Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Iprodione

MRID #: 419836-01

Matrix: Sediment

Analysis: HPLC/UV

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IPRODIONE, RP-30228 AND RP-32490 RESIDUES - A HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC METHOD FOR MEASUREMENT IN FIELD SEDIMENT

Submitted To:

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ABSTRACT

A high performance liquid chromatographic (HPLC) method is described for the analysis of Iprodione and its degradates RP-30228 and RP-32490 in field sediment. The method employs a solvent extraction of the sediment sample followed by filtration and a solvent-solvent back partition of the extract. The extract is purified by preparative florisil chromatography and analyzed by HPLC utilizing UV detection at 200 nm. Recoveries of Iprodione from field sediment fortified in the range of 20.0 - 100 ppb resulted in a mean recovery of 80% with a recovery range of 73%-91%. Control values were less than 10.4 and 8.9 ppb in two separate studies. Recoveries of RP-30228 from field sediment fortified in the range of 20.0 - 100 ppb resulted in a mean recovery of 78% with a recovery range of 60%-101%. Control values were less than 8.2 and 7.2 ppb in two studies. Recoveries of RP-32490 from field sediment fortified in the range of 20.0 - 100 ppb resulted in a mean recovery of 73% with a recovery range of 48%-103%. Control values were less than 11.2 and 9.73 ppb in two studies.

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INTRODUCTION

A method for determining Iprodione, RP-30228 and RP-32490 residues in field sediment is presented. The methodology for extraction, purification and detection of Iprodione and its degradates was developed for freshwater and seawater sediments.

PRINCIPLE AND APPLICATION

Iprodione and its degradates are extracted from field sediment by acidifying with hydrochloric acid and partitioning on a shaker table with 50% dichloromethane : 25% acetone : 25% ethyl acetate. The extract is filtered through a glass fiber membrane, dried through sodium sulfate, rotary evaporated to approximately 0.5 mL, evaporated to dryness under nitrogen and the residues dissolved in acetonitrile. The acetonitrile solution is partitioned twice with an equal volume of hexane, the acetonitrile is rotary evaporated to 0.5 mL and subsequently evaporated to dryness under nitrogen. The extracted residue is dissolved in 50% hexane : 50% dichloromethane. This solution is then submitted to florisil column chromatography and the Iprodione and RP-30228 residues are collected separately from the RP-32490 fraction. The solvent in each of the samples is evaporated to dryness, the residues are dissolved in a requisite volume of acetonitrile:water and analyzed by HPLC. Iprodione and RP-30228 are separated isocratically from coextracted artifacts using two HPLC columns in series and visualized with UV detection. The second degradate (RP-32490) is separated using a single column system. Linear regression analysis of Iprodione, RP-30228 and RP-32490 peak heights for samples and reference standards permits calculation of residue sample concentrations.

ANALYTICAL METHOD

Reagents

Acetonitrile, Burdick and Jackson, HPLC grade, UV cutoff @ 188 nm

Water, Barnstead NANOpure, HPLC grade

Sodium Sulfate, Mallinckrodt, Analytical Reagent

Hydrochloric acid, Mallinckrodt, AR Select

Dichloromethane, Fisher Scientific, Optima
Ethyl acetate, Burdick and Jackson, Residue grade
Hexane, Burdick and Jackson, HPLC grade
Toluene, Burdick and Jackson, UV cutoff @ 283 nm
Acetone, Burdick and Jackson, Reagent grade
Florisil, Fisher, 60-100 mesh, activated at 120 °C overnight
Glass wool, washed with dichloromethane
Iprodione, Lot No. EA 2002 SD8, 99.9% active ingredient, supplied by Rhone-Poulenc
RP-30228, Lot No. EA 2025 RFI, 100% active ingredient, supplied by Rhone-Poulenc
RP-32490, Lot No. EA 2026 RFI, 100% active ingredient, supplied by Rhone-Poulenc

Equipment

Balance, Ohaus Galaxy 160, four-place analytical balance
Balance, Ohaus MB 200, for moisture determination
Nalgene bottles, Nalgene, HDLPE, 500 mL
Flasks, volumetric, assorted sizes
Filter flasks, 1000 mL
Buchner funnels, 11.0 cm diameter
Glass fiber filters, Whatman, 934-AH, 11.0 cm diameter
Separatory funnels, 250 mL
Roundbottom flasks, assorted sizes
Chromatographic columns, glass, 270 mm (length) x 10 mm (ID)
Pipets, volumetric, assorted sizes
Serum bottles, Wheaton, assorted sizes, with Teflon-lined lids and metal crimp caps
Syringes, Hamilton, assorted sizes
Rotary evaporator, Buchii Model R110, with vacuum pump, 40 °C water bath
Shaker table, Eberbach, 0-500 rpm, or equivalent

