)-(1*RS*,3*RS*)-3-(2-chloro-3-3-3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

CAS Number : 82657-04-3

CAS Name : (2-methyl[1,1'-biphenyl]-3-(2-chloro-3-3-3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

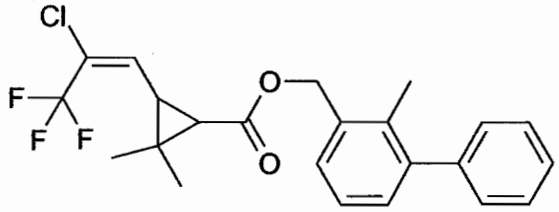


Figure 2

Compound : Cypermethrin

IUPAC Name : (*RS*)-α-cyano-3-phenoxybenxyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate

CAS Number : 52315-07-8

CAS Name : Cyano(3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate

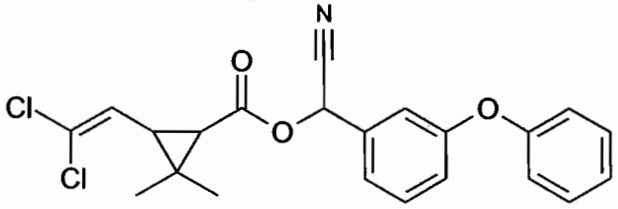


Figure 3

Compound : Cyfluthrin

IUPAC Name : *RS*)-α-cyano-4-fluoro-3-phenoxybenzyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate

CAS Number : 68359-37-5

CAS Name : Cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate

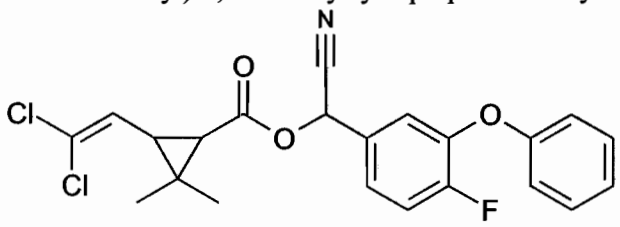


Figure 4

Compound : Deltamethrin

IUPAC Name : (*S*)-α-cyano-3-phenoxybenzyl (1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate

CAS Number : 52918-63-5

CAS Name : 1-[*R*-[1-α(*S**),3α]]-cyano(3-phenoxyphenyl)methyl 3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate

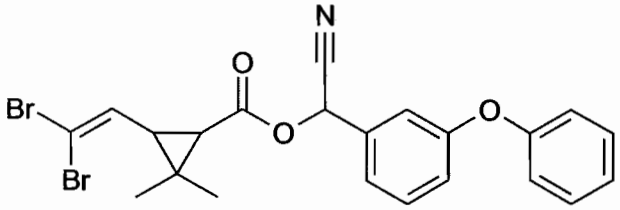


Figure 5

Compound	:	Esfenvalerate
IUPAC Name	:	(*S*)-α-cyano-3-phenoxybenzyl (*S*)-2-(4-chlorophenyl)-3-methylbutyrate
CAS Number	:	66230-04-4
CAS Name	:	[*S*-(*R**,*R**)]-cyano(3-phenoxyphenyl)methyl 4-chloro-2-(1-methylethyl)benzeneacetate

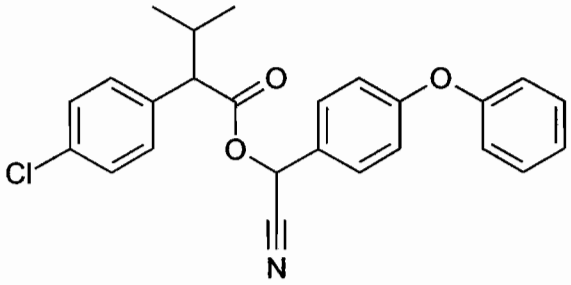


Figure 6

Compound	:	Fenpropathrin
IUPAC Name	:	(*RS*)-α-cyano-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate
CAS Number	:	64257-84-7
CAS Name	:	Cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate

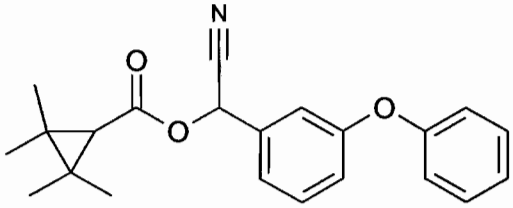


Figure 7

Compound : Lambda-cyhalothrin

IUPAC Name : A reaction product containing equal quantities of (*S*)-α-cyano-3-phenoxybenzyl (*Z*)-(1*R*,3*R*)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate and (*R*)-α-cyano-3-phenoxybenzyl (*Z*)-(1*R*,3*R*)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

CAS Number : 91465-08-6

CAS Name : [1α(*S**),3α(*Z*)]-(±)-cyano(3-phenoxyphenyl)methyl 3-(2-chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethylcyclopropanecarboxylate

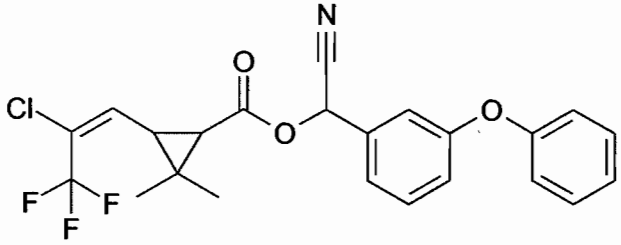


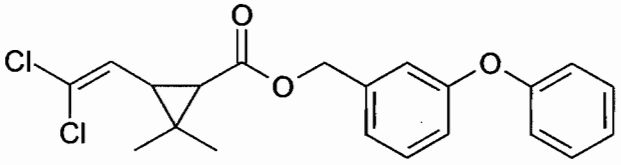
Figure 8

Compound : Permethrin

IUPAC Name : 3-phenoxybenzyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate

CAS Number : 52645-53-1

CAS Name : (3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate



1.2 Method Summary

Bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin residues are extracted from sediment by shaking with methanol/water mixture and hexane for one hour. The sample is centrifuged and an aliquot of the upper hexane layer evaporated to dryness and re-dissolved in a small volume of hexane. The hexane sample is then subjected to a silica solid phase extraction (SPE) procedure prior to residue determination by gas

chromatography with mass selective detection using negative ion chemical ionisation (GC-MS/NICI). The limit of quantitation of the method is 0.1 $\mu g\ kg^{-1}$ for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and 1.0 $\mu g\ kg^{-1}$ for permethrin.

2. Materials

The recommended equipment and reagents are described in Appendices 1 and 2. Equipment with equivalent performance specifications and reagents of comparable purity can be substituted provided that they can be shown to be suitable.

2.1 Apparatus

See Appendix 1 for a list of apparatus used during this method.

2.2 Reagents

All solvents and other reagents must be of high purity, i.e. pesticide grade solvents and analytical grade reagents. Extreme care must be taken to avoid contamination of the reagents used. See Appendix 2 for a list of reagents used in this method.

2.3 Preparation of Analytical Standards

It is recommended that the following precautions should be taken when weighing the analytical materials.

1. Ensure good ventilation.
2. Wear gloves and laboratory coat.
3. Prevent inhalation and contact with mouth.
4. Wash any contaminated area immediately.

Weigh out accurately, using a 5 figure balance, sufficient bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin analytical standards to allow dilution in acetone to give a 200 $\mu g\ mL^{-1}$ stock solutions in a volumetric flasks. Make serial dilutions of these stock solutions to give 100 $\mu g\ mL^{-1}$, 10 $\mu g\ mL^{-1}$, 1.0 $\mu g\ mL^{-1}$, 0.1 $\mu g\ mL^{-1}$ and 0.01 $\mu g\ mL^{-1}$ standards in acetone. If required, mixed standards can also be prepared to 0.01 $\mu g\ mL^{-1}$ in acetone.

When not in use, always store the standard solutions in a refrigerator at ≤7°C to prevent decomposition and/or concentration of the standard. Analytical standards should be replaced with freshly prepared standards after six months.

2.4 Safety Precautions and Hazards

The following information is included as an indication to the analyst of the nature and hazards of the reagents used in this procedure. If in any doubt, consult a monograph such as 'Hazards in the Chemical Laboratory', Edited by S G Luxon, The Chemical Society, London (Reference 1).

Reagent Hazards

	Acetone	Hexane	Methanol	Diethyl Ether
Harmful Vapour	✓	✓	✓	✓
Highly Flammable	✓	✓	✓	✓
Harmful by Skin Absorption	×	✓	×	×
OES Short Term (mg m^{-3})	3560	3600	310	1500

In all cases avoid breathing vapour. Avoid contact with eyes and skin.

2.5 Time Required for Analysis

The methodology is normally performed with a batch of 20 samples over the course of 1 day.

2.6 Work Stoppages

The analytical procedure can be stopped at various points for overnight and weekend breaks except where specified in the analytical procedure. Acceptable external standard recoveries will validate the work stoppages. Samples should be stored in sealed vessels at a temperature of ≤7°C.

3. Analytical Procedure

3.1 Sample Preparation

Sediment samples should be thoroughly mixed prior to taking an aliquot for analysis to ensure sample homogeneity.

3.2 Extraction

a) Weigh a representative amount of aquatic sediment (50 g) into a plastic screw capped centrifuge bottle (250 ml size). At least one untreated control and two control samples fortified with known amounts of bifenthrin, cypermethrin, cyfluthrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin in acetone should be analysed alongside each batch of samples to demonstrate acceptable performance of the method and allow recovery corrections to be made if desired.

Note : To avoid sample cross contamination, extraction vessels should be used only once and discarded after use.

b) Add methanol/water solution [1:1 v/v, 75 mL] and hexane (50 mL) to the sample and shake the sample using a mechanical shaker for 60 minutes.

c) Centrifuge the sample at a speed that disperses any emulsions formed on shaking e.g. 4000 rpm for five minutes.

3.3 Solid Phase Extraction

a) Transfer an aliquot of the upper hexane layer from the sediment extract equivalent to 10 g of sediment (10 mL) into a test tube (10 mL size). Evaporate the sample to dryness under a stream of clean, dry air in a heating block set to 40°C. Re-dissolve the sample in hexane (2 mL), with ultrasonication.

b) Place a Varian Silica Bond Elut™ solid phase extraction cartridge (500 mg, 3 mL size) on a suitable vacuum manifold. Add hexane (3 mL) and draw through under vacuum to the level of the top frit at a rate of approximately 2 mL min^{-1}, discarding the eluate.

c) Transfer the sample aliquot from section 3.3 (a) onto the cartridge and allow to percolate through under gravity or low vacuum (approx. 200 mbar). Discard the eluate.

d) Add hexane (1 mL) to the cartridge and allow to percolate through under gravity or low vacuum. Discard the eluate.

e) Place a collection tube (10 mL) in the manifold rack. Elute the analytes from the column with hexane/diethyl ether solution [9:1 v/v, 6 mL] drawing through under gravity or low vacuum at a rate of approximately 2 mL min^{-1}, collecting the eluate in the tube.

f) Evaporate the column eluate to dryness under a stream of clean, dry air in a heating block with the temperature set to 40 °C. Re-dissolve the sample in acetone

+ 0.1% (v/v) peanut oil solution (1 mL) with ultrasonication. Transfer the sample to an autosampler vial ready for final determination by GC-MS/NICI.

Note : The 0.1% peanut oil in acetone solution is used to minimise the effect of matrix related GC-MSD response enhancement and to minimise possible peak tailing due to adsorption.

3.4 Preparation of GC-MSD Calibration Standards

GC-MSD calibration standards should be prepared in acetone + 0.1% (v/v) peanut oil solution. The 0.1% peanut oil in acetone solution is used to minimise the effect of matrix related GC-MSD response enhancement and to minimise possible peak tailing due to adsorption.

For example, to prepare a 1.0 ng mL^{-1} calibration standard, transfer 1 mL of a 0.01 µg mL^{-1} bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin mixed standard in acetone to a volumetric flask (10 mL) and dilute to 10 mL volume with acetone.

4. Final Determination by GC-MSD

The following instrumentation and conditions have been found to be suitable for this analysis in this laboratory. Other instrumentation can also be used, however optimisation may be required to achieve the desired separation and sensitivity. The operating manuals for the instruments should always be consulted to ensure safe and optimum use.

Instrument Conditions

GC system	:	Agilent 6890 with split/splitless injector
MSD system	:	Agilent 5973 with negative ion chemical ionization
Injection temperature	:	275°C
Injection liner	:	4 mm i.d. double gooseneck splitless liner (unpacked)
Column	:	Varian CPSil 8 30 m × 0.25 mm, 0.25 µm film thickness (5% diphenyl, 95% dimethylpolysiloxane)
Column flow rate	:	0.9 mL min^{-1} constant flow
Injection mode	:	Pulsed splitless, 30 psi for 1 min, purge flow to split vent 50 psi @2 min
Injection volume	:	2 µL
Column temperature program	:	80°C for 1 min then program at 40°C/min to 180°C, hold for 0 min then program at 5 °C/min to 305 °C, hold for 0 min.

MS transfer line temp	:	280°C
Ionization mode	:	Negative ion CI
Reagent gas	:	Methane
System calibration	:	Autotune
System resolution	:	Low
Acquisition type	:	Selected Ion Monitoring (SIM)

	Target Ion	Qualifier 1	Qualifier 2
Bifenthrin	*m/z* = 386	*m/z* = 387	*m/z* = 241
Fenpropathrin	*m/z* = 141	-	-
Lambda-cyhalothrin	*m/z* = 205	*m/z* = 241	*m/z* = 243
Permethrin	*m/z* = 207	*m/z* = 209	-
Cyfluthrin	*m/z* = 207	*m/z* = 209	*m/z* = 171
Cypermethrin	*m/z* = 207	*m/z* = 209	*m/z* = 171
Esfenvalerate	*m/z* = 211	*m/z* = 213	-
Deltamethrin	*m/z* = 297	*m/z* = 81	*m/z* = 296

Retention Times

Compound Name	Peak	Retention Time (min)
Bifenthrin	1	18.1
Fenpropathrin	1	18.5
Lambda-cyhalothrin	1 2	19.6 19.9
Permethrin	1 2	21.5 21.8
Cyfluthrin	1 2 3 4	22.5 22.7 22.8 22.9
Cypermethrin	1 2 3 4	23.2 23.4 23.5 23.6
Esfenvalerate	1 2	24.9 25.3
Deltamethrin	1 2	25.9 26.3

Typical chromatograms are shown in Appendix 4.

5. Calculation of Results

Residues may be calculated using an external standardisation procedure. For lambda-cyhalothrin, permethrin, cyfluthrin, cypermethrin, esfenvalerate and deltamethrin, isomer peaks are resolved in the GC chromatogram. The peak areas of the individual isomer peaks for each pyrethroid should be summed together and a total residue value calculated for each compound.

5.1 Using Mean Bracketed Single-Point Calibration

a) Make repeated injections of a standard containing bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin at an appropriate concentration into the GC-MSD operated under conditions as described in Section 4. When a consistent response is obtained, measure the peak areas obtained for each peak.

b) Make an injection of each sample solution and measure the peak areas of the peaks corresponding to the each analyte.

c) Re-inject the standard solution after a maximum of four injections of sample solutions.

d) Calculate the residues in the sample, expressed as $\mu g\ kg^{-1}$, using a mean standard response from each of the injections bracketing the sample as follows.

$$\text{Residue} = \frac{\text{PK area (SA)}}{\text{PK area (STD)}} \times \frac{\text{Standard Conc.}}{\text{Sample Conc.}}$$

PK area (SA) = Peak response for sample
PK area (STD) = Average peak response for bracketing standards
Standard Conc. = Concentration of standard ($ng\ mL^{-1}$)
Sample Conc. = Sample concentration ($g\ mL^{-1}$)

If residues need to be corrected for average percentage recovery, then the equation below should be used.

$$\text{Corrected Residue} = \frac{\text{Residue} \times 100}{\text{Average percentage Recovery}} (\mu g\ kg^{-1})$$

When the average percentage recovery is greater than 100%, the sample residue values should not be corrected.

5.2 Using Multi-Point Calibration

Bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin residues may be calculated in $\mu g\ kg^{-1}$ for each sample as follows.

a) Prepare standard solutions containing bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin over a concentration range appropriate to the expected residues in the samples (for example, 50% LOQ to 10x LOQ). An appropriate number of different concentrations within this range should be prepared (at least four). Injections of these standard solutions should be interspersed throughout the analysis, after a maximum of four injections of sample solutions.

b) Make an injection of each standard and sample solution into the GC-MSD operated under conditions as described in Section 4 and measure the peak areas of the peaks corresponding to each analyte.

c) Generate calibration curve parameters using an appropriate regression package.

d) The following equation can be rearranged and used to calculate residues as follows:

$$y = mx + c$$

Where y is the instrument response value, x is the standard concentration, m is the gradient of the line of best fit ("X-variable 1" in MS Excel) and c is the intercept value. An example of this equation generated using the experimental values of m and c should be included in the raw data, as should the "R-Square" value for the regression.

Re-arrangement for x gives

$$x = \frac{y - c}{m}$$

e) Alternatively (depending on the regression analysis software available) a quadratic equation may be used to fit the data. In this case the following general equation should be re-arranged and used to calculate residues:

$$y = a + bx + cx^2$$

Where y is the instrument response value, x is the standard concentration and a, b, c are constants.

g) Calculate the residues in the sample, expressed as $\mu g\ kg^{-1}$, as follows

$$\text{Residue}\ (\mu g\ kg^{-1}) = \frac{\text{Analyte found}\ (ng\ mL^{-1})}{\text{Sample conc.}\ (g\ mL^{-1})}$$

Where analyte found ($ng\ mL^{-1}$) is calculated from the standard calibration curve and sample conc. is the final sample concentration in $g\ mL^{-1}$.

If residues need to be corrected for average percentage recovery, then the equation below should be used.

$$\text{Corrected Residue} = \frac{\text{Residue} \times 100}{\text{Average percentage Recovery}}\ (\mu g\ kg^{-1})$$

When the average percentage recovery is greater than 100%, the sample residue values should not be corrected.

6. Control and Recovery Experiments

Control experiments should be completed as Section 3 for each set of samples analysed to verify that samples are free from bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin contamination. A minimum of one control should be analysed with each batch of samples.

At least two recovery experiments (untreated samples accurately fortified with a known amount of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin prior to extraction) should also be completed alongside each batch of samples. Provided the recovery values are acceptable they may be used to correct any residues found. The recovery levels should be appropriate to the residue levels expected.

Recovery data is generally considered acceptable when the mean values are between 70% and 120% and with a relative standard deviation of $\leq$20%.

7. Interference

7.1 Matrix

Due to the high selectivity of the detection technique, no interference arising from the sediment matrix has been observed.

7.2 Reagent and Solvent Interference

Using high purity solvents and reagents no interference has been found, however it is recommended that each batch of reagents or solvent is checked for contamination prior to use.

7.3 Labware Interference

The method uses disposable labware which minimises the possibility of cross contamination. All labware should be discarded after use and not re-used.

8. Method Validation

8.1 Recoveries

Method validation has been carried out on the procedures described in Section 3. (Reference 2) A summary of the method validation data is shown in Appendix 3.

8.2 Limit of Quantification and Limit of Detection

8.2.1 Limit of Quantification (LOQ)

The limit of quantification of the method is defined as the lowest analyte concentration in a sample at which the methodology has been validated and a mean recovery of 70-110% with a relative standard deviation of ≤ 20% has been obtained.

Generally, for accurate quantification, the response for an analyte peak should be no lower than four times the mean amplitude of the background noise in an untreated sample at the corresponding retention time.

The limit of quantification has been set at 0.1 $\mu g\ kg^{-1}$ for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and 1.0 $\mu g\ kg^{-1}$ for permethrin.

8.2.2 Limit of Detection (LOD)

The limit of detection of the method is defined as the lowest analyte concentration detectable above the mean amplitude of the background noise in an untreated sample at the corresponding retention time. An estimate of the LOD can be taken as three times background noise. Note that the LOD may vary between runs and from instrument to instrument. The limit of detection for this procedure is estimated at 0.001 $\mu g\ L^{-1}$ for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and 0.2 $\mu g\ kg^{-1}$ for permethrin.

8.3 Detector Linearity

For accurate quantification of residue concentrations, analyses should be carried out within the linear range of detector responses. Detector linearity graphs are given in Appendix 5.

In these laboratories the linearity of the GC-MSD detector response for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin and lambda-cyhalothrin was tested in the range from 1 pg to 40 pg injected on column (equivalent to 0.5 ng mL^{-1} to 20 ng mL^{-1} standards when using a 2 µL injection volume) and were found to be linear. Permethrin linearity was tested in the range from 5 pg to 400 pg injected on column (equivalent to 5 ng mL^{-1} to 200 ng mL^{-1} standards when using a 2 µL injection volume) and was also found to be linear. If residues beyond the tested concentration ranges are expected, dilute the extract appropriately to bring it within the tested linear range prior to quantification.

9. Limitations

The method has been tested on representative sediment types. It can reasonably be assumed that the method can be applied for other sediment types not tested in this method provided successful recovery tests at the relevant levels validate the suitability of the method.

10. Conclusions

The method described is suitable for the analysis of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin residues in aquatic sediment. Only commercially available laboratory equipment and reagents are required. The analysis of a batch of 20 samples can be completed by one person in 1 day (8 working hour period). Untreated and fortified samples should be analysed with each set of samples to demonstrate absence of any interference and adequate recovery, if possible. The limit of quantification of the method is 0.1 $\mu g\ kg^{-1}$ for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and 1.0 $\mu g\ kg^{-1}$ for permethrin.

11. References

1. Luxon S G (1992): Hazards in the Chemical Laboratory 5th Edition. The Royal Society of Chemistry. Thomas Graham House, The Science Park, Cambridge CB4 4WF, UK. ISBN 0-85186-229-2.

2. Validation Report.

Appendix 1 : Apparatus

General laboratory glassware e.g. volumetric flasks, available from Fisher Scientific, Liberty Lane, Hampton, NH 03842, USA.

250 ml screw capped polypropylene centrifuge bottles, available from Fisher Scientific, Liberty Lane, Hampton, NH 03842, USA.

10 mL disposable glass test tubes, available from Fisher Scientific, Liberty Lane, Hampton, NH 03842, USA

Varian Bond Elut™ Silica solid phase extraction columns 500 mg, 3 mL size, available from Varian Inc. 24021 Frampton Avenue, Harbor City, CA 90710, USA.

SPE sample processing station, available from Varian Inc. 24021 Frampton Avenue, Harbor City, CA 90710, USA.

Gas chromatograph fitted with a mass selective detector e.g. Agilent 6890 GC fitted with a 5973 series mass selective detector, available from Agilent Technologies, 395 Page Mill Road, Palo Alto, CA 94304 USA.

CP SIL-8 CB Low bleed MS capillary column 30 m × 0.25 mm id, 0.25 µm film thickness, available from Varian Inc. 24021 Frampton Avenue, Harbor City, CA 90710, USA.

Double gooseneck injection liner for Agilent splitless injectors (4 mm i.d.), available from Restek Corporation, 110 Benner Circle, Bellefonte, PA 168230-8812, USA.

Crimp cap autosampler vials and caps available from Fisher Scientific, Liberty Lane, Hampton, NH 03842, USA.

Appendix 2 : Reagents

All solvents and other reagents must be of high purity, e.g. glass distilled/HPLC grade solvents and analytical grade reagents. Particular care must be taken to avoid contamination of the reagents used.

Hexane, methanol, acetone and diethyl ether, pesticide grade, available from B & J Brand Solvents, from Scientific Products Division of Baxter Healthcare Corporation, USA (Tel: 312-689-8410).

Peanut oil, cooking grade, available from Planters Company, East Hanover, NJ 07936.

Bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin analytical standards, available from Dr. Ehrenstorfer GmbH, Bgm.-Schlosser-Str. 6 A, 86199 Augsburg, Germany.

Appendix 3 : Method Validation Data

Table 1 : Sediment Samples Used For Validation

Sample Identification	Sediment Type	Total Organic Carbon (%)
BUCGR	Fresh Water	1.31
Paradise Cove	Estuarine	0.86

Table 2 : Pyrethroid Recovery Data Obtained During Method Validation

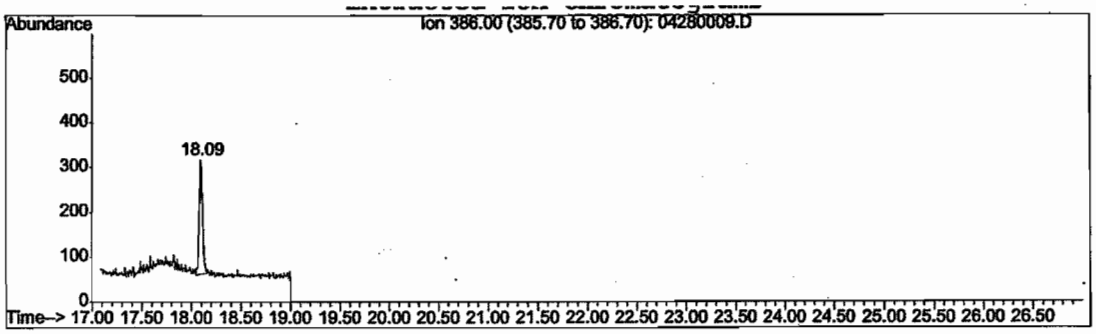
Summary of Method Validation Recoveries					
Matrix	**Analyte**	**Spike level (µg/kg)**	**Sample size (n)**	**Net Recoveries[a] (%)**	**Overall Mean ± std dev (%)**
Sediment, Fresh Water	Bifenthrin	0.1	5	103, 106, 107, 107, 109	107 ± 4.5
		1.0	5	100, 111, 109, 102, 115	
	Cypermethrin	0.1	5	107, 102, 105, 104, 110	112 ± 9.4
		1.0	5	111, 130, 120, 105, 123	
	Cyfluthrin	0.1	5	107, 105, 107, 101, 108	111 ± 8.1
		1.0	5	111, 127, 119, 108, 120	
	Deltamethrin	0.1	5	89, 78, 84, 86, 102	98 ± 13
		1.0	5	104, 116, 111, 99, 109	
	Esfenvalerate	0.1	5	76, 77, 82, 73, 84	95 ± 19
		1.0	5	105, 122, 117, 99, 115	
	Fenpropathrin	0.1	5	102, 94, 107, 108, 107	108 ± 6.8
		1.0	5	110, 115, 116, 108, 116	
	Lambda-Cyhalothrin	0.1	5	84, 83, 96, 93, 108	103 ± 13
		1.0	5	106, 121, 113, 104, 118	
	Permethrin	1.0	5	97, 99, 94, 100, 108	104 ± 10
		10	5	97, 124, 107, 93, 117	
Sediment, Estuarine	Bifenthrin	0.1	5	91, 94, 90, 91, 87	95 ± 5.7
		1.0	5	93, 98, 106, 101, 95	
	Cypermethrin	0.1	5	98, 114, 108, 96, 97	106 ± 6.9
		1.0	5	102, 110, 114, 111, 105	
	Cyfluthrin	0.1	5	89, 108, 92, 107, 98	102 ± 7.8
		1.0	5	98, 107, 113, 108, 102	
	Deltamethrin	0.1	5	87, 78, 89, 84, 86	85 ± 7.7
		1.0	5	74, 81, 96, 97, 77	
	Esfenvalerate	0.1	5	97, 111, 112, 109, 118	107 ± 7.2
		1.0	5	95, 109, 112, 107, 102	
	Fenpropathrin	0.1	5	95, 109, 106, 111, 105	105 ± 5.9
		1.0	5	98, 105, 114, 107, 100	
	Lambda-Cyhalothrin	0.1	5	90, 106, 102, 104, 105	102 ± 5.9
		1.0	5	97, 105, 111, 106, 99	
	Permethrin	1.0	5	98, 111, 102, 107, 111	105 ± 5.5
		10	5	96, 106, 111, 108, 101	

[a]Calculated following correction by the average residue concentration measured in the corresponding unfortified control samples

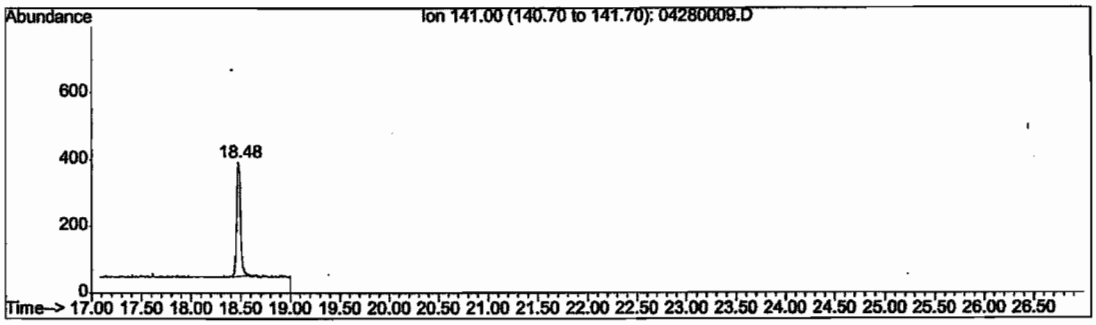
Appendix 4 : Representative Chromatograms

Figure 9 : Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



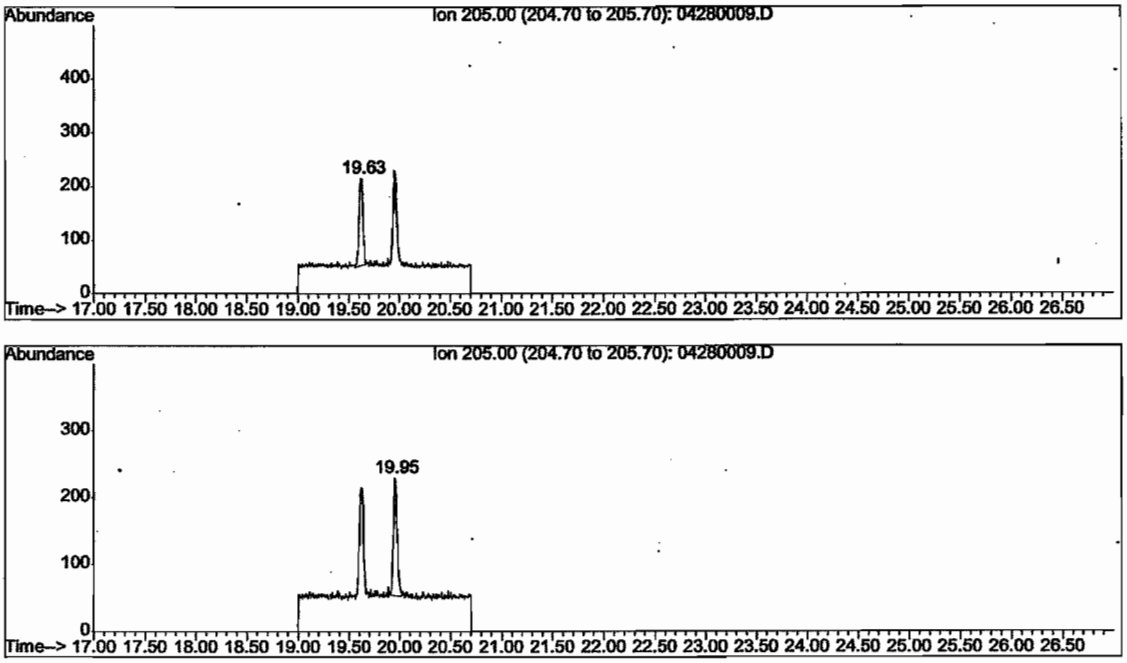
Bifenthrin
1.0 ng/mL
Peak Response (area): 6326



Fenpropathrin
1.0 ng/mL
Peak Response (area): 9369

Figure 9. (con't) **Bracketing Standards**

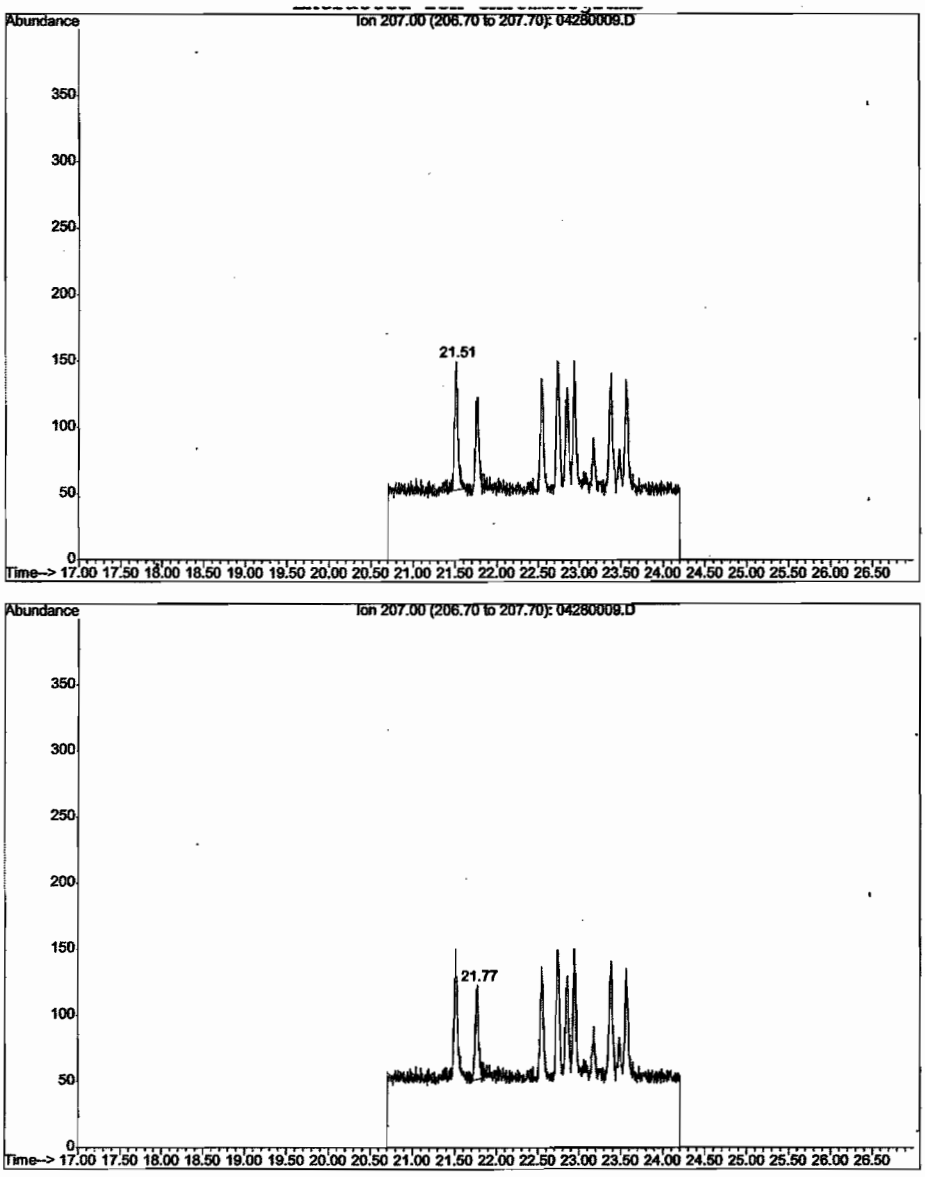
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 8917

Figure 9. (con't) **Bracketing Standards**

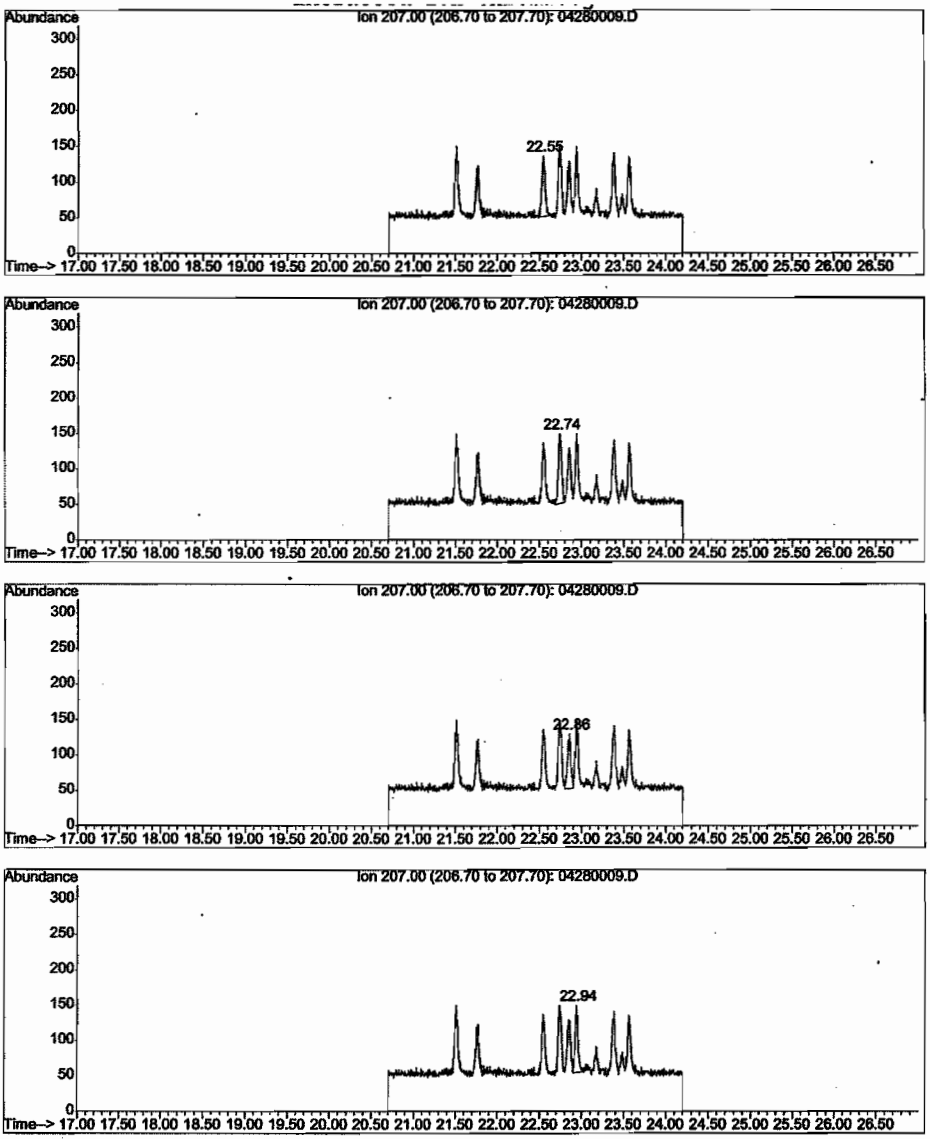
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Permethrin
10 ng/mL
Peak Response (area): 4519

Figure 9. (con't) **Bracketing Standards**

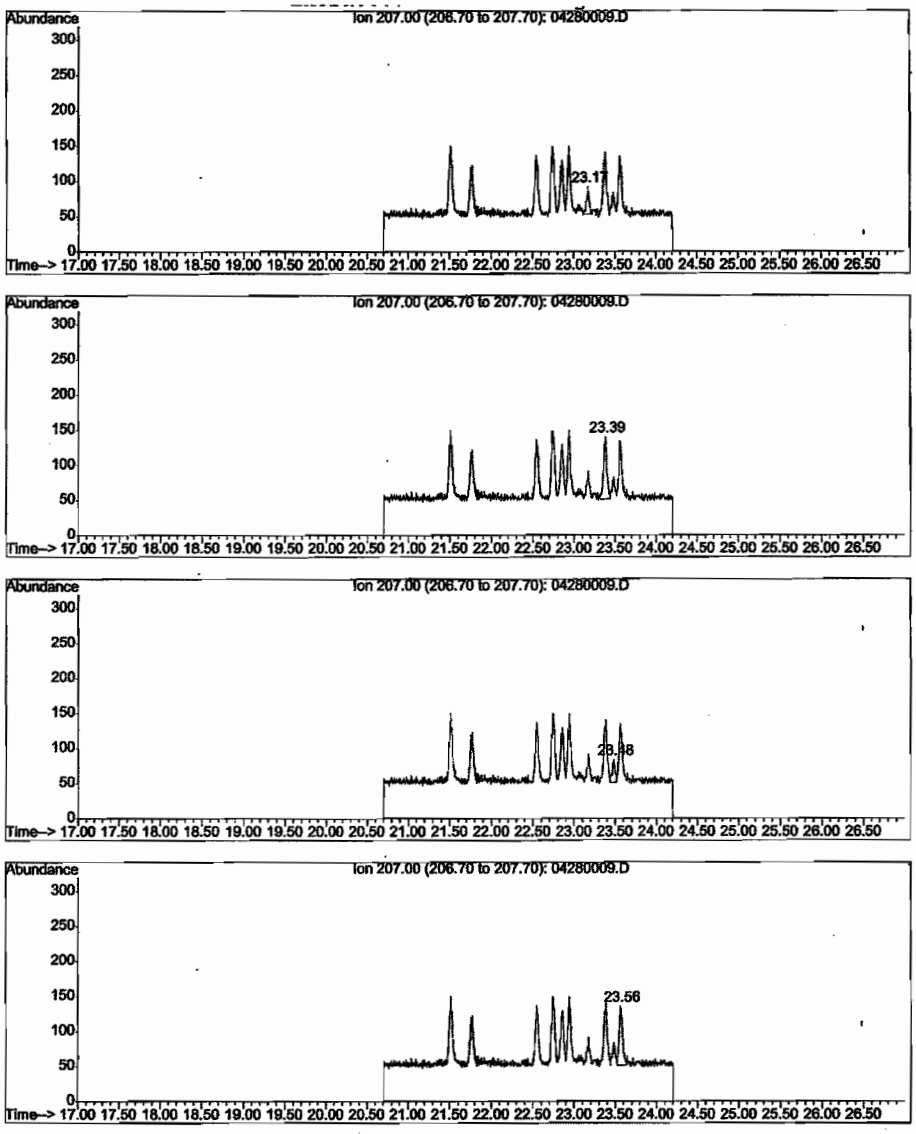
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cyfluthrin
1.0 ng/mL
Peak Response (area): 9501

Figure 9. (con't) **Bracketing Standards**

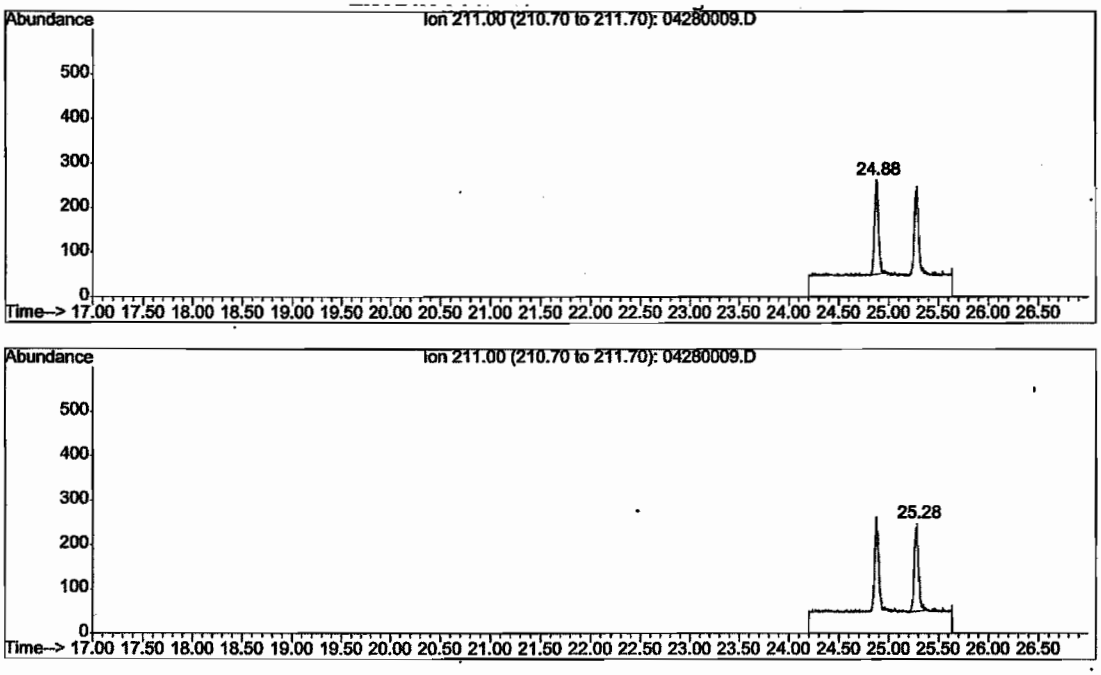
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cypermethrin
1.0 ng/mL
Peak Response (area): 6464

Figure 9. (con't) **Bracketing Standards**

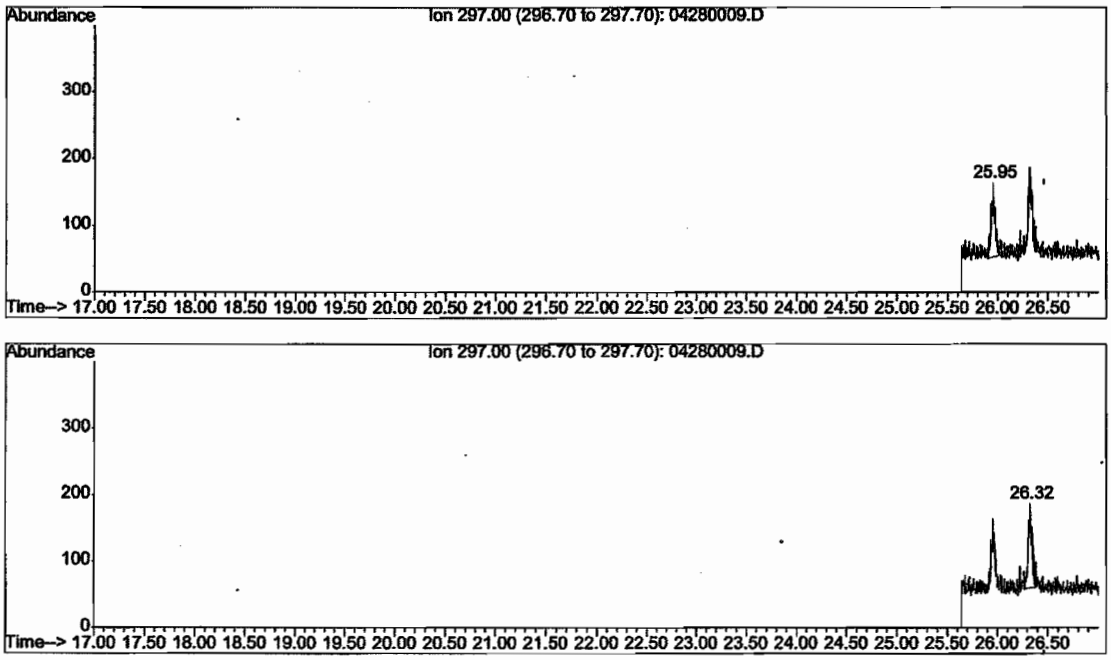
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Esfenvalerate
1.0 ng/mL
Peak Response (area): 11274

Figure 9. (con't) **Bracketing Standards**

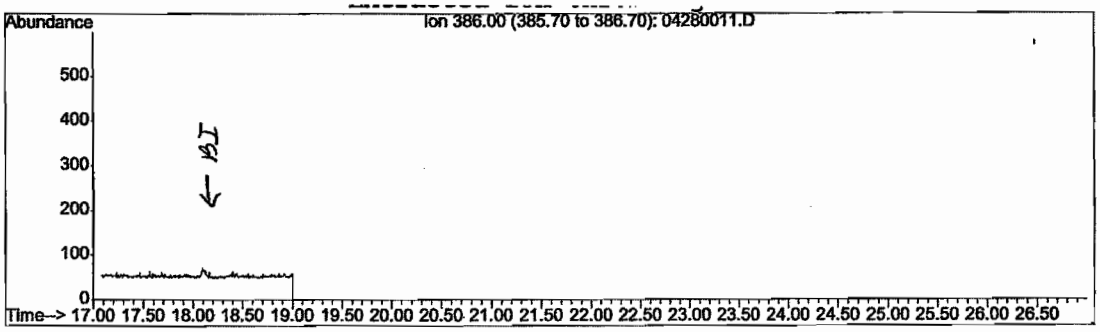
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin, Set #2



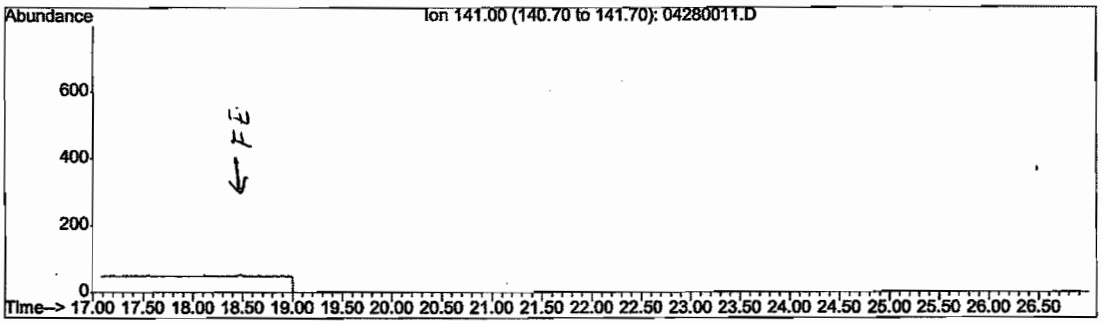
Deltamethrin
1.0 ng/mL
Peak Response (area): 5996

Figure 10. Fresh Water Sediment, Untreated Control

Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL.



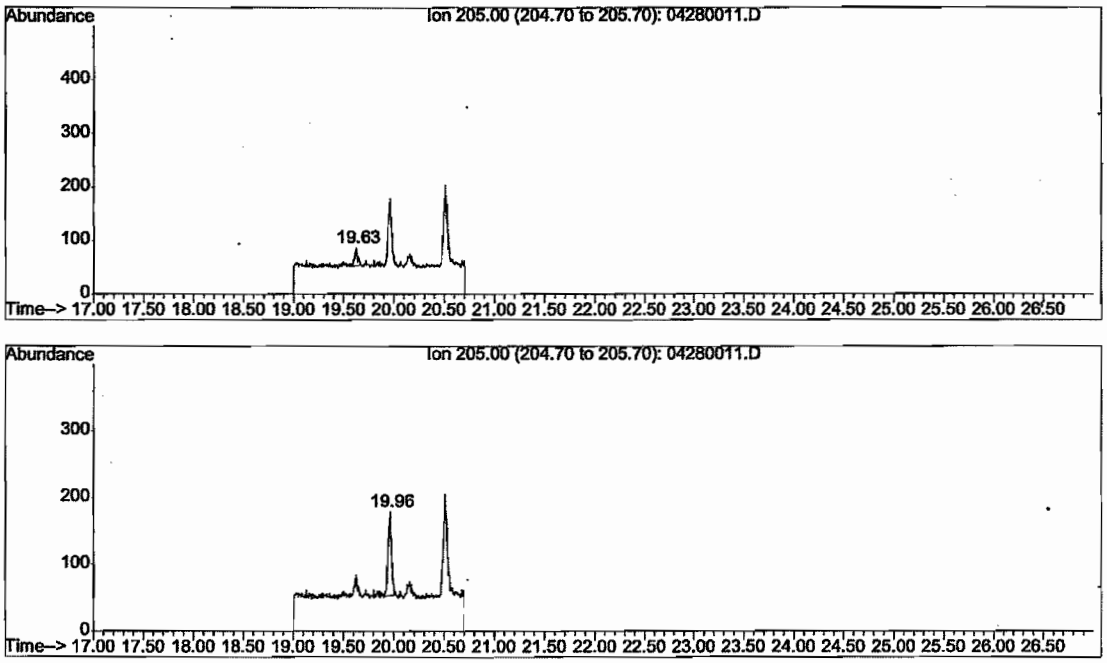
Bifenthrin
Peak Response (area): 0
Bifenthrin Reported: None Detected



Fenpropathrin
Peak Response (area): 0
Fenpropathrin Reported: None Detected

Figure 10. **Fresh Water Sediment, Untreated Control**

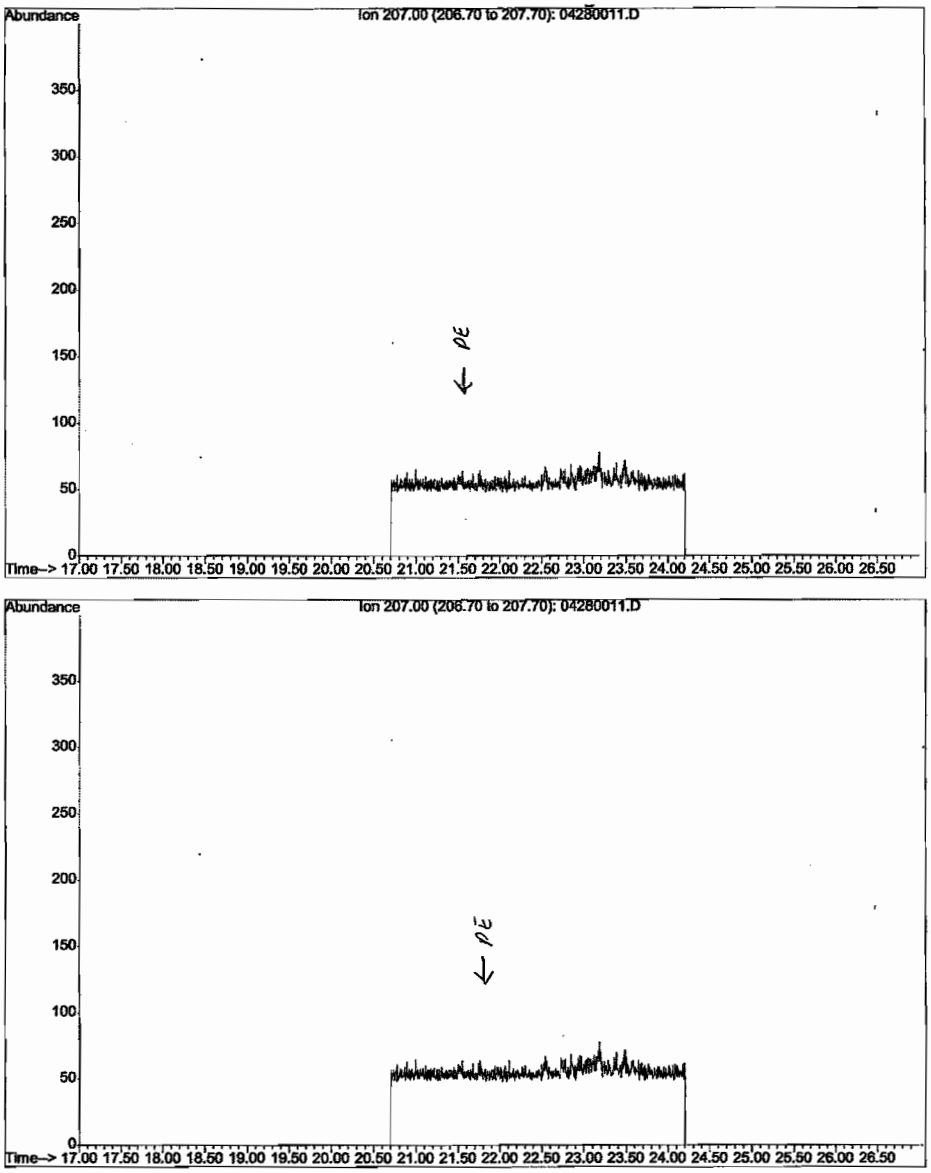
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL.



Lambda-cyhalothrin
Peak Response (area): 3484
Lambda-cyhalothrin Reported: <0.1 µg/kg

Figure 10. **Fresh Water Sediment, Untreated Control**

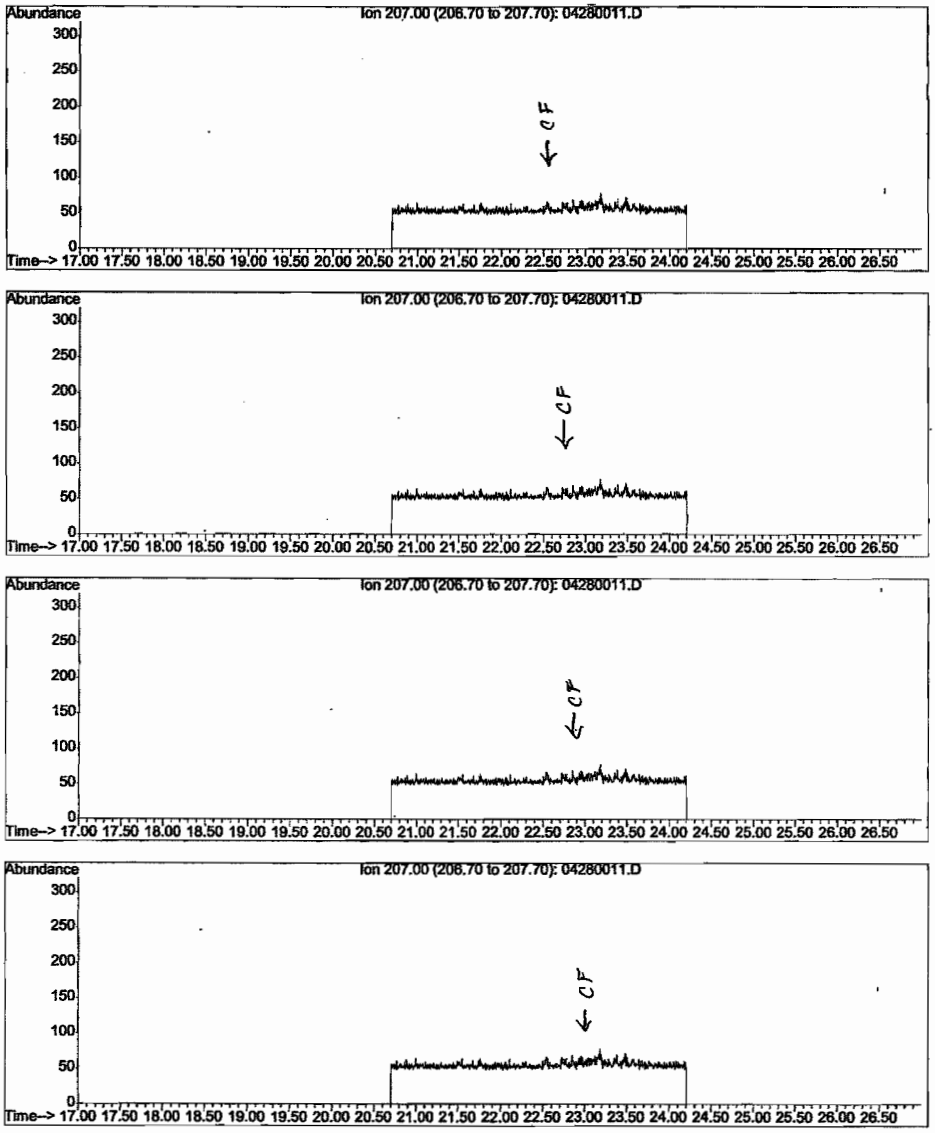
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL.



Permethrin
Peak Response (area): 0
Permethrin Reported: None Detected

Figure 10. **Fresh Water Sediment, Untreated Control**

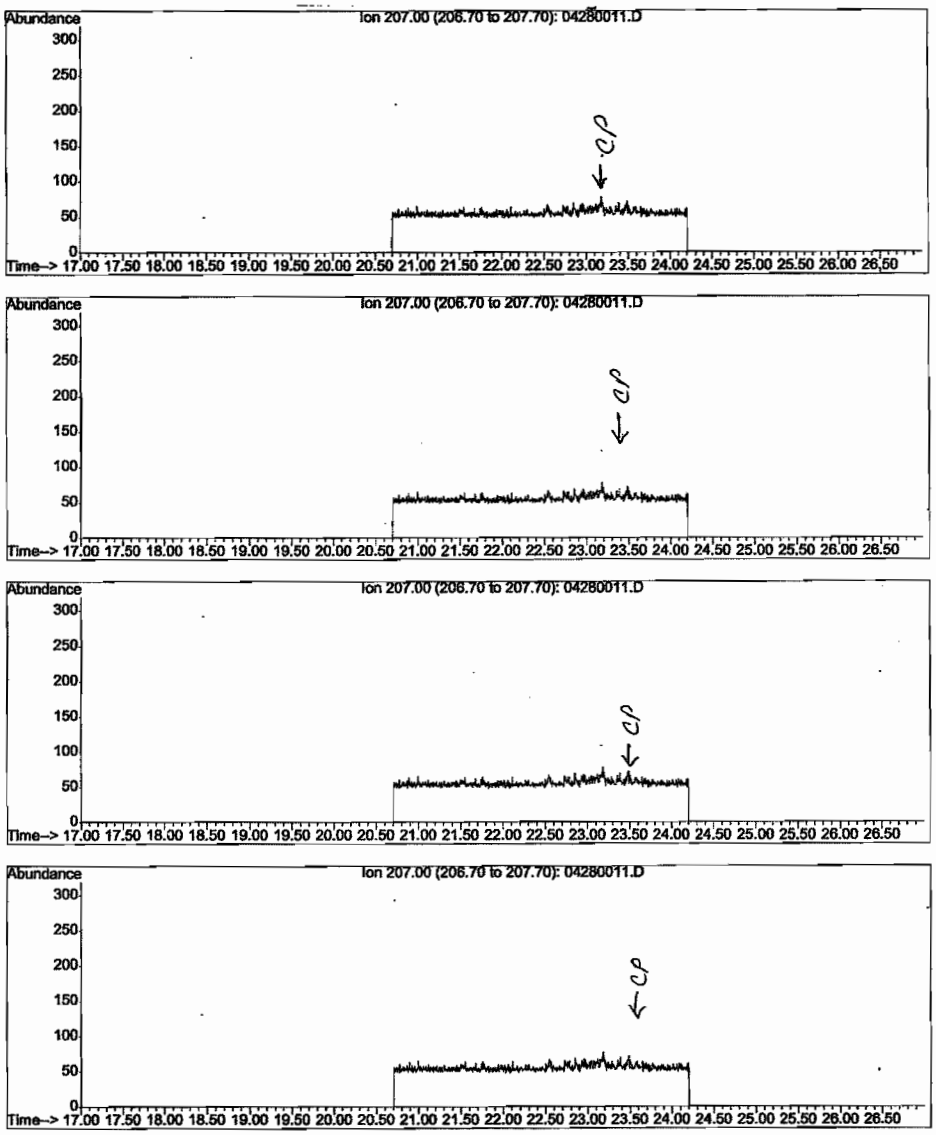
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL.



Cyfluthrin
Peak Response (area): 0
Cyfluthrin Reported: None Detected

Figure 10. **Fresh Water Sediment, Untreated Control**

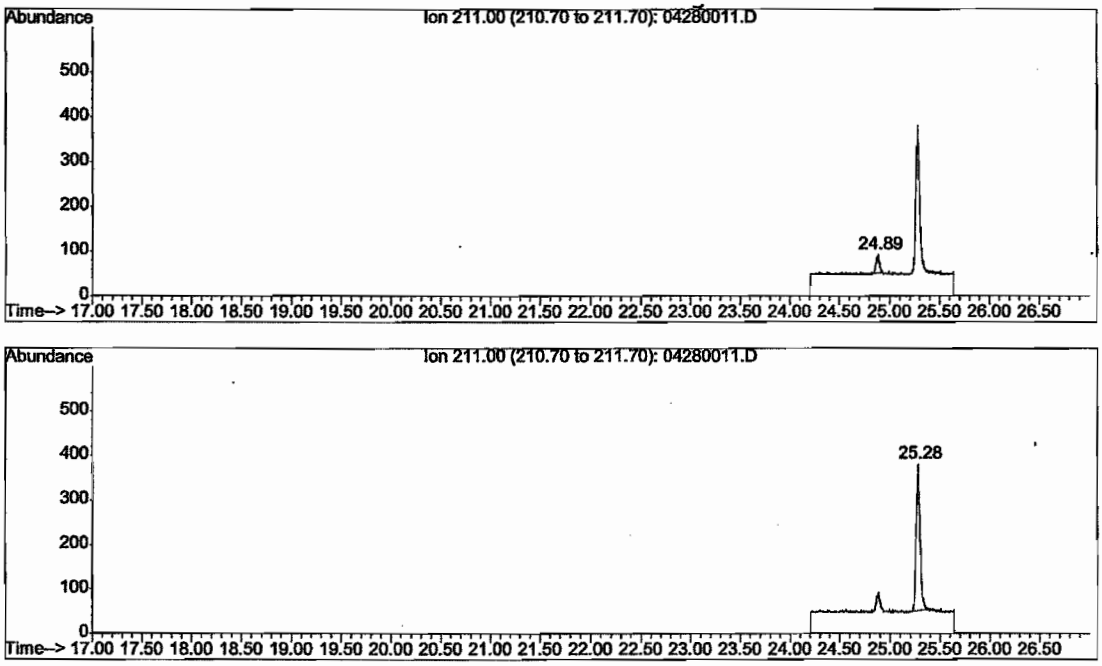
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL.



