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Study Title

A Method for the Determination of Folpan and Phthalimide Residues in Non-Oily Crops

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Study Director's Statement

The following report is a true and accurate record of the analytical work pertaining to the method for the determination of folpan and phthalimide in non-oily crops.

The work was commissioned by Makhteshim Chemical Manufacturers Ltd., the study's sponsor, and was performed in accordance with protocol no. 14/91.

The work was carried out under my supervision in the laboratories of Analyst Ltd., Kiryat Weizmann, Rehovot, Israel.

All original data, including notebooks, worksheets and chromatograms are available for inspection at the archives of Analyst Ltd.

H.M. Schlesinger, Ph.D.		4 march 1992
Name	Signature	Date

Good Laboratory Practice Statement

The study described in this final report was conducted in accordance with Good Laboratory Practice U.S. EPA Good Laboratory Practices, 40 CFR 160. To the best of my knowledge, there were no deviations from GLP which may have affected the integrity of the data.

Tishel 4/March/92

Dr. Moshe Tishel
Quality Assurance

Date

A Residue Analytical Method for Folpan and Phthalimide in Non Oily Crops

Analyst Report No. FP/15/91

Quality Assurance Statement

	Dates (Day, Month, Year)	
	Inspection	Report to management and study director
PROTOCOL		
Protocol was reviewed on:	07/07/91	07/07/91
Amendment was reviewed on:	03/09/91	03/09/91
ANALYTICAL WORK		
Analytical work of this type of study, including procedures and data, were audited as follows:	20/08/91	20/08/91
	20/10/91	20/10/91
	15/11/91	15/11/91
	20/12/91	20/12/91

This report, including its raw data, has been reviewed by Analyst Ltd. Quality Assurance Unit according to S.O.P. 0005. This report has been found to be a true and accurate presentation of the analytical work performed and of the results obtained.

This review was completed on: 12/1/92.

Tishel

Dr. Moshe Tishel
Quality Assurance

4/March/92

Date

1. Summary

A method is presented for the determination of folpan and phthalimide residues in non-oily crops including lettuce, tomatoes, melons, apples, squash, potatoes, grapes, and strawberries.

Samples are chopped up and extracted with ethyl acetate followed by clean-up on a Florisil column for folpan analysis or by partition between n-hexane and phosphate buffer for phthalimide analysis.

Samples extracts after clean-up are analyzed by gas liquid chromatography using a Megabore capillary column. A N_{63} electron capture detector is used in analyzing for folpan, and a thermionic specific detector in analyzing for phthalimide.

The methods are specific for folpan and phthalimide in the presence of 25 other pesticides.

The limits of detection are: 0.02 ppm for folpan, 0.1 ppm for phthalimide; the limits of quantitation are 0.05 and 0.2 ppm, respectively.

The recovery for folpan ranged from 76% to 112% and for phthalimide from 70% to 130%.

2. Chemicals

2.1. Folpan: Standard 99.2%; Lot 147-16/1; Lab. no. 553 supplied by sponsor.

2.2. Phthalimide: Standard 98%; Lot 1526-0; Lab. no. 554 supplied by sponsor.

Identification and purity are the responsibility of the sponsor.

3. Materials and Reagents

3.1.	Ethyl acetate	AR No. 0540515 Bio-Lab (Israel)
3.2.	Ethyl acetate	For residue analysis Merck No. 10972 (Germany)
3.3.	n-Hexane	HPLC grade No. C-2516 Lab-Scan (Ireland)
3.4.	Dichloromethane	For residue analysis Merck No. 6054 (Germany)
3.5.	Methanol	HPLC grade No. 9093-02 Baker (USA)
3.6.	Acetone	For residue analysis Merck No. 12 (Germany)
3.7.	Water	HPLC grade Purified by Nanopure Apparatus, Barnstead (USA
3.8.	Orthophosphoric acid	AR Merck No. 573 (Germany)
3.9.	Sodium sulfate, anhydrous	For synthesis, Merck No. 822286 (Germany)
3.10.	Sodium hydroxide	AR Merck No. 6482 (Germany)
3.11.	Florisil Bond Elute Cartridge	Varian No. 1211-3049 (USA)
3.12.	Buffer phosphate	0.2M pH 7.0
3.13.	Elution solution	1% methanol in dichloromethane

4. Apparatus

4.1.	Waring Blender	(USA)
4.2.	Food chopper	Magimix Food Processor (France)
4.3.	Rotary flask evaporator	Büchi Rotovapor (Switzerland)
4.4.	Separatory funnels	125 and 250 ml equipped with teflon stoppers
4.5.	Filter paper	Whatman No. 1
4.6.	Usual laboratory glassware	(Class A)
4.7.	Gas liquid chromatographs	see section 5.1.3, 5.2.3

5. Analytical Methods

5.1. Folpan

5.1.1. Sample preparation and clean-up

About 500g of sample are chopped in a food processor. 25 grams are transferred to the blender to which 100g sodium sulfate, 220 mL ethyl acetate and 2 mL of o-phosphoric acid are added. The mixture is blended for 2 minutes and is then passed through a Whatman No. 1 filter. The blender and filter are rinsed with 30-40 mL ethyl acetate and the final solution volume is brought to 250 mL.

Fifty (50) mL of the filtered solution is transferred to a 125 mL separatory funnel. 10 mL of acidified water (0.75% v/v phosphoric acid) is added and the funnel is shaken for 1 minute. The layers are allowed to separate and the lower aqueous phase is discarded.