Detailed Procedure

I. Preparation of Stock Solution

A. Iprodione (0.10 mg/mL)

1. Weigh 10.0 milligrams (A.I.) of Iprodione on an analytical balance.
2. Transfer the Iprodione to a 100-mL volumetric flask and dissolve to the mark with acetone.
3. Transfer the stock solution to a 100-mL amber serum vial and seal with a Teflon-lined crimp cap.
4. Store this stock solution in a refrigerator maintained at 4 °C.

B. RP-30228 and RP-32490 (0.10 mg/mL)

1. Weigh, dissolve, seal and refrigerate as previously described for Iprodione.

II. Quality Control Sample Fortification

A. Quality Control

1. Rinse all glassware with 50% dichloromethane : 25% acetone : 25% ethyl acetate prior to fortification.
2. To each 500 mL Nalgene bottle add approximately 50 grams (wet weight) of field sediment; see section IIIC.
3. To each sediment sample add 2.0 mL of 1.0 M HCl. Stir the sample in order to ensure homogeneity.
4. For a method validation or quality control sample, fortify each sample with Iprodione, RP-30228 and RP-32490 by volumetric addition of dilutions of the primary stock solutions.

NOTE: The fortification levels produced in the control field sediment samples for the method validation/recovery were 100, 50.0, and 20.0 ppb (three replicates at each level). An additional three field sediment samples were left unfortified and utilized as control samples.

III. Extraction

A. Field Sediment

1. To each Nalgene bottle, add approximately 50 grams (wet weight) of homogenized field sediment. Record the sediment weight to the nearest 0.01 grams.

   NOTE: At this stage, a secondary subsample should be taken and weighed in order to determine the percent soil content; see section IIIC.

2. To each sediment sample add 2.0 mL of 1.0 M HCl. Stir the sample in order to ensure homogeneity.

3. Add 300 mL of 50% dichloromethane : 25% ethyl acetate : 25% acetone to the 50 gram sample.

4. Place the samples on a shaker table; adjust the rpm to 300 and allow the samples to shake for 50-55 minutes.

5. Filter the sample through a Whatman 934-AH 11.0 cm glass fiber filter. Add 25 mL of the extraction solvent to the Nalgene bottle, cap and shake for approximately 15 seconds; pour the rinse through the sediment (on the filter paper) and collect the eluent in the filter flask. Repeat this 25 mL rinse two additional times.

6. Dry the organic extract through sodium sulfate and collect the eluent in a 1000 mL roundbottom flask. Rinse the filter flask with 25 mL of the extraction solvent. Pour this rinse through the sodium sulfate and collect the eluant in the roundbottom flask. Repeat this rinse one additional time; combine rinses in the roundbottom flask.

7. Evaporate the sample to approximately 0.5 mL on a rotary evaporator.

8. Evaporate the remaining solvent under a gentle stream of nitrogen.

9. To the flask add 50 mL of acetonitrile and transfer the dissolved residues into a 250 mL separatory funnel. Rinse the flask with 10-15 mL of acetonitrile and transfer the solution into the separatory funnel. Repeat this rinse one additional time.

10. To the separatory funnel add 50 mL of hexane. Shake the separatory funnel for 1-2 minutes. Drain the lower acetonitrile layer into a 250 mL beaker; discard the hexane layer. Return the acetonitrile extract (with rinsing) to the separatory funnel. Repeat the 50 mL hexane back-partition; drain the acetonitrile into the original 1000 mL roundbottom flask. Discard the hexane layer.

11. Rotary evaporate the acetonitrile to approximately 0.5 mL and evaporate the remaining acetonitrile under a gentle stream of nitrogen.

12. To the flask add 10 mL of 50% dichloromethane : 50% hexane. Swirl the solution in order to dissolve the extracted residues. Proceed to Section III B, Column Chromatography.

B. Column Chromatography

1. Place (and tamp) a plug of glass wool in a glass chromatographic column (270 mm length x 10 mm ID) fitted with a teflon stopcock.
2. Add approximately 1 inch of sodium sulfate to the column.
3. To the column add enough 100% activated Florisil to form a 3 inch column (excluding the one inch of sodium sulfate). Add an additional 1 inch of sodium sulfate to the top of the Florisil column.
4. To the column add 25 mL of acetone (as a rinse); drain the column completely dry and discard the eluent.