Cypermethrin
Peak Response (area): 0
Cypermethrin Reported: None Detected

Figure 10. **Fresh Water Sediment, Untreated Control**

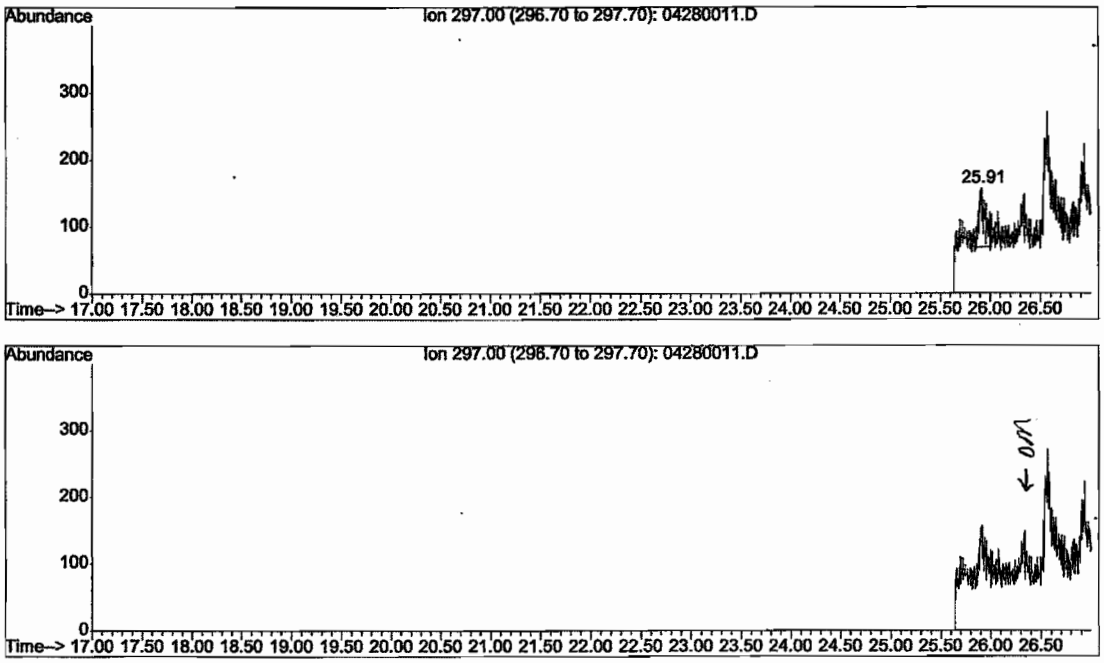
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



Esfenvalerate
Peak Response (area): 9601
Esfenvalerate Reported: <0.1 µg/kg

Figure 10. **Fresh Water Sediment, Untreated Control**

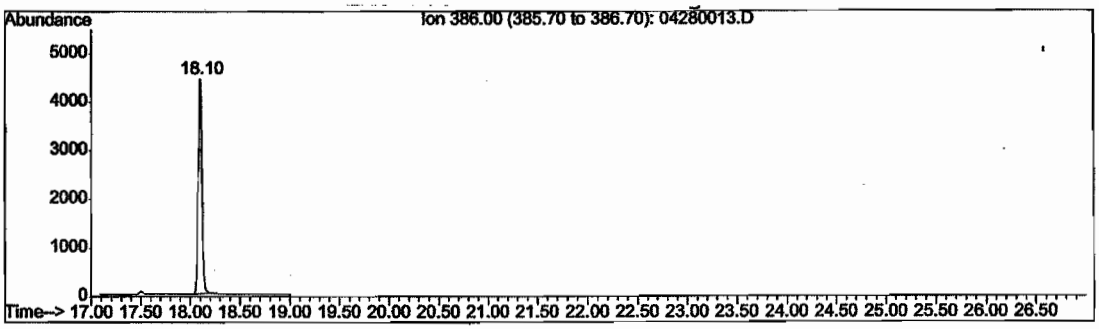
Untreated Control, Fresh Water Sediment, BUCGR, Control 3, 10.0 g/mL, Set #2



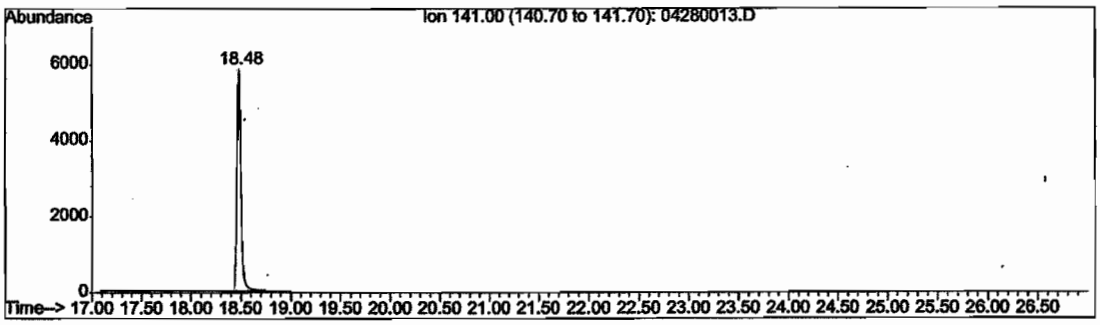
Deltamethrin
Peak Response (area): 3496
Deltamethrin Reported: <0.1 µg/kg

Figure 11. Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



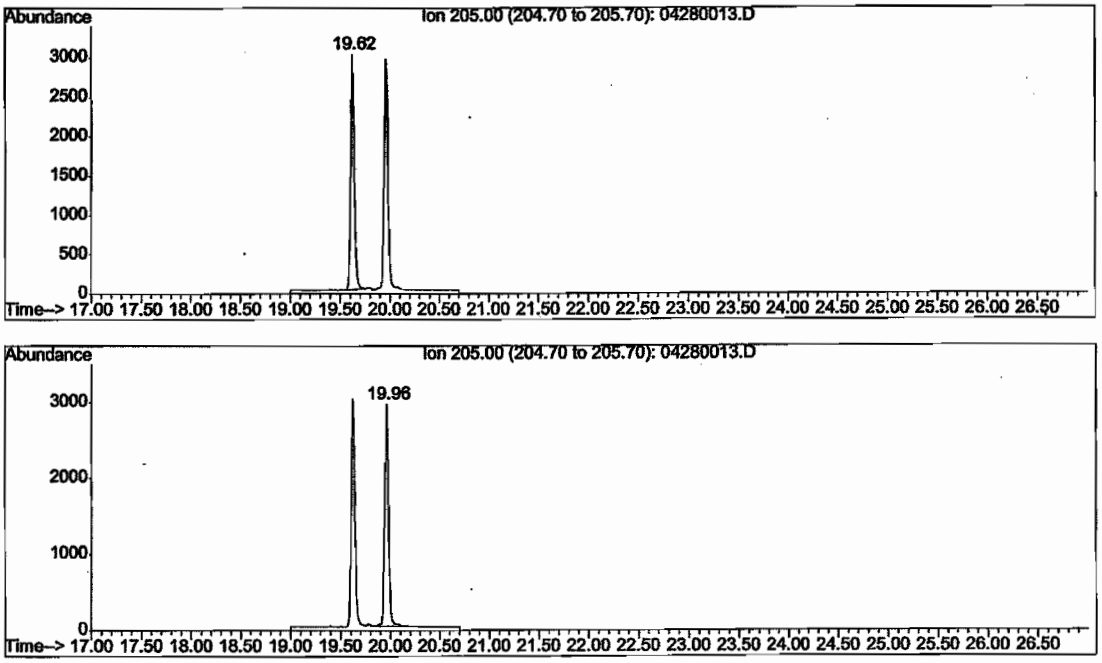
Bifenthrin
20 ng/mL
Peak Response (area): 115423



Fenpropathrin
20 ng/mL
Peak Response (area): 170037

Figure 11. (con't) Bracketing Standards

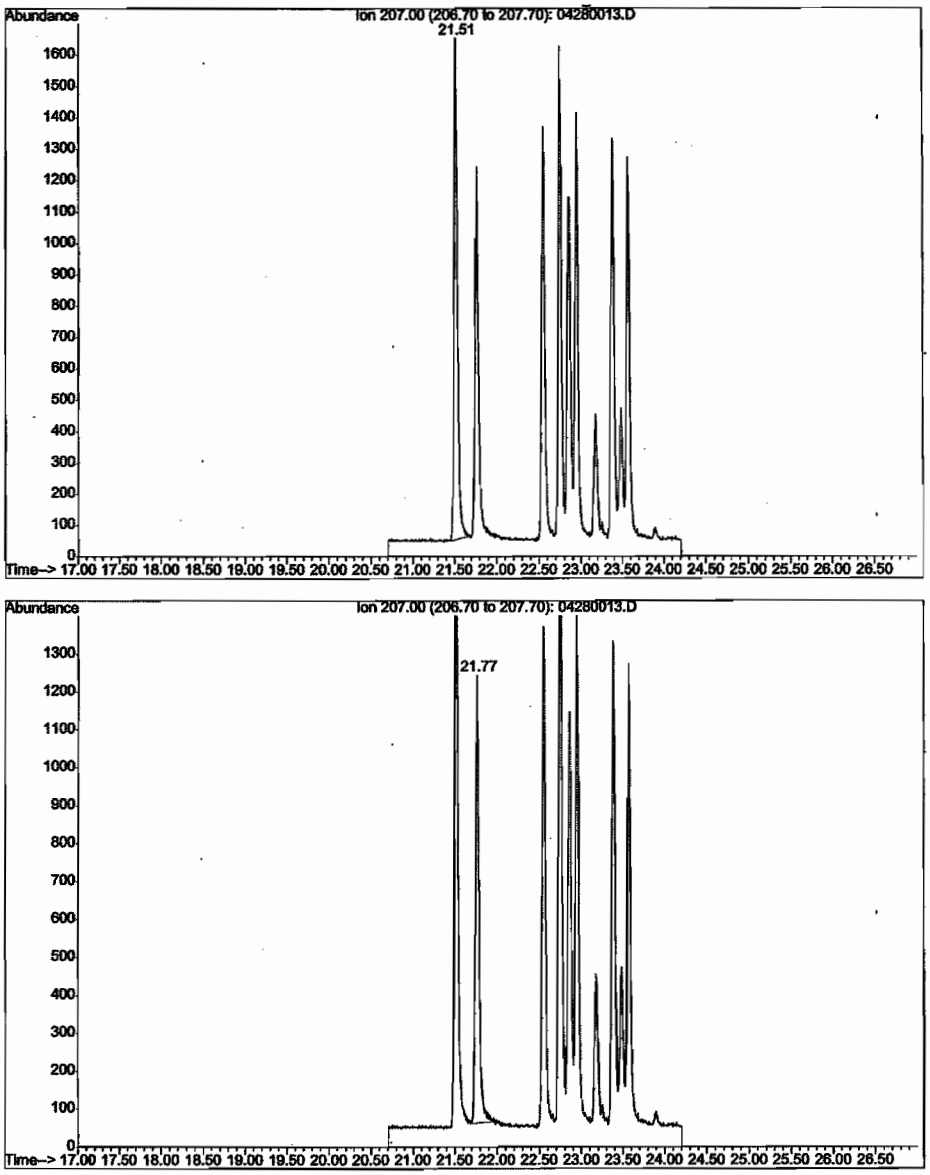
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 153812

Figure 11. (con't) Bracketing Standards

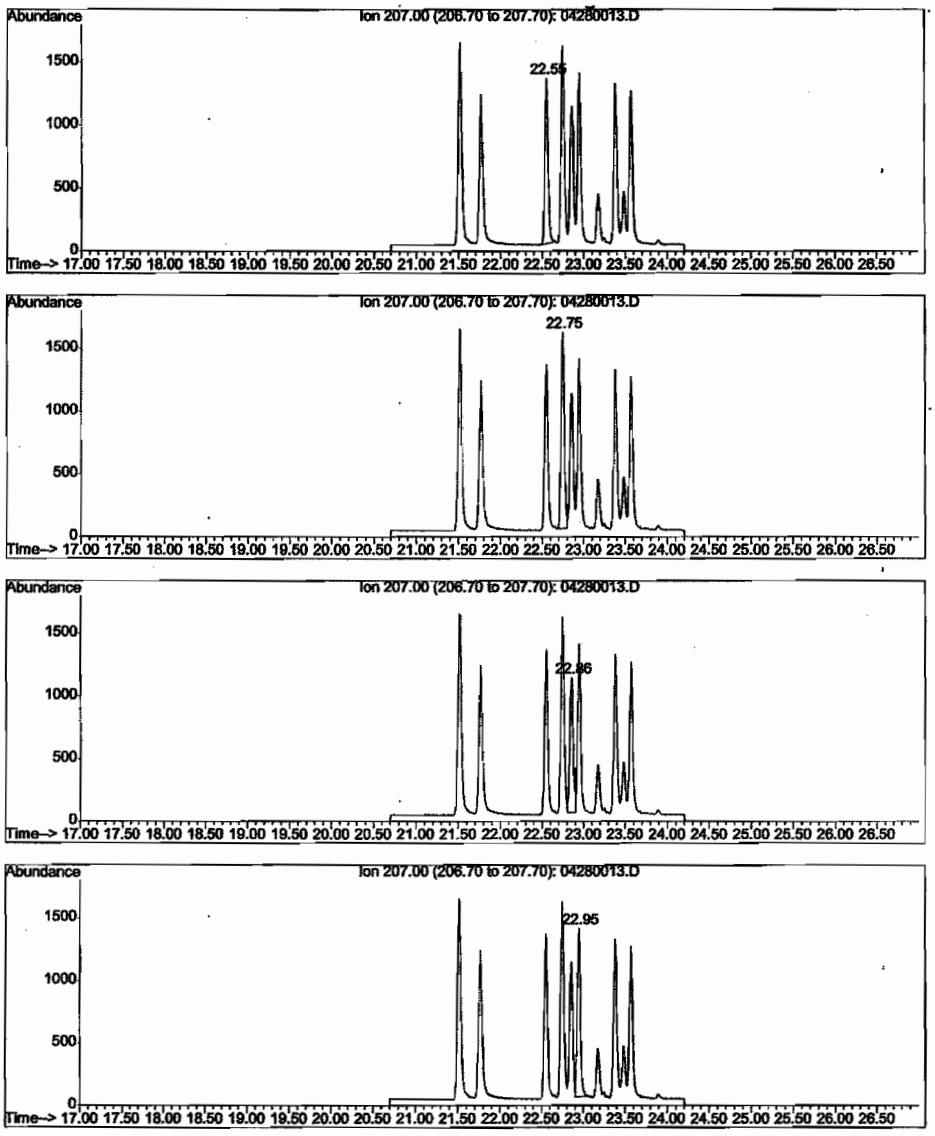
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Permethrin
200 ng/mL
Peak Response (area): 80094

Figure 11. (con't) Bracketing Standards

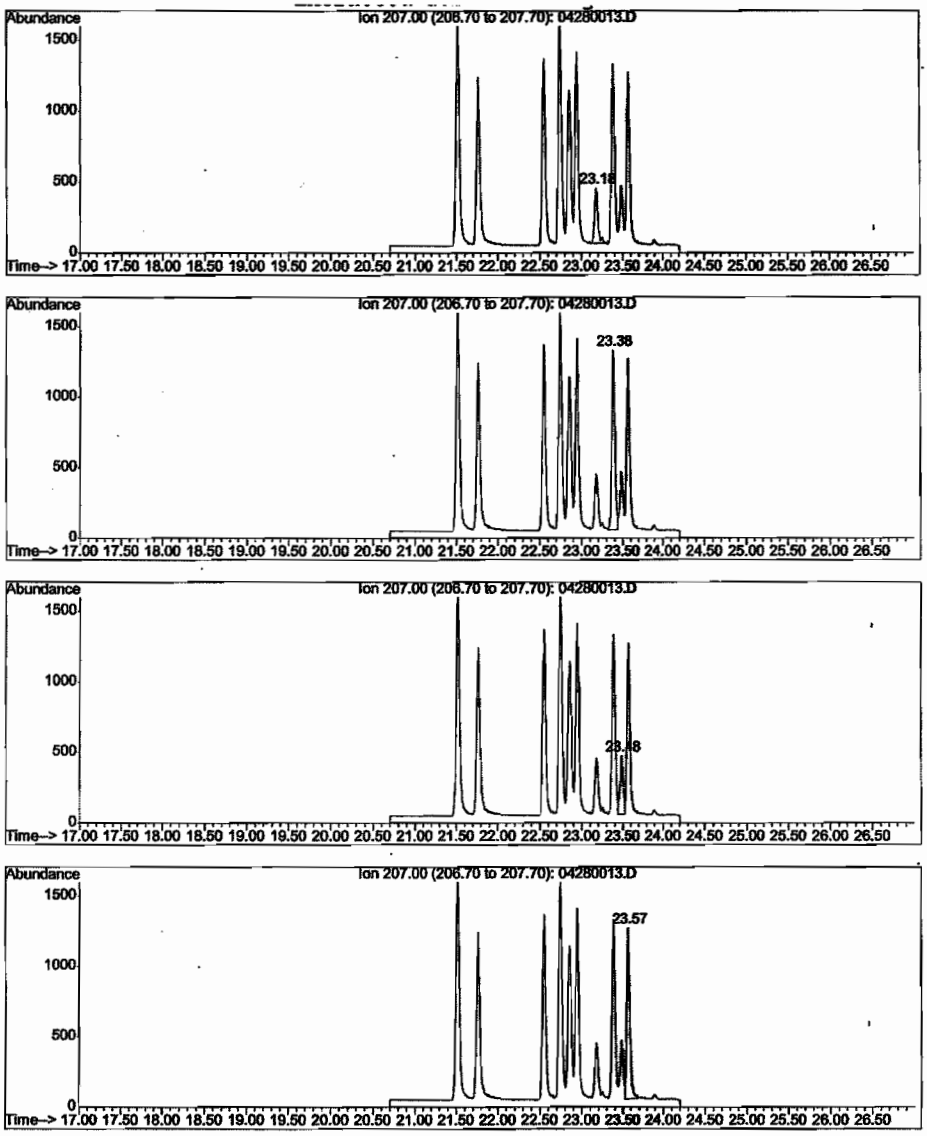
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cyfluthrin
20 ng/mL
Peak Response (area): 146068

Figure 11. (con't) Bracketing Standards

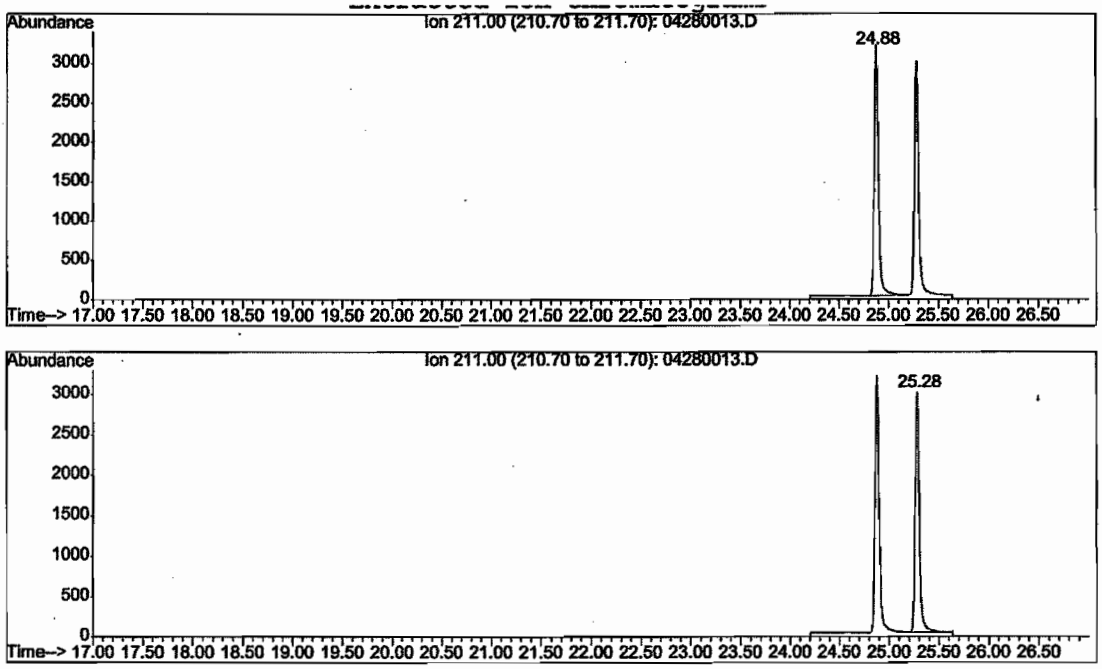
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cypermethrin
20 ng/mL
Peak Response (area): 90994

Figure 11. (con't) Bracketing Standards

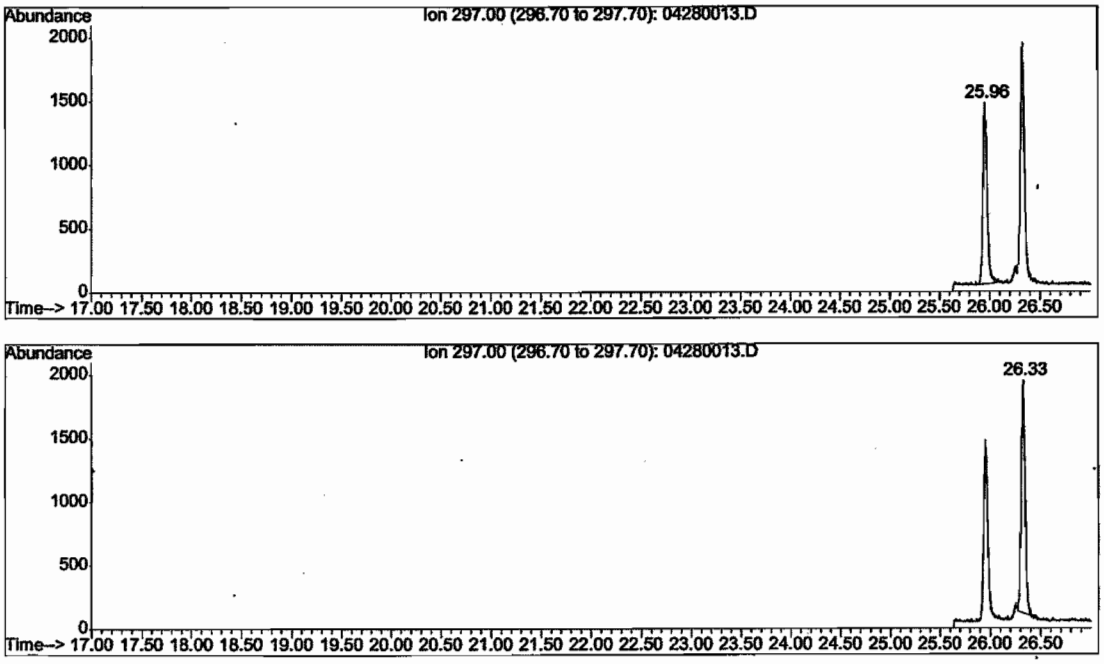
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Esfenvalerate
20 ng/mL
Peak Response (area): 185356

Figure 11. (con't) Bracketing Standards

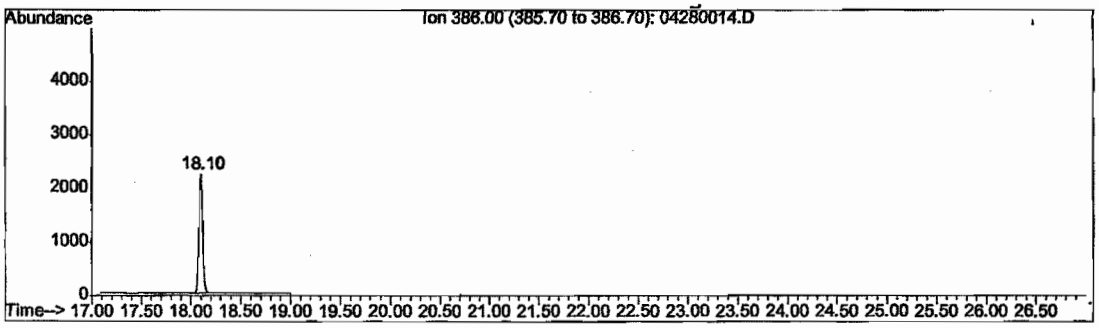
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



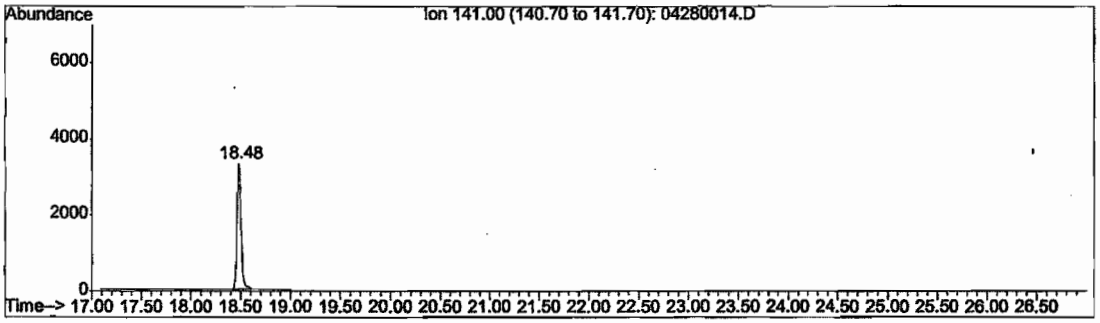
Deltamethrin
20 ng/mL
Peak Response (area): 90699

Figure 12. Fresh Water Sediment, Fortified Control (10×LOQ)

Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



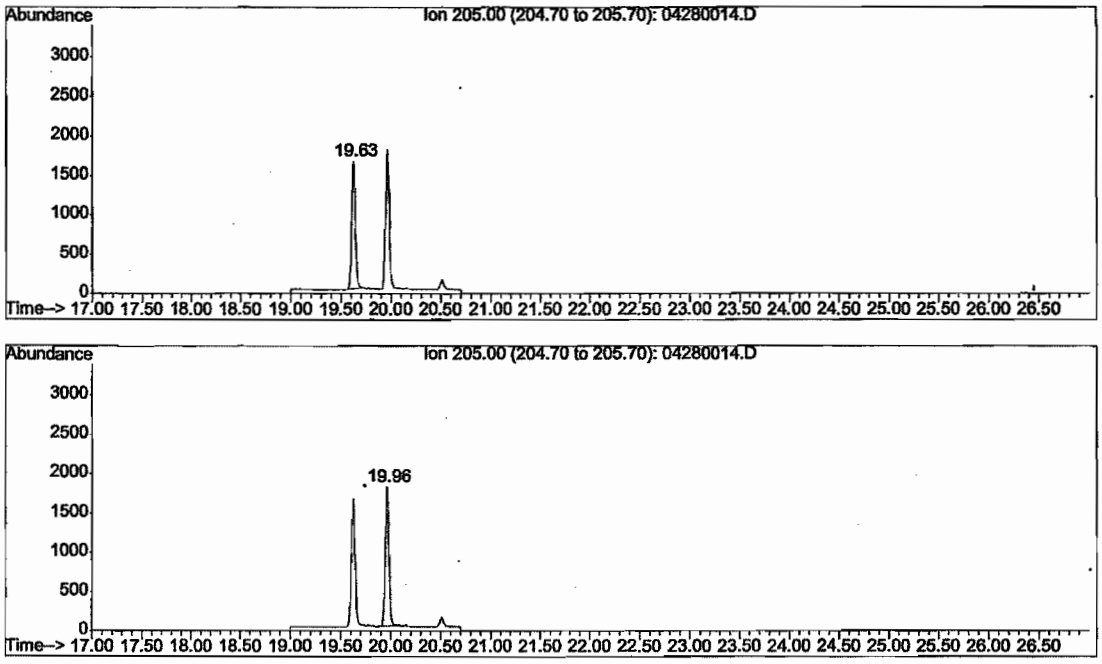
Bifenthrin
Peak Response (area): 57760
Bifenthrin Reported Recovery: 100%



Fenpropathrin
Peak Response (area): 93570
Fenpropathrin Reported Recovery: 110%

Figure 12. (con't) Fresh Water Sediment, Fortified Control (10×LOQ)

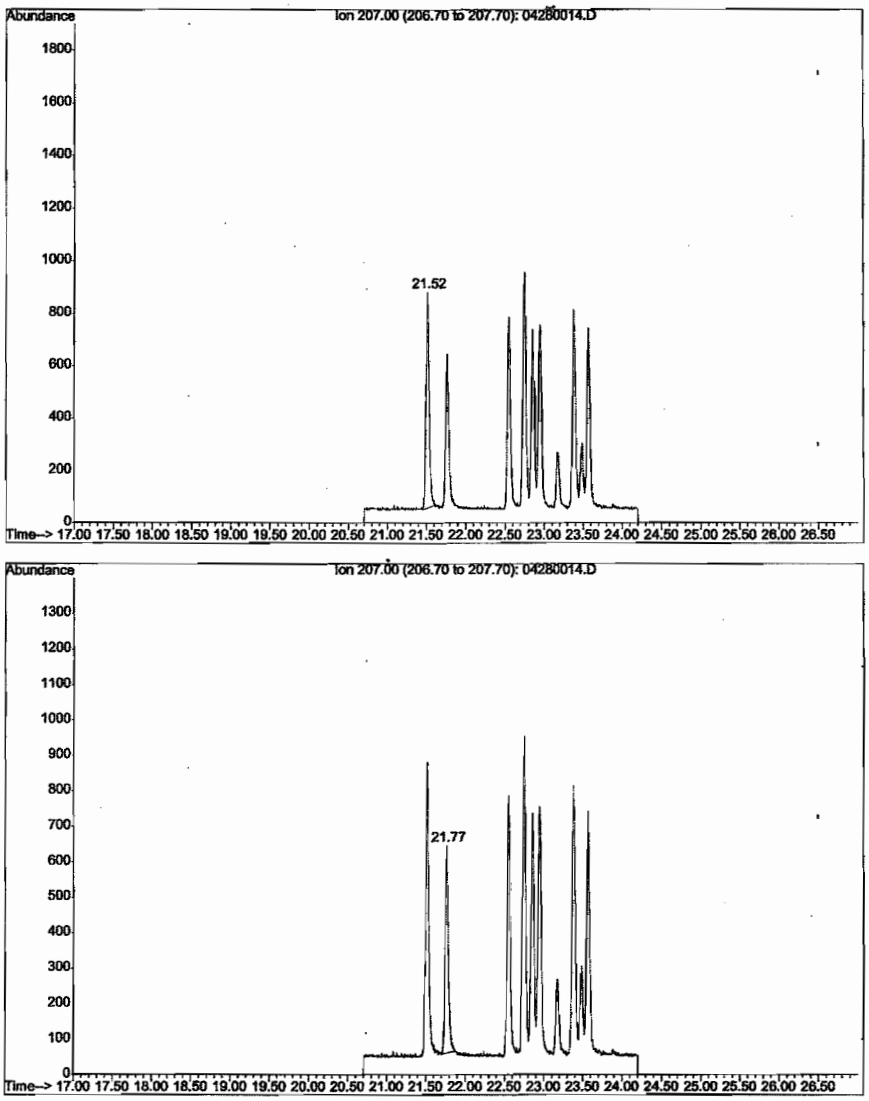
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Lambda-cyhalothrin
Peak Response (area): 85102
Lambda-cyhalothrin Reported Recovery: 106%

Figure 12. (con't) Fresh Water Sediment, Fortified Control (10×LOQ)

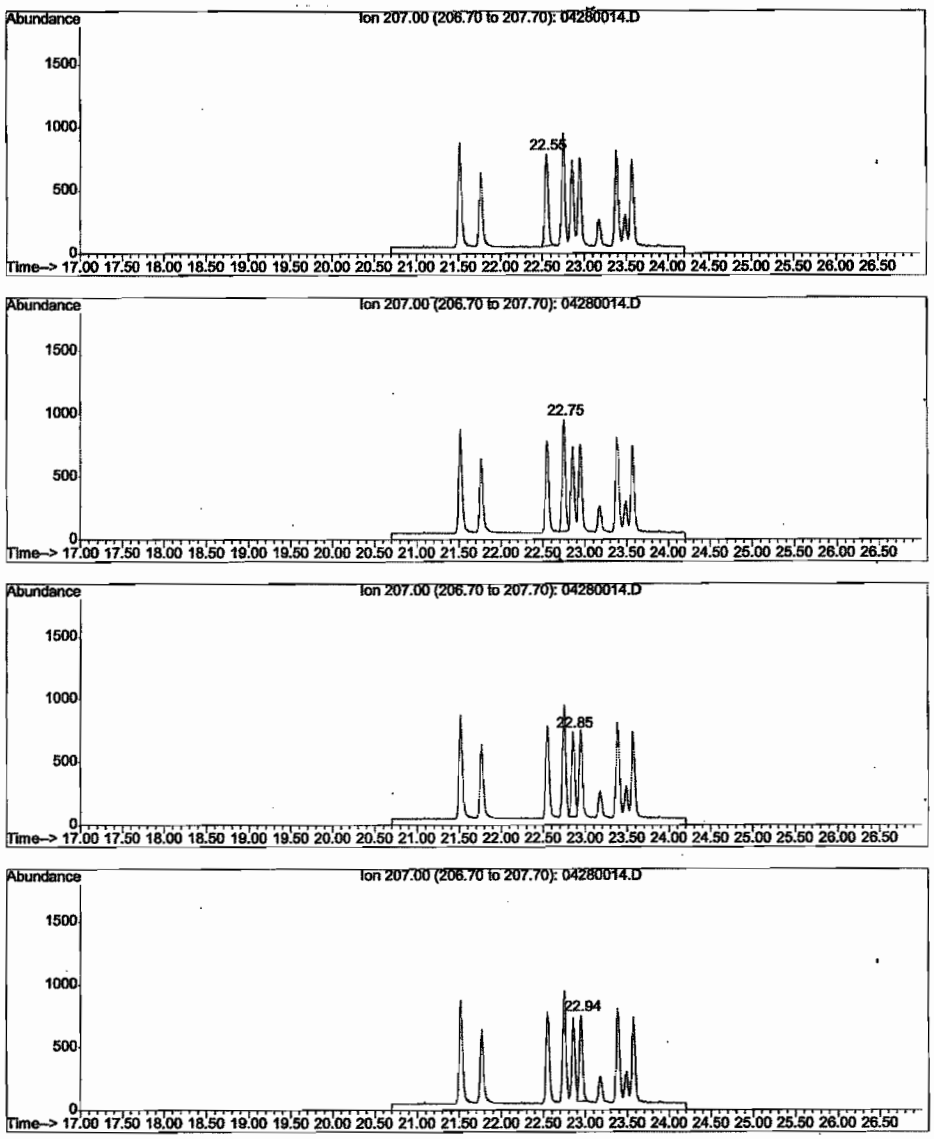
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Permethrin
Peak Response (area): 39075
Permethrin Reported Recovery: 97%

Figure 12. (con't) Fresh Water Sediment, Fortified Control (10×LOQ)

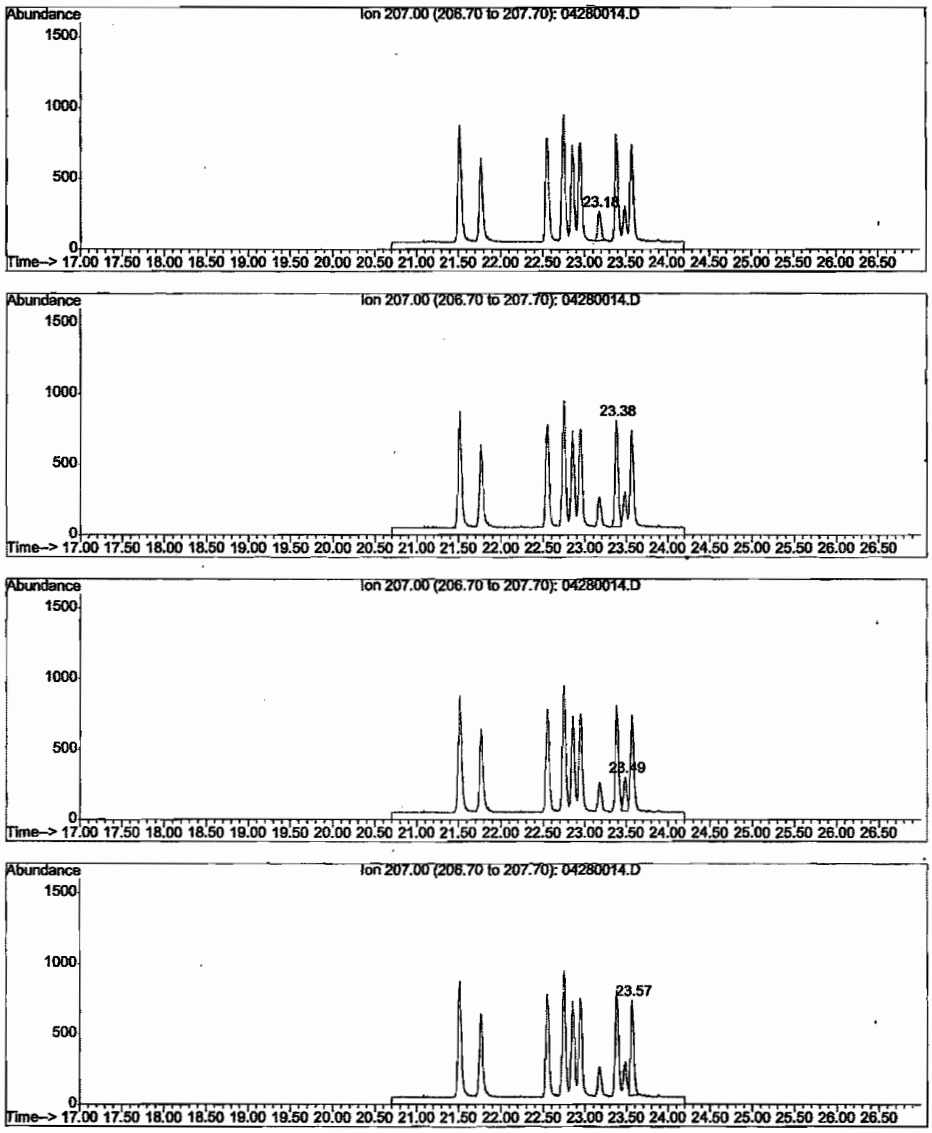
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Cyfluthrin
Peak Response (area): 80574
Cyfluthrin Reported Recovery: 111%

Figure 12. (con't) Fresh Water Sediment, Fortified Control (10×LOQ)

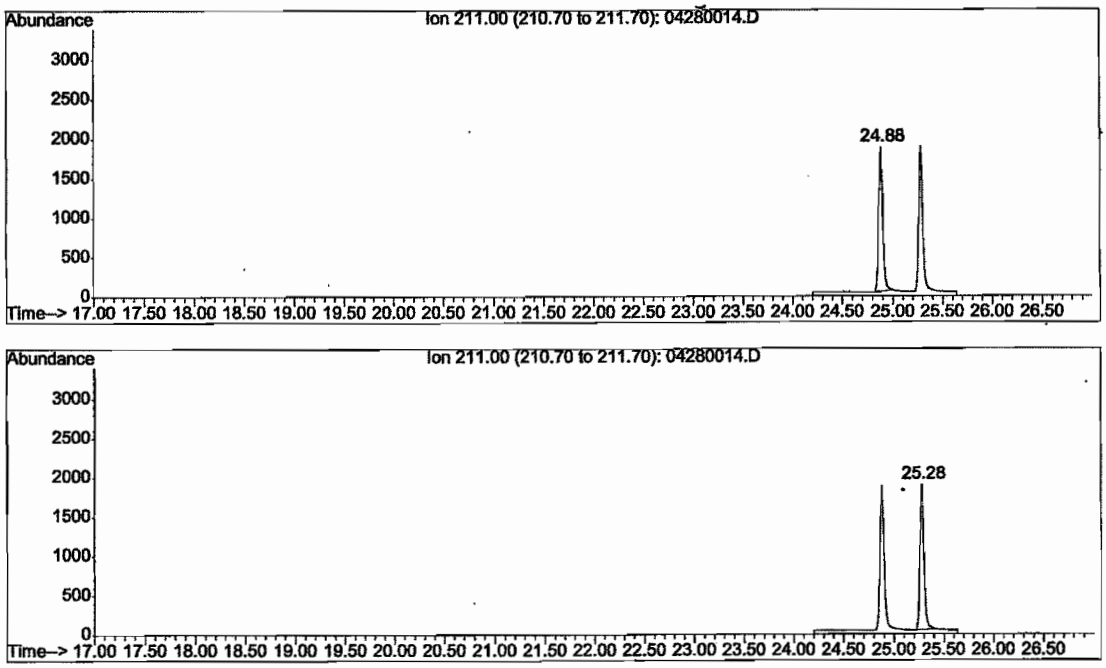
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Cypermethrin
Peak Response (area): 50676
Cypermethrin Reported Recovery: 111%

Figure 12. (con't) Fresh Water Sediment, Fortified Control (10×LOQ)

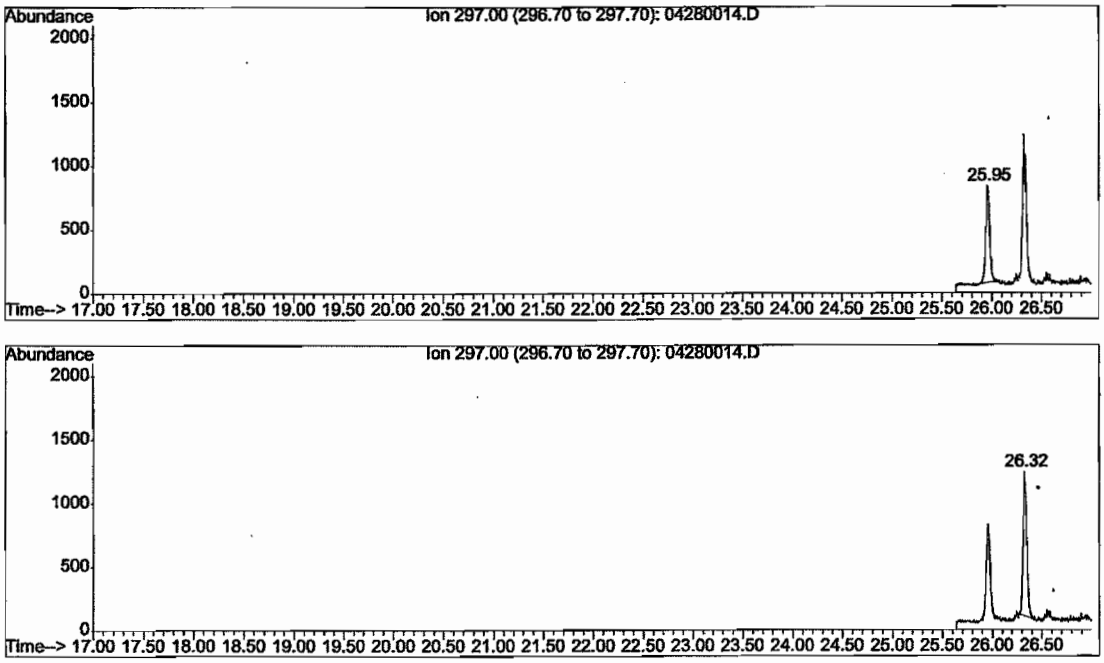
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Esfenvalerate
Peak Response (area): 102955
Esfenvalerate Reported Recovery: 105%

Figure 12. (con't) Fresh Water Sediment, Fortified Control (10×LOQ)

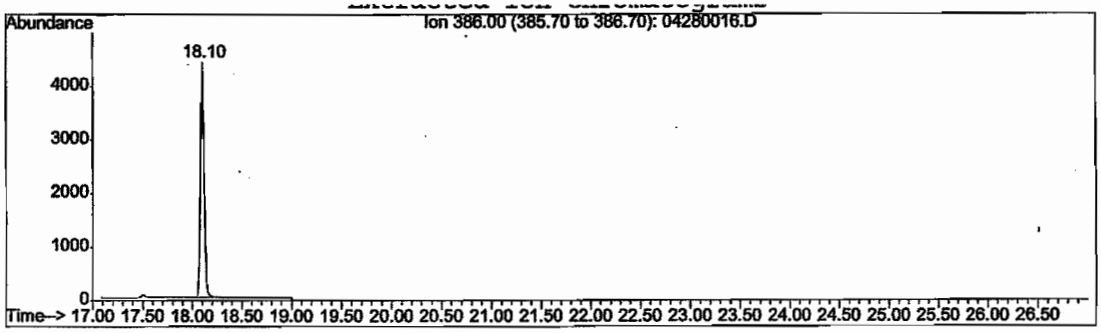
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 16 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



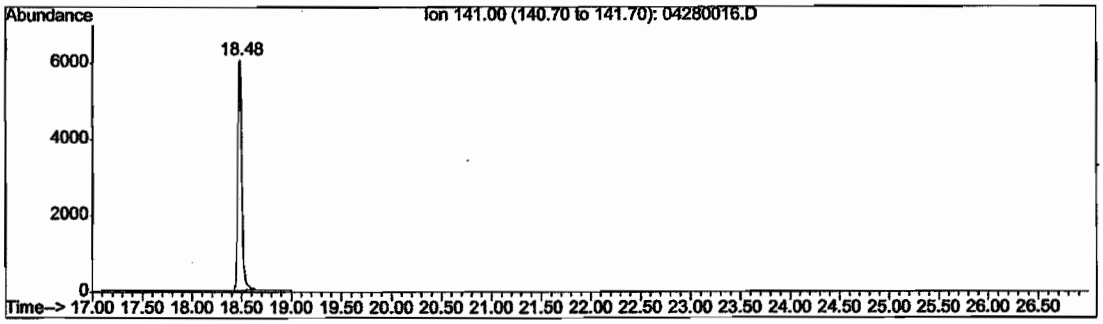
Deltamethrin
Peak Response (area): 48891
Deltamethrin Reported Recovery: 104%

Figure 13. Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



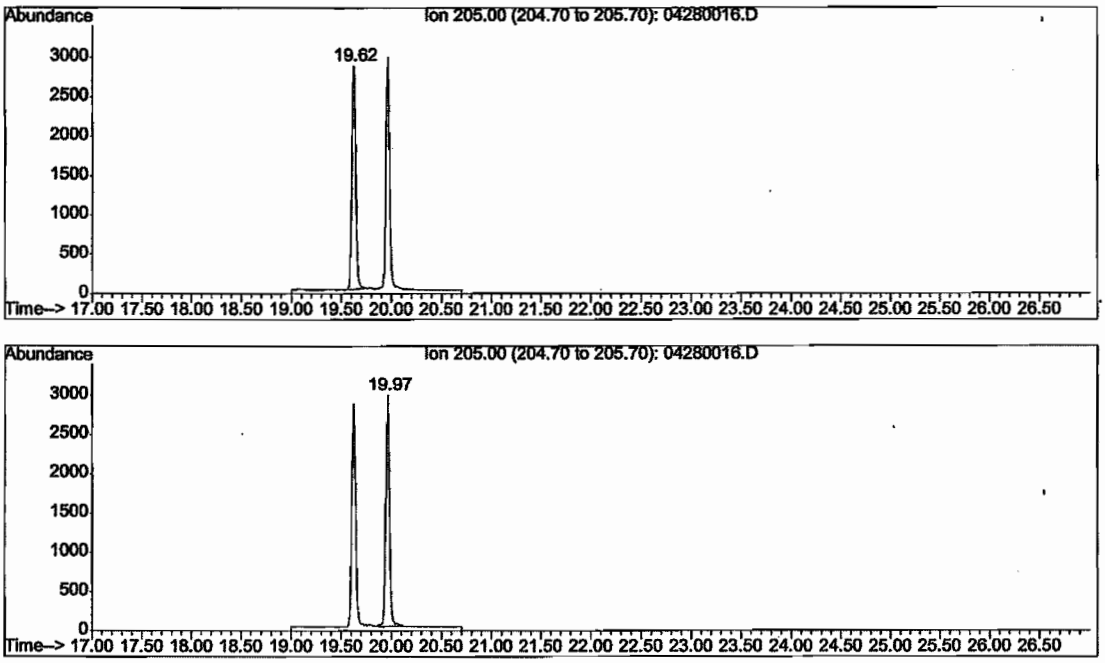
Bifenthrin
20 ng/mL
Peak Response (area): 116412



Fenpropathrin
20 ng/mL
Peak Response (area): 169884

Figure 13. (con't) Bracketing Standards

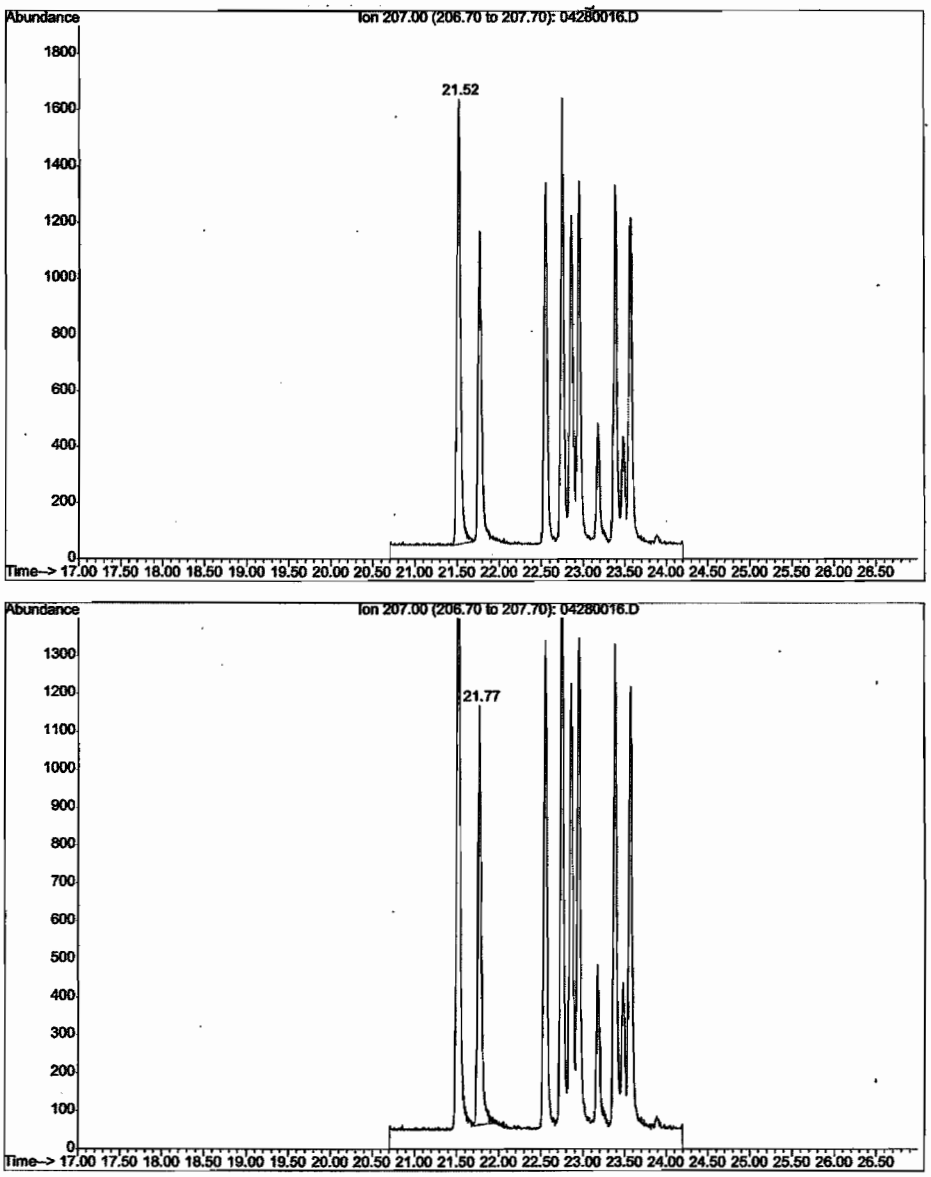
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 154546

Figure 13. (con't) Bracketing Standards

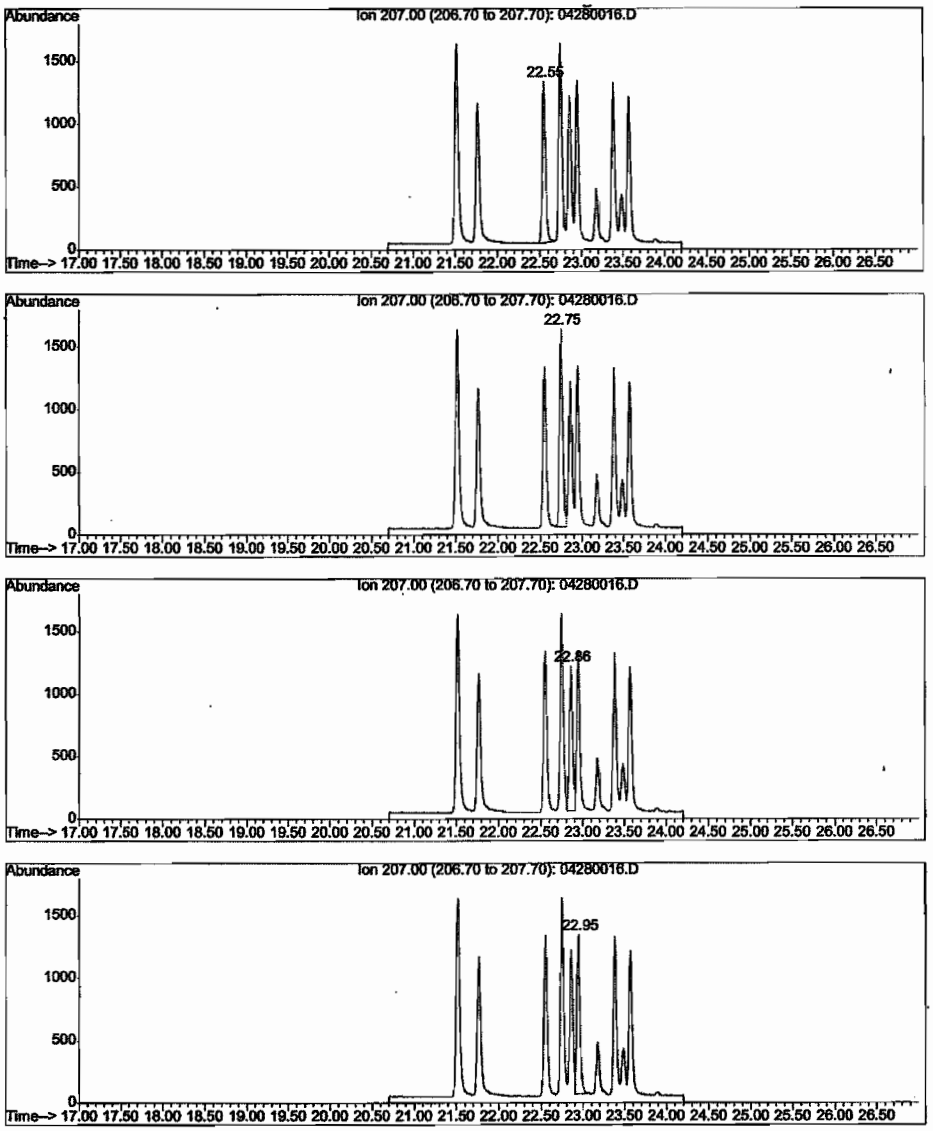
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Permethrin
200 ng/mL
Peak Response (area): 80924

Figure 13. (con't) Bracketing Standards

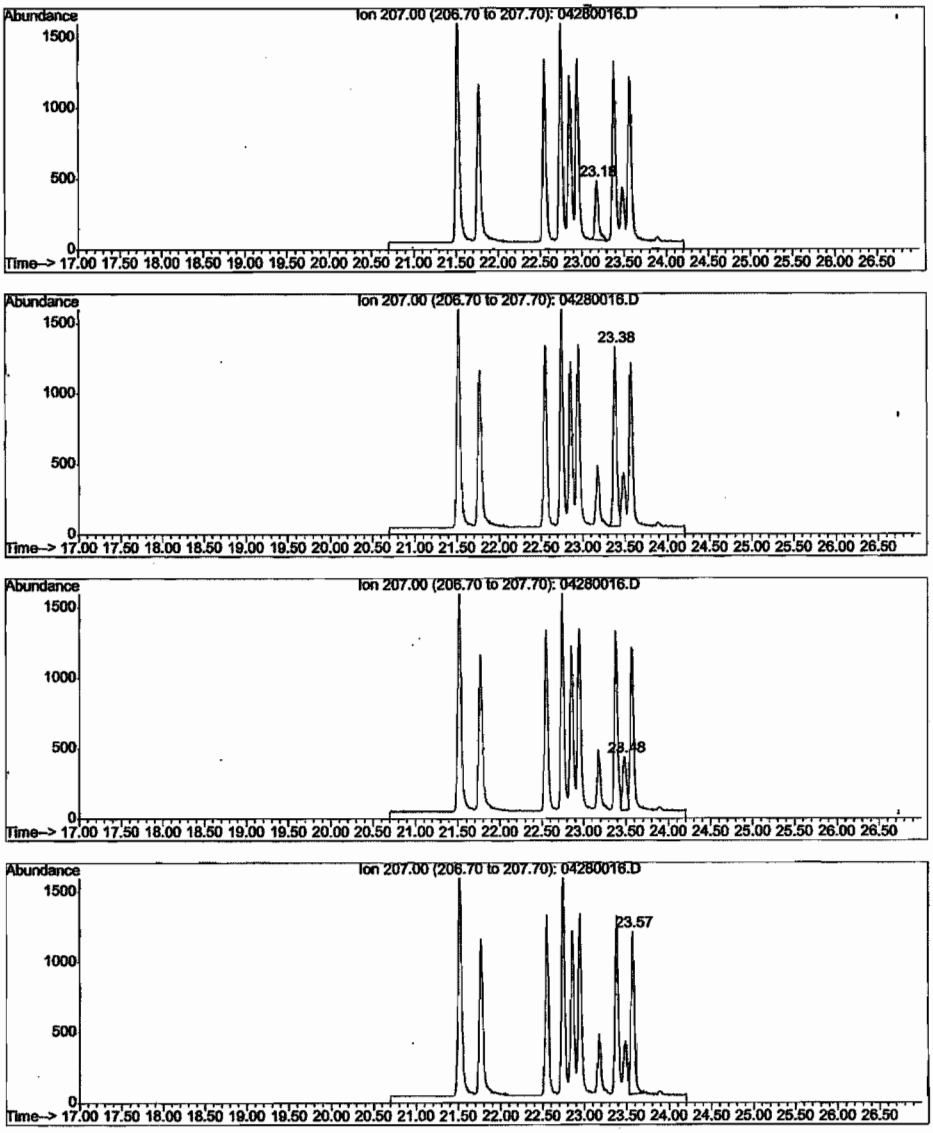
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cyfluthrin
20 ng/mL
Peak Response (area): 144415

Figure 13. (con't) Bracketing Standards

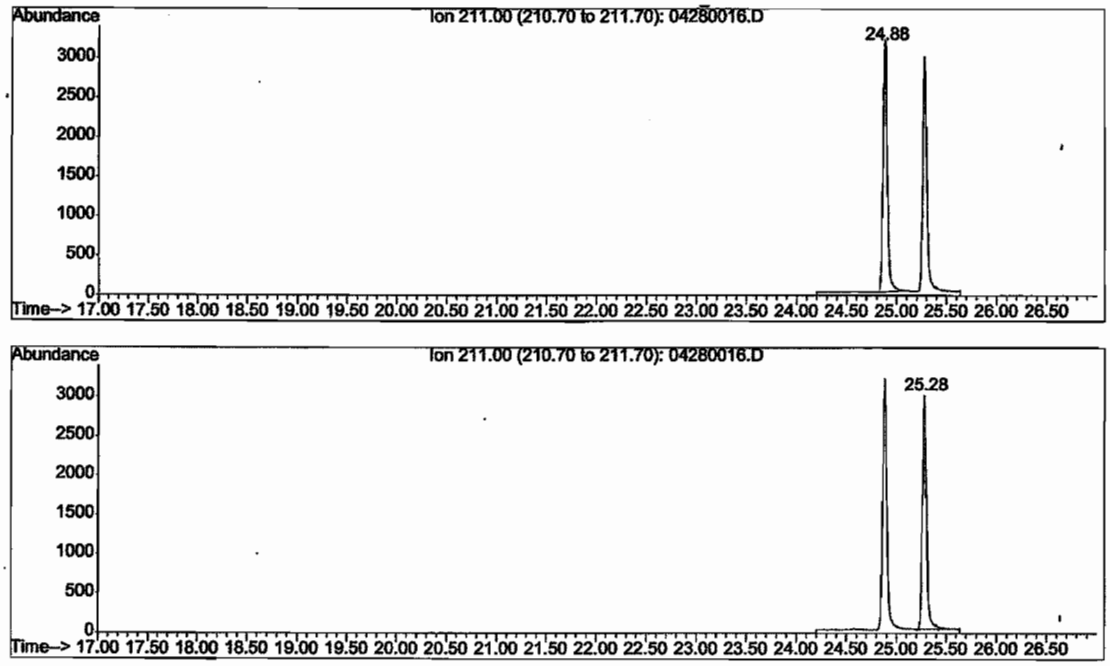
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cypermethrin
20 ng/mL
Peak Response (area): 91267

Figure 13. (con't) Bracketing Standards

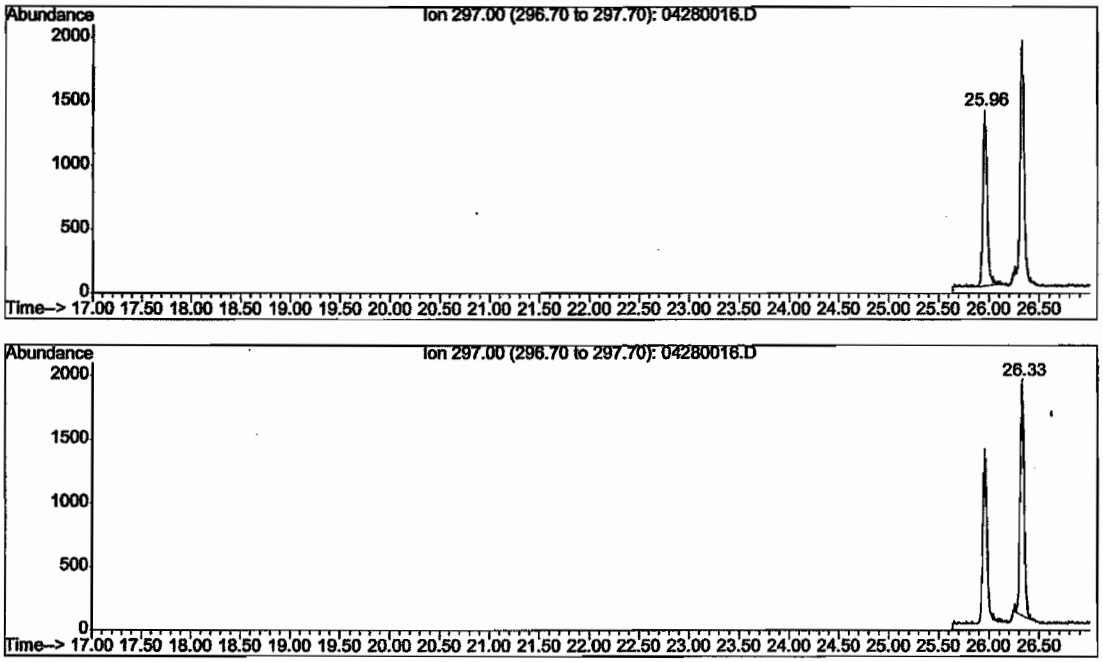
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Esfenvalerate
20 ng/mL
Peak Response (area): 181974

Figure 13. (con't) Bracketing Standards

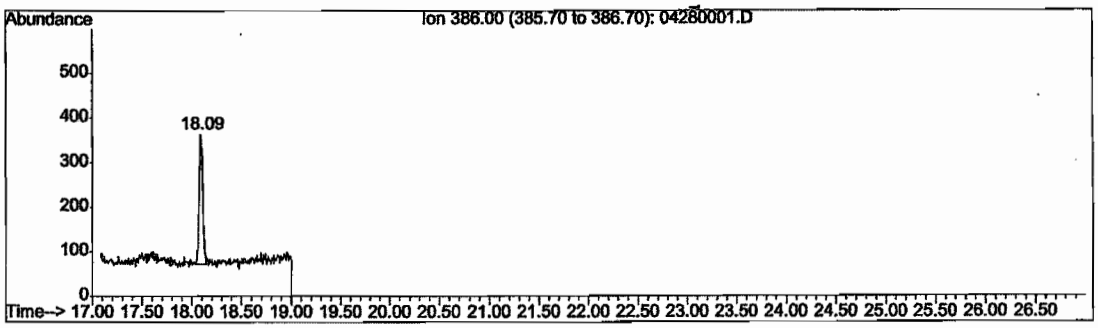
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



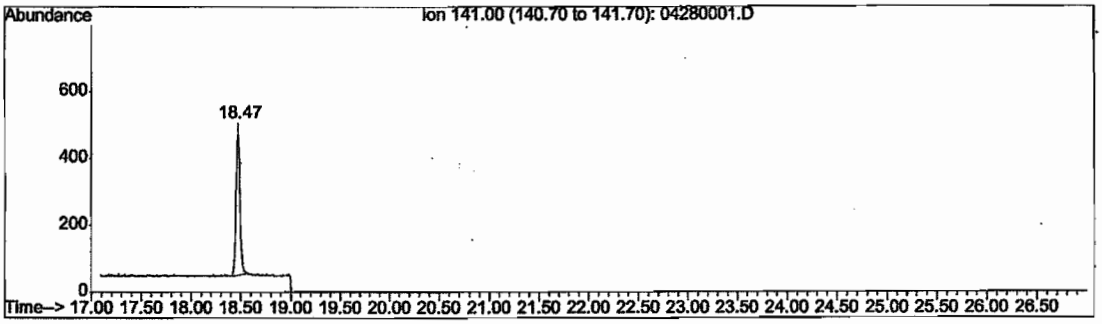
Deltamethrin
20 ng/mL
Peak Response (area): 88712

Figure 14. Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



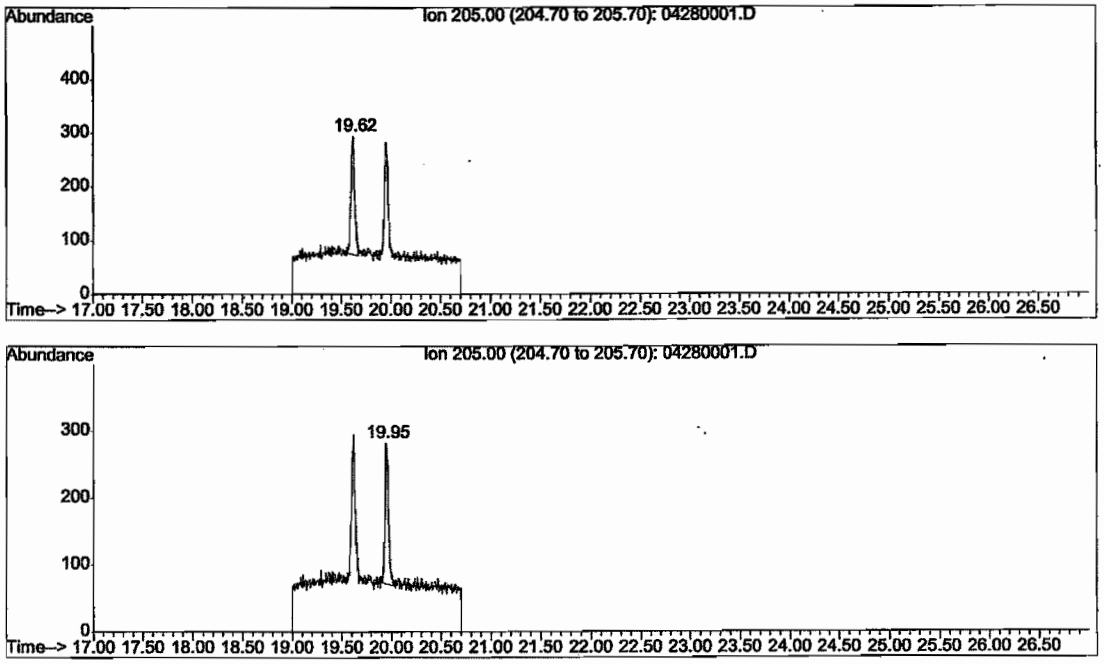
Bifenthrin
1.0 ng/mL
Peak Response (area): 7454



Fenpropathrin
1.0 ng/mL
Peak Response (area): 11706

Figure 14. (con't) Bracketing Standards

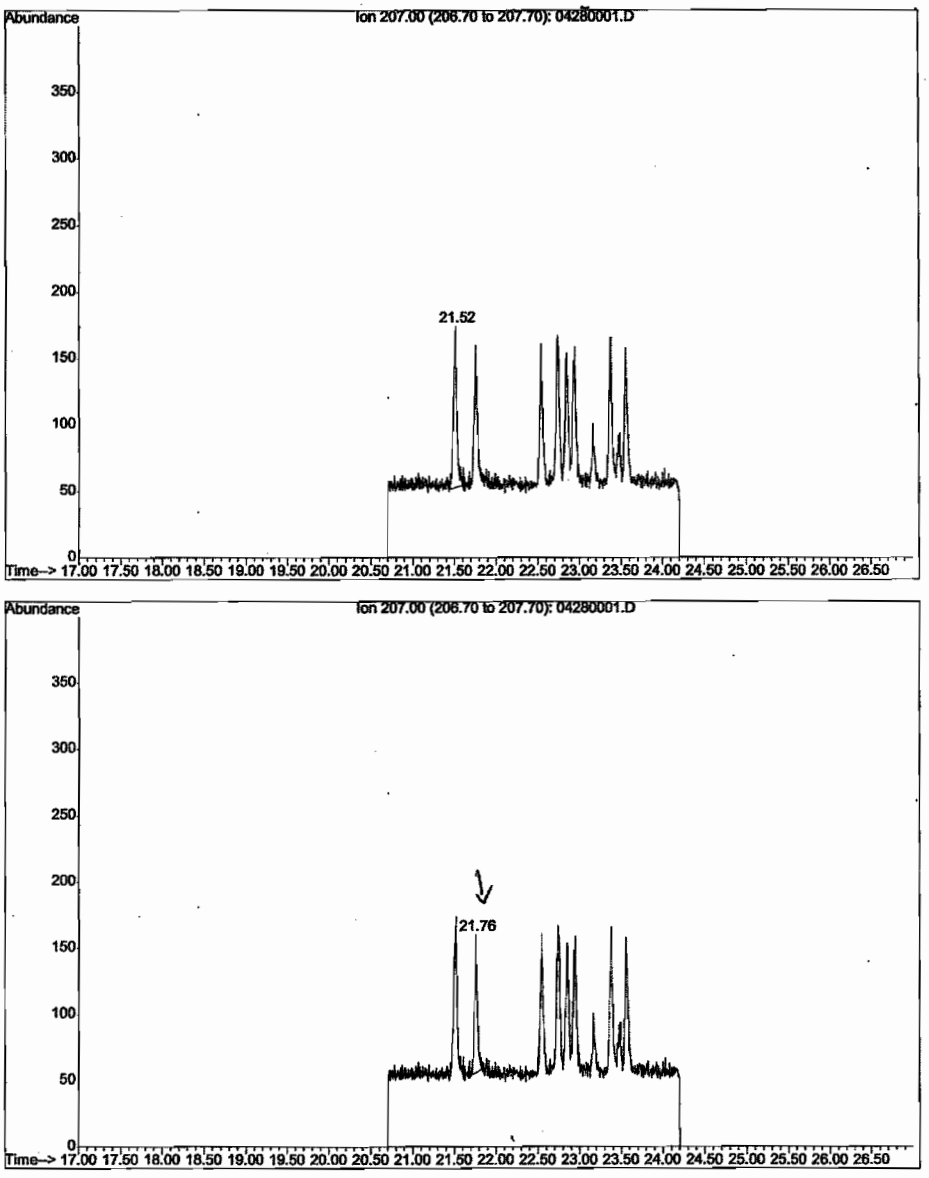
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 10784

Figure 14. (con't) Bracketing Standards

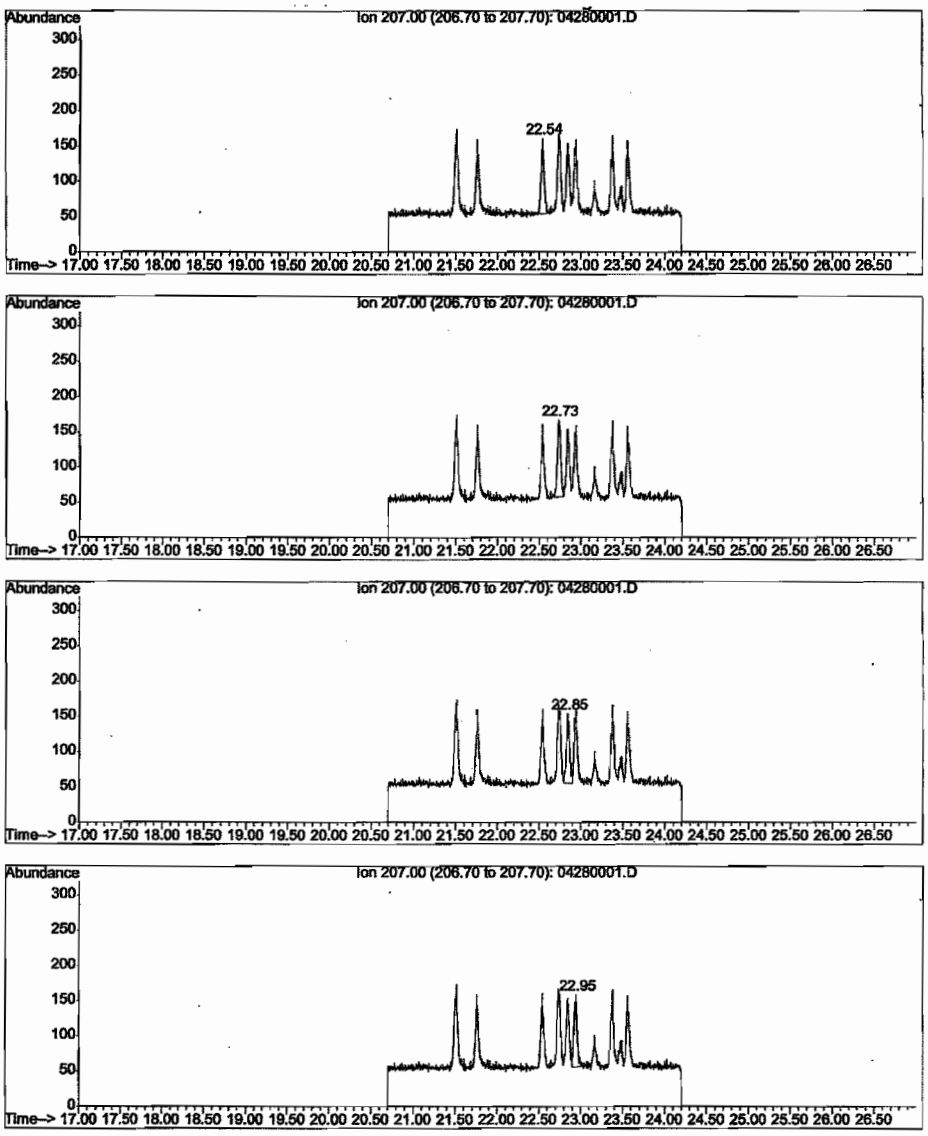
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Permethrin
10 ng/mL
Peak Response (area): 5857

Figure 14. (con't) Bracketing Standards

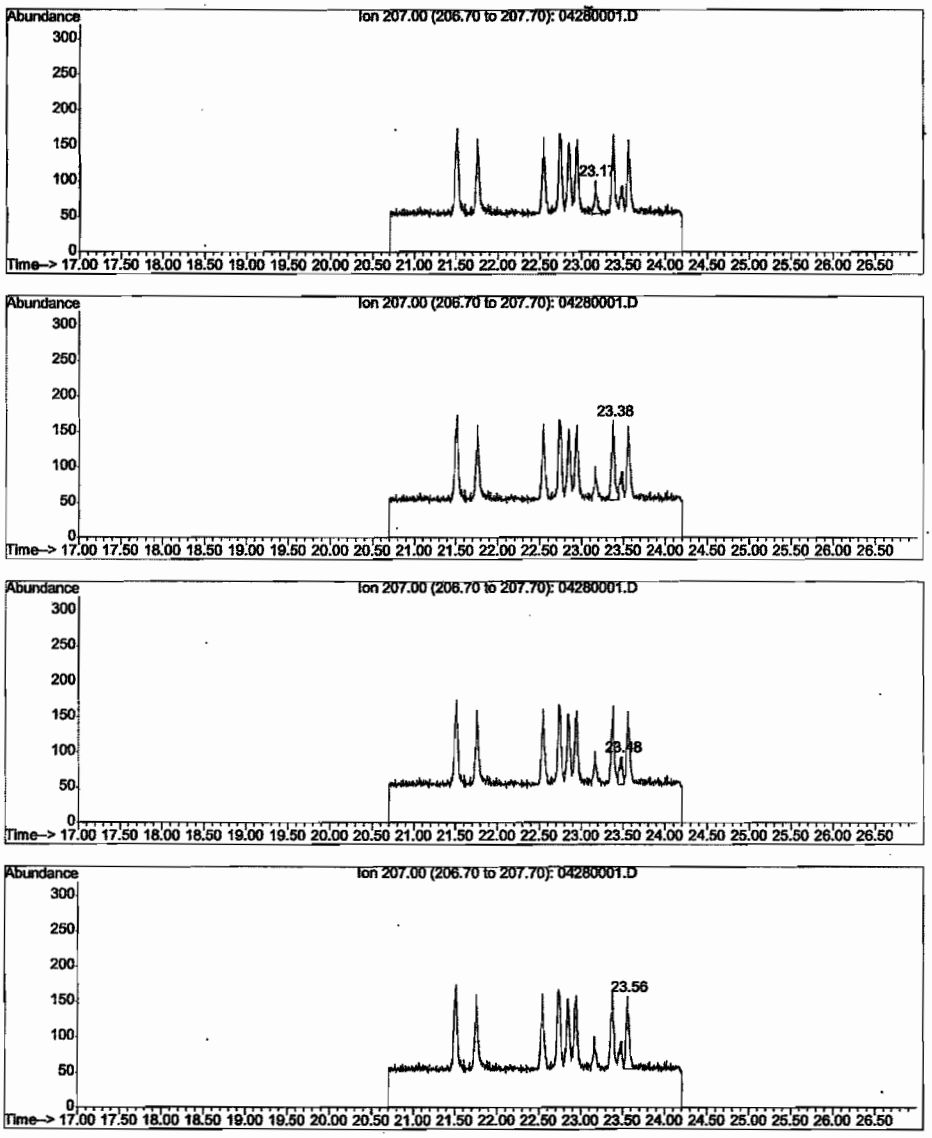
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cyfluthrin
1.0 ng/mL
Peak Response (area): 10870

