

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Iprodione & Metab

MRID #: 452392-03

Matrix: Water

Analysis: LC/MS/MS

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Method of Analysis for Iprodione and Its Metabolites in Water

I. INTRODUCTION

A. Scope

This method sets forth the procedure for determining the residues of iprodione and its metabolites RP 30228 and RP 32596 in ground water.

B. Principle

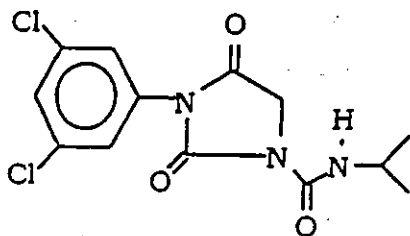
An analytical method is described for the determination of residues of iprodione and its metabolites RP 30228 and RP 32596 in ground water. Residues of iprodione, RP 30228 and RP 32596 are extracted from water using a RP-102 resin cartridge, then removed with acetonitrile.

All residue analysis is accomplished by LC-MS-MS on a C8 column. Quantification of results is based on a comparison of peak areas with those of known standards.

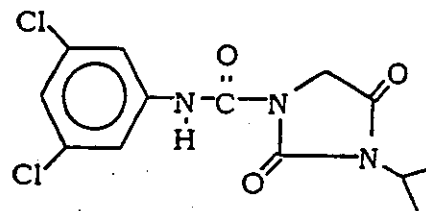
C. Method Limits

The method detection limits (MDL) and limits of quantification (LOQ) for iprodione (RP 26019) and its metabolites RP 30228 and RP 32596 in water have not been determined. This information will be obtained from the subsequent validation study. Target level LOQ is 50 ppt.

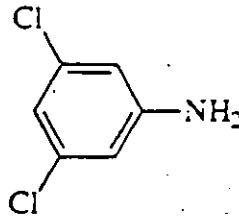
D. Chemical Structures



RP26019 (Iprodione)



RP30228



RP 32596

II. MATERIALS

Unless otherwise noted, equivalent brands and/or suppliers can be used.

A. Reagents/Solvents

Acetonitrile Omni-Solv (EM Science, Cat. No. AX0142-1)
Formic Acid Suprapur (EM Science, Cat. No. 11670-1)
Water Omni-Solv (EM Science, Cat. No. WX0004-1)

B. Equipment and Supplies

Adaptors, 1,3 and 6 mL (Varian, 1213-1001)

Balance :

accuracy ± 0.1 mg (analytical standards) (Mettler AT 201 or equiv)
accuracy ± 0.1 g (samples and chemicals)(Mettler PC 4000 or equiv)

Bottles, amber, 4 oz. (Qorpak)

Cartridges ,Spe-ed™ SPE, RP-102 Resin (100mg/1mL)
(Applied Separations, Cat. No. 4207, *no substitute*)

Cartridge Adaptors, SPE
(University Research Glass, Cat. No. URG-2440-SPECA)

Disposable pipettes

Glass wool

Graduated cylinders

Column. HPLC, Columbus C8, 4.6x100 mm, 5 μ m. *no substitute*
(Phenomenex, Cat. No. 00D-4187-E0, *no substitute*)

Magnetic stirrer

Pipette bulb

Precolumn HPLC Filter, Ultra Low Dead Volume, 0.5 μ m frit
(Upchurch, A-318)

Reservoirs, 75 mL volume (Varian, 1213-1012)

Solvent jugs, 4 L brown glass

Stopcocks, Luer Lock (Varian, 1213-1005)

Stoppers, glass, 24/40

Vacuum Gauges

Volumetric flasks

Volumetric pipettes

Vial, clear, 1.5mL; cap, open top; septa, split
(Sun, 200-250; 200-292, 500-870)

C. Solutions

The following is a list of the solutions used in the analyses of ground water. Example procedures for the preparation of each solution are also provided.

Note that the reagent water used in the preparations should be HPLC grade.

1. Solution of -pH 3 Formic acid in Water
Pipet formic acid into a stirring volume of HPLC grade water until the pH is 3 ± 0.4 . Check the pH with a pH meter. (about 0.5 mL formic acid/liter water)
2. 90:10 Solution of pH 3 Water (formic):Acetonitrile
Using a graduated cylinder, transfer 900 mL of a solution of pH 3 formic acid in H₂O and 100 mL CH₃CN to a 4 L brown glass solvent jug that is clean and dry or a jug which was previously used for this solution. Repeat until the desired quantity has been made.
3. Solution of 50:50 Water:Acetonitrile
Using a graduated cylinder, transfer 500 mL of HPLC grade H₂O and 500 mL CH₃CN to a 4 L brown glass solvent jug that is clean and dry or a jug which was previously used for this solution. Repeat until the desired quantity has been made.
4. 50:50 Solution of pH 3 Water (formic):Acetonitrile
Using a graduated cylinder, transfer 500 mL of a solution of pH 3 formic acid in H₂O and 500 mL CH₃CN to a 4 L brown glass solvent jug that is clean and dry or a jug which was previously used for this solution. Repeat until the desired quantity has been made.

D. Analytical Standards

Common name/alias: Iprodione, RP 26019

Chemical name: 3-(3,5-dichlorophenyl)-N-isopropyl-2,4-dioximidazolidine-1-carboxamide

*Solubility*¹:

acetone:	34.2(unit : g/100 ml)
acetonitrile:	16.8
hexane:	0.059
dichloromethane:	45.0
distilled water:	0.00122

Common name/alias: RP 30228

Chemical name: 1-(3,5-dichlorophenyl)carbamoyl-3-isopropyl hydantoin

Common name/alias: 3,5- DCA/ RP 32596

Chemical name: 3,5-dichloroaniline
(CAS No. 626-43-7)

III. FORTIFICATION AND CALIBRATION STANDARD SOLUTIONS

A. Preparation

All the standard solutions must be stored in amber glass bottles. Stock solutions will be stored at about -10°C . All other standards solutions will be stored at $4^{\circ}\text{C} \pm 3^{\circ}\text{C}$ when not in use. Solutions should be allowed to warm to room temperature prior to use. The following is an example of a procedure to follow in preparing standard solutions. Alternate or additional standards of appropriate weight and volume may be prepared as needed. The "~" symbol indicates approximately.

Stock solutions:

1. Weigh $\sim 0.1000\text{g}$ (corrected for purity) each of iprodione and RP 32596 and $\sim 0.0100\text{g}$ (corrected for purity) of RP 30228 into separate 100-mL volumetric flasks and dilute with $\sim 50\text{ mL}$ acetonitrile. Sonicate for approximately 5 minutes if necessary. Add $\sim 45\text{ mL}$ of pH 3 water and mix by inversion. Allow the solution to reach ambient temperature before diluting to the mark with more pH 3 water. The concentration of these stock standards is $\sim 1000\text{ }\mu\text{g/mL}$ of iprodione and RP 32596 and $\sim 100\text{ }\mu\text{g/mL}$ of RP 30228.

