UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

ENVIRONMENTAL CHEMISTRY SECTION
BUILDING 1105—JOHN C. STENNIS SPACE CENTER
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SEP 27 1993

MEMORANDUM

SUBJECT: KARATE® Method Evaluation - Report No. ECM004W1

FROM: Aubry E. Dupuy, Jr., Chief
BEAD/ACB/Environmental Chemistry Section

TO: Anthony F. Maciorowski, Chief
EFED/Ecological Effects Branch (H7507C)

THRU: Donald A. Marlow, Chief
BEAD/Analytical Chemistry Branch (H7503W)

The EFED/Ecological Effects Branch has requested an Environmental Chemistry Method Evaluation (ECME) on KARATE® in pond water using the ICI Americas Inc. analytical method - "Appendix IV, Part 5, Determination of PP321 Residues in Pond Water by Gas-Liquid Chromatography (PPRAM 125)" contained in the study - PP321: Evaluation of the Impact of Run-Off and Spray-Drift on Aquatic Ecosystems, Using USA Experimental Ponds (Mesocosms). ECS used a condensed version provided by ICI Agrochemicals entitled "ICI Agrochemicals Residue Analytical Method Number 125b - The Determination of Residues of PP321 in Water Following Sampling by a Solid-Phase Extraction Technique." The method evaluation has specified a method blank and triplicate analyses on pond water samples fortified at the limit of detection (LOD), 10 x limit of detection, and 100 x limit of detection.

The attached method evaluation report includes three parts:

Part I: Summary and Conclusions

In this section any problems encountered with the method and how they were handled are discussed. ECS's opinion of how well the method performed is also presented.

Part II: Analytical Results

In this section the individual results of each samples at each spiking level for each matrix are listed. The relative standard deviation (RSD) for each spiking level is also presented here.

Part III: Experimental Details

In this section any modification(s) that were made to this section, instrument parameters, spiking levels, explanation of instrument calibration, representative sample and standard chromatograms and standard curves are listed and/or discussed.

If you have any questions concerning this report, please contact Christian Byrne at (601) 688-3213 or me at (601) 688-3212.

Attachments

cc: Danny McDaniel, QA Coordinator
BEAD/ACB/Environmental Chemistry Section
Dan Rieder
EFED/Ecological Effects Branch (H7507C)

Environmental Chemistry Method Validation Report Number ECM 0004W1

KARATE® [lambda-Cyhalothrin (PP321)] in Pond Water

Environmental Chemistry Section
Analytical Chemistry Branch
Biological and Economic Analysis Division

September 15, 1993

Prepared by: Christian Byrne
Christian Byrne, ECS Chemist

Reviewed by: Danny McDaniel
Danny McDaniel, QA Coordinator

TABLE of CONTENTS

PART I

Summary and Conclusions

We have completed the Environmental Chemistry Method Evaluation (ECME) on KARATE® [lambda-Cyhalothrin (PP321)] for pond water. The method appears to be suitable to gather residue data for KARATE® [lambda-Cyhalothrin (PP321)] at levels at or greater than 10 ppt (parts-per-trillion).

The method used for the ECME is entitled **ICI Agrochemicals Residue Analytical Method Number 125b - The Determination of Residues of PP321 in Water Following Sampling by a Solid-Phase Extraction Technique** (Appendix 1). This is a condensed version of **Appendix IV Part 5 Determination of PP321 Residues in Pond Water by Gas-Liquid Chromatography (PPRAM 125)** of the Study **PP321: EVALUATION OF THE IMPACT OF RUN-OFF AND SPRAY-DRIFT ON AQUATIC ECOSYSTEMS, USING USA EXPERIMENTAL PONDS (MESOCOSMS).** The performing laboratory was Jealotts Hill Research Station, ICI Plant Protection Division, Bracknell, Berkshire, U.K.

The analytical method involves the loading of the water sample onto a pre-conditioned C_8/SAX (strong anion exchange) solid phase extraction cartridge. The cartridge is then fortified with a known amount of internal standard (R171554). The cartridge is then eluted with acetonitrile, diethyl ether: hexane (70:30), and acetonitrile washes, co-extractives removed, and the analyte concentrated onto an additional C_8 solid phase extraction cartridge. The analyte is eluted with hexane and the extract concentrated to volume. Final quantitative results are determined using on-column capillary gas-liquid chromatography using internal standardization.

The Limit of Determination (LOD) of the registrant was set at 1 ng/L (ppt). ECS calculated the Limit of Determination (LOD) to be 10 ppt, as calculated from the results of the analysis of pond water spiked at concentrations of 1, 10, and 100 ppt with lambda-Cyhalothrin (PP321). The accuracy and precision of the results between ECS and the registrant at spiking concentrations of 10 ppt were comparable . The mean recovery of lambda-Cyhalothrin in pond water spiked at 10 ppt for ECS was 97.5% with a relative standard deviation (RSD) of 17.6. The mean recovery of lambda-Cyhalothrin in pond water spiked at 10 ppt for ICI

Agrochemicals was 100% with an RSD of 16. The mean recovery of the internal standard, R171554, spiked at 10 ppt for ECS was 109% with a RSD of 17.9. The mean recovery of R171554 for ICI Agrochemicals was 70% with a RSD of 21.

ECS estimates that it takes approximately three (3) working days to start and finish one set of five (5) samples with appropriate blanks and standards.

The analytical method validation proceeded very smoothly; however, there are several problems with the comparison of the data between the results of ECS and the registrant.

Problems Discovered During ECS Method Evaluation

Problem 1:

The limit of determination of the method, as stated by the registrant, was "determined by fortification of untreated samples at low levels and subjecting them to the complete analytical procedure. The chromatographic response obtained for these recoveries at the retention time of PP321 (enantiomer B) should exceed the background signal noise by a factor of at least four to be considered an acceptable quantitative limit of determination. In addition, the precision of measurement at this level should not exceed a coefficient of variation of ± 15%. The LOD for enantiomer B (PP321) was set at 1 ng/L (ppt)." There was neither accuracy or precision data nor chromatographic results provided at this designed LOD concentration. This LOD was calculated from the recovery data determined at higher concentrations.

Recoveries of three replicate samples of pond water spiked with lambda-Cyhalothrin (PP321) at a concentration of 1 ppt were 125%, 95%, and Not Detected (ND). One of the three replicate samples contained no measurable concentration of lambda-Cyhalothrin above background noise. In addition, none of the three replicates possessed signal to noise ratio greater than the four to one ratio as stated as necessary for an acceptable quantitative limit of determination. Recoveries of three replicate samples of pond water spiked with lambda-Cyhalothrin at a concentration of 10 ppt met the definition of "an acceptable quantitative limit of determination (LOD).

Problem 2:

The representative chromatograms in the **ICI Agrochemicals Residue Analytical Method Number 125b** apparently do not include a matrix control (blank). The chromatogram for the control (untreated) sample (Figure 49c) possesses a baseline that appears to be that of a solvent blank. The chromatogram of the pond water spiked with PP321 (Figure 49d) appears to have a degree of background noise that would be attributed to the matrix effects of the solvents, solid phase extraction cartridges, and pond water.

The chromatograms of the reagent water matrix control and pond water matrix control, in addition to the reagent matrix control, of the ECS ECME revealed varying degrees of matrix background noise. These results would call into question the exact manner in which the ICI Agrochemicals LOD of 1 ppt was determined.

PART II

Analytical Results

Method: On-Column Gas Chromatography Determination for KARATE® [lambda-Cyhalothrin (PP321)], **ICI Agrochemicals Residue Analytical Method Number 125b,** Jealotts Hill Research Station, Bracknell, Berkshire, U.K.

Results:

lambda-CYHALOTHRIN (PP321)

Added (1 ppt)	Found (ppt)		Found (% Recovery)
Pond Water Sample 1	1.25		125%
Pond Water Sample 2	0.95		95%
Pond Water Sample 3	ND		ND
		Mean	73.5%
		SD	65.3%
		RSD	89.0

Added (10 ppt)	Found (ppt)		Found (% Recovery)
Pond Water Sample 1	8.1		81.2%
Pond Water Sample 2	9.6		96.0%
Pond Water Sample 3	11.5		115.4%
		Mean	97.5%
		SD	17.1%
		RSD	17.6

Added (100 ppt)	Found (ppt)	Found (% Recovery)
Pond Water Sample 1	86.9	86.9%
Pond Water Sample 2	82.3	82.3%
Pond Water Sample 3	77.9	77.9%
	Mean	82.3%
	SD	4.5%
	RSD	5.5

NOTES:

(1) Sample 1,2,3... are separate analyses using the same pond water source

(2) SD - Standard Deviation; RSD - Relative Standard Deviation

PART III

Experimental Details

General description of method:

A 250 ml aliquot of pond water (1000 ml aliquot for spiked pond water samples at concentrations of 1 ppt) was filtered through a pre-conditioned C_8/SAX solid phase extraction cartridge(s) at a flow rate of 50 ml/min. The cartridge is fortified with the internal standard (R171554) at an appropriate level. The cartridge is eluted with acetonitrile, ethyl ether:hexane (70:30), and finally acetonitrile. The organic eluates are concentrated by rotary evaporation, dilute to volume, and mixed with ultra-pure water. This mixture is filtered through an additional pre-conditioned C_8 solid phase extraction cartridge at a flow rate of 1 ml/min. The cartridge is eluted with hexane. This extract is concentrated by rotary evaporation, transferred to a 5 ml centrifuge tube, and diluted to volume. The extract is stored at 4°C for analysis by gas chromatography.

Special precautions to be taken:

None

Source of analytical reference standards:

Standards were received from two sources:

(1) Pesticides and Industrial Chemical Repository (MD-8), United States Environmental Protection Agency (USEPA), Research Triangle Park, North Carolina 27709

lambda-Cyhalothrin, Lot #FYV2, 99.7% purity, dated May 3, 1990, 100 mg (Appendix 2)

(2) ICI Agrochemicals, Jealotts Hill Research Station, Plant Protection Division, Bracknell, Berkshire, United Kingdom.

R171554, Lot ISJ/R171554/No. 1, 95% purity, 100 mg (Appendix 2)

Source of sample matrix:

Pond water was taken from a series of gravel pit ponds on site at John C. Stennis Space Center, Mississippi. The water quality parameters are listed in Appendix 3.

Instrumentation for quantitation:

Gas Chromatograph: Hewlett Packard Model 5890 Series II

Inlet: Hewlett Packard 5890A Series II Programmable Cool On-Column Inlet

Injector: Hewlett Packard 7673A Automatic Sampler

Detector: Hewlett Packard Ni^{63} Electron Capture Detector

Column: One meter uncoated deactivated fused silica retention gap of 0.52 mm ID attached to the front of a 30 meter x 0.32 mm ID fused silica column with a 0.25 μm of 5% phenyl/95% methylsilicone liquid phase (J&W Scientific DB-5).

Data Handling: Hewlett Packard 3359A Chromatographic Worksystem

Instrument for confirmation: None applicable

Instrument parameters:

Instrument: Hewlett Packard 5890A

Temperature Program:	Oven - 50°C programmed at 40°C/min to 90°C (hold 1 minute), ramp at 10°C/min to 240°C (hold 21 minutes), ramp at 10°C/min to 250°C (hold 10 minutes)
Temperature Program:	Injector - 40°C programmed at 150°C/min to 250°C (hold 35 minutes)

Detector Temperature:	300°C
Carrier Gas:	Helium at 1.5 ml/min
Make-Up Gas:	Nitrogen at 30 ml/min
Retention Times:	lambda-Cyhalothrin - 22.46 minutes
	R171554 - 24.68 minutes

Notes on Analytical Procedure:

(1) In the **ICI Agrochemicals Residue Analytical Method Number 125b**, the water was sampled through the sorbent cartridge using a hand pump at a flow rate of 50 ml/min. This was modified in the ECS ECME by transferring the water sample through the pretreated sorbent cartridge vertically without dissolvation occurring.

(2) In the **ICI Agrochemicals Residue Analytical Method Number 125b**, a water sample of 250 ml would be filtered through a pre-conditioned solid phase extraction cartridge. In water samples with anticipated concentrations of lambda-Cyhalothrin of approximately 1 ppt, a 1000 ml water sample would be required and the water would be filtered through three pre-conditioned solid phase extraction cartridges with each cartridge filtering approximately 335 ml at a flow rate of 50 ml/min. The three eluate mixtures would be combined and concentrated for continuation through the method.

Chromatograms and Calibration Curves:

A. Calibration Standards (lambda-Cyhalothrin & R171554)

CS-0:	0	ppt equivalent	(0 pg/µl)
CS-1:	1	ppt equivalent	(1 pg/µl)
CS-2:	2.5	ppt equivalent	(2.5 pg/µl)
CS-3:	4	ppt equivalent	(4 pg/µl)
CS-4:	5	ppt equivalent	(5 pg/µl)
CS-5:	10	ppt equivalent	(10 pg/µl)
CS-6:	25	ppt equivalent	(25 pg/µl)
CS-7:	50	ppt equivalent	(50 pg/µl)
CS-8:	100	ppt equivalent	(100 pg/µl)

B. lambda-Cyhalothrin Fortification, Pond Water @ 1 ppt

LC-0-1-1000	Pond Water Matrix Control		1000 ml
LC-1-1-1000	Pond Water Fortified @	1.0 ppt	1000 ml
LC-1-2-1000	Pond Water Fortified @	1.0 ppt	1000 ml
LC-1-3-1000	Pond Water Fortified @	1.0 ppt	1000 ml

C. lambda-Cyhalothrin Fortification, Pond Water @ 10 ppt

LC-0-1-250	Pond Water Matrix Control		250 ml
LC-10-1-250	Pond Water Fortified @	10.0 ppt	250 ml
LC-10-2-250	Pond Water Fortified @	10.0 ppt	250 ml
LC-10-3-250	Pond Water Fortified @	10.0 ppt	250 ml

D. lambda-Cyhalothrin Fortification, Pond Water @ 100 ppt

LC-100-1-250	Pond Water Fortified @ 100.0 ppt	250 ml
LC-100-2-250	Pond Water Fortified @ 100.0 ppt	250 ml
LC-100-3-250	Pond Water Fortified @ 100.0 ppt	250 ml

Notes:

(1) Abbreviations -

CS -

Calibration Standards

LC-0-1-1000 -

lambda-Cyhalothrin-Matrix Control-Replicate #1-
Sample Volume 1000 ml

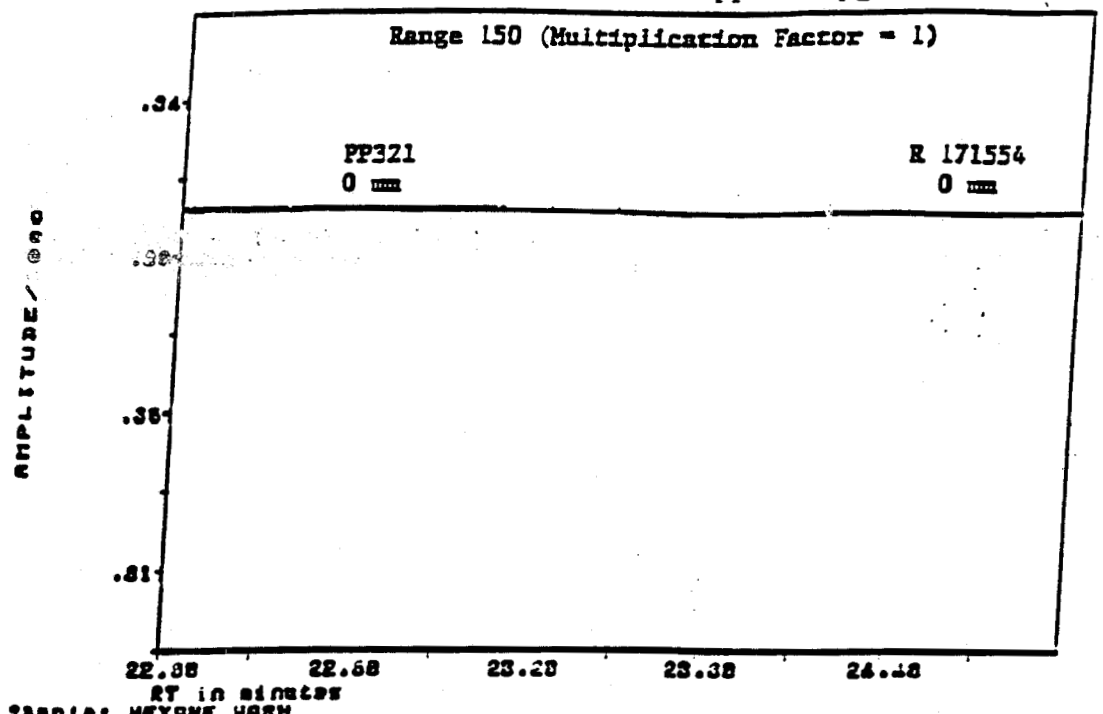
LC-1-1-250 -

lambda-Cyhalothrin-Fortified Sample @ 1 ppt-Replicate #1-
Sample Volume 250 ml

LC-10-3-250 -

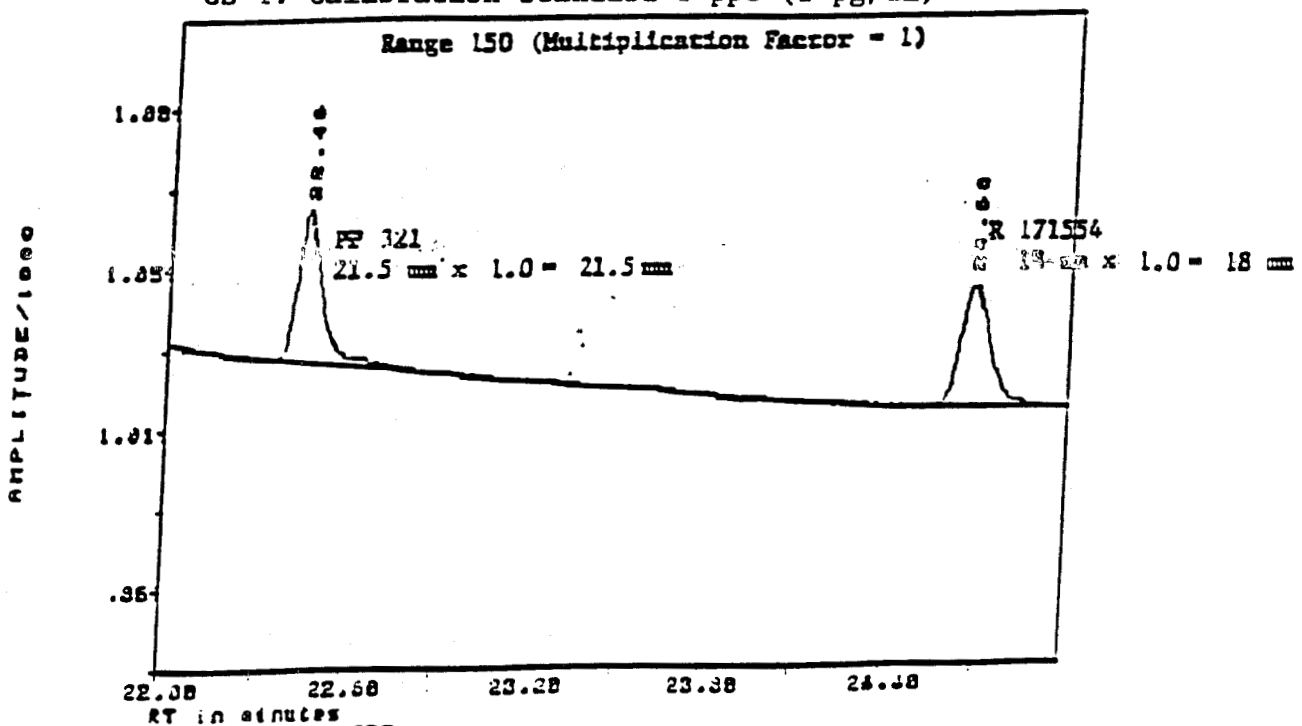
lambda-Cyhalothrin-Fortified Sample @ 10 ppt-Replicate #3-
Sample Volume 250 ml

CS-0: Calibration Standard 0 ppt (0 pg/ul)



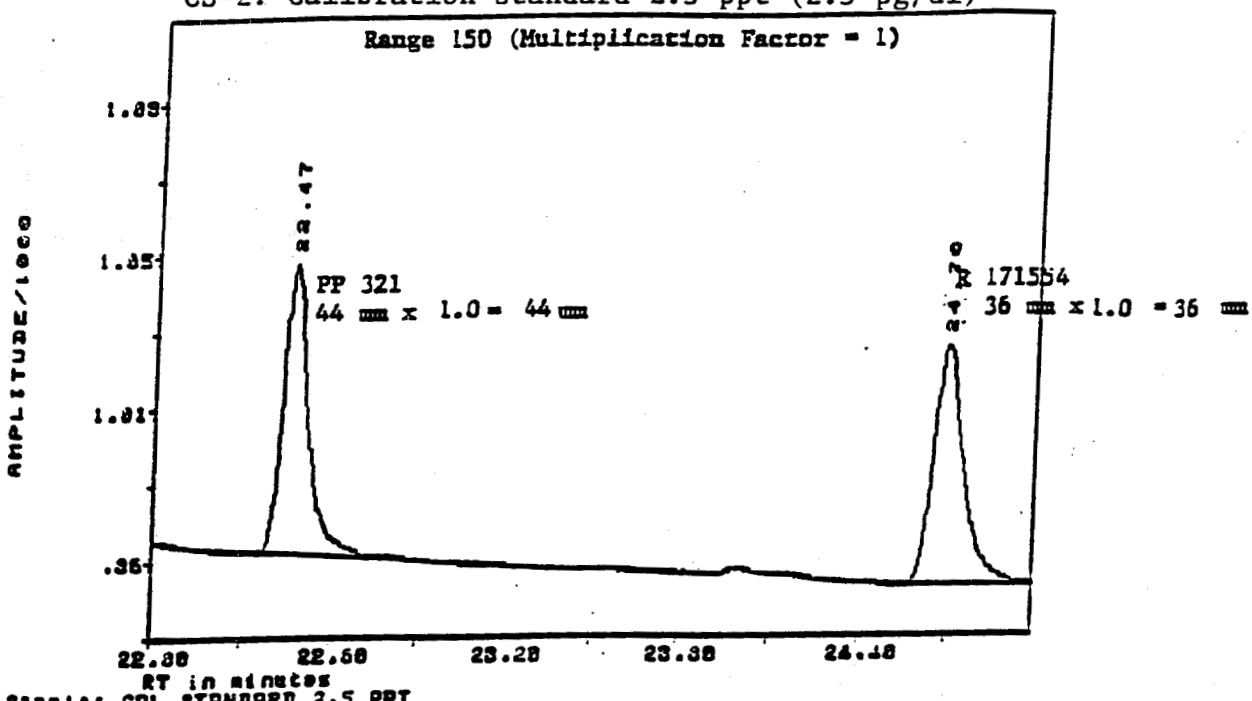
Sample: HEXANE WASH
Method: /DATA/MRID4651S9012.MTH
Result: /DATA/PP321ECMM20022.RES
P.Data: /DATA/PP321ECMM20022.RES
Injected on Sat Jun 13, 1992 7:46:39 pm

CS-1: Calibration Standard 1 ppt (1 pg/ul)



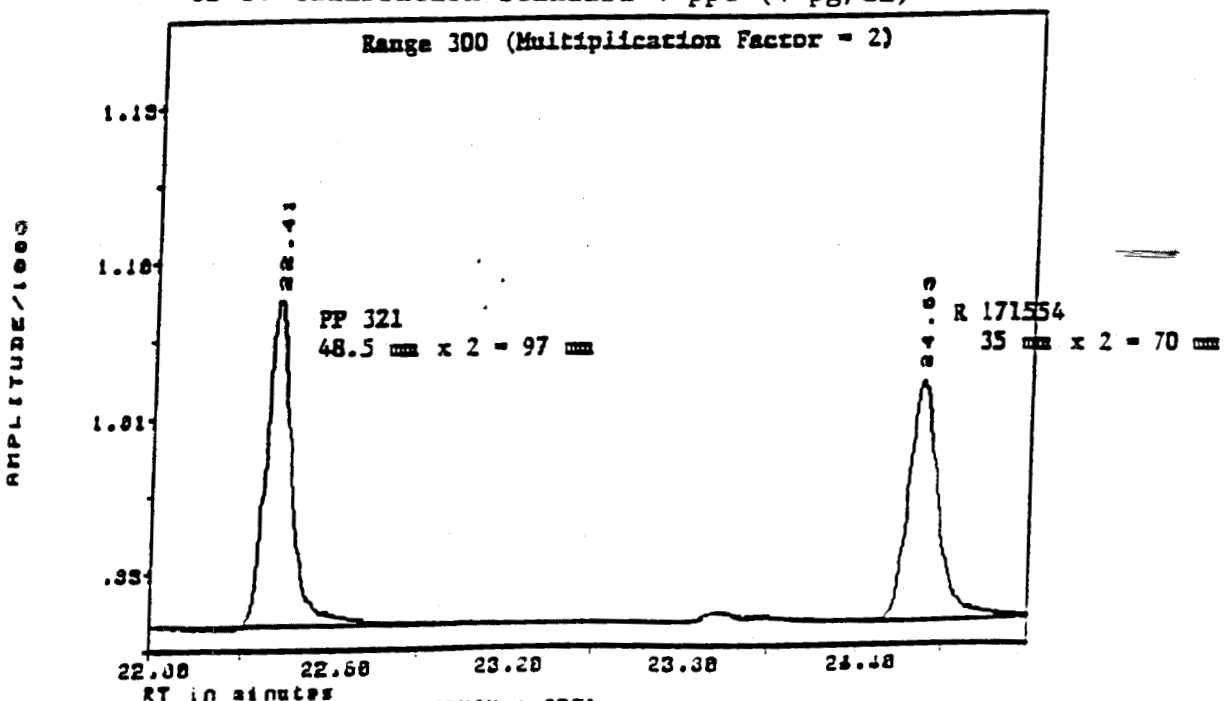
Sample: CAL STANDARD 1 PPT
Method: /DATA/MRID4651S9012.MTH
Result: /DATA/PP321ECMM20017.RES
Injected on Sat Jun 13, 1992 1:01:10 pm

CS-2: Calibration Standard 2.5 ppt (2.5 pg/ul)



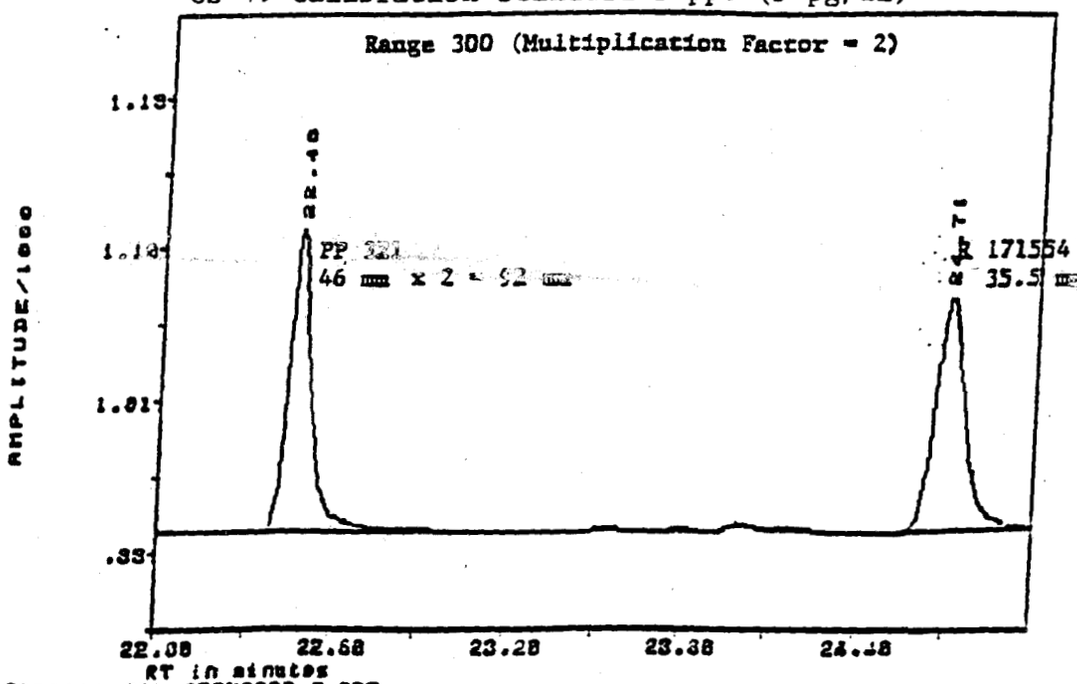
Sample: CAL STANDARD 2.5 PPT
Method: /DATA/MRID405159012.MTH
Result: /DATA/PP321ECMM20010.RES
Injected on Sat Jun 13, 1992 2:22:48 pm

CS-3: Calibration Standard 4 ppt (4 pg/ul)



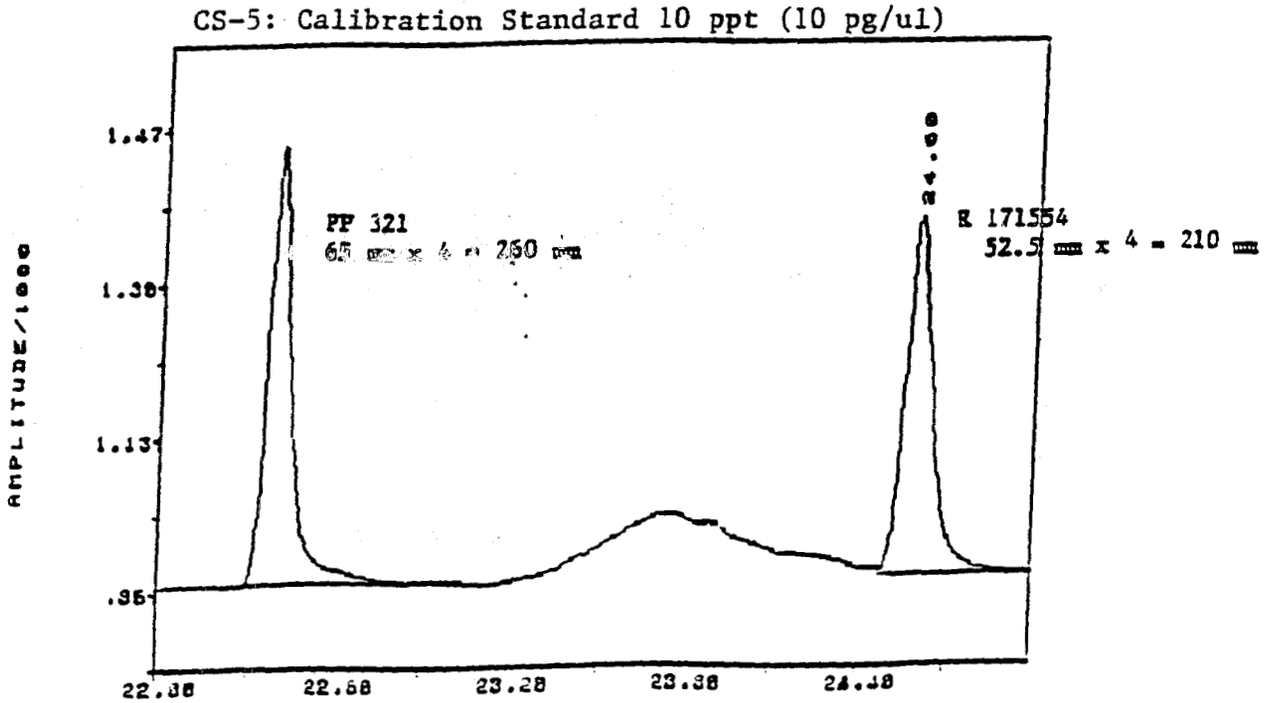
Sample: CAL STANDARD 4 PPT (EQUIV=4 PPT)
Method: /DATA/MRID405159012.MTH
Result: /DATA/PP321ECMM20023.RES
Injected on Sat Jun 13, 1992 9:10:26 pm

CS-4: Calibration Standard 5 ppt (5 pg/ul)



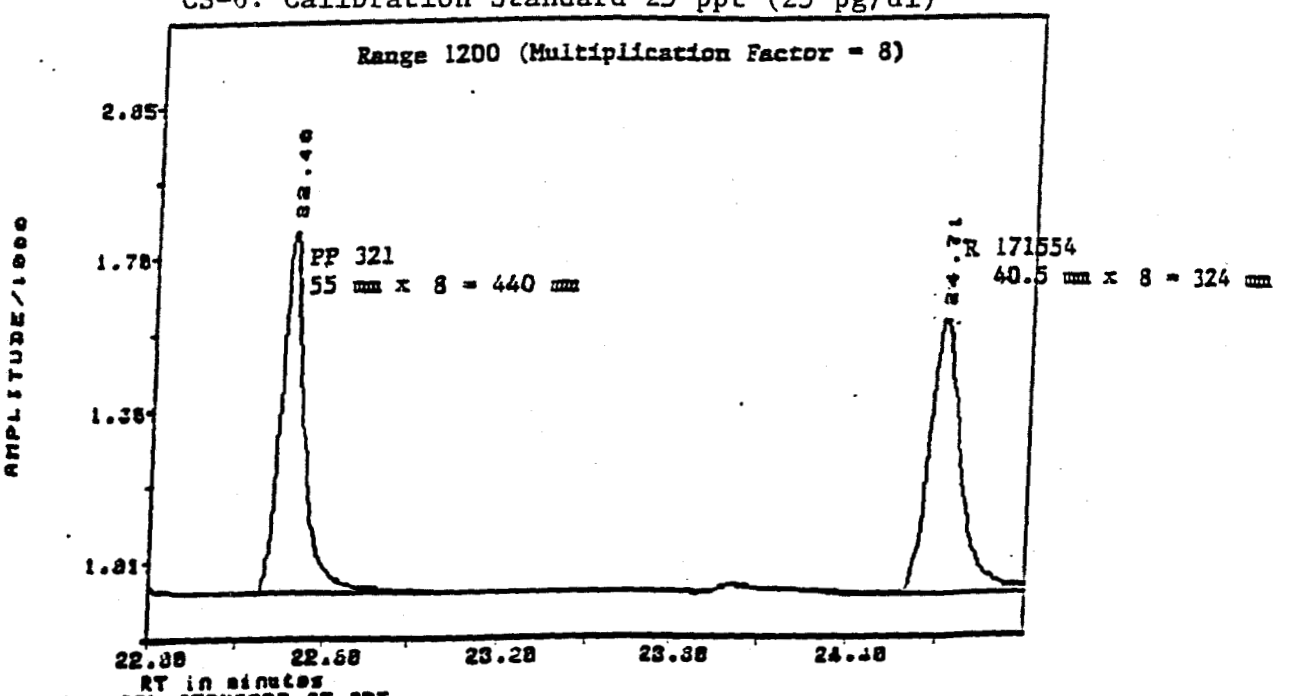
Sample: CAL STANDARD 5 PPT
Method: /DATA/MKID40515B012.MTH
Result: /DATA/PP321ECHM20015.RES
Injected on Sat Jun 13, 1992 3:44:07 pm