   Note: This step is necessary in order to clean the florisil of UV absorbing components. Add 25-30 mL of 50% dichloromethane : 50% hexane to the column and drain the solvent just into the upper sodium sulfate layer. Do not allow the florisil to go dry after this stage.
5. Transfer the dissolved residues from section A (step 8) to the column and open the stopcock fully. Rinse the roundbottom flask with 10 mL of 50% dichloromethane : 50% hexane and transfer the rinse onto the column. Drain the solvent into the upper sodium sulfate layer. Repeat this 10 mL

rinse two additional times and drain into the sodium sulfate. Close the stopcock. Discard all rinses.

6. Add 20 mL of dichloromethane to the column and drain into the sodium sulfate. Discard the eluent.
7. Place a 50 mL roundbottom flask under the column. Add 20 mL of 5% acetone : 95% dichloromethane to the column and drain into the sodium sulfate. Collect this fraction which contains Iprodione and RP-30228.
8. To the column add 20 mL of 15% acetone : 85 % dichloromethane; drain into the sodium sulfate. Collect this fraction separately which contains RP-32490.
9. Rotary evaporate the column eluents (separately) to approximately 0.5 mL. Evaporate the solution to dryness under a gentle stream of nitrogen.
10. Dissolve the residues in a requisite volume of 50% acetonitrile:50% HPLC grade water. Proceed to section IV for HPLC analysis of Iprodione, RP-30228 and RP-32490 residues.

C. Percent Soil Determination

1. Weigh approximately 10 grams of the sediment sample into an aluminum weigh dish.
2. Place the sample into an Ohaus MB 200 moisture determination balance and close the cover.
3. Bake the sample at 205 °C for 10 minutes.
4. Document the percent solids read from the LED.

IV. High Pressure Liquid Chromatography

A. Method:

Fractions containing Iprodione and RP-30228 are analyzed isocratically on a two-column system. Fractions containing RP-32490 are analyzed isocratically on a one column system. Both analyses utilize UV detection at 200 nm.

Instrumental conditions for the analysis of iprodione and RP-30228 are as follows:

Instrument:	Waters Model 510 liquid chromatograph solvent pump equipped with Waters Intelligent Sample Processor Model 710 B, ABI Model 783 Variable Wavelength Detector and Hewlett-Packard Model 3396A integrator.
Column:	Phenomenex Ultremex 5 µm ODS, 250 mm (length) x 4.6 mm ID; 2 columns in series
Mobile Phase:	80% acetonitrile:20% HPLC grade water
Mobile Phase Flowrate:	1.5 mL/minute
Pressure:	ca 2500 psi
Chartspeed:	0.3 cm/minute
Injection Volume:	125 µL
Wavelength:	200 nm
Sensitivity:	0.010 AUFS
Rise Time:	0.1 seconds
Attenuation:	2^6
Threshold:	6
Peak Width:	0.10 seconds
Area Reject:	500

Instrumental conditions for the analysis of RP-32490 are as follows:

Instrument:	Waters Model 510 liquid chromatograph solvent pump equipped with Waters Intelligent Sample Processor Model 710 B, ABI Model 783 Variable Wavelength Detector and Hewlett-Packard Model 3396A integrator.
Column:	Phenomenex Ultremex 5 µm ODS, 250 mm (length) x 4.6 mm ID
Mobile Phase:	60% acetonitrile:40% HPLC grade water
Mobile Phase Flowrate:	1.2 mL/minute

Pressure:	ca 1000 psi
Chartspeed:	0.3 cm/minute
Injection Volume:	100 µL
Wavelength:	200 nm
Sensitivity:	0.010 AUFS
Rise Time:	0.1 seconds
Attenuation:	2^6
Threshold:	6
Peak Width:	0.15 seconds
Area Reject:	500

B. Analysis

1. Prepare standard solutions containing both Iprodione and RP-30228 and separately RP-32490. Standard solution concentrations used for the recovery study were 125, 250, 500, 750 and 1000 µg/L.
2. Inject 125 µL (or 100 µL for RP-32490) of the 125 µg/L standard solution. Adjust the attenuation so that the peak signal results in at least a fifteen percent deflection from the baseline.
3. Inject 125 µL of each of the mixed standards, document the peak heights, and determine the correlation coefficient of the line. Proceed to step 4 if the correlation coefficient is greater than or equal to 0.985.
4. Inject 125 µL of several samples.
5. Identify each analyte by its retention time and document the respective peak heights.
6. After each set of samples, reinject 125 µL of each of the mixed standards and document the peak heights.
7. Construct a standard curve for each analyte (using all standard results) by plotting peak height observed versus the concentration (µg/L) of the standard injected.
8. The standard linear regression analysis for Iprodione, RP-30228 and RP-32490 is used to determine the concentration in each sample.