Standards solutions:

2. Transfer 10 mL of the $\sim 1000\text{ }\mu\text{g/mL}$ iprodione and RP 32596 standard solutions, via volumetric class "A" pipettes, to one 100 mL volumetric flask. Dilute to mark with a 50:50 solution of pH 3 formic acid in $\text{H}_2\text{O}:\text{CH}_3\text{CN}$. Cap and mix by inversion. The concentration of this standard is $\sim 100\text{ }\mu\text{g/mL}$ of iprodione and RP 32596.
3. Transfer 20 mL of the $\sim 100\text{ }\mu\text{g/mL}$ RP 30228 standard solution, via a volumetric class "A" pipette, to a 100 mL volumetric flask. Dilute to mark with a 50:50 solution of pH 3 formic acid in $\text{H}_2\text{O}:\text{CH}_3\text{CN}$. Cap

and mix by inversion. The concentration of this standard is ~20 $\mu\text{g}/\text{mL}$ of RP 30228.

4. Transfer 20 mL of the ~100 $\mu\text{g}/\text{mL}$ iprodione and RP 32596 standard solutions, via volumetric class "A" pipettes, to one 100 mL volumetric flask. Dilute to mark with a 50:50 solution of pH 3 formic acid in $\text{H}_2\text{O}:\text{CH}_3\text{CN}$. Cap and mix by inversion. The concentration of this mixed standard is ~20 $\mu\text{g}/\text{mL}$ of iprodione and RP 32596.

Mixed standard solutions:

5. Transfer 10 mL of the ~20 $\mu\text{g}/\text{mL}$ iprodione, RP 32596 (III.A.2) and RP 30228 (III.A.3) standard solutions, via volumetric class "A" pipettes, to one 100 mL volumetric flask. Dilute to mark with a 90:10 solution of pH 3 formic acid in $\text{H}_2\text{O}:\text{CH}_3\text{CN}$. Cap and mix by inversion. The concentration of this mixed standard is ~2 $\mu\text{g}/\text{mL}$.
6. Transfer 10 mL of the ~2 $\mu\text{g}/\text{mL}$ mixed standard solution of iprodione, RP 32596 and RP 30228 via a volumetric class "A" pipette, to one 100 mL volumetric flask. Dilute to mark with a 90:10 solution of pH 3 formic acid in $\text{H}_2\text{O}:\text{CH}_3\text{CN}$. Cap and mix by inversion. The concentration of this mixed standard is ~0.2 $\mu\text{g}/\text{mL}$.
7. Using a class "A" volumetric pipette, transfer 10 mL of the mixed standard (step III.A.6.) to a 100-mL volumetric flask. Dilute to mark with a 90:10 solution of pH 3 formic acid in $\text{H}_2\text{O}:\text{CH}_3\text{CN}$. Cap and mix by inversion. The concentration of this mixed standard is ~20 ng/mL iprodione, RP 30228 and RP 32596.
8. Using a class "A" volumetric pipette, transfer 3 mL of the mixed standard (step III.A.6.) to a 100-mL volumetric flask. Dilute to mark with a 90:10 solution of pH 3 formic acid in $\text{H}_2\text{O}:\text{CH}_3\text{CN}$. Cap and mix by inversion. The concentration of this mixed standard is ~6 ng/mL iprodione, RP 30228 and RP 32596.
9. Using a class "A" volumetric pipette, transfer 25 mL of the ~20 ng/mL mixed standard (step III.A.7.) to a 100-mL volumetric flask. Dilute to mark with a 90:10 solution of pH 3 formic acid in $\text{H}_2\text{O}:\text{CH}_3\text{CN}$. Cap and mix by inversion. The concentration of this mixed standard is ~5 ng/mL iprodione, RP 30228 and RP 32596.
10. Using a class "A" volumetric pipette, transfer 2 mL of the mixed standard (step III.A.6.) to a 100-mL volumetric flask. Dilute to mark

with a 90:10 solution of pH 3 formic acid in H₂O:CH₃CN. Cap and mix by inversion. The concentration of this mixed standard is ~4 ng/mL iprodione, RP 30228 and RP 32596.

11. Using a class "A" volumetric pipette, transfer 3 mL of the mixed standard (step III.A.6.) to a 100-mL volumetric flask. Dilute to mark with a 90:10 solution of pH 3 formic acid in H₂O:CH₃CN. Cap and mix by inversion. The concentration of this mixed standard is ~2 ng/mL iprodione, RP 30228 and RP 32596.

B. Stability

1. To evaluate the stability, the following formula has been used :

$$\text{percent stability} = [1 - (\text{old std. soln.} / \text{new std. soln.})] \times 100$$

The old standard solution should give detector responses within 10% of those of the new standard solution in order for the given standard solution to be considered stable under the storage conditions.

2. Stock solutions: Each product prepared in acetonitrile and stored at about -10°C was stable for up to 6 months².
3. Standard solutions: Mixed standard solutions of iprodione , RP30228 and RP 32596 prepared in acidified water:acetonitrile and stored at 4°C ± 3°C was stable for up to 6 months².

IV. METHOD PROCEDURES

A. General Notes

- A1. The "~" symbol indicates 'approximately.'
- A2. The pH of samples to be analyzed may be measured with a pH meter or narrow range pH paper. Adjust pH between 2-4 with formic acid.
- A3. If the samples are turbid, glass wool can be used in the SPE cartridges to aid filtration. The glass wool must be washed with acetonitrile prior to use. Place ~ 35 g glass wool in a 500 mL Nalgene[®] bottle, add ~300 mL acetonitrile and shake on a platform

shaker for ~ 15 minutes. Filter through a 9 cm GF/C filter paper on a Buchner funnel, vacuum dry. Rinse with ~ 100mL acetonitrile and vacuum dry on the filter. Store in a wide-mouthed bottle.

- A4. Conditioning of the cartridges in step B4 can be started earlier and does not have to be done after the completion of steps B1-B3. However, the cartridges should be used the day of conditioning.
- A5. Throughout the conditioning and elution process (unless otherwise specified) cartridges should not be allowed to run dry.
- A6. The flow rate for loading the water sample on the cartridges (step B5) is faster than the conditioning and elution flow rate.