CS-5: Calibration Standard 10 ppt (10 pg/ul)



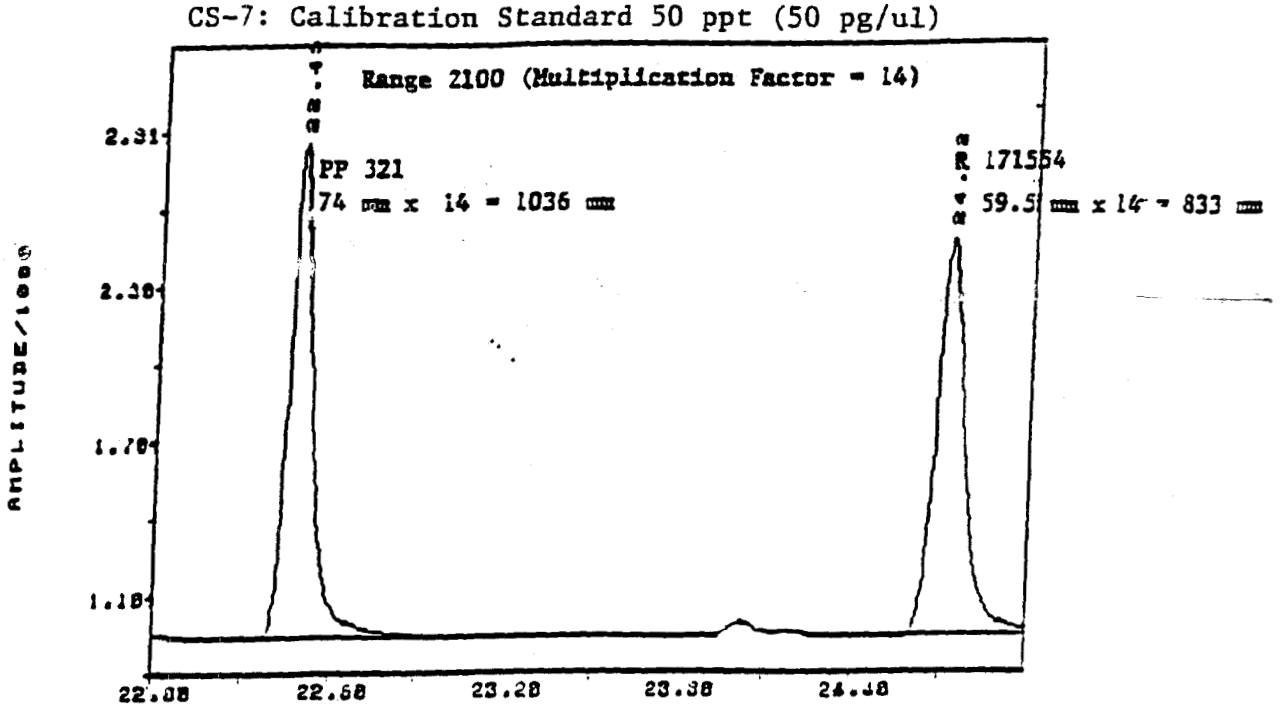
Sample: CAL STANDARD 10 PPT
Method: /DATA/MKID40515B01.MTH
Result: /DATA/PP321ECHM20005.RES
Injected on Fri Jun 12, 1992 8:43:08 pm

CS-6: Calibration Standard 25 ppt (25 pg/ul)



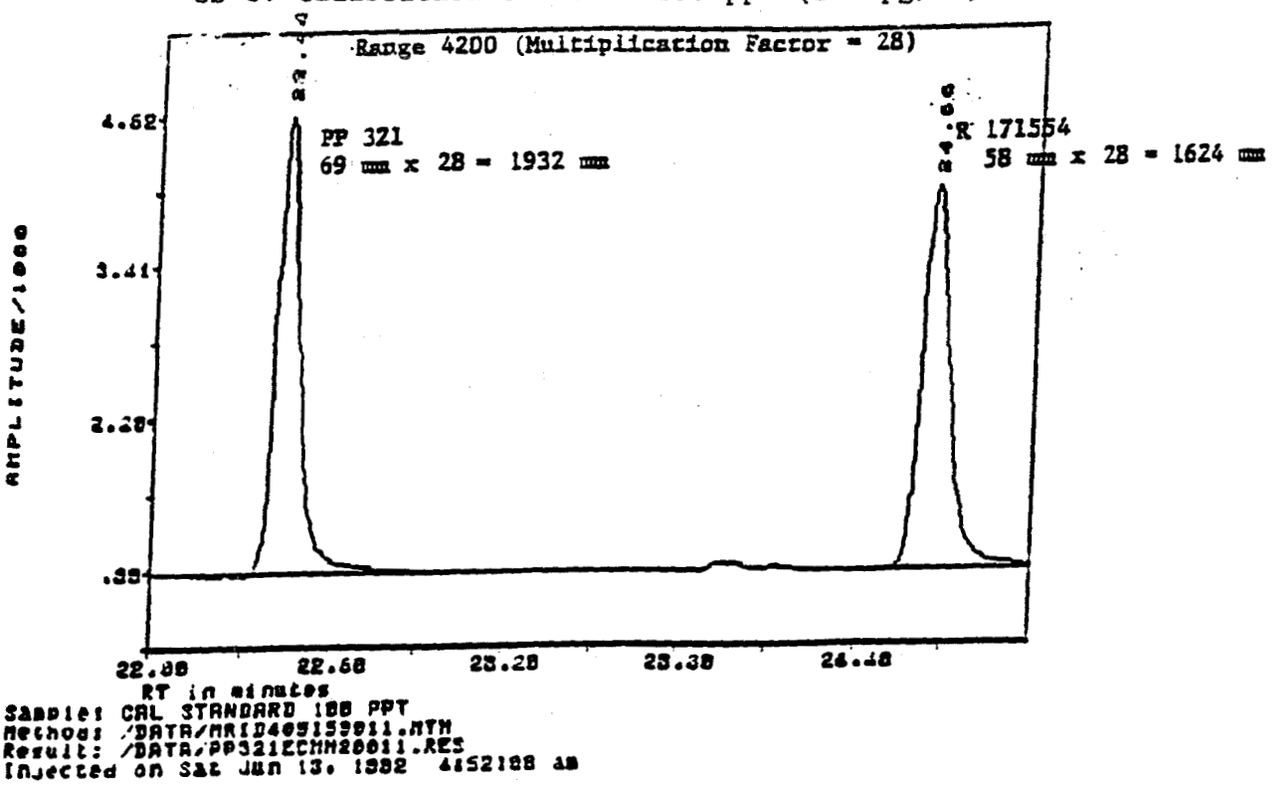
Sample: CAL STANDARD 25 PPT
Method: /DATA/MRID465159012.MTH
Result: /DATA/PP321ECMM20020.RES
Injected on Sat Jun 13, 1992 5185139 pa

CS-7: Calibration Standard 50 ppt (50 pg/ul)



Sample: CAL STANDARD 50 PPT
Method: /DATA/MRID465159012.MTH
Result: /DATA/PP321ECMM20021.RES
Injected on Sat Jun 13, 1992 6127109 pa

CS-8: Calibration Standard 100 ppt (100 pg/ul)

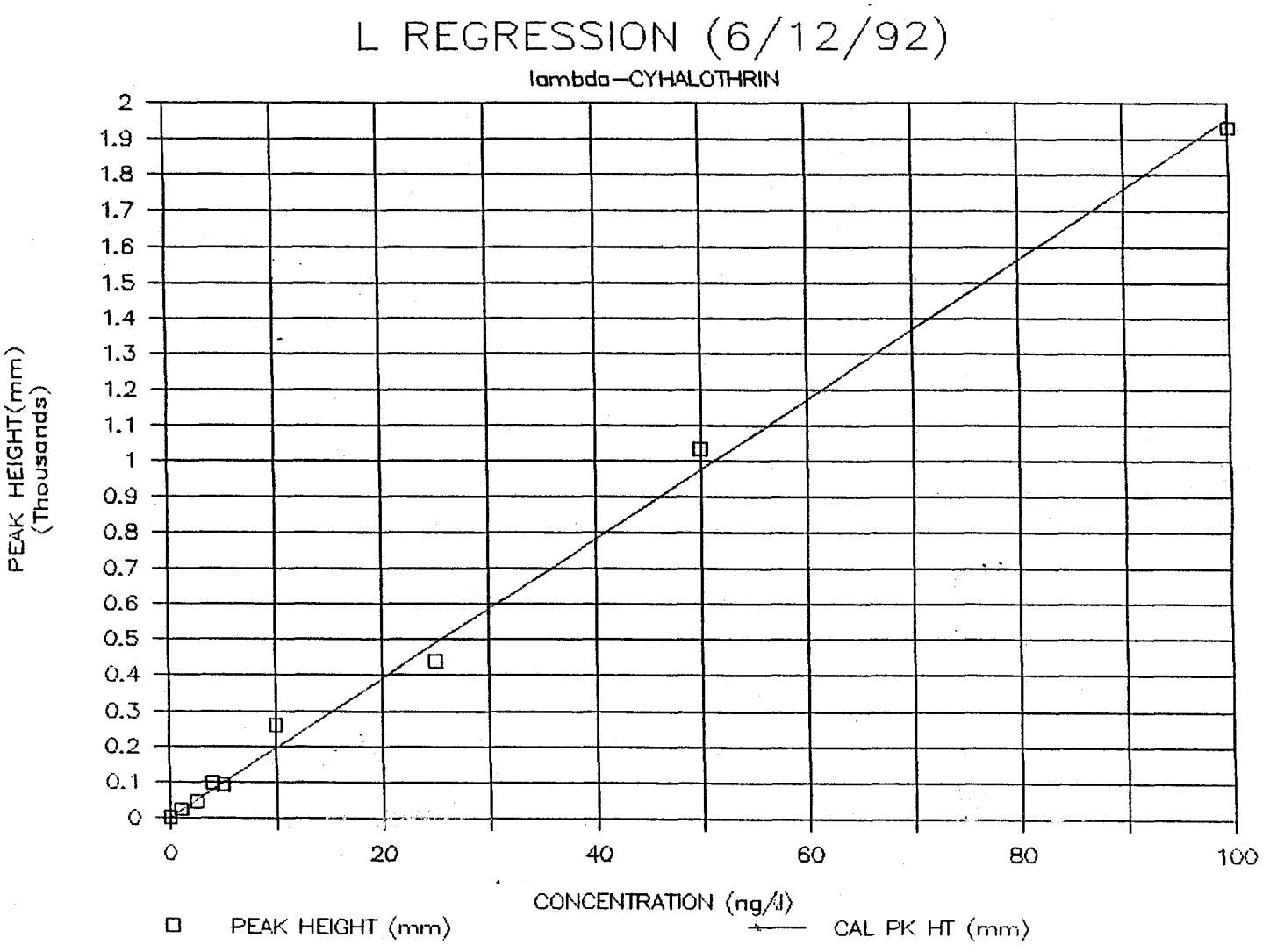


LINEAR REGRESSION OF THE RESPONSE OF lambda-CYHALOTHRIN

CONCENTRATION (ppt)	PEAK HEIGHT (mm)	CALCULATED PEAK HEIGHT (mm)
0.0	0	0
1.0	21.5	20
2.5	44	49
4.0	97	78
5.0	92	98
10.0	260	196
25.0	440	489
50.0	1036	978
100.0	1932	1956

Regression Output:

Constant	0
Std Err of Y Est	36.91469
R Squared	0.996772
No. of Observations	9
Degrees of Freedom	8
X Coefficient(s)	19.55658
Std Err of Coef.	0.320413

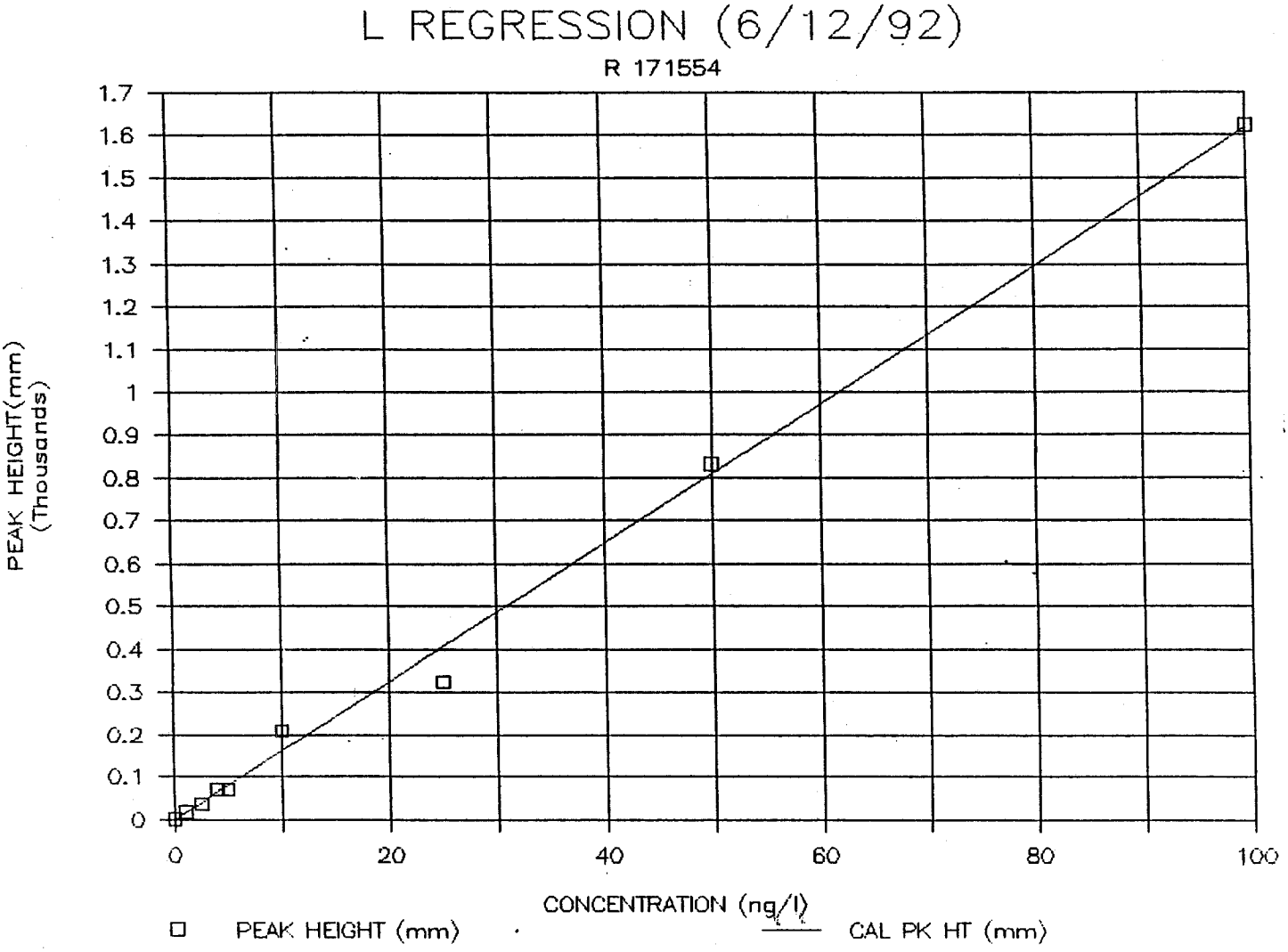


LINEAR REGRESSION OF THE RESPONSE OF R-171554 (I.S.)