Figure 14. (con't) **Bracketing Standards**

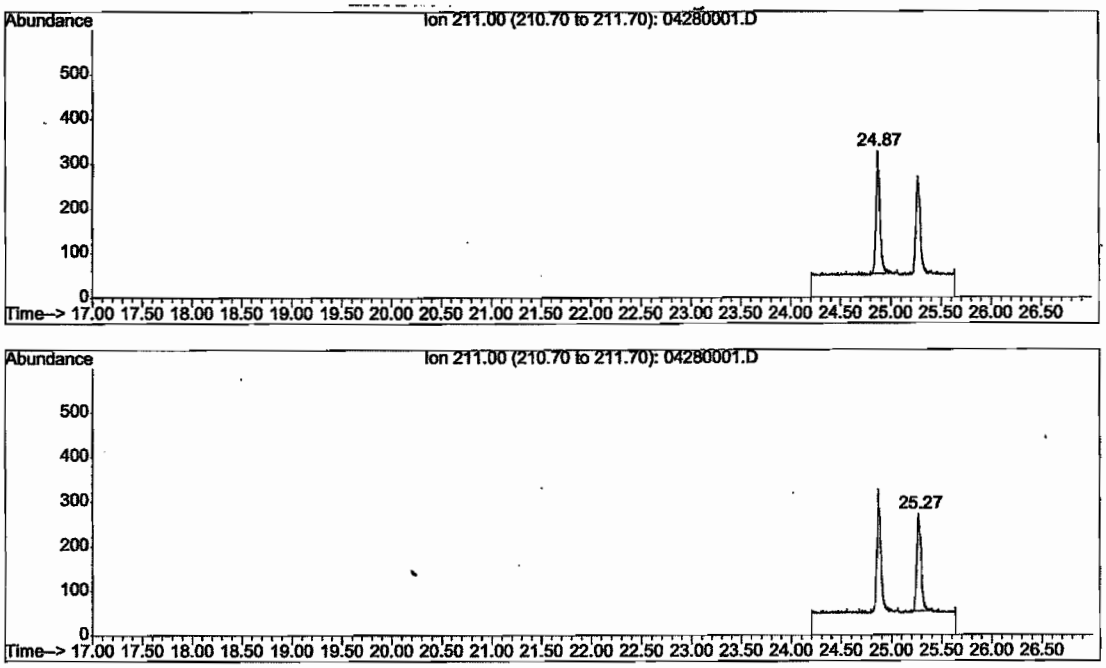
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cypermethrin
1.0 ng/mL
Peak Response (area): 7257

Figure 14. (con't) **Bracketing Standards**

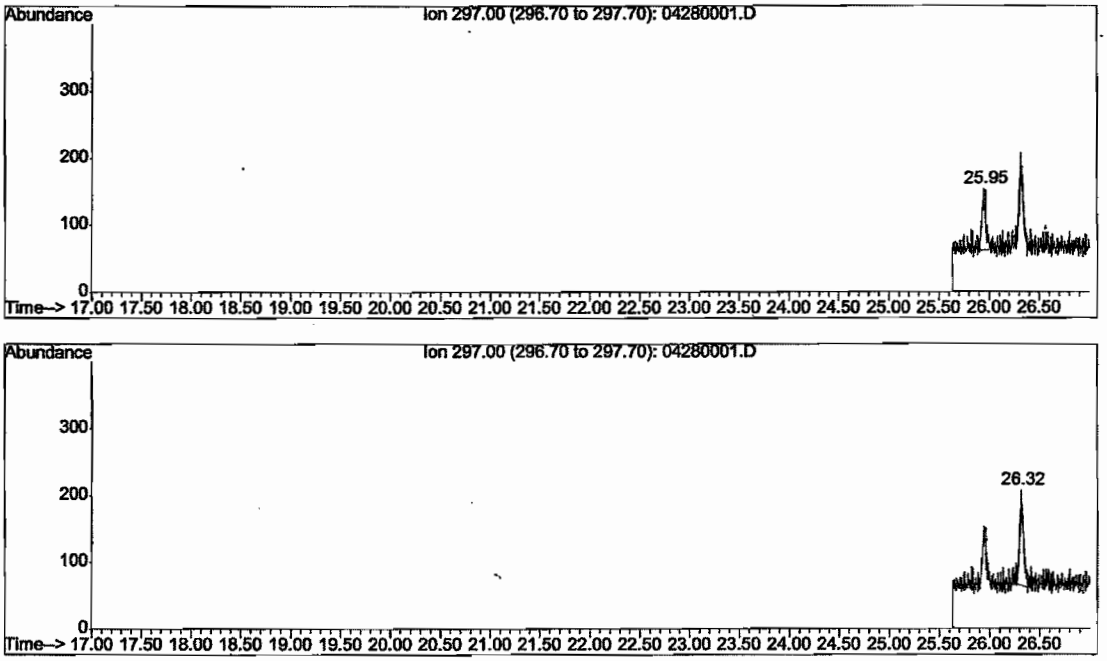
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Esfenvalerate
1.0 ng/mL
Peak Response (area): 13421

Figure 14. (con't) Bracketing Standards

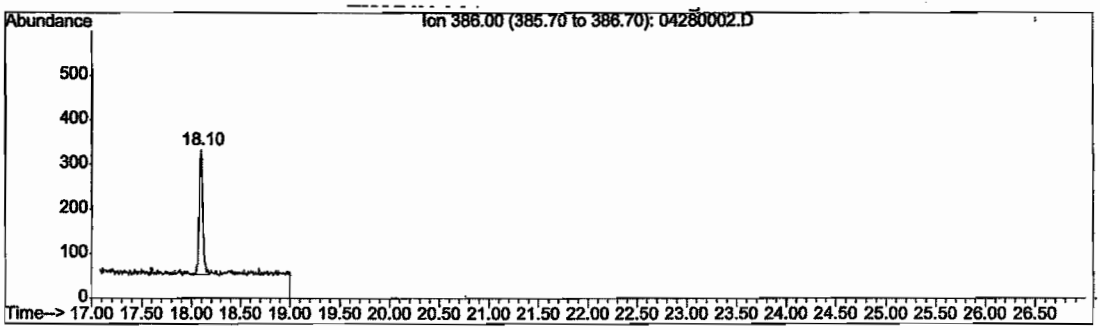
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



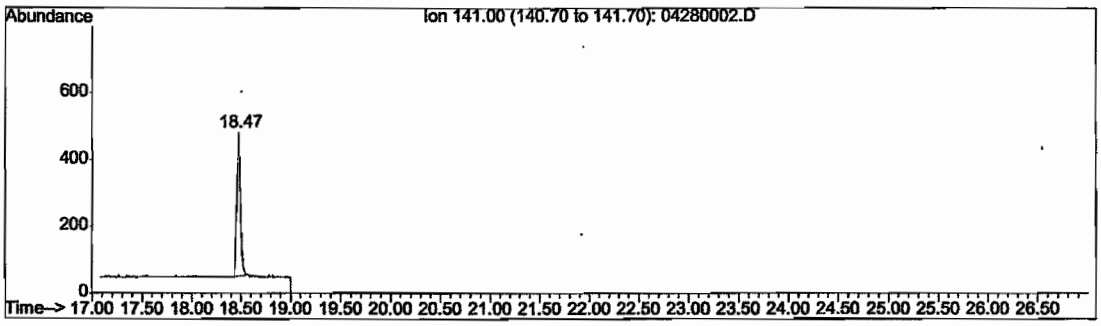
Deltamethrin
1.0 ng/mL
Peak Response (area): 6022

Figure 15. Fresh Water Sediment, Fortified Control (LOQ)

Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



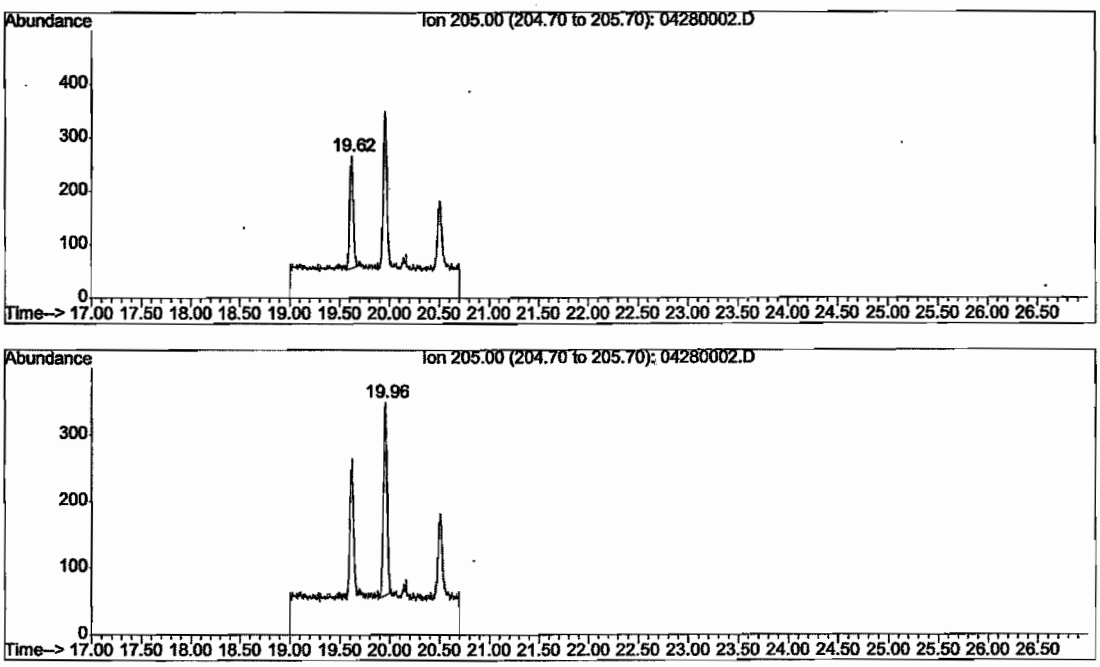
Bifenthrin
Peak Response (area): 7355
Bifenthrin Reported Recovery: 103%



Fenpropathrin
Peak Response (area): 11040
Fenpropathrin Reported Recovery: 102%

Figure 15. (con't) Fresh Water Sediment, Fortified Control (LOQ)

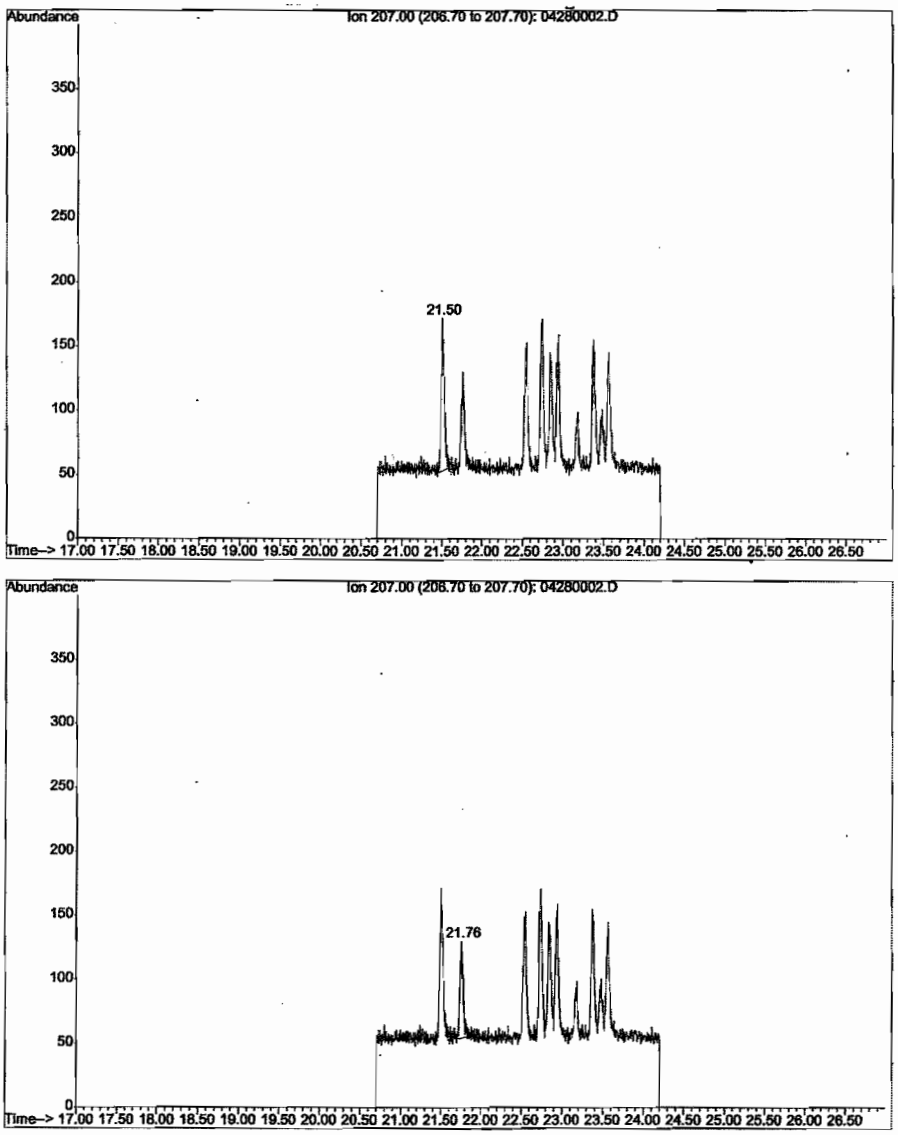
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



Lambda-cyhalothrin
Peak Response (area): 12470
Lambda-cyhalothrin Reported Recovery: 84%

Figure 15. (con't) Fresh Water Sediment, Fortified Control (LOQ)

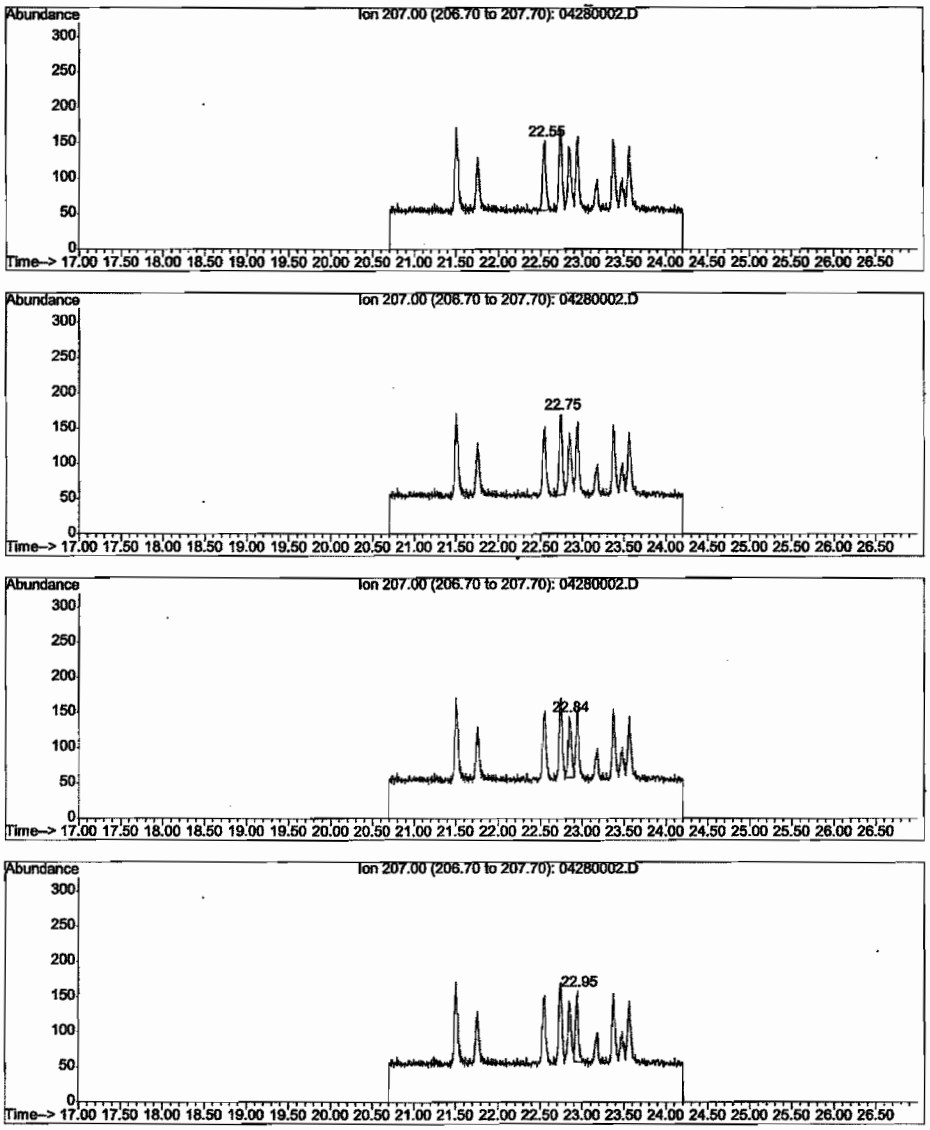
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



Permethrin
Peak Response (area): 5246
Permethrin Reported Recovery: 97%

Figure 15. (con't) Fresh Water Sediment, Fortified Control (LOQ)

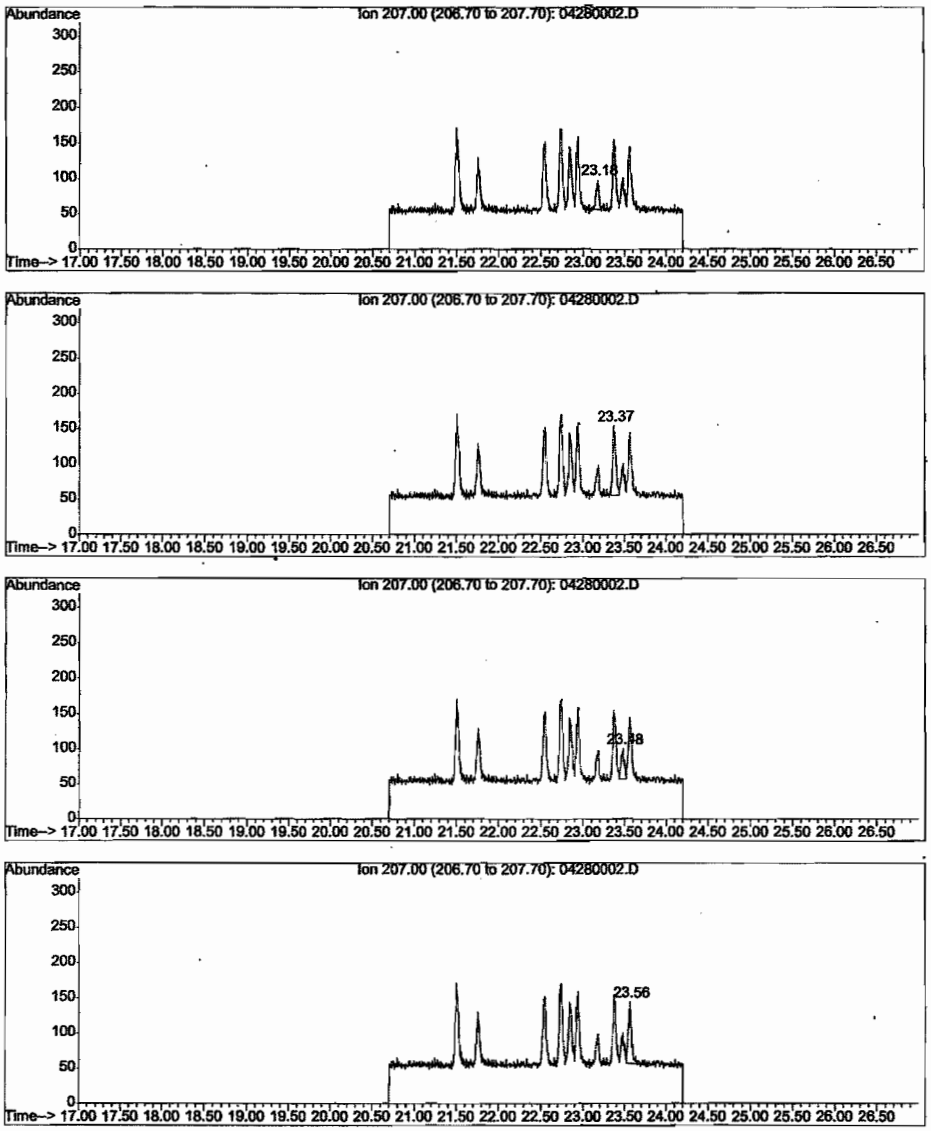
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



Cyfluthrin
Peak Response (area): 10542
Cyfluthrin Reported Recovery: 107%

Figure 15. (con't) Fresh Water Sediment, Fortified Control (LOQ)

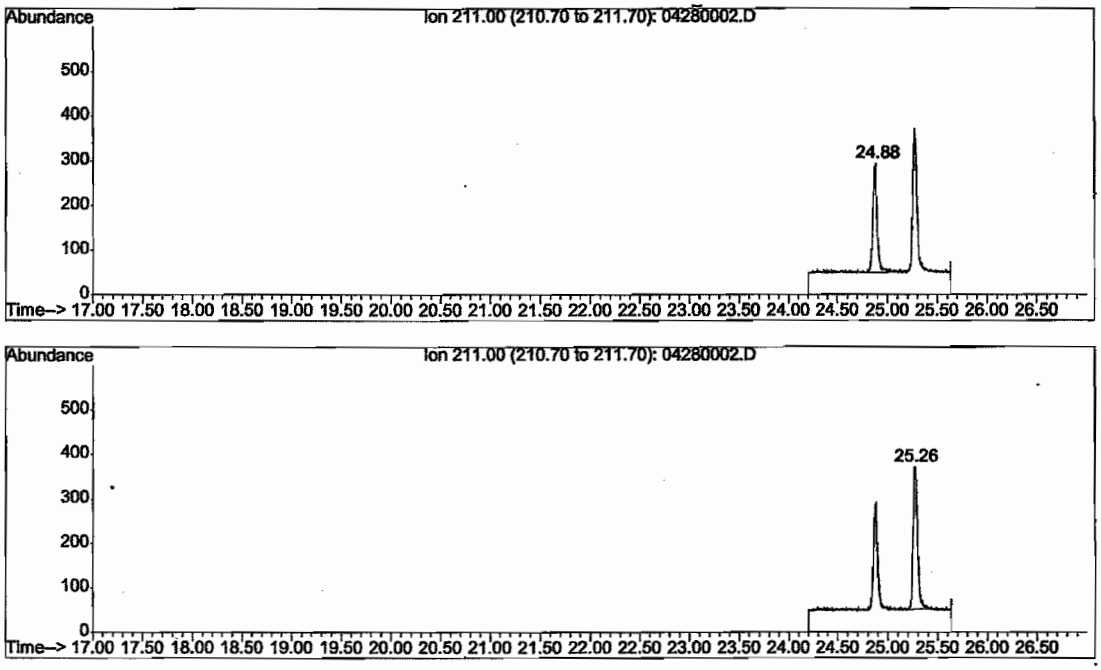
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



Cypermethrin
Peak Response (area): 7080
Cypermethrin Reported Recovery: 107%

Figure 15. (con't) Fresh Water Sediment, Fortified Control (LOQ)

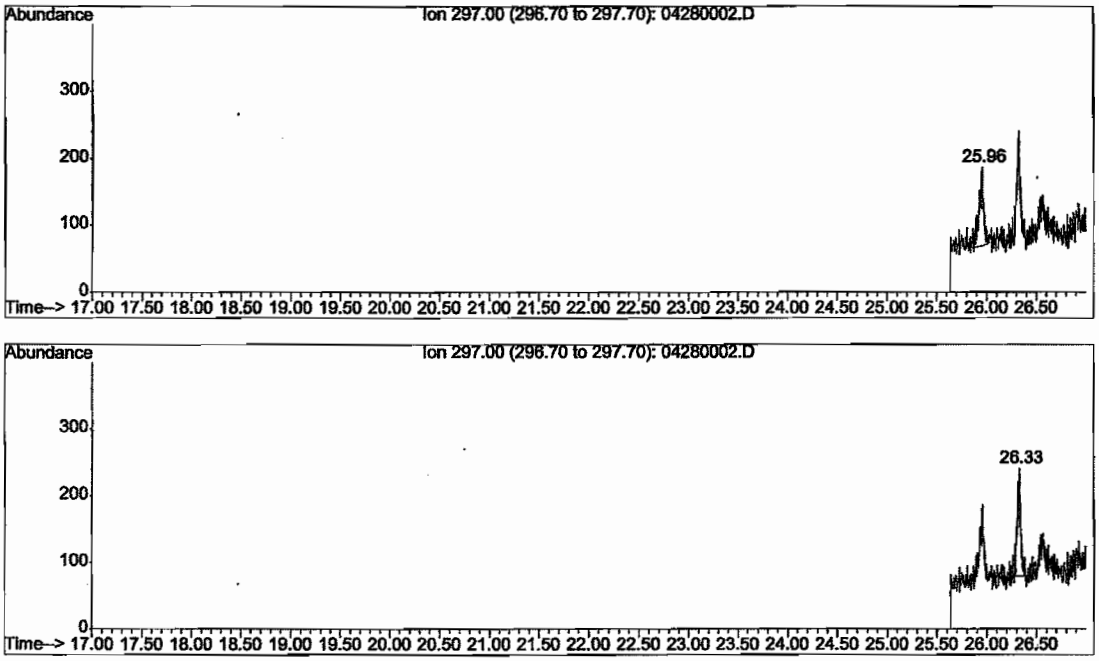
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



Esfenvalerate
Peak Response (area): 16887
Esfenvalerate Reported Recovery: 76%

Figure 15. (con't) Fresh Water Sediment, Fortified Control (LOQ)

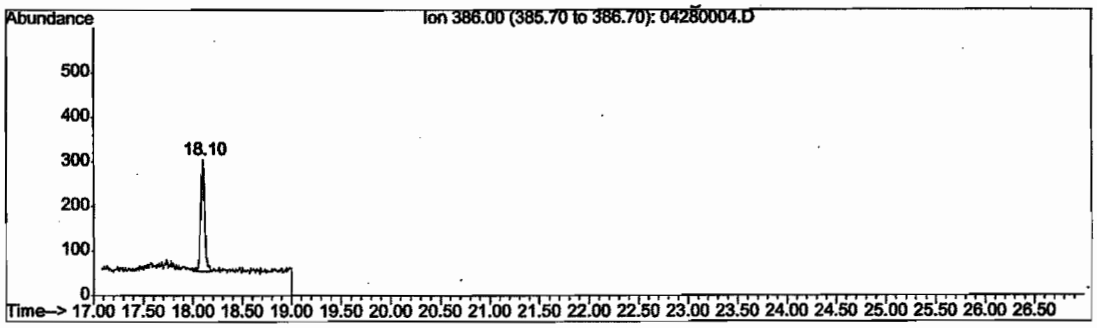
Fortified Control, Fresh Water Sediment, BUCGR, Fortified Control 11 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



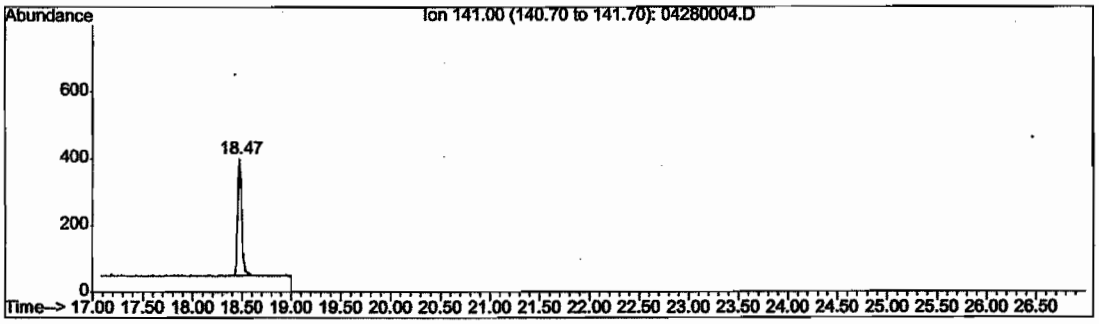
Deltamethrin
Peak Response (area): 7633
Deltamethrin Reported Recovery: 89%

Figure 16. Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



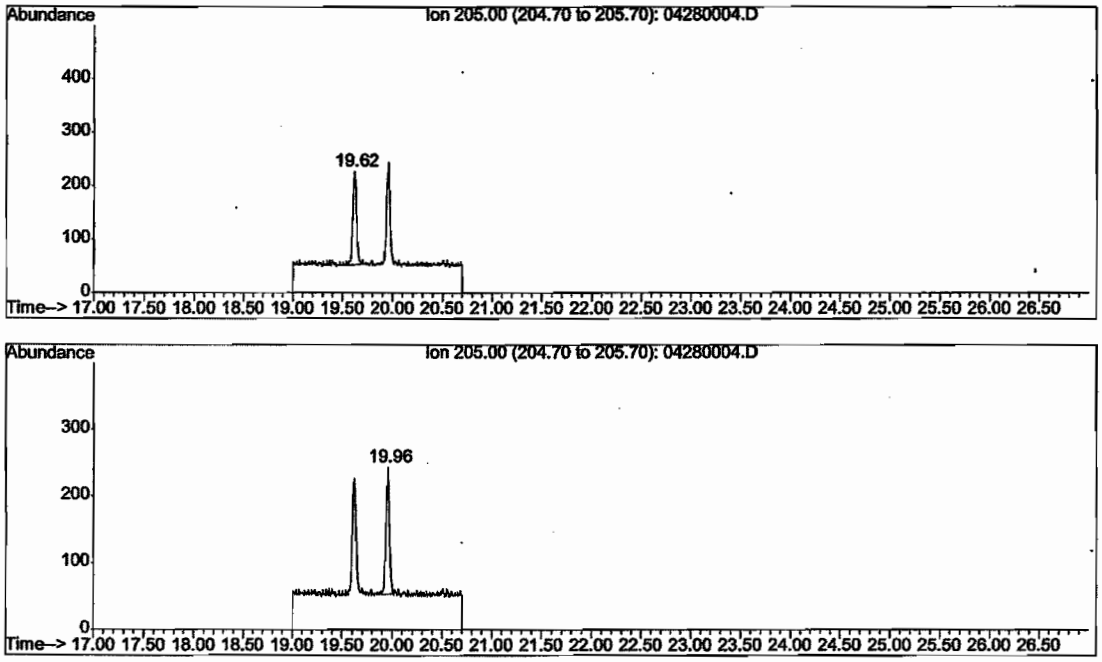
Bifenthrin
1.0 ng/mL
Peak Response (area): 6804



Fenpropathrin
1.0 ng/mL
Peak Response (area): 9881

Figure 16. (con't) **Bracketing Standards**

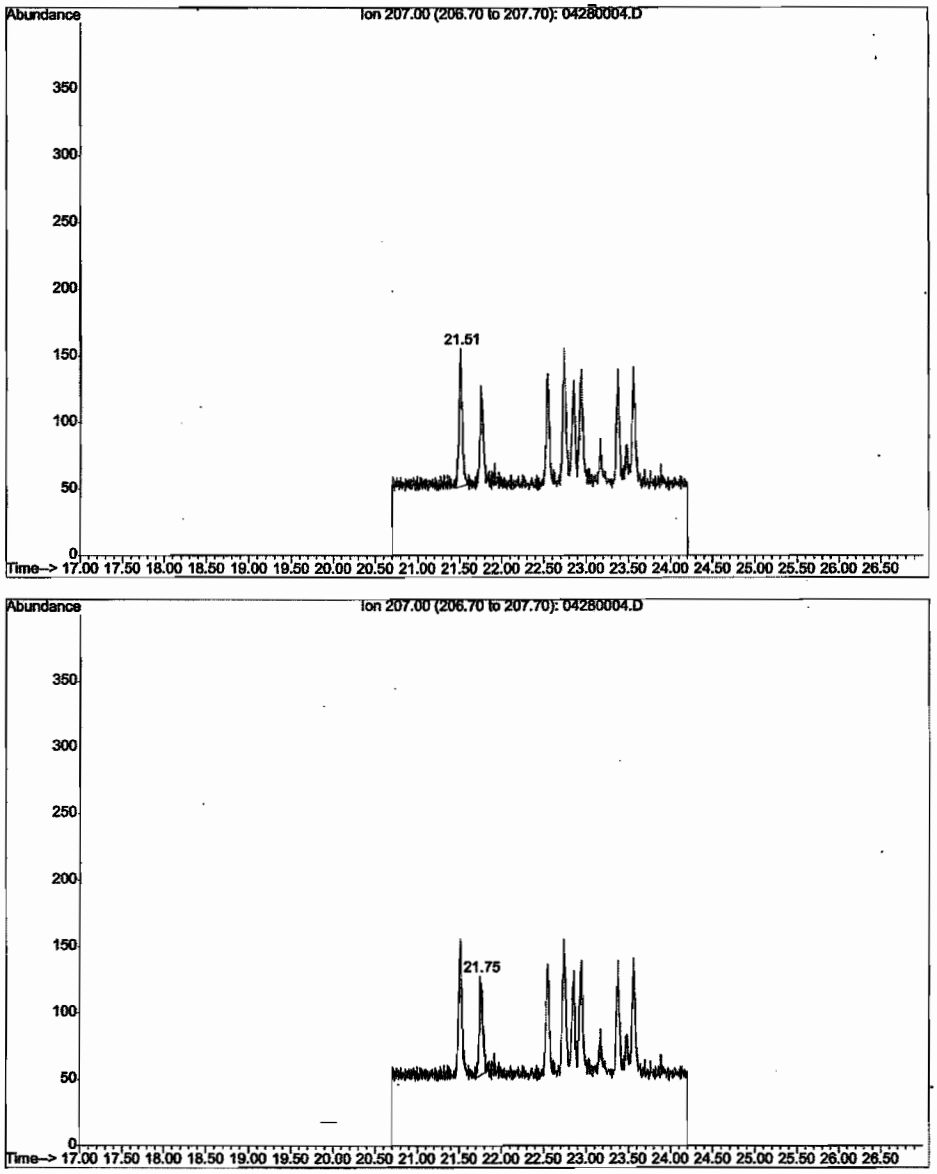
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 9119

Figure 16. (con't) Bracketing Standards

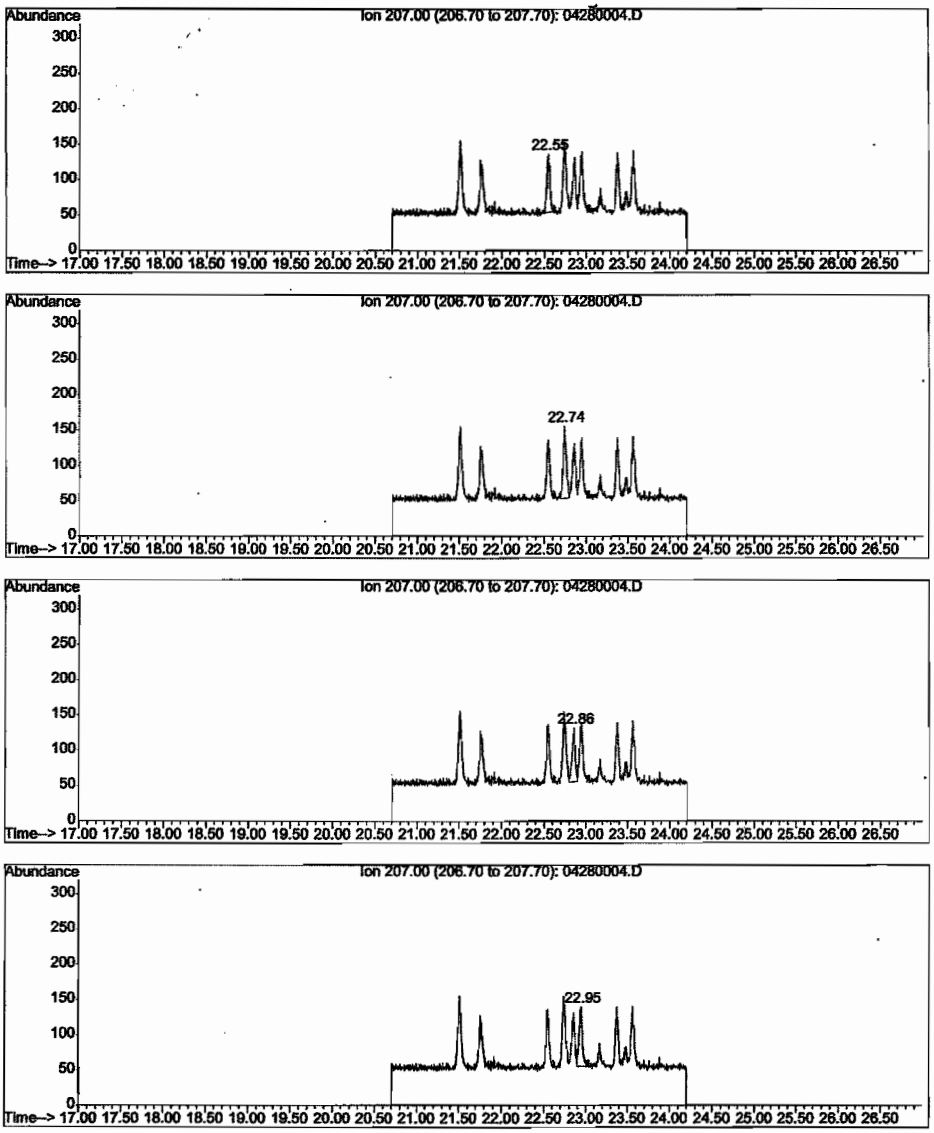
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Permethrin
10 ng/mL
Peak Response (area): 4918

Figure 16. (con't) Bracketing Standards

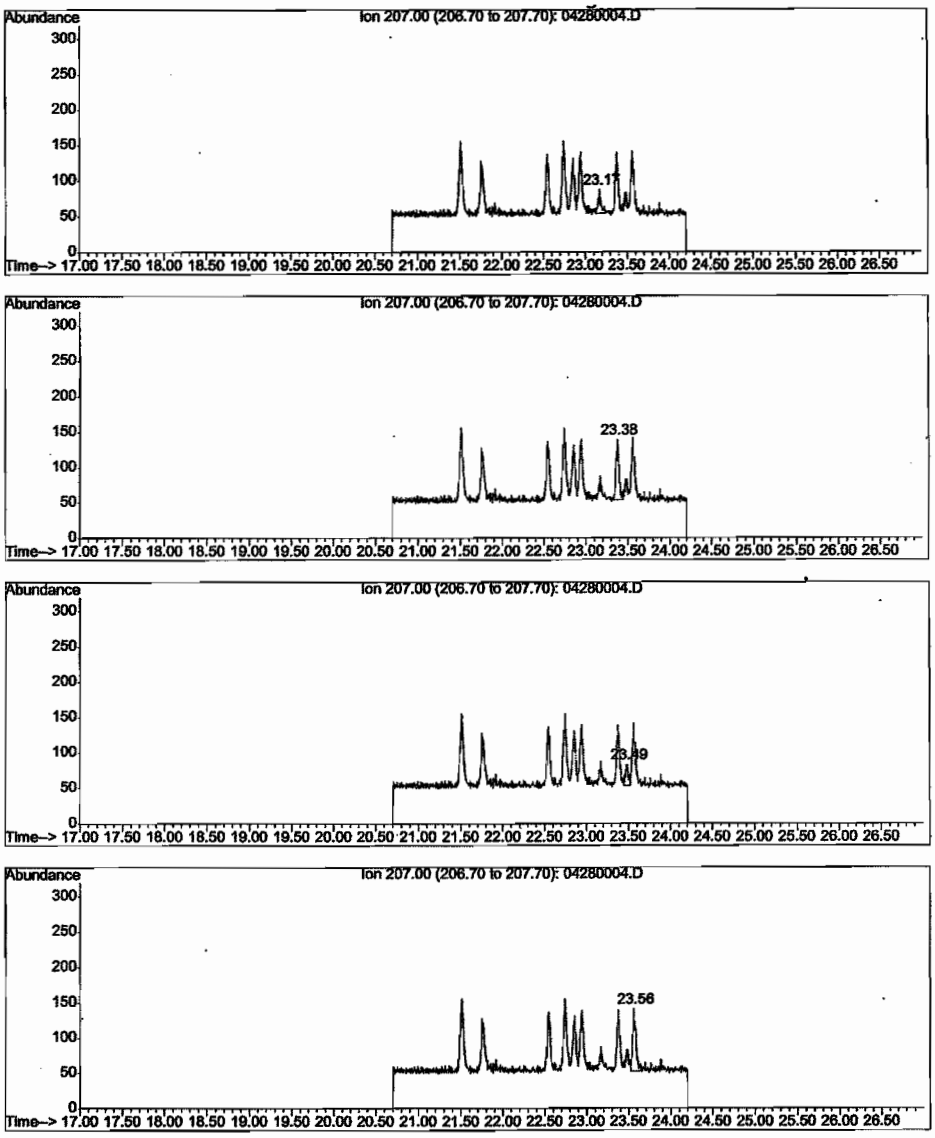
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cyfluthrin
1.0 ng/mL
Peak Response (area): 8872

Figure 16. (con't) Bracketing Standards

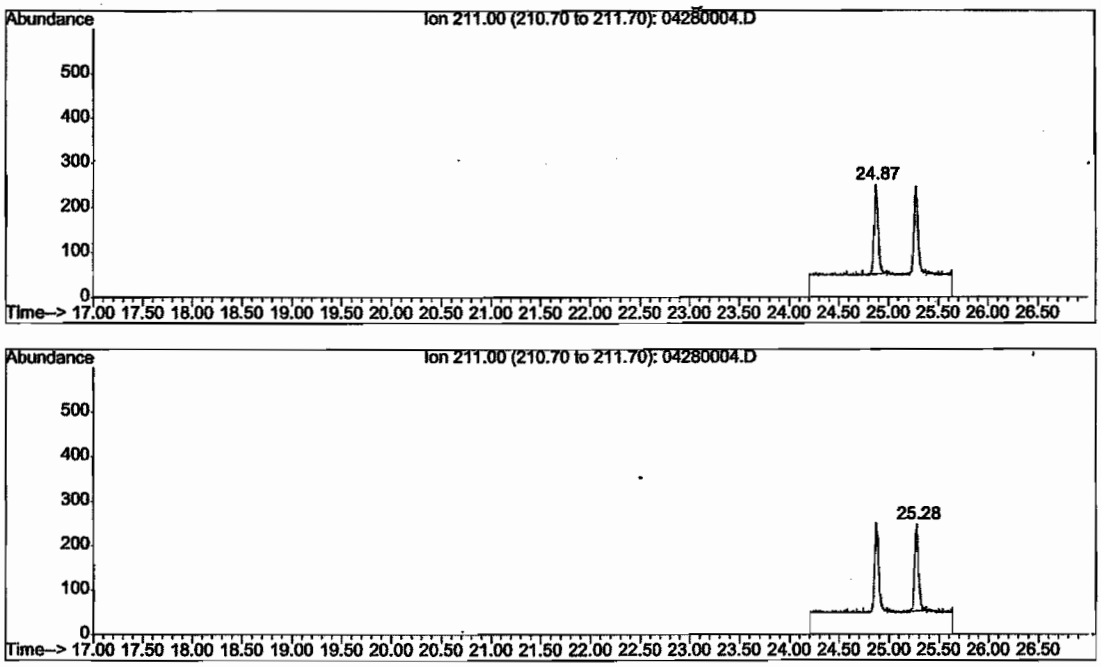
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cypermethrin
1.0 ng/mL
Peak Response (area): 6027

Figure 16. (con't) Bracketing Standards

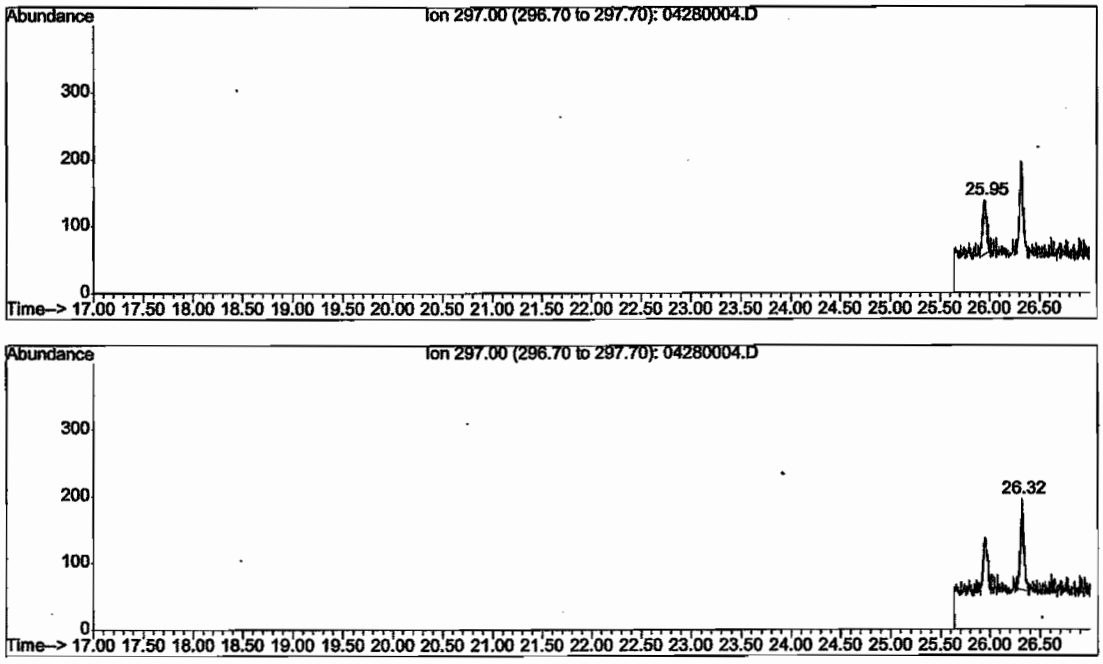
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Esfenvalerate
1.0 ng/mL
Peak Response (area): 10554

Figure 16. (con't) Bracketing Standards

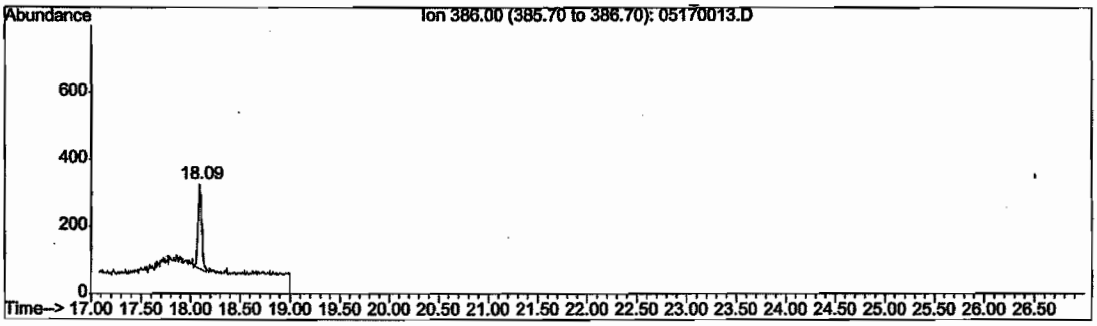
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



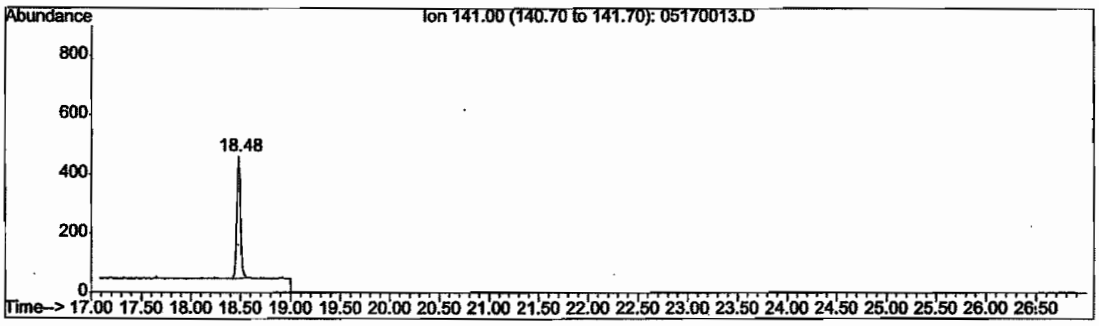
Deltamethrin
1.0 ng/mL
Peak Response (area): 5406

Figure 17. Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



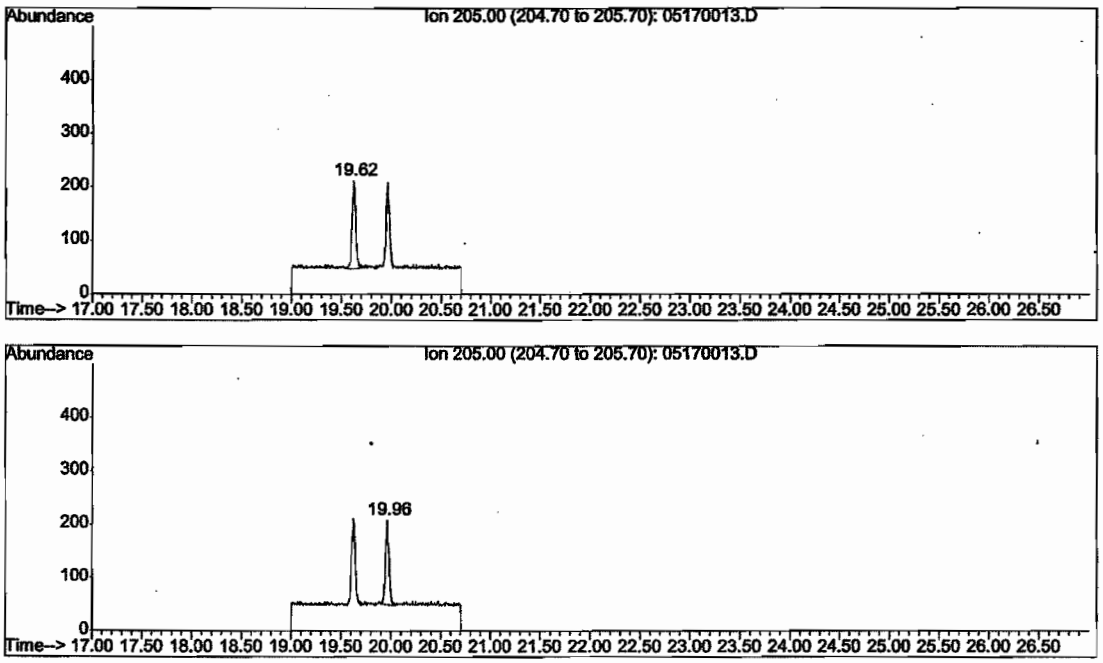
Bifenthrin
1.0 ng/mL
Peak Response (area): 6282



Fenpropathrin
1.0 ng/mL
Peak Response (area): 10454

Figure 17. (con't) Bracketing Standards

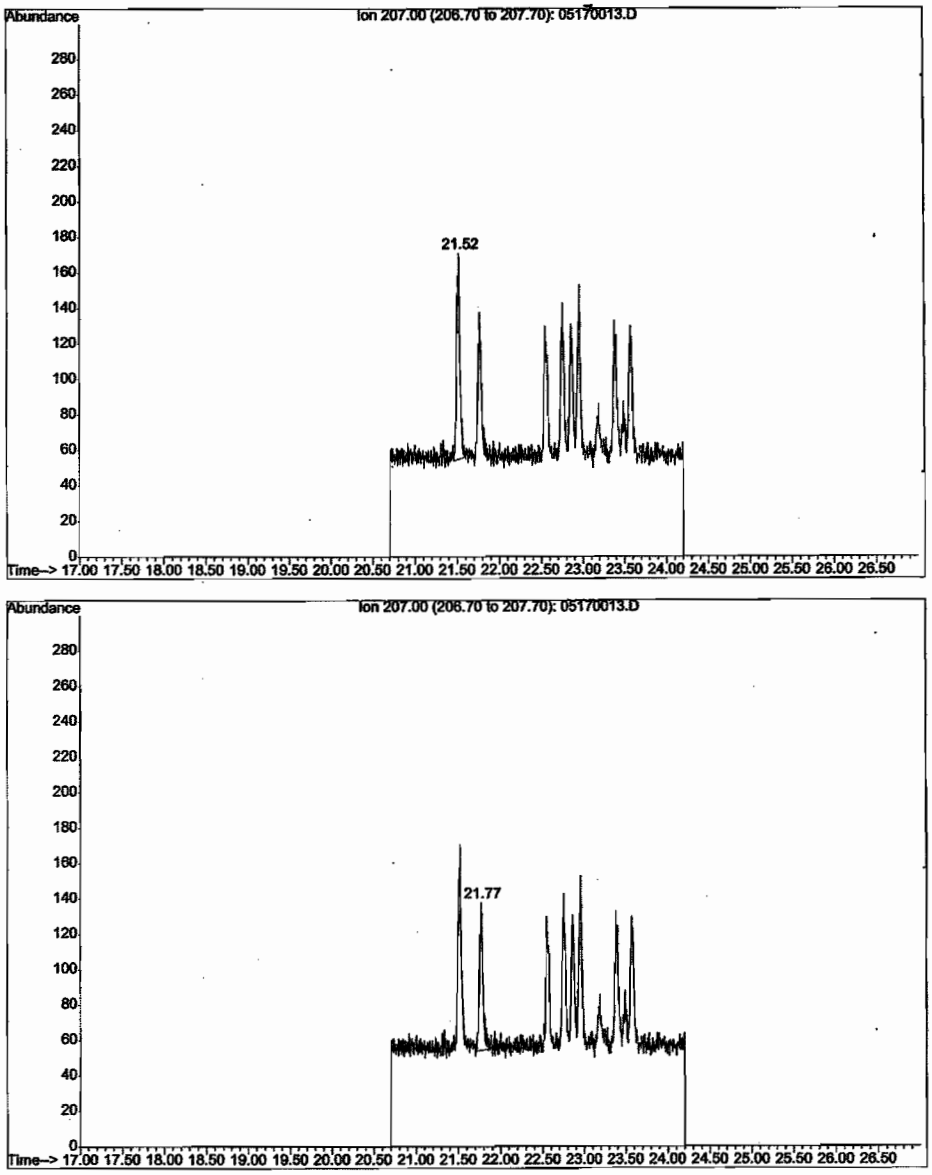
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 7973

Figure 17. (con't) Bracketing Standards

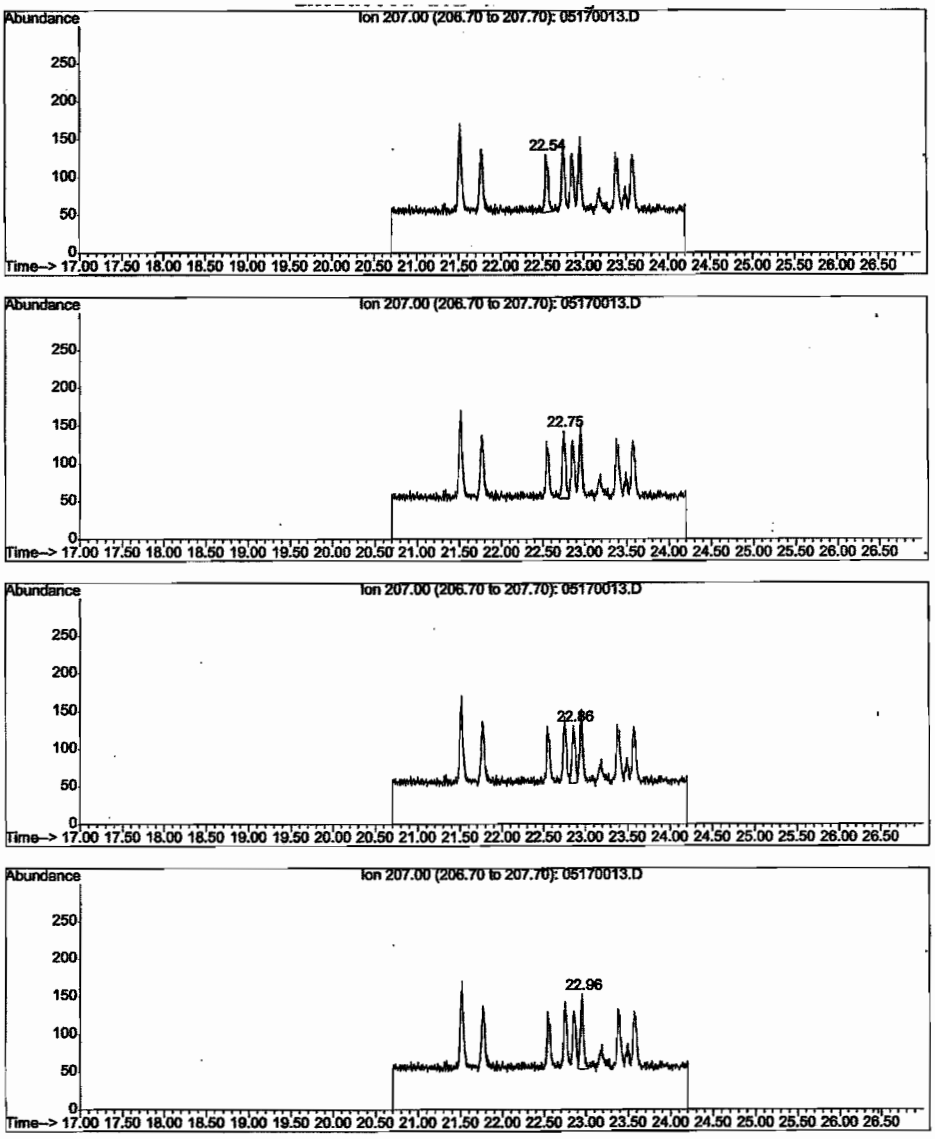
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Permethrin
10 ng/mL
Peak Response (area): 5389

Figure 17. (con't) Bracketing Standards

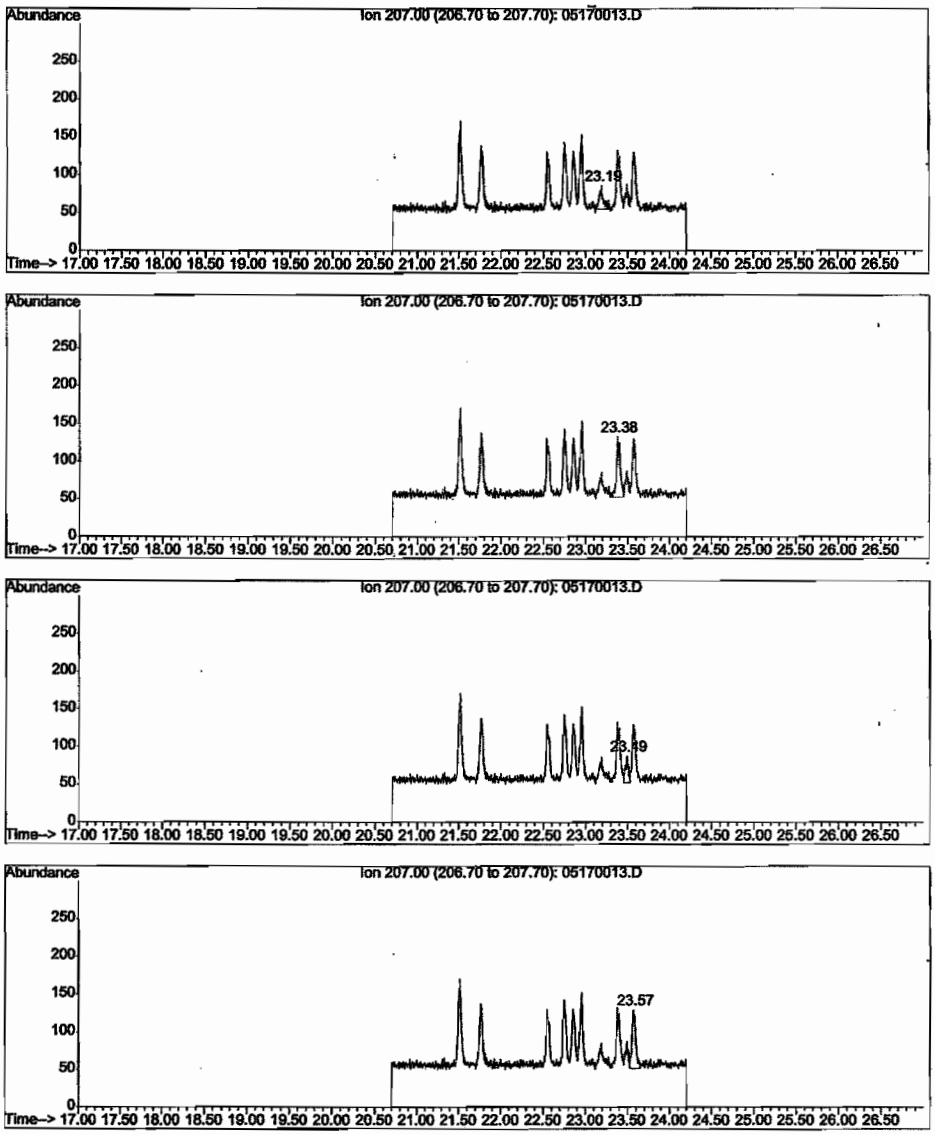
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cyfluthrin
1.0 ng/mL
Peak Response (area): 8943

Figure 17. (con't) Bracketing Standards

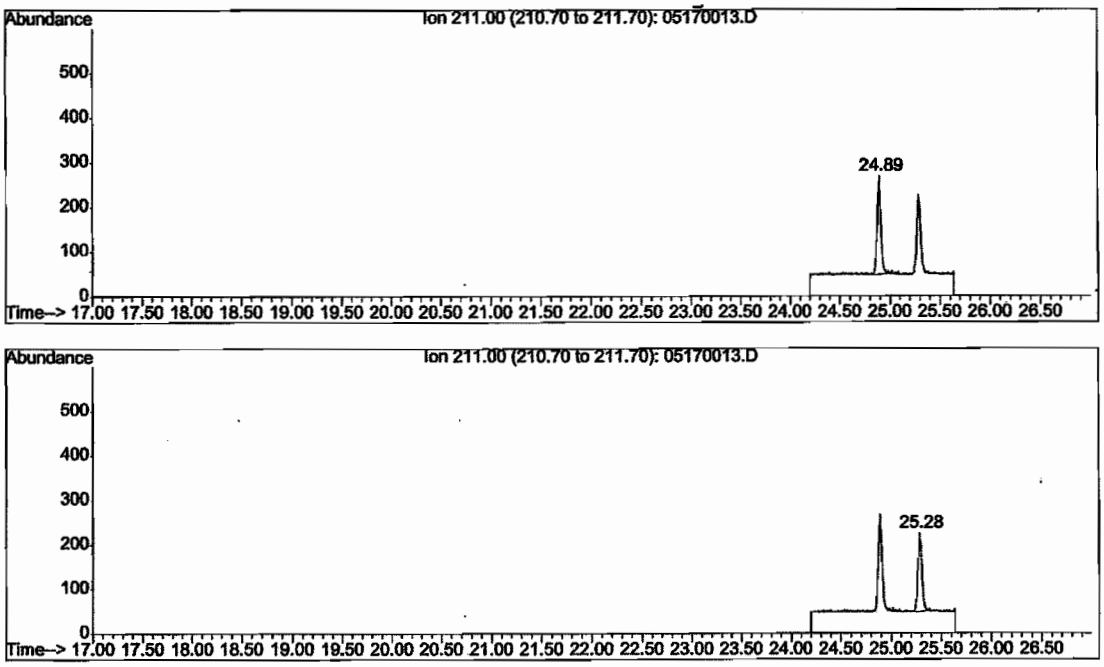
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cypermethrin
1.0 ng/mL
Peak Response (area): 6684

Figure 17. (con't) Bracketing Standards

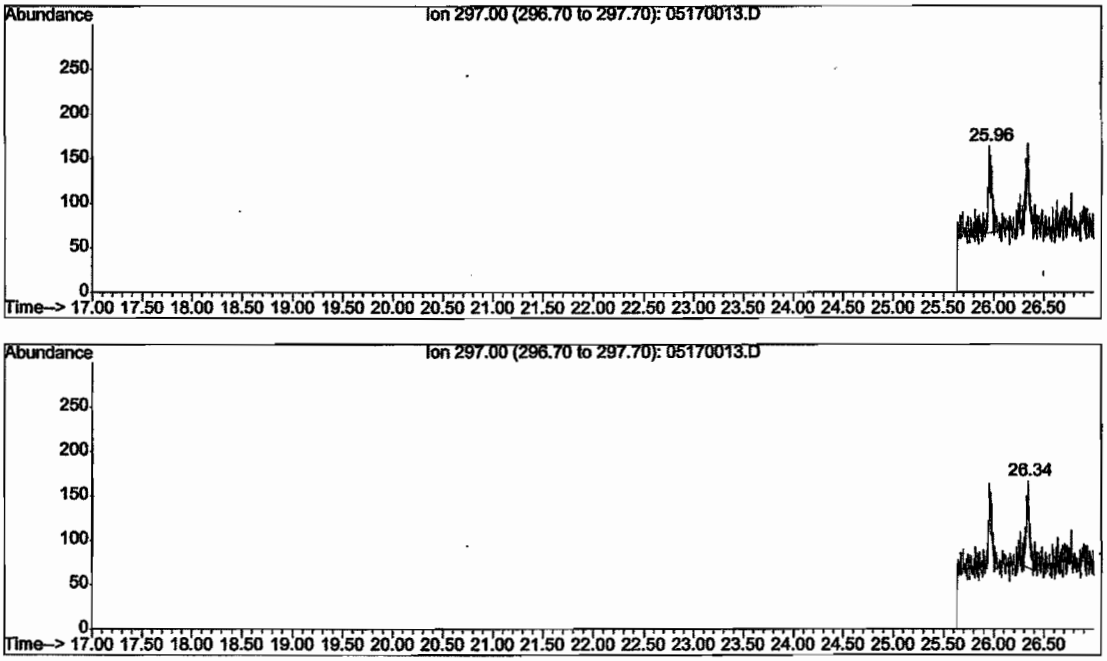
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Esfenvalerate
1.0 ng/mL
Peak Response (area): 10532

Figure 17. (con't) Bracketing Standards

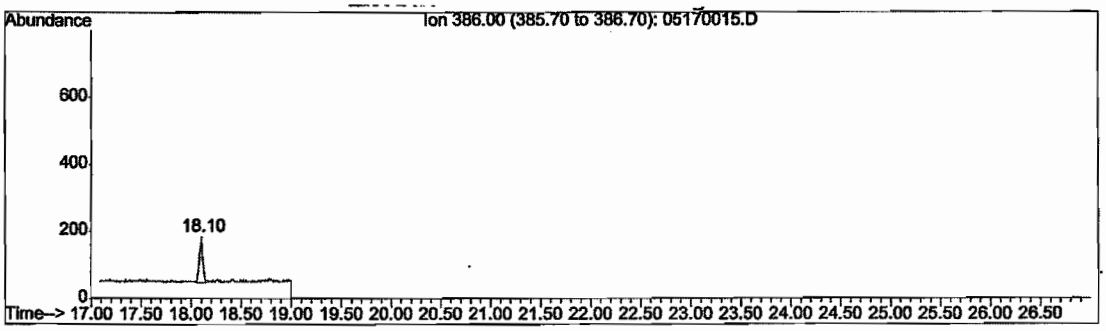
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



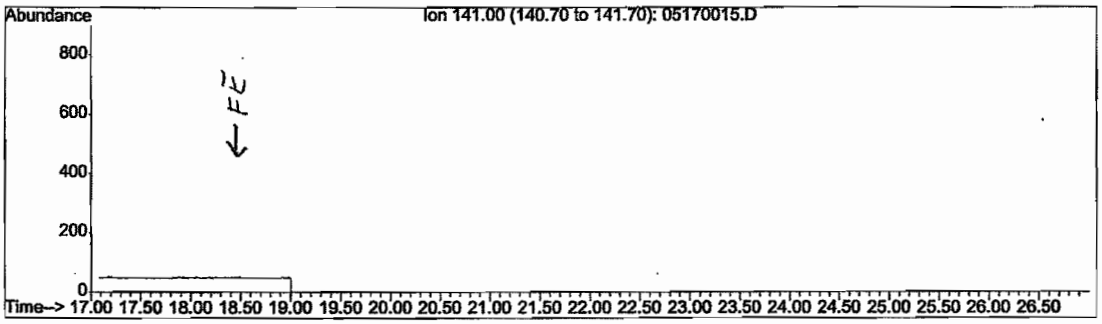
Deltamethrin
1.0 ng/mL
Peak Response (area): 4210

Figure 18. Estuarine Sediment, Untreated Control

Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL.