9. In order to determine the analytical result for each sample, the following equation is used:

Analytical Result (ppb) = A X D.F.

where:

Analytical Result = concentration of Iprodione, RP-30228 or RP-32490

A = concentration (µg/L) of sample from the regression analysis

D.F. = dilution factor, ratio of the final volume (mL) of the extracted sample to the initial mass (g) of sample

RESULTS AND DISCUSSION

Recovery samples were prepared in sediment collected from Live Oak, Louisiana, East Bernard, Texas, Matagorda, Texas, Bayou Bartholomew, Arkansas and Canal 43, Arkansas. Although the sediment types from the five sites varied substantially, recovery of Iprodione and its degradates from each of the sites was comparable.

Sediment samples were prepared which contained Iprodione, RP-30228 and RP-32490. Each sample was acidified with HCl, partitioned into 25% ethyl acetate : 25% acetone : 50% dichloromethane and filtered. The filtrate was evaporated to dryness, the residues dissolved in acetonitrile and back partitioned with hexane. The acetonitrile was evaporated and the residues dissolved in 50% hexane : 50% dichloromethane. The resulting solution was fractionated on a florisil column; one fraction contained Iprodione and RP-30228 and a separate fraction contained RP-32490. The organic extracts were rotary evaporated to approximately 0.5 mL, evaporated to dryness under nitrogen and dissolved in an acetonitrile/water mixture.

Recoveries from sediment fortified with Iprodione at 100 ppb ranged from 73.5% to 82.4% with an average recovery of 77.8% (N=7); recoveries at 50.0 ppb ranged from 80.9% to 84.1% with an average recovery of 82.6 (N=3); recoveries at 20.0 ppb ranged from 72.5% to 91.1% with an average recovery of 80.6% (N=7). The minimum detectable level for Iprodione was determined by linear regression analysis of the calculated value of one-half the mean of the peak

height of the lowest standard, resulting in an M.D.L. of less than 8.92 and 10.4 ppb in two studies.

Recoveries from sediment fortified with RP-30228 at 100 ppb ranged from 74.3% to 83.0% with an average recovery of 78.7% (N=7); recoveries at 50.0 ppb ranged from 77.7% to 83.9% with an average recovery of 81.1% (N=3); recoveries at 20.0 ppb ranged from 60.3% to 101% with an average recovery of 75.4% (N=7). The minimum detectable level for RP-30228 was determined by linear regression analysis of the calculated value of one-half the mean of the peak height of the lowest standard, resulting in an M.D.L of less than 8.25 and 7.16 ppb in two studies.

Recoveries from sediment fortified with RP-32490 at 100 ppb ranged from 48.1% to 84.2% with an average recovery of 68.8% (N=7); recoveries at 50.0 ppb ranged from 76.7% to 80.9% with an average recovery of 79.0% (N=3); recoveries at 20.0 ppb ranged from 48.9% to 103% with an average recovery of 73.8% (N=6). One of the 20.0 ppb recovery samples was unquantifiable due excessive contamination. The minimum detectable level for RP-32490 was determined by linear regression analysis of the calculated value of one-half the mean of the peak height of the lowest standard, resulting in an M.D.L of less than 9.73 and 11.2 ppb in two studies.

Linear regression analyses for the response of Iprodione, RP-30228 and RP-32490 are shown in Figures 1, 2 and 3, respectively. Recovery results for Iprodione, RP-30228 and RP-32490 are shown in Tables 1, 2 and 3, respectively. Representative chromatograms of Iprodione and RP-30228 at the 100 ppb, 50.0, 20.0 ppb and control levels using sediment collected from East Bernard, Texas are shown in Figures 4-7, respectively. Representative chromatograms of RP-32490 at the 100 ppb, 50.0 ppb, 20.0 ppb and control levels are shown in Figures 8-11, respectively.

SUMMARY

A novel procedure for the measurement of trace levels of Iprodione, RP-30228 and RP-32490 in sediment has been developed. Iprodione and its degradates are extracted from the sediment sample, preparatively isolated from coextracted artifacts and analyzed by high performance liquid chromatography.

Table 1. Analytical results for the recovery of Iprodione from sediment.