B. Ground Waters

(Analysis for Iprodione (RP 26019), RP 30228 and RP 32596)

- B1. Weigh ~200 g of sample into a 500 mL Nalgene[®] bottle. The sample may be stored in a refrigerator until needed.
- B2. For recoveries, fortify the sample with the appropriate standard solution. Immediately adjust the pH to 2-4 with formic acid. Cap and mix on a platform shaker for ~5 minutes.
- B3. Immediately set-up a RP-102 cartridge (100 mg) on a purification system connected to a vacuum. (If samples are turbid - insert a plug of ~0.1 g of acetonitrile-washed glass wool into the cartridge). Place a reservoir on top of the cartridge.
- B4. Condition the cartridge with ~4 ml of acetonitrile followed by ~4 ml of pH3 water. (~1 drop/3-4 seconds. Do not allow the cartridge to dry).
- B5. Apply prepared sample to the cartridge (~1 drop/second). Do not allow the cartridge to dry. *Note that the flow rate for loading the water sample on the cartridges is faster than the conditioning and elution flow rate.*
- B6. Add ~2mL of pH3 formic acid in water to the cartridge. Elute and discard the effluent. (~1 drop/3-4 sec. Do not allow the cartridge to dry).

- B7. Add ~ 1 mL of a 50:50 solution of water:acetonitrile to the cartridge. Elute and discard the effluent. (~1 drop/2 sec. Do not allow the cartridge to dry).
- B8. Air dry the cartridge under high vacuum (~20 inches of mercury) for ~2 minutes.
- B9. Add 1 mL of acetonitrile to the cartridge. Apply positive pressure and push ~1/3 of the solvent through the cartridge. *Positive pressure can be applied via a handheld nitrogen line.* Vent the pressure and allow the cartridge to soak for 1-2 minutes. Reapply pressure and elute the solvent (~1 drop/second) into an appropriately sized volumetric flask.
- B10. Dilute to the mark with pH3 formic acid in H₂O. Mix by inversion. Samples are ready for LC-MS-MS analysis. Suggested final dilution volumes are 2mL for samples fortified at the LOQ level and 25mL for samples fortified at the 10 x LOQ level.

V. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY-MASS SPECTROMETRY (LC-MS)

A. Conditions

<u>Instrument used:</u>	Perkin Elmer Sciex API III+ LC/MS/MS system coupled to a Hitachi L6200 HPLC pump via PE. Hitachi AS2000 autosampler
<u>MS Mode:</u>	MS/MS multiple reaction monitoring (MRM)
<u>Ionization:</u>	Atmospheric Pressure Chemical Ionization (APCI) using Heated Nebulizer Interface
<u>Heated Nebulizer Settings:</u>	Heated air at ~4.00 L/min, 450°C
<u>Nebulizer pressure:</u>	80 psi
<u>Curtain gas flow:</u>	Nitrogen at ~1.2 L/min
<u>Collision gas:</u>	Argon at approximately 275×10^{13} atoms/cm ²

Period 1 Positive Mode:

Orifice voltage: 65 V
Collision energy (R2-R0): 11V - 30V = -19V
Mass Transition: RP32596: 162.1/127.1

Period 2 Negative Mode:

Orifice voltage: -57 V
Collision energy (R2-R0): -7V - 30V = -37V
Mass Transition: iprodione(RP 26019): 243.1/42.0

Period 3 Negative Mode:

Orifice voltage: -38 V
Collision energy (R2-R0): -20V - 30V = -50V
Mass Transition: RP 30228: 328.1/141.0

Column: Phenomenex, Columbus C8, 4.6 x 100mm, 5µm particle size

Column In-Line Filter: Upchurch, Ultra Low Dead Volume, 0.5µm frit

Mobile phase flow rate: 1.0 mL/min

Mobile phase composition: isocratic
60% acetonitrile
40% 0.1% acetic acid in HPLC grade water
- 8 minutes between injections (set autosampler to 7.5 minutes run time)

Injection volume: 40 µl

Note the indicated LC-MS-MS parameters are guidelines and should be optimized for the instrument and column actually used. Instrument parameters and mobile phase compositions may be adjusted to improve separation from interfering peaks.

APPROXIMATE RETENTION TIMES

RP 32596	3.3 minutes
RP 26019	4.4 minutes
RP 30228	7.0 minutes

Retention times may vary from those presented above.

Example chromatograms are attached (see section X). Note that the retention times may vary from system to system.

B. Performance Criteria

The following criterion should be met before analysis of samples begins. Once the criterion has been met it is not necessary to perform them again.

First criterion:

Run a standard solution corresponding to a level at or below the estimated LOQ and obtain a signal to noise ratio of at least 9:1.

If this criterion cannot be met, optimize instrument operating parameters or change instrument method parameters such as injection size until a signal to noise ratio of 9:1 is obtained.

If this criterion still cannot be met by changing operating parameters, run higher level standards until a signal to noise ratio of 9:1 is obtained. This will require adjusting the method final sample dilution such that this standard level corresponds to the required LOQ.

Second criterion:

Run a set of standards of four or more concentration levels, from at or below the LOQ, up to the highest concentration level to be included in the analysis. Generate a calibration curve for each analyte and obtain a linear regression with a correlation coefficient of at least 0.90 for each analyte. If this criterion is met, the samples may be run with standards interspersed. Do not use any sample run data if the combined regression for standards run immediately before, during and after the samples do not meet this criterion.

Note:

To stabilize the response of the instrument, it has been found useful to run at least one standard and/or a sample or untreated control solutions as "wake up" runs before the actual runs to be used in calculations are commenced.

VI. CALCULATIONS

Linear regression should be used to generate calibration curves for RP 26019 and RP 30228 and RP 32596. After the instrument performance criteria are met, a minimum of four standards over a range of concentration levels should be included with a set of samples. Standards should be interspersed with samples to compensate for any minor change in instrument response. Samples should be diluted such that any peak areas or heights are within the area or height range between the lowest and highest standards injected.

Linear regression coefficients should be calculated on standard concentration (ng/mL) versus peak area or height. The data from the analytical standards should then be fit to the linear model,

$$Y = A + BX.$$

The equation to be used to estimate the residues in the samples is:

$$E = \frac{(Y - A) \times C}{B \times D}$$

where: Y = response of analyte of interest (peak area or height)

A = intercept from linear regression analysis (peak area or height)

B = slope from linear regression analysis (response per concentration)

C = final sample volume (mL)

D = starting weight in grams of sample in final volume (g)

E = concentration of analyte in sample in parts per billion (ppb or ng/mL)

VII. SAFETY

All available appropriate Material Safety Data Sheets should be available to the study personnel during the conduct of the study. General laboratory safety precautions should be taken. This method does not present any specific risks.