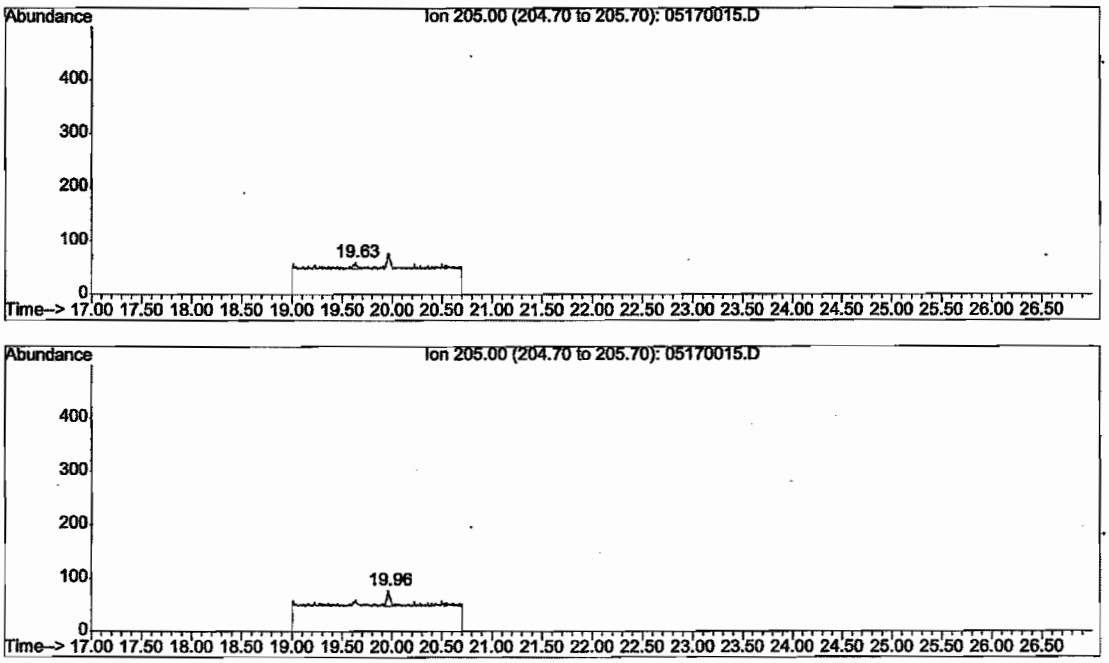
Bifenthrin
Peak Response (area): 3071
Bifenthrin Reported: <0.1 µg/kg



Fenpropathrin
Peak Response (area): 0
Fenpropathrin Reported: None Detected

Figure 18. (con't) **Estuarine Sediment, Untreated Control**

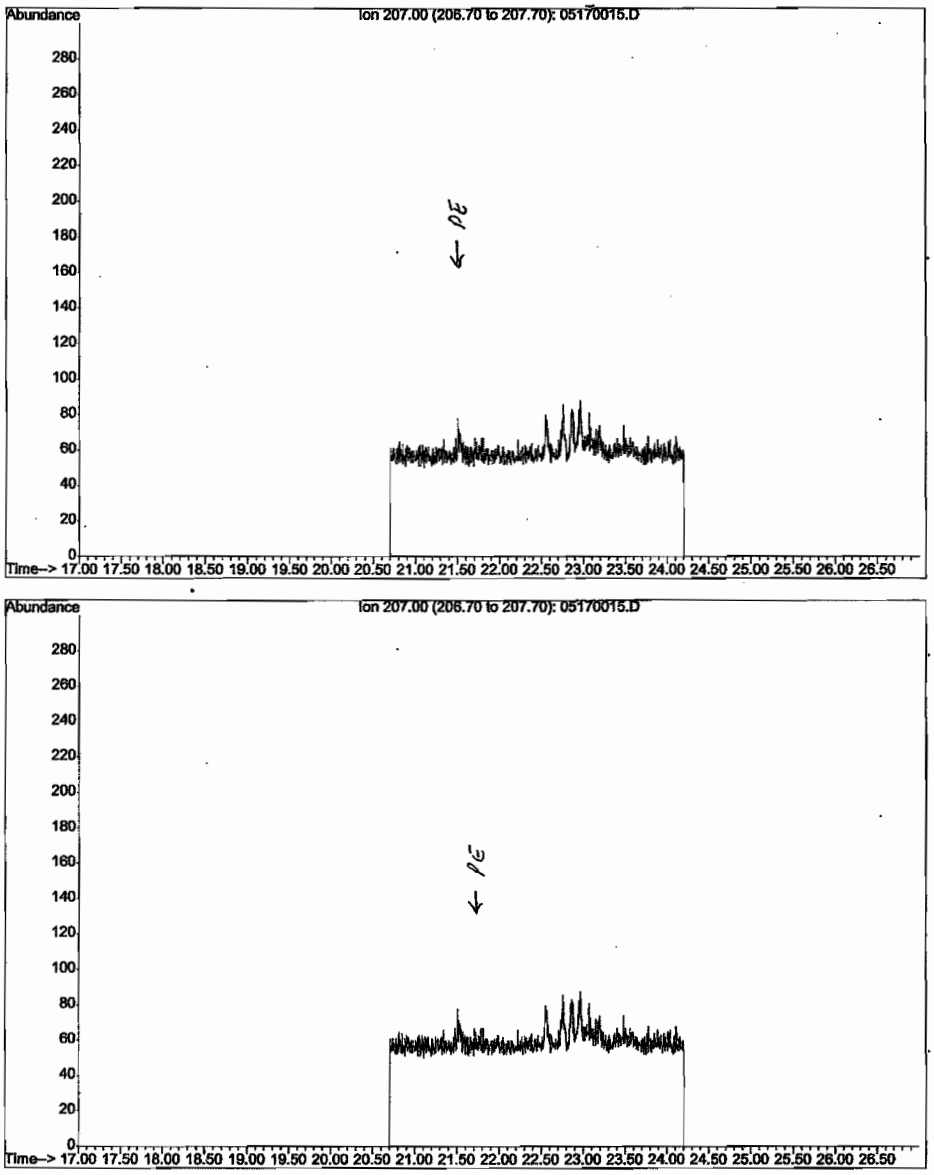
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL.



Lambda-cyhalothrin
Peak Response (area): 920
Lambda-cyhalothrin Reported: <0.1 µg/kg

Figure 18. (con't) **Estuarine Sediment, Untreated Control**

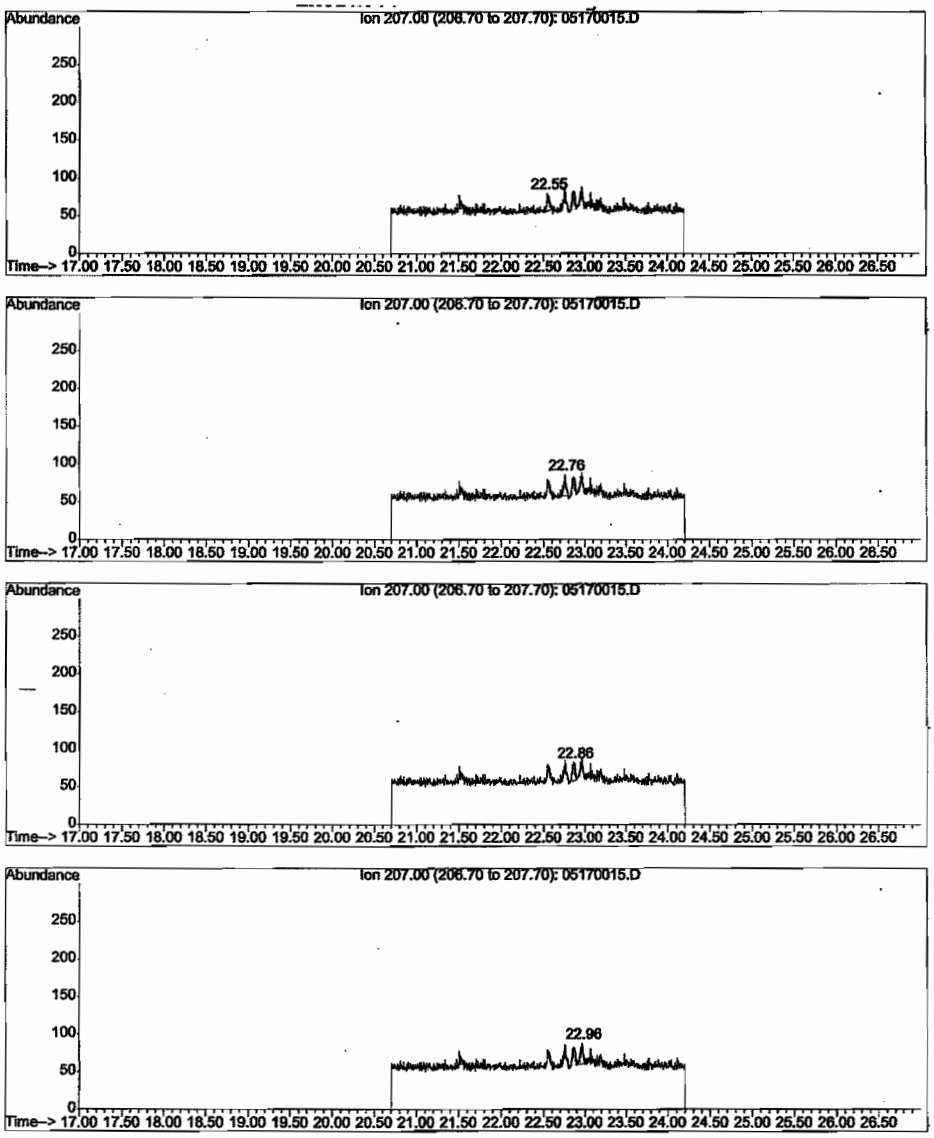
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL.



Permethrin
Peak Response (area): 0
Permethrin Reported: None Detected

Figure 18. (con't) **Estuarine Sediment, Untreated Control**

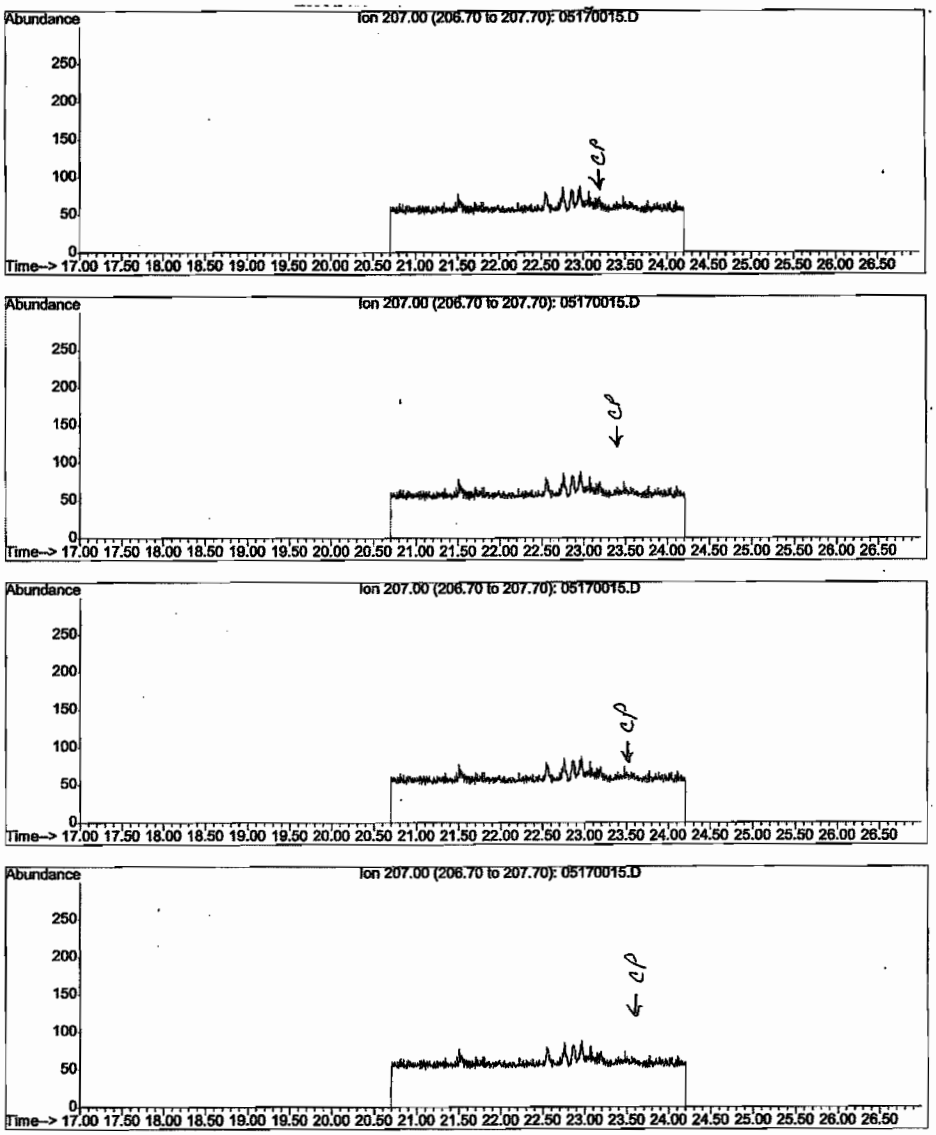
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL.



Cyfluthrin
Peak Response (area): 2029
Cyfluthrin Reported: <0.1 µg/kg

Figure 18. (con't) **Estuarine Sediment, Untreated Control**

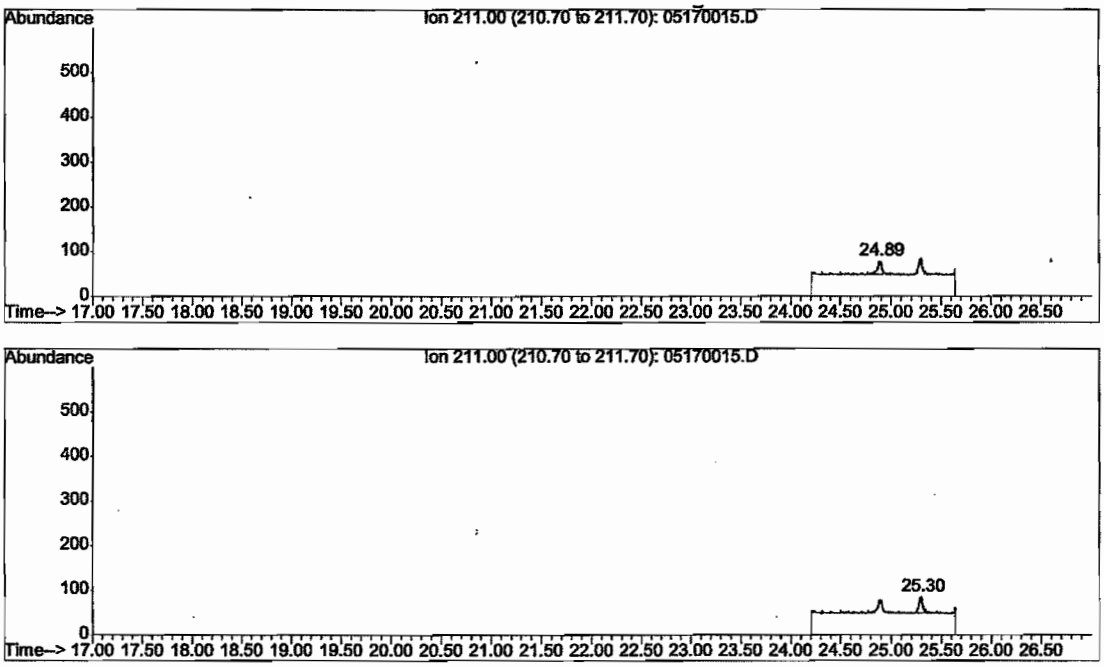
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL.



Cypermethrin
Peak Response (area): 0
Cypermethrin Reported: None Detected

Figure 18. (con't) **Estuarine Sediment, Untreated Control**

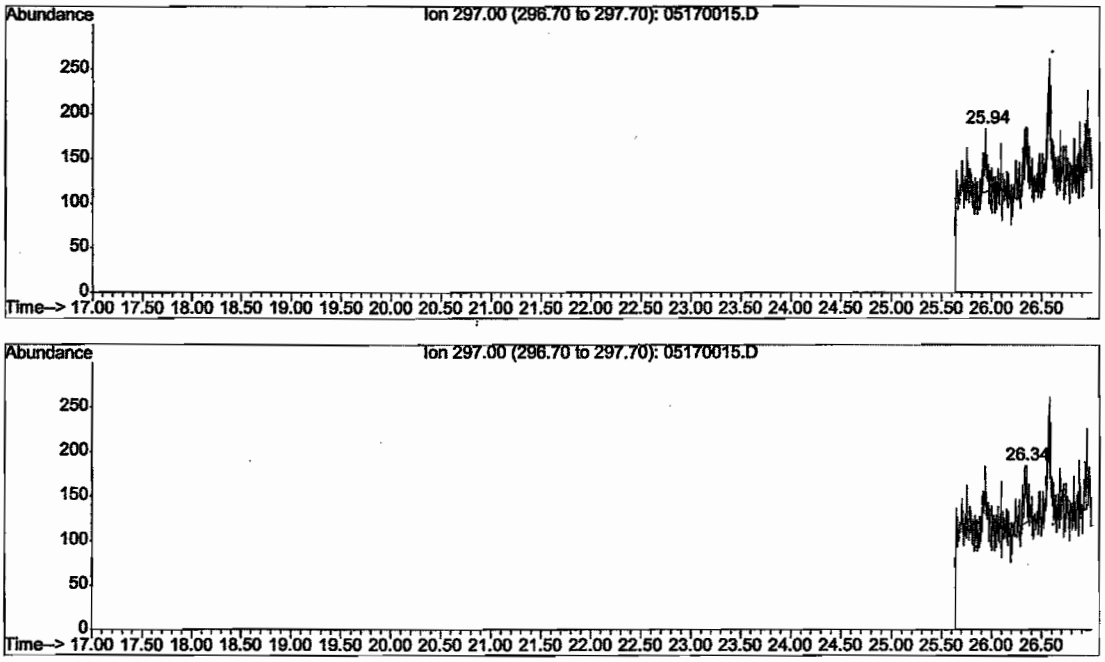
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL.



Esfenvalerate
Peak Response (area): 1671
Esfenvalerate Reported: <0.1 µg/kg

Figure 18. (con't) **Estuarine Sediment, Untreated Control**

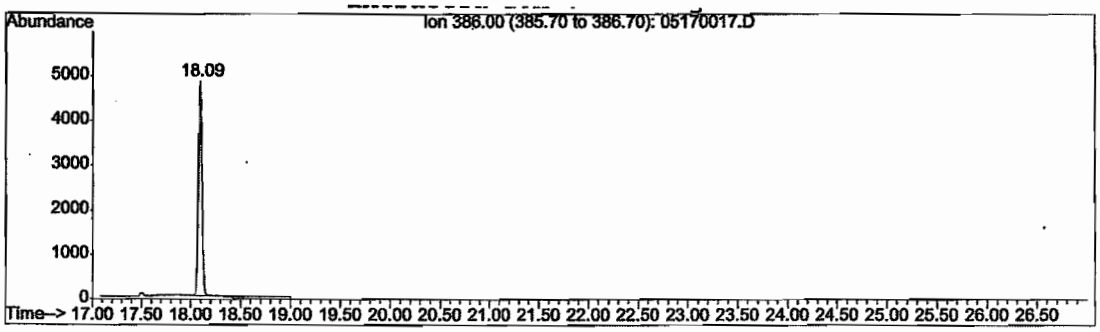
Untreated Control, Estuarine Sediment, Paradise Cove, Control 5, 10.0 g/mL.



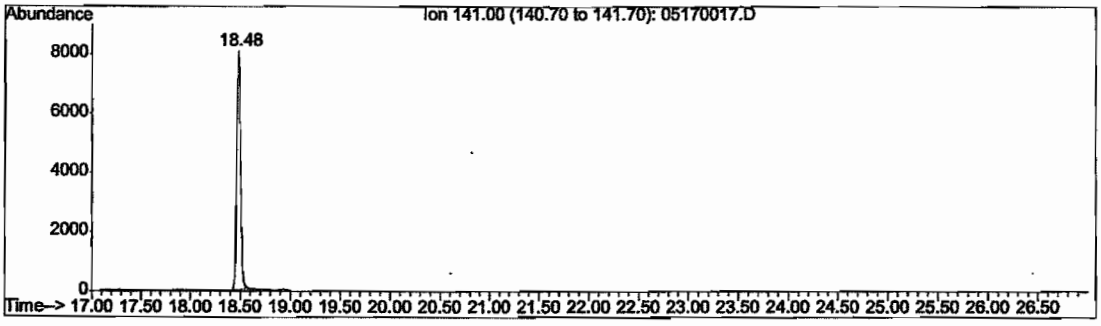
Deltamethrin
Peak Response (area): 2717
Deltamethrin Reported: <0.1 µg/kg

Figure 19. Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



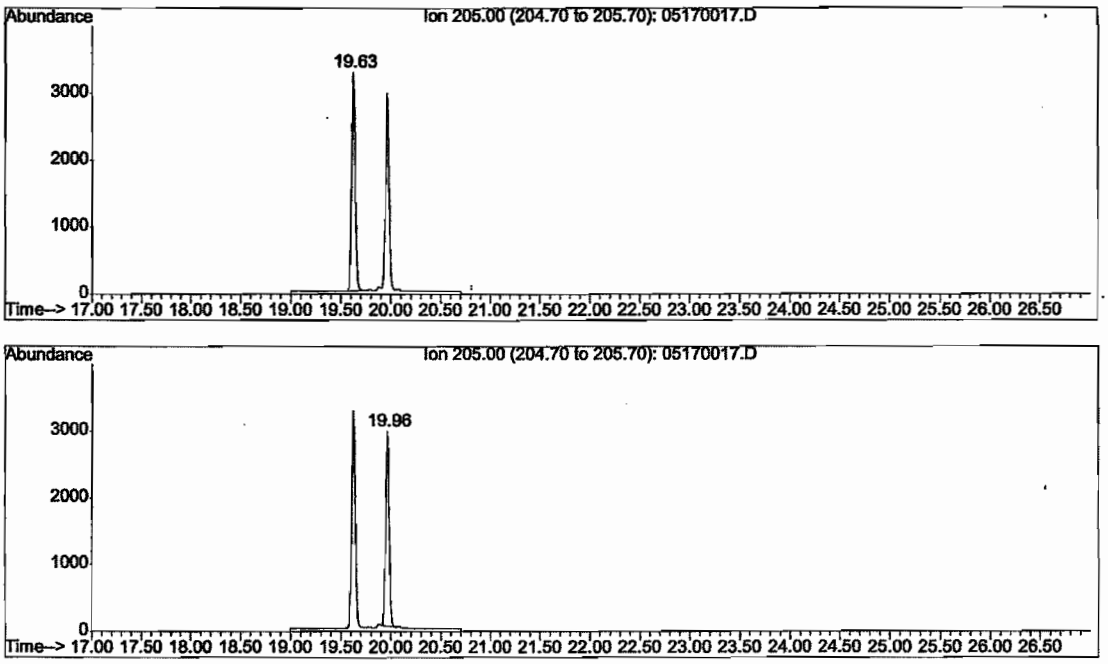
Bifenthrin
20 ng/mL
Peak Response (area): 121315



Fenpropathrin
20 ng/mL
Peak Response (area): 216355

Figure 19. (con't) Bracketing Standards

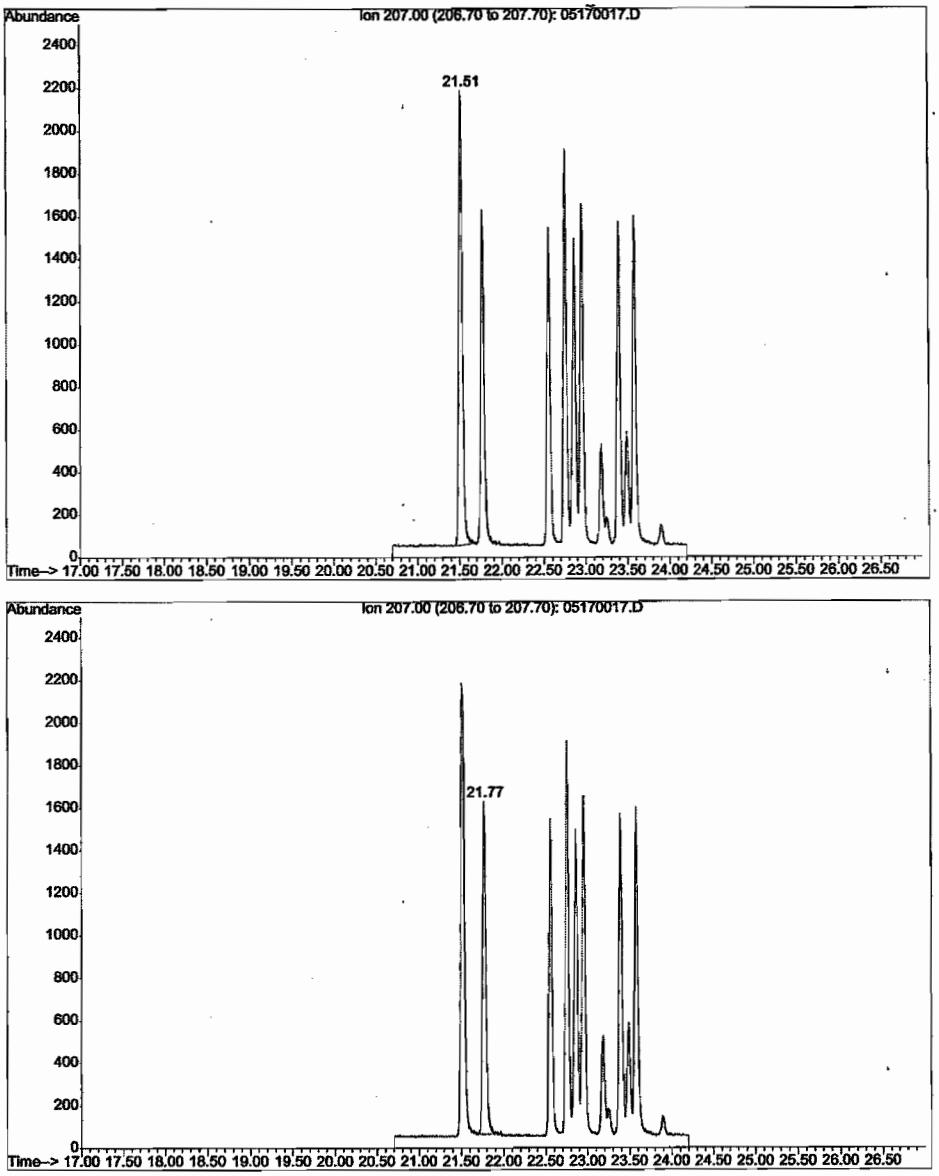
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 154964

Figure 19. (con't) Bracketing Standards

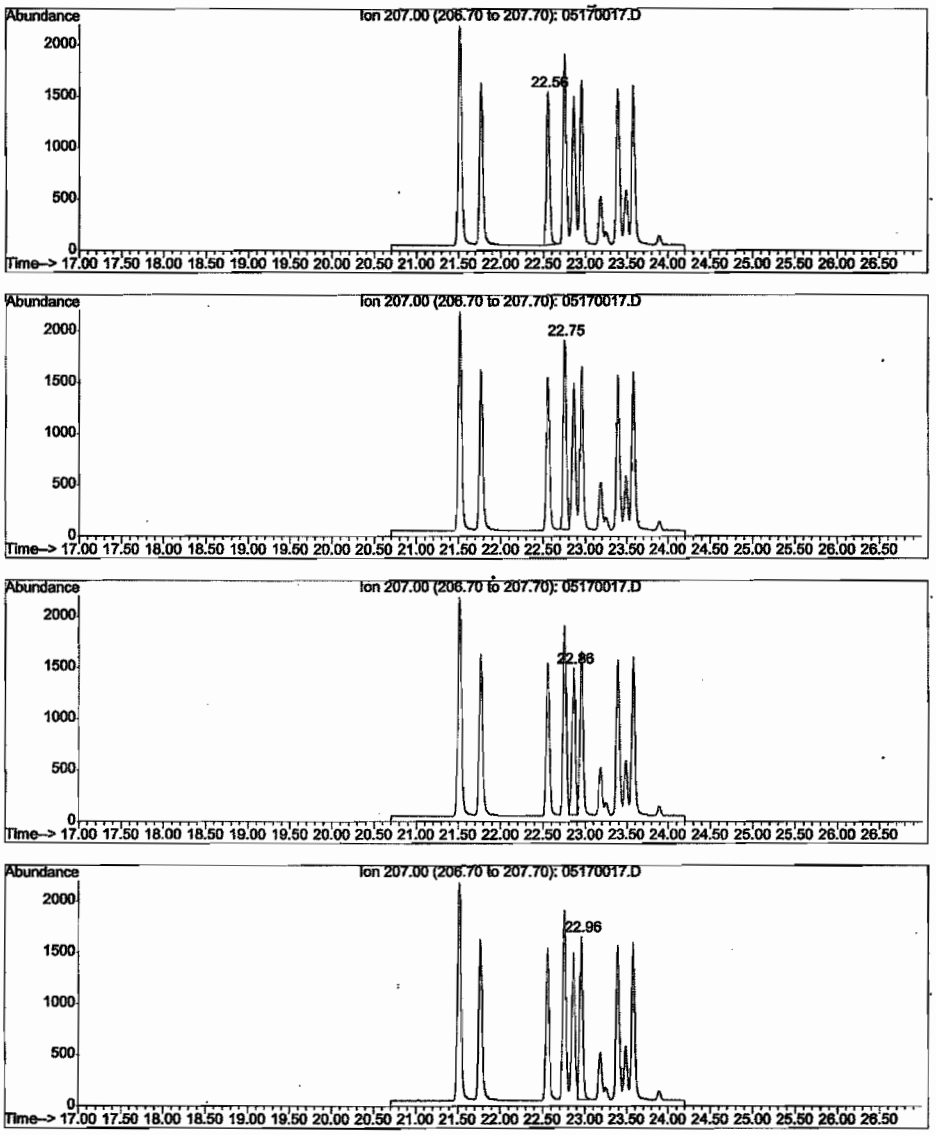
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Permethrin
200 ng/mL
Peak Response (area): 105832

Figure 19. (con't) **Bracketing Standards**

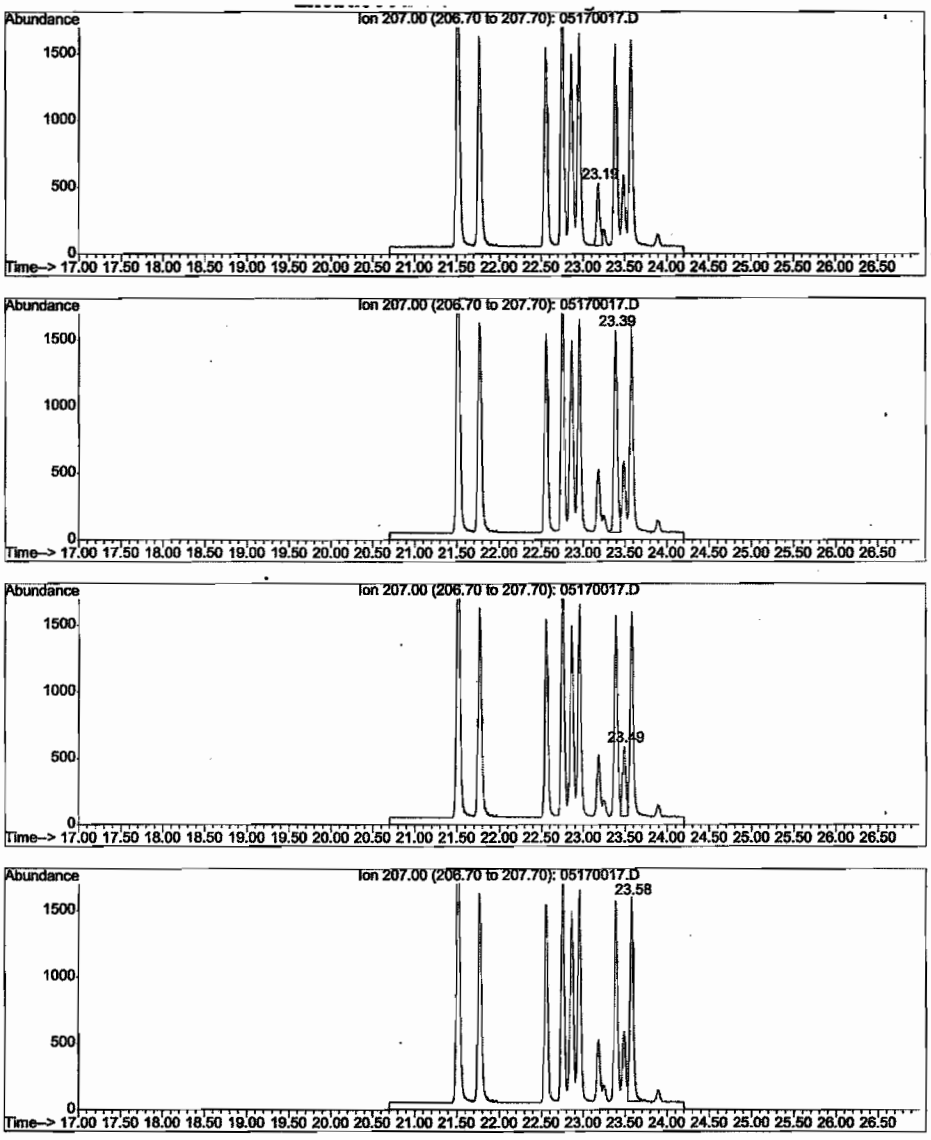
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cyfluthrin
20 ng/mL
Peak Response (area): 168645

Figure 19. (con't) Bracketing Standards

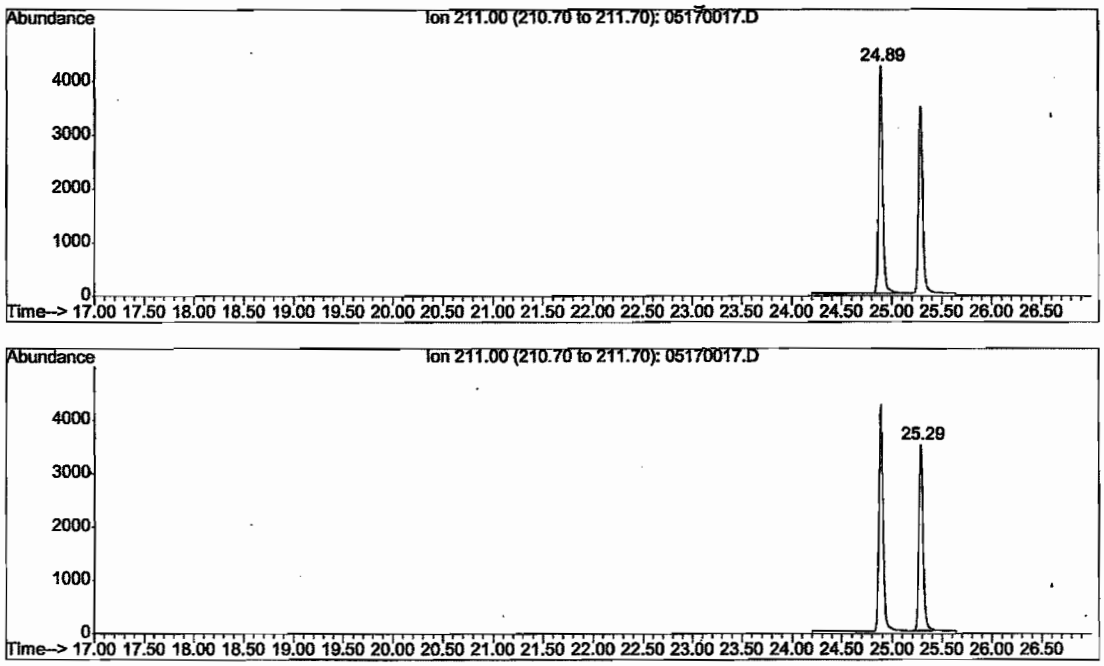
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cypermethrin
20 ng/mL
Peak Response (area): 111862

Figure 19. (con't) **Bracketing Standards**

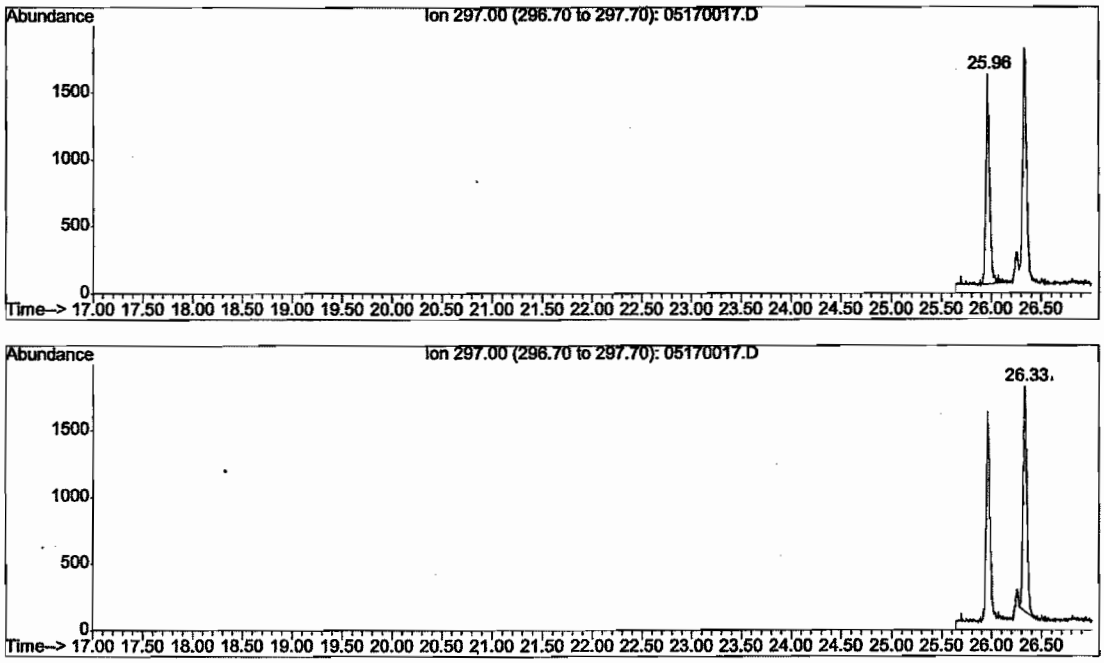
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Esfenvalerate
20 ng/mL
Peak Response (area): 221658

Figure 19. (con't) **Bracketing Standards**

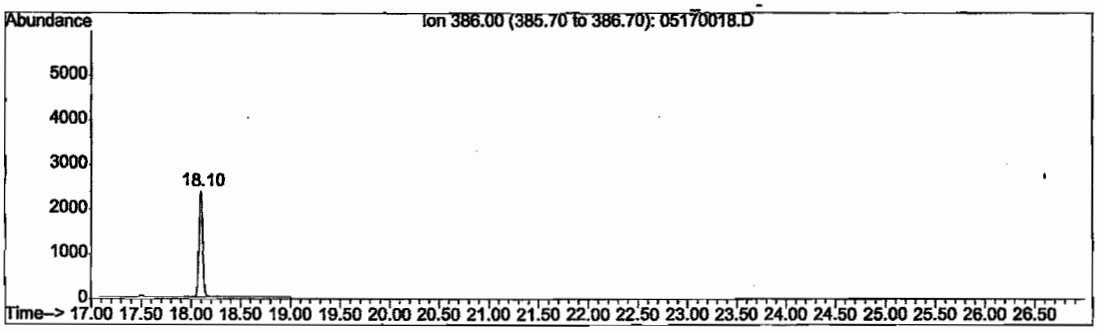
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



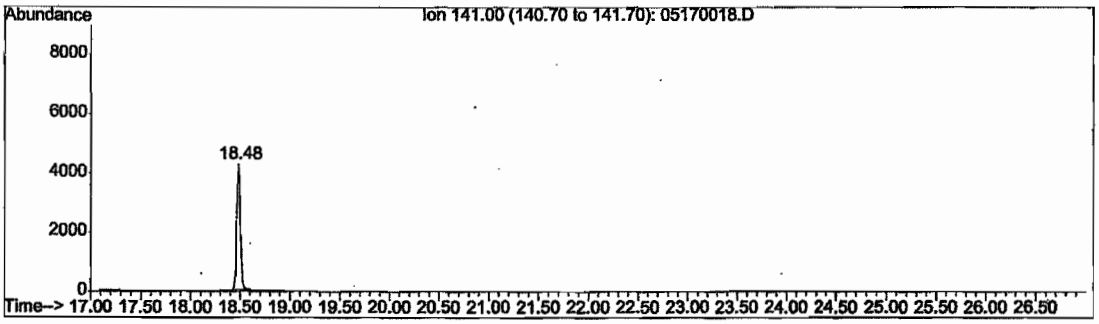
Deltamethrin
20 ng/mL
Peak Response (area): 85238

Figure 20. Estuarine Sediment, Fortified Control (10×LOQ)

Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



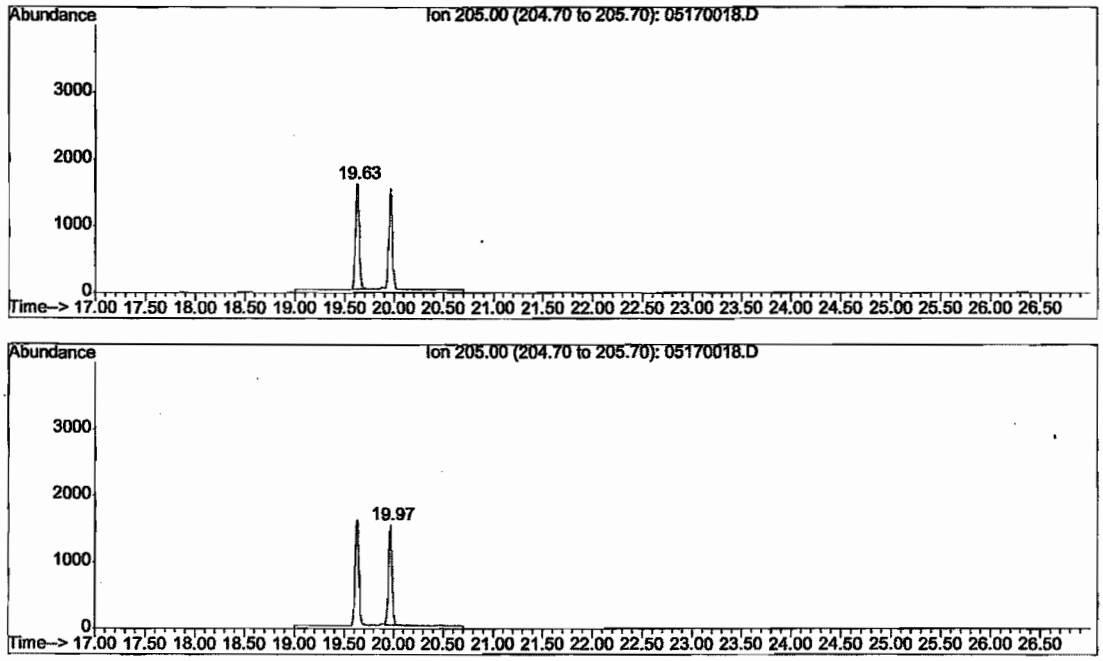
Bifenthrin
Peak Response (area): 60105
Bifenthrin Reported Recovery: 93%



Fenpropathrin
Peak Response (area): 108530
Fenpropathrin Reported Recovery: 98%

Figure 20. (con't) **Estuarine Sediment, Fortified Control (10×LOQ)**

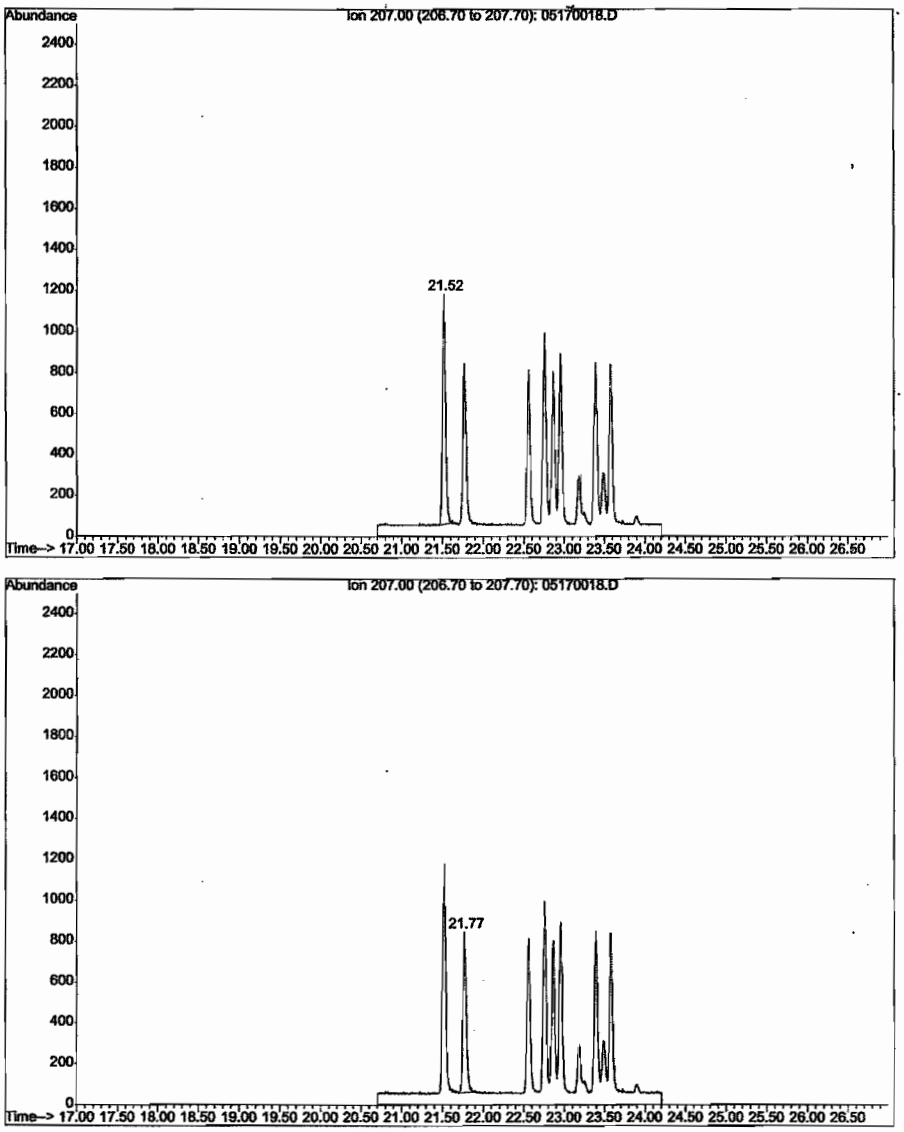
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Lambda-cyhalothrin
Peak Response (area): 77659
Lambda-cyhalothrin Reported Recovery: 97%

Figure 20. (con't) Estuarine Sediment, Fortified Control (10×LOQ)

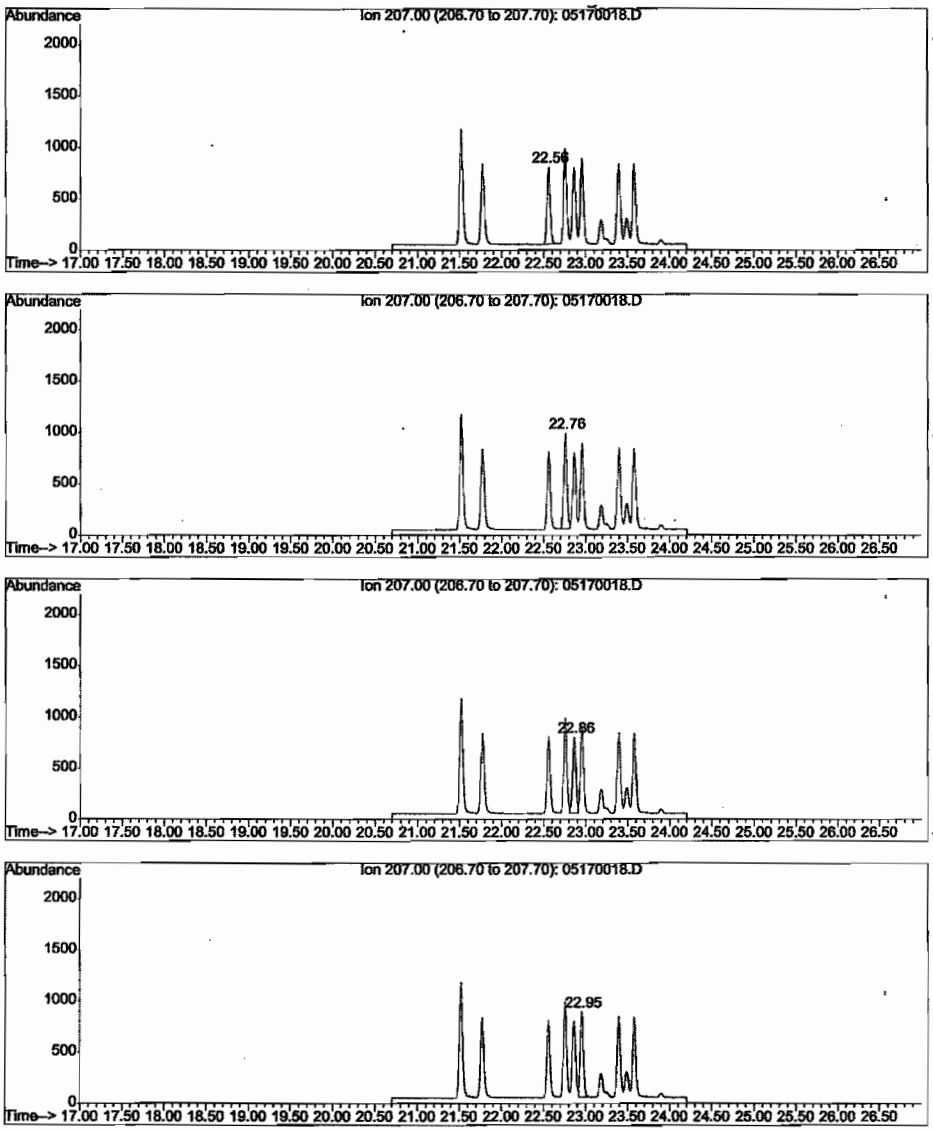
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Permethrin
Peak Response (area): 51714
Permethrin Reported Recovery: 96%

Figure 20. (con't) **Estuarine Sediment, Fortified Control (10×LOQ)**

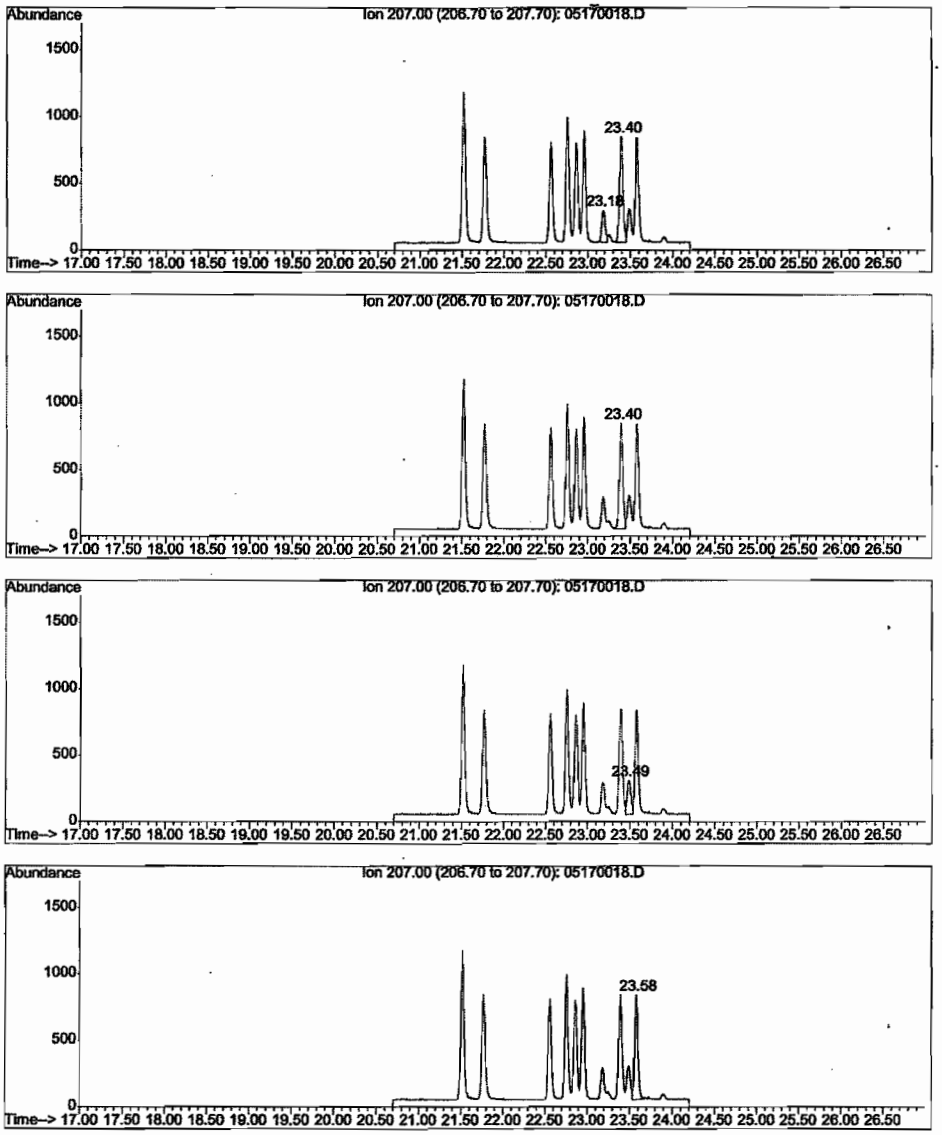
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Cyfluthrin
Peak Response (area): 85979
Cyfluthrin Reported Recovery: 98%

Figure 20. (con't) **Estuarine Sediment, Fortified Control (10×LOQ)**

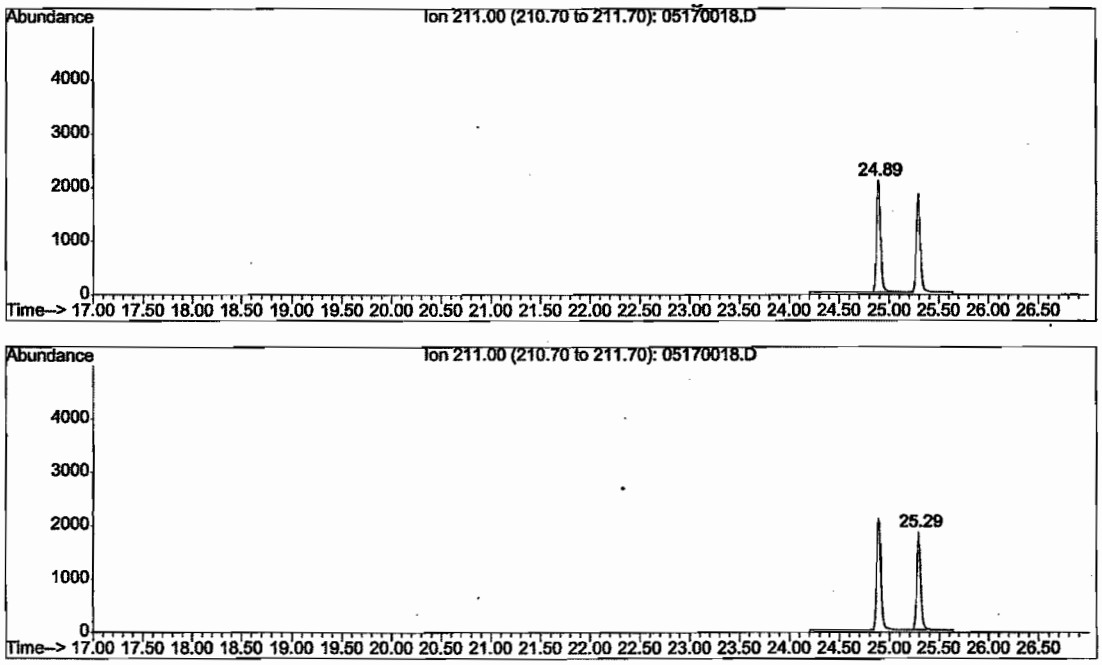
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Cypermethrin
Peak Response (area): 58299
Cypermethrin Reported Recovery: 102%

Figure 20. (con't) **Estuarine Sediment, Fortified Control (10×LOQ)**

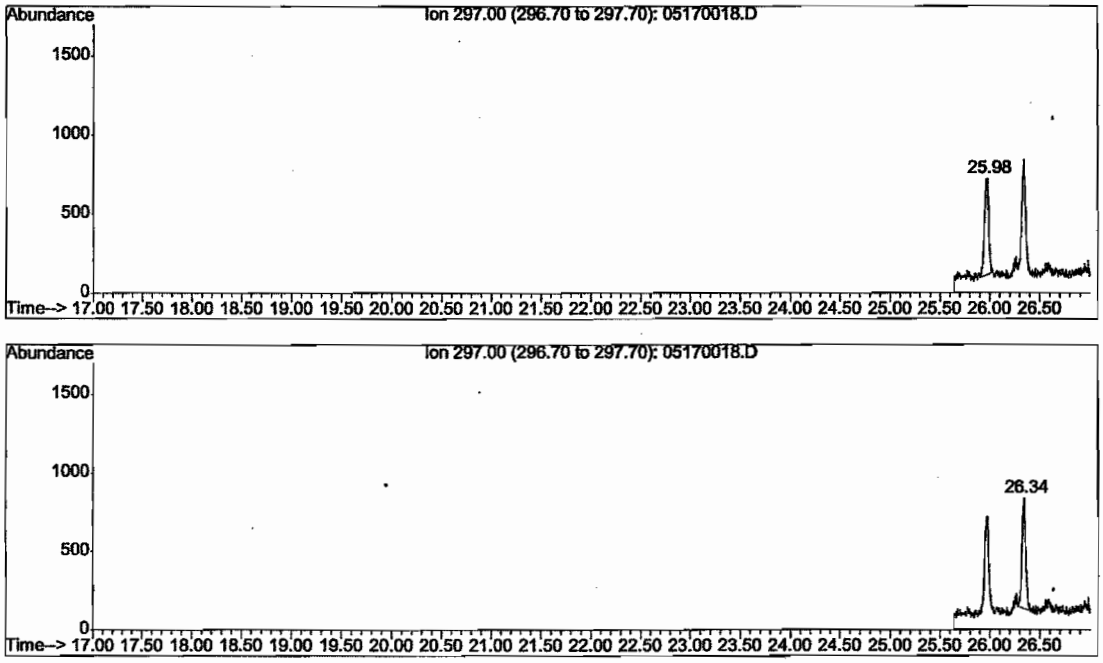
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



Esfenvalerate
Peak Response (area): 110281
Esfenvalerate Reported Recovery: 95%

Figure 20. (con't) **Estuarine Sediment, Fortified Control (10×LOQ)**

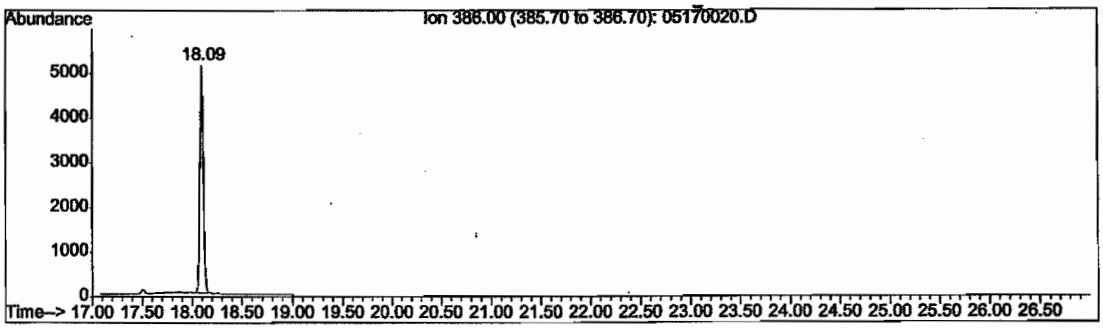
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 26 @ 1.0 µg/kg for all analytes except Permethrin @ 10.0 µg/kg, 10.0 g/mL.



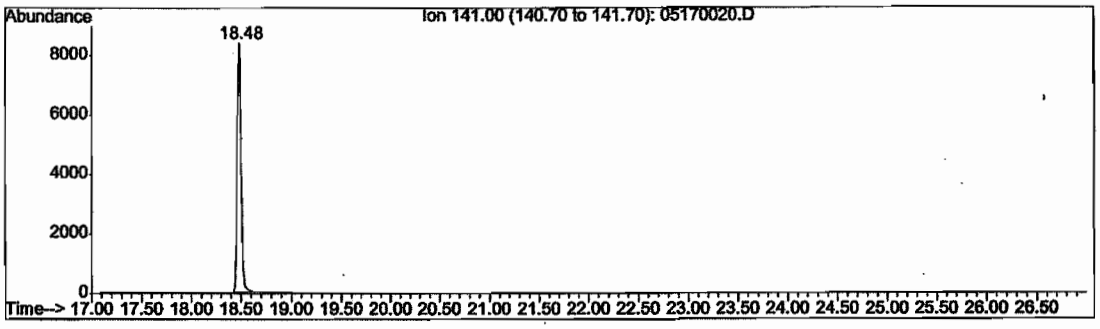
Deltamethrin
Peak Response (area): 35683
Deltamethrin Reported Recovery: 74 %

Figure 21. Bracketing Standards

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



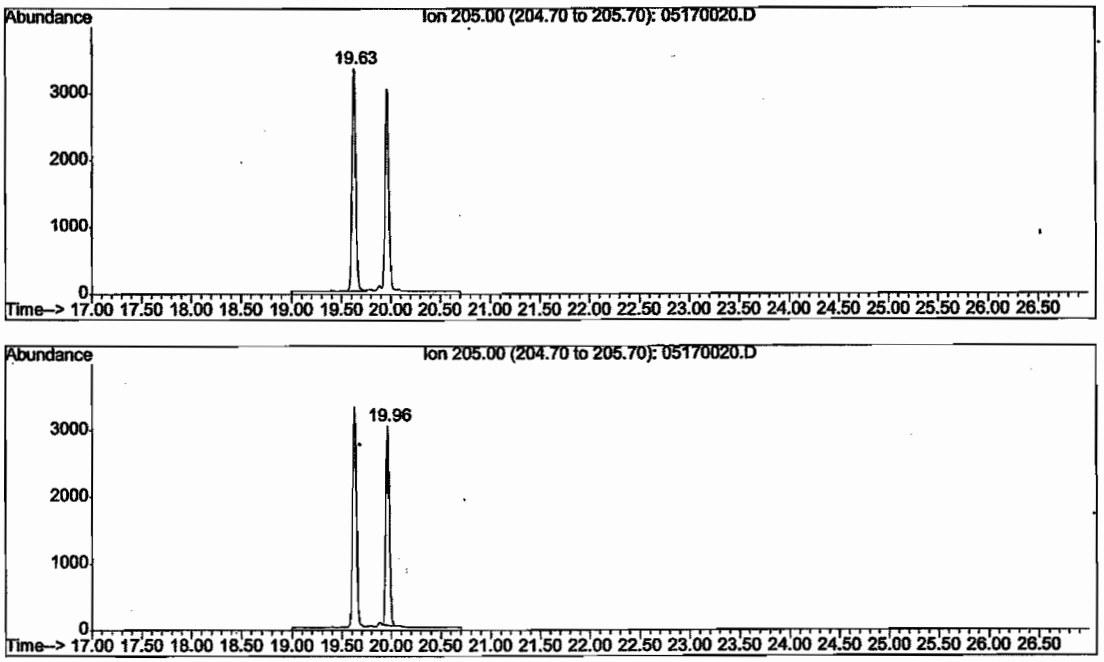
Bifenthrin
20 ng/mL
Peak Response (area): 124239



Fenpropathrin
20 ng/mL
Peak Response (area): 224573

Figure 21. (con't) Bracketing Standards

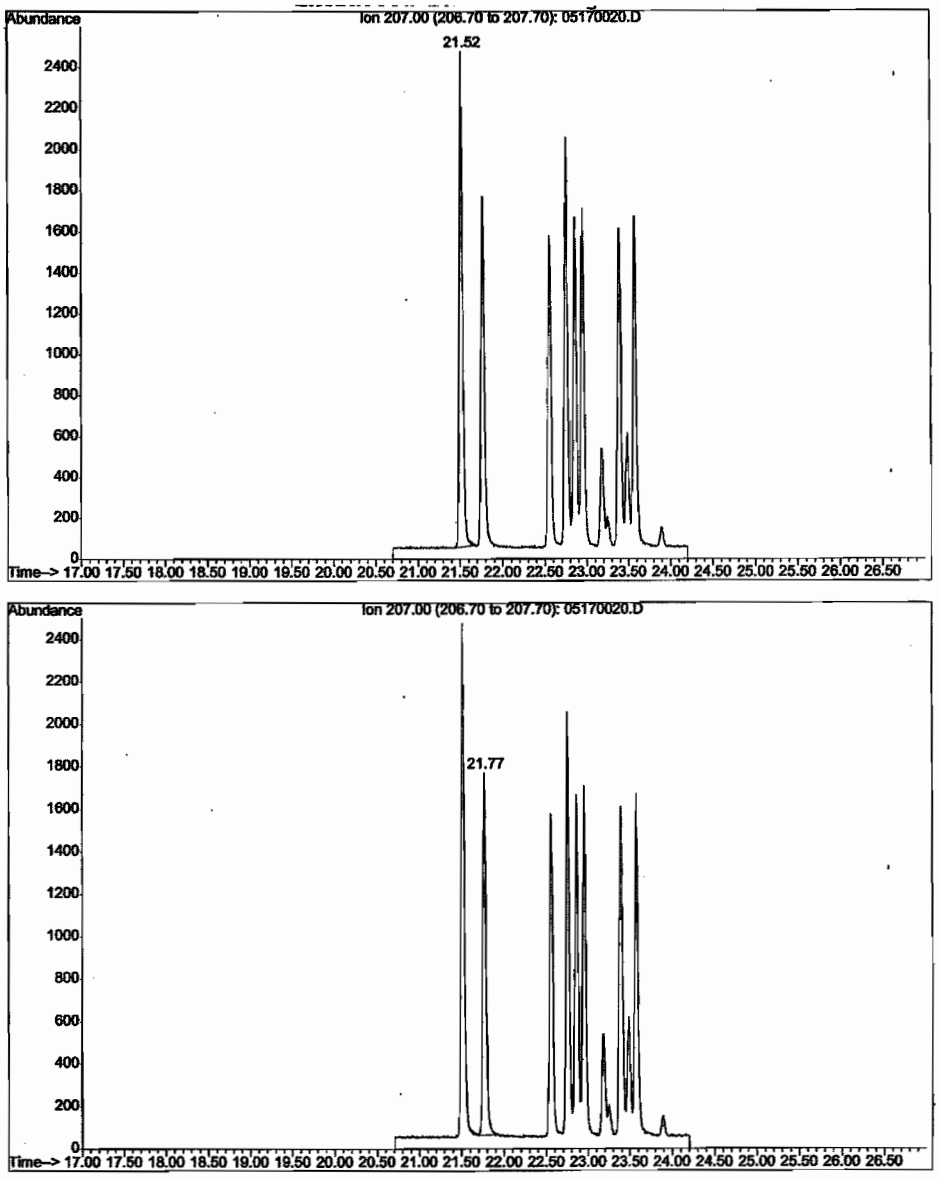
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 162527

Figure 21. (con't) **Bracketing Standards**

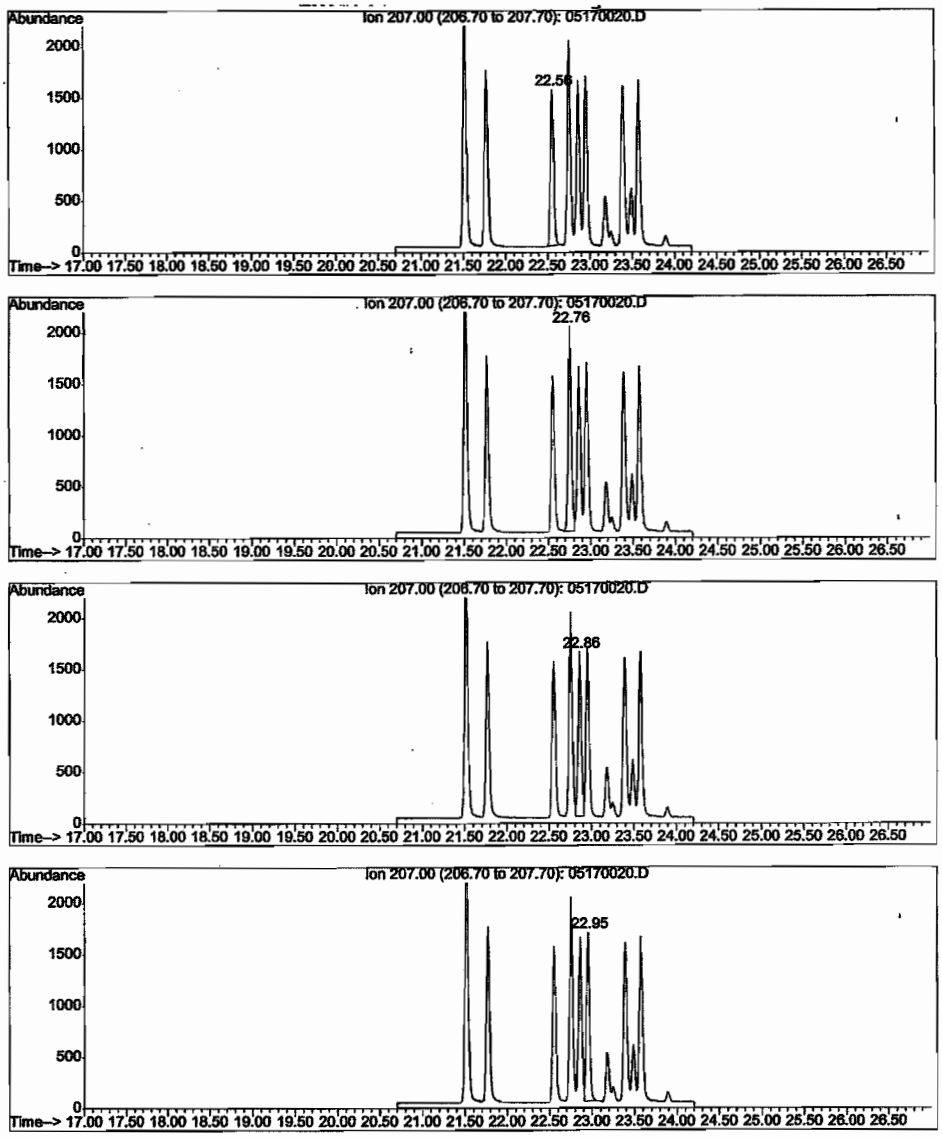
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Permethrin
200 ng/mL
Peak Response (area): 110099

Figure 21. (con't) Bracketing Standards

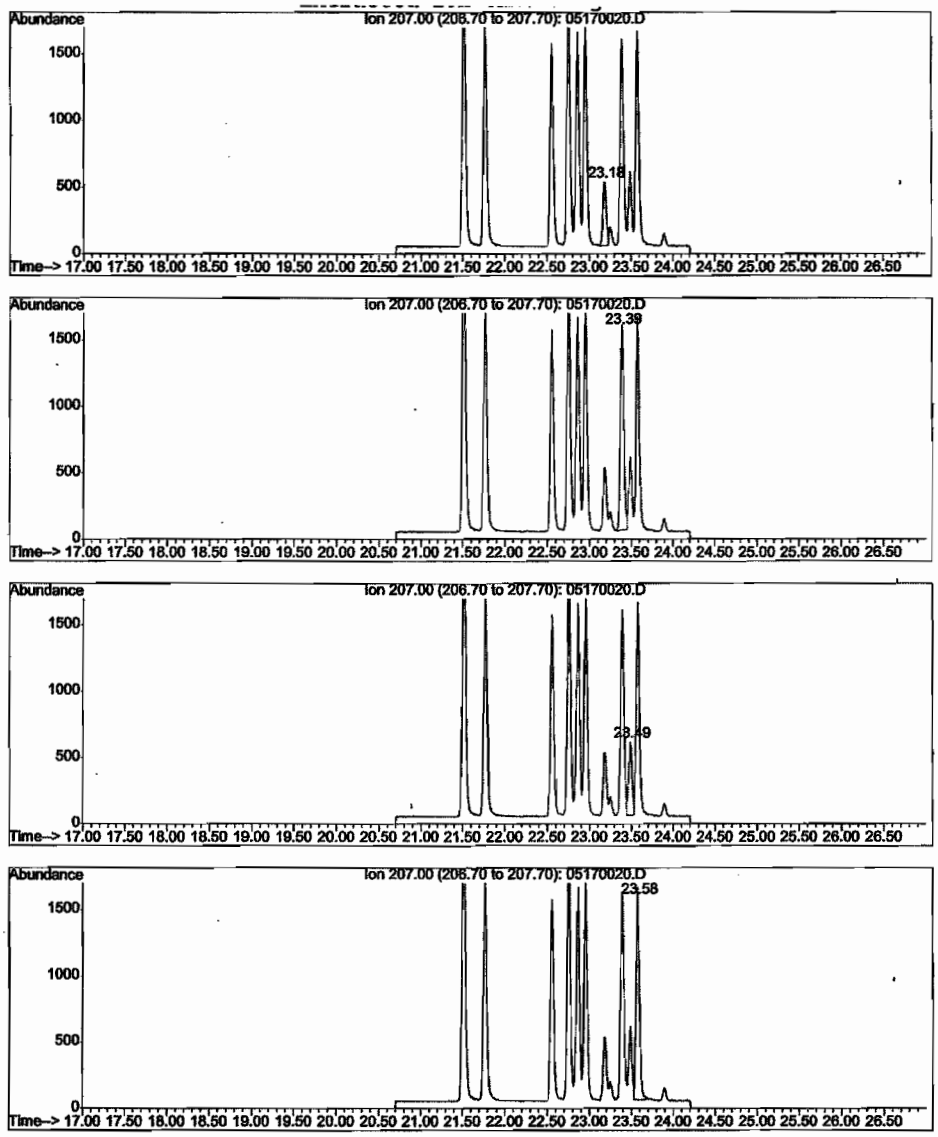
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cyfluthrin
20 ng/mL
Peak Response (area): 178012