Fortified Level (ppb)	Sample Wet Weight (g)	Level Found (ppb)	Percent Recovery (%)
100 EB	50.0	80.3	80.3
100 EB	50.0	82.4	82.4
100 EB	50.0	76.7	76.7
100 MG	50.0	73.5	73.5
100 LO	50.0	74.9	74.9
100 BB	50.0	78.4	78.4
100 C4	50.0	78.1	78.1
50 EB	50.0	40.5	80.9
50 EB	50.0	42.1	84.1
50 EB	50.0	40.5	80.9
20 EB	50.0	16.5	82.3
20 EB	50.0	17.7	88.6
20 EB	50.0	15.7	78.3
20 MG	50.0	15.2	75.8
20 LO	50.0	14.5	72.5
20 BB	50.0	18.2	91.1
20 C4	50.0	15.1	75.7
CONT. EB	50.0	< 10.4	NA
CONT. EB	50.0	< 10.4	NA
CONT. EB	50.0	< 10.4	NA
CONT. MG	50.0	< 8.92	NA
CONT. LO	50.0	< 8.92	NA
CONT. BB	50.0	< 8.92	NA
CONT. C4	50.0	< 8.92	NA

Average Recovery: 79.7% ± 5.03 (N=17)
EB : East Bernard, Texas
MG : Matagorda, Texas
LO : Live Oak, Louisiana
BB : Bayou Bartholomew, Arkansas
C4 : Canal 43, Arkansas

Table 2. Analytical results for the recovery of RP-30228 from sediment.

Fortified Level (ppb)	Sample Wet Weight (g)	Level Found (ppb)	Percent Recovery (%)
100 EB	50.0	75.9	75.9
100 EB	50.0	83.0	83.0
100 EB	50.0	80.1	80.1
100 MG	50.0	74.3	74.3
100 LO	50.0	77.4	77.4
100 BB	50.0	81.1	81.1
100 C4	50.0	79.2	79.2
50 EB	50.0	38.9	77.7
50 EB	50.0	40.8	81.6
50 EB	50.0	41.9	83.9
20 EB	50.0	12.6	63.1
20 EB	50.0	14.4	71.9
20 EB	50.0	20.2	101
20 MG	50.0	14.7	73.7
20 LO	50.0	15.3	76.4
20 BB	50.0	12.1	60.3
20 C4	50.0	16.3	81.4
CONT. EB	50.0	< 8.25	NA
CONT. EB	50.0	< 8.25	NA
CONT. EB	50.0	< 8.25	NA
CONT. MG	50.0	< 7.16	NA
CONT. LO	50.0	< 7.16	NA
CONT. BB	50.0	< 7.16	NA
CONT. C4	50.0	< 7.16	NA

Average Recovery: 77.8% ± 8.77 (N=17)
EB : East Bernard, Texas
MG : Matagorda, Texas
LO : Live Oak, Louisiana
BB : Bayou Bartholomew, Arkansas
C4 : Canal 43, Arkansas

Table 3. Analytical results for the recovery of RP-32490 from sediment.

Fortified Level (ppb)	Sample Wet Weight (g)	Level Found (ppb)	Percent Recovery (%)
100 EB	50.0	82.5	82.5
100 EB	50.0	84.2	84.2
100 EB	50.0	79.1	79.1
100 MG	50.0	58.1	58.1
100 LO	50.0	63.8	63.8
100 BB	50.0	48.1	48.1
100 C4	50.0	65.8	65.8
50 EB	50.0	38.4	76.7
50 EB	50.0	39.7	79.4
50 EB	50.0	40.5	80.9
20 EB	50.0	*	*
20 EB	50.0	20.5	103
20 EB	50.0	20.1	100
20 MG	50.0	12.9	64.5
20 LO	50.0	12.6	62.8
20 BB	50.0	9.77	48.9
20 C4	50.0	12.8	63.6
CONT. EB	50.0	< 11.2	NA
CONT. EB	50.0	< 11.2	NA
CONT. EB	50.0	< 11.2	NA
CONT. MG	50.0	< 9.73	NA
CONT. LO	50.0	< 9.73	NA
CONT. BB	50.0	< 9.73	NA
CONT. C4	50.0	< 9.73	NA

Average Recovery: 72.6% ± 15.9 (N=16)
* : This sample was unquantifiable due to interferences
EB : East Bernard, Texas
MG : Matagorda, Texas
LO : Live Oak, Louisiana
BB : Bayou Bartholomew, Arkansas
C4 : Canal 43, Arkansas

Figure 1. **The linear regression analysis for iprodione standards used in the recovery study.**