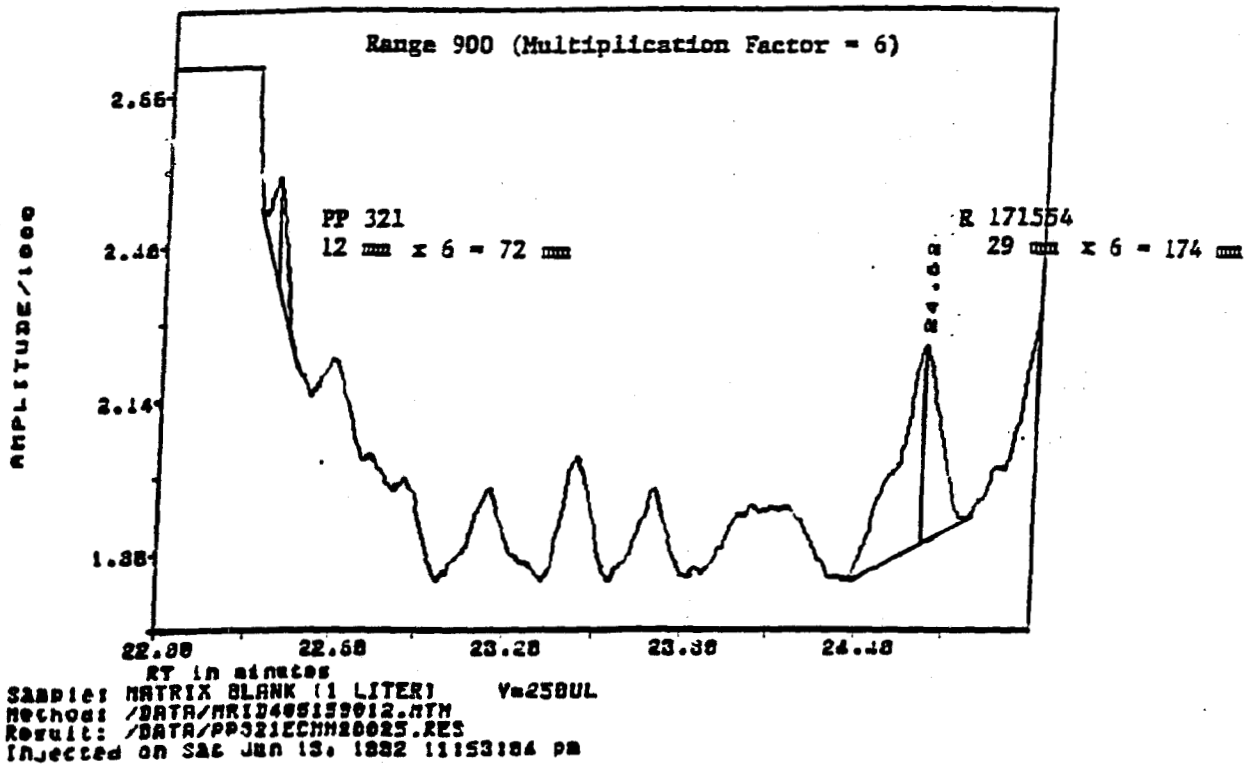
CONCENTRATION (ppt)	PEAK HEIGHT (mm)	CALCULATED PEAK HEIGHT (mm)
0.0	0	0
1.0	17	16
2.5	36	40
4.0	70	65
5.0	71	81
10.0	210	162
25.0	324	405
50.0	833	810
100.0	1624	1620

Regression Output:

Constant	0
Std Err of Y Est	34.56486
R Squared	0.995958
No. of Observations	9
Degrees of Freedom	8
X Coefficient(s)	16.19738
Std Err of Coef.	0.300017

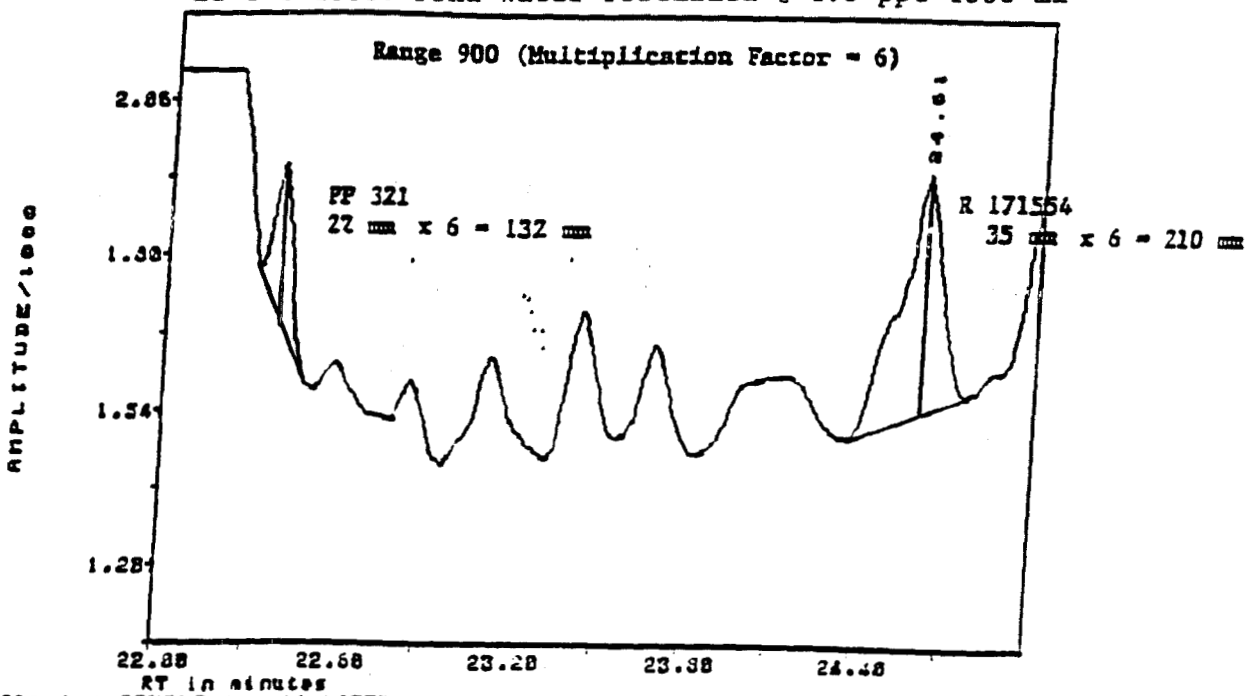


LC-0-1-1000: Pond Water Matrix Control 1000 ml



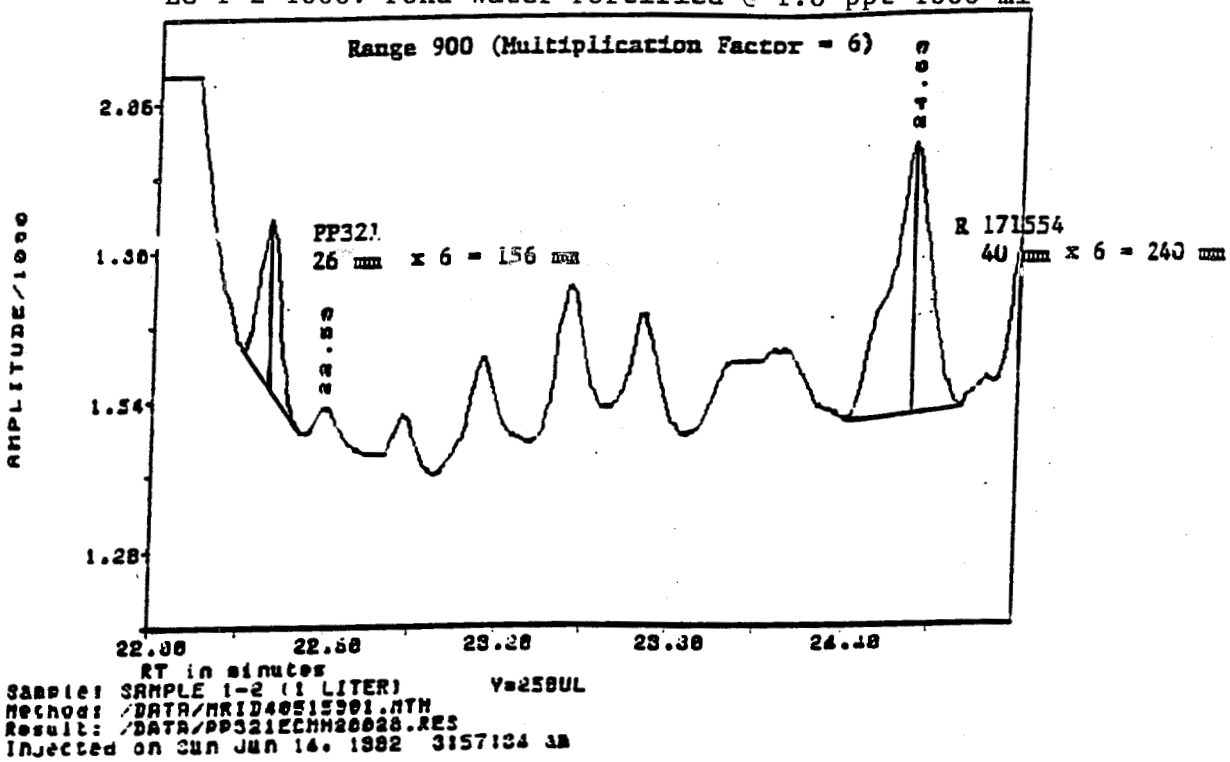
Sample: MATRIX BLANK (1 LITER) Vx=250UL
Method: /DATA/MRID405159912.MTH
Result: /DATA/PP321ECHM20025.RES
Injected on Sat Jun 13, 1992 11:53:04 pm

LC-1-1-1000: Pond Water Fortified @ 1.0 ppt 1000 ml

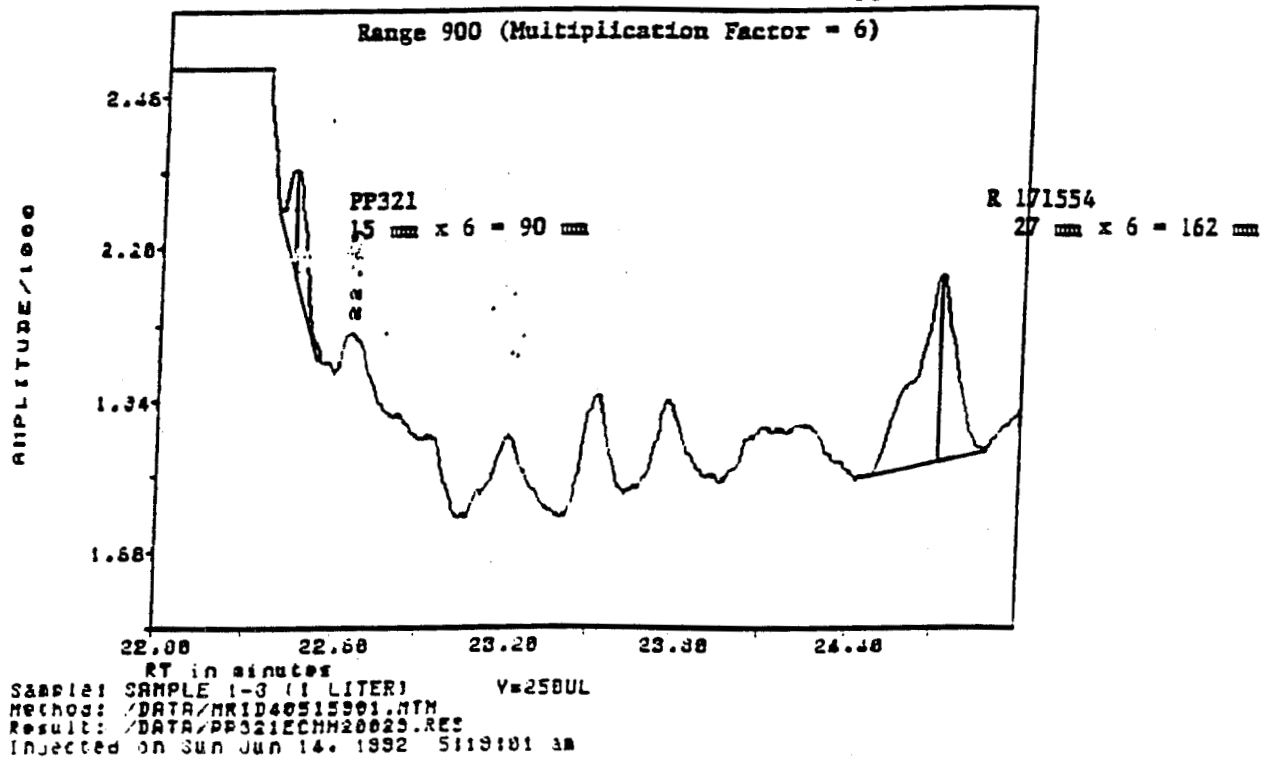


Sample: SAMPLE 1-1 (1 LITER) Vx=250UL
Method: /DATA/MRID405159912.MTH
Result: /DATA/PP321ECHM20027.RES
Injected on Sun Jun 14, 1992 2:35:05 am

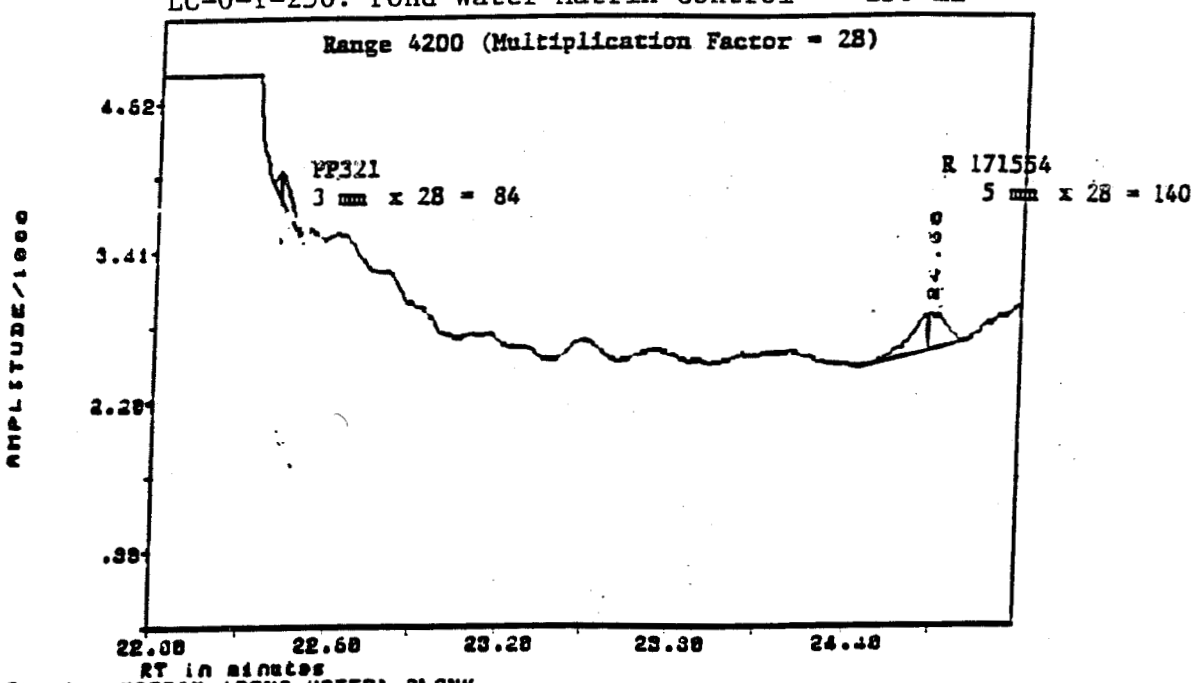
LC-1-2-1000: Pond Water Fortified @ 1.0 ppt 1000 ml