Figure 21. (con't) Bracketing Standards

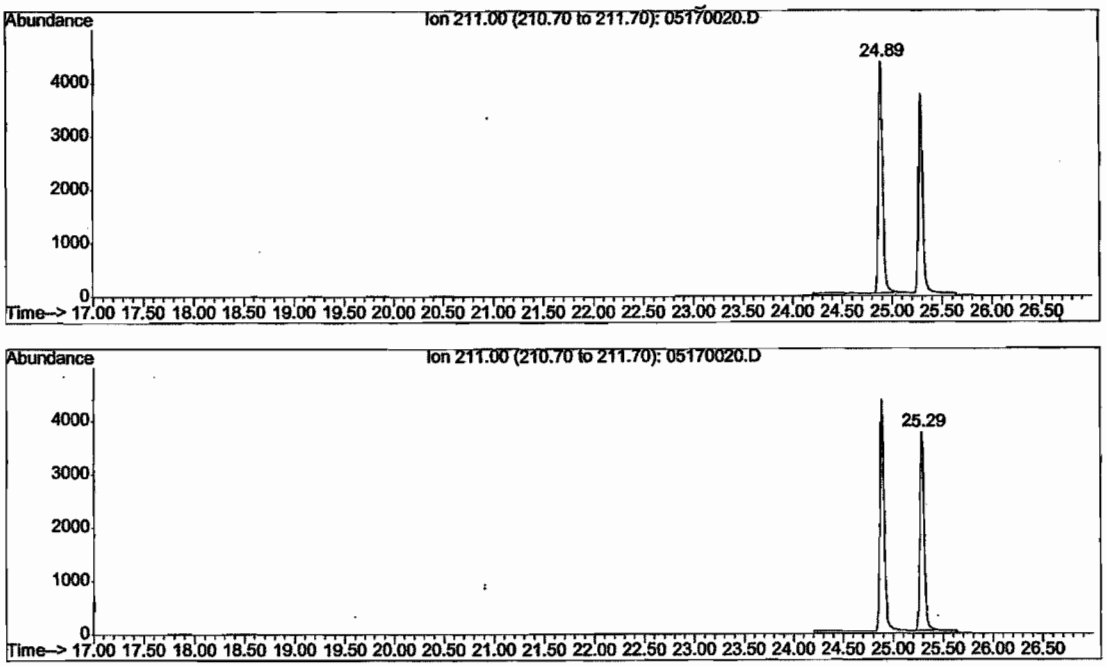
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cypermethrin
20 ng/mL
Peak Response (area): 117395

Figure 21. (con't) Bracketing Standards

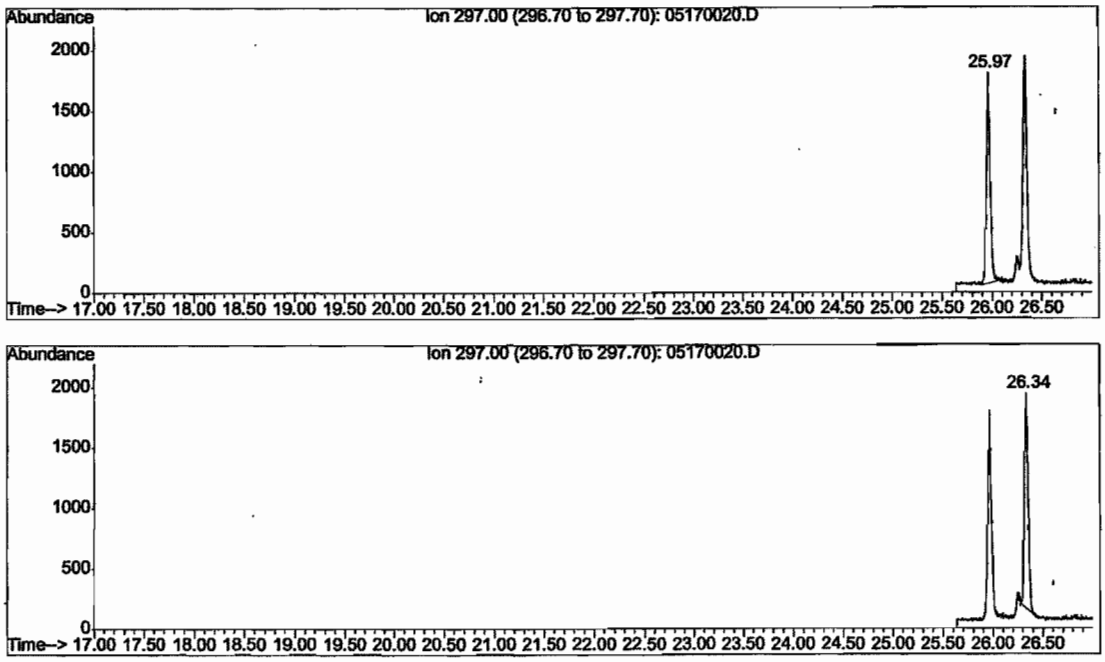
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Esfenvalerate
20 ng/mL
Peak Response (area): 234713

Figure 21. (con't) Bracketing Standards

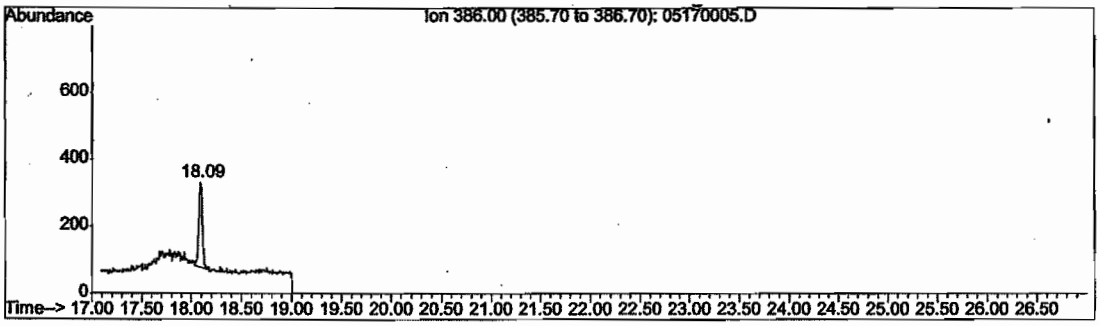
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



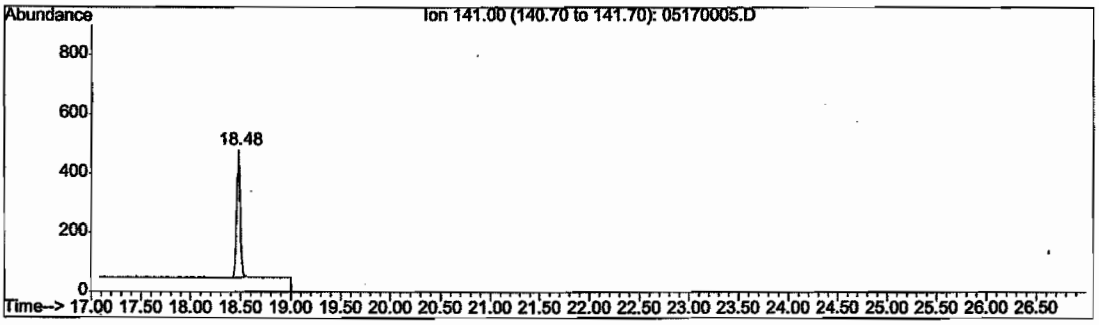
Deltamethrin
20 ng/mL
Peak Response (area): 90585

Figure 22. Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



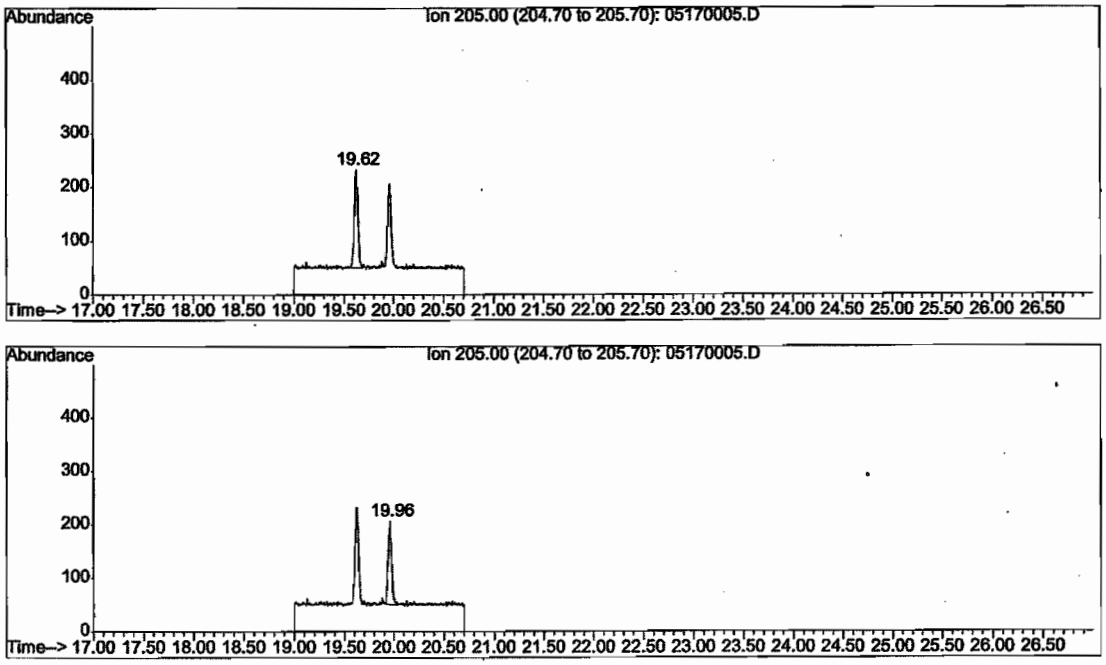
Bifenthrin
1.0 ng/mL
Peak Response (area): 6312



Fenpropathrin
1.0 ng/mL
Peak Response (area): 10783

Figure 22. (con't) Bracketing Standards

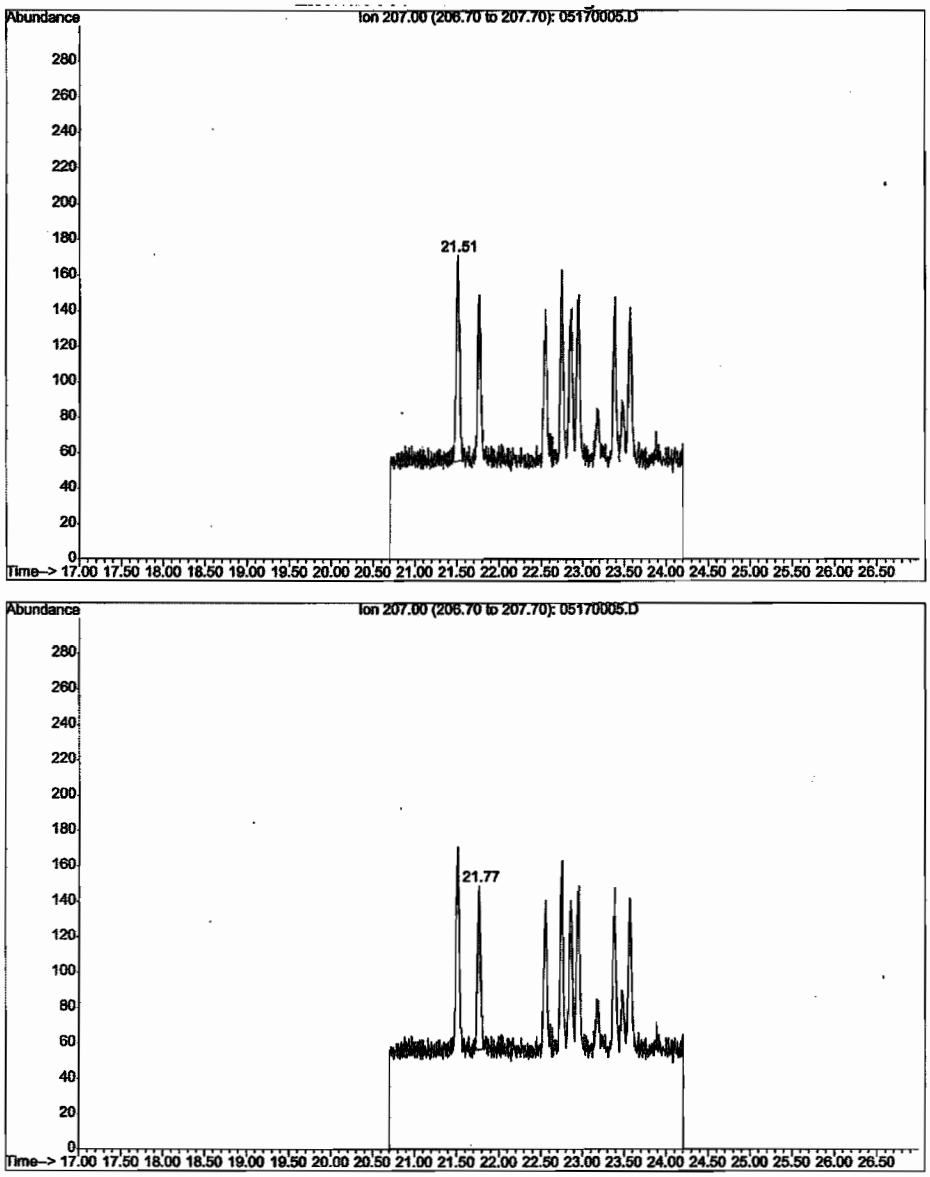
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 8164

Figure 22. (con't) Bracketing Standards

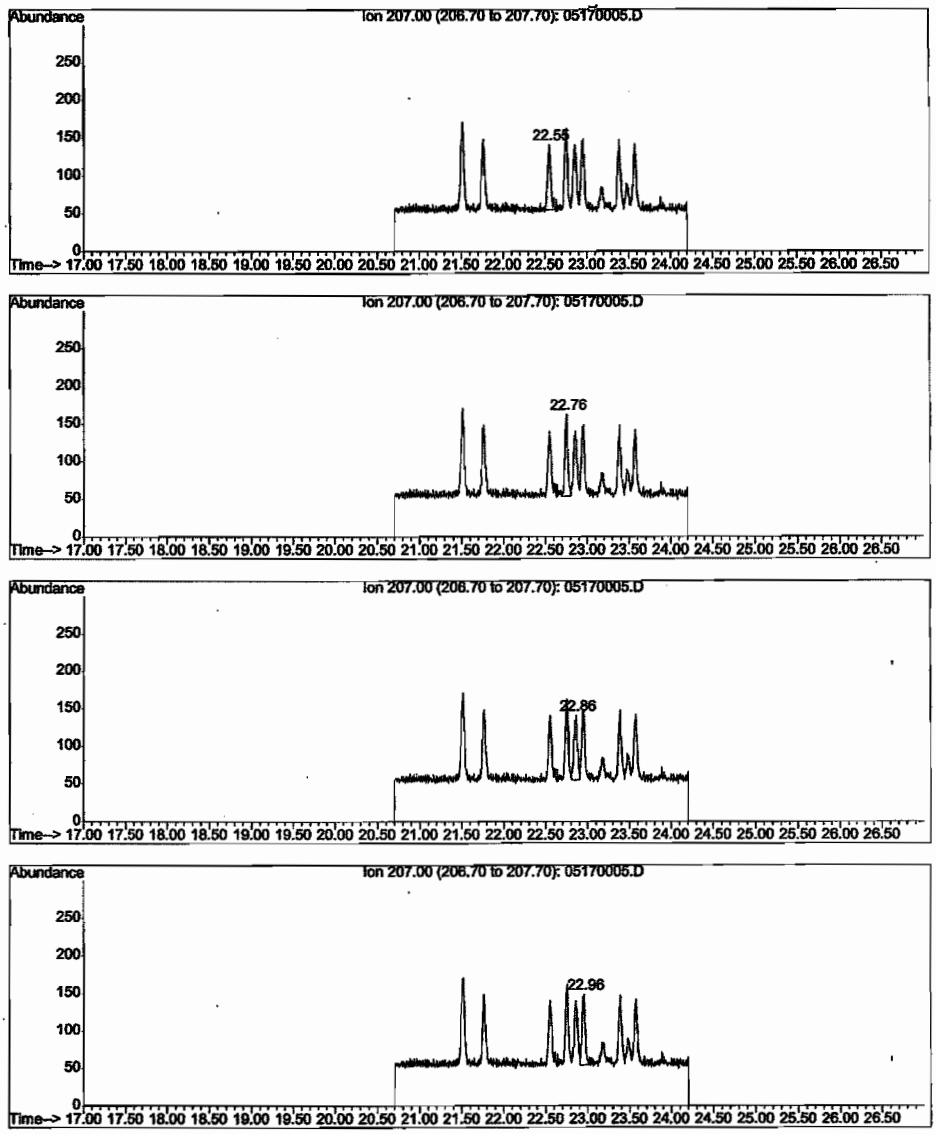
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Permethrin
10 ng/mL
Peak Response (area): 5435

Figure 22. (con't) Bracketing Standards

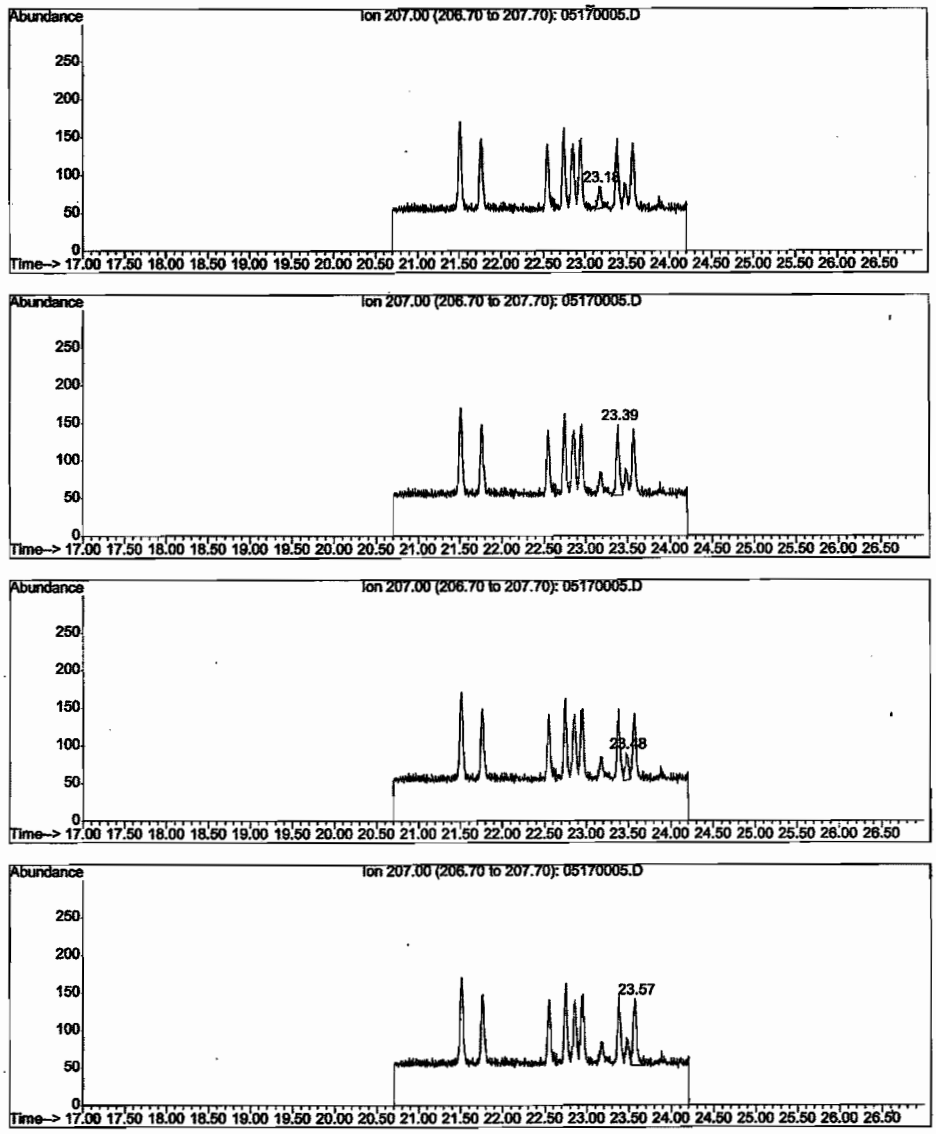
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cyfluthrin
1.0 ng/mL
Peak Response (area): 10000

Figure 22. (con't) Bracketing Standards

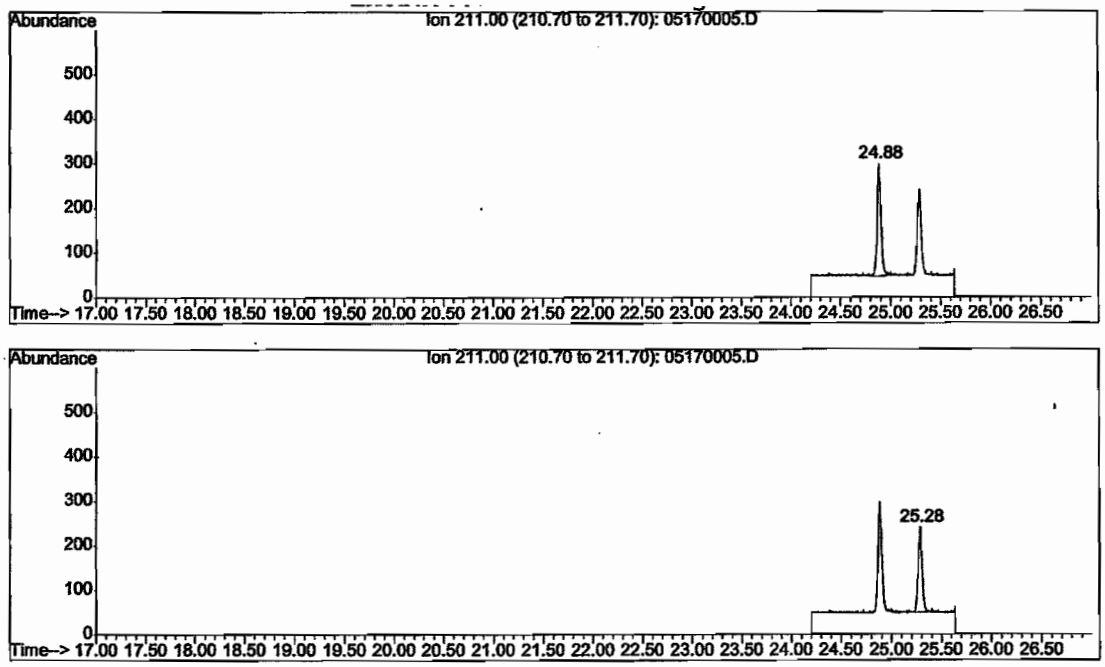
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cypermethrin
1.0 ng/mL
Peak Response (area): 6869

Figure 22. (con't) **Bracketing Standards**

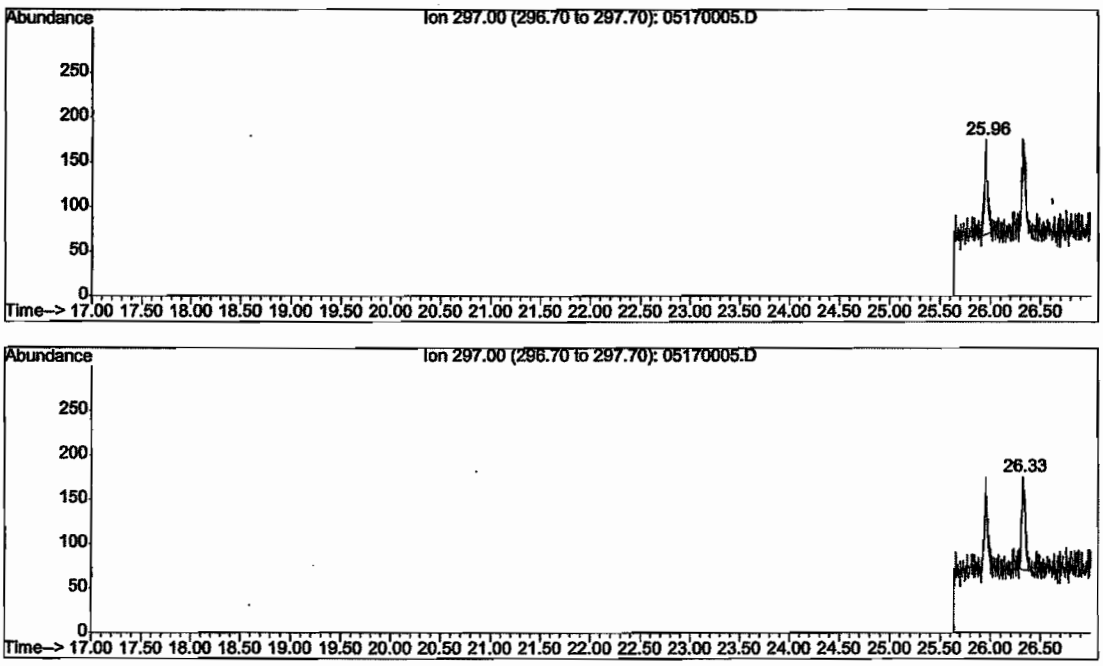
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Esfenvalerate
1.0 ng/mL
Peak Response (area): 12026

Figure 22. (con't) **Bracketing Standards**

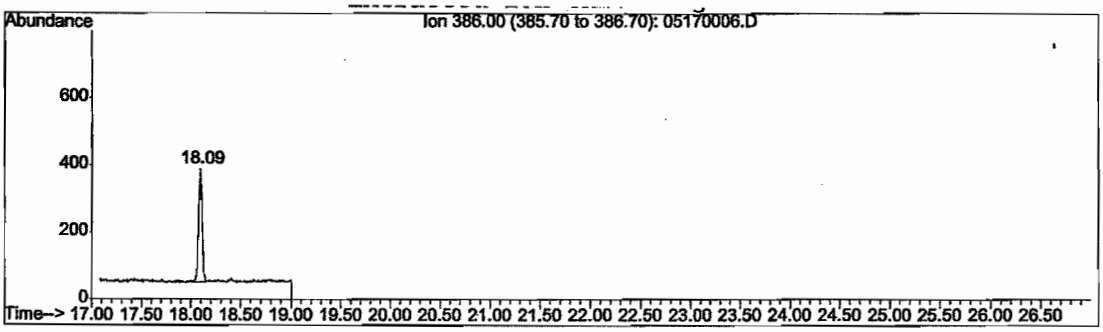
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



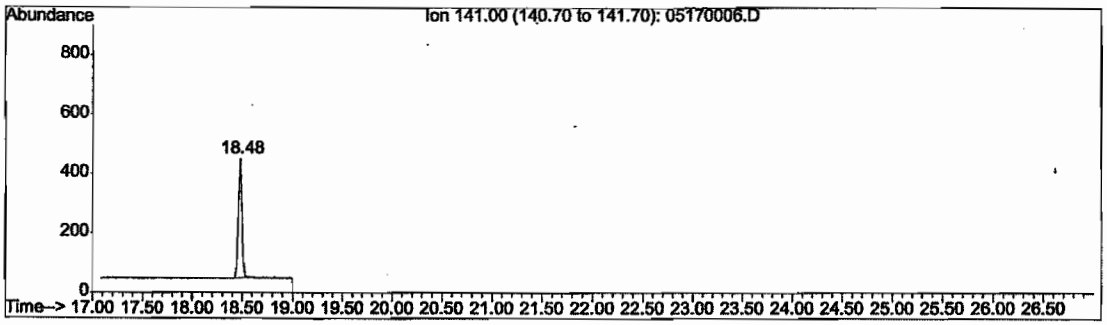
Deltamethrin
1.0 ng/mL
Peak Response (area): 4855

Figure 23. Estuarine Sediment, Fortified Control (LOQ)

Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



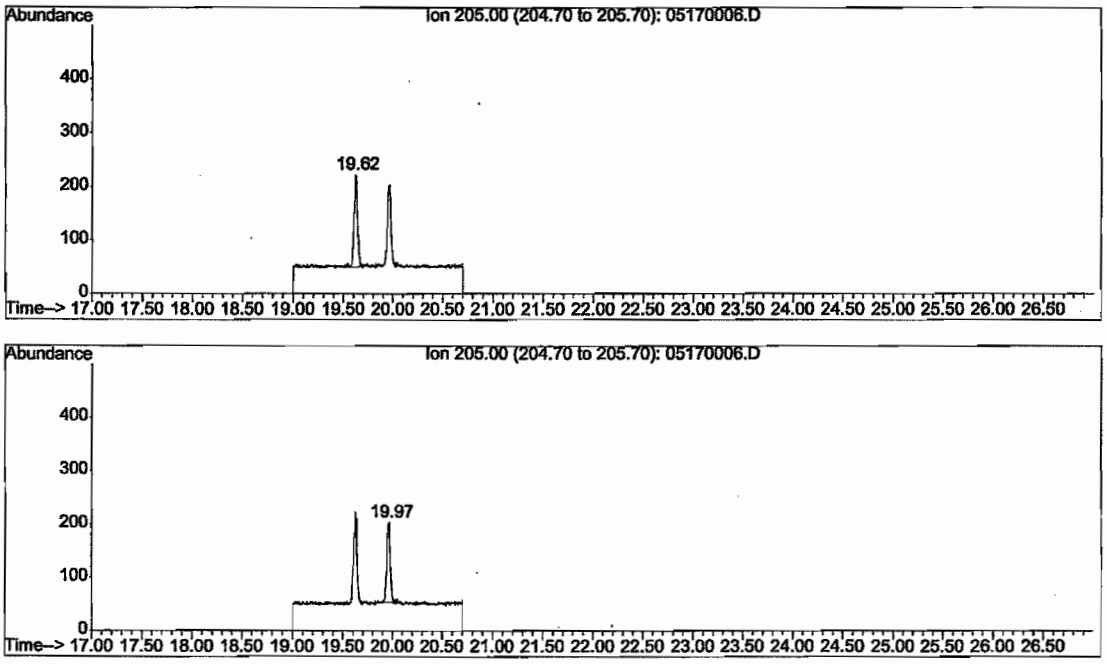
Bifenthrin
Peak Response (area): 8465
Bifenthrin Reported Recovery: 91%



Fenpropathrin
Peak Response (area): 10014
Fenpropathrin Reported Recovery: 95%

Figure 23. (con't) **Estuarine Sediment, Fortified Control (LOQ)**

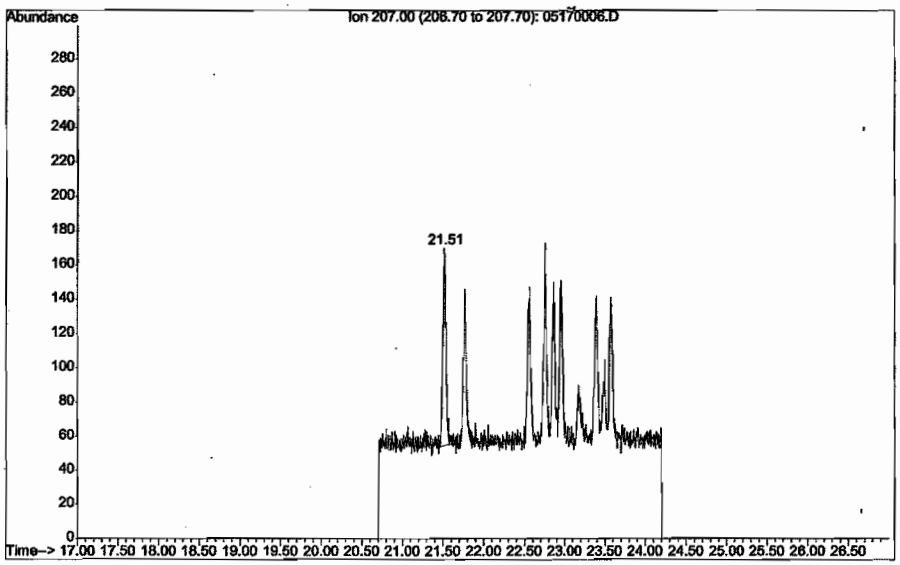
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.

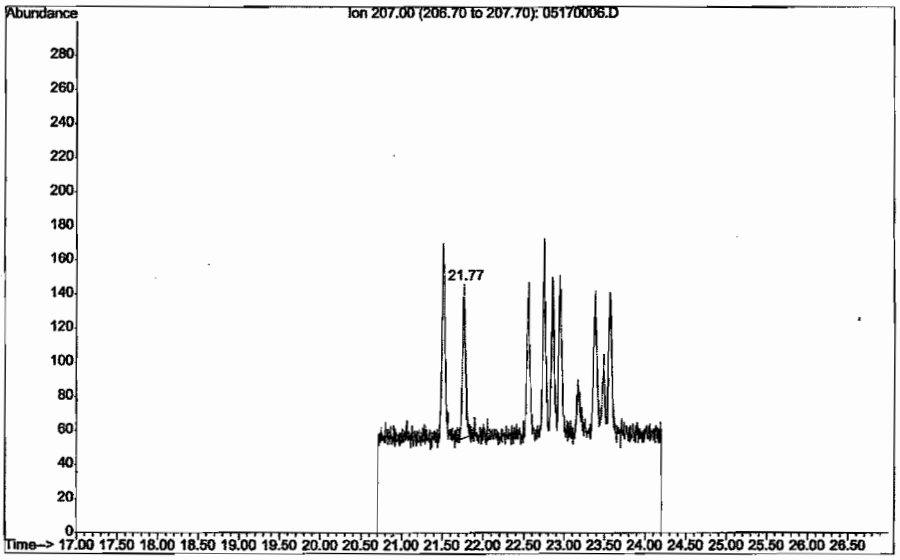


Lambda-cyhalothrin
Peak Response (area): 7959
Lambda-cyhalothrin Reported Recovery: 90%

Figure 23. (con't) **Estuarine Sediment, Fortified Control (LOQ)**

Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.

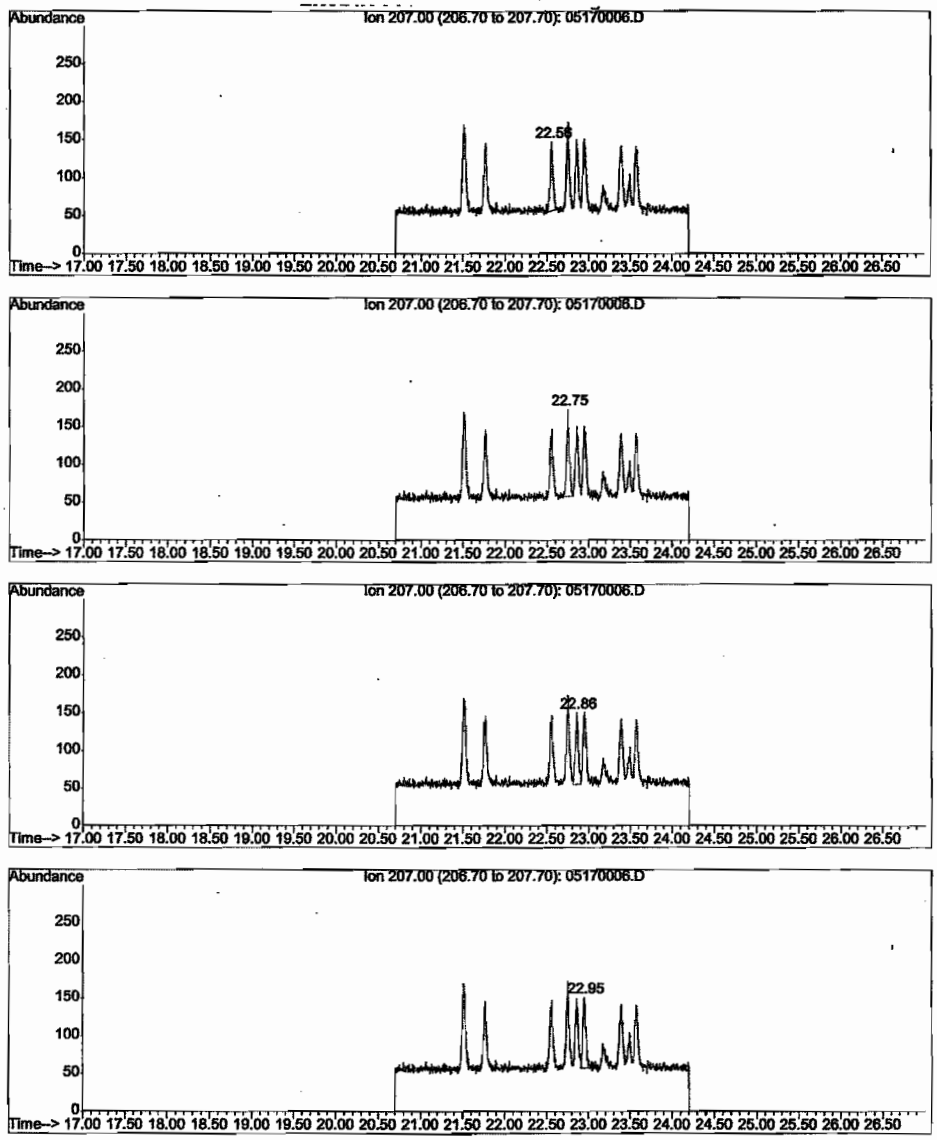




Permethrin
Peak Response (area): 5433
Permethrin Reported Recovery: 98%

Figure 23. (con't) **Estuarine Sediment, Fortified Control (LOQ)**

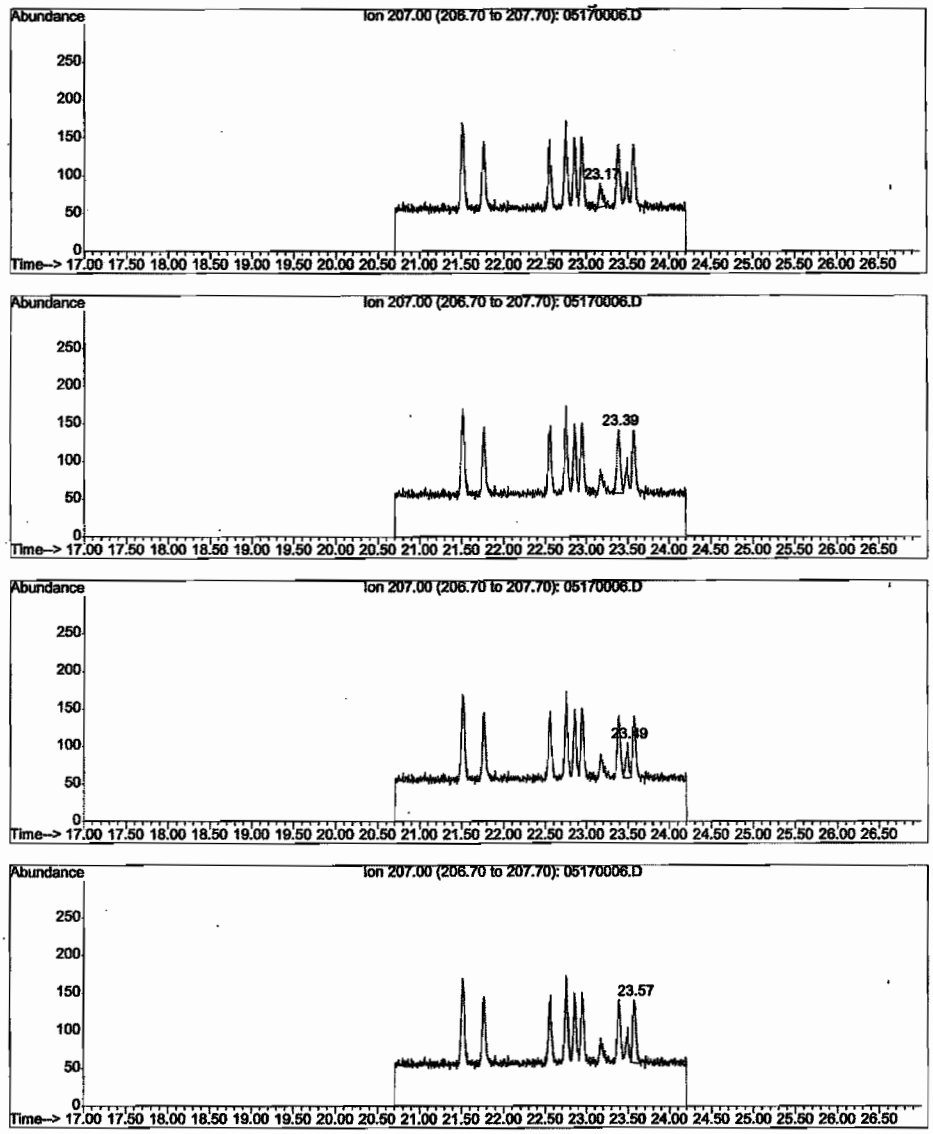
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



Cyfluthrin
Peak Response (area): 9613
Cyfluthrin Reported Recovery: 89%

Figure 23. (con't) **Estuarine Sediment, Fortified Control (LOQ)**

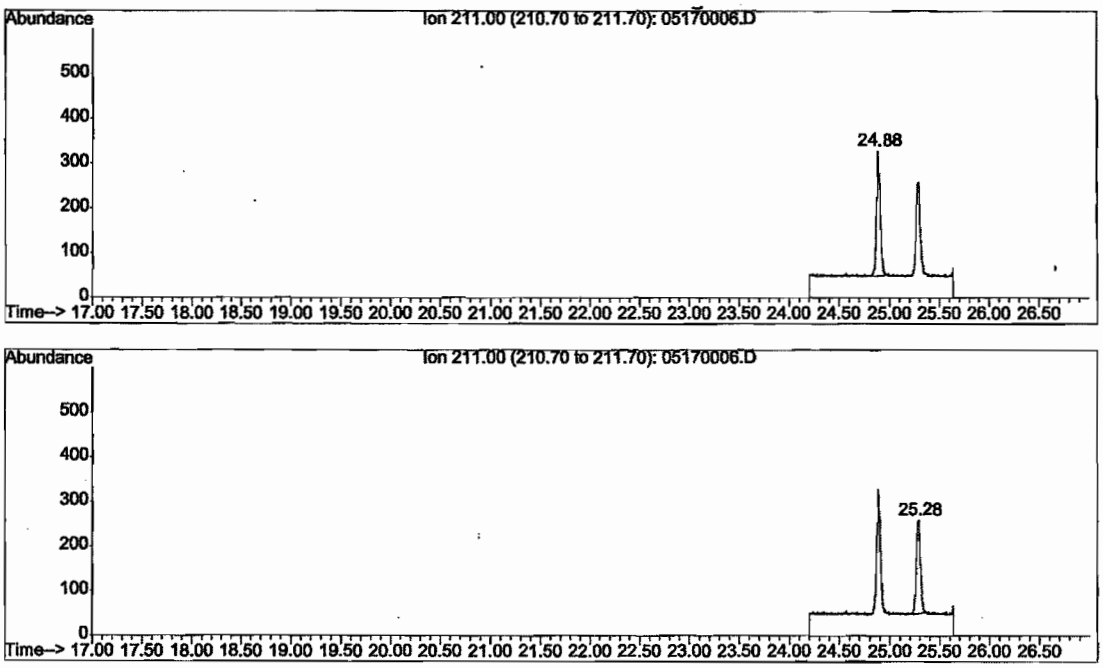
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



Cypermethrin
Peak Response (area): 6423
Cypermethrin Reported Recovery: 98%

Figure 23. (con't) Estuarine Sediment, Fortified Control (LOQ)

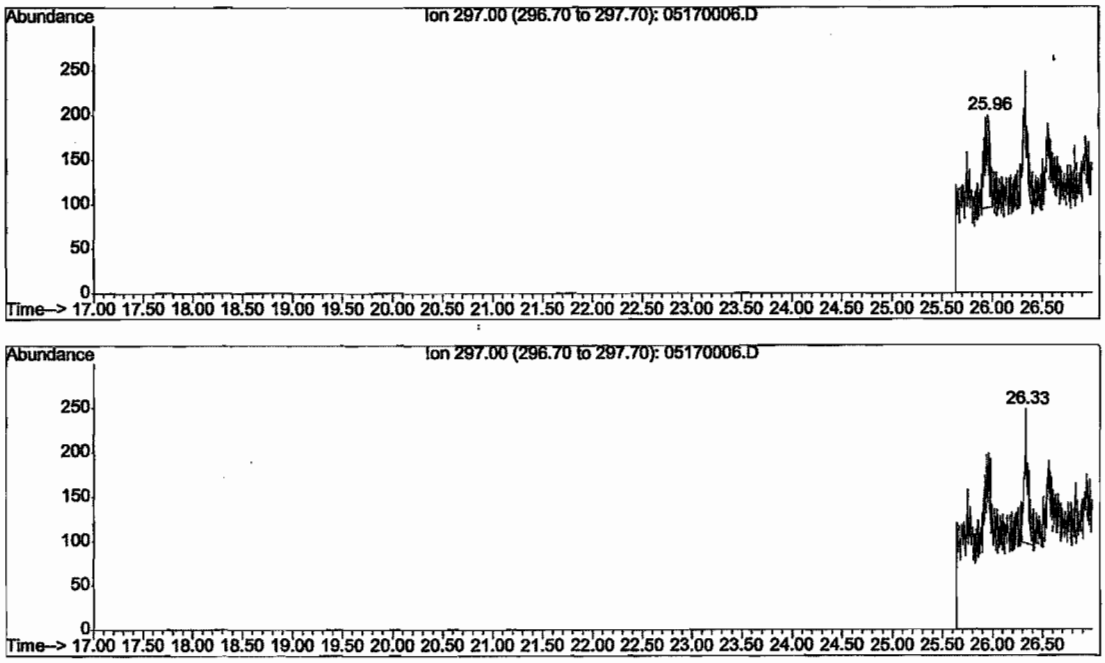
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



Esfenvalerate
Peak Response (area): 12911
Esfenvalerate Reported Recovery: 97%

Figure 23. (con't) **Estuarine Sediment, Fortified Control (LOQ)**

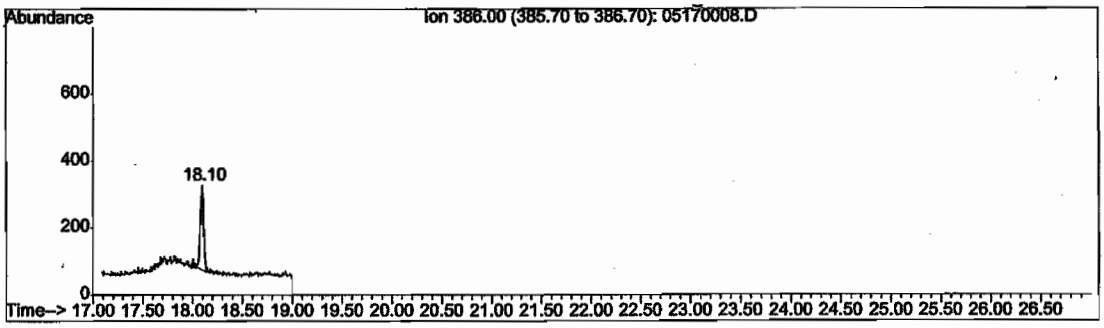
Fortified Control, Estuarine Sediment, Paradise Cove, Fortified Control 21 @ 0.1 µg/kg for all analytes except Permethrin @ 1.0 µg/kg, 10.0 g/mL.



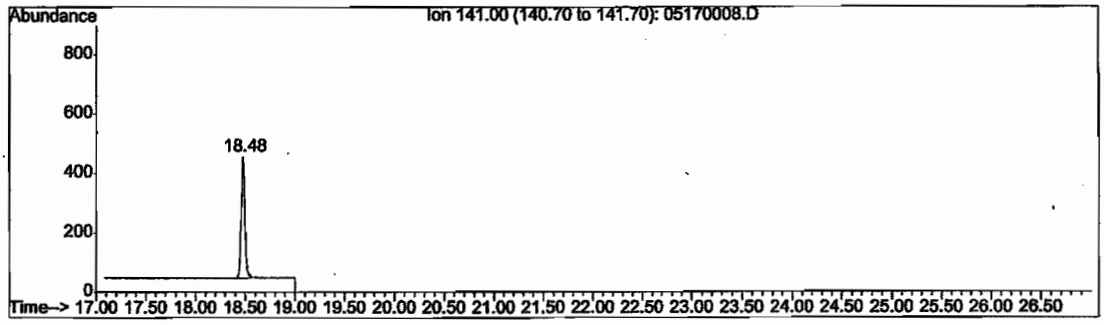
Deltamethrin
Peak Response (area): 7418
Deltamethrin Reported Recovery: 87%

Figure 24. Bracketing Standards

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



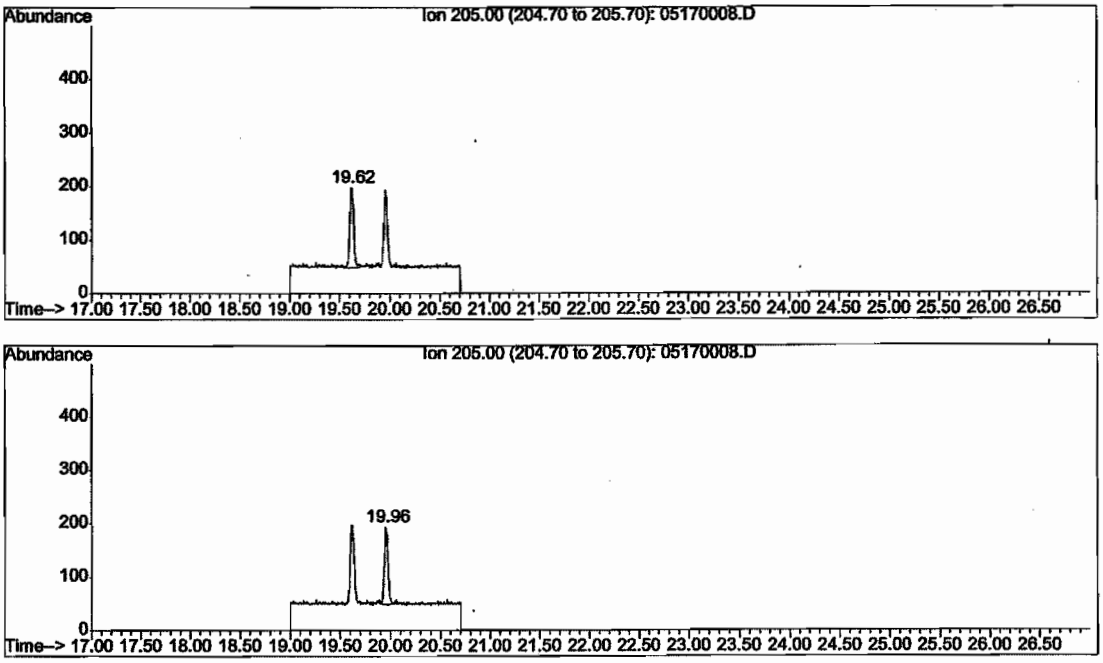
Bifenthrin
1.0 ng/mL
Peak Response (area): 5926



Fenpropathrin
1.0 ng/mL
Peak Response (area): 10414

Figure 24. (con't) Bracketing Standards

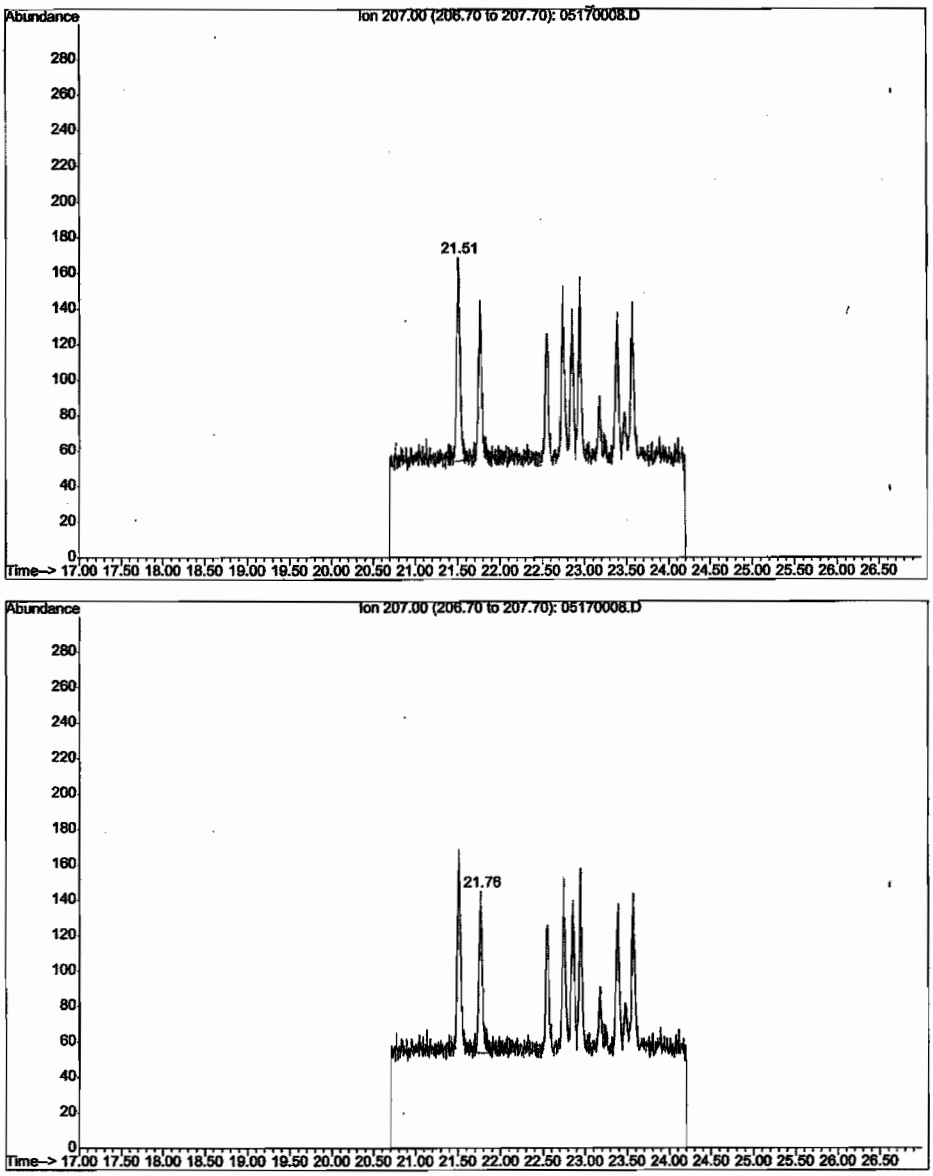
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 7569

Figure 24. (con't) Bracketing Standards

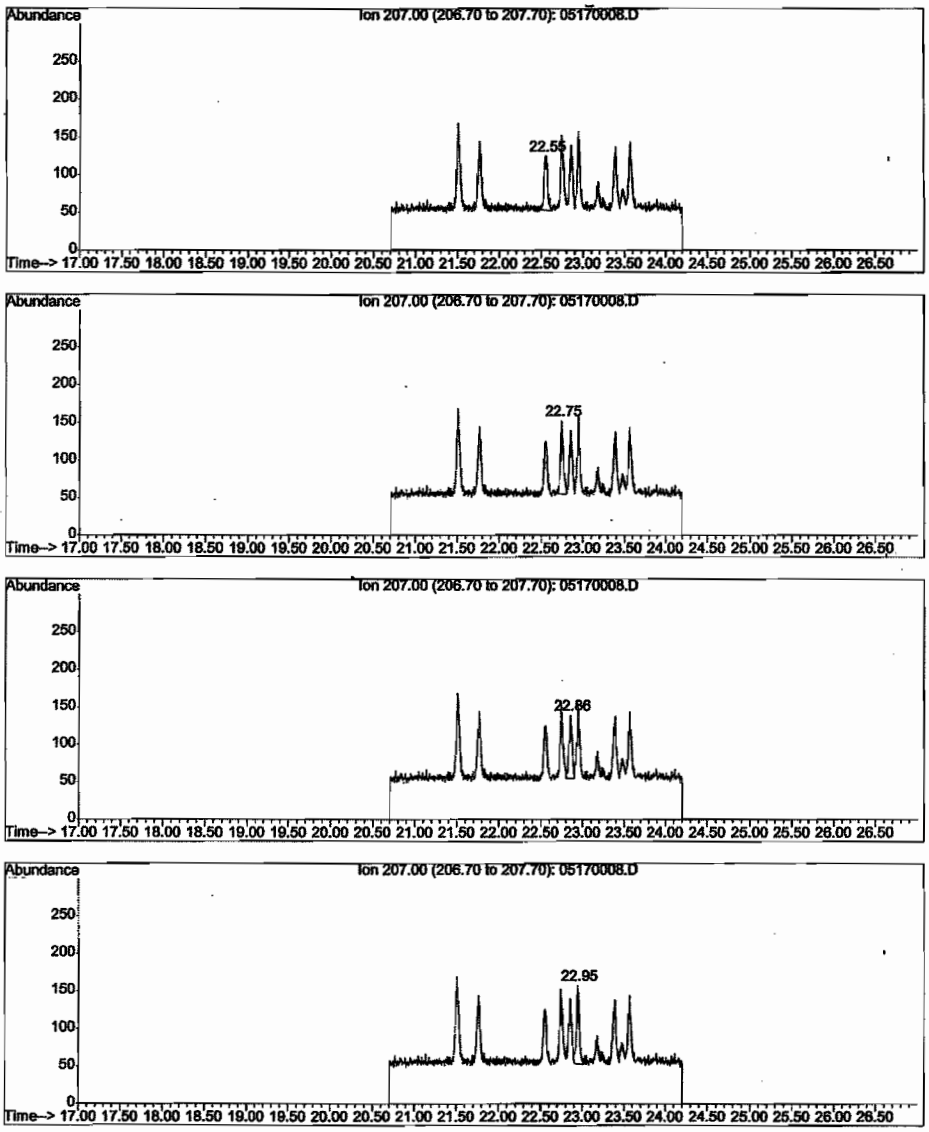
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Permethrin
10 ng/mL
Peak Response (area): 5606

Figure 24. (con't) Bracketing Standards

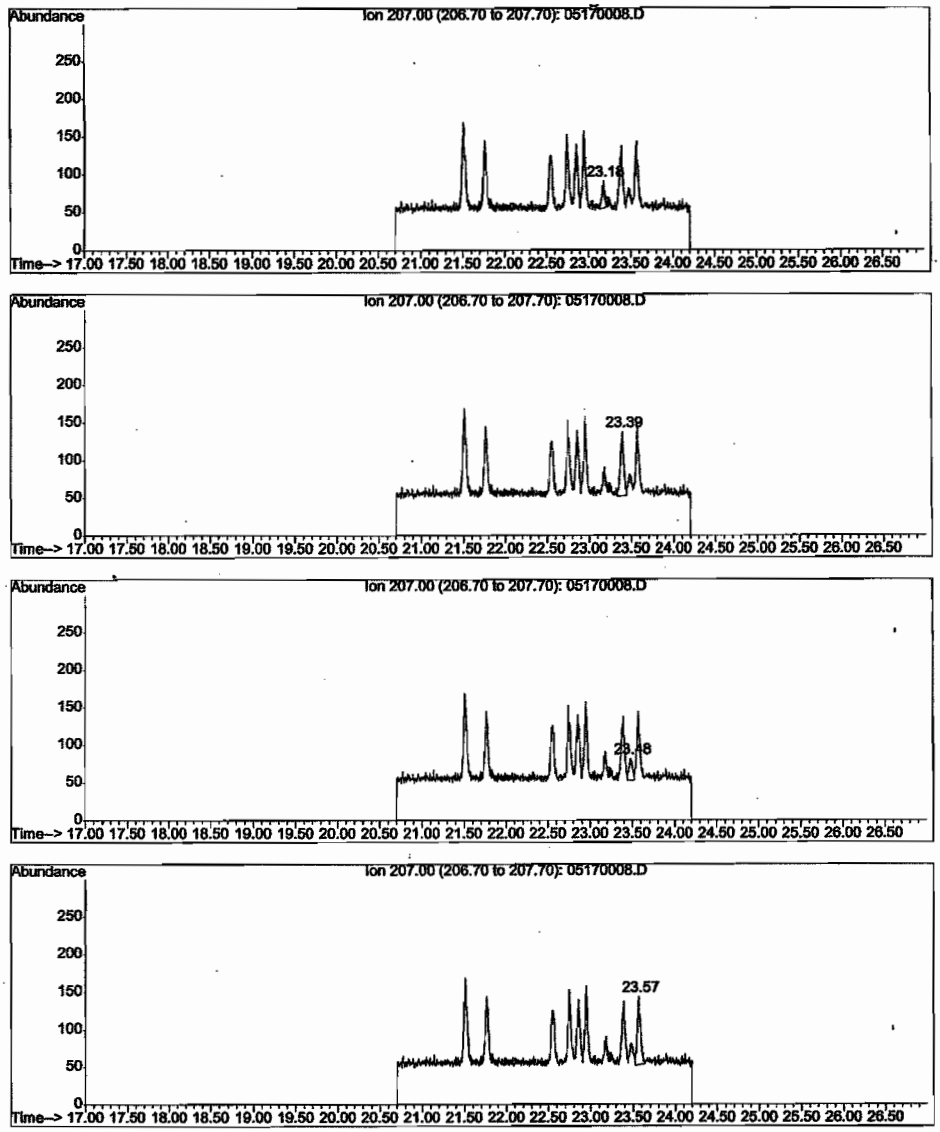
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cyfluthrin
1.0 ng/mL
Peak Response (area): 9051

Figure 24. (con't) Bracketing Standards

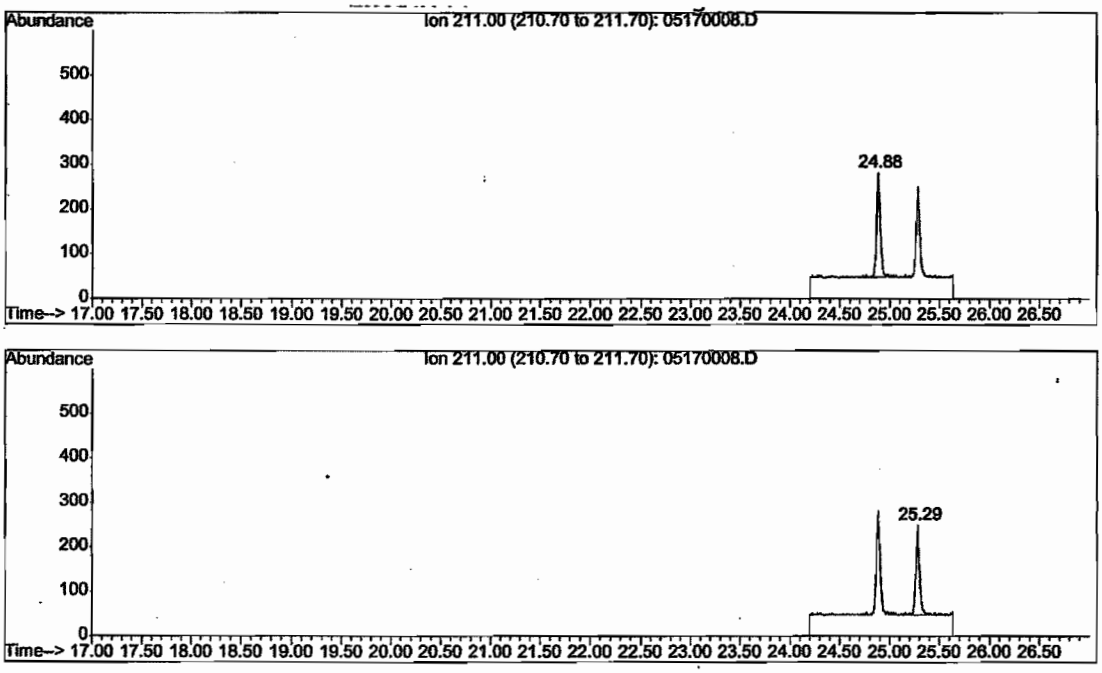
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cypermethrin
1.0 ng/mL
Peak Response (area): 6254

Figure 24. (con't) Bracketing Standards

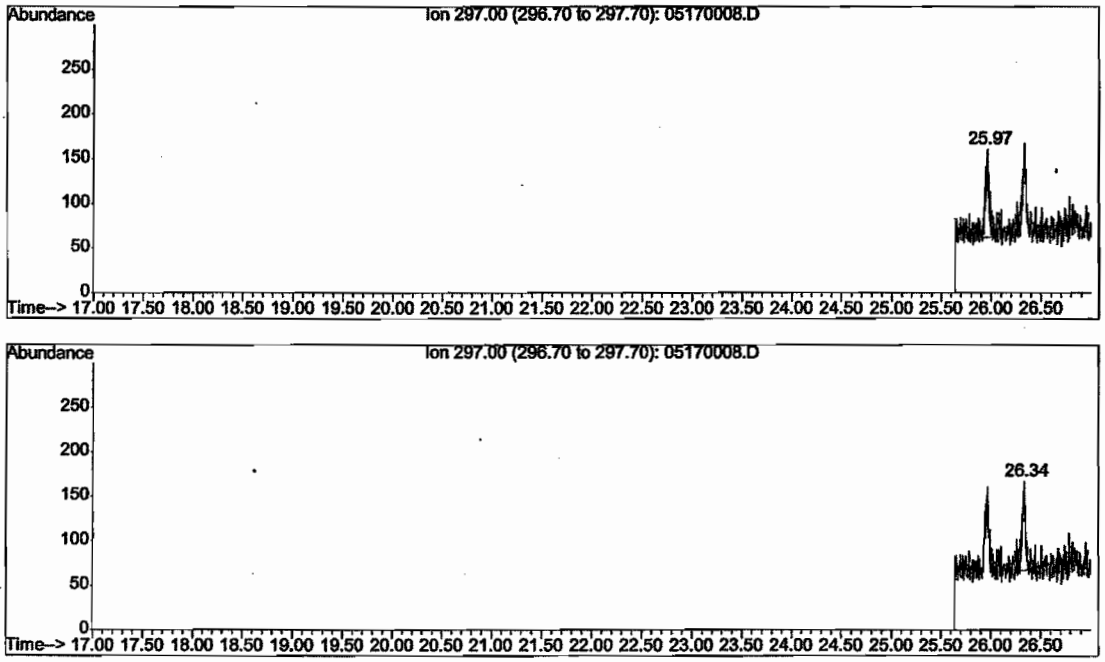
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Esfenvalerate
1.0 ng/mL
Peak Response (area): 10940

Figure 24. (con't) Bracketing Standards

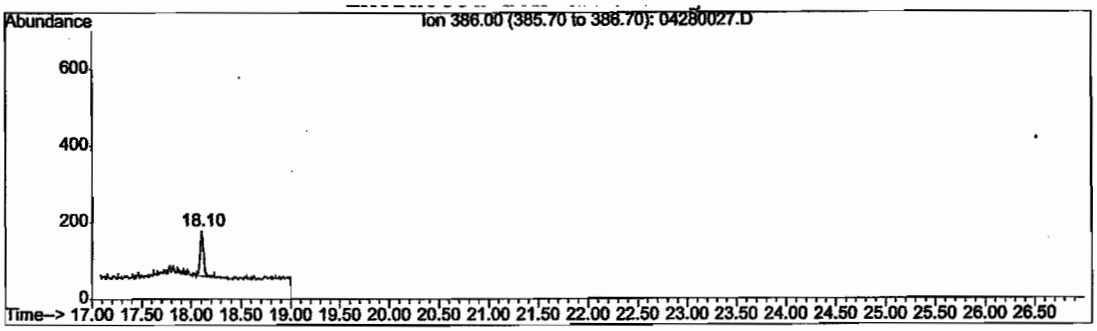
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



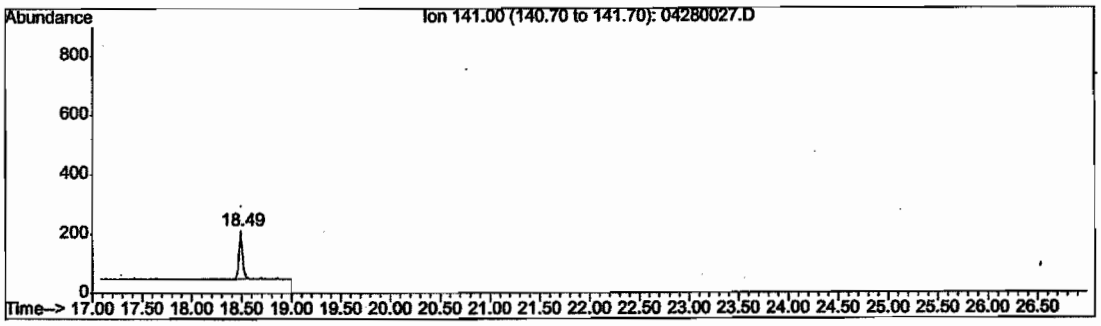
Deltamethrin
1.0 ng/mL
Peak Response (area): 4457

Figure 25. Calibration Standards @ 0.5/5 ng/mL

Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin.



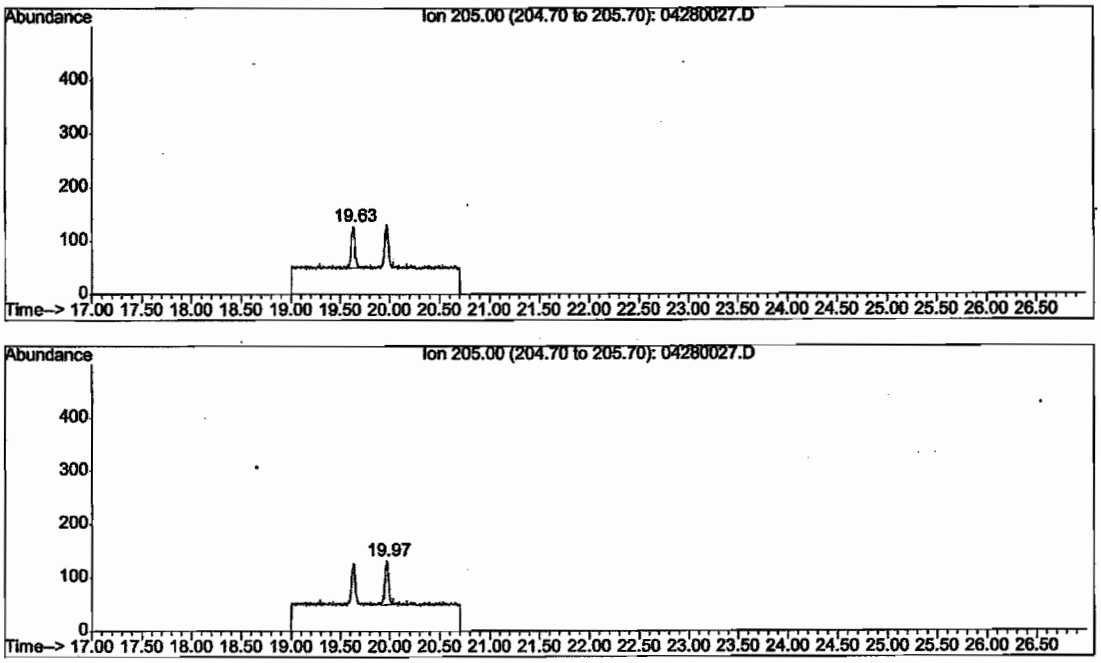
Bifenthrin
0.5 ng/mL
Peak Response (area): 2681



Fenpropathrin
0.5 ng/mL
Peak Response (area): 3802

Figure 25.(con't) Calibration Standards @ 0.5/5 ng/mL

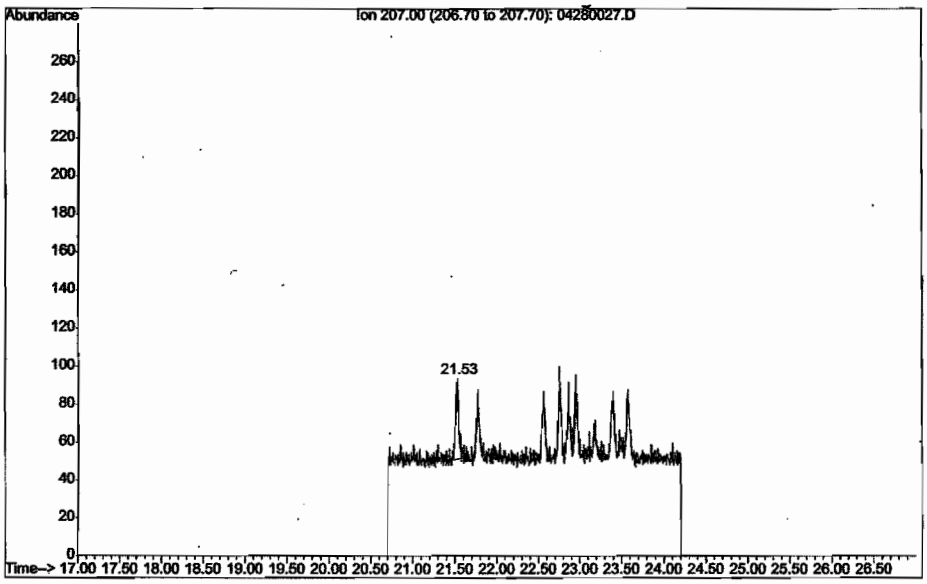
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin.