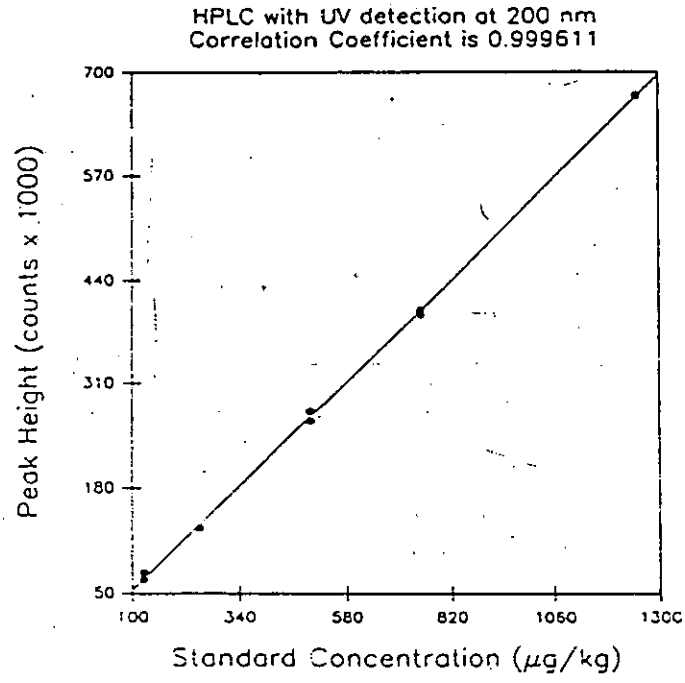


Figure 2. The linear regression analysis for RP-30228 standards used in the recovery study.

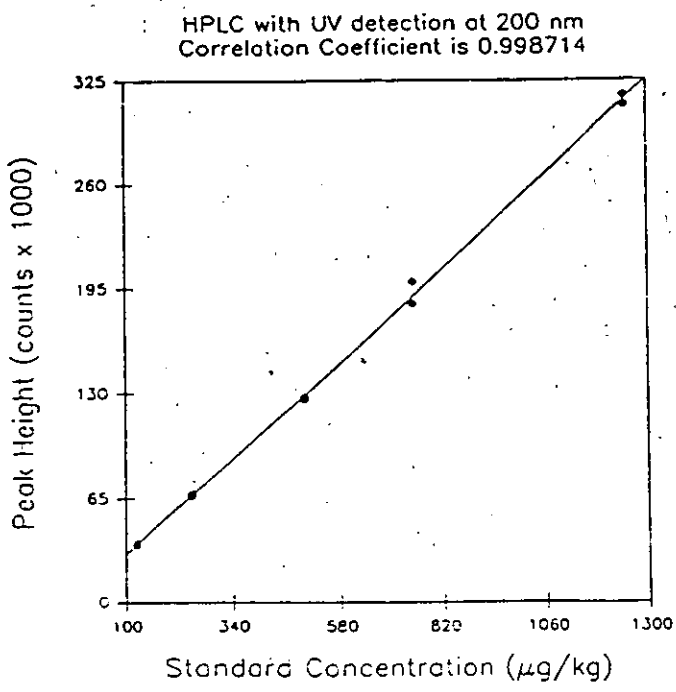


Figure 3. **The linear regression analysis for RP-32490 standards used in the recovery study.**

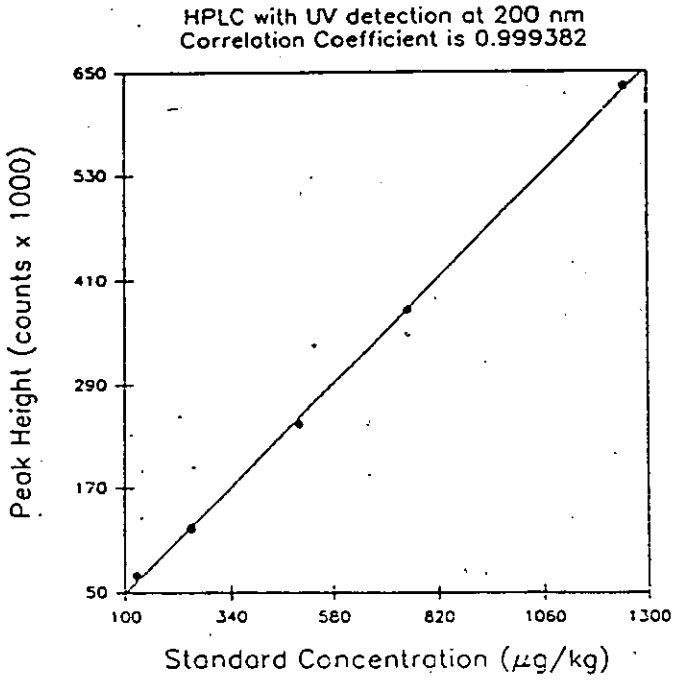
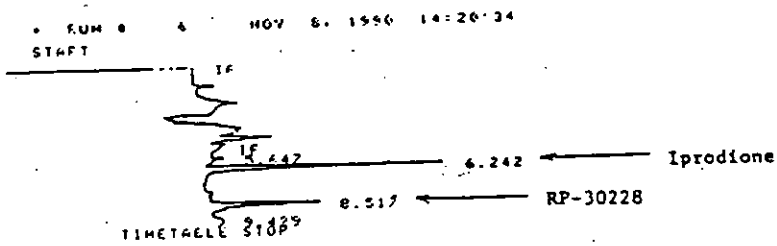