VIII. REFERENCES

1. "Iprodione technical grade. Solubility at 20°C" Chabassol, Y. & Gomez, J.L. AG/CRLD/AN 9115375, April 9, 1991
2. "Storage Stability of Iprodione (RP-26019), its Isomer (RP 30228), and its Metabolite (RP-32596) in Various Raw Agricultural Commodities and Processing Fractions" RPAC file# 44327 R. S. Plaisance, June 13, 1994.

IX. RECOVERY DATA

Ground Water Recovery Data

(Clovis, California water)

Sample Identification	Fortification Level (ppt)	Recovery (%)		
		RP 26019	RP 30228	RP 32596
641	0	0	0	0
643	0	0	0	0
642	50	92	80	83
681	0	0	0	0
682	50	94	76	85
683	50	96	84	89

X. EXAMPLE CHROMATOGRAMS

A. Calibration Data

Figure 1. Standard: 6.0 ng/ml - RP 26109, RP 30228, RP 32596

MacQuan, version 1.5

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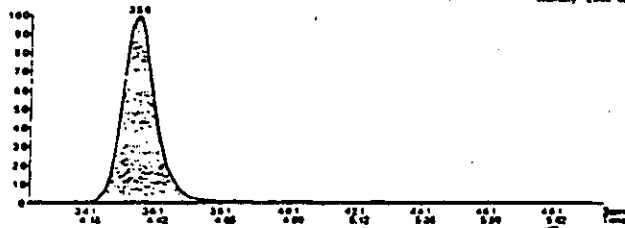
Printed: Tue, Jun 16, 1998 17:29

Calibration File: cal_june98_posneg Path: Office G3:DRJS: Iprodione Water: CAS Iprod Water:june98:98june08:

Comments: No Comments

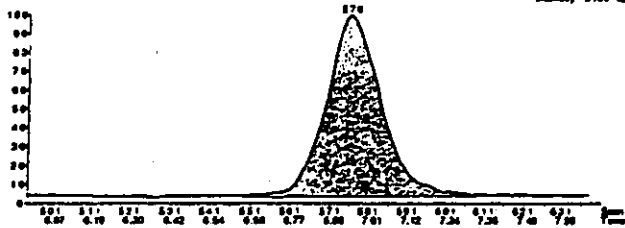
00jun08004 Iprodione related004 Mon, Jun 8, 1998 17:24
6 ng/ml MS2

7.70 in 3 peaks
No Internal Standard
Use Area
2 1.00 MHz, 171 scans
242.1-42.9
Name Three 5.0
Queue Three 1.0
Min. Width 0
Max. Width 1.5
Scan Width 100
RT Min. (secs) 2.0
Smooth 2
Expected RT 4.40
Area 2074.3
Height 25.20
Start Time 4.14
End Time 4.76
Integration Width 0.62
Retention Time 4.20
Integration Type A - 00



00jun08004 Iprodione related004 Mon, Jun 8, 1998 17:24
6 ng/ml MS2

7.70 in 3 peaks
No Internal Standard
Use Area
3 1.00 MHz, 142 scans
222.1-42.9
Name Three 4.0
Queue Three 2.0
Min. Width 0
Max. Width 1.5
Scan Width 100
RT Min. (secs) 2.0
Smooth 2
Expected RT 7.10
Area 34002
Height 2000
Start Time 6.52
End Time 7.31
Integration Width 0.60
Retention Time 6.80
Integration Type A - 00



00jun08004 Iprodione related004 Mon, Jun 8, 1998 17:24
6 ng/ml MS2

7.70 in 3 peaks
No Internal Standard
Use Area
1 1.70 MHz, 324 scans
152.1-127.1
Name Three 5.0
Queue Three 2.0
Min. Width 0
Max. Width 2
Scan Width 100
RT Min. (secs) 0
Smooth 0
Expected RT 3.42
Area 20014
Height 2270
Start Time 3.22
End Time 3.53
Integration Width 0.20
Retention Time 3.30
Integration Type A - 00

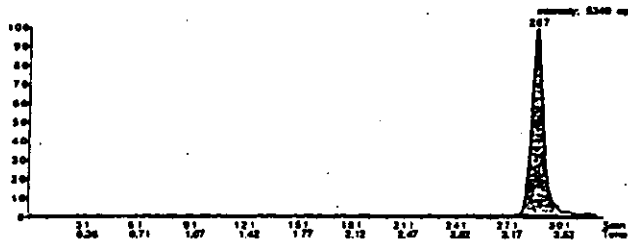


Figure 2. Standard: 5.0 ng/ml - RP-26109, RP 30228, RP 32596

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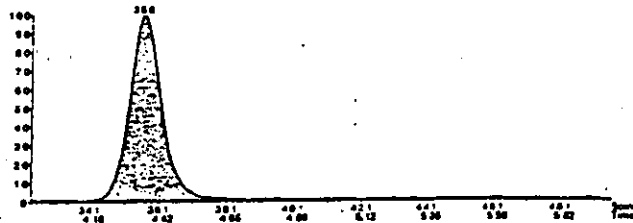
Printed: Tue, Jun 16, 1998 17:21

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Comments: No Comments

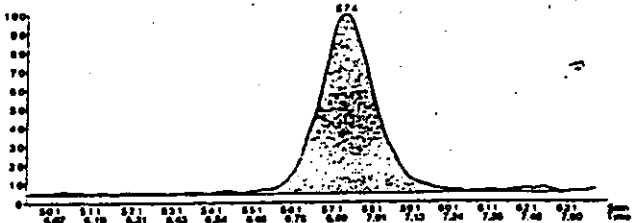
00jun0805 Iprodione r0100005 Mon, Jun 8, 1998 17:32
5 signal MS2

7.70 in 3 peaks
No Internal Standard
Use Area
2.100 u/min, 177.000 m/min
Area: 2164
Chrom. Time: 4.70
Min. Width: 0.71
Max. Width: 0.71
Peak Width: 1.5
RT Min. (min): 2.0
Smooth: 3
ScanRate: 4.00
Area: 10540
Height: 2164
Start Time: 4.70
End Time: 4.71
Integration Width: 0.51
Resolution Time: 4.36
Integration Type: A - 50



00jun0805 Iprodione r0100005 Mon, Jun 8, 1998 17:32
5 signal MS2

7.70 in 3 peaks
RP 30228
No Internal Standard
Use Area
3.100 u/min, 146.000 m/min
Area: 27781
Height: 2411
Chrom. Time: 6.66
Min. Width: 7.23
Max. Width: 6.63
Peak Width: 15
RT Min. (min): 2.0
Smooth: 3
ScanRate: 7.00
Area: 27781
Height: 2411
Start Time: 6.66
End Time: 7.23
Integration Width: 0.63
Resolution Time: 6.03
Integration Type: A - 50