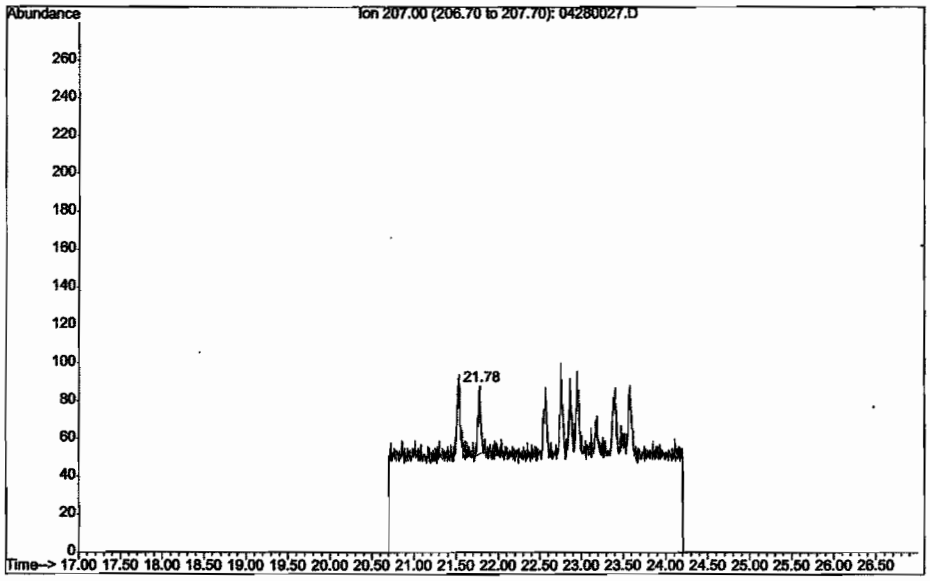


Lambda-cyhalothrin
0.5 ng/mL
Peak Response (area): 4057

Figure 25.(con't) Calibration Standards @ 0.5/5 ng/mL

Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin.

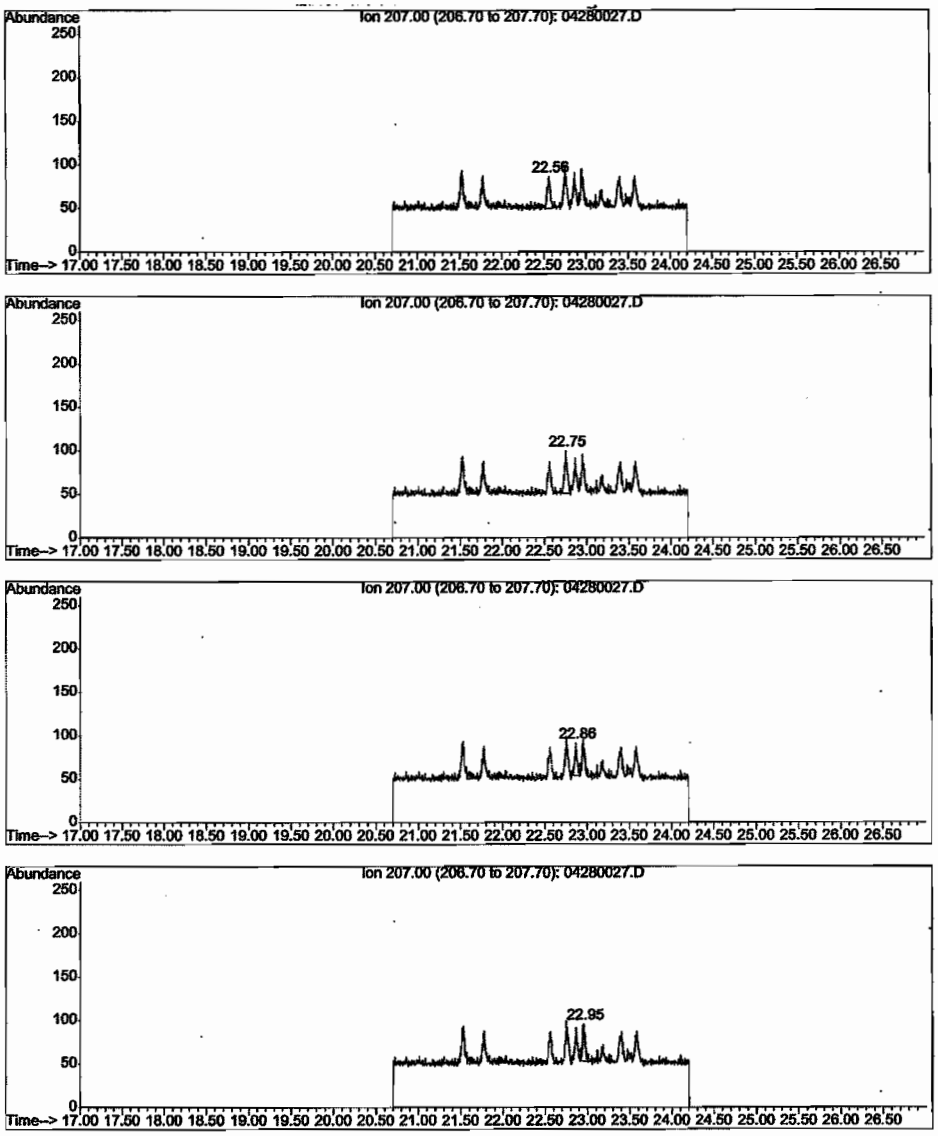




Permethrin
5.0 ng/mL
Peak Response (area): 1788

Figure 25.(con't) Calibration Standards @ 0.5/5 ng/mL

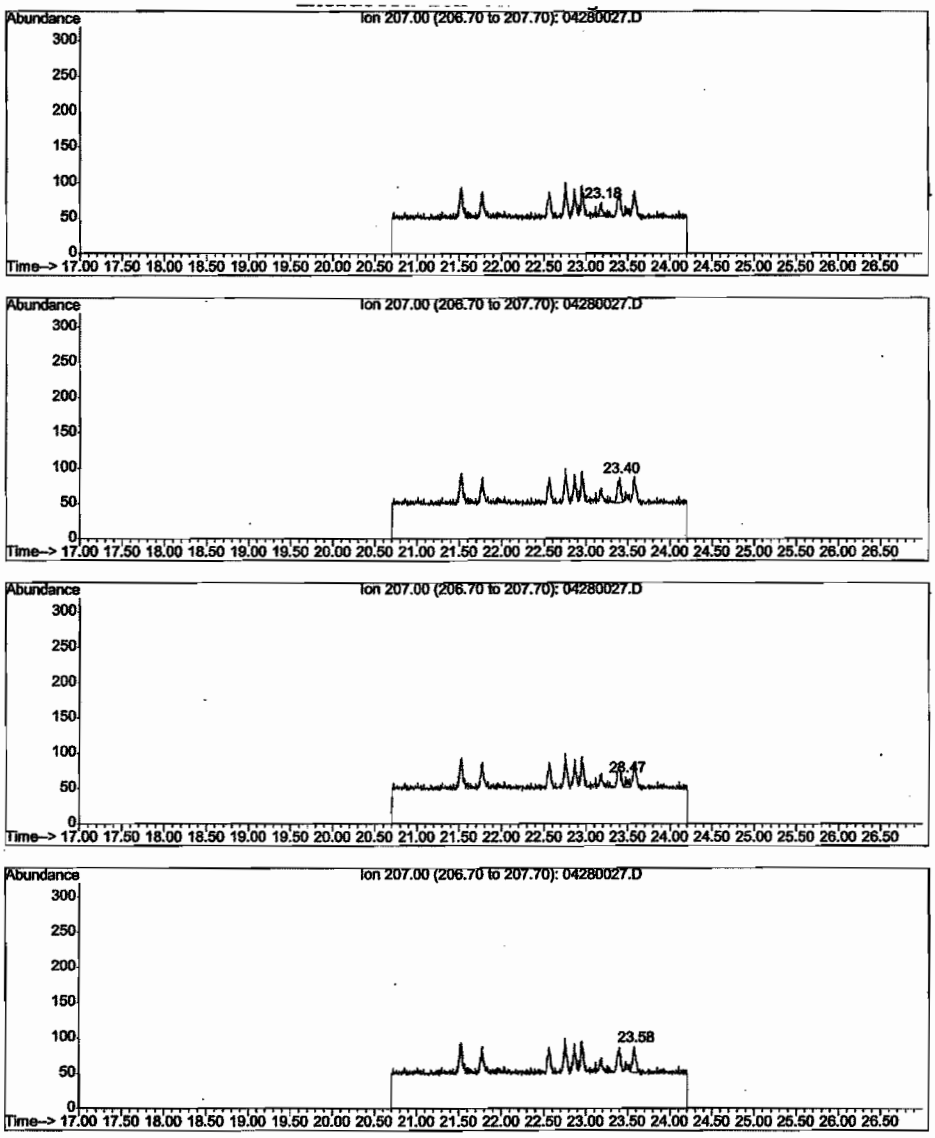
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin.



Cyfluthrin
0.5 ng/mL
Peak Response (area): 3564

Figure 25.(con't) Calibration Standards @ 0.5/5 ng/mL

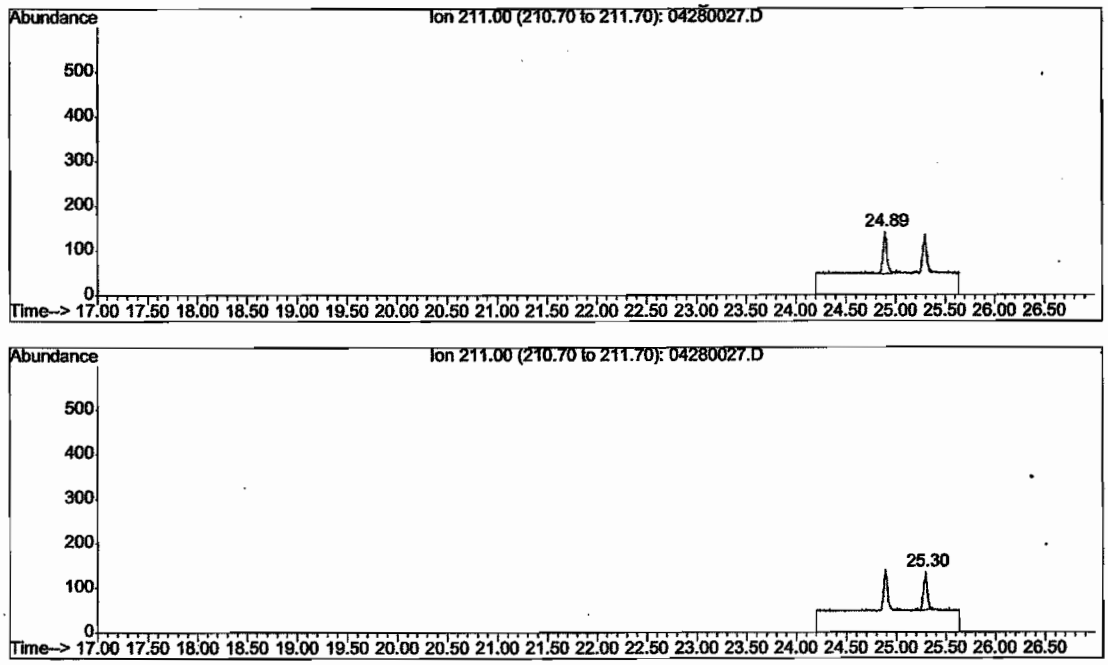
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin.



Cypermethrin
0.5 ng/mL
Peak Response (area): 2452

Figure 25.(con't) Calibration Standards @ 0.5/5 ng/mL

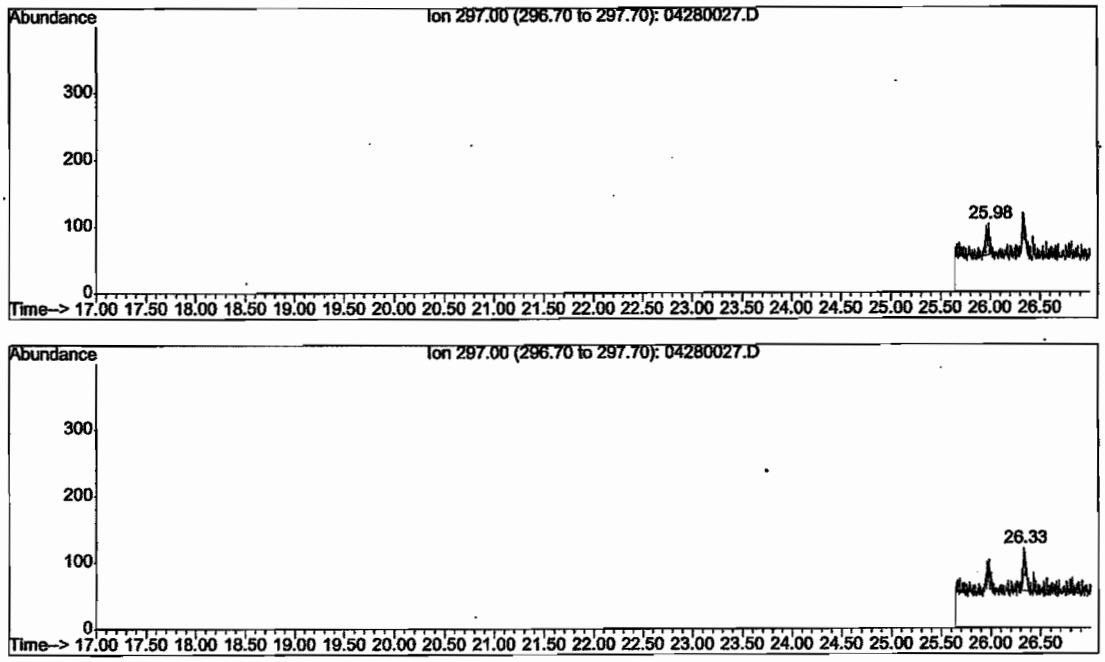
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin.



Esfenvalerate
0.5 ng/mL
Peak Response (area): 4521

Figure 25.(con't) Calibration Standards @ 0.5/5 ng/mL

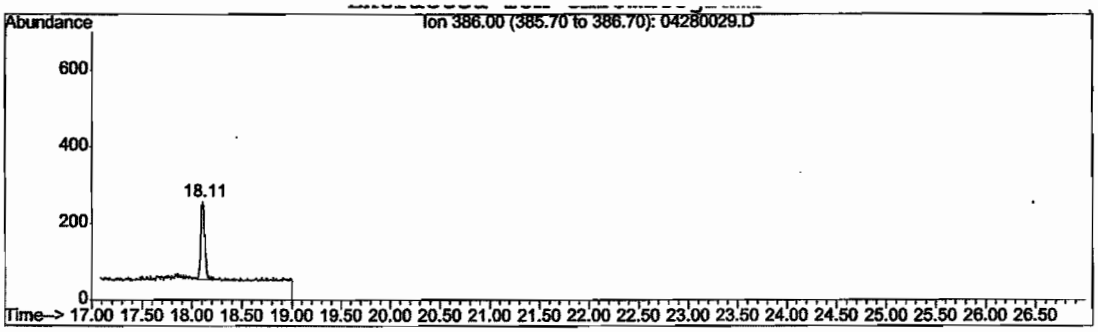
Pyrethroids, standard mix @ 0.5 ng/mL for all analytes except Permethrin; 5.0 ng/mL for Permethrin.



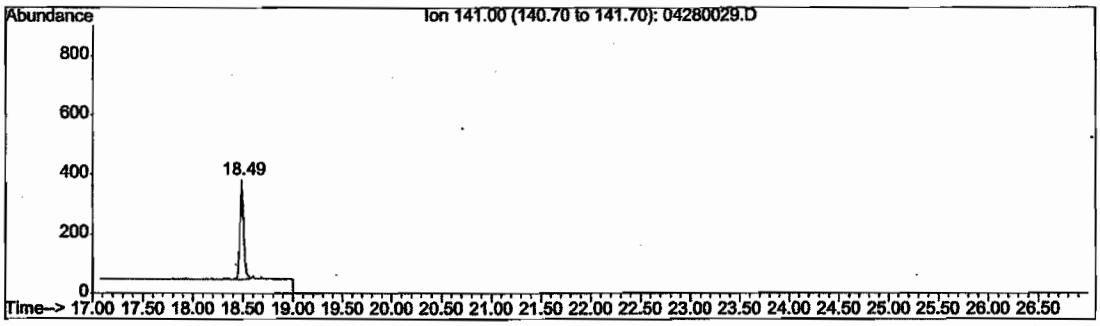
Deltamethrin
0.5 ng/mL
Peak Response (area): 2141

Figure 26. Calibration Standards @ 1.0/10 ng/mL

Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



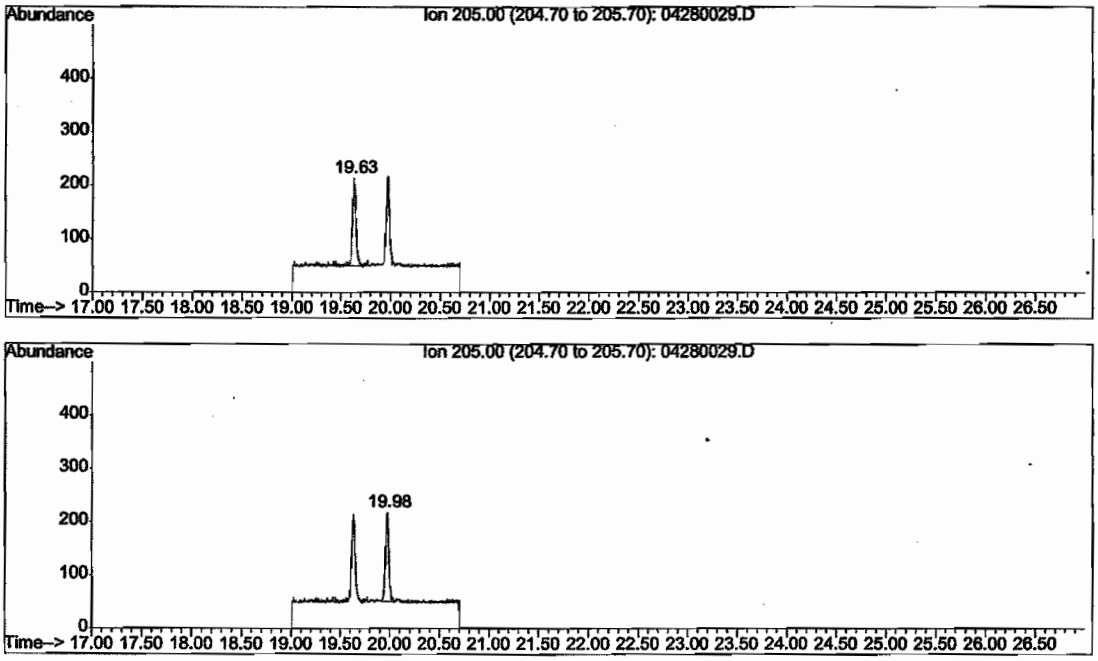
Bifenthrin
1.0 ng/mL
Peak Response (area): 5493



Fenpropathrin
1.0 ng/mL
Peak Response (area): 8251

Figure 26.(con't) **Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL**

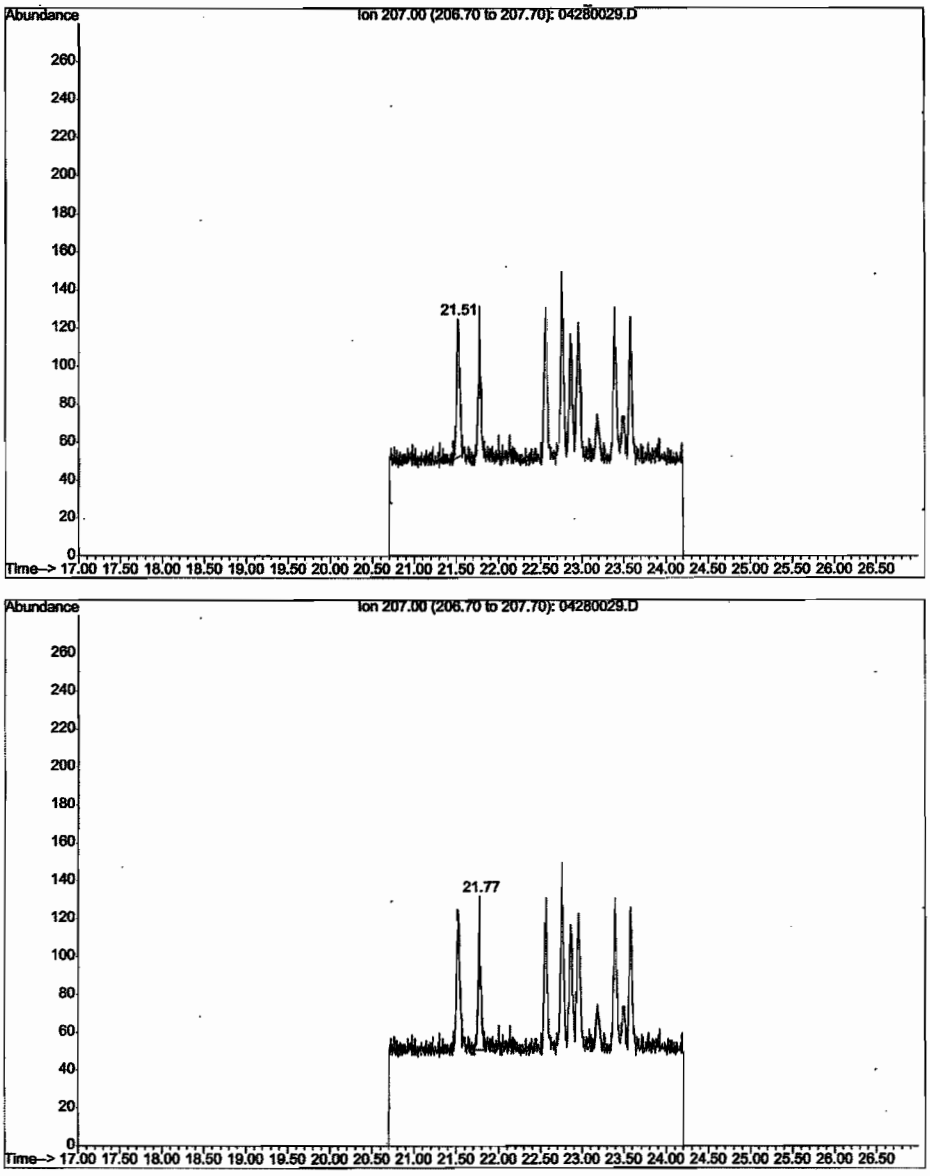
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Lambda-cyhalothrin
1.0 ng/mL
Peak Response (area): 8036

Figure 26.(con't) **Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL**

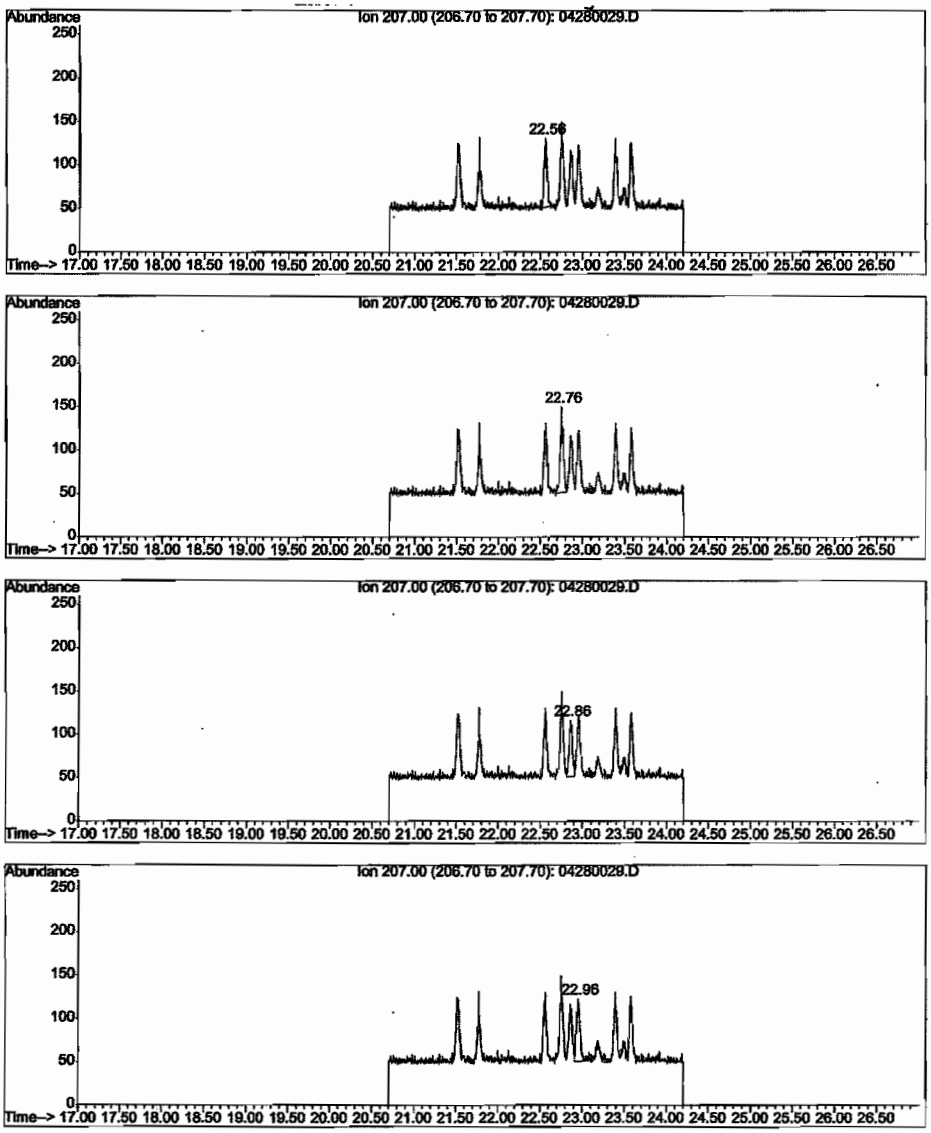
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Permethrin
10 ng/mL
Peak Response (area): 3434

Figure 26.(con't) **Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL**

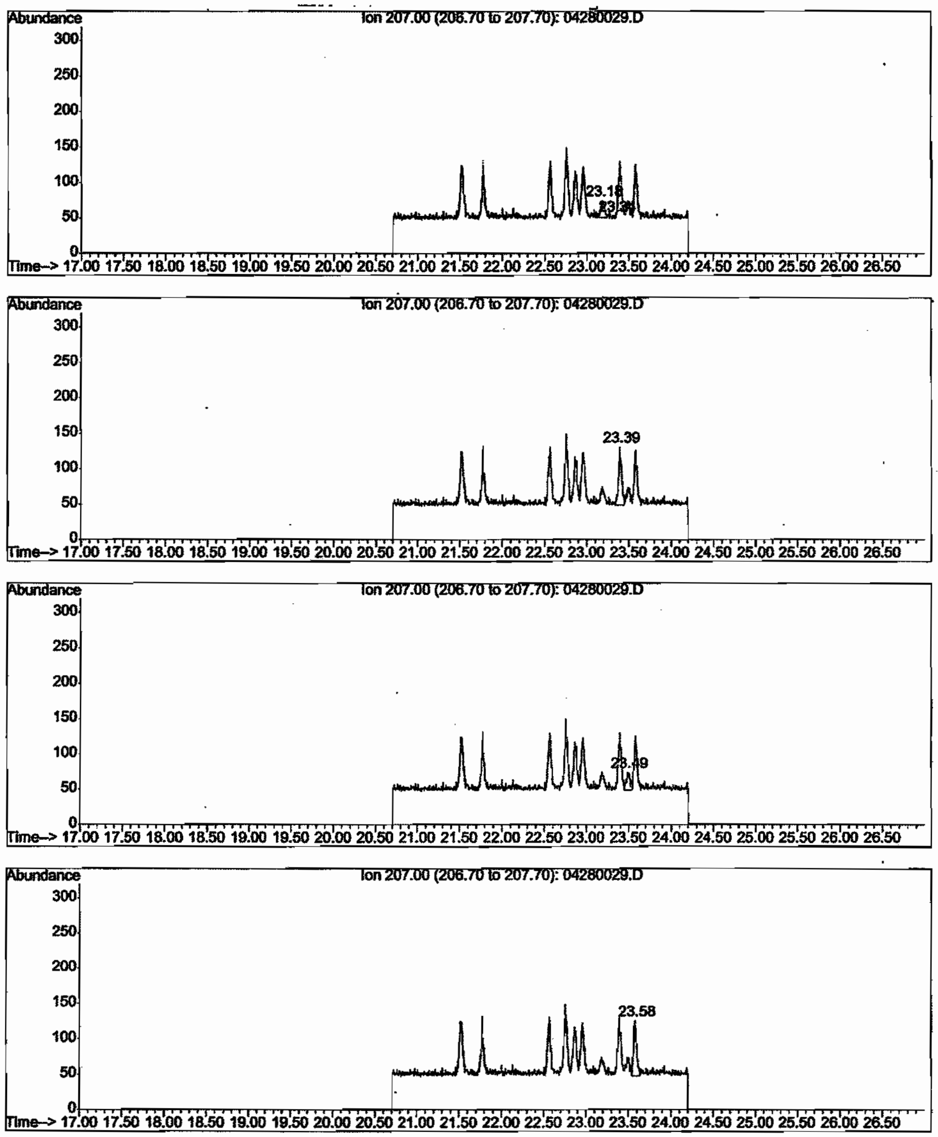
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cyfluthrin
1.0 ng/mL
Peak Response (area): 8056

Figure 26.(con't) **Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL**

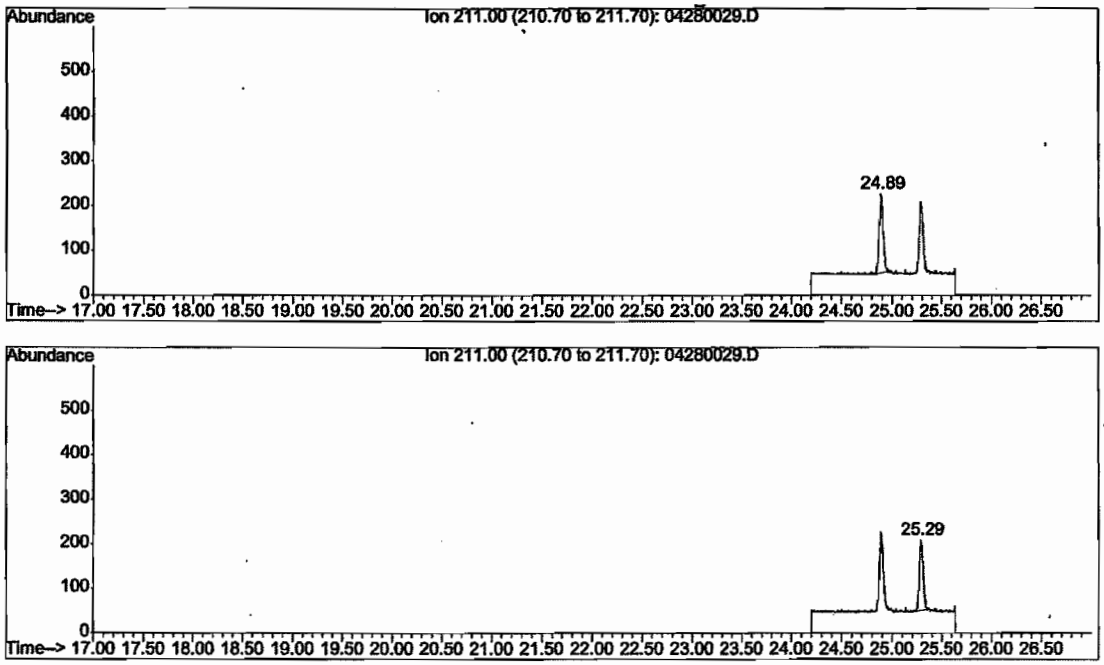
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Cypermethrin
1.0 ng/mL
Peak Response (area): 5474

Figure 26.(con't) **Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL**

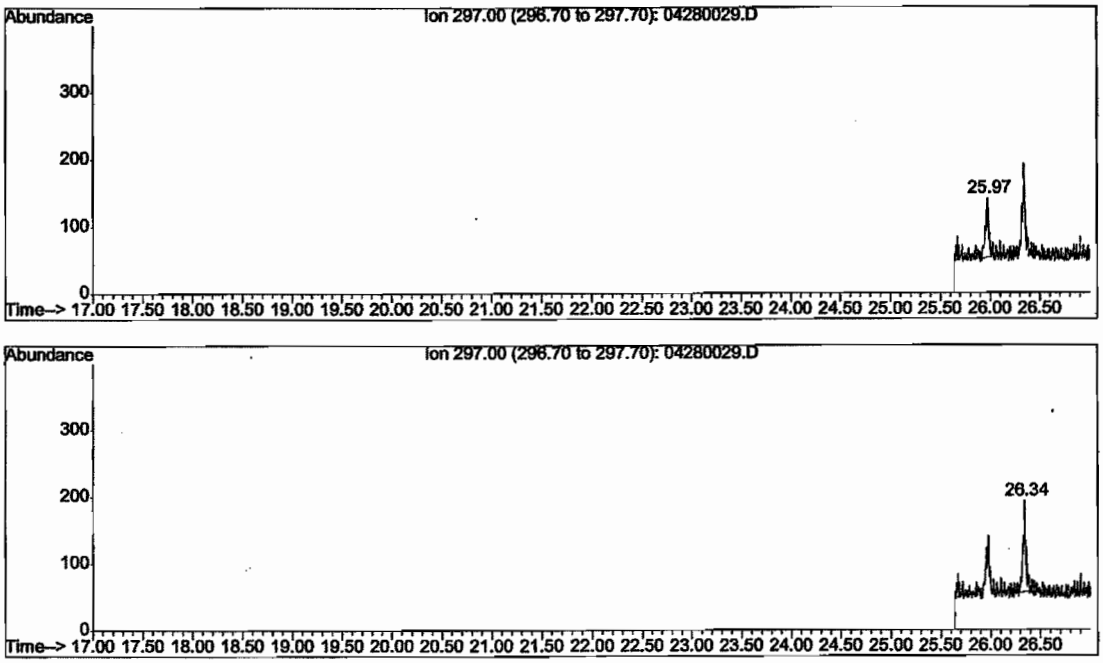
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



Esfenvalerate
1.0 ng/mL
Peak Response (area): 8960

Figure 26.(con't) **Representative Chromatograms - Calibration Standards @ 1.0/10 ng/mL**

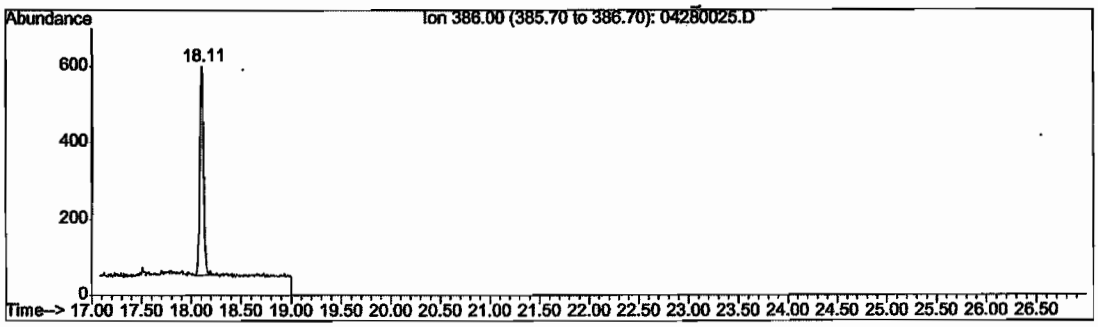
Pyrethroids, standard mix @ 1.0 ng/mL for all analytes except Permethrin; 10 ng/mL for Permethrin.



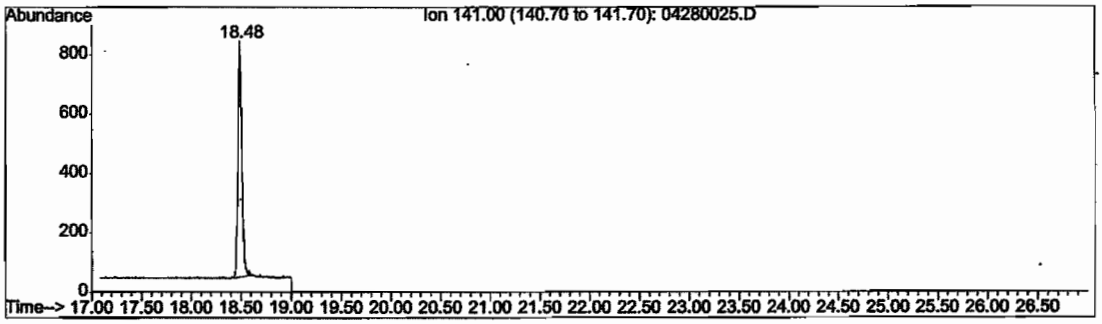
Deltamethrin
1.0 ng/mL
Peak Response (area): 4572

Figure 27. Calibration Standards @ 2.5/25 ng/mL

Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin.



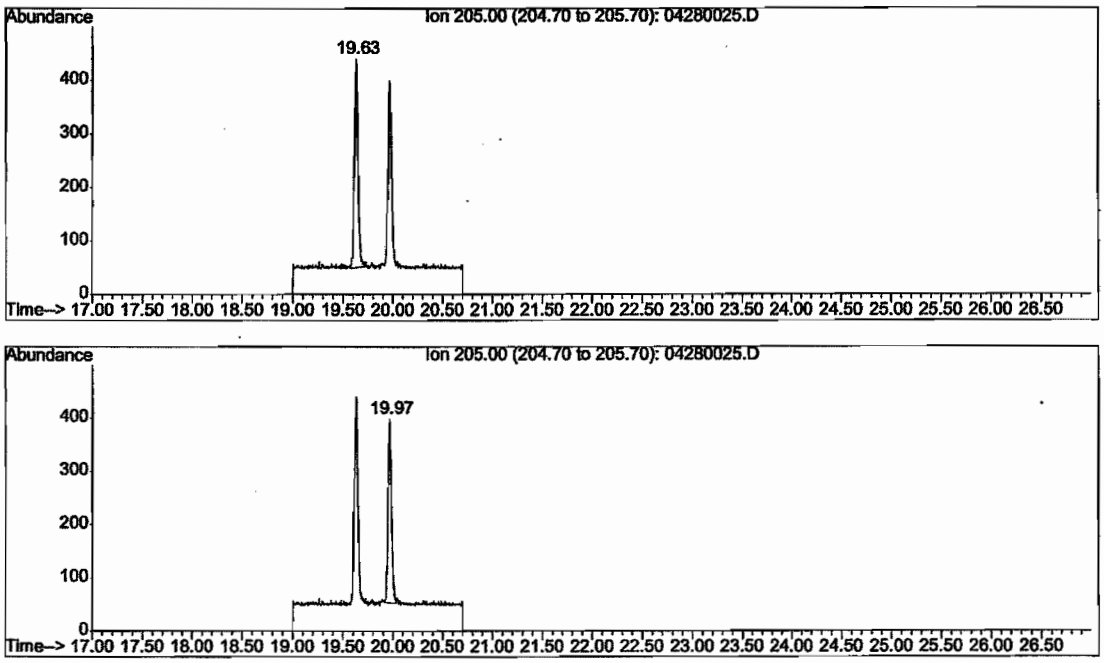
Bifenthrin
2.5 ng/mL
Peak Response (area): 14349



Fenpropathrin
2.5 ng/mL
Peak Response (area): 21056

Figure 27. (con't) **Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL**

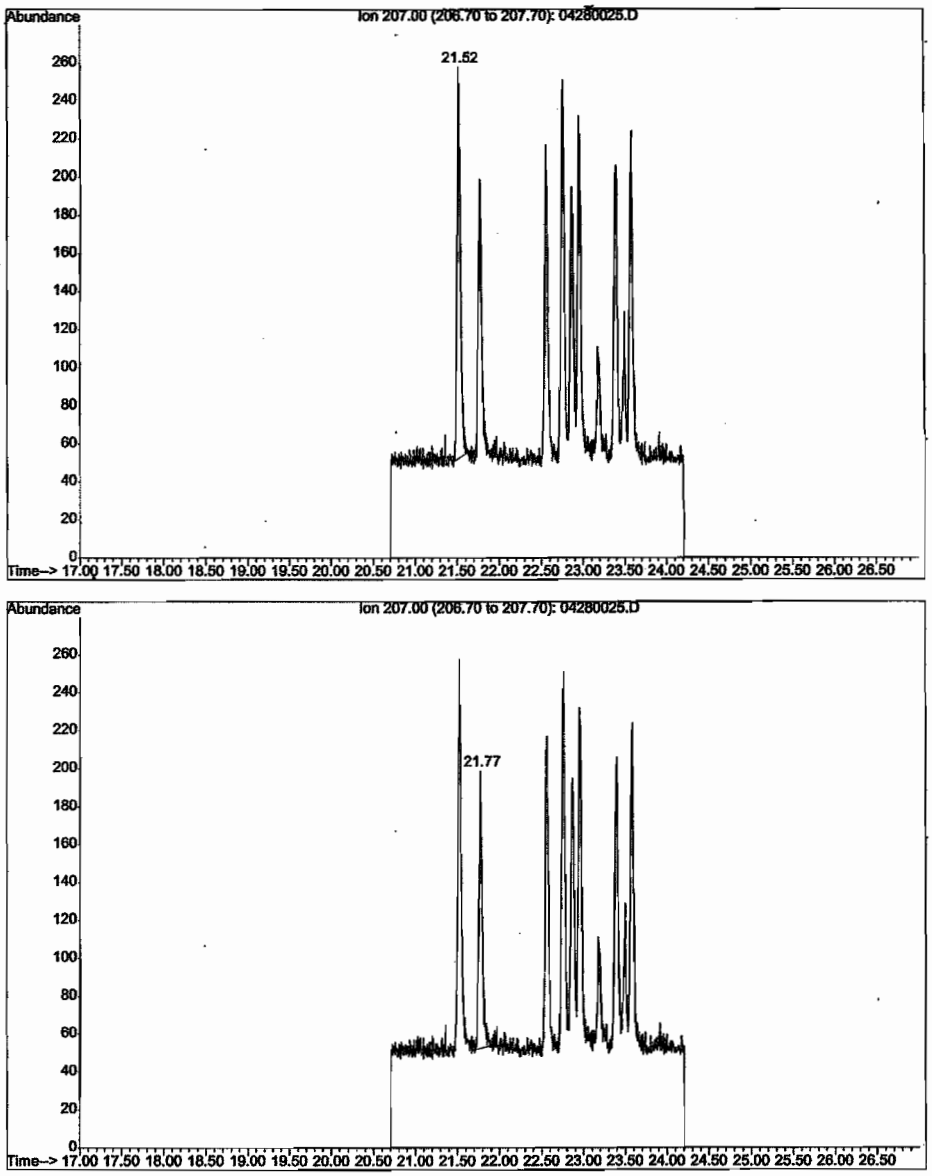
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin.



Lambda-cyhalothrin
2.5 ng/mL
Peak Response (area): 18359

Figure 27. (con't) **Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL**

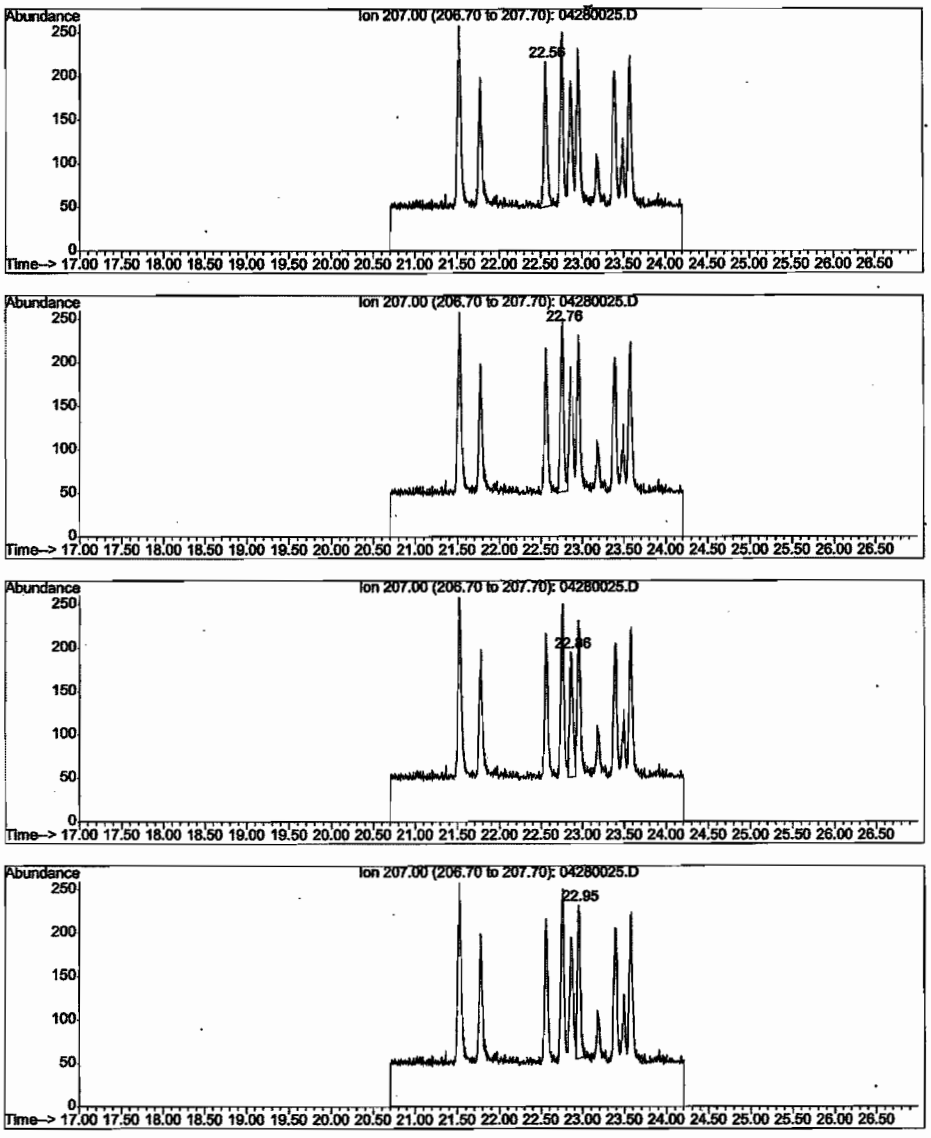
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin.



Permethrin
25.0 ng/mL
Peak Response (area): 9434

Figure 27. (con't) **Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL**

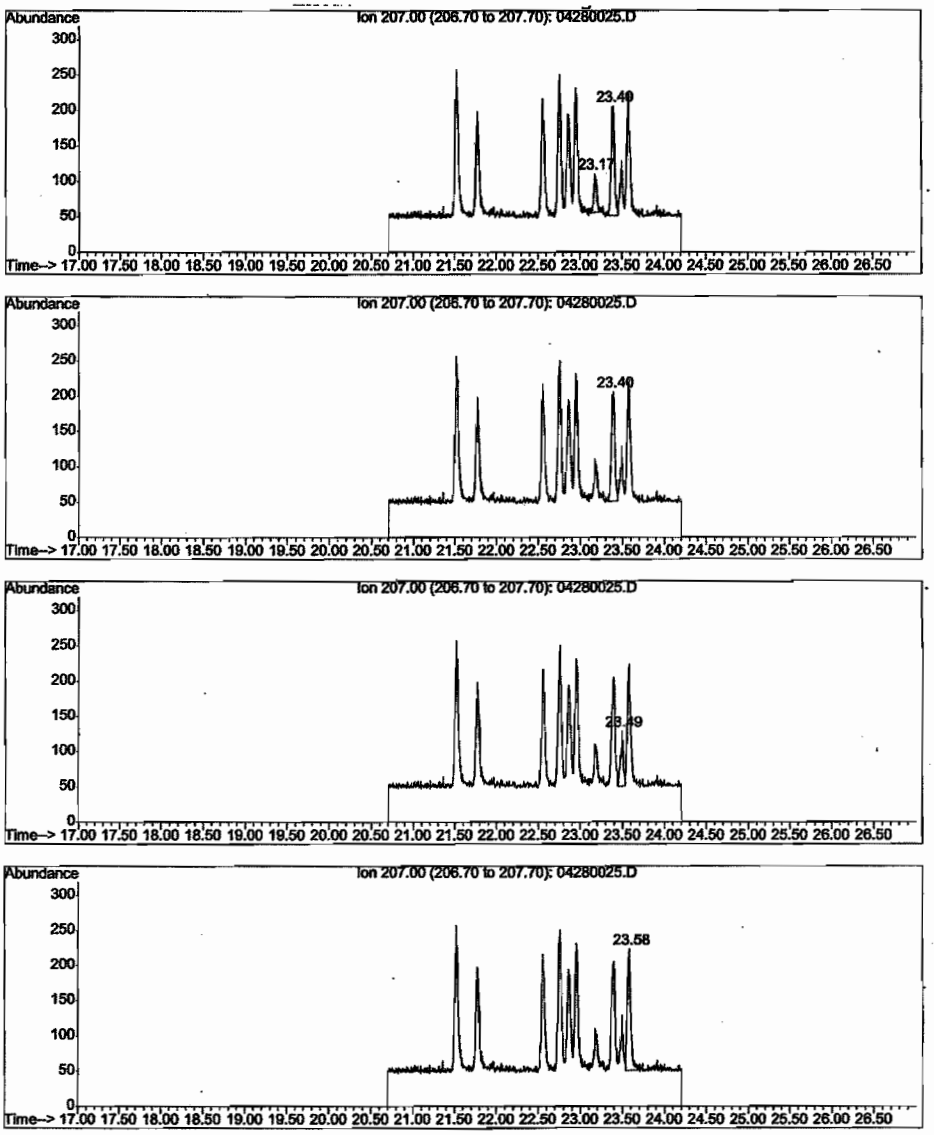
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin.



Cyfluthrin
2.5 ng/mL
Peak Response (area): 18640

Figure 27. (con't) **Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL**

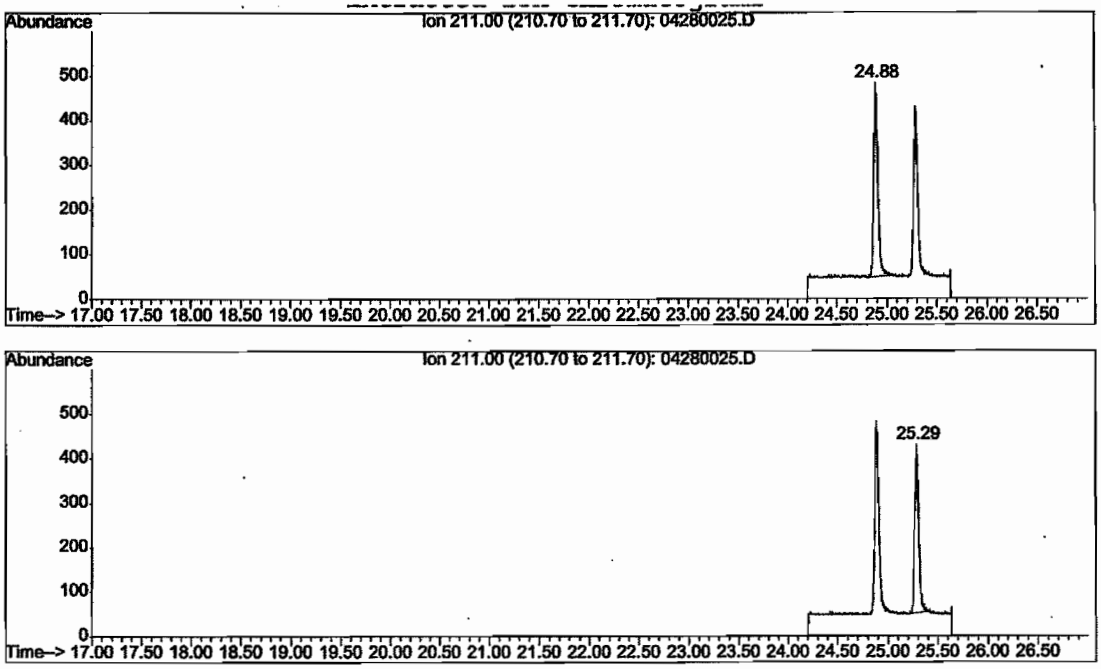
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin.



Cypermethrin
2.5 ng/mL
Peak Response (area): 12184

Figure 27. (con't) **Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL**

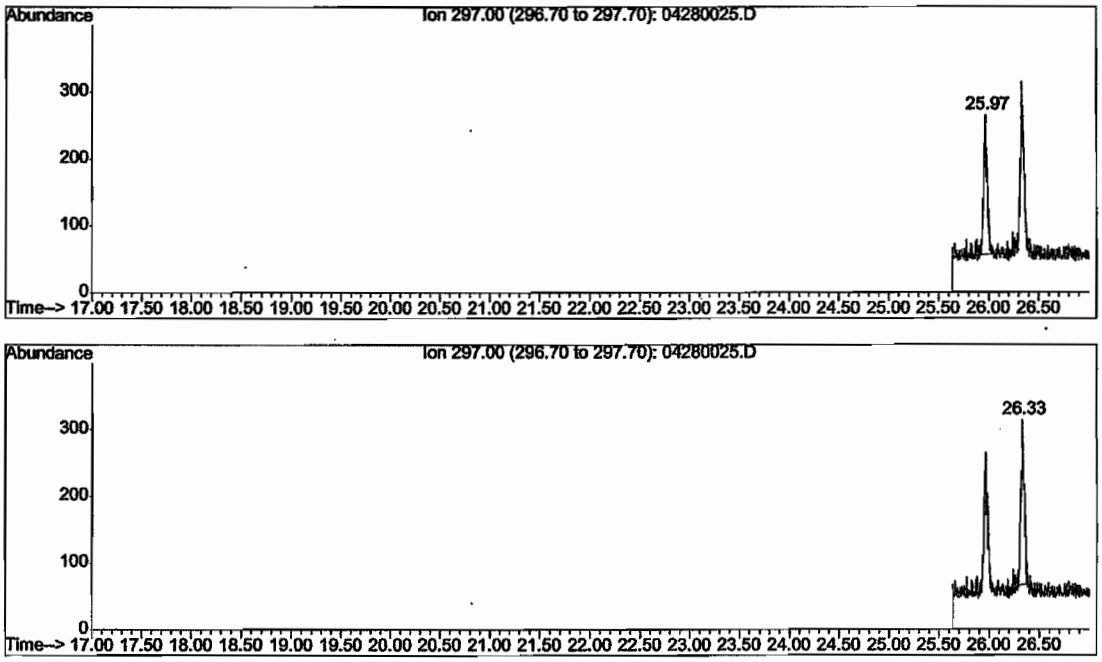
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin.



Esfenvalerate
2.5 ng/mL
Peak Response (area): 22472

Figure 27. (con't) **Representative Chromatograms - Calibration Standards @ 2.5/25 ng/mL**

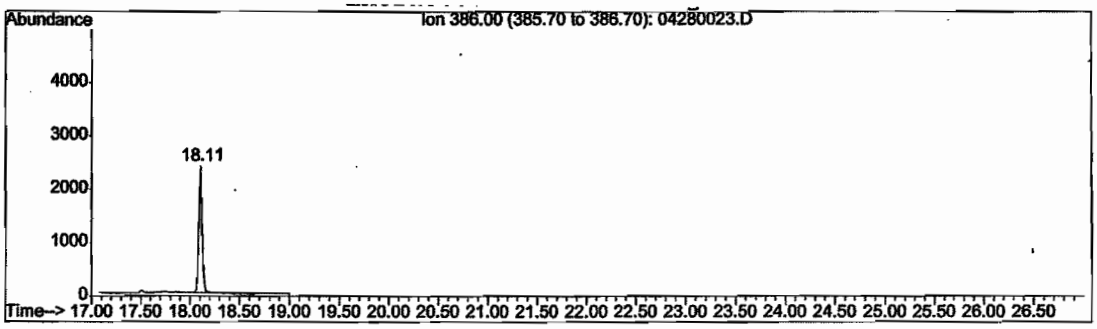
Pyrethroids, standard mix @ 2.5 ng/mL for all analytes except Permethrin; 25.0 ng/mL for Permethrin.



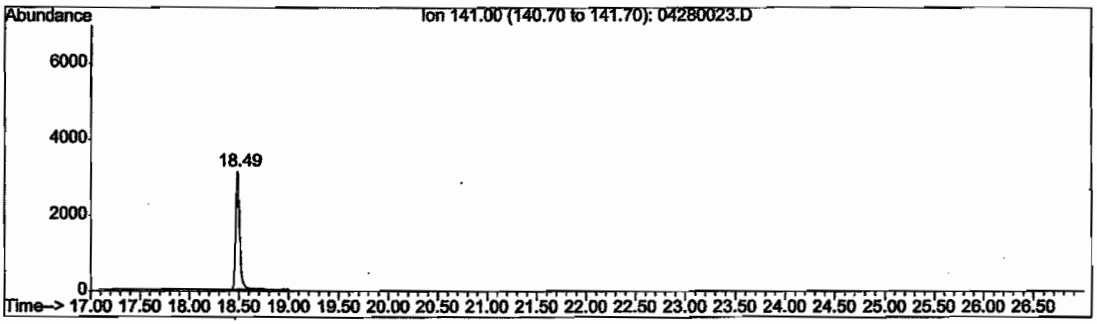
Deltamethrin
2.5 ng/mL
Peak Response (area): 10511

Figure 28. Calibration Standards @ 10/100 ng/mL

Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin.



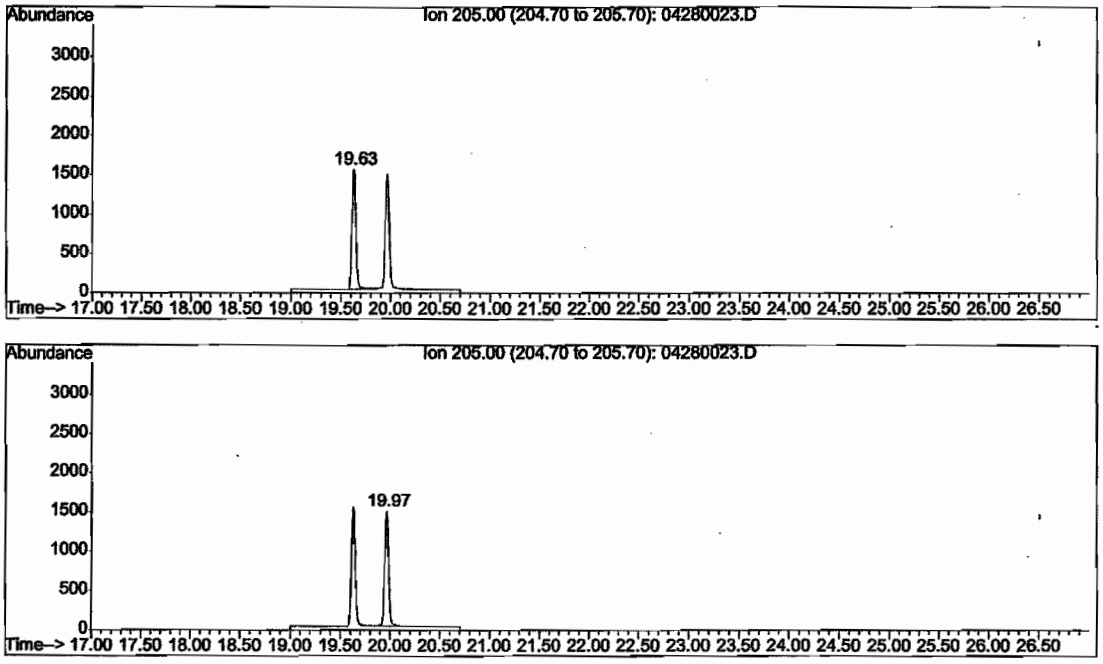
Bifenthrin
10 ng/mL
Peak Response (area): 58285



Fenpropathrin
10 ng/mL
Peak Response (area): 86322

Figure 28. (con't) **Calibration Standards @ 10/100 ng/mL**

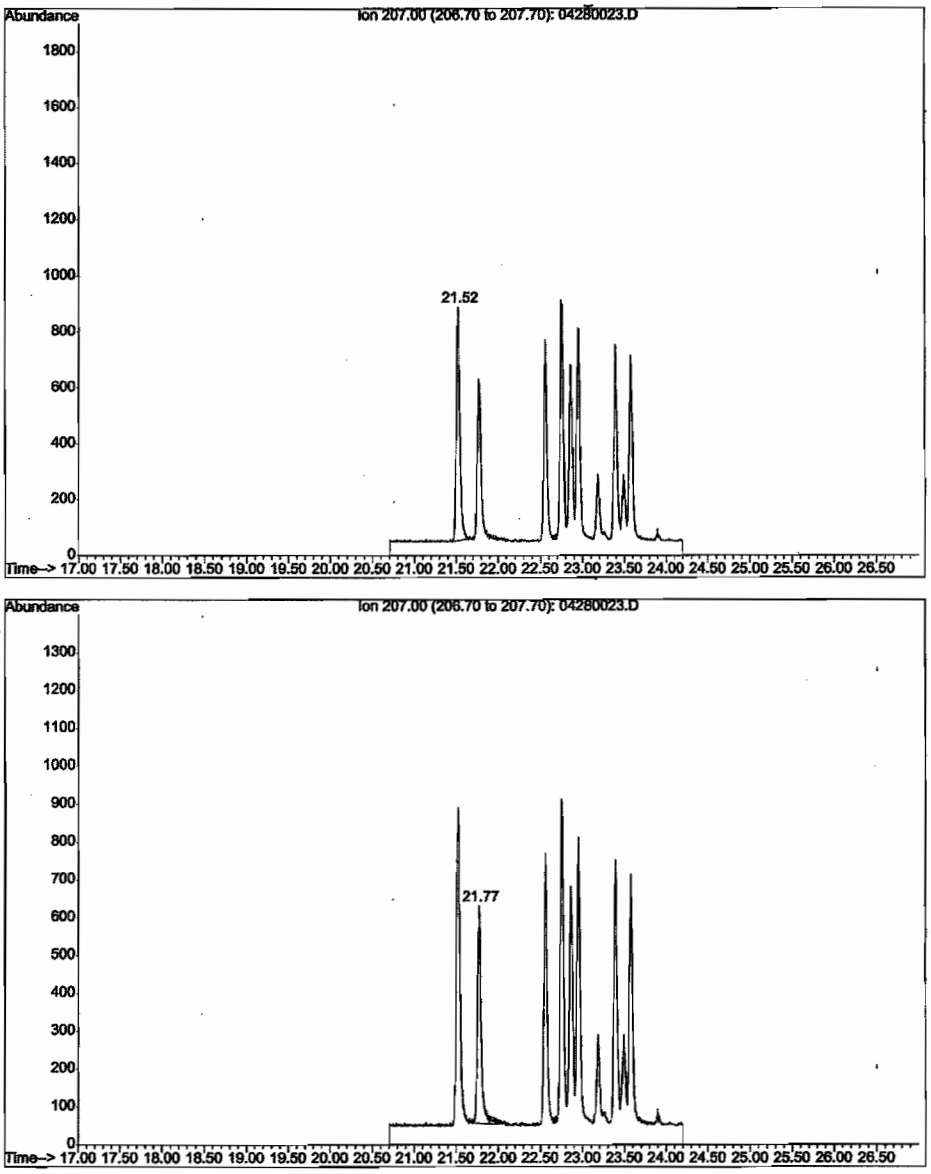
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin.



Lambda-cyhalothrin
10 ng/mL
Peak Response (area): 81386

Figure 28. (con't) Calibration Standards @ 10/100 ng/mL

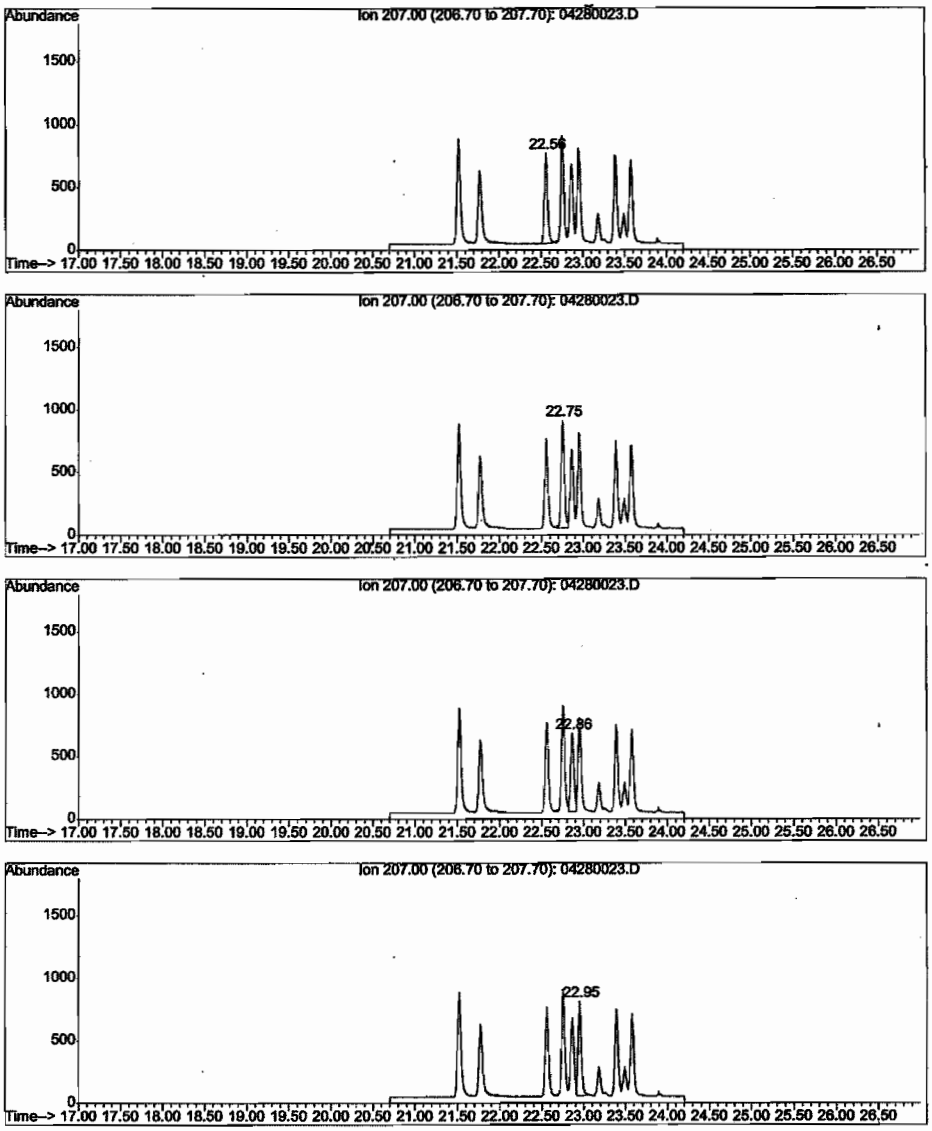
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin.



Permethrin
100 ng/mL
Peak Response (area): 42339

Figure 28. (con't) Calibration Standards @ 10/100 ng/mL

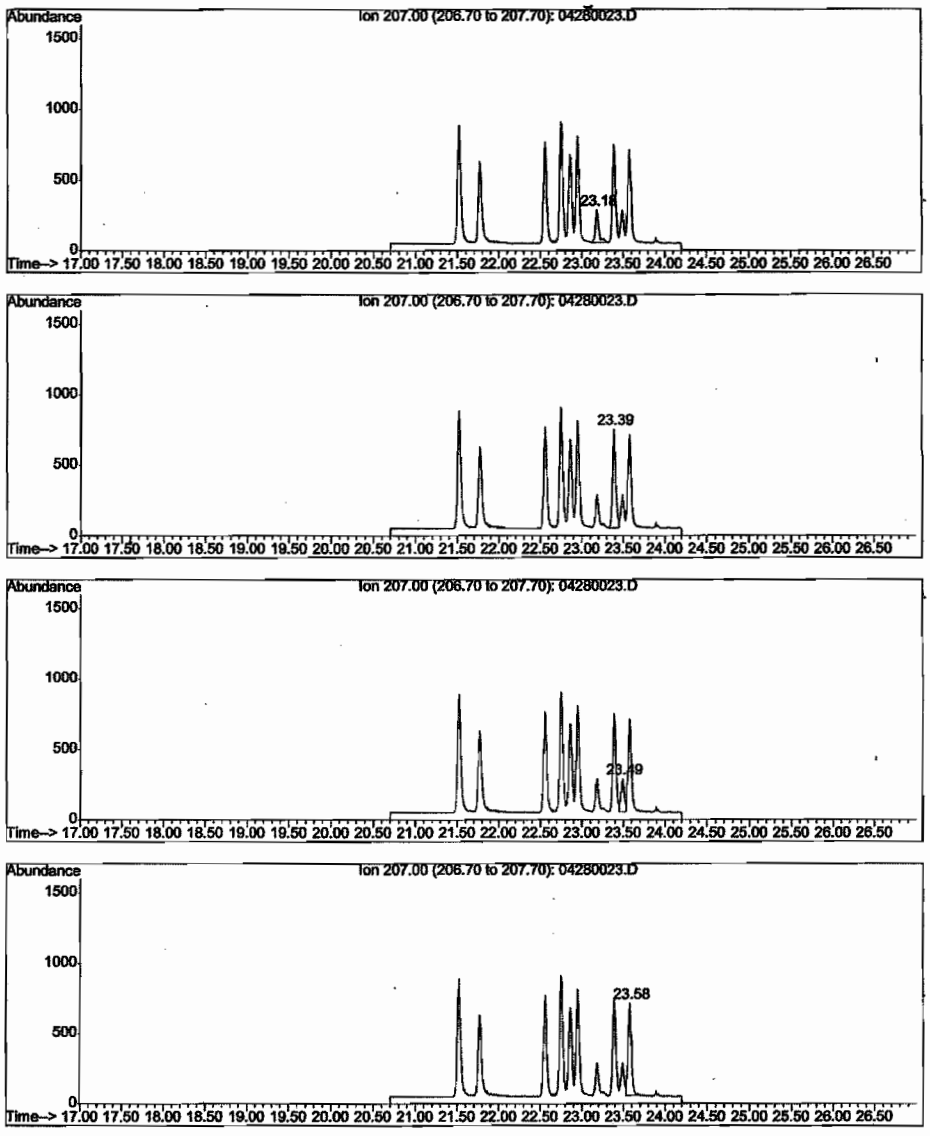
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin.