Figure 4. A representative chromatogram of a sediment extract (reflecting 100 ppb Iprodione and RP-30228) utilizing UV detection.



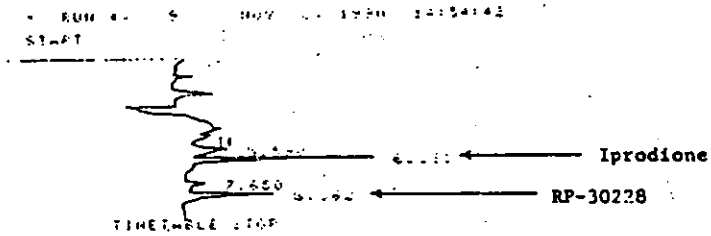
RUN# 6 NOV 8, 1990 14:20:34

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
5.647	9465	BP	.162	4.20812
6.242	144106	PB	.143	64.86931
8.517	67863	VV	.177	26.17160
9.429	3488	PB	.128	1.55076

TOTAL HEIGHT= 224922
MUL FACTOR=1.0000E+00

Figure 5. **A representative chromatogram of a sediment extract (reflecting 50.0 ppb Iprodione and RP-30228) utilizing UV detection.**



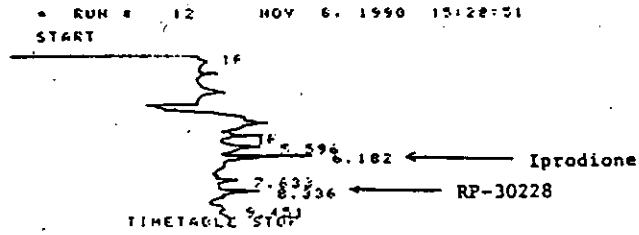
RUN # 9 Nov 15 1990 14:54:42

HEIGHTS

RT	HEIGHT	Type	WIDTH	HEIGHT%
5.590	26310	B	.331	11.34324
6.191	199216	VB	.157	85.93968
7.650	11757	B	.173	5.07270
9.362	53074	BB	.201	22.89938

TOTAL HEIGHT= 231657
MUL FACTOR=1.0000E+00

Figure 6. A representative chromatogram of a sediment extract (reflecting 20.0 ppb Iprodione and RP-30228) utilizing UV detection.



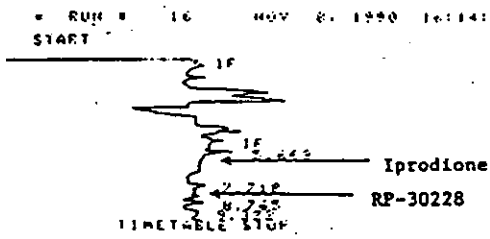
RUN# 12 NOV 6, 1990 15:28:51

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
5.556	23177	BV	.296	19.50119
6.182	56047	VV	.157	44.38243
7.633	13394	PP	.153	10.56730
8.336	24941	VV	.178	19.75417
9.451	6501	BV	.376	5.45491

TOTAL HEIGHT= 126510
MUL FACTOR=1.0000E+00

Figure 7. **A representative chromatogram of an unfortified (control) sediment extract utilizing UV detection.**



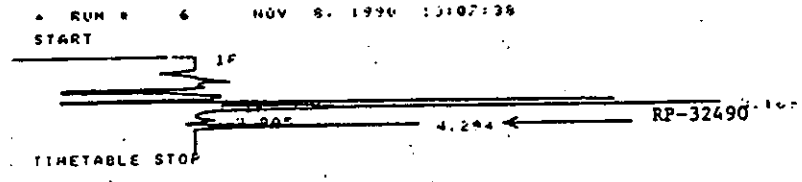
RUN# 16 NOV 8, 1990 16:14:22

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
5.669	18398	BP	.118	39.79754
7.718	11062	PV	.196	23.93066
8.765	12545	PB	.165	27.13664
9.322	4225	PB	.067	9.13496

TOTAL HEIGHT= 46229
MUL FACTOR=1.0000E+00

Figure 8. A representative chromatogram of a sediment extract (reflecting 100 ppb RP-32490) utilizing UV detection.