LC-1-3-1000: Pond Water Fortified @ 1.0 ppt 1000 ml

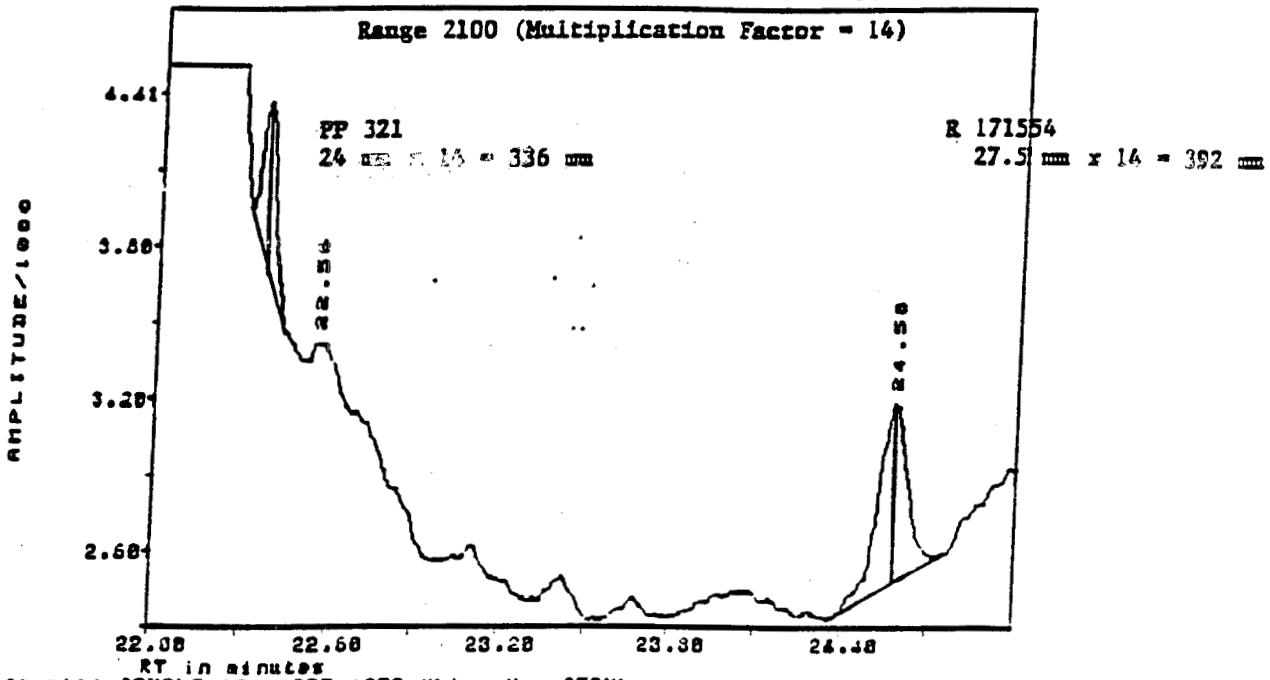


LC-0-1-250: Pond Water Matrix Control 250 ml



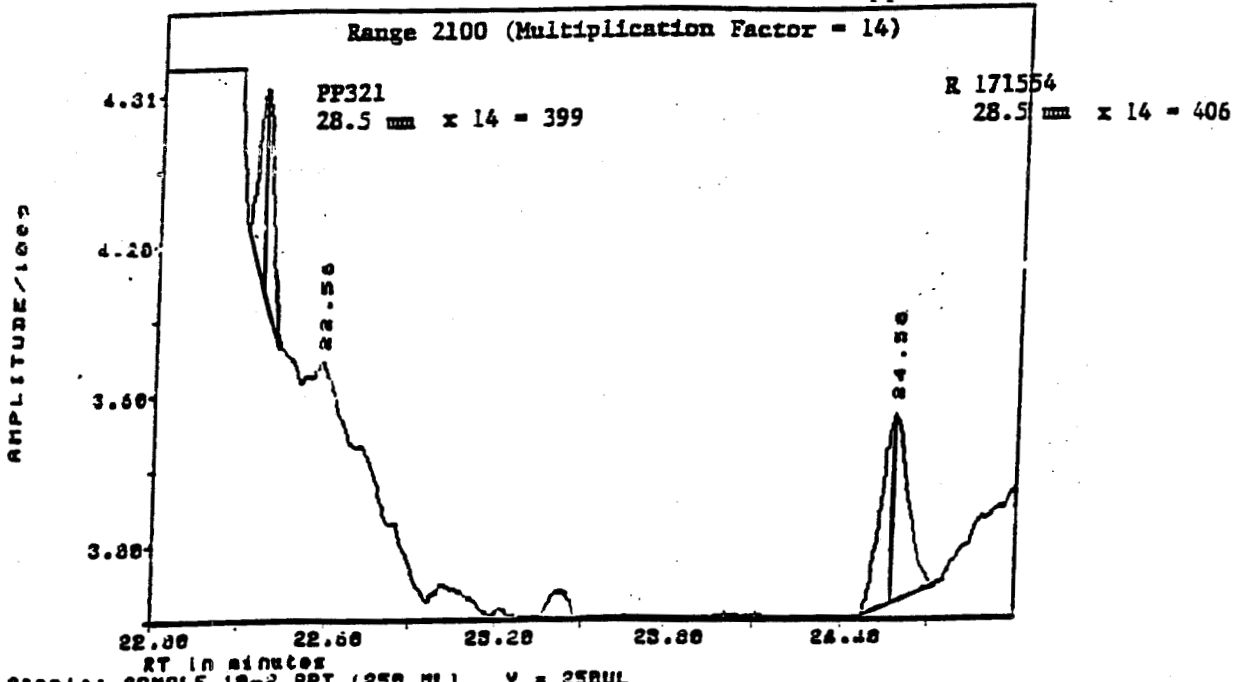
Sample: MATRIX (POND WATER) BLANK
Method: /DATA/MRID40515901.MTH
Result: /DATA/PP321ECMM20004.RES
Injected on Fri Jun 12, 1992 7:21:29 pm

LC-10-1-250: Pond Water Fortified @ 10.0 ppt 250 ml



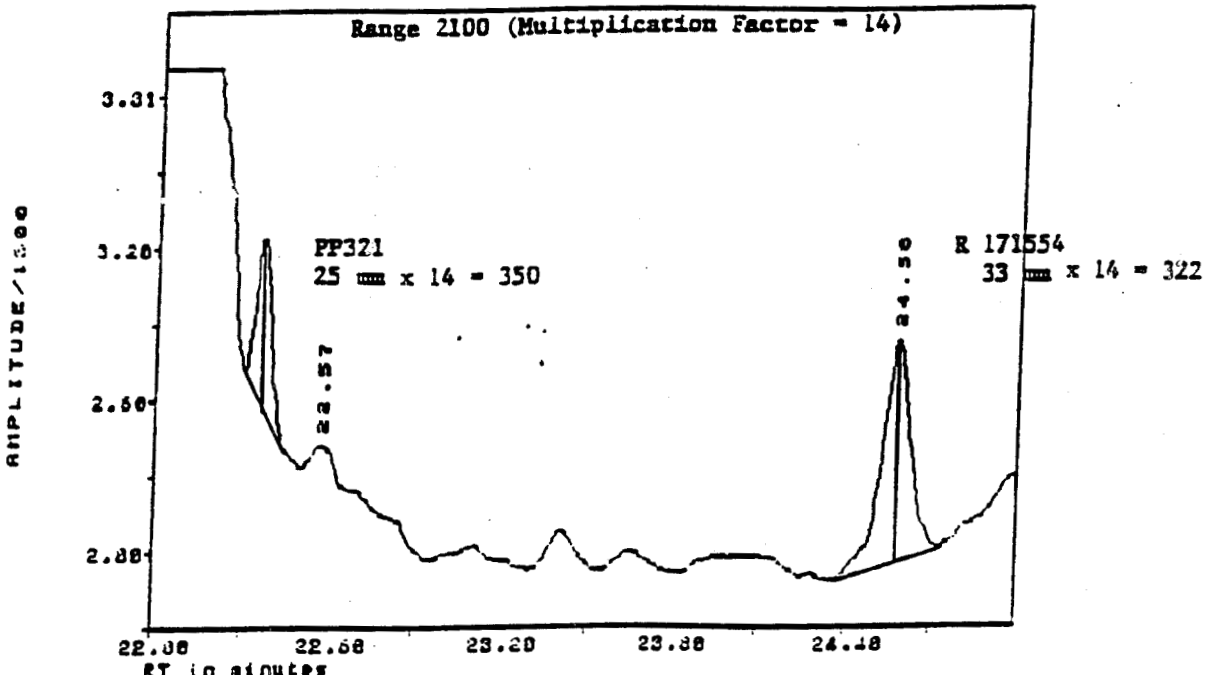
Sample: SAMPLE 10-1 PPT (250 ML) V = 250UL
Method: /DATA/MRID40515901.MTH
Result: /DATA/PP321ECMM20007.RES
Injected on Fri Jun 12, 1992 11:25:03 pm

LC-10-2-250: Pond Water Fortified @ 10.0 ppt 250 ml



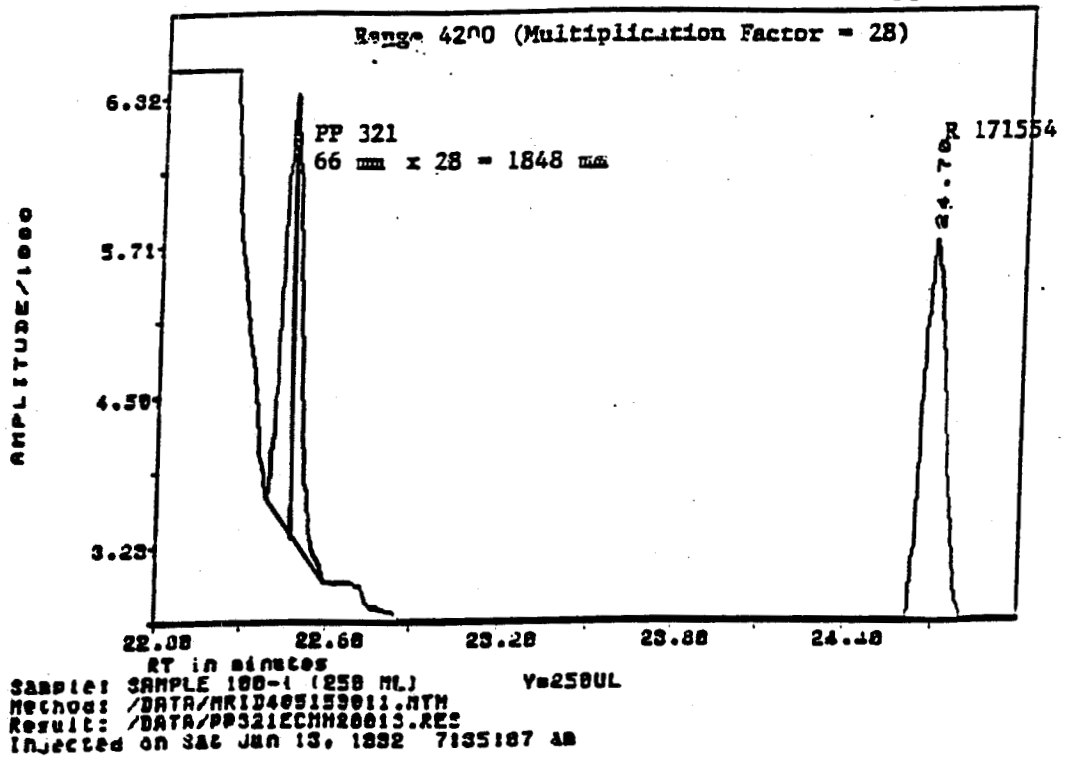
Sample: SAMPLE 10-2 PPT (250 ML) Y = 250UL
Method: /DATA/MRID40515901.MTH
Result: /DATA/PP321ECHH20000.RES
Injected on Sat Jun 13, 1992 12:47:35 AM

LC-10-3-250: Pond Water Fortified @ 10.0 ppt 250 ml

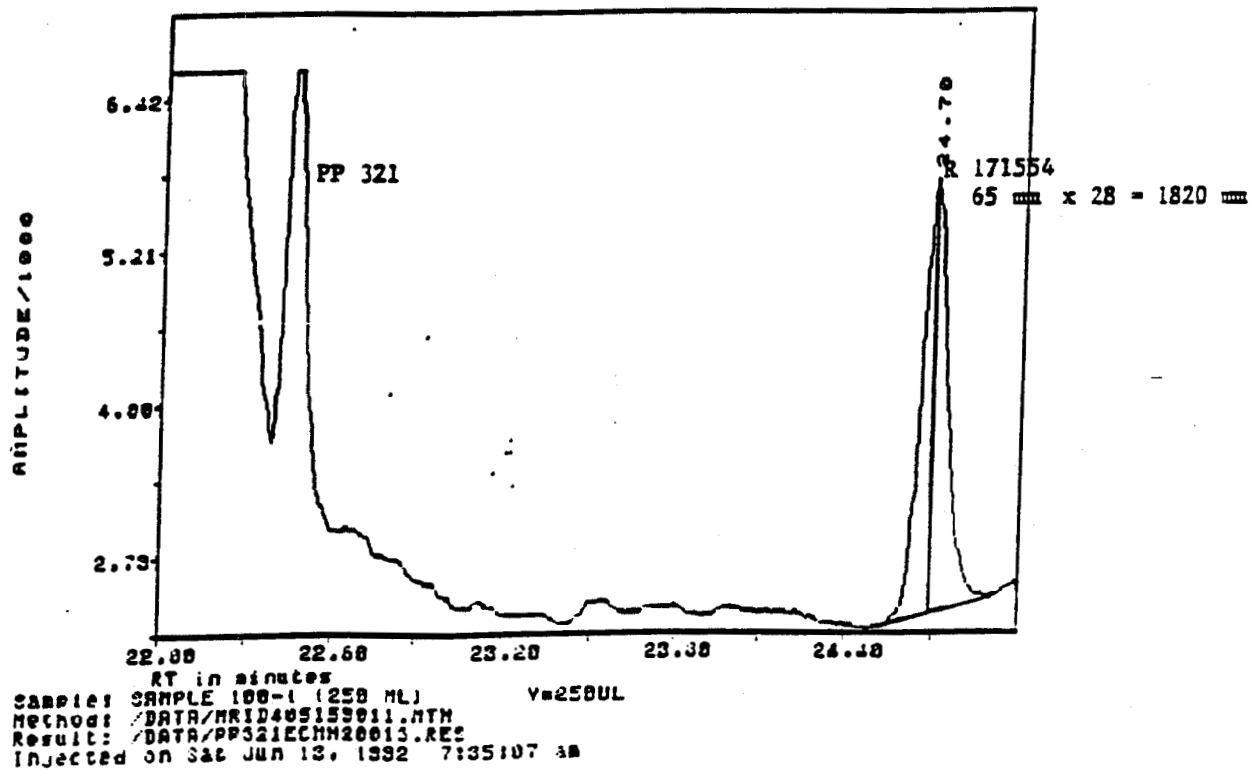


Sample: SAMPLE 10-3 PPT (250 ML) Y = 250UL
Method: /DATA/MRID40515901.MTH
Result: /DATA/PP321ECHH20005.RES
Injected on Sat Jun 13, 1992 21:09:03 AM

LC-100-1-250: Pond Water Fortified @ 100.0 ppt 250 ml

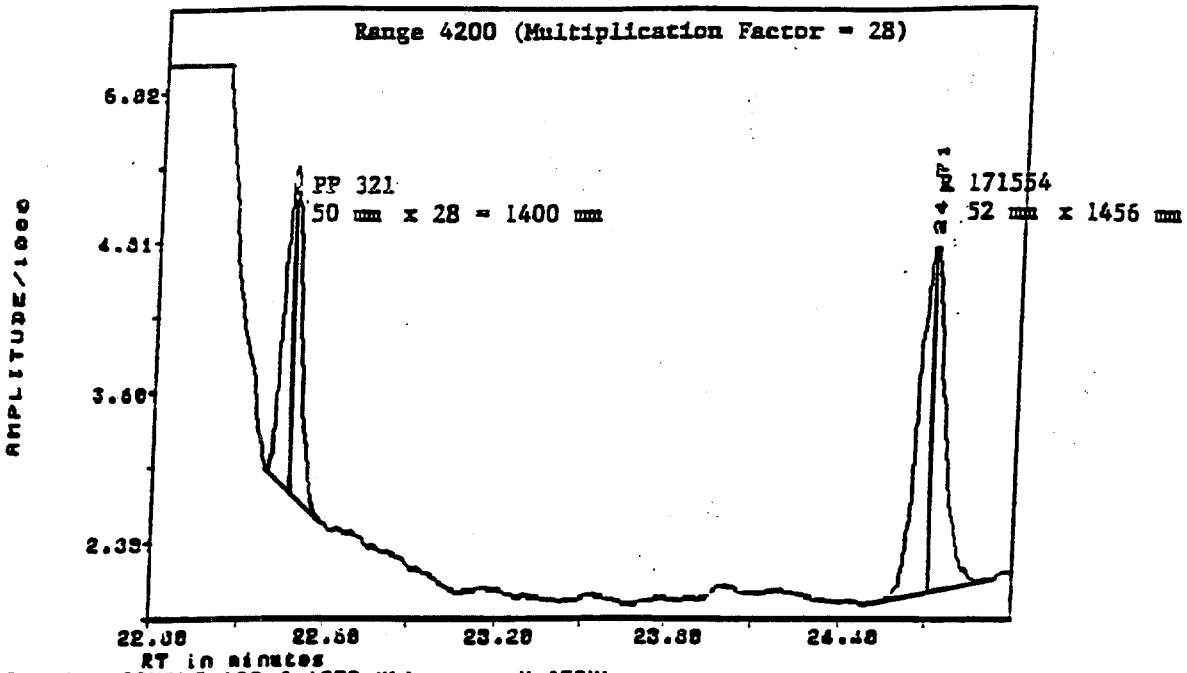


Sample: SAMPLE 100-1 (250 ML) V=250UL
Method: /DATA/MRID405159011.MTH
Result: /DATA/PP321ECHM20013.RES
Injected on Sat Jun 13, 1992 7:35:07 AM



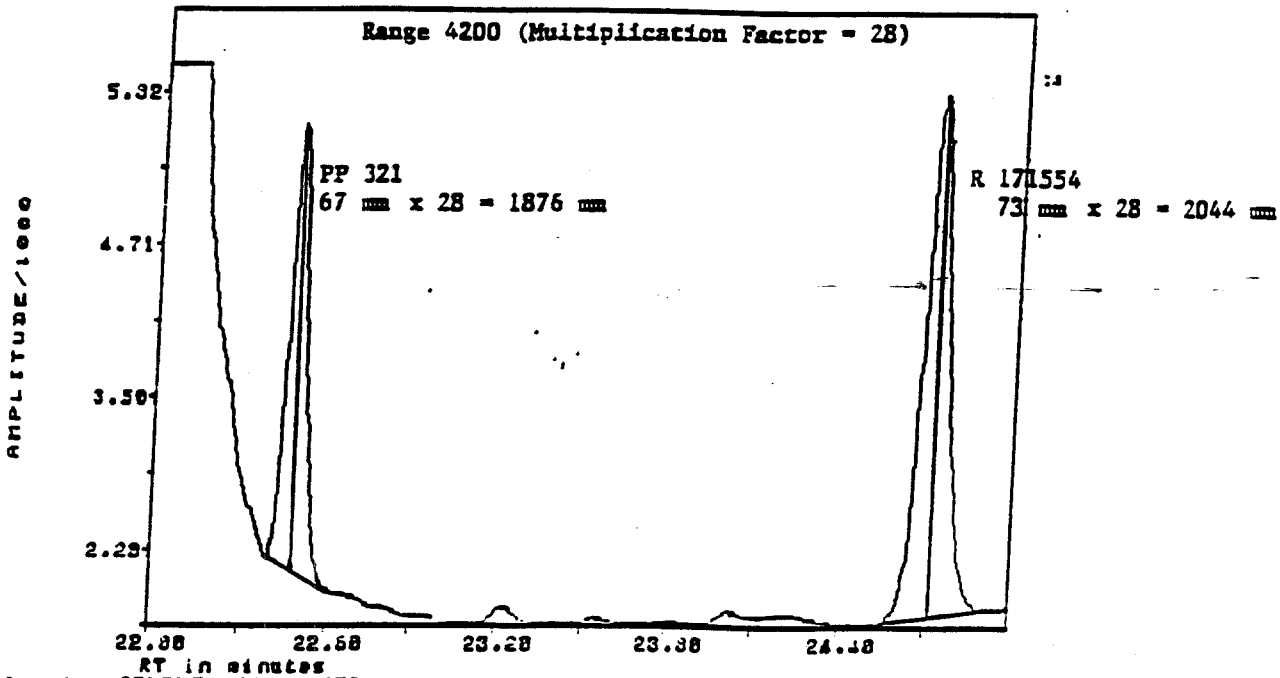
Sample: SAMPLE 100-1 (250 ML) V=250UL
Method: /DATA/MRID405159011.MTH
Result: /DATA/PP321ECHM20013.RES
Injected on Sat Jun 13, 1992 7:35:07 AM

LC-100-2-250: Pond Water Fortified @ 100.0 ppt 250 ml



Sample: SAMPLE 100-2 (250 ML) Vm258UL
Method: /DATA/MRID405159011.MTH
Result: /DATA/PP321ECHH20814.RES
Injected on Sat Jun 13, 1992 8:56:38 am

LC-100-3-250: Pond Water Fortified @ 100.0 ppt 250 ml



Sample: SAMPLE 100-3 (250 ML) Vm258UL
Method: /DATA/MRID405159011.MTH
Result: /DATA/PP321ECHH20815.RES
P.Data: /DATA/PP321ECHH20815.RES
Injected on Sat Jun 13, 1992 10:13:07 am

Example of Calculations:

1. Gas Chromatographic Intensity:

   The peak intensity is expressed in terms of peak height. The peak height, in millimeters, is measured manually from the apex of the peak to the baseline drawn across the bottom of the peak.