Cyfluthrin
10 ng/mL
Peak Response (area): 79513

Figure 28. (con't) **Calibration Standards @ 10/100 ng/mL**

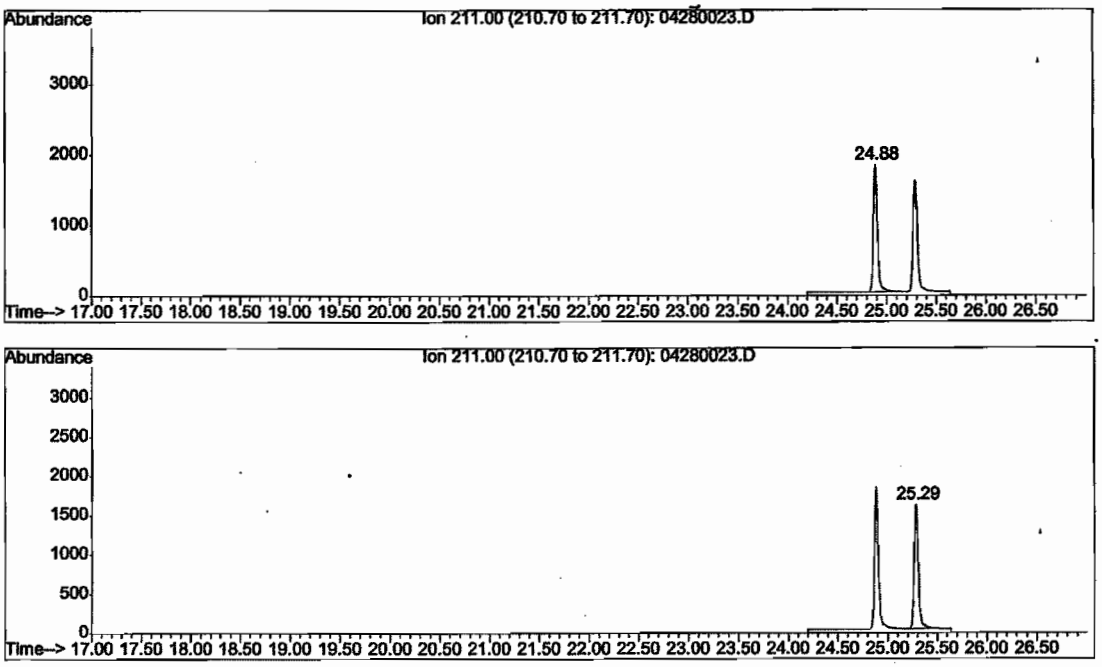
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin.



Cypermethrin
10 ng/mL
Peak Response (area): 50030

Figure 28. (con't) **Calibration Standards @ 10/100 ng/mL**

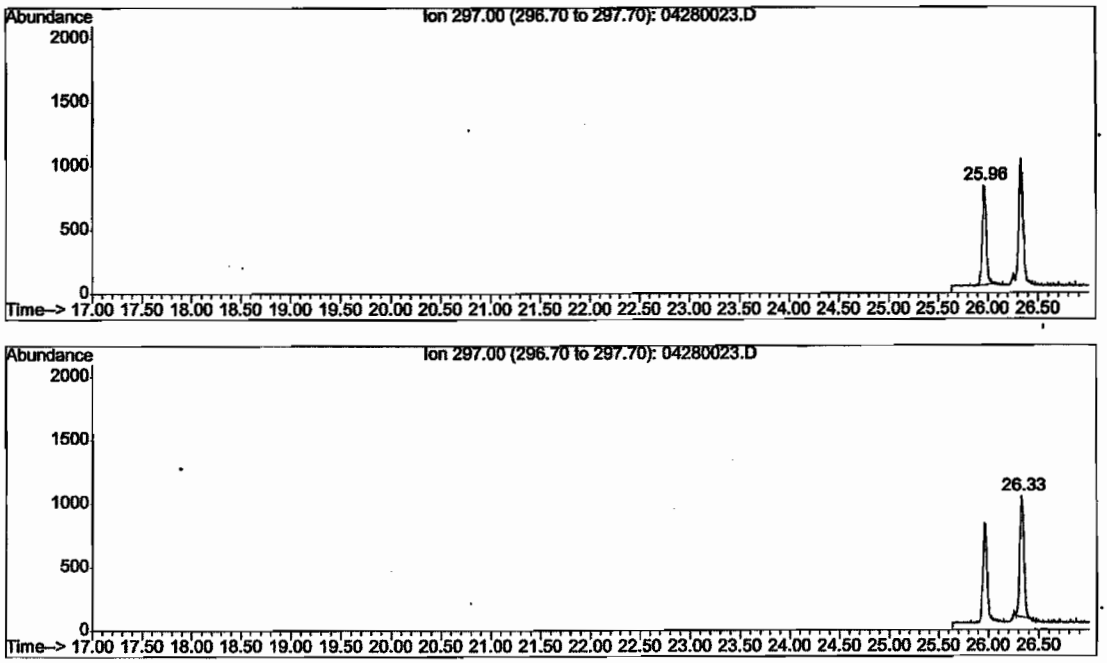
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin.



Esfenvalerate
10 ng/mL
Peak Response (area): 99991

Figure 28. (con't) **Calibration Standards @ 10/100 ng/mL**

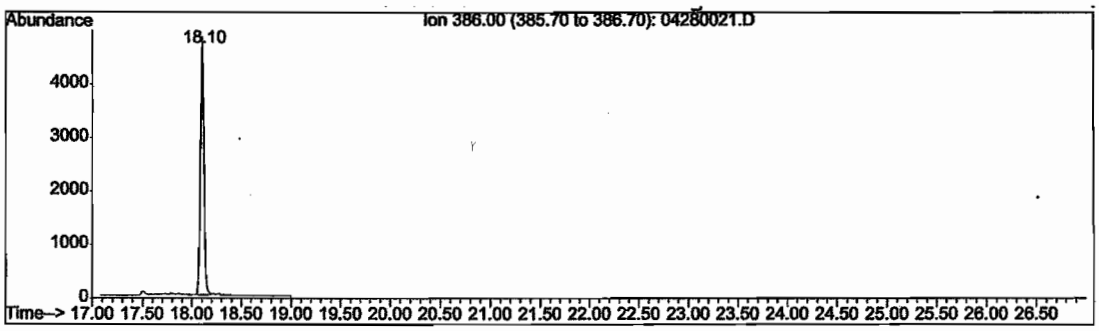
Pyrethroids, standard mix @ 10 ng/mL for all analytes except Permethrin; 100 ng/mL for Permethrin.



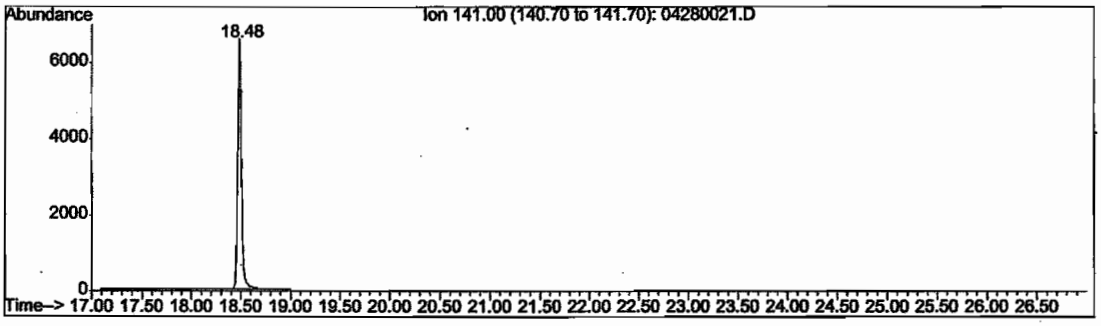
Deltamethrin
10 ng/mL
Peak Response (area): 47234

Figure 29. Calibration Standards @ 20/200 ng/mL

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



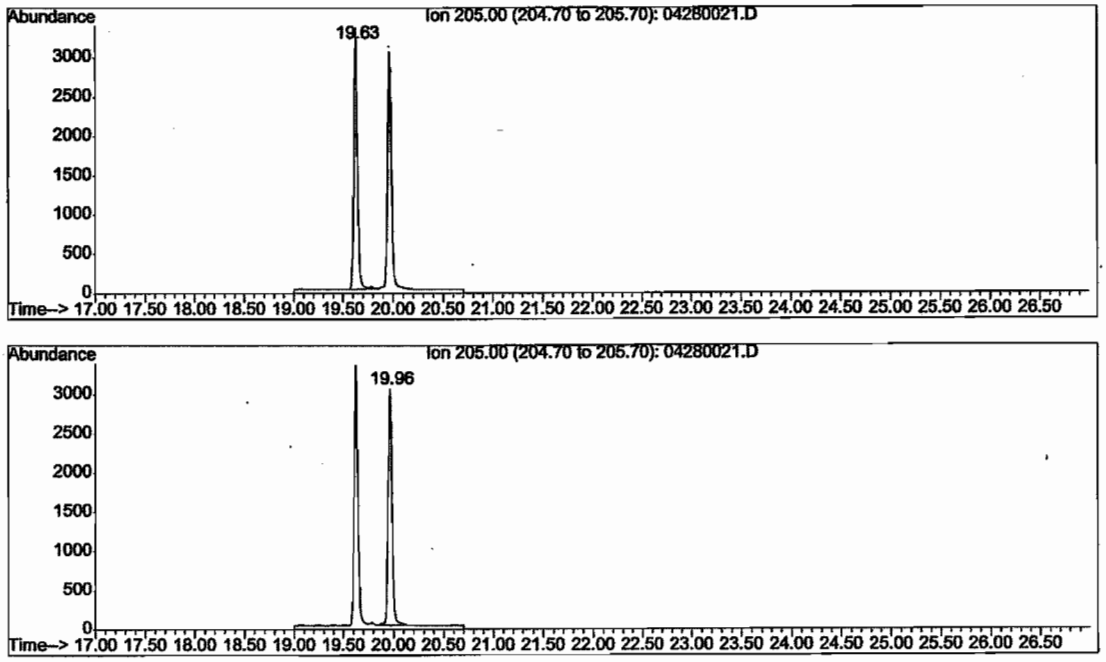
Bifenthrin
20 ng/mL
Peak Response (area): 119698



Fenpropathrin
20 ng/mL
Peak Response (area): 180408

Figure 29.(con't) Calibration Standards @ 20/200 ng/mL

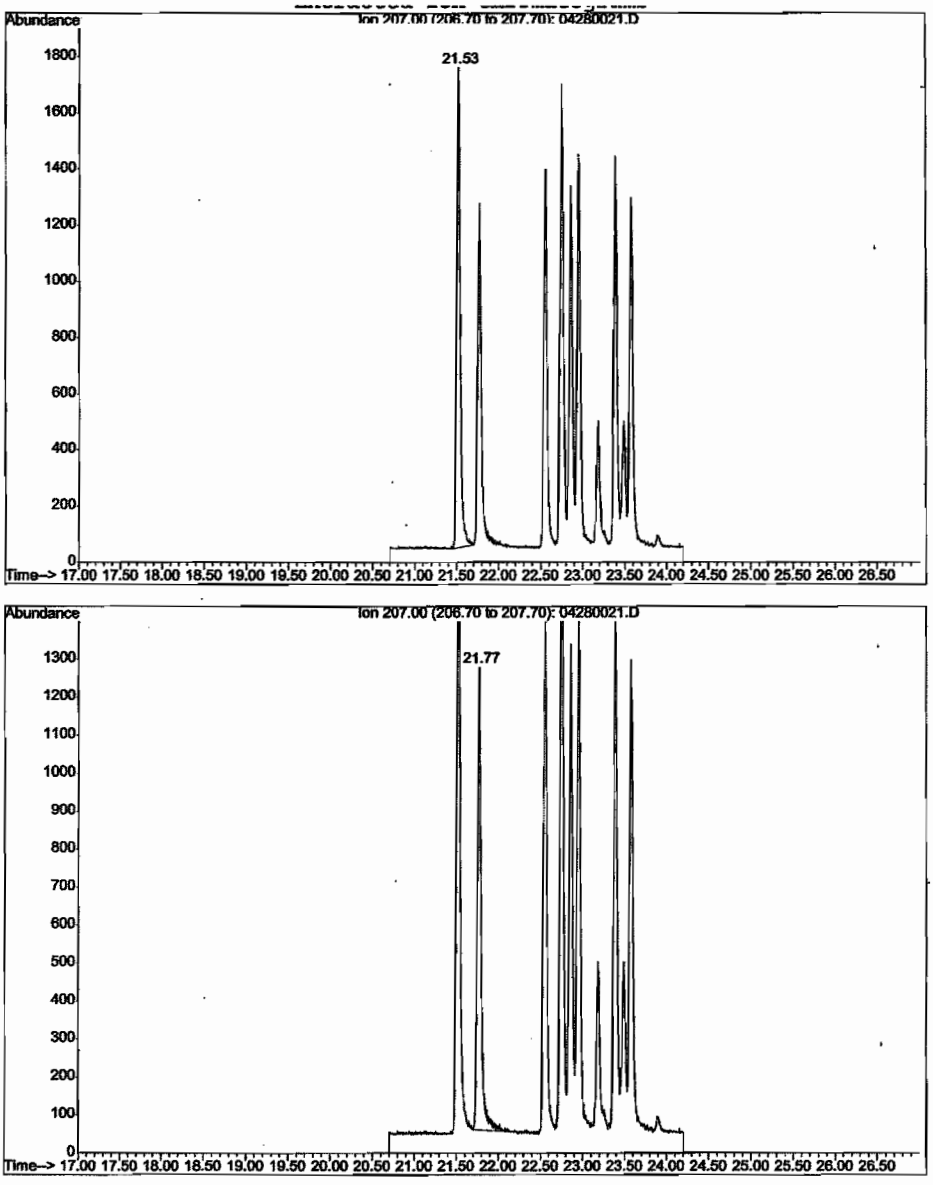
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Lambda-cyhalothrin
20 ng/mL
Peak Response (area): 165637

Figure 29.(con't) Calibration Standards @ 20/200 ng/mL

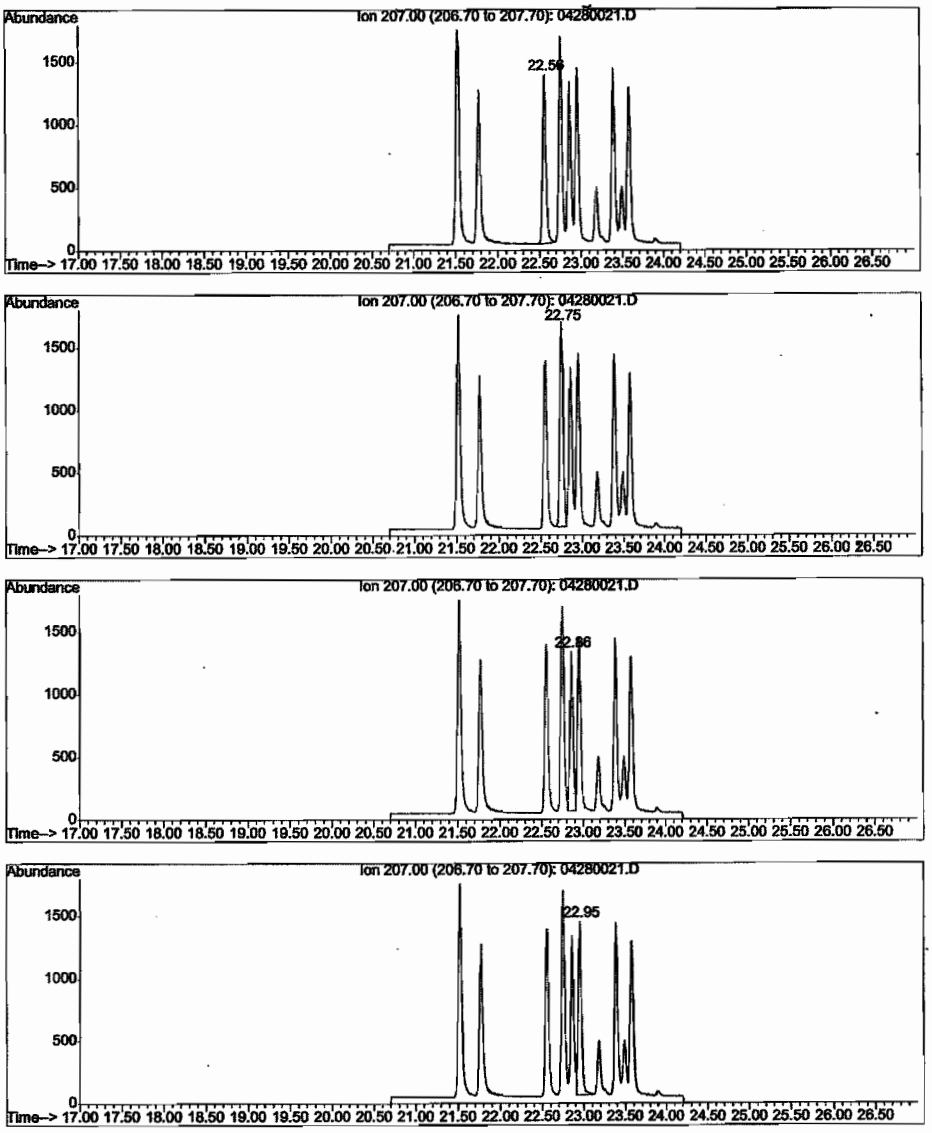
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Permethrin
200 ng/mL
Peak Response (area): 88355

Figure 29.(con't) Calibration Standards @ 20/200 ng/mL

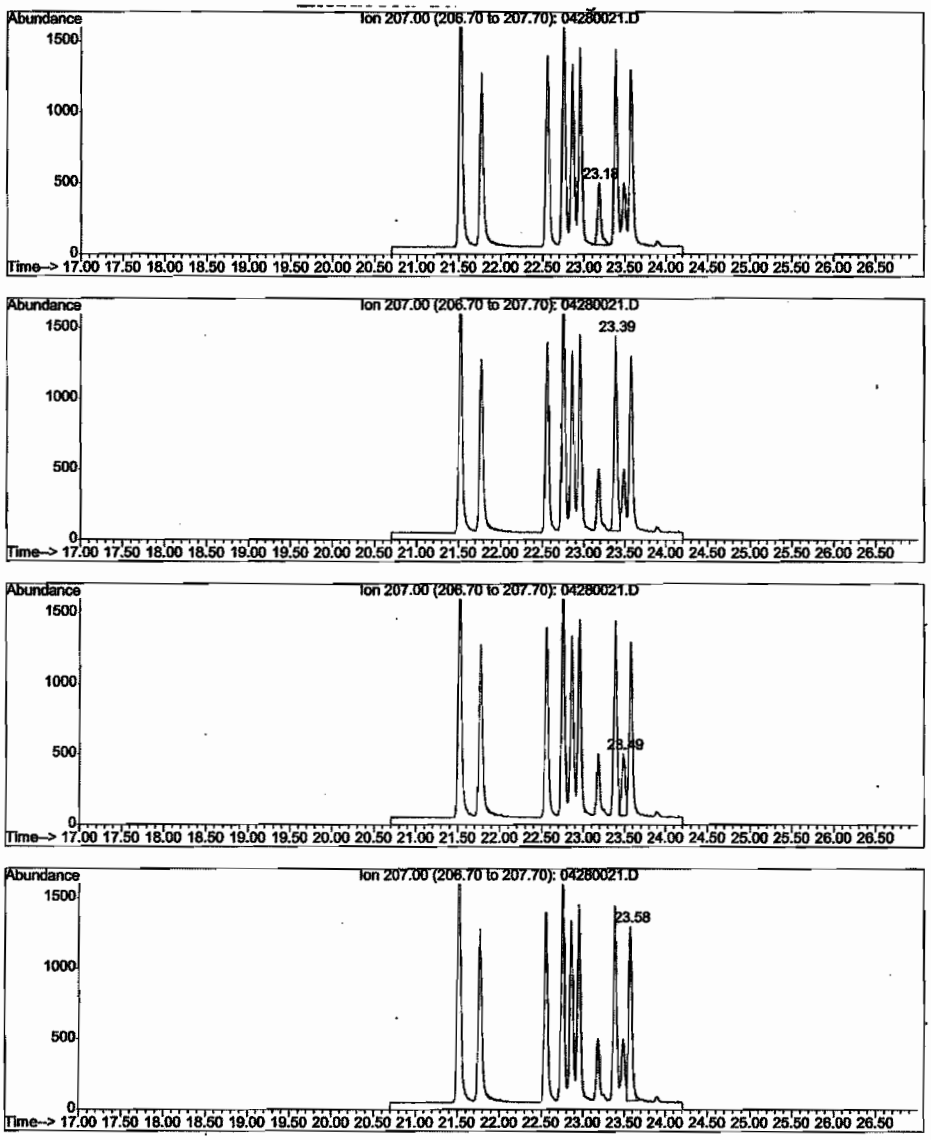
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cyfluthrin
20 ng/mL
Peak Response (area): 156003

Figure 29.(con't) Calibration Standards @ 20/200 ng/mL

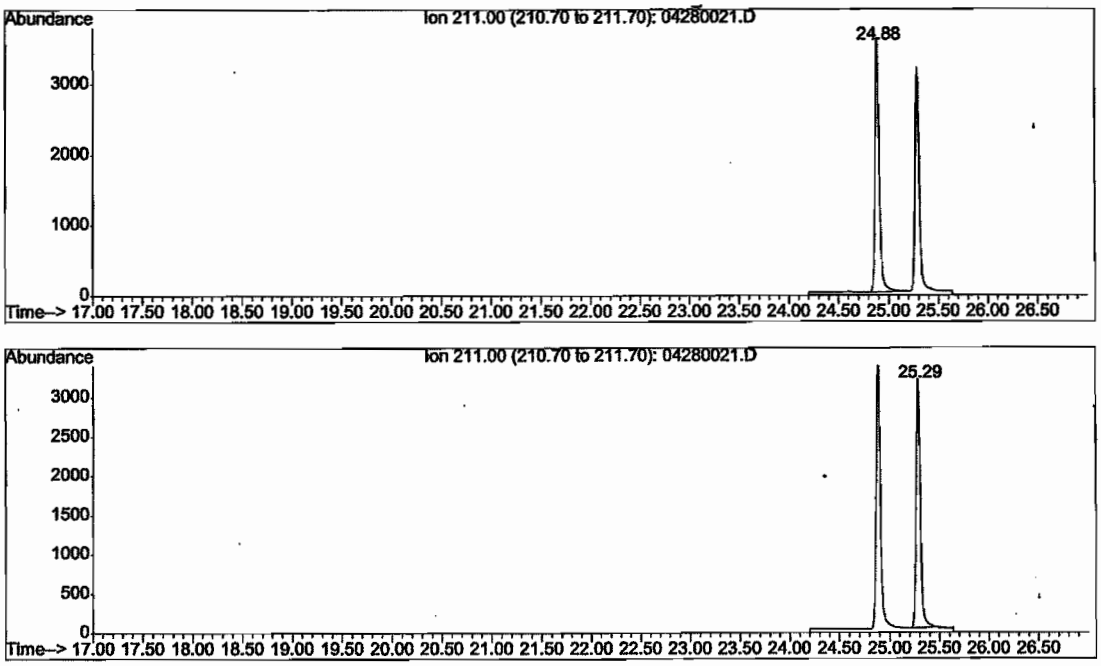
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Cypermethrin
20 ng/mL
Peak Response (area): 97707

Figure 29.(con't) Calibration Standards @ 20/200 ng/mL

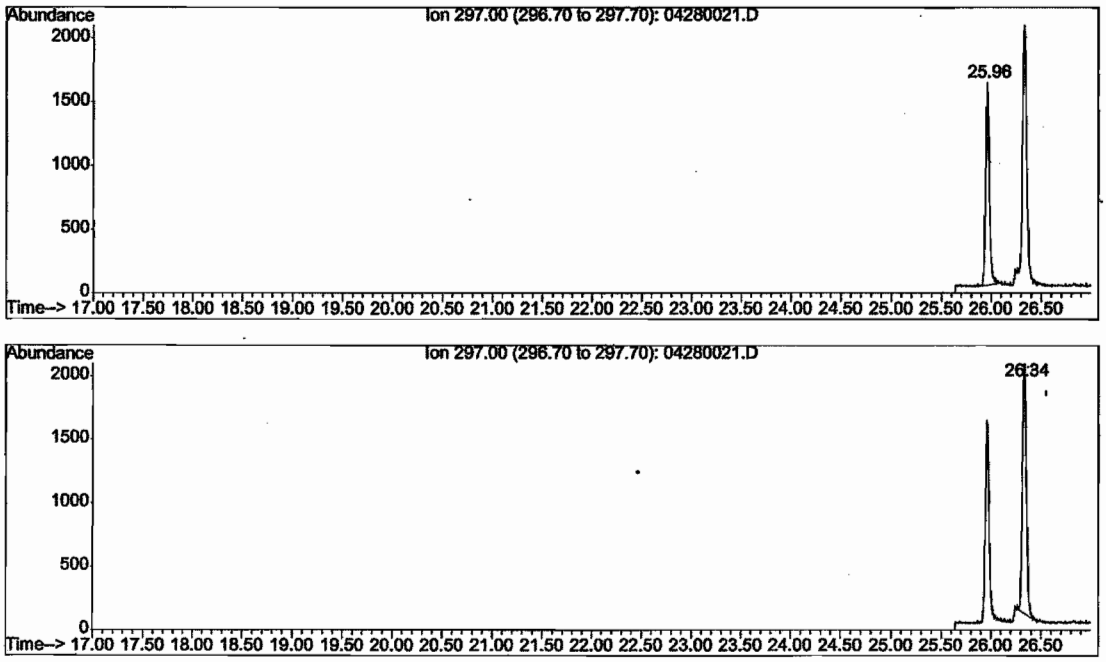
Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Esfenvalerate
20 ng/mL
Peak Response (area): 197023

Figure 29.(con't) Calibration Standards @ 20/200 ng/mL

Pyrethroids, standard mix @ 20 ng/mL for all analytes except Permethrin; 200 ng/mL for Permethrin.



Deltamethrin
20 ng/mL
Peak Response (area): 96652

Appendix 5 : Detector Linearity Graphs

Figure 30 : GC-MSD Calibration Graph for Bifenthrin

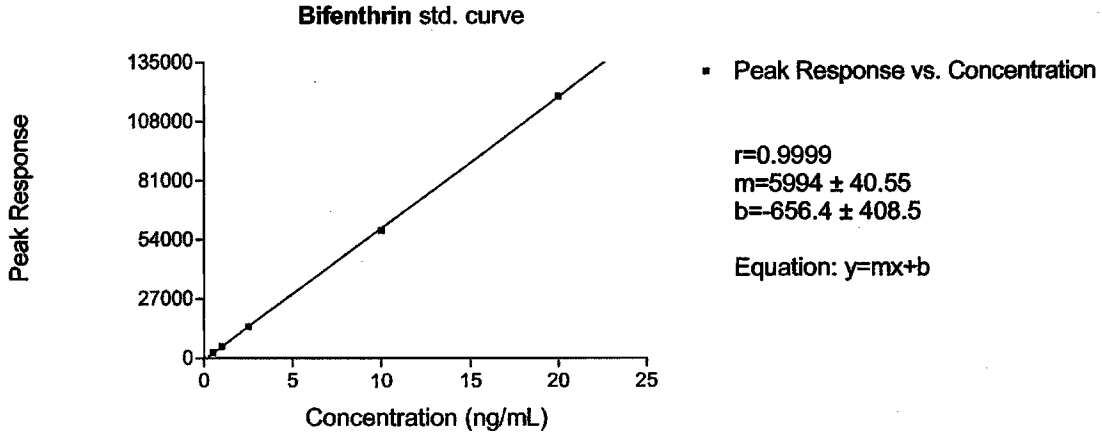


Figure 31 : GC-MSD Calibration Graph for Fenpropathrin

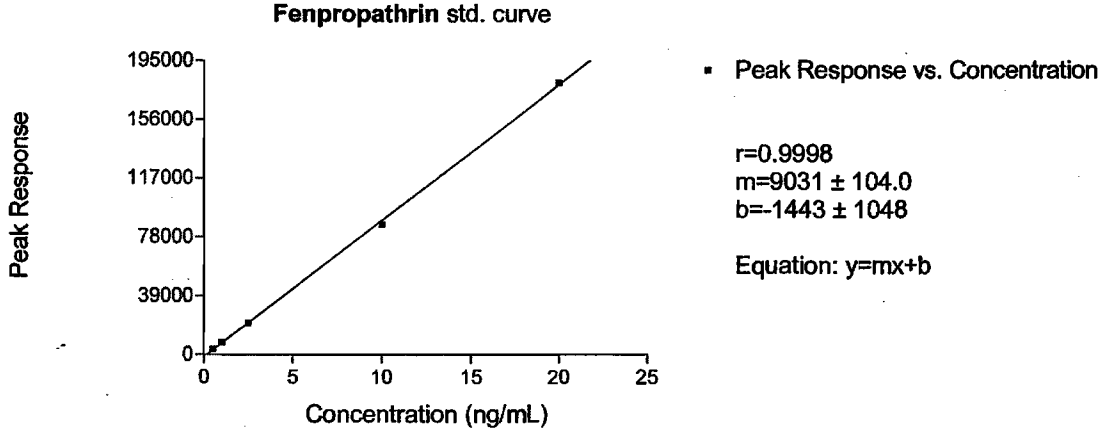


Figure 32 : GC-MSD Calibration Graph for Lambda-cyhalothrin

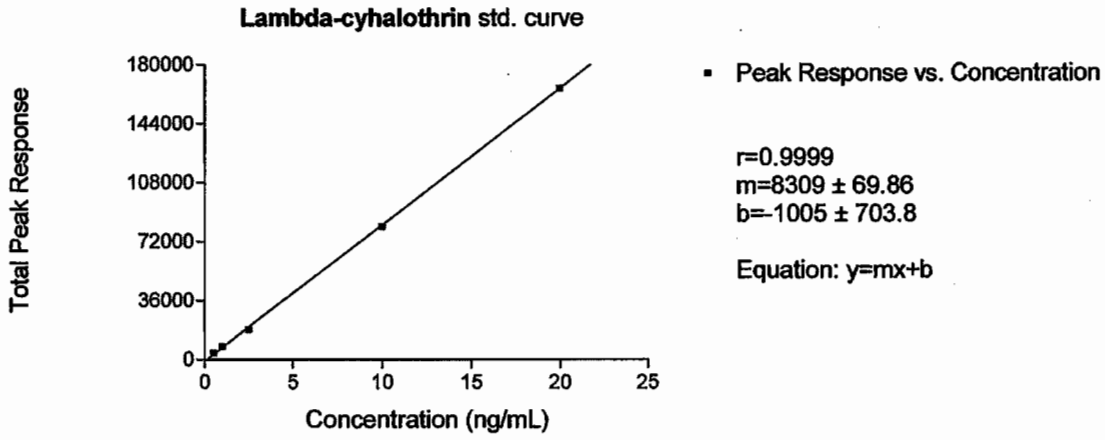


Figure 33: GC-MSD Calibration Graph for Permethrin

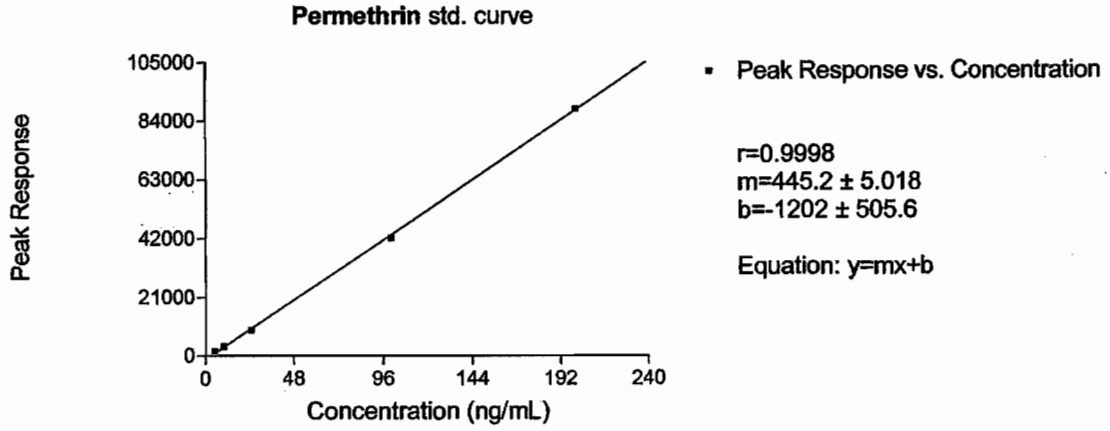


Figure 34 : GC-MSD Calibration Graph for Cyfluthrin

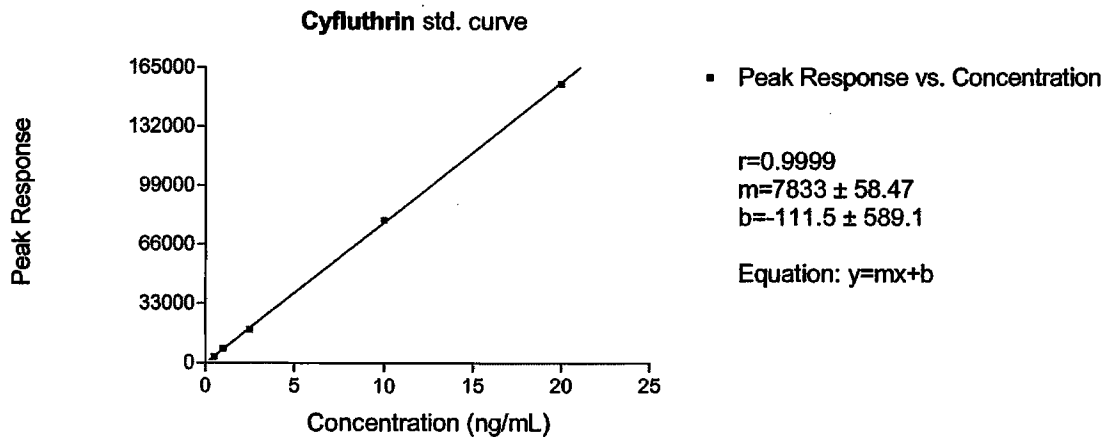


Figure 35 : GC-MSD Calibration Graph for Cypermethrin

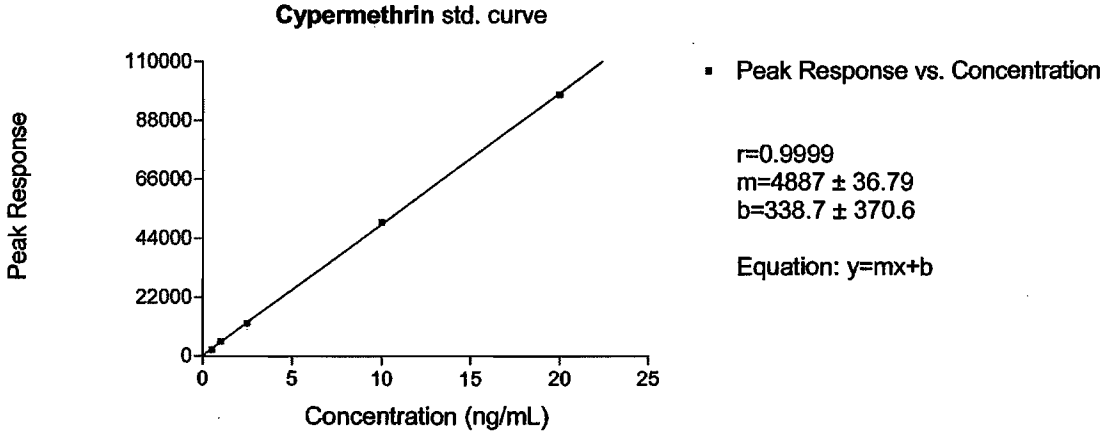


Figure 36 : GC-MSD Calibration Graph for Esfenvalerate

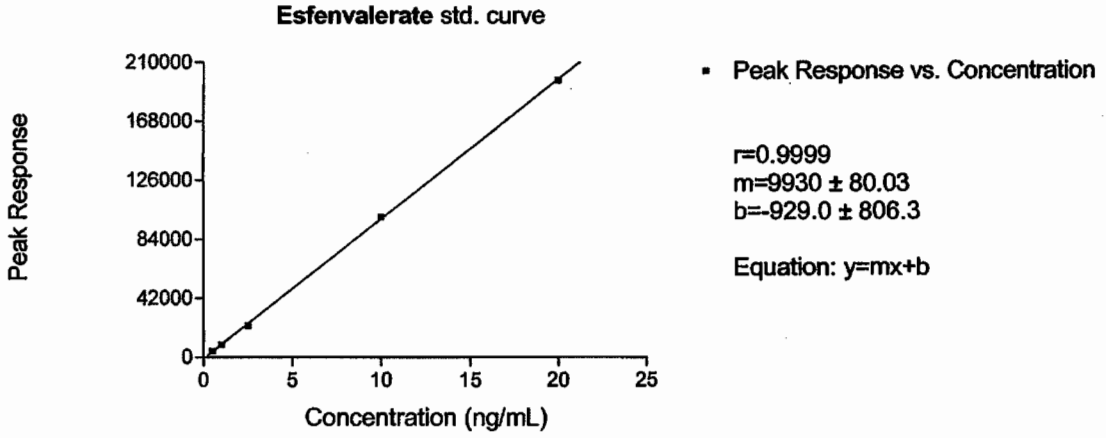
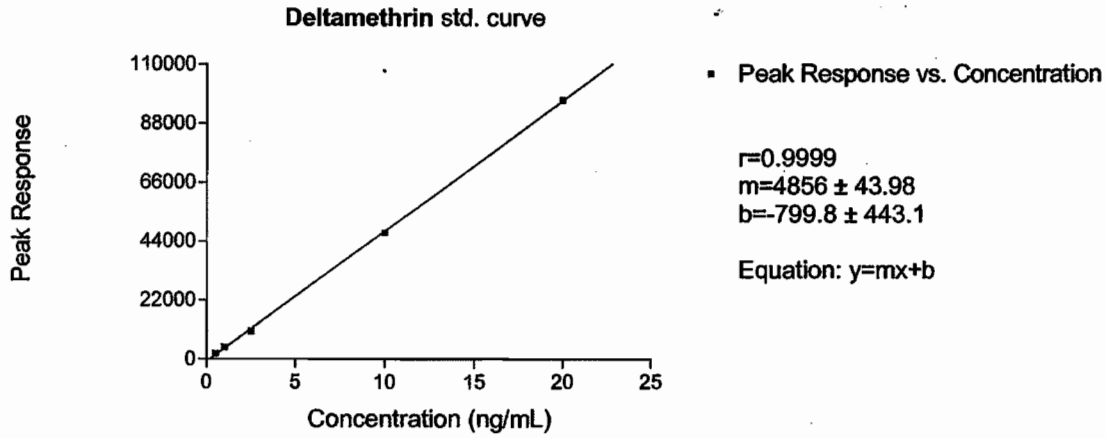


Figure 37 : GC-MSD Calibration Graph for Deltamethrin



Appendix 6 : GC-MS/NICI Full Scan Spectra

Figure 38 : Bifenthrin

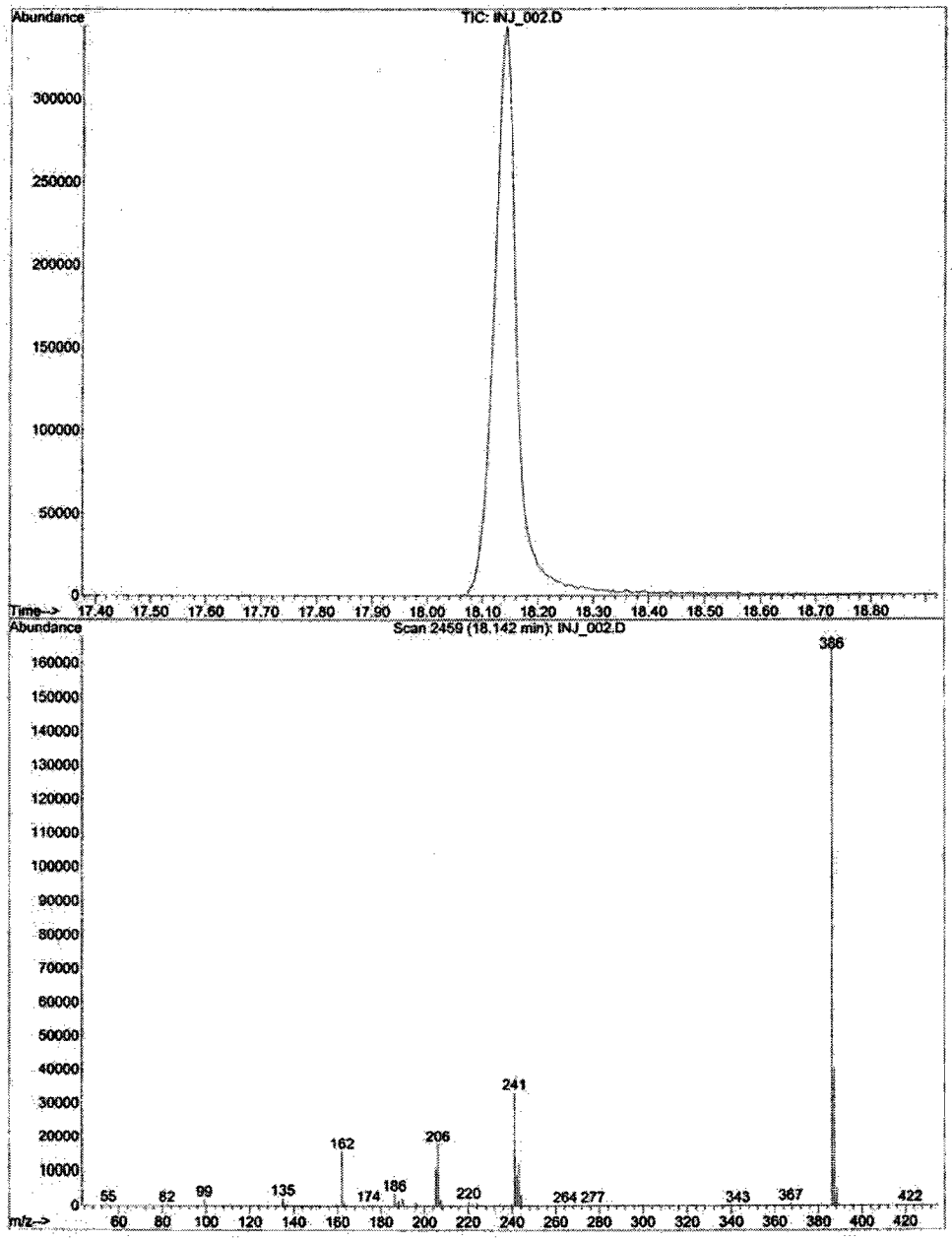


Figure 39 : Fenpropathrin

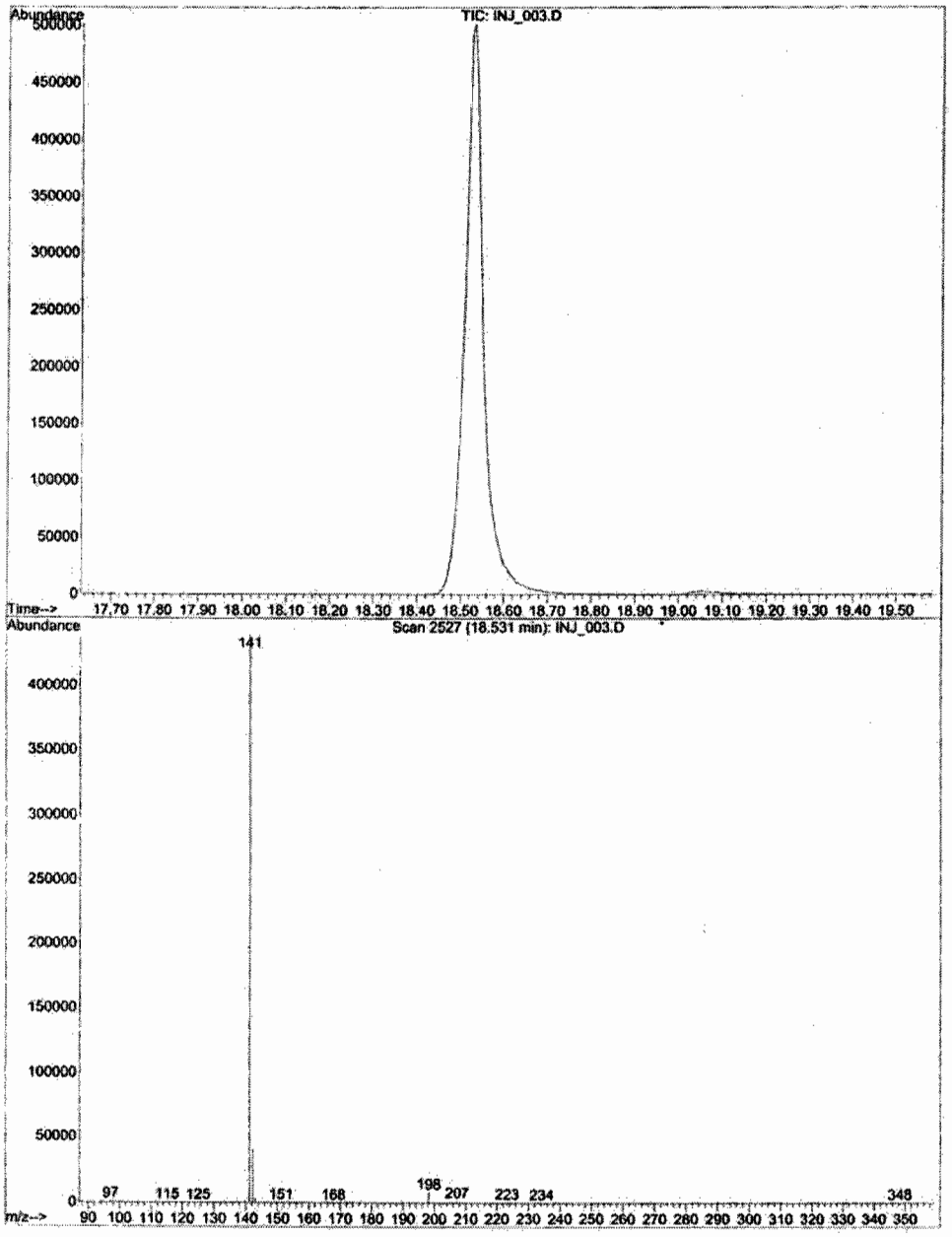


Figure 40 : Lambda-cyhalothrin

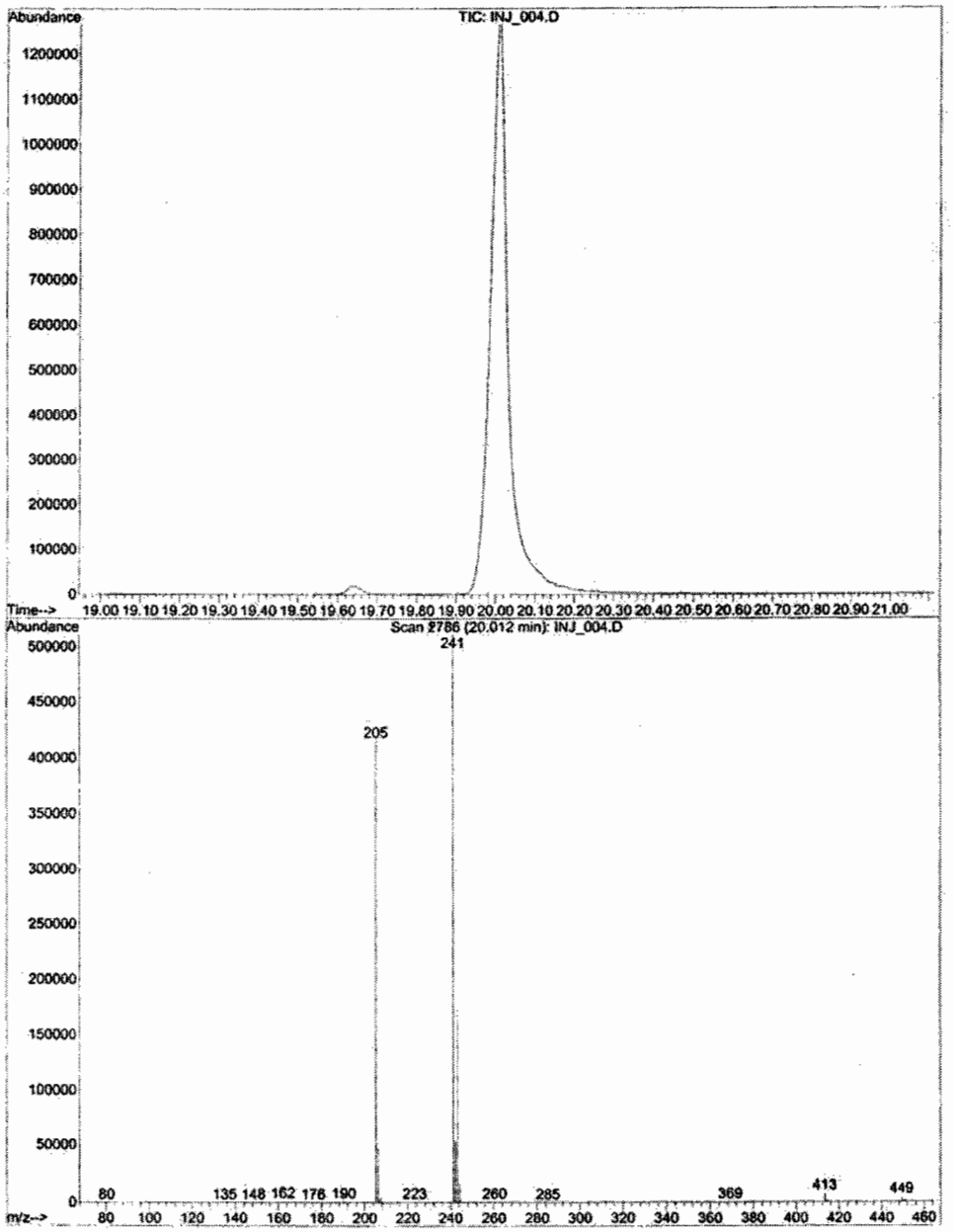


Figure 41 : Permethrin

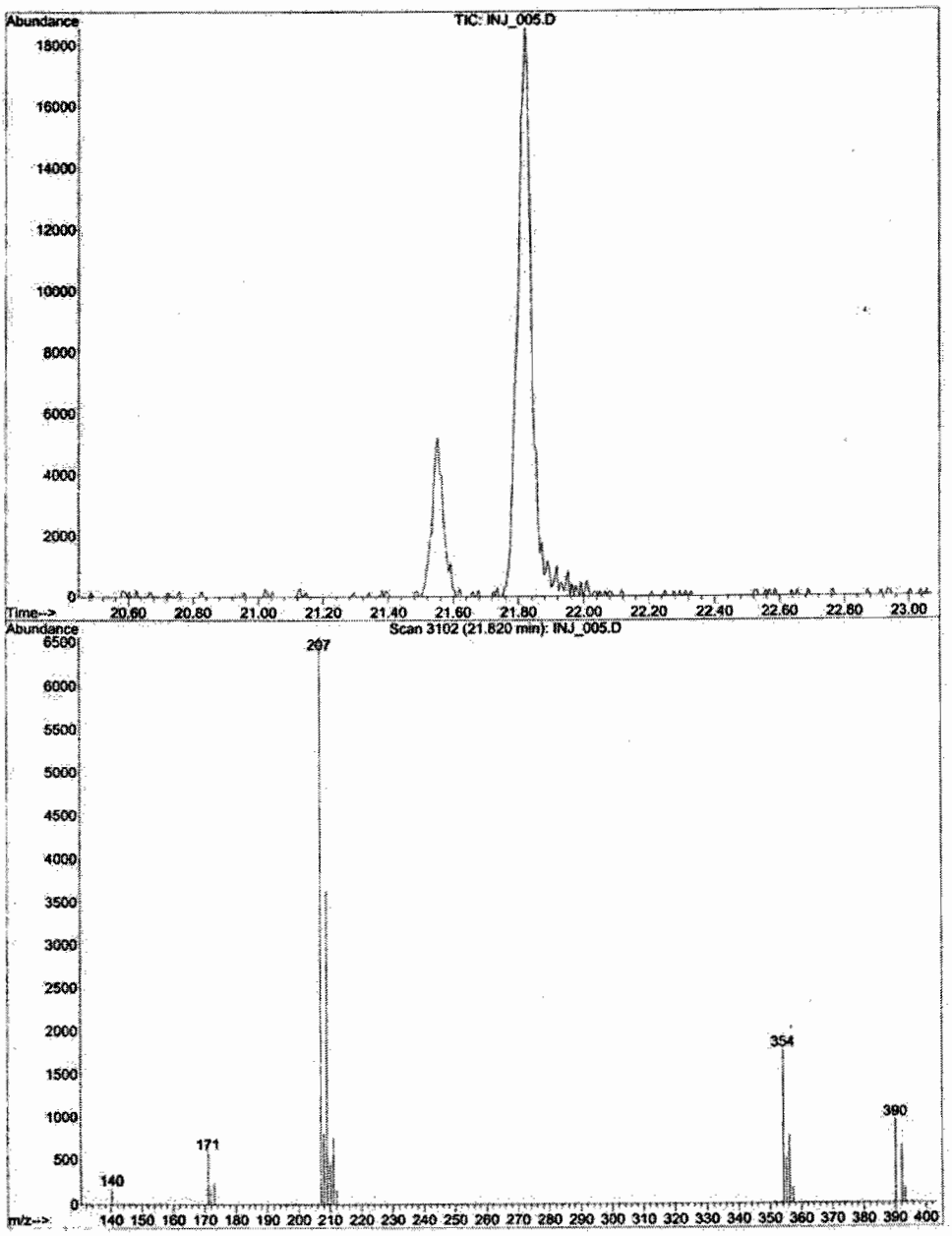


Figure 42 : Cyfluthrin

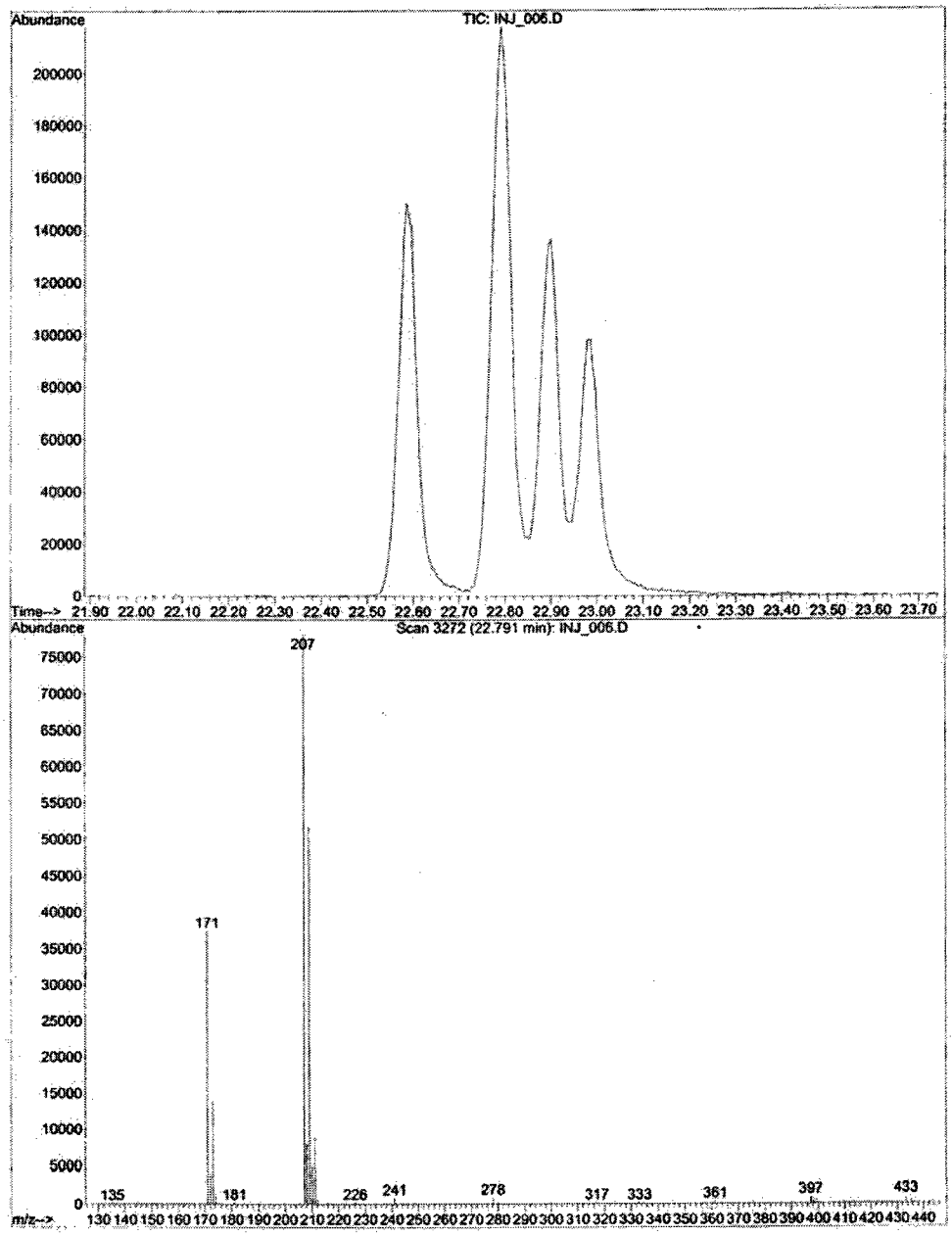


Figure 43 : Cypermethrin

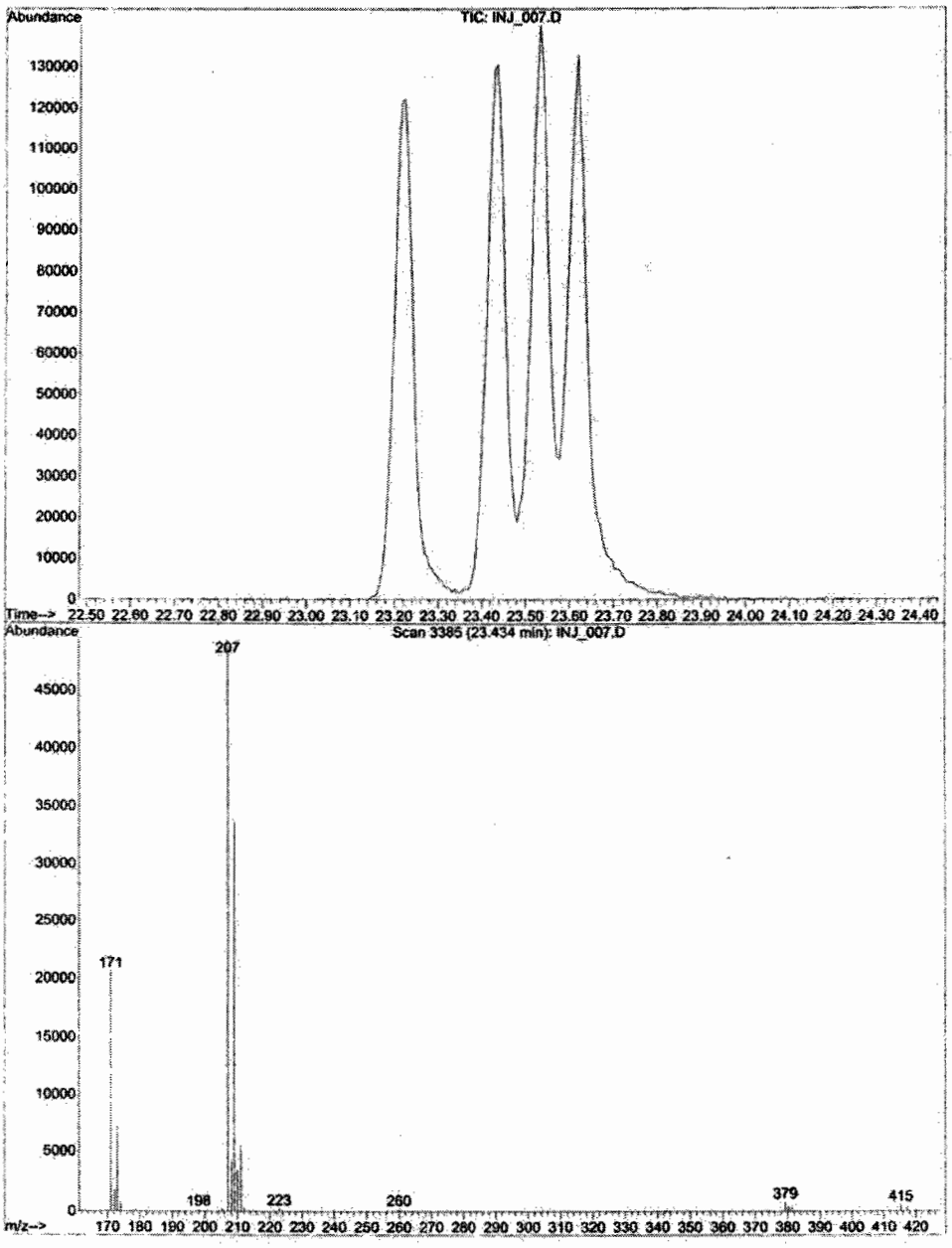


Figure 44 : Esfenvalerate

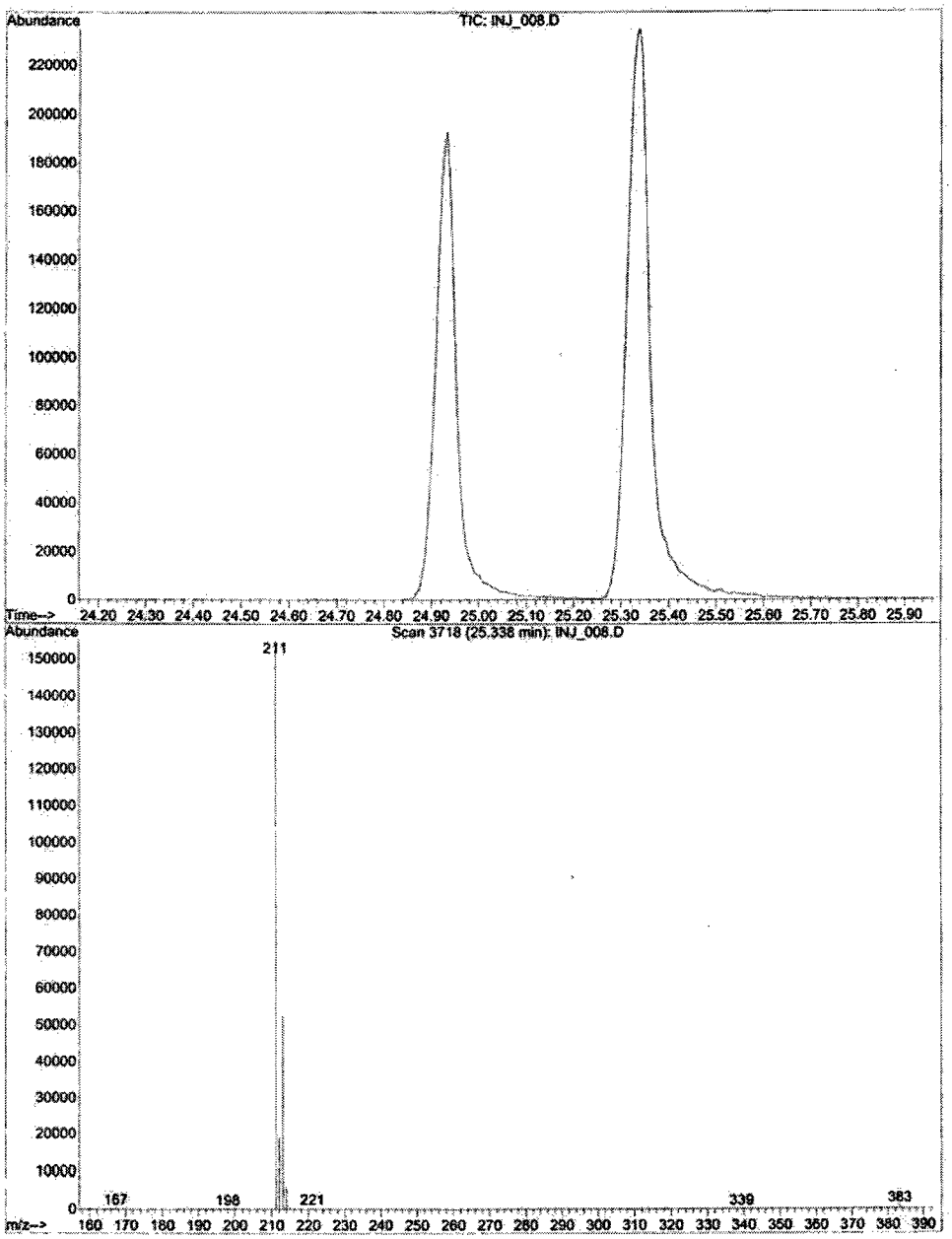
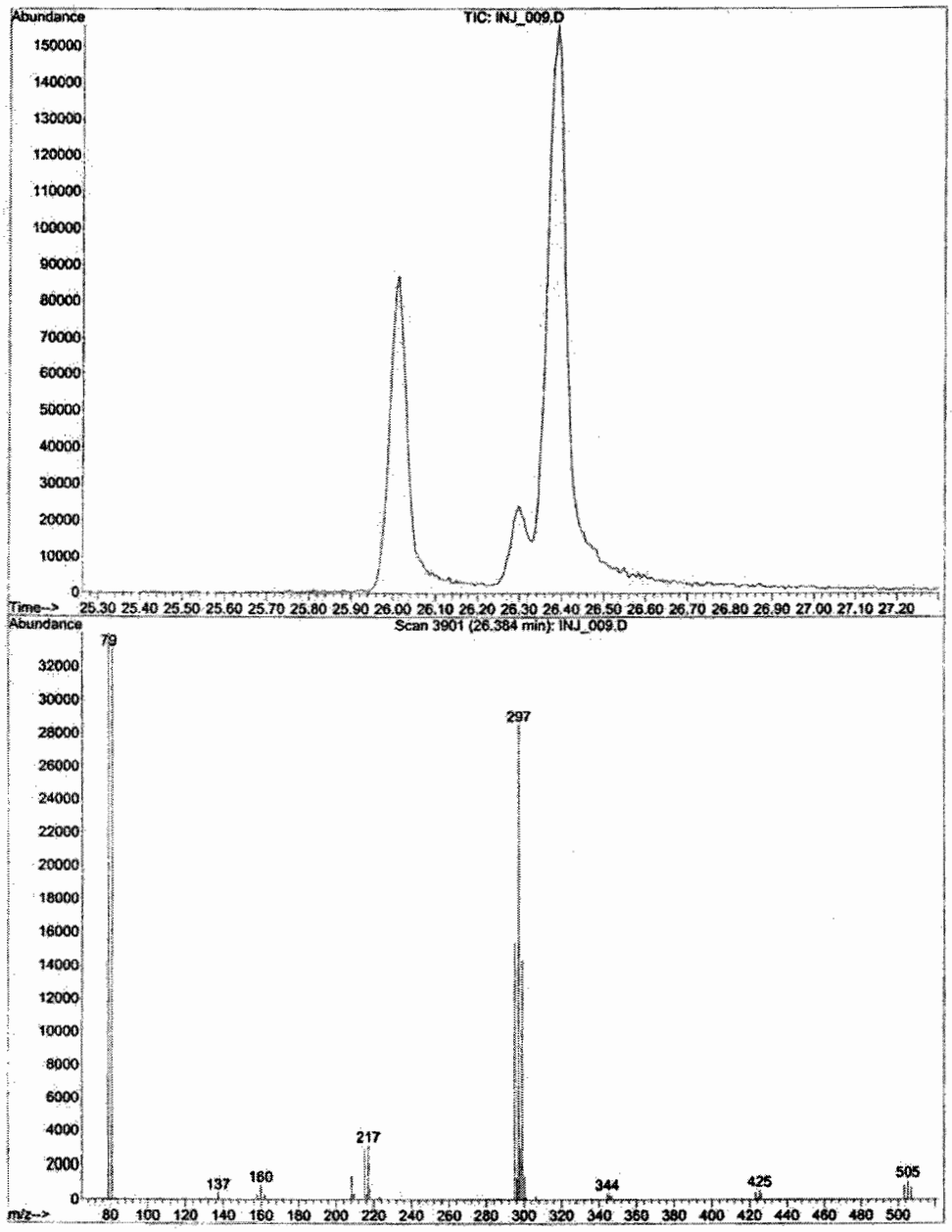


Figure 45 : Deltamethrin



APPENDIX 2. Protocol and Amendments

PROTOCOL

STUDY TITLE

Validation of the Residue Analytical Method:
"Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment," Draft

DATA REQUIREMENT

EPA: Ecological Effects Test Guidelines
OPPTS 850.7100 Data Reporting for Environmental Chemistry Methods

SPONSOR

Pyrethroid Working Group
c/o Fred Pearson
Chair PWG Coordination Committee
Syngenta Crop Protection
410 Swing Road
Greensboro, NC 27409

STUDY DIRECTOR

Richard L. Reed II
Morse Laboratories, Inc.

PERFORMING LABORATORY

Morse Laboratories, Inc.
1525 Fulton Ave.
Sacramento, CA 95825

STUDY IDENTIFICATION

Protocol No.: MLI-06-02
Project No.: ML06-1286-PWG

SIGNATURES

Study Director:

Richard L. Reed II
Morse Laboratories, Inc.

Date: April 12, 2006

Laboratory Director:

Gary L. Westberg
Morse Laboratories, Inc.

Date: Apr 12, 2006

Quality Assurance:

Sherita Normington
Morse Laboratories, Inc.

Date: April 12 2006

Study Monitor:

N. Robinson
Neil Robinson
Syngenta Crop Protection, Inc.
Jealott's Hill Research Centre
Bracknell, Berkshire
RG42 6EY, UK

Date: April 17, 2006

Sponsor Representative:

Fred Pearson
Chair PWG Coordination Committee

Date: April 11 2006

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1 GENERAL INFORMATION

Study Title:	Validation of the Residue Analytical Method: "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment," Draft
Sponsor:	Pyrethroid Working Group c/o Fred Pearson Chair PWG Coordination Committee Syngenta Crop Protection 410 Swing Road Greensboro, NC 27409 USA
Study Monitor:	Neil Robinson Syngenta Crop Protection, Inc. Jealott's Hill Research Centre Bracknell, Berkshire RG42 6EY, UK
Testing Facility:	Morse Laboratories, Inc. 1525 Fulton Ave. Sacramento, CA 95825 USA
Testing Facility Management:	Gary L. Westberg Laboratory Director Morse Laboratories, Inc.
Study Director:	Richard L. Reed II Morse Laboratories, Inc.
Protocol No.:	MLI-06-02
Morse Laboratories Project No.:	ML06-1286-PWG
Test Items:	Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin, and Permethrin
Test System:	Sediment

2 RESPONSIBILITIES

Project Staff

Study Director: Richard L. Reed II

Quality Assurance

Head of Quality Assurance: Sherita Normington

3 PURPOSE OF STUDY

The purpose of this study is to validate an analytical method developed by Syngenta Crop Protection on behalf of the Pyrethroid Working Group for the determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin and permethrin in sediment samples.