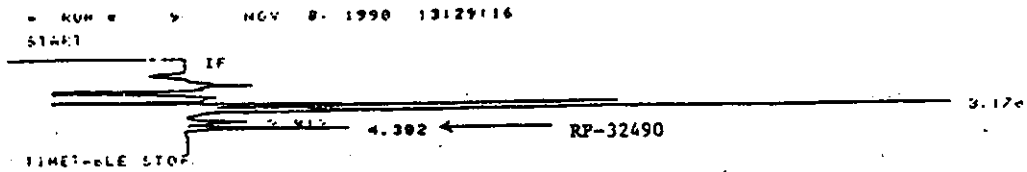
RUN# 6 NOV 8, 1990 13:07:38

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
3.169	367913	BB	.111	66.68970
3.985	15646	BP	.22[illegible]	2.38859
4.294	135161	PB	.[illegible]	29.91279

TOTAL HEIGHT= 468726
MUL FACTOR=1.0000E+00

Figure 9. **A representative chromatogram of a sediment extract (reflecting 50.0 ppb RP-32490) utilizing UV detection.**




```
* RUN #    *      NOV  8, 1990  13:29:16
START
TIME-TABLE STOP


RUN#    *          NOV  8, 1990  13:29:16

HEIGHTS
      RT     HEIGHT TYPE   WIDTH    HEIGHT%
   3.170     478416   BB    .110   78.36694
   3.412      35895   BV    .191    5.88044
   4.302      96167   VB    .128   15.75263

TOTAL HEIGHT= 610478
MUL FACTOR=1.0000E+00
```

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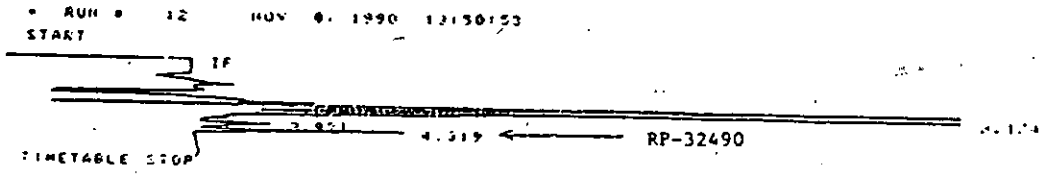
Figure 10. **A representative chromatogram of a sediment extract (reflecting 20.0 ppb RP-32490) utilizing UV detection.**



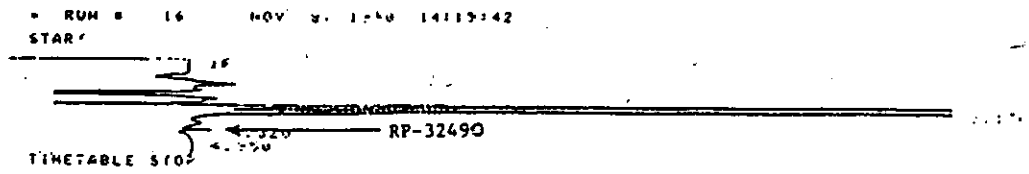
*RUN# 12 NOV 6, 1990 13:50:53

*HEIGHTS

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
3.174	593382	BP	.168	78.16909
3.921	42815	PV	.164	5.63453
4.319	123071	VP	.125	16.19637

TOTAL HEIGHT= 759268.
MUL FACTOR=1.0000E+00

Figure 11. **A representative chromatogram of an unfortified (control) sediment extract utilizing UV detection.**




```
* RUN #   16      NOV  9. 1990  14:19:42
STAR
TIMETABLE STOP

RUN#    16          NOV  9. 1990  14:19:42

HEIGHT%
      RT     HEIGHT TYPE   WIDTH    HEIGHT%
   3.170     542558   BB    .199    95.98854
   4.320      18706   PP    .142     3.30944
   4.950       3902   FB    .023      .70201

TOTAL HEIGHT= 565232
AUL FACTOR=1.0000E+00
```

APPENDIX VI: ANALYTICAL RESULTS FOR SEDIMENT DRY WEIGHT