00jun0805 Iprodione r0100005 Mon, Jun 8, 1998 17:32
5 signal MS2

7.70 in 3 peaks
32696
No Internal Standard
Use Area
1.270 u/min, 32.000 m/min
Area: 4314
Height: 4314
Chrom. Time: 2.96
Min. Width: 2.96
Max. Width: 0
Peak Width: 2
RT Min. (min): 1.00
Smooth: 0
ScanRate: 2.00
Area: 24000
Height: 4314
Start Time: 2.96
End Time: 2.96
Integration Width: 0.30
Resolution Time: 2.30
Integration Type: A - 50

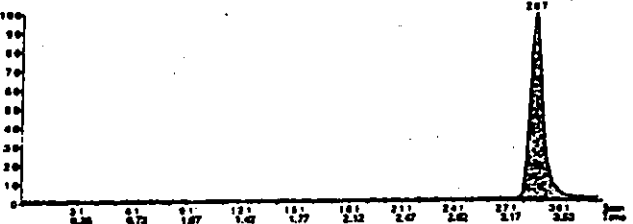


Figure 3. Standard: 4.0 ng/ml - RP 26109, RP 30228, RP 32596

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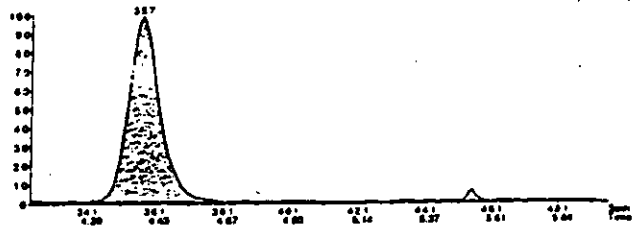
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Comments: No Comments

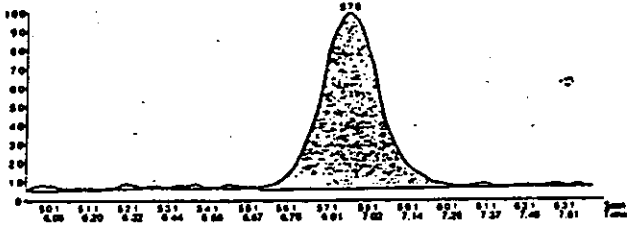
00|000000 Iprodione related000 Mon, Jun 8, 1998 17:40
 4 ng/ml MSX

7.76 in 3 peaks
 No Internal Standard
 Use Area
 2.100 MPAL 171 mass
 242.1--42.0
 Noise Thresh: 5.0
 Quant Thresh: 1.0
 Min. Width: 0
 Max. Width: 1.5
 Base Width: 100
 RT Min. (secs): 2.0
 Smooth: 2
 Expected RT: 4.40
 Area: 13309
 Height: 1764
 Start Time: 4.21
 End Time: 4.56
 Integration Width: 0.44
 Resolution Time: 4.20
 Integration Type: A - 00



00|000000 Iprodione related000 Mon, Jun 8, 1998 17:40
 4 ng/ml MSX

7.76 in 3 peaks
 No Internal Standard
 Use Area
 3.100 MPAL 146 mass
 224.1--42.0
 Noise Thresh: 4.0
 Quant Thresh: 2.0
 Min. Width: 0
 Max. Width: 1.0
 Base Width: 100
 RT Min. (secs): 2.0
 Smooth: 2
 Expected RT: 7.10
 Area: 21007
 Height: 1944
 Start Time: 6.72
 End Time: 7.26
 Integration Width: 0.52
 Resolution Time: 6.97
 Integration Type: A - 00



00|000000 Iprodione related000 Mon, Jun 8, 1998 17:40
 4 ng/ml MSX

7.76 in 3 peaks
 No Internal Standard
 Use Area
 1.250 MPAL 226 mass
 162.1--127.1
 Noise Thresh: 5.0
 Quant Thresh: 2.0
 Min. Width: 0
 Max. Width: 2
 Base Width: 100
 RT Min. (secs): 2.0
 Smooth: 0
 Expected RT: 3.40
 Area: 16207
 Height: 2481
 Start Time: 3.20
 End Time: 3.56
 Integration Width: 0.26
 Resolution Time: 3.27
 Integration Type: A - 00

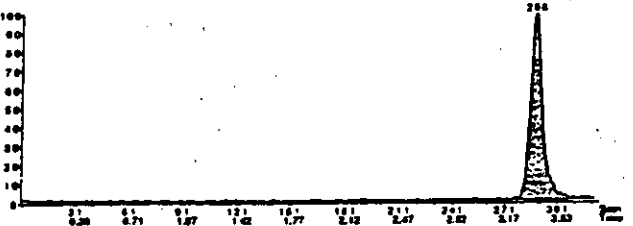


Figure 4. Standard: 2.0 ng/ml - RP 26109, RP 30228, RP 32596

MacQuan, version 1.5

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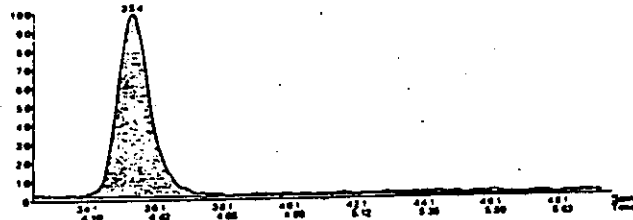
Printed: Tue, Jun 16, 1998 17:21

Calibration File: cal_june98_posneg Path: Office G3:ORJS: Iprodione Water: CAS Iprod Water:june98:98june08:

Comments: No Comments

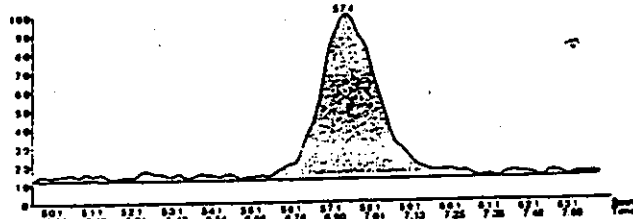
08|060007 Iprodione related007 Mon, Jun 8, 1998 17:48
2 ng/ml MSX

772 in 3 peaks
No Internal Standard
Use Area
2 1.00 0.000, 171 scans
242 1.00 0.000
Name: Iprodione
Chrom. Time: 3.07
Min. Width: 0.15
Max. Width: 1.00
RT Min. (sec): 2.90
Summit RT: 3.07
Area: 6566
Height: 823
Start Time: 0.10
End Time: 4.52
Integration Width: 0.50
Retention Time: 3.04
Integration Type: A - 00