A limit of quantitation (LOQ) of 0.1 µg/kg for all targeted analytes except permethrin, which is 1.0 µg/kg, and a working range from 0.1 µg/kg to 1.0 µg/kg for all targeted analytes except permethrin, which is from 1.0 µg/kg to 10 µg/kg, will be validated in sediment. The validation will be performed on two different types of sediment samples (freshwater and estuarine) collected from sites in California.

4 GUIDELINES

This study will be performed in compliance with :

EPA: Ecological Effects Test Guidelines
OPPTS 850.7100-Data Reporting for Environmental Chemistry Methods

5 GOOD LABORATORY PRACTICE

The study will be performed in compliance with EPA's Good Laboratory Practice Standards as specified in 40 CFR 160. The Study Director is responsible for the overall conduct of the study.

6 AMENDMENT PROCEDURES

This protocol can be amended by mutual consent of the Study Director and the Study Monitor. Detailed descriptions of all amendments will be signed by the Study Director, Management and the Study Monitor. The amendment will be

effective at the time of the Study Director's signature. The original amendment(s) will be maintained with the original protocol in the study file(s).

7 DEVIATION PROCEDURES

Unplanned changes to the protocol will be detailed in the raw data, signed and dated by the Study Director. These deviations, both protocol and SOP, will be evaluated by the Study Director and the appropriate documentation will be issued if necessary. The procedure section of the report will reflect the deviations.

8 TEST SYSTEM JUSTIFICATION

The matrix employed in this method validation is representative of the matrix this method was designed to analyze.

9 SCHEDULE

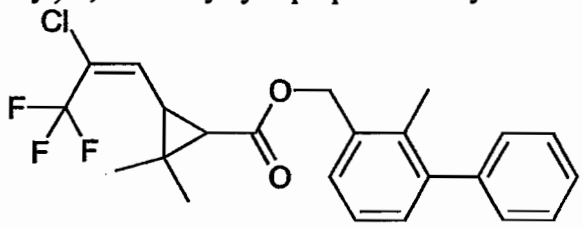
Estimated Start of Experiments:	April, 2006
Estimated Completion of Experiments:	May, 2006
Estimated Date of Final Report:	June, 2006

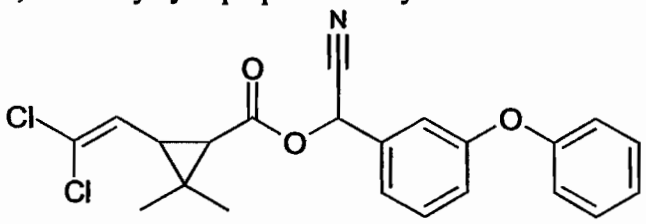
10 MATERIALS AND METHODS

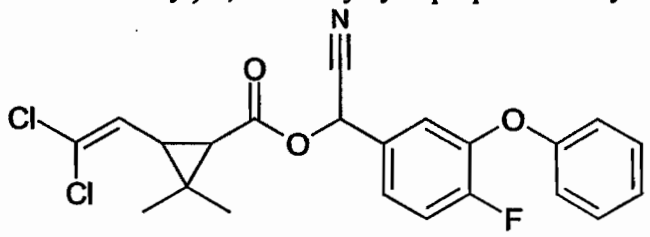
10.1 Test Items

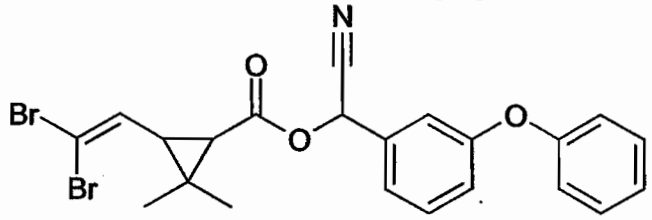
The test items and the information concerning the test items are provided by the Pyrethroid Working Group.

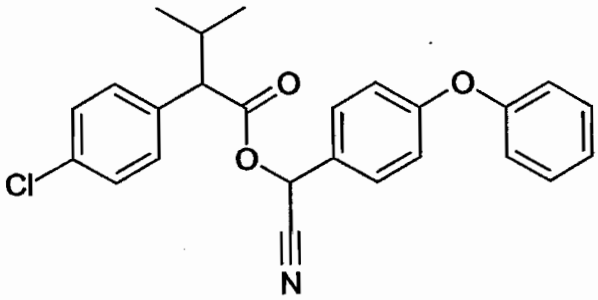
The test items are:

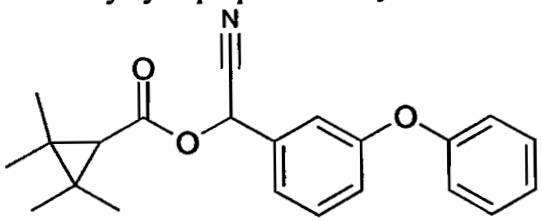
Compound	Bifenthrin
IUPAC Name:	2-methylbiphenyl-3-ylmethyl (*Z*)-(1*RS*,3*RS*)-3-(2-chloro-3-3-3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	82657-04-3
CAS Name:	(2-methyl[1,1'-biphenyl]-3-(2-chloro-3-3-3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	97.8
Lot No.:	BI-29
Source:	FMC Agricultural Products
Expiration Date:	8/2007
Storage:	-8°C to -22°C

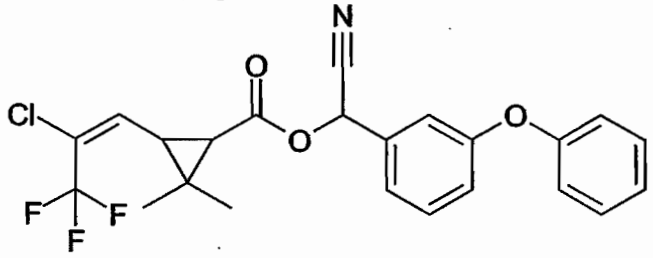
Compound	Cypermethrin
IUPAC Name:	(*RS*)-α-cyano-3-phenoxybenxyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	52315-07-8
CAS Name:	Cyano(3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	99.4
Lot No.:	AMS 202/102
Source:	Syngenta Crop Protection
Expiration Date:	3/2009
Storage:	Ambient

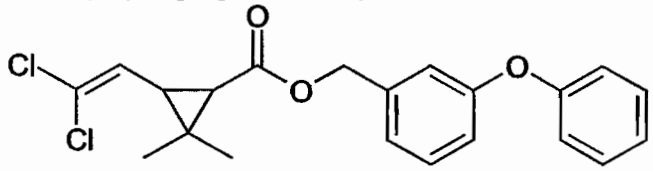
Compound	Cyfluthrin
IUPAC Name:	*RS*)-α-cyano-4-fluoro-3-phenoxybenzyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	68359-37-5
CAS Name:	Cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	50.2
Lot No.:	K-618
Source:	Bayer CropScience
Expiration Date:	12/31/13
Storage:	-8°C to -22°C

Compound	Deltamethrin
IUPAC Name:	(*S*)-α-cyano-3-phenoxybenzyl (1*R*,3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	52918-63-5
CAS Name:	1-[*R*-[1-α(*S**),3α]]-cyano(3-phenoxyphenyl)methyl 3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	99.4
Lot No.:	K-1375
Source:	Bayer CropScience
Expiration Date:	9/8/10
Storage:	-8°C to -22°C

Compound	Esfenvalerate
IUPAC Name:	(*S*)-α-cyano-3-phenoxybenzyl (*S*)-2-(4-chlorophenyl)-3-methylbutyrate
CAS Number:	66230-04-4
CAS Name:	[*S*-(*R**,*R**)]-cyano(3-phenoxyphenyl)methyl 4-chloro-2-(1-methylethyl)benzeneacetate
Structure:	
% Purity:	98.7
Lot No.:	058
Source:	DuPont
Expiration Date:	11/26/07
Storage:	Ambient

Compound	Fenpropathrin
IUPAC Name:	(*RS*)-α-cyano-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate
CAS Number:	64257-84-7
CAS Name:	Cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate
Structure:	
% Purity:	99.7
Lot No.:	AS 459h
Source:	Valent
Expiration Date:	8/5/06
Storage:	-8°C to -22°C

Compound	Lambda-cyhalothrin
IUPAC Name:	A reaction product containing equal quantities of (*S*)-α-cyano-3-phenoxybenzyl (*Z*)-(1*R*,3*R*)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate and (*R*)-α-cyano-3-phenoxybenzyl (*Z*)-(1*R*,3*R*)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	91465-08-6
CAS Name:	[1α(*S**),3α(*Z*)]-(±)-cyano(3-phenoxyphenyl)methyl 3-(2-chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	98.7
Lot No.:	ASJ10012-04
Source:	Syngenta Crop Protection
Expiration Date:	8/2006
Storage:	1°C to 8°C

Compound	Permethrin
IUPAC Name:	3-phenoxybenzyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Number:	52645-53-1
CAS Name:	(3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate
Structure:	
% Purity:	99.7
Lot No.:	S01-2613
Source:	Syngenta Crop Protection
Expiration Date:	7/2006
Storage:	-8°C to -22°C

10.2 Test System

The test system is sediment.

10.3 Characterization

Only percent organic matter and particle size will be required. This data will be provided by the Sponsor.

10.4 Source of Sediment and Identification

One sample of untreated sediment from each of two sites in California, each representing a different type of sediment, will be provided by the Sponsor. A freshwater sediment sample from Butte Creek, Butte Co., CA and an estuarine sediment sample from Belvedere Cove, Marin Co., CA will be submitted to the laboratory.

Upon receipt, the untreated sediment samples will be stored frozen at all times in a temperature-monitored freezer at less than or equal to -15°C except when removed from the freezer to prepare for analysis. Sediment receipt, condition upon receipt, and storage conditions during the validation will be documented in the study records.

The untreated sediment will be identified with a unique label or number to identify the test system, study, nature of study, and the date of collection. Such identification will be either on the sediment container(s) or cross-referenced in the raw data to the sediment as provided by the Sponsor.

10.5 Method of Analysis

The analytical method to be validated in this study is Syngenta Crop Protection analytical method entitled "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment," Draft, by N. J. Robinson.

All modifications to the method after the validation begins shall be documented in the raw data and the final report. Major modifications must be approved through oral or written communication with the Study Monitor.

10.6 Experimental Procedures

10.6.1 Sample processing

Upon receipt, the untreated control sediment sample will be processed for analysis by transferring the entire sample to a large metal bowl, then removing by hand any rocks or plant debris that may be present. The sample will be mixed by hand using a large metal spoon. Following processing, the sample will be divided into several equal portions, with each portion placed into a plastic bag. The bagged portions will be placed into frozen storage (at less than or equal to -15 °C) and remain there until removed for use at a later date.

10.6.2 Fortification procedures

Aliquots of untreated control sample will be fortified with microliter amounts of appropriate mixed or individual standard solutions of the test items. During actual fortification, the standard solution(s) will be evenly distributed over the exposed sample as much as possible.

Subsamples from each type of untreated control sediment sample will be fortified according to the following scheme:

Matrix	Sample Type	Fortifying Compounds	Fortification Level	# of Samples
None	Reagent blank	None	0.0 mg/kg	1
Sediment	Control	None	0.0 mg/kg	2
Sediment	Spike	All targeted analytes except Permethrin	0.1 µg/kg (LOQ) and Permethrin 1.0 µg/kg (LOQ)	5
Sediment	Spike	All targeted analytes except Permethrin	1.0 µg/kg (10 x LOQ) and Permethrin 10 µg/kg (10 x LOQ)	5

10.6.3 Analysis scheme

All samples from one sediment type will be analyzed in one sample set.

10.6.4 Calibration procedure

The instrument calibration standards (mixed) for this study will be prepared by making appropriate dilutions of stock standard solutions of the test items. The standard solution mixes will bracket the working range. The lowest standard of the working range will be no less than half the concentration of the lowest sample concentration expected.

Calibration standards and samples will be injected according to the schemes outlined in the method. Regardless of the scheme used, a calibration curve (minimum 4 points) for each analyte will be constructed to demonstrate appropriate system linearity.

Determination and quantitation of the targeted analyte residues will be conducted using gas chromatography (GC) employing negative ion chemical ionization mass selective detection (MSD).

11 EVALUATION OF RESULTS

The quantity of the residues in each sample shall be determined in accordance with the provisions of the analytical method, using either of the two calculation options provided.

Use of alternate regression approaches, which may be more appropriate for the actual detector employed, is permissible. Other alternate techniques may be used following consultation with the Study Monitor. The actual percent recovery for the analytes will be determined as follows:

$$\%\,Recovery = \frac{\mu g/kg\ found\ in\ fortified\ control}{\mu g/kg\ added} x\ 100$$

Recoveries are not to be corrected for control contribution.

For a successful validation, mean recovery values for each fortification level will be within the range of 70-120%. The relative standard deviation (RSD) of replicate recovery measurements will not exceed the level of ±20% at or above the LOQ. The values found in the control samples will not be higher than 30% of the LOQ. If any of the aforementioned requirements is not met, the Study Director will contact the Study Monitor to determine how to proceed.

12 STATISTICAL METHODS

Only descriptive statistics, including means, standard deviations, and relative standard deviations will be used to evaluate the data.

13 CONTROL OF BIAS

Bias will be controlled by use of representative samples from a homogeneous mixture for analysis and replicate analysis of all fortified samples.

14 ARCHIVING

All original copies of raw data for this study not specific to the operation of Morse Laboratories, Inc. will be shipped directly to an archive facility to be specified by the Sponsor and identified in the final report. When this is not possible, verified exact copies should be used.

15 REPORTING

The final report will be formatted following PR Notice 86-5 (2). The information to be reported will include, but will not be limited to, the following:

1. The name and address of the Sponsor, the testing facility and the study schedule.

2. The names of personnel involved with the study including Study Director, other scientists and supervisory personnel.

3. The signatures of Study Director, laboratory director and other individual scientists.

4. The signed and dated reports of each of the individual scientists or other professionals involved in the study, as appropriate.

5. A list of all deviations from the protocol, if any.

6. The quality assurance statement, signed by a member of the Quality Assurance Unit.

7. The statement of compliance, signed by the Study Director.

8. The storage locations of all archived data, specimen, and samples.

9. The test item identification, either by name and/or code number.

10. The purity, storage conditions (such as temperature and illumination), and other appropriate characteristics of the test item, as provided by the Sponsor.

11. A detailed description of the analytical method.

12. The individual and mean recovery values for the analyte.

13. A description of all other methods used with references.

14. A description, discussion, and interpretation of all results and, if necessary, a statistical evaluation.

15. Chromatograms of at least one representative calibration standard solution, one control sample, and one procedural recovery sample at each fortification level for each matrix evaluated.

16. Chromatograms should clearly indicate with an arrow the peak or region of interest.

17. A copy of the protocol as well as copies of all protocol amendments/deviations will be attached to the report.

16 REFERENCES

1. Syngenta Crop Protection analytical method entitled *"Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment,"* Draft, by N. J. Robinson.

2. *Ecological Effects Test Guidelines, OPPTS 850.7100, Data Reporting for Environmental Chemistry Methods,* U.S. Environmental Protection Agency. U.S. Government Printing Office: Washington, DC, April 1996; EPA-712-C-96-348.

3. *Pesticide Regulation Notice 86-5,* U.S. Environmental Protection Agency, Office of Pesticide Programs: Washington, DC, 1986.

PROTOCOL AMENDMENT 1

Study Title: Validation of the Residue Analytical Method: "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment," Draft.

Protocol No.: MLI-06-02

MLI Project No.: ML06-1286-PWG

Date: May 23, 2006

CHANGES TO THE STUDY

Change 1: Page 12 of 16, Section 10.4, Source of Sediment and Identification:

To change estuarine sediment sampling location from Belvedere Cove to Paradise Cove.

Change 2: Page 13 of 16, Section 10.6.2, Fortification procedures:

To change Reagent Blank and Control fortification level from 0.0 mg/kg to µg/kg.

Change 3: Page 14 of 16, Section II, Evaluation of Results:

a) To remove statement: Recoveries are not to be corrected for control contribution.

b) Calculation change. The actual percent recovery for the analytes will be determined as follows:

$$\%\ Recovery = \frac{\mu g/kg\ found\ in\ fortified\ control - mean\ \mu g/kg\ found\ in\ control}{\mu g/kg\ added}$$

REASONS FOR CHANGES

Change 1. The estuarine sediment source was changed due to Sponsor having more historical data available for Paradise Cove.

Change 2. Typographical error.

Change 3. Due to the presence of some targeted analytes in control samples chosen and provided by the Sponsor to be used in the study, it became necessary to correct recoveries for control contribution in order to get a true assessment of the accuracy of the method.

IMPACT ON STUDY: There will be no impact on the Study.

Richard L. Reed II

Richard L. Reed II
MORSE LABORATORIES, INC.
Study Director

May 24, 2006
Date

N. Robinson

Neil Robinson
SYNGENTA CROP PROTECTION, INC.
Study Monitor

May 31, 2006
Date

PROTOCOL AMENDMENT 2

Study Title: Validation of the Residue Analytical Method: "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment," Draft.

Protocol No.: MLI-06-02

MLI Project No.: ML06-1286-PWG

Date: October 24, 2006

CHANGE TO THE STUDY

Change 1: Page 1 of 16, STUDY TITLE:

To remove the word "Draft" from the **Study Title.** The **Study Title** is thus changed to:

Validation of the Residue Analytical Method: "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment"

REASON FOR CHANGE

Change 1. To reflect the actual version (final) of the method being appended to the Final Report.

IMPACT ON STUDY: There will be no impact on the Study.

Richard L. Reed II
MORSE LABORATORIES, INC.
Study Director

Oct. 30, 2006
Date

N. Robinson
Neil Robinson
SYNGENTA CROP PROTECTION, INC.
Study Monitor

26th Oct 2006
Date

APPENDIX 3. Protocol Deviations

PROTOCOL DEVIATION

MORSE LABORATORIES, INC. PROJECT #: **ML06-1286-PWG**

PROTOCOL #: **MLI-06-02**

DEVIATION #: **1**

DATE: **May 23, 2006**

SUBJECT: Set #2: Freshwater Sediment

The fresh water control sample used for the validation set contained mean residues of 0.0414 µg/kg, 0.0655 µg/kg ad 0.0453 µg/kg for lambda-cyhalothrin, esfenvalerate and deltamethrin, respectively. These are greater than the 30% of the limit of quantitation required by the protocol. The sample identification is BUCGR (Morse Laboratories, Inc. Ticket #83858.

REASONS: Unknown.

IMPACT ON STUDY: There is no adverse impact on the study. The level in the control subtracted from the fortified control still had acceptable recovery.

Richard L. Reed II

Richard L. Reed II
MORSE LABORATORIES, INC.
Analytical Project Coordinator

May 24, 2006
Date

N. Robinson

Neil Robinson
SYNGENTA CROP PROTECTION, INC.
Study Monitor

May 31, 2006
Date

PROTOCOL DEVIATION

MORSE LABORATORIES, INC. PROJECT #: **ML06-1286-PWG**

PROTOCOL #: **MLI-06-02**

DEVIATION #: **2**

DATE: **August 4, 2006** ③ RR 8-18-06

SUBJECT: Set ~~#1~~ 3: Estuarine

The estuarine water control sample used for the validation set contained mean residues of 0.0469 µg/kg, and 0.0720 µg/kg for bifenthrin and deltamethrin, respectively. These are greater than the 30% of the limit of quantitation required by the protocol. The sample identification is Paradise Cove (Morse Laboratories, Inc. Ticket #83942).

REASONS: Unknown.

IMPACT ON STUDY: There is no adverse impact on the study. The level in the control subtracted from the fortified control still had acceptable recovery.

Richard L. Reed II

Richard L. Reed II
MORSE LABORATORIES, INC.
Analytical Project Coordinator

Aug 8, 2006
Date

N. Robinson

Neil Robinson
SYNGENTA CROP PROTECTION, INC.
Study Monitor

Aug 16, 2006
Date

PROTOCOL DEVIATION

MORSE LABORATORIES, INC. PROJECT #: **ML06-1286-PWG**

PROTOCOL #: **MLI-06-02**

DEVIATION #: **3**

DATE: **September 25, 2006**

SUBJECT: **Sediment Characterization**

Percent organic matter and particle size determinations were required for each sediment type per protocol Section 10.3 "Characterization". In deviation from the protocol, particle size determination was not performed.

REASON: Through oversight by the Sponsor, particle size analysis was not scheduled to be performed.

IMPACT ON STUDY: There is no adverse impact on the study. This study was performed to validate the analytical method, regardless of particle size of the sample analyzed.

Richard L. Reed II — Sept. 26, 2006

Richard L. Reed II
MORSE LABORATORIES, INC.
Study Director

Date

N. Robinson — October 2, 2006

Neil Robinson
SYNGENTA CROP PROTECTION, INC
Study Monitor

Date

APPENDIX 4. Spreadsheets

Residue Results - Method Validation
Analyte: Bifenthrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Fresh Water																		
1.0		2				04/28/06						7454	7454					
	BUCGR fort. cont. 11	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	7355		7129	0.10317015	0.103	0.000	103
	BUCGR fort. cont. 12	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	7557		7129	0.106003647	0.106	0.000	106
1.0		2				04/28/06						6804	6804					
	BUCGR fort. cont. 13	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	6922		6486	0.106722171	0.107	0.000	107
1.0		2				04/28/06						6169	6169					
	BUCGR fort. cont. 14	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	6712		6248	0.107426376	0.107	0.000	107
	BUCGR fort. cont. 15	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	6815		6248	0.109074904	0.109	0.000	109
1.0		2				04/28/06						6326	6326					
	Reagent Blank 2	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		6048	0	ND		
	BUCGR control 3	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		6048	0	ND		
	BUCGR control 4	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		6048	0	ND		
20		2				04/28/06						115423	5771					
	BUCGR fort. cont. 16	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	57760		5796	0.996549344	1.00	0.000	100
	BUCGR fort. cont. 17	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	64094		5796	1.105831608	1.11	0.000	111
20		2				04/28/06						116412	5821					
	BUCGR fort. cont. 18	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	61614		5636	1.093222143	1.09	0.000	109
20		2				04/29/06						108990	5450					
	BUCGR fort. cont. 19	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	58424		5718	1.021755859	1.02	0.000	102
	BUCGR fort. cont. 20	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	65620		5718	1.147604057	1.15	0.000	115
20		2				04/29/06						119698	5985					
10		2				04/29/06						58285	5829					
2.5		2				04/29/06						14349	5740					
0.5		2				04/29/06						2681	5362					
1.0		2				04/29/06						5493	5493					

Residue Results - Method Validation
Analyte: Bifenthrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Estuarine																		
1.0		3				05/17/06						6312	6312					
	Paradise Cove fort. cont. 21	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	8465		6119	0.138339598	0.138	0.0469	91
	Paradise Cove fort. cont. 22	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	8600		6119	0.140545841	0.141	0.0469	94
1.0		3				05/17/06						5926	5926					
	Paradise Cove fort. cont. 23	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	8535		6250	0.13656	0.137	0.0469	90
1.0		3				05/17/06						6574	6574					
	Paradise Cove fort. cont. 24	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	8874		6428	0.138052271	0.138	0.0469	91
	Paradise Cove fort. cont. 25	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	8622		6428	0.134131923	0.134	0.0469	87
1.0		3				05/17/06						6282	6282					
	Reagent Blank 3	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		6174	0	ND		
	Paradise Cove control 5	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	3071		6174	0.049740849	<0.1		
	Paradise Cove control 6	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	2724		6174	0.044120505	<0.1		
20		3				05/17/06						121315	6066					
	Paradise Cove fort. cont. 26	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	60105		6139	0.979068252	0.979	0.0469	93
	Paradise Cove fort. cont. 27	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	63430		6139	1.033230168	1.03	0.0469	98
20		3				05/17/06						124239	6212					
	Paradise Cove fort. cont. 28	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	66536		6020	1.105249169	1.11	0.0469	106
20		3				05/17/06						116551	5828					
	Paradise Cove fort. cont. 29	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	64597		6108	1.057580223	1.06	0.0469	101
	Paradise Cove fort. cont. 30	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	61267		6108	1.003061559	1.00	0.0469	95
20		3				05/17/06						127745	6387					
10		3				05/18/06						64487	6449					
2.5		3				05/18/06						15988	6395					
1.0		3				05/18/06						6264	6264					
0.5		3				05/18/06						3251	6502					

Residue Results - Method Validation
Analyte: Cypermethrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Fresh Water																		
1.0		2				04/28/06						7257	7257					
	BUCGR fort. cont. 11	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	7080		6642	0.106594399	0.107	0.000	107
	BUCGR fort. cont. 12	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	6763		6642	0.10182174	0.102	0.000	102
1.0		2				04/28/06						6027	6027					
	BUCGR fort. cont. 13	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	6404		6094	0.105086971	0.105	0.000	105
1.0		2				04/28/06						6160	6160					
	BUCGR fort. cont. 14	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	6554		6312	0.103833967	0.104	0.000	104
	BUCGR fort. cont. 15	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	6938		6312	0.109917617	0.110	0.000	110
1.0		2				04/28/06						6464	6464					
	Reagent Blank 2	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		5507	0	ND		
	BUCGR control 3	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		5507	0	ND		
	BUCGR control 4	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		5507	0	ND		
20		2				04/28/06						90994	4550					
	BUCGR fort. cont. 16	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	50676		4556	1.112291484	1.11	0.000	111
	BUCGR fort. cont. 17	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	59344		4556	1.302546093	1.30	0.000	130
20		2				04/28/06						91267	4563					
	BUCGR fort. cont. 18	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	53990		4502	1.19924478	1.20	0.000	120
20		2				04/29/06						88804	4440					
	BUCGR fort. cont. 19	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	48910		4662	1.049120549	1.05	0.000	105
	BUCGR fort. cont. 20	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	57196		4662	1.226855427	1.23	0.000	123
20		2				04/29/06						97707	4885					
10		2				04/29/06						50030	5003					
2.5		2				04/29/06						12184	4874					
0.5		2				04/29/06						2452	4904					
1.0		2				04/29/06						5474	5474					

Residue Results - Method Validation
Analyte: Cypermethrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Estuarine																		
1.0		3				05/17/06						6869	6869					
	Paradise Cove fort. cont. 21	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	6423		6562	0.097881743	0.0979	0.000	98
	Paradise Cove fort. cont. 22	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	7498		6562	0.114263944	0.114	0.000	114
1.0		3				05/17/06						6254	6254					
	Paradise Cove fort. cont. 23	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	6949		6452	0.107703038	0.108	0.000	108
1.0		3				05/17/06						6650	6650					
	Paradise Cove fort. cont. 24	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	6366		6667	0.095485226	0.0955	0.000	96
	Paradise Cove fort. cont. 25	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	6464		6667	0.096955152	0.0970	0.000	97
1.0		3				05/17/06						6684	6684					
	Reagent Blank 3	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		6138	0	ND		
	Paradise Cove control 5	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		6138	0	ND		
	Paradise Cove control 6	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		6138	0	ND		
20		3				05/17/06						111862	5593					
	Paradise Cove fort. cont. 26	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	58299		5732	1.017079553	1.02	0.000	102
	Paradise Cove fort. cont. 27	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	62987		5732	1.098866015	1.10	0.000	110
20		3				05/17/06						117395	5870					
	Paradise Cove fort. cont. 28	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	63690		5584	1.140580229	1.14	0.000	114
20		3				05/17/06						105968	5298					
	Paradise Cove fort. cont. 29	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	64187		5779	1.110693892	1.11	0.000	111
	Paradise Cove fort. cont. 30	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	60636		5779	1.049247275	1.05	0.000	105
20		3				05/17/06						125194	6260					
10		3				05/18/06						61415	6142					
2.5		3				05/18/06						14171	5668					
1.0		3				05/18/06						6589	6589					
0.5		3				05/18/06						2860	5720					

Residue Results - Method Validation
Analyte: Cyfluthrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Fresh Water																		
1.0		2				04/28/06						10870	10870					
	BUCGR fort. cont. 11	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	10542		9871	0.10679769	0.107	0.000	107
	BUCGR fort. cont. 12	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	10374		9871	0.105095735	0.105	0.000	105
1.0		2				04/28/06						8872	8872					
	BUCGR fort. cont. 13	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	9630		9020	0.106762749	0.107	0.000	107
1.0		2				04/28/06						9169	9169					
	BUCGR fort. cont. 14	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	9405		9335	0.100749866	0.101	0.000	101
	BUCGR fort. cont. 15	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	10058		9335	0.107745046	0.108	0.000	108
1.0		2				04/28/06						9501	9501					
	Reagent Blank 2	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		8402	0	ND		
	BUCGR control 3	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		8402	0	ND		
	BUCGR control 4	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		8402	0	ND		
20		2				04/28/06						146068	7303					
	BUCGR fort. cont. 16	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	80574		7262	1.109529055	1.11	0.000	111
	BUCGR fort. cont. 17	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	92456		7262	1.273147893	1.27	0.000	127
20		2				04/28/06						144415	7221					
	BUCGR fort. cont. 18	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	85617		7200	1.189125	1.19	0.000	119
20		2				04/29/06						143555	7178					
	BUCGR fort. cont. 19	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	80747		7489	1.078208038	1.08	0.000	108
	BUCGR fort. cont. 20	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	89927		7489	1.200787822	1.20	0.000	120
20		2				04/29/06						156003	7800					
10		2				04/29/06						79513	7951					
2.5		2				04/29/06						18640	7456					
0.5		2				04/29/06						3564	7128					
1.0		2				04/29/06						8056	8056					

Residue Results - Method Validation
Analyte: Cyfluthrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Estuarine																		
1.0		3				05/17/06						10000	10000					
	Paradise Cove fort. cont. 21	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	9613		9526	0.10091329	0.101	0.0117	89
	Paradise Cove fort. cont. 22	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	11450		9526	0.120197355	0.120	0.0117	108
1.0		3				05/17/06						9051	9051					
	Paradise Cove fort. cont. 23	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	9809		9463	0.103656346	0.104	0.0117	92
1.0		3				05/17/06						9875	9875					
	Paradise Cove fort. cont. 24	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	11181		9409	0.118833032	0.119	0.0117	107
	Paradise Cove fort. cont. 25	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	10352		9409	0.110022319	0.110	0.0117	98
1.0		3				05/17/06						8943	8943					
	Reagent Blank 3	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		8688	0	ND		
	Paradise Cove control 5	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	2029		8688	0.023354052	<0.1		
	Paradise Cove control 6	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		8688	0	ND		
20		3				05/17/06						168645	8432					
	Paradise Cove fort. cont. 26	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	85979		8666	0.992141703	0.992	0.0117	98
	Paradise Cove fort. cont. 27	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	93771		8666	1.082056312	1.08	0.0117	107
20		3				05/17/06						178012	8901					
	Paradise Cove fort. cont. 28	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	96994		8518	1.138694529	1.14	0.0117	113
20		3				05/17/06						162722	8136					
	Paradise Cove fort. cont. 29	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	96203		8803	1.092843349	1.09	0.0117	108
	Paradise Cove fort. cont. 30	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	90802		8803	1.031489265	1.03	0.0117	102
20		3				05/17/06						189405	9470					
10		3				05/18/06						91721	9172					
2.5		3				05/18/06						20454	8182					
1.0		3				05/18/06						9816	9816					
0.5		3				05/18/06						4560	9120					

Residue Results - Method Validation
Analyte: Deltamethrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Fresh Water																		
1.0		2				04/28/06						6022	6022					
	BUCGR fort. cont. 11	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	7633		5714	0.133584179	0.134	0.0453	89
	BUCGR fort. cont. 12	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	7027		5714	0.122978649	0.123	0.0453	78
1.0		2				04/28/06						5406	5406					
	BUCGR fort. cont. 13	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	7456		5794	0.128684846	0.129	0.0453	84
1.0		2				04/28/06						6181	6181					
	BUCGR fort. cont. 14	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	7946		6088	0.130519054	0.131	0.0453	86
	BUCGR fort. cont. 15	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	8943		6088	0.146895532	0.147	0.0453	102
1.0		2				04/28/06						5996	5996					
	Reagent Blank 2	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		5266	0	ND		
	BUCGR control 3	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	3496		5266	0.06638815	<0.1		
	BUCGR control 4	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	1280		5266	0.024306874	<0.1		
20		2				04/28/06						90699	4535					
	BUCGR fort. cont. 16	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	48891		4486	1.089857334	1.09	0.0453	104
	BUCGR fort. cont. 17	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	54153		4486	1.207155595	1.21	0.0453	116
20		2				04/28/06						88712	4436					
	BUCGR fort. cont. 18	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	51226		4428	1.156865402	1.16	0.0453	111
20		2				04/29/06						88416	4421					
	BUCGR fort. cont. 19	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	47963		4627	1.036589583	1.04	0.0453	99
	BUCGR fort. cont. 20	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	52973		4627	1.144867085	1.14	0.0453	109
20		2				04/29/06						96652	4833					
10		2				04/29/06						47234	4723					
2.5		2				04/29/06						10511	4204					
0.5		2				04/29/06						2141	4282					
1.0		2				04/29/06						4572	4572					

Residue Results - Method Validation
Analyte: Deltamethrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Estuarine																		
1.0		3				05/17/06						4855	4855					
	Paradise Cove fort. cont. 21	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	7418		4656	0.159321306	0.159	0.0720	87
	Paradise Cove fort. cont. 22	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	7005		4656	0.150451031	0.150	0.0720	78
1.0		3				05/17/06						4457	4457					
	Paradise Cove fort. cont. 23	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	7594		4722	0.160821686	0.161	0.0720	89
1.0		3				05/17/06						4988	4988					
	Paradise Cove fort. cont. 24	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	7158		4599	0.155642531	0.156	0.0720	84
	Paradise Cove fort. cont. 25	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	7274		4599	0.158164818	0.158	0.0720	86
1.0		3				05/17/06						4210	4210					
	Reagent Blank 3	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		4236	0	ND		
	Paradise Cove control 5	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	2717		4236	0.064140699	<0.1		
	Paradise Cove control 6	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	3380		4236	0.079792257	<0.1		
20		3				05/17/06						85238	4262					
	Paradise Cove fort. cont. 26	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	35683		4396	0.811715196	0.812	0.0720	74
	Paradise Cove fort. cont. 27	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	38638		4396	0.878935396	0.879	0.0720	81
20		3				05/17/06						90585	4529					
	Paradise Cove fort. cont. 28	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	43286		4220	1.025734597	1.03	0.0720	96
20		3				05/17/06						78236	3912					
	Paradise Cove fort. cont. 29	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	47034		4506	1.043808256	1.04	0.0720	97
	Paradise Cove fort. cont. 30	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	38015		4506	0.843652907	0.844	0.0720	77
20		3				05/17/06						101988	5099					
10		3				05/18/06						47617	4762					
2.5		3				05/18/06						10176	4070					
1.0		3				05/18/06						4978	4978					
0.5		3				05/18/06						2285	4570					

Residue Results - Method Validation
Analyte: Esfenvalerate
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Fresh Water																		
1.0		2				04/28/06						13421	13421					
	BUCGR fort. cont. 11	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	16887		11988	0.140865866	0.141	0.0655	76
	BUCGR fort. cont. 12	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	17032		11988	0.142075409	0.142	0.0655	77
1.0		2				04/28/06						10554	10554					
	BUCGR fort. cont. 13	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	15612		10653	0.146550268	0.147	0.0655	82
1.0		2				04/28/06						10752	10752					
	BUCGR fort. cont. 14	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	15218		11013	0.138182148	0.138	0.0655	73
	BUCGR fort. cont. 15	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	16418		11013	0.149078362	0.149	0.0655	84
1.0		2				04/28/06						11274	11274					
	Reagent Blank 2	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		10271	0	ND		
	BUCGR control 3	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	9601		10271	0.093476779	<0.1		
	BUCGR control 4	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	3862		10271	0.037601013	<0.1		
20		2				04/28/06						185356	9268					
	BUCGR fort. cont. 16	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	102955		9184	1.121025697	1.12	0.0655	105
	BUCGR fort. cont. 17	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	118777		9184	1.293303571	1.29	0.0655	122
20		2				04/28/06						181974	9099					
	BUCGR fort. cont. 18	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	112487		9096	1.236664468	1.24	0.0655	117
20		2				04/29/06						181881	9094					
	BUCGR fort. cont. 19	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	100492		9472	1.0609375	1.06	0.0655	99
	BUCGR fort. cont. 20	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	115747		9472	1.221991132	1.22	0.0655	115
20		2				04/29/06						197023	9851					
10		2				04/29/06						99991	9999					
2.5		2				04/29/06						22472	8989					
0.5		2				04/29/06						4521	9042					
1.0		2				04/29/06						8960	8960					

Residue Results - Method Validation
Analyte: Esfenvalerate
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Estuarine																		
1.0		3				05/17/06						12026	12026					
	Paradise Cove fort. cont. 21	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	12911		11483	0.112435775	0.112	0.0148	97
	Paradise Cove fort. cont. 22	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	14513		11483	0.126386833	0.126	0.0148	111
1.0		3				05/17/06						10940	10940					
	Paradise Cove fort. cont. 23	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	14013		11044	0.126883376	0.127	0.0148	112
1.0		3				05/17/06						11147	11147					
	Paradise Cove fort. cont. 24	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	13454		10840	0.124114391	0.124	0.0148	109
	Paradise Cove fort. cont. 25	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	14364		10840	0.132509225	0.133	0.0148	118
1.0		3				05/17/06						10532	10532					
	Reagent Blank 3	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		10808	0	ND		
	Paradise Cove control 5	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	1671		10808	0.01546077	<0.1		
	Paradise Cove control 6	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	1525		10808	0.014109919	<0.1		
20		3				05/17/06						221658	11083					
	Paradise Cove fort. cont. 26	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	110281		11410	0.96652936	0.967	0.0148	95
	Paradise Cove fort. cont. 27	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	124965		11410	1.095223488	1.10	0.0148	109
20		3				05/17/06						234713	11736					
	Paradise Cove fort. cont. 28	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	125695		11161	1.126198369	1.13	0.0148	112
20		3				05/17/06						211718	10586					
	Paradise Cove fort. cont. 29	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	123423		11450	1.077930131	1.08	0.0148	107
	Paradise Cove fort. cont. 30	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	117523		11450	1.026401747	1.03	0.0148	102
20		3				05/17/06						246266	12313					
10		3				05/18/06						116640	11664					
2.5		3				05/18/06						25297	10119					
1.0		3				05/18/06						11665	11665					
0.5		3				05/18/06						5384	10768					

Residue Results - Method Validation
Analyte: Fenpropathrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Fresh Water																		
1.0		2				04/28/06						11706	11706					
	BUCGR fort. cont. 11	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	11040		10794	0.102279044	0.102	0.000	102
	BUCGR fort. cont. 12	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	10122		10794	0.093774319	0.0938	0.000	94
1.0		2				04/28/06						9881	9881					
	BUCGR fort. cont. 13	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	10373		9668	0.107292098	0.107	0.000	107
1.0		2				04/28/06						9456	9456					
	BUCGR fort. cont. 14	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	10165		9412	0.108000425	0.108	0.000	108
	BUCGR fort. cont. 15	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	10026		9412	0.106523587	0.107	0.000	107
1.0		2				04/28/06						9369	9369					
	Reagent Blank 2	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		8936	0	ND		
	BUCGR control 3	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		8936	0	ND		
	BUCGR control 4	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		8936	0	ND		
20		2				04/28/06						170037	8502					
	BUCGR fort. cont. 16	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	93570		8498	1.101082608	1.10	0.000	110
	BUCGR fort. cont. 17	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	97778		8498	1.150600141	1.15	0.000	115
20		2				04/28/06						169884	8494					
	BUCGR fort. cont. 18	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	96174		8276	1.162083132	1.16	0.000	116
20		2				04/29/06						161155	8058					
	BUCGR fort. cont. 19	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	91843		8539	1.07557091	1.08	0.000	108
	BUCGR fort. cont. 20	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	98848		8539	1.157606277	1.16	0.000	116
20		2				04/29/06						180408	9020					
10		2				04/29/06						86322	8632					
2.5		2				04/29/06						21056	8422					
0.5		2				04/29/06						3802	7604					
1.0		2				04/29/06						8251	8251					

Residue Results - Method Validation
Analyte: Fenpropathrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Estuarine																		
1.0		3				05/17/06						10783	10783					
	Paradise Cove fort. cont. 21	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	10014		10598	0.094489526	0.0945	0.000	95
	Paradise Cove fort. cont. 22	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	11587		10598	0.109331949	0.109	0.000	109
1.0		3				05/17/06						10414	10414					
	Paradise Cove fort. cont. 23	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	11176		10536	0.106074412	0.106	0.000	106
1.0		3				05/17/06						10657	10657					
	Paradise Cove fort. cont. 24	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	11682		10556	0.110666919	0.111	0.000	111
	Paradise Cove fort. cont. 25	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	11080		10556	0.104964002	0.105	0.000	105
1.0		3				05/17/06						10454	10454					
	Reagent Blank 3	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		10636	0	ND		
	Paradise Cove control 5	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		10636	0	ND		
	Paradise Cove control 6	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		10636	0	ND		
20		3				05/17/06						216355	10818					
	Paradise Cove fort. cont. 26	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	108530		11024	0.984488389	0.984	0.000	98
	Paradise Cove fort. cont. 27	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	115828		11024	1.050689405	1.05	0.000	105
20		3				05/17/06						224573	11229					
	Paradise Cove fort. cont. 28	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	123259		10828	1.138335796	1.14	0.000	114
20		3				05/17/06						208516	10426					
	Paradise Cove fort. cont. 29	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	117783		10978	1.072900346	1.07	0.000	107
	Paradise Cove fort. cont. 30	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	109714		10978	0.999398798	0.999	0.000	100
20		3				05/17/06						230594	11530					
10		3				05/18/06						114717	11472					
2.5		3				05/18/06						26839	10736					
1.0		3				05/18/06						11613	11613					
0.5		3				05/18/06						5319	10638					

Residue Results - Method Validation
Analyte: Lambda-cyhalothrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Fresh Water																		
1.0		2				04/28/06						10784	10784					
	BUCGR fort. cont. 11	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	12470		9952	0.125301447	0.125	0.0414	84
	BUCGR fort. cont. 12	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	12378		9952	0.12437701	0.124	0.0414	83
1.0		2				04/28/06						9119	9119					
	BUCGR fort. cont. 13	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	12173		8899	0.136790651	0.137	0.0414	96
1.0		2				04/28/06						8679	8679					
	BUCGR fort. cont. 14	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	11780		8798	0.133894067	0.134	0.0414	93
	BUCGR fort. cont. 15	2	50.0	0.1	04/28/06	04/28/06	50	10.0	1.0	2	1	13143		8798	0.149386224	0.149	0.0414	108
1.0		2				04/28/06						8917	8917					
	Reagent Blank 2	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		8304	0	ND		
	BUCGR control 3	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	3484		8304	0.041955684	<0.1		
	BUCGR control 4	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	3397		8304	0.040907996	<0.1		
20		2				04/28/06						153812	7691					
	BUCGR fort. cont. 16	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	85102		7709	1.103930471	1.10	0.0414	106
	BUCGR fort. cont. 17	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	96043		7709	1.245855494	1.25	0.0414	121
20		2				04/28/06						154546	7727					
	BUCGR fort. cont. 18	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	89416		7670	1.165788787	1.17	0.0414	113
20		2				04/29/06						152275	7614					
	BUCGR fort. cont. 19	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	85676		7948	1.077956719	1.08	0.0414	104
	BUCGR fort. cont. 20	2	50.0	1.0	04/28/06	04/29/06	50	10.0	1.0	2	1	96649		7948	1.216016608	1.22	0.0414	118
20		2				04/29/06						165637	8282					
10		2				04/29/06						81386	8139					
2.5		2				04/29/06						18359	7344					
0.5		2				04/29/06						4057	8114					
1.0		2				04/29/06						8036	8036					

Residue Results - Method Validation
Analyte: Lambda-cyhalothrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Estuarine																		
1.0		3				05/17/06						8164	8164					
	Paradise Cove fort. cont. 21	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	7959		7866	0.101182304	0.101	0.0111	90
	Paradise Cove fort. cont. 22	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	9214		7866	0.117137046	0.117	0.0111	106
1.0		3				05/17/06						7569	7569					
	Paradise Cove fort. cont. 23	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	8695		7697	0.112966091	0.113	0.0111	102
1.0		3				05/17/06						7825	7825					
	Paradise Cove fort. cont. 24	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	9121		7899	0.115470313	0.115	0.0111	104
	Paradise Cove fort. cont. 25	3	50.0	0.1	05/16/06	05/17/06	50	10.0	1.0	2	1	9138		7899	0.11568553	0.116	0.0111	105
1.0		3				05/17/06						7973	7973					
	Reagent Blank 3	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		7860	0	ND		
	Paradise Cove control 5	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	920		7860	0.011704835	<0.1		
	Paradise Cove control 6	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	819		7860	0.010419847	<0.1		
20		3				05/17/06						154964	7748					
	Paradise Cove fort. cont. 26	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	77659		7937	0.978442737	0.978	0.0111	97
	Paradise Cove fort. cont. 27	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	83913		7937	1.057238251	1.06	0.0111	105
20		3				05/17/06						162527	8126					
	Paradise Cove fort. cont. 28	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	87480		7808	1.120389344	1.12	0.0111	111
20		3				05/17/06						149828	7491					
	Paradise Cove fort. cont. 29	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	85156		7978	1.06738531	1.07	0.0111	106
	Paradise Cove fort. cont. 30	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	80028		7978	1.003108549	1.00	0.0111	99
20		3				05/17/06						169287	8464					
10		3				05/18/06						82045	8205					
2.5		3				05/18/06						17663	7065					
1.0		3				05/18/06						8271	8271					
0.5		3				05/18/06						3687	7374					

Residue Results - Method Validation
Analyte: Permethrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Fresh Water																		
10		2				04/28/06						5857	586					
	BUCGR fort. cont. 11	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	5246		539	0.973283859	0.973	0.000	97
	BUCGR fort. cont. 12	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	5326		539	0.98812616	0.988	0.000	99
10		2				04/28/06						4918	492					
	BUCGR fort. cont. 13	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	4462		475	0.939368421	0.939	0.000	94
10		2				04/28/06						4575	458					
	BUCGR fort. cont. 14	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	4526		455	0.994725275	0.995	0.000	100
	BUCGR fort. cont. 15	2	50.0	1.0	04/28/06	04/28/06	50	10.0	1.0	2	1	4929		455	1.083296703	1.08	0.000	108
10		2				04/28/06						4519	452					
	Reagent Blank 2	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		426	0	ND		
	BUCGR control 3	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		426	0	ND		
	BUCGR control 4	2	50.0		04/28/06	04/28/06	50	10.0	1.0	2	1	0		426	0	ND		
200		2				04/28/06						80094	400					
	BUCGR fort. cont. 16	2	50.0	10.0	04/28/06	04/28/06	50	10.0	1.0	2	1	39075		402	9.720149254	9.72	0.000	97
	BUCGR fort. cont. 17	2	50.0	10.0	04/28/06	04/28/06	50	10.0	1.0	2	1	49737		402	12.37238806	12.4	0.000	124
200		2				04/28/06						80924	405					
	BUCGR fort. cont. 18	2	50.0	10.0	04/28/06	04/28/06	50	10.0	1.0	2	1	42642		398	10.71407035	10.7	0.000	107
200		2				04/29/06						78326	392					
	BUCGR fort. cont. 19	2	50.0	10.0	04/28/06	04/29/06	50	10.0	1.0	2	1	38848		417	9.316067146	9.32	0.000	93
	BUCGR fort. cont. 20	2	50.0	10.0	04/28/06	04/29/06	50	10.0	1.0	2	1	48814		417	11.7059952	11.7	0.000	117
200		2				04/29/06						88355	442					
100		2				04/29/06						42339	423					
25		2				04/29/06						9434	377					
5.0		2				04/29/06						1788	358					
10		2				04/29/06						3434	343					

Residue Results - Method Validation
Analyte: Permethrin
Matrix: Sediment

Std Conc ng/mL	Sample Identification	Set #	Samp Wt g	Fort Level ug/kg	Extraction Date	Injection Date	mL solv	mL aliq	FV mL	Inj vol uL	GC Dil Fact	Total Peak Resp	Std Resp Factor	Avg Resp Fact (Brack Stds)	ug/kg	rept ug/kg	Avg. ug/kg cont.	% Rec
Sediment, Estuarine																		
10		3				05/17/06						5435	544					
	Paradise Cove fort. cont. 21	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	5433		552	0.98423913	0.984	0.000	98
	Paradise Cove fort. cont. 22	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	6119		552	1.108514493	1.11	0.000	111
10		3				05/17/06						5606	561					
	Paradise Cove fort. cont. 23	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	5523		542	1.01900369	1.02	0.000	102
10		3				05/17/06						5223	522					
	Paradise Cove fort. cont. 24	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	5667		530	1.069245283	1.07	0.000	107
	Paradise Cove fort. cont. 25	3	50.0	1.0	05/16/06	05/17/06	50	10.0	1.0	2	1	5888		530	1.110943396	1.11	0.000	111
10		3				05/17/06						5389	539					
	Reagent Blank 3	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		534	0	ND		
	Paradise Cove control 5	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		534	0	ND		
	Paradise Cove control 6	3	50.0		05/16/06	05/17/06	50	10.0	1.0	2	1	0		534	0	ND		
200		3				05/17/06						105832	529					
	Paradise Cove fort. cont. 26	3	50.0	10.0	05/16/06	05/17/06	50	10.0	1.0	2	1	51714		540	9.576666667	9.58	0.000	96
	Paradise Cove fort. cont. 27	3	50.0	10.0	05/16/06	05/17/06	50	10.0	1.0	2	1	57407		540	10.63092593	10.6	0.000	106
200		3				05/17/06						110099	550					
	Paradise Cove fort. cont. 28	3	50.0	10.0	05/16/06	05/17/06	50	10.0	1.0	2	1	58326		524	11.13091603	11.1	0.000	111
200		3				05/17/06						99368	497					
	Paradise Cove fort. cont. 29	3	50.0	10.0	05/16/06	05/17/06	50	10.0	1.0	2	1	57945		536	10.81063433	10.8	0.000	108
	Paradise Cove fort. cont. 30	3	50.0	10.0	05/16/06	05/17/06	50	10.0	1.0	2	1	54232		536	10.11791045	10.1	0.000	101
200		3				05/17/06						115111	576					
100		3				05/18/06						56440	564					
25		3				05/18/06						13142	526					
10		3				05/18/06						5491	549					
5.0		3				05/18/06						2633	527					

APPENDIX 5. Characterization Analytical Report

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client : Pacific Eco-Risk Laboratories
Project Name : NA
Project Number : 10952
Sample Matrix : SEDIMENT

Service Request : K0602611
Date Collected : 03/29/06
Date Received : 04/05/06

Carbon, Total Organic

Analysis Method : ASTM D4129-98M
Test Notes :

Units : Percent
Basis : Dry

Sample Name	Lab Code	MRL	Dilution Factor	Date Analyzed	Result	Result Notes
BUCGR	K0602611-001	0.05	1	04/12/06	0.86	
Method Blank	K0602611-MB	0.05	1	04/12/06	ND	

M Modified for analysis of soil.

00010

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client : Pacific Eco-Risk Laboratories
Project Name : Paradise Cove
Project Number : 10952
Sample Matrix : Sediment

Service Request : K0603866
Date Collected : 05/11/06
Date Received : 05/12/06

Carbon, Total Organic

Analysis Method : ASTM D4129-98M
Test Notes :

Units : PERCENT
Basis : Dry

Sample Name	Lab Code	MRL	Dilution Factor	Date Prepared	Date Analyzed	Result	Result Notes
Paradise Cove	K0603866-001	0.05	1	05/15/06	05/23/06	1.31	
Method Blank	K0603866-MB	0.05	1	05/15/06	05/23/06	ND	

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Report By:tpoyfair

GCMS-NCI SIM / SW-846 8270 Method Validation Submittal

Initial Calibration Linearity and ICAL Verification Data

Report Date : 21-Jul-2009 15:47

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CalTest

INITIAL CALIBRATION DATA

Start Cal Date : 17-JUL-2009 14:06
End Cal Date : 17-JUL-2009 20:25
Quant Method : ISTD
Target Version : 4.14
Integrator : HP RTE
Method file : \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Last Edit : 21-Jul-2009 14:59 nta

Pyr curve
7/21/09

Calibration File Names:
Level 1: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_13.D
Level 2: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_12.D
Level 3: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_11.D
Level 4: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_10.D
Level 5: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_09.D
Level 6: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_08.D
Level 7: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_07.D
Level 8: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_06.D
Level 9: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_05.D
Level 10: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_04.D
Level 11: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_03.D

Compound	0.0001000 Level 1	0.0002500 Level 2	0.0005000 Level 3	0.0010000 Level 4	0.0020000 Level 5	0.0050000 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2	
	0.0100000 Level 7	0.0150000 Level 8	0.0200000 Level 9	0.0300000 Level 10	0.0500000 Level 11							
1 StartList	+++++	+++++	+++++	+++++	+++++	+++++						
	+++++	+++++	+++++	+++++	+++++		QUAD	0.000e+000	0.000e+000	0.000e+000	0.000e+000	<-
4 Diazinon	21.00000	18.00000	41.00000	79.00000	130	357						
	687	1026	1367	2460	4446		QUAD	-0.13261	101	-44.92189	0.99982	

Report Date : 21-Jul-2009 15:47

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CalTest

INITIAL CALIBRATION DATA

Start Cal Date : 17-JUL-2009 14:06
End Cal Date : 17-JUL-2009 20:25
Quant Method : ISTD
Target Version : 4.14
Integrator : HP RTE
Method file : \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Last Edit : 21-Jul-2009 14:59 nta

Compound	0.0001000 Level 1	0.0002500 Level 2	0.0005000 Level 3	0.0010000 Level 4	0.0020000 Level 5	0.0050000 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
	0.0100000 Level 7	0.0150000 Level 8	0.0200000 Level 9	0.0300000 Level 10	0.0500000 Level 11						
5 Chlorpyrifos	107	216	452	896	1769	4606					
	9709	13042	17563	27374	46875		QUAD	-0.29021	8.00625	-0.20249	0.99829
6 Allethrin	161	309	574	1052	2031	5516					
	11449	15428	20286	31059	52137		QUAD	-0.34304	6.83557	-0.12085	0.99759
7 Bifenthrin	20.00000	44.00000	88.00000	140	258	698					
	1525	2045	2782	4188	7246		QUAD	-0.31136	51.39287	-8.11203	0.99743
8 Tetramethrin	21.00000	53.00000	124	228	421	1078					
	2323	3134	4121	6226	10360		QUAD	-0.32618	33.55016	-2.57330	0.99734
9 Phenothrin	+++++	+++++	+++++	+++++	89.00000	113					
	186	267	351	529	782		QUAD	-3.31728	450	-339	0.99726
10 Fenpropathrin(Danitol)	59.00000	168	269	500	1117	2958					
	6299	8516	11392	17555	29943		QUAD	-0.24819	12.29038	-0.44868	0.99794
11 L-Cyhalothrin(ISO#1&2)	131	270	520	970	2048	5512					
	11312	15932	20862	31836	53785		QUAD	-0.25652	6.66803	-0.11944	0.99760

Report Date : 21-Jul-2009 15:47

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CalTest

INITIAL CALIBRATION DATA

Start Cal Date : 17-JUL-2009 14:06
End Cal Date : 17-JUL-2009 20:25
Quant Method : ISTD
Target Version : 4.14
Integrator : HP RTE
Method file : \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Last Edit : 21-Jul-2009 14:59 nta

Compound	0.0001000 Level 1	0.0002500 Level 2	0.0005000 Level 3	0.0010000 Level 4	0.0020000 Level 5	0.0050000 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2	
	0.0100000 Level 7	0.0150000 Level 8	0.0200000 Level 9	0.0300000 Level 10	0.0500000 Level 11							
12 Permethrin(ISO#1&2)	+++++	+++++	+++++	+++++	96.00000	129						
	222	274	389	514	932		QUAD	-5.68637	520	-1048	0.99120	
13 Cyfluthrin(ISO#1-4)	90.00000	215	359	683	1315	3554						
	7214	10413	13468	20699	32379		QUAD	-0.17725	9.81867	-0.10218	0.99845	
14 Cypermethrin(ISO#1-4)	109	170	252	449	873	2315						
	4805	6800	8603	14013	20980		QUAD	-0.16091	14.70024	-0.12517	0.99947	
15 Fenvalerate/Esfenvalerate	230	576	1025	2028	4084	10690						
	22555	32150	42361	62626	101201		QUAD	-0.36137	6.43108	-0.02925	0.99728	
16 Tau-Fluvalinate(ISO#1&2)	144	370	641	1347	2736	7291						
	15332	21920	28653	41634	66502		QUAD	-0.15769	4.68887	-0.01548	0.99670	
17 Tralomethrin/Deltamethrin	59.00000	137	230	407	913	2215						
	5195	7328	9935	14140	22634		QUAD	-0.12156	27.44616	-0.25730	0.99640	
18 EndList	+++++	+++++	+++++	+++++	+++++	+++++						
	+++++	+++++	+++++	+++++	+++++		QUAD	0.000e+000	0.000e+000	0.000e+000	0.000e+000	<-

Report Date : 21-Jul-2009 15:47

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CalTest

INITIAL CALIBRATION DATA

Start Cal Date : 17-JUL-2009 14:06
End Cal Date : 17-JUL-2009 20:25
Quant Method : ISTD
Target Version : 4.14
Integrator : HP RTE
Method file : \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Last Edit : 21-Jul-2009 14:59 nta

Compound	0.0001000 Level 1	0.0002500 Level 2	0.0005000 Level 3	0.0010000 Level 4	0.0020000 Level 5	0.0050000 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
	0.0100000 Level 7	0.0150000 Level 8	0.0200000 Level 9	0.0300000 Level 10	0.0500000 Level 11						
2 Decachlorobiphenyl (SS)	39.00000	80.00000	127	285	417	1127					
	2892	3441	4366	+++++	11562		QUAD	-0.12458	27.98010	-1.11994	0.99816

Report Date : 21-Jul-2009 15:47

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CalTest

INITIAL CALIBRATION DATA

Start Cal Date : 17-JUL-2009 14:06
End Cal Date : 17-JUL-2009 20:25
Quant Method : ISTD
Target Version : 4.14
Integrator : HP RTE
Method file : \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Last Edit : 21-Jul-2009 14:59 nta

Average %RSD Results.
Calculated Average %RSD = 0.000e+000
Maximun Average %RSD = 15.00000
Passed Average %RSD Test.

Curve	Formula	Units
Quad	Amt = b + m1*Rsp + m2*Rsp^2	Response

Data File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_14.D
Report Date: 21-Jul-2009 11:53

ICV

CalTest

RECOVERY REPORT

Client Name:
Sample Matrix: NONE
Lab Smp Id: pyr 2* std @ 0.01pp
Level:
Data Type: MS DATA
SpikeList File: PYR_SEC.spk
Sublist File: SECPYR.sub
Method File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Misc Info: 3;3;;;;;S-070709-3

Client SDG: 0204d
Fraction: SV

Operator: RLH
SampleType: BLANK
Quant Type: ISTD

Pass 7/21/09

SPIKE COMPOUND	AMOUNT ADDED ug/mL	AMOUNT RECOVERED ug/mL	% RECOVERED	LIMITS
4 Diazinon	0.0100	0.00875	87.47	
5 Chlorpyrifos	0.0100	0.00915	91.46	
6 Allethrin	0.0100	0.00931	93.07	
7 Bifenthrin	0.0100	0.00786	78.63	
8 Tetramethrin	0.0100	0.00848	84.77	
9 Phenothrin	0.0100	0.00720	72.03	
10 Fenpropathrin(Dani	0.0100	0.00806	80.60	
11 L-Cyhalothrin(ISO#	0.0100	0.00815	81.50	
12 Permethrin(ISO#1&2	0.0100	0.00898	89.81	
13 Cyfluthrin(ISO#1-4	0.0100	0.00771	77.09	
14 Cypermethrin(ISO#1	0.0100	0.00789	78.90	
15 Fenvalerate/Esfenv	0.0200	0.0156	77.76	
16 Tau-Fluvalinate(IS	0.0100	0.00807	80.70	
17 Tralomethrin/Delta	0.0200	0.0149	74.48	

Data File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_14.D
Report Date: 21-Jul-2009 11:53

CalTest

ICV

INTERNAL STANDARD COMPOUNDS
AREA AND RT SUMMARY

Instrument ID: MSDF.i
Lab File ID: 0717F_14.D
Lab Smp Id: pyr 2* std @ 0.01pp
Analysis Type: SV
Quant Type: ISTD
Operator: RLH
Method File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Misc Info: 3;3;;;;;S-070709-3

Calibration Date: 17-JUL-2009
Calibration Time: 16:56

Level:
Sample Type:

Test Mode:
Use Last Continuing Calibrator.
If Continuing Cal. use Initial Cal. Level 5

		AREA LIMIT			
COMPOUND	STANDARD	LOWER	UPPER	SAMPLE	%DIFF
3 4,4'-DBOB (IS)	6793	3397	13586	6768	-0.37

		RT LIMIT			
COMPOUND	STANDARD	LOWER	UPPER	SAMPLE	%DIFF
3 4,4'-DBOB (IS)	8.27	8.17	8.37	8.26	-0.06

AREA UPPER LIMIT = +100% of internal standard area.
AREA LOWER LIMIT = - 50% of internal standard area.
RT UPPER LIMIT = + 0.10 minutes of internal standard RT.
RT LOWER LIMIT = - 0.10 minutes of internal standard RT.

GCMS-NCI SIM / SW-846 8270 Method Validation Submittal

Initial Method Detection Limit (MDL) Study

Initial Precision & Recovery (IPR)

QC Sample Data

CalTest
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Batch File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b
Inst ID: MSDF.i

ID:	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07
FILENAME:	0717F_22	0717F_23	0717F_24	0717F_25	0717F_26	0717F_27	0717F_28
INJ.DATE:	18-JUL-2009	18-JUL-2009	18-JUL-2009	18-JUL-2009	18-JUL-2009	18-JUL-2009	18-JUL-2009
INJ.TIME:	01:37	02:12	02:46	03:21	03:56	04:31	05:05

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	AVG CONC	STD DEV	MDL	
4 Diazinon	0.09	0.14	0.09	0.12	0.14	0.08	0.08	0.11	0.03	0.09	
5 Chlorpyrifos	0.10	0.13	0.13	0.14	0.14	0.04	0.06	0.11	0.04	0.13	
6 Allethrin	0.07	0.07	0.07	0.08	0.09	0.05	0.05	0.07	0.02	0.05	
7 Bifenthrin	0.11	0.11	0.10	0.09	0.12	0.04	0.06	0.09	0.03	0.10	
8 Tetramethrin	0.02	0.04	0.01	0.06	0.05	0.01	0.02	0.03	0.02	0.06	
10 Fenpropathrin(Danitol)	0.11	0.13	0.10	0.09	0.13	0.08	0.08	0.10	0.02	0.07	
11 L-Cyhalothrin(ISO#1&2)	0.08	0.11	0.09	0.07	0.10	0.06	0.07	0.08	0.02	0.06	
12 Permethrin(ISO#1&2)	0.06	0.12	0.09	0.12	0.11	0.04	0.07	0.09	0.03	0.11	
13 Cyfluthrin(ISO#1-4)	0.10	0.18	0.12	0.12	0.12	0.08	0.08	0.12	0.03	0.11	
14 Cypermethrin(ISO#1-4)	0.12	0.17	0.14	0.15	0.15	0.09	0.09	0.13	0.03	0.10	
15 Fenvalerate/Esfenvaler	0.21	0.26	0.21	0.20	0.24	0.15	0.15	0.20	0.04	0.13	÷ 2
16 Tau-Fluvalinate(ISO#1&	0.01	0.02	0.04	0.01	0.01	0.02	0.02	0.02	0.01	0.04	
17 Tralomethrin/Deltameth	0.22	0.25	0.20	0.22	0.24	0.16	0.15	0.21	0.04	0.12	÷ 2

Results are in ug/kg

INSTRUMENT: MSDF (5:1 split ratio nci-msd-sim/micro ecd)

PREP METHOD: EPA 3540
PREP DATE: 7/13/09
PREP TECH: NTA/MDT

MATRIX: SL
BATCH: S090162
SPIKE LEVEL: 0.13ug/kg
SPIKE CONC: .25ug/ml

METHOD:CALTEST PYRETHROIDS
ANALYST : RLH/NTA

MDL RANGE : .013 - .13ug/kg
RL : .5ug/kg

Reviewer 1 Richard Hennes Date: 7-29-09

Reviewer 2 Agrelles Date: 7/29/09

Caltest Analytical Laboratory
** RSD Spike Study **

IPR

Instrument ID: MSDF.i
Analyst: RLH

T-Statistic used at the 99% confidence level: 4.5410

Files Used:

(1) \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_18.D 17-JUL-2009 23:18
(2) \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_19.D 17-JUL-2009 23:53
(3) \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_20.D 18-JUL-2009 00:27
(4) \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_21.D 18-JUL-2009 01:02

*** RSD Spike Study (Compound Concentrations ug/Kg)***

Compound Name	1	2	3	4	Avg.	St.Dev.	%RSD	Rec Avg.
Bifenthrin	0.427	0.439	0.464	0.417	0.437	0.020	4.6	65.51
L-Cyhalothrin(ISO#1&2)	0.405	0.412	0.344	0.402	0.391	0.031	8.0	58.61
Permethrin(ISO#1&2)	0.511	0.392	0.425	0.422	0.438	0.051	11.7	65.63
Cyfluthrin(ISO#1-4)	0.540	0.463	0.335	0.363	0.425	0.094	22.1	63.79
Cypermethrin(ISO#1-4)	0.516	0.508	0.350	0.377	0.438	0.087	19.8	65.66
Fenvalerate/Esfenvalerate	0.793	0.774	0.700	0.733	0.750	0.042	5.6	56.25
Tau-Fluvalinate(ISO#1&2)	0.263	0.269	0.130	0.267	0.232	0.068	29.4	34.84
Tralomethrin/Deltamethrin	0.695	0.807	0.724	0.807	0.758	0.058	7.6	56.87
Allethrin	0.397	0.442	0.373	0.386	0.399	0.030	7.5	59.93
Tetramethrin	0.124	0.194	0.079	0.161	0.139	0.050	35.5	20.92
Phenothrin	0.059	0.076	0.054	0.060	0.062	0.010	15.5	9.35
Chlorpyrifos	0.423	0.530	0.514	0.397	0.466	0.066	14.1	69.90
Diazinon	0.379	0.436	0.467	0.396	0.419	0.040	9.5	62.93
Fenpropathrin(Danitol)	0.408	0.431	0.428	0.422	0.422	0.010	2.4	63.34

Results are in ug/kg

INSTRUMENT: MSDF (5:1 split ratio nci-msd-sim/micro ecd)

PREP METHOD: EPA 3540
PREP DATE: 7/13/09
PREP TECH: NTA/MDT

MATRIX: SL
BATCH: S090162
SPIKE LEVEL: 0.67ug/kg
SPIKE CONC: .25ug/ml

INSTRUMENT: MSDF
METHOD:CALTEST PYRETHROIDS
ANALYST : RLH/NTA

Reviewer 1 Richard Heeries Date: 7-29-09

Reviewer 2 [signature] Date: 7/29/09

Data File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_17.D
Report Date: 21-Jul-2009 16:25

Method Blank

CalTest

Semivolatile BNA'S

Data file : \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_17.D
Lab Smp Id: mb-soil
Inj Date : 17-JUL-2009 22:43 MS Autotune Date: 03-OCT-2008 14:31
Operator : RLH Inst ID: MSDF.i
Smp Info : mb-soil
Misc Info : 3;2;;30.0;2;;s090162;
Comment : QUEUE: SMS
Method : \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Meth Date : 21-Jul-2009 16:25 nta Quant Type: ISTD
Cal Date : 17-JUL-2009 16:56 Cal File: 0717F_07.D
Als bottle: 14 QC Sample: BLANK
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: all.sub
Target Version: 4.14
Processing Host: MISSISSIPPI

Concentration Formula: Amt * DF * Vt/ Ws * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Vt	2.000	Volume of final extract (mL)
Ws	30.000	Weight of sample (g)
Cpnd Variable		Local Compound Variable

7/21/09

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS ON-COLUMN (ug/mL)	FINAL (mg/Kg)	SIMILARITY
1	StartList	0	Compound Not Detected.						
$ 2	Decachlorobiphenyl (SS)	394	25.436	25.436	(3.076)	2534	0.00859	0.000572	9917
* 3	4,4'-DBOB (IS)	376	8.269	8.269	(1.000)	8035	0.00100		9505
4	Diazinon	169	Compound Not Detected.						
5	Chlorpyrifos	315	Compound Not Detected.						
6	Allethrin	167	Compound Not Detected.						
7	Bifenthrin	386	Compound Not Detected.						
8	Tetramethrin	331	Compound Not Detected.						
9	Phenothrin	167	Compound Not Detected.						
10	Fenpropathrin(Danitol)	141	Compound Not Detected.						
11	L-Cyhalothrin(ISO#1&2)	241	Compound Not Detected.						
12	Permethrin(ISO#1&2)	207	Compound Not Detected.						
13	Cyfluthrin(ISO#1-4)	207	Compound Not Detected.						
14	Cypermethrin(ISO#1-4)	207	Compound Not Detected.						
15	Fenvalerate/Esfenvalerate	211	Compound Not Detected.						
16	Tau-Fluvalinate(ISO#1&2)	294	Compound Not Detected.						
17	Tralomethrin/Deltamethrin	81	Compound Not Detected.						
18	EndList	0	Compound Not Detected.						

Data File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_17.D
Report Date: 21-Jul-2009 16:25

CalTest

INTERNAL STANDARD COMPOUNDS
AREA AND RT SUMMARY

Instrument ID: MSDF.i
Lab File ID: 0717F_17.D
Lab Smp Id: mb-soil
Analysis Type: SV
Quant Type: ISTD
Operator: RLH
Method File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Misc Info: 3;2;;30.0;2;;s090162;

Calibration Date: 17-JUL-2009
Calibration Time: 16:56
Level: LOW
Sample Type: SOIL

Test Mode:
Use Last Continuing Calibrator.
If Continuing Cal. use Initial Cal. Level 5

COMPOUND	STANDARD	AREA LIMIT LOWER	AREA LIMIT UPPER	SAMPLE	%DIFF
3 4,4'-DBOB (IS)	6793	3397	13586	8035	18.28

COMPOUND	STANDARD	RT LIMIT LOWER	RT LIMIT UPPER	SAMPLE	%DIFF
3 4,4'-DBOB (IS)	8.27	8.17	8.37	8.27	0.00

AREA UPPER LIMIT = +100% of internal standard area.
AREA LOWER LIMIT = - 50% of internal standard area.
RT UPPER LIMIT = + 0.10 minutes of internal standard RT.
RT LOWER LIMIT = - 0.10 minutes of internal standard RT.

Data File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_17.D
Report Date: 21-Jul-2009 16:25

CalTest

RECOVERY REPORT

Client Name:
Sample Matrix: SOLID
Lab Smp Id: mb-soil
Level: LOW
Data Type: MS DATA
SpikeList File:
Sublist File: all.sub
Method File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\PYR_MSD.m
Misc Info: 3;2;;30.0;2;;s090162;

Client SDG: 0204d
Fraction: SV

Operator: RLH
SampleType: BLANK
Quant Type: ISTD

SURROGATE COMPOUND	CONC ADDED mg/Kg	CONC RECOVERED mg/Kg	% RECOVERED	LIMITS
$ 2 Decachlorobiphenyl	0.000667	0.000572	85.88	50-150

Data File: \\delaware\SVOL_D\chem\MSDF.i\0717FMSD.b\0717F_17.D
Date : 17-JUL-2009 22:43
Client ID:
Sample Info: mb-soil

Instrument: MSDF.i

Operator: RLH
Column diameter: 0.25

Column phase: DB-5MS