   Injection volume for all standards and samples was 2 µl; therefore, no injection volume adjustment is necessary in the calculations.

   The samples, which were extracted using 250 ml of pond water, were concentrated to 250 µl before gas chromatographic analysis. Therefore, no sample volume adjustment is necessary in the calculations.

   There is a background correction for the samples as there was a matrix effect noted in the matrix controls. The corrected peak heights for lambda-Cyhalothrin (PP321) and the corrected peak heights for R171554 were obtained by the difference between the sample peak height and the matrix control.

2. Calculation Formula:

   Relative Factor Ratio =

$$\frac{\text{Calibration Standard Peak Height PP321 (mm)}}{\text{Calibration Standard Concentration PP321 (ppt)}} \Big/ \frac{\text{Calibration Standard Peak Height R171554 (mm)}}{\text{Calibration Standard Concentration R171554 (ppt)}}$$

   Concentration Found of PP321 (ppt) =

$$\frac{\text{Fortified Sample Corrected Peak Height PP321 (mm)}}{\text{RF Ratio}} \Big/ \frac{\text{Fortified Sample Concentration R171554 (ppt)}}{\text{Fortified Sample Corrected Peak Height R171554 (mm)}}$$

Found (% Recovery) =

$$\frac{\text{Concentration Found (ppt)}}{\text{Concentration Fortified (ppt)}} \times 100\ \%$$

4. Calculations for the Determination of the Concentration and Recovery of lambda-Cyhalothrin (PP321) in Fortified Pond Water Sample LC-10-1-250

Calibration Standard (10 ppt equivalent)

Concentration	Peak Height PP321	Peak Height R171554	RF
10 ppt	266 mm	210 mm	1.27

LC-0-1-250
Pond Water Matrix Control

Peak Height PP321	Peak Height R171554
84 mm	140 mm

LC-10-1-250
Pond Water, Fortified @ 10 ppt

Peak Height PP321	Peak Height R171554
336 mm	385 mm

Corrected LC-10-1-250

Difference (Peak Height) PP321	Difference (Peak Height) R171554
(336 - 84 mm = 252 mm)	(385 - 140 mm = 245 mm)

Concentration Found of PP321 (ppt) in LC-10-1-250 =

$$\frac{(252\text{ mm PP321})}{1.27} \times \frac{(10\text{ ppt R171554})}{(245\text{ mm R171554})} = 8.1\text{ ppt}$$

$$\text{Found (\% Recovery)} = \frac{8.1\text{ ppt PP321 Found}}{10.0\text{ ppt PP321 Fortified}} \times 100\%$$

$$= 81\ \%$$

Appendix 1

ICI Agrochemicals Residue Analytical Method Number 125b

The Determination of Residues of PP321 in Water Following Sampling by a Solid-Phase Extraction Technique

Agrochemicals

ICI Agrochemicals
Jealott's Hill Research Station
Bracknell
Berkshire RG12 6EY
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Your ref | Our ref | Direct line | Date

ICI AGROCHEMICALS RESIDUE ANALYTICAL METHOD NUMBER 125b

THE DETERMINATION OF RESIDUES OF PP321 IN WATER

FOLLOWING SAMPLING BY A SOLID-PHASE EXTRACTION TECHNIQUE

- A gas-liquid chromatographic method using an internal standard

Author : J Sadler, E Bolygo

Study Scientist : S T Hadfield

Date of Issue : September '89

CONTENTS

CONTENTS PAGE NO

CHEMICAL PROPERTIES

CYHALOTHRIN (PP563)

Chemical Name : (RS)-α-cyano-3-phenoxybenzyl-(Z)-(1RS, 3RS)(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate.
PP563 consists of two enantiomer pairs designated A and B.

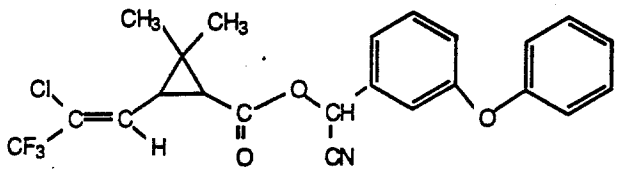
LAMBDA-CYHALOTHRIN (PP321)

Chemical Name : α-cyano-3-phenoxybenzyl-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate
a 1:1 mixture of the (Z)-(1R,3R), S-ester and (Z)-(1S,3S),R-ester
PP321 consists of one enantiomer pair designated B.

Emperical Formula : C_{23} H_{19} ClF_3 NO_3

Molecular Weight : 449.9

Structural Formula :



1. SCOPE

The analytical procedures described are suitable for the determination of residues of the synthetic pyrethroid insecticides cyhalothrin - PP563, and lambda-cyhalothrin - PP321 in natural waters including pond water.

In water at pH values greater than 7, PP321 (enantiomer pair B) rapidly epimerises to enantiomer pair A. Therefore the determination of both enantiomer pairs is necessary.

The internal standard used, R171554 has also been found to epimerise during the analytical procedure. This method is suitable for the determination of both epimers found.

Since pyrethroids are of low water solubility and will readily adsorb onto glassware, sampling and subsequent storage in water bottles is not recommended.

The recommended sampling procedure is to sample at source as shown in Fig 2.

The limit of determination for the method has been set at 1 ng/litre water (for each of the enantiomer pairs A and B).

2. SUMMARY

The water sample is loaded onto a pre-conditioned solid phase extraction cartridge, (cartridge as shown in Figure 1). The cartridges are frozen immediately after sampling and should be maintained frozen during transportation and storage, prior to analysis.

After thawing, the cartridges are accurately fortified with a known amount of internal standard. Cartridges are then eluted with organic solvent, and co-extractives removed and the analyte concentrated by further solid-phase sorbent extraction.

Final quantitative determination is by on-column capillary gas-liquid chromatography using internal standardisation.

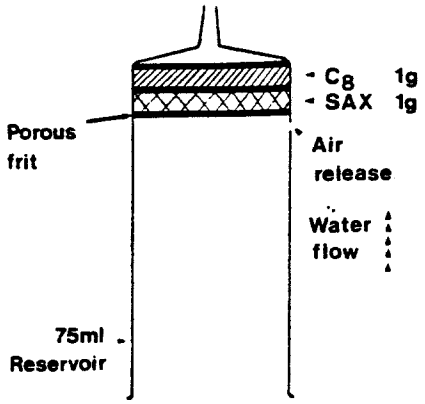
A flow chart summary of the method is shown is Appendix 5.

3. SAMPLING PROCEDURE

3.1 Extraction Cartridge Preparation

Prepare extraction cartridges as shown in Figure 1 overleaf:-

Figure 1



a) Place one 20 μm porous polyethylene frit in the base of a 75 cm^3 polypropylene reservoir.

b) Add C_8 Sepralyte (reverse-phase) sorbent (1 g).

c) Place another frit on top of the C_8 sorbent and add SAX (strong anion exchange) sorbent (1 g).

d) Add a further frit to the top of the sorbent bed.

3.2 **Extraction Cartridge Conditioning**

a) Before use all cartridges must be conditioned with methanol followed by deionised water.

b) All cartridges must be used as soon as possible after conditioning (within one hour).

c) Cartridges can be conditioned by placing in a rack supported over a collection vessel and allowing the solvent to pass through under gravity.

d) Apply methanol (25 cm^3) to each cartridge and allow to drain under gravity.

e) When the methanol has drained from each cartridge (i.e. has stopped dripping) apply deionised water (5 cm^3) and allow to drain under gravity.

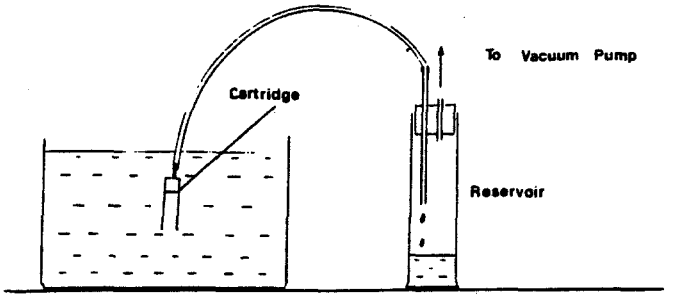
f) A hole must be drilled in the side of each cartridge (1 cm from the sorbent bed) in order to release air when the cartridge is lowered into the water. This prevents desolvation of the cartridge prior to the water reaching the sorbent bed, when pumping commences.

g) The cartridges should then be transferred into individual plastic bags until required for sampling.

3.3 **Water Sampling**

Water can be sampled through the cartridge using the apparatus shown below:

Figure 2



b) Water can be pumped through the cartridge using a hand held vacuum pump (approximately 25 cm^3/stroke). A flow of 50 cm^3/min should be achieved.

c) The total volume of water sampled through the cartridge can then be measured on the cylinder and recorded.

Desolvation of the sorbent bed reduces the retention efficiency of the analyte on the cartridge and therefore there is a maximum volume of water that can be sampled. This maximum is 350 cm^3 water.
Since for a limit of 1 ppt (1 ng/litre), one litre of water is required, then three cartridges are taken per sample. For higher limits of determination fewer cartridges can be used.

d) Immediately following sampling the cartridge should be removed carefully from the tubing and placed in its original plastic bag. The bags should be sealed, labelled and placed in a freezer as soon as possible after sampling.

3.4 **Prevention of Contamination**

In order to avoid contamination of samples during the course of sampling the following procedures are necessary.

a) Cartridges should be conditioned and drilled in a contamination free zone (ie. an area well away from the sampling area).

b) All equipment used for drilling or conditioning should also be stored away from the sampling area.

c) Immediately after sampling, the cartridges should be replaced in their individual plastic bags, labelled appropriately, then sealed and stored in a temperature-monitored freezer at <-18°C until required for analysis.

d) Handling of the cartridges should be minimised and contact with the insides of the cartridges avoided at all times.

4. ANALYTICAL PROCEDURE

4.1 Sample Preparation

a) After removal from the freezer and thawing, sample matrix cartridges should be taken to dryness under vacuum on a vacuum manifold system (eg. Supelco) for 30 minutes.

b) Sample matrix cartridges should be fortified with the internal standard (R171554 see Appendix 3.2) at an appropriate level.

4.2 Extraction

a) Extract matrix cartridges with acetonitrile (3 x 5 cm^3) on a manifold system, aiding percolation of solvent through matrix with a vacuum if necessary. Collect all the eluate.

b) Apply a vacuum to cartridges (approximately 10 minutes) to remove any remaining eluate.

c) Add buffer (Clark and Lubs pH 1, see Appendix 3.2) (2 cm^3) to cartridge, allow to percolate into cartridge then take to dryness under vacuum. Discard all eluate from this step.

d) Continue extraction with diethyl ether: hexane 70:30 (5 cm^3) followed by acetonitrile (5 cm^3). Remove any excess eluate by applying a vacuum and then combine extracts from steps a, b, and d.

e) Repeat steps a-d for any further cartridges remaining for each sample. (see 3.3c)

f) Combine all organic eluates in a round bottom flask and concentrate by rotary evaporation until between 5-10 cm^3 remain.

g) Ultrasonicate round bottom flask, measure volume remaining and make to 15 cm^3 with acetonitrile.

h) Add ultra-pure water (50 cm^3) to each flask and shake thoroughly (this should only be done immediately before application to the C_8 sorbent cartridge in 4.3).

4.3 Sorbent Clean-Up

a) Prepare matrix cartridges (1g of C_8 Sepralyte sorbent) in a reservoir (75 cm^3). Place on a vacuum manifold system with individual valve control eg. Supelco.

b) Condition cartridges with methanol (25 cm^3) followed by water (5 cm^3) allowing each to percolate under gravity.

(N.B. Cartridges must be conditioned within half an hour of use and must not be allowed to dry out before use).

c) Apply analyte solution 4.2 (h) to C_8 cartridge and allow to percolate under gravity, controlling the rate, (no greater than 1 cm^3/min). Discard the eluate.

d) Wash cartridge with water (5 cm^3) followed by acetonitrile:water 50:50 (2 x 2.5 cm^3). Discard the eluate.

e) Take cartridges to dryness under vacuum (approximately 1 hour).

f) Elute cartridges with hexane (4 x 2.5 cm^3) and collect the eluate.

g) Make the hexane eluate to an appropriate volume for analysis by GLC.

5. GAS-LIQUID CHROMATOGRAPHY

The conditions for the analysis by GLC will depend upon the equipment available. A highly sensitive detector is required for analysis at low levels, and the following conditions have been found to be satisfactory.

5.1 Instrumentation

Varian 3500 capillary gas chromatograph with automated on-column injector and fitted with a Ni^{63} (8 mCi) electron capture detector.

5.2 GLC Conditions

a) SE54 bonded phase fused silica open tubular capillary column 25m x 0.32mm ID.

b) Temperature programs:-
Column temperature:- 89°C (hold 1 minute) program at 10°C/min to 240°C (hold 21 minutes) program at 10°C/min to 250°C (hold 10 minutes).

Injector temperature:- 40°C program at 150°C/min to 250° (hold 35 minutes).

c) Detector temperature 300°C.

d) Carrier gas, helium at 2 cm^3/min. Make up gas nitrogen at 28 cm^3/min.

Under these conditions PP563 is resolved into its two enantiomer pairs, A with retention time 24.1 mins and B (PP321) with retention time 24.85. The internal standard used -R171554 (see Appendix 3.2) has a retention time of 27.1 minutes. Sensitivity is such that 2.4 pg enantiomer pair B (PP321) injected on column with electrometer attenuation at 10 x 8 and recorder on 1mV gives approximately 40% full scale deflection.

6. CALCULATION OF RESIDUES

Residues of enantiomer pairs A and B in the final extract are calculated using the internal standardisation procedure.

Prior to extraction each sample is accurately fortified with a known concentration of internal standard. Each sample residue can thus be individually corrected by measuring the percentage recovery of internal standard through the analytical method.

The calculation for the determination of cyhalothrin (PP563) residues may be performed using a 'single point ratio calibration' (Cardone and Palermo, 1980). It should be noted that such calibrations are feasible only when the chosen internal standard meets certain criteria (see Ref 1). The calculations below are shown for PP321 (enantiomer pair B) but are equally valid for enantiomer pair A.

a) Using the GLC operated under conditions described in 5.2 above, make repeated injections of 2 µl of a standard solution containing a mixture of R171554 and PP563 each at 2.5 ng/cm^3 until a consistent response is obtained.

b) Measure the peak heights or area obtained and calculate the detector 'Response factor ratio'.

$$\text{i.e. Rf ratio (Std)} = \frac{\text{Peak ht PP321 (mm)}}{\text{Conc'n PP321 (ng/cm}^3\text{)}} \div \frac{\text{Peak ht internal standard (mm)}}{\text{Conc'n internal standard (ng/cm}^3\text{)}}$$

c) Make an injection of the sample solution (SA) and measure the peak heights or area obtained for PP321 and internal standard (R171554).

d) Since the response factor ratio will be constant for a detector with a linear response, the PP321 residue in the sample can thus be calculated:-

$$\text{Rf ratio (Std)} = \frac{\text{Peak ht PP321}_{SA}}{\text{Conc'n PP321}_{SA}} \div \frac{\text{Peak ht R171554}_{SA}}{\text{Conc' R171554}_{SA}}$$

$$\text{Therefore PP321 conc'n} = \frac{\text{Peak ht PP321}_{SA} \times \text{Conc'n R171554}_{SA}}{\text{Rf} \times \text{Peak ht R171554}}$$

$$\text{Units (ng/cm}^3\text{)} = \frac{\text{mm} \times \text{ng/cm}^3}{\text{mm}}$$

e) This concentration must then be corrected in order to express the residue in terms of the concentration of analyte in a known amount of matrix.

$$\text{Residue in ng/l} = \frac{\text{PP321 conc'n}}{\text{(Sample/matrix) conc'n in final solution}}$$

$$\text{Units (ng/l)} = \frac{\text{ng/cm}^3}{\text{l/cm}^3}$$

Note:- Injections of sample solutions (3 maximum) should be bracketed between standards.