08|060007 Iprodione related007 Mon, Jun 8, 1998 17:48
2 ng/ml MSX

772 in 3 peaks
No Internal Standard
Use Area
2 1.00 0.000, 148 scans
228 1.00 0.000
Name: Iprodione
Chrom. Time: 4.00
Min. Width: 0.15
Max. Width: 1.00
RT Min. (sec): 3.80
Summit RT: 4.00
Area: 10630
Height: 900
Start Time: 0.72
End Time: 7.20
Integration Width: 0.50
Retention Time: 4.02
Integration Type: A - 00



08|060007 Iprodione related007 Mon, Jun 8, 1998 17:48
2 ng/ml MSX

772 in 3 peaks
No Internal Standard
Use Area
1 2.70 0.000, 328 scans
122 1.00 0.000
Name: Iprodione
Chrom. Time: 2.00
Min. Width: 0.15
Max. Width: 1.00
RT Min. (sec): 1.80
Summit RT: 2.00
Area: 9902
Height: 1032
Start Time: 0.24
End Time: 3.31
Integration Width: 0.27
Retention Time: 2.06
Integration Type: A - VV

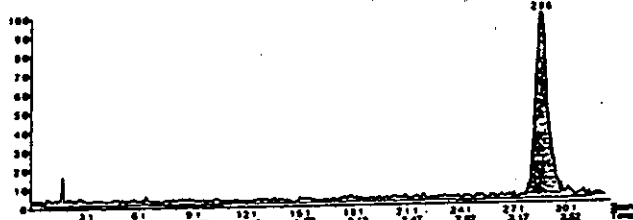


Figure 5. Standard Calibration Curve for RP 26019

MacQuan, version 1.5

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Printed: Tue, Jun 16, 1998 17:22

Calibration File: cal_june98_posneg Path: Office G3:0RJS: Iprodione Water: CAS Iprod Water:june98:98june98:

Comments: No Comments

Iprodione 243.1->42.0 No Internal Standard
Linear

Intercept = -57.659
Slope = 3296.779
Correlation Coeff. = 0.998

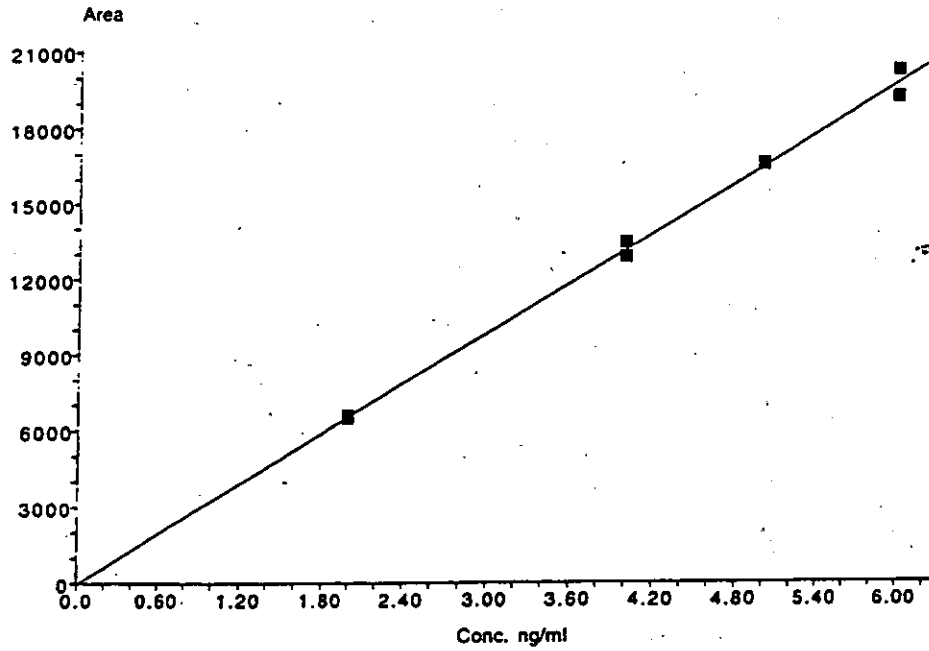


Figure 6. Standard Calibration Curve for RP 30228

MacQuan, version 1.5

Page 1 of 1

Printed: Tue, Jun 16, 1998 17:22

Calibration File: cal_june98_posneg Path: Office G3:0RJS: Iprodione Water: CAS Iprod Water:june98:96june98:

Comments: No Comments

RP30228 328.1->141.0 No Internal Standard

Linear

Intercept = -1021.486

Slope = 5663.843

Correlation Coeff. = 0.992

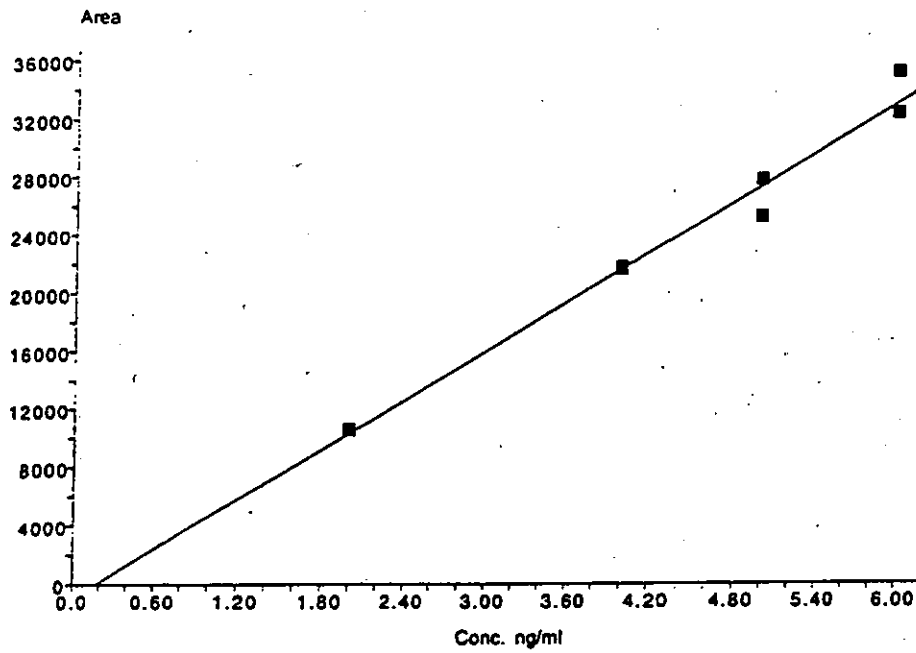


Figure 7. Standard Calibration Curve for RP 32596

MacQuan, version 1.5

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Printed: Tue, Jun 16, 1998 17:22

Calibration File: cal_june08_posneg Path: Office G3:ORJS: Iprodione Water: CAS Iprod Water:june98:98june08:

Comments: No Comments

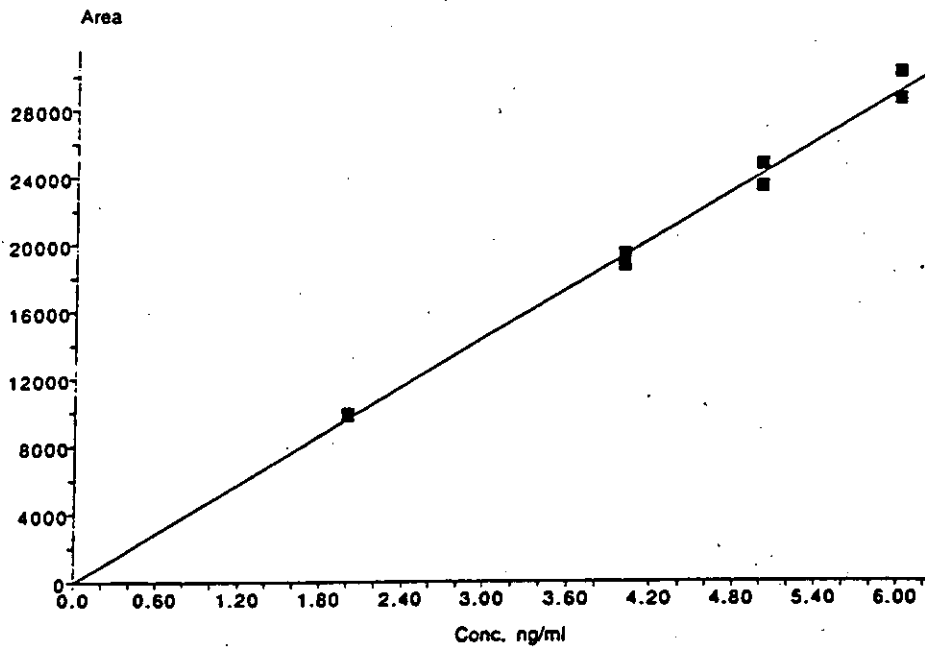
32596 162.1->127.1 No Internal Standard

Linear

Intercept = -25.450

Slope = 4841.314

Correlation Coeff. = 0.997



B. Results Tables

RP 26019 Results Table

MacQin, version 1.3
 Project: Test, Jun 18, 1998 17:41
 Calibration File: cal June98.pctmg Path: Office G3,DRJ5 Iprodione Water: CAS and Water June98-98June08
 Comments: No Comments

Iprodione
 No Internal Standard
 243.1142 0
 Linear
 Intercept = -57.653
 Slope = 3295.779
 Correlation Coeff. = 0.998
 Use Area

Element	Element	Sample Desc.	Amount	Conc. Conc.	Conc.	BL	Area	Height
98Jun08001	CC	20 ng/ml MIX	96.212	19.242	20.000	4.49	63360	7917
98Jun08002	CC	20 ng/ml MIX	96.292	19.256	20.000	4.40	63433	8272
98Jun08003	CC	20 ng/ml MIX	102.786	20.557	20.000	4.36	67713	8643
98Jun08004	Standard	6 ng/ml MIX	102.326	6.140	6.000	4.36	20183	2539
98Jun08005	Standard	3 ng/ml MIX	100.741	5.637	3.000	4.36	16548	2156
98Jun08006	Standard	4 ng/ml MIX	102.046	4.062	4.000	4.39	13399	1768
98Jun08007	Standard	2 ng/ml MIX	100.337	2.007	2.000	4.34	9358	823
98Jun08008	CC	681 ulc	n/a	n/a	0.0	n/a	n/a	n/a
98Jun08009	CC	681 M	109.396	3.469	3.000	4.34	17973	2213
98Jun08010	CC	682	93.936	4.697	3.000	4.33	13427	1918
98Jun08011	CC	683	93.996	4.790	3.000	4.37	15700	1945
98Jun08013	CC	BLANK	n/a	n/a	0.0	n/a	n/a	n/a
98Jun08019	CC	LCSFK	96.436	4.822	3.000	4.33	15838	2002
98Jun08020	CC	20 ng/ml MIX	98.049	19.610	20.000	4.37	63591	8197
98Jun08021	Standard	6 ng/ml MIX	97.060	3.824	6.000	4.35	19141	2427
98Jun08022	Standard	3 ng/ml MIX	106.315	3.016	3.000	4.36	16478	2074
98Jun08023	Standard	4 ng/ml MIX	97.818	3.913	4.000	4.39	12842	1649
98Jun08024	Standard	2 ng/ml MIX	99.139	1.963	2.000	4.36	6478	881

RP 30228 Results Table

MacQuan version 1.5
 Printed: Tue, Jun 16, 1998 17:41
 Calculation File: Z:\0000\jonesq Path: Office G:\PLS\Iprodione Water CAS\prod Water\prod 98\RPAC00
 Comments: No Comments

RP30228
 No Internal Standard
 328.1 -> 141.0
 Linear
 Intercept = -1021.486
 Slope = 5643.843
 Correlation Coeff = 0.992
 Use Area

Sample	Filter	Sample Desc	Accuracy	Calc Conc	Conc	R.T.	Area	Height
98jun08001	CC	20 ng/ml MIX	103.141	20.828	20.000	7.16	115814	9892
98jun08002	CC	20 ng/ml MIX	106.052	21.210	20.000	6.96	118111	10377
98jun08003	CC	20 ng/ml MIX	102.760	20.532	20.000	6.96	115382	9668
98jun08004	Standard	6 ng/ml MIX	105.975	6.358	6.000	6.95	34992	3080
98jun08005	Standard	5 ng/ml MIX	101.423	5.071	5.000	6.93	27701	2411
98jun08006	Standard	4 ng/ml MIX	100.279	4.011	4.000	6.97	21897	1840
98jun08007	Standard	2 ng/ml MIX	102.049	2.041	2.000	6.93	10538	908
98jun08008	CC	681 unc	n / s	n / s	0.0	n / s	n / s	n / s
98jun08009	CC	681 M	105.235	5.282	5.000	6.96	28780	2370
98jun08010	CC	682	75.798	3.790	5.000	6.91	20444	1751
98jun08011	CC	683	83.524	4.178	5.000	6.97	22832	2017
98jun08012	CC	BLANK	n / s	0.275	0.0	7.00	539	50
98jun08018	CC	LCSPK	93.071	4.654	5.000	6.93	25335	2226
98jun08020	CC	20 ng/ml MIX	90.794	18.159	20.000	6.98	101827	8643
98jun08021	Standard	6 ng/ml MIX	87.877	5.873	6.000	6.93	32240	2820
98jun08022	Standard	5 ng/ml MIX	92.385	4.619	5.000	6.98	25141	2338
98jun08023	Standard	4 ng/ml MIX	98.775	3.991	4.000	6.99	27583	1947
98jun08024	Standard	2 ng/ml MIX	101.787	2.035	2.000	6.96	10506	945