7. **LIMIT OF DETERMINATION**

A true assessment of the limit of determination of the method may be determined by fortification of untreated samples at low levels and subjecting them to the complete analytical procedure. The chromatographic response obtained for these recoveries at the retention time of PP321 (enantiomer pair B) or enantiomer pair A should exceed the background signal noise by a factor of at least four to be considered an acceptable quantitative limit of determination. In addition the precision of measurement at this level should not exceed a coefficient of variation of ± 15%.

In these laboratories the LOD for each of enantiomer pairs A and B has been set at 1 ng/l.

8. **CONTROL EXPERIMENTS**

a) To ensure that no observed contamination of the samples occurs during analysis at least one untreated sample should be analysed alongside a set of samples using exactly the same procedure.

b) In these laboratories in order to ensure that any interference peaks observed at the retention time of PP321 or PP563 are not due to solvent/reagents a reagent blank sample is analysed alongside any set of samples ie. complete procedure carried out in absence of sample matrix.

9. **RECOVERY EXPERIMENTS**

a) Before extraction each sample must be accurately fortified with a concentration of internal standard within the range of the residue levels expected.

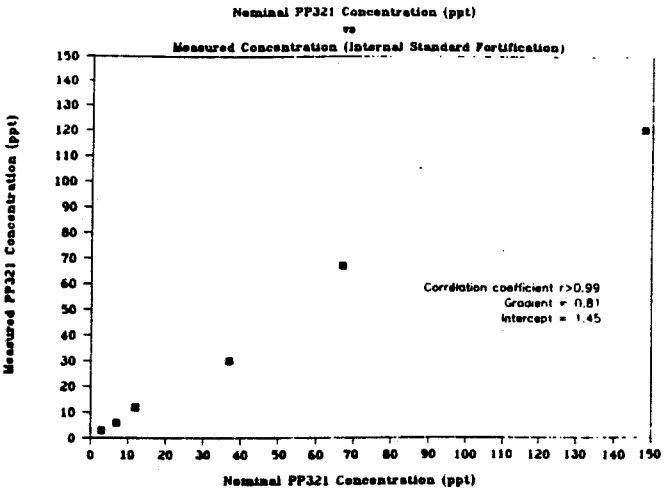
b) At least one untreated sample should be accurately fortified with suitable concentrations of PP321 and R171554 within the range of the expected residue level in order to check the correlation between the analyte and internal standard recoveries during analysis.

10. **METHOD VALIDATION**

10.1 **Internal Standardisation Procedure**

The validity of the internal standardisation procedure has been demonstrated by plotting a calibration graph for the residues calculated by the internal standardisation procedure, against concentration of PP321 in accurately fortified control samples with constant internal standard concentration.

The resultant plot is shown overleaf.



10.2 **Recovery Experiments**

For authentic water samples (115) fortified with internal standard R171554 at 10 ng/l (10 ppt) and analysed using this analytical procedure, the mean internal standard recovery was found to be 70% with a standard deviation of ± 15.

10.3 **Estimation of the Precision of the Method**

For twenty six untreated samples accurately fortified with PP321 and internal standard R171554 at 10 ng/l (10 ppt) and then taken through the analytical method, the mean recovery by internal standardisation was calculated to be 10 ng/l with a standard deviation of <2.

10.4 **Extractability**

Extractibility of ^{14}C-PP321 from authentic sample extraction cartridges sampled at source was found to be greater than 88% in all cases.

10.5 **Linearity**

For accurate quantitiation of residue concentrations, all analyses should be carried out within the linear range of the detector response.

In these laboratories the detector responses for PP321 and R171554 were shown to be linear over the concentration range 1.25 ng/ml - 100 ng/ml (Appendix 4).

11. **CONFIRMATION OF RESIDUES**

Capillary gas chromatography-mass spectrometry (GCMS) operated in the selected ion monitoring mode (SIM) may be used for the qualitative and quantitative confirmation of residues. Samples obtained from the residue analytical method are examined by SIM; ie. 2 or more of the most abundant m/z values present in the mass spectrum are continuously monitored throughout the gas chromatographic run and collected on a data capture system.

Qualitative confirmation of residues is given by the appearance of a peak at the correct gas chromatographic retention time for all the specific m/z values monitored.

In these laboratories GCMS was used to qualitatively and semi-quantitatively confirm the presence of residues of PP563 and also the epimer of the internal standard R171554, formed by inversion of the optically active α-CN position.

11.1 **GC-MS Operating Conditions**

a) Chromatography

Instrument : Finnigan 9610 Gas Chromatograph

Column : RSL 150, 25 m column, ID 0.25 mm

Conditions : 45°C (hold 2 minutes); program at 15°C minute^{-1} to 220°C (hold 17.5 minutes); program at 5°C minute^{-1} to 260°C (hold 8 minutes)

Carrier Gas : Helium, at 10 psi back pressure

The mass chromatogram of PP563 is shown in Appendix 2.

b) Mass Spectrometer

Instrument : Finnigan 4600 quadrapole mass spectrometer

Mode : Negative ion methane electron capture

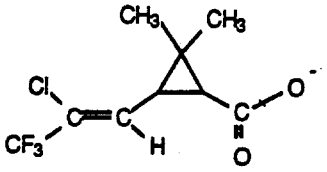
Conditions :

Source Pressure : 2.1 x 10^{-5} TORR
Electron energy : 100 eV
Filament current : 0.3 AMPS
EM voltage : 1300 VOLTS
Ioniser pressure : 0.22 TORR
Ioniser temp : 120°C
Sensitivity : 10^{-7} AMPS/VOLT

C) Selected Ion Monitoring

Compound PP563

m/z 241 structure of ion:

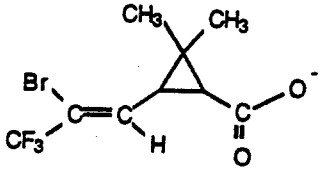


m/z 205 structure of ion:

241-HCl

Compound R171554

m/z 285 structure of ion



m/z 205 structure of ion

285 - HBr

m/z 79/81 structure of ion

Br^-

The mass spectrum of PP563 is shown in Appendix 2.

12. EXAMPLES OF CHROMATOGRAPHIC TRACES

These are shown in Appendix 1.

13. **REFERENCES**

1. Cardone M J, Palermo P J and Sybrandt L B : Potential error in single point ratio calculations based on linear calibration curves with a significiant intercept, Anal. Chem., 52, pp 1187-1191, 1980.

FIGURE 1 : 2.5 ng/cm^3 PP563 and Internal Standard

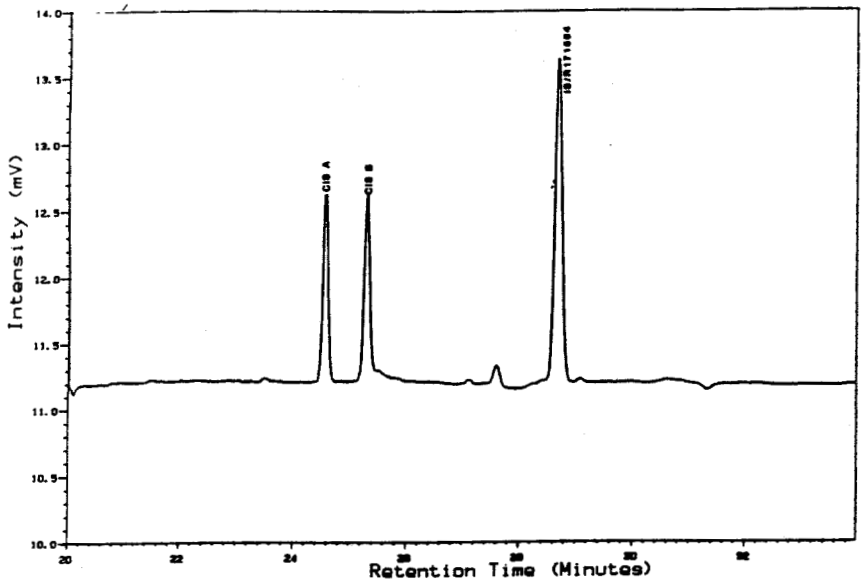
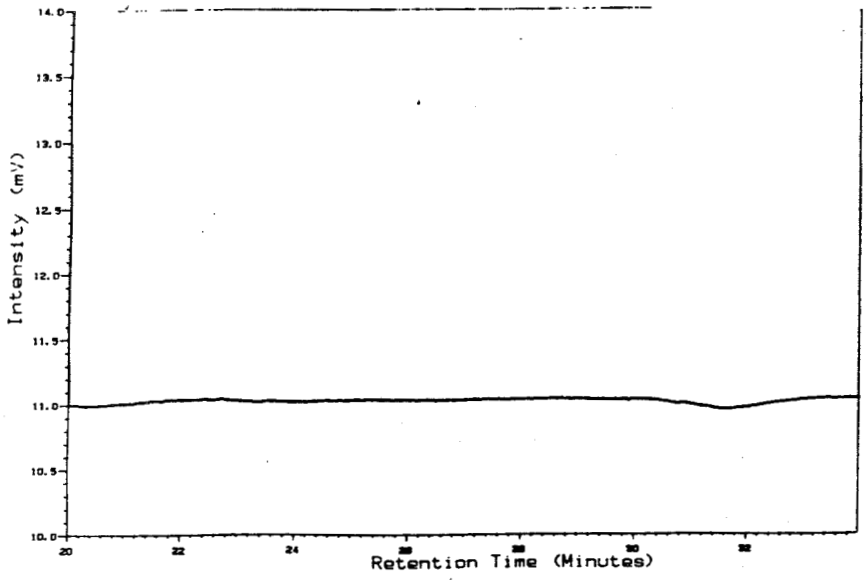


FIGURE 2 : Untreated Sample at 0.25 l/cm^3



APPENDIX 1

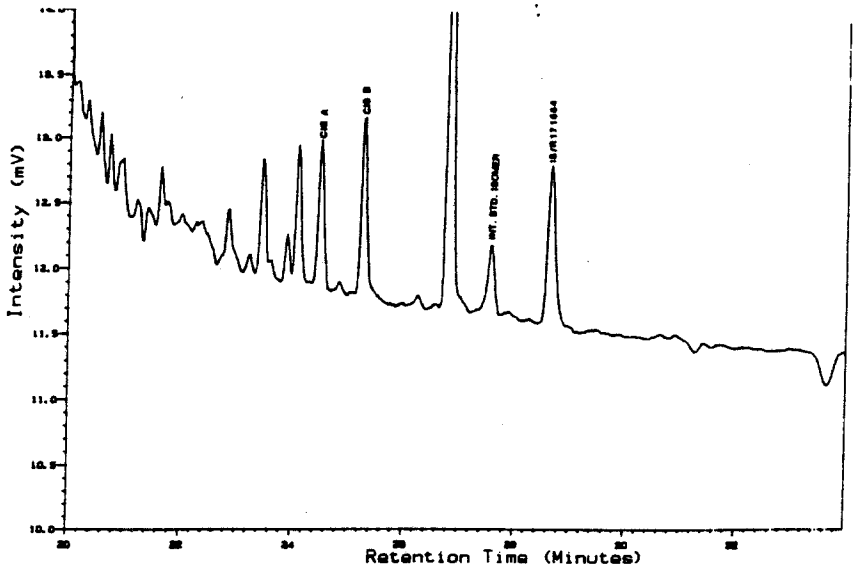
Typical Gas Chromatograms for PP563 Residue Determination in Pond Water

Figure 1 : 2.5 ng/cm^3 cyhalothrin and internal standard

Figure 2 : Untreated sample at 0.25 l/cm^3

Figure 3 : Water residue sample at 0.25 l/cm^3

FIGURE 3 : Water Residue Sample at 0.25/l cm^3
Residue : cis A = 6 ng/l
cis B = 7 ng/l



APPENDIX 2

Figure 4 : Mass chromatogram of PP563

Figure 5 : Mass spectrum of PP563

Figure 4 : Mass chromatogram of PP563 and R171554.