RP 32596 Results Table

MacQuant, version 1.3
 Printed: Tue, Jun 16, 1998 17:41
 Calibration File: C:\JUN98\jprodmg.Pac; Path: Office G3-ORLIS; Iprodione Water; CAS Used: Water; 45593; 26 Jun 98
 Comments: No Comments

32596
 No Internal Standard
 162.1 -> 127.1
 Linear
 Intercept = -25.450
 Slope = 4841.314
 Correlation Coeff. = 0.997
 Use Area

ESIDNR	Element	Sample Desc.	ACTUAL	Cal. Conc.	Conc.	R ²	AREA	Height
98Jun08001	CC	20 ng/ml MIX	113.024	22.805	20.000	3.42	109411	18389
98Jun08002	CC	20 ng/ml MIX	107.788	21.558	20.000	3.37	104341	17913
98Jun08003	CC	20 ng/ml MIX	106.830	21.388	20.000	3.35	103414	17341
98Jun08004	Standard	8 ng/ml MIX	103.855	8.219	8.000	3.36	30064	5276
98Jun08005	Standard	3 ng/ml MIX	102.134	5.107	5.000	3.36	24898	4238
98Jun08006	Standard	4 ng/ml MIX	100.284	4.012	4.000	3.37	19387	3461
98Jun08007	Standard	2 ng/ml MIX	102.522	2.051	2.000	3.35	8902	1632
98Jun08008	CC	681 ml	n / a	n / a	0.0	n / a	n / a	n / a
98Jun08009	CC	681 ml	100.913	5.048	5.000	3.34	24402	4164
98Jun08010	CC	682	85.147	4.237	5.000	3.33	22386	3515
98Jun08011	CC	683	89.000	4.450	5.000	3.35	21318	3626
98Jun08013	CC	BLANK	n / a	n / a	0.0	n / a	n / a	n / a
98Jun08018	CC	LCSPK	90.808	4.340	5.000	3.33	21956	3629
98Jun08020	CC	20 ng/ml MIX	98.284	19.619	20.000	3.36	94956	18347
98Jun08021	Standard	8 ng/ml MIX	98.184	3.891	8.000	3.35	28485	4794
98Jun08022	Standard	5 ng/ml MIX	98.614	4.831	5.000	3.35	23261	3984
98Jun08023	Standard	4 ng/ml MIX	98.527	3.861	4.000	3.36	19069	3274
98Jun08024	Standard	2 ng/ml MIX	101.420	2.028	2.000	3.37	9795	1758

C. Chromatograms of Samples

Figure 8. Ground water (Clovis, Ca. utc)-
 Untreated Control

MacQuen, version 1.5

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Printed: Tue, Jun 16, 1998 17:40

Calibration File: cal_june08_posneg Path: Office G3:0RJS: Iprodione Water: CAS Iprod Water:june98:june08:

Comments: No Comments

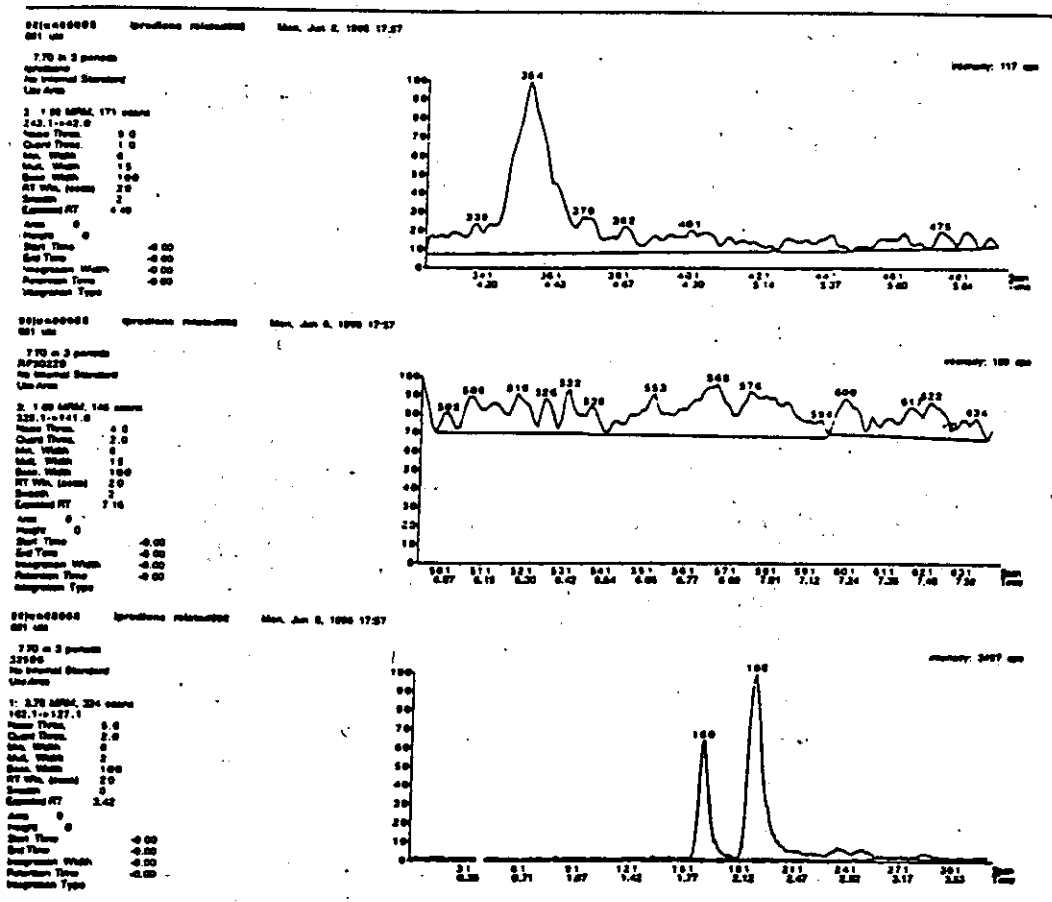


Figure 9. Ground water (Clovis, Ca.)-
50 ppt RP 26109, RP 30228, RP 32596

MacQuan, version 1.5

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Printed: Tue, Jun 16, 1998 17:32

Calibration File: cal_june98_posneg Path: Office G3:0RJS: Iprodione Water: CAS Iprod Water:june98:98june08:

Comments: No Comments