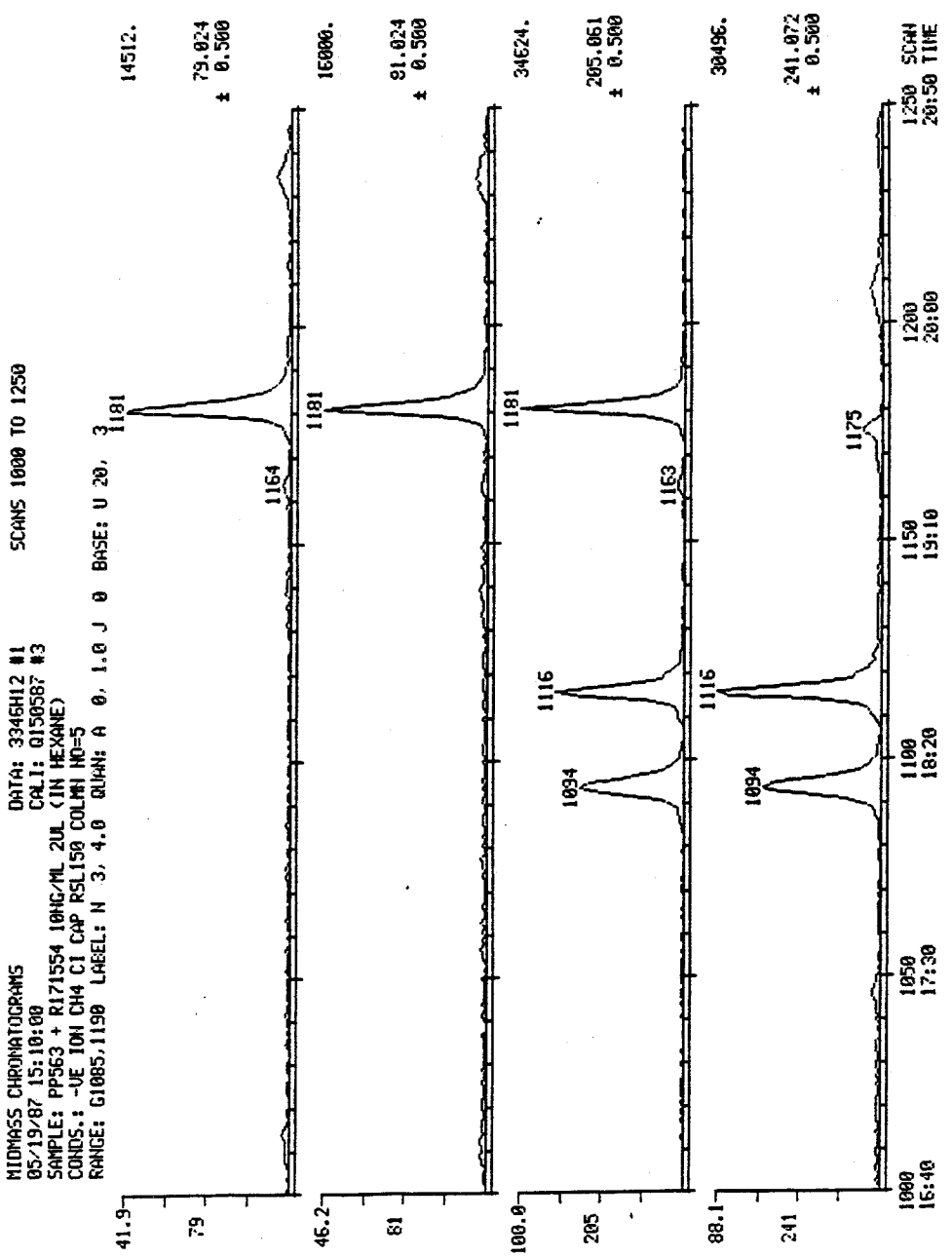


Figure 5 : Mass spectrum of PP563

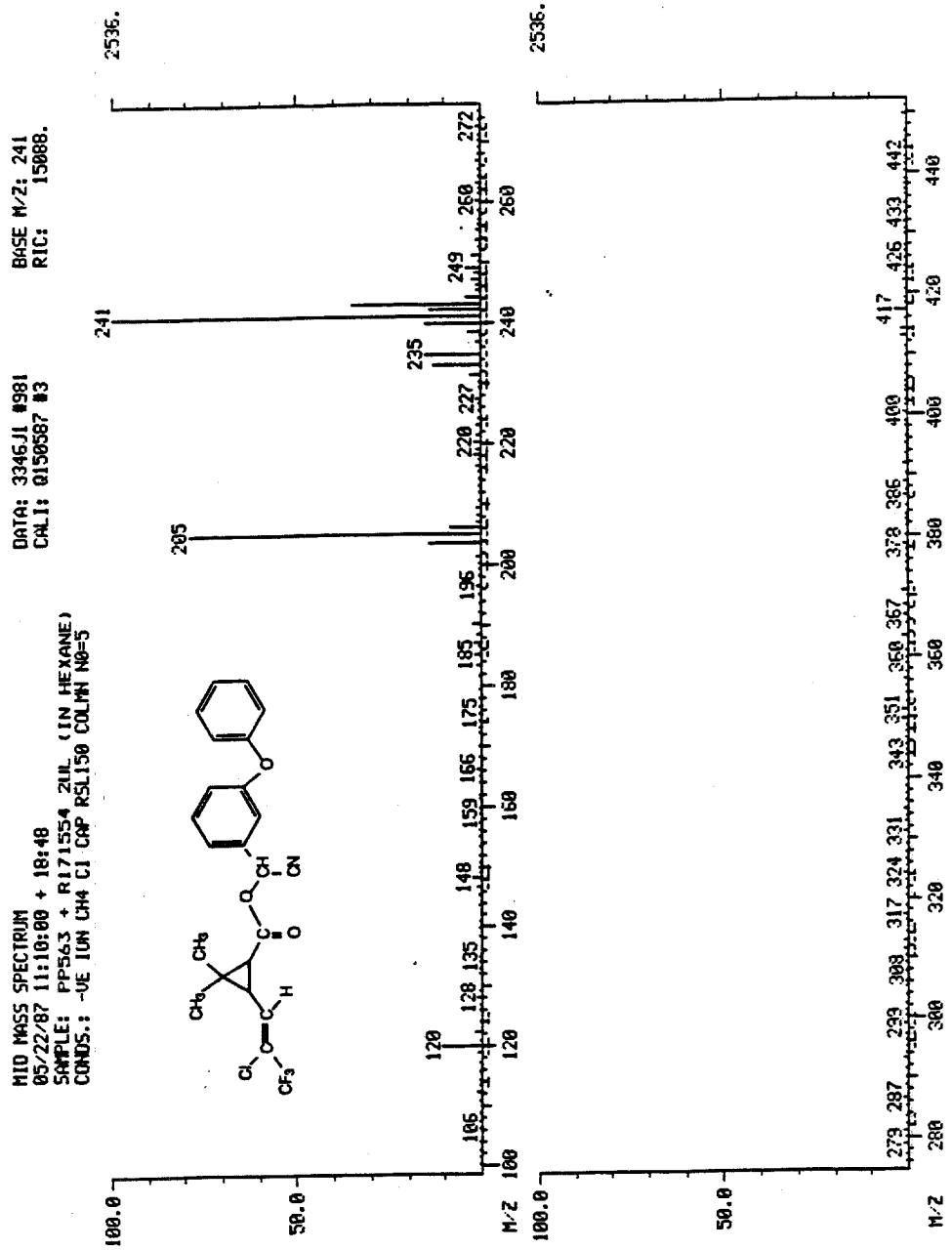
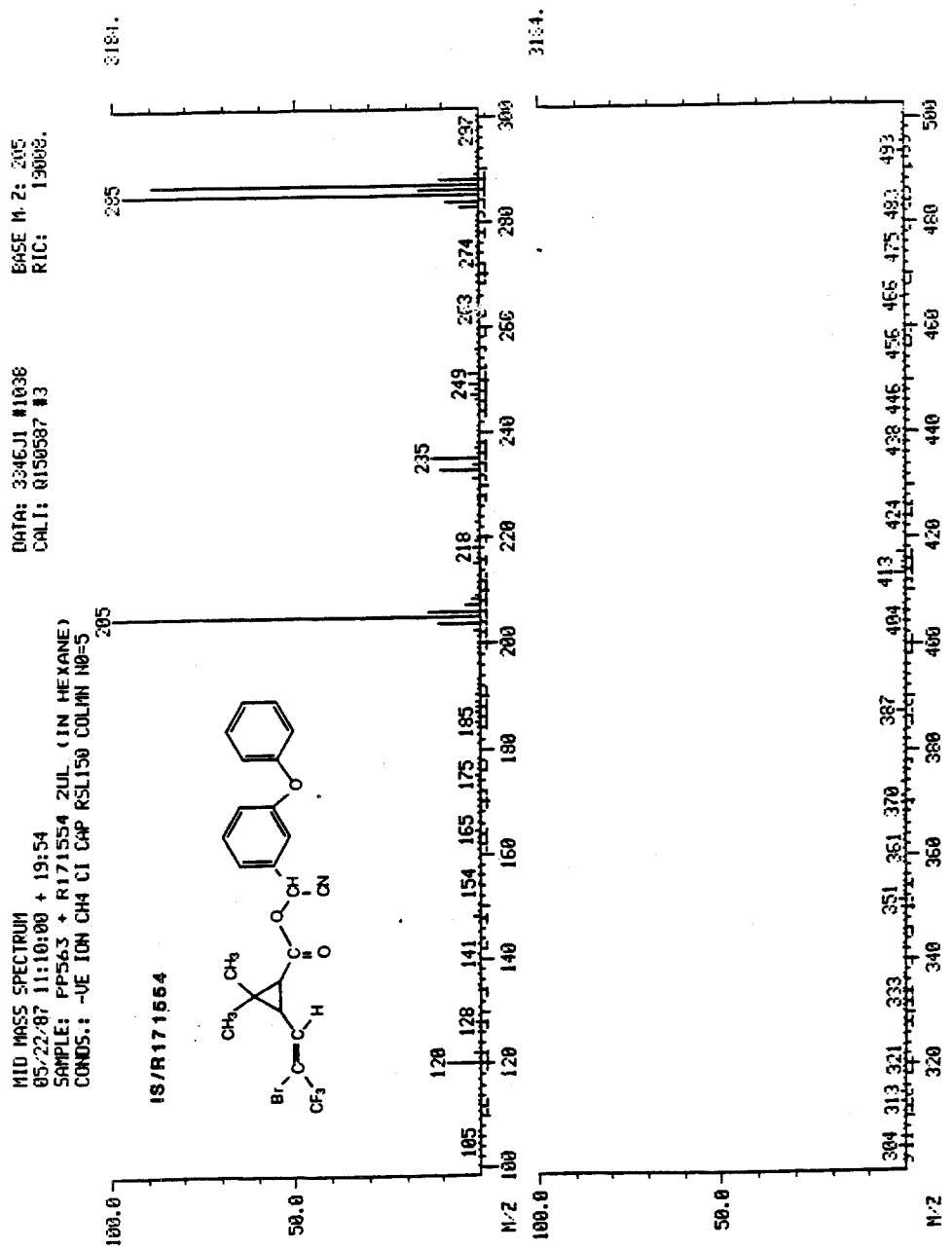


Figure 5 : Mass spectrum of R171554



APPENDIX 3

1. APPARATUS

a) Vacuum manifold system (Supelco).

b) Collection vessels (eg. measuring cylinders).

c) Graduated tubes of 10cm^3 calibrated down to 1.0cm^3 in 0.1cm^3 units.

d) Capillary gas chromatograph fitted with an electron capture detector which will meet the sensitivity requirements.

N.B. An autosampler may be used with the gas chromatograph providing:-

1) That it is ensured it is not a source of cross-contamination to samples.

2) Suitably precise injections can be achieved with a reproducibility better than ± 5%.

e) Reservoirs (polypropylene) 75 cm^3 capacity, porous polyethylene frits (20 μm). (Supplier:Jones Chromatography).

2. REAGENTS

a) Solvents: Re-distilled methanol, acetonitrile, diethyl ether, n-hexane, and ultrapure water.

b) C_8 octyl sepralyte sorbent (Supplier:- Jones Chromatography).

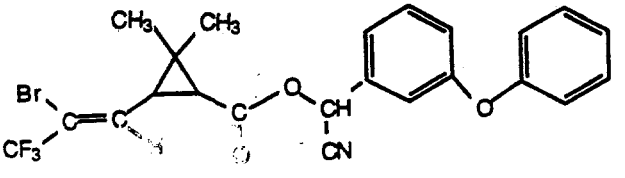
c) Clark and Lubs buffer solution (pH1). Mix 0.2 M KCl (25 cm^3) + 0.2 M HCl (67 cm^3) make to 100 cm^3 with water.

d) A sample of PP321 or cyhalothrin of known purity.

e) A sample of R171554 for use as an internal standard.

The internal standard used is the resolved cis isomer assumed by analogy to the chromatographic GLC characteristics of PP563 and other synthetic pyrethroids to be:

(RS)-α-cyano-3-phenoxybenzyl-(1RS)-cis-3-(Z-2-bromo-3, 3, 3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate.



3. PREVENTION OF CONTAMINATION

Great care must be taken when working at low levels to minimise the risk of contamination.

a) To achieve the set limits of determination, whenever possible all analysis should be carried out in a designated 'low level' area.

b) All glassware must be segregated and used solely for the low level analysis. If possible a washing machine should be designated for such glassware.

c) All glassware should be thoroughly soaked and rinsed with appropriate solvents before use.

d) Plastic and glassware for the analysis should be minimised.

e) All new 'batches' of solvent and sorbent should be analysed (for interfering contaminants in ECD analysis) before use.

f) Solvents should be freshly dispensed into glass vessels for each analysis and any solution which is required, should be freshly prepared.

g) Staff must not come into contact with high residues or work in areas exposed to these.

4. 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 doubt, consult the appropriate safety manual (eg, ICI Laboratory Safety Manual) containing recommendations and procedures for handling chemicals, and monograph such as 'Hazards in the Chemical Laboratory', Ed G D Muir, The Chemical Society, London.

a) Solvent Hazards

	Acetone	Acetonitrile	Diethyl ether	Hexane	Methanol
Harmful vapour	Yes	Yes	Yes	Yes	Yes
Highly flammable	Yes	Yes	Yes	Yes	Yes
Harmful by skin absorption	-	Yes	-	-	Yes
TLV mg m^{-3}	2400	70	1200	180	260

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

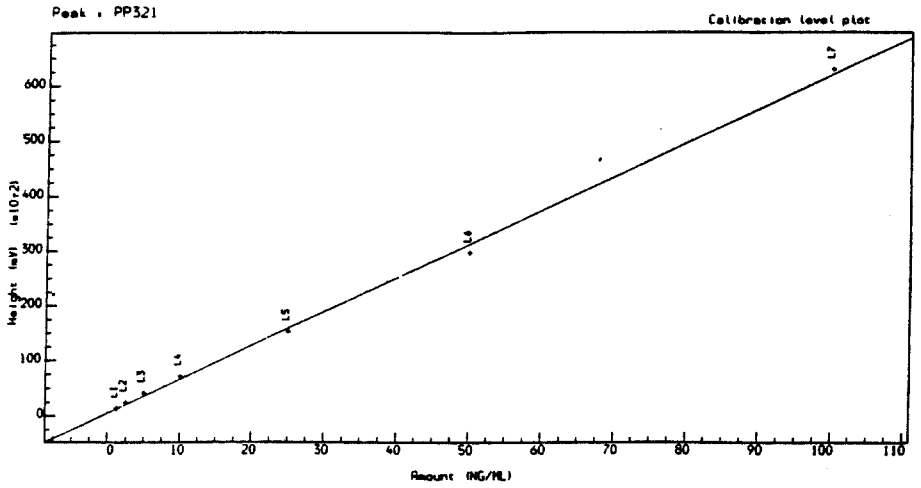
b) PP321, PP563 and R171554 are synthetic pyrethroid insecticides with a mammalian toxicity (acute oral LD_{50}) in the rat in the order of 50-60 mg kg^{-1} (PP321).

5. **PREPARATION OF ANALYTICAL STANDARDS**

Weigh out accurately, using a 'five-figure' balance, sufficient PP321, PP563 or R171554 to allow dilution in acetone to give a 1000 $\mu g/cm^3$ stock solution in a volumetric flask. Make serial dilutions of this stock to give 100 $\mu g/cm^3$, 10 $\mu g/cm^3$ and 1.0 $\mu g/cm^3$ standard solutions in hexane. Prepare serial dilutions of mixed standards of PP321 and internal standard in hexane (for use as GLC reference standards) down to 2.5 ng/cm^3.

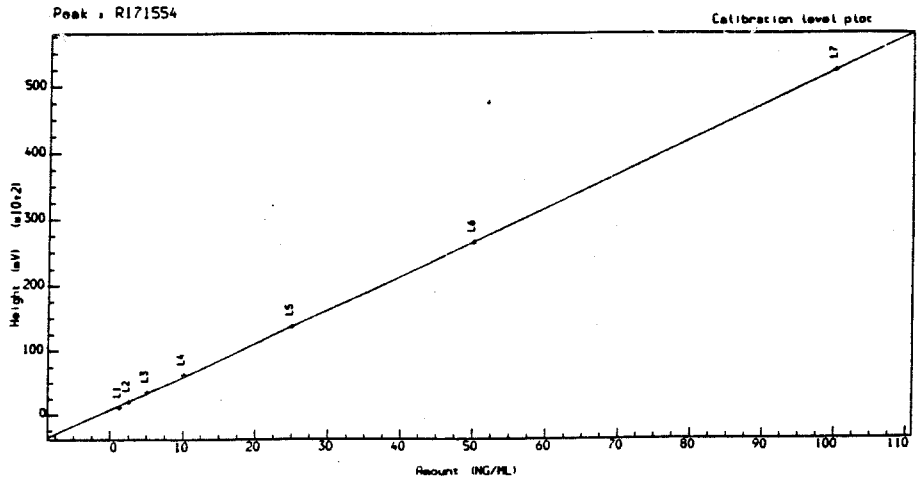
When not in use, always store the standard solutions in a refrigerator at <4°C to prevent decomposition/evaporation/ concentration of the standard strength. Analytical standards should be replaced with freshly prepared standards after 3 months of use.

APPENDIX 4 - Linearity Graphs



Constant : 5.00576E+2
1er degree : 6.15341E+2

Curve fit : Linear
Correlation coefficient : 0.99926



Constant : 8.38244E+2
1er degree : 5.16477E+2

Curve fit : Linear
Correlation coefficient : 0.99994

APPENDIX 5 - Flow Chart Summary of Method

C_8+SAX Cartridge Conditioning

i) Methanol (25 cm^3)
ii) Water (5 cm^3)

C_8+SAX Cartridge Sampling

Load at 50 cm^3 min (up to 350 cm^3 water).

Sample Storage

Freeze cartridges at <-18°C until required for analysis

Sample Analysis

Elution : Acetonitrile (3 x 5 cm^3) - Collect
Remove excess with vacuum - Collect
Clark + Lubs buffer (1 x 2 cm^3) - Discard
Remove excess with vacuum - Discard
Diethyl ether:hexane 70:30 (5 cm^3) - Collect
Acetonitrile (1 x 5 cm^3) - Collect

Combine

i) Rotary evaporate to <10 cm^3

ii) Make to 15 cm^3 with acetonitrile

C_8 Clean Up

i) Add ultra pure water (50 cm^3)

ii) Load into 1 g C_8 cartridge at 1 cm^3/min

iii) Wash with water (5 cm^3) followed by acetonitrile:water 50:50 (2 x 2.5 cm^3) Discard

iv) Dry under vacuum for one half hour

v) Elute with hexane (4 x 2.5 cm^3)

vi) Make to an appropriate volume for gas-liquid chromatographic determination.

Appendix 2

Structure of lambda-Cyhalothrin & R171554

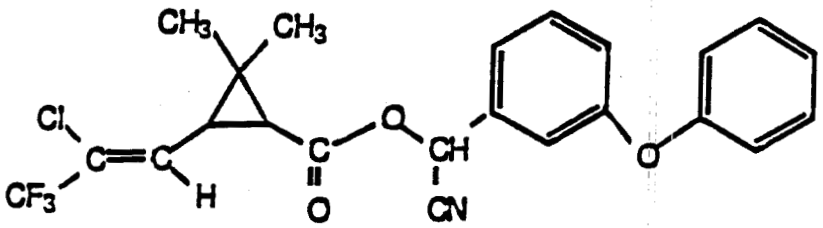
LAMBDA-CYHALOTHRIN (PP321)

Chemical Name : α-cyano-3-phenoxybenzyl-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate
a 1:1 mixture of the (Z)-(1R,3R), S-ester and (Z)-(1S,3S),R-ester
PP321 consists of one enantiomer pair designated B.

Emperical Formula : C_{23} H_{19} ClF_3 NO_3

Molecular Weight : 449.9

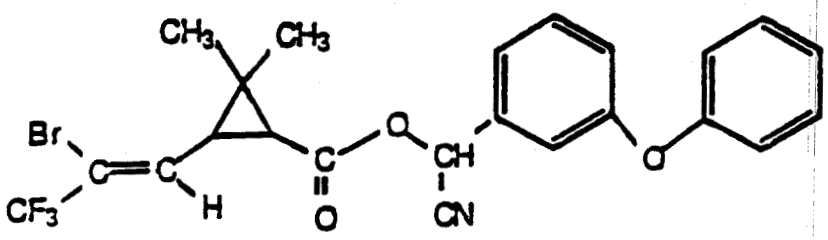
Structural Formula :



A sample of R171554 for use as an internal standard.

The internal standard used is the resolved cis isomer assumed by analogy to the chromatographic GLC characteristics of PP563 and other synthetic pyrethroids to be:

(RS)-α-cyano-3-phenoxybenzyl-(1RS)-cis-3-(Z-2-bromo-3, 3, 3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate.



Appendix 3

Water Quality Parameters of Gravel Pit Pond Water
(Stennis Space Center, MS)

Water quality Parameters of Gravel Pit Pond Water Collected at John C. Stennis Space Center

pH = 5.44

Suspended Solids (mg/L) = 8

Percent (%) Organic Matter in Suspended Solids (mg/L) = 88.3