The organic phase is passed through sodium sulfate into a 100 round bottomed flask and the ethyl acetate is evaporated at 45°C under reduced pressure.

The residue is taken in 4-10 mL n-hexane, depending on the expected folpan concentration. Two mL of the solution is passed through a Florisil column which has been first pre-washed with 1 mL hexane. The column is washed with 6 mL hexane. Next, the column is washed with 5 mL dichloromethane containing 1% (v/v) methanol which will elute any folpan.

The eluate is evaporated to dryness, taken up in 1-50 mL (depending on residue level) hexane for injection into the GLC system.

5.1.2. Standard curve preparation

A stock solution containing 500 ppm folpan (25 mg/50 ml) in acetone is prepared. The stock solution is diluted with hexane to prepare the following working solutions: 0.025; 0.05; 0.10; 0.20; 0.30; and 0.50 ppm.

5.1.3. Gas chromatography

- Instrument: Perkin Elmer model Autosystem equipped with a Ni_{63} electron capture detector and an autosampler-autoinjector.

- Column: Megabore, 30 m, 0.53 mm ID, 1.5µm film thickness, DB-1, J&W No. 125-1032
- Temperature: Column: 200°C
  Injector: 210°C
  Detector: 300°C
- Carrier gas: Helium
- Head pressure: 7 PSI
- Makeup gas: Nitrogen
- Injector mode: Splitless
- Injection volume: 1 µl
- Integrator: Nelson Model 1020 (USA)

5.1.4. Calculations

The concentration of folpan was calculated by using the standard curve regression equation:

$$S = \frac{P - A}{B} \times D$$

where

S = Test substance residue amount in the tested crop (ppm=mg/kg)
P = Peak area as given in the GLC chromatograms
A = Constant value (intercept) of the regression equation
B = Slope value of the regression equation
D = Dilution/concentration factor

5.2. Phthalimide

5.2.1. Sample preparation and clean-up

About 500 g of sample are chopped in a food processor. 25 g are transferred to the blender of which 100g sodium sulfate, 220 mL ethyl acetate and 2 mL of o-phosphoric acid are added. The mixture is blended for 2 minutes and is then passed through a Whatman No. 1 filter. The blender and filter are rinsed with 30-40 mL ethyl acetate and the final solution volume is brought to 250 mL.

Fifty (50) mL of the filtered solution is transferred to a round bottomed flask and is evaporated to dryness. The residue is taken up in 50 mL n-hexane and transferred to a 125 mL separatory funnel. The flask is rinsed with 50 mL 0.2M phosphate buffer, pH 7.0 which are transferred to the separatory funnel. The funnel is shaken vigorously for about 1 minute and about 5 g sodium sulfate is added. The funnel is shaken again for 20 sec and the layers are allowed to separate. The aqueous phase is drained into a 250 mL separatory funnel. The hexane phase is reextracted with an additional 50 mL of phosphate buffer and is then discarded. Two mL of phosphoric acid are added to the combined aqueous phase which is extracted twice with 35 mL dichloromethane. The aqueous phase is discarded and the dichloromethane is passed through sodium sulfate into a

round bottomed flask and is evaporated to dryness at 45°C under reduced pressure. The residue is taken up in 1-4 mL (depending on residue level) ethyl acetate for injection into the GLC system.

5.2.2. Standard curve preparation

A stock solution containing 500 ppm phthalimide (25 mg/50 ml) in acetone is prepared. The stock solution is diluted with ethyl acetate to prepare the following working solutions: 0.25; 0.50; 1.0; 1.5; 2.0; and 3.0 ppm.

5.2.3. Gas chromatography

The study began with the Varian model 3700 GLC. However, most of the residue analysis was carried out with the new H.P. 5890 GLC instrument.

- Instrument: Varian model 3700 equipped with a thermionic specific detector and a model D-2000 Merck-Hitachi integrator. For columns and operating conditions, see H.P. model 5890.
- Instrument: Hewlitt Packard Model 5890-II equipped with a thermionic specific detector and an autosampler (autoinjector).
- Column: Megabore, 15 m, 0.53 mm ID, 1.0µm film thickness, DB-Wax, J&W No. 125-7012
- Temperature: Column - Programmed
  Initial 140°C, 2 min
  Prog. Rate 30°C/min
  Final 230°C, 7 min
  Injector: 250°C
  Detector: 260°C
- Carrier gas: Helium
- Makeup gas: Nitrogen
- Injector mode: Splitless
- Injection volume: 1 µl
- Integrator: Hewlett Packard Model 3396 (USA)

5.2.4. Calculations

The concentration of phthalimide was calculated by using the standard curve regression equation:

$$S = \frac{P - A}{B} \times D$$

where

S = Test substance residue amount in the tested crop (ppm=mg/kg)
P = Peak area as given in the GLC chromatograms
A = Constant value (intercept) of the regression equation
B = Slope value of the regression equation
D = Dilution/concentration factor

6. Method Validation

6.1. Folpan

6.1.1. Recovery

The recovery test was carried out by spiking clean control samples with known amounts of folpan. Samples were spiked at several levels of folpan including: limit of detection, limit of quantitation, and three more levels covering the folpan residues. The results as shown in tables no. I-VIII give an average recovery of 95%.

Table I. Recovery test results for folpan in lettuce

Folpan			
Lab ID	ppm spike	ppm found	% recovery
1	Control (0)	ND	—
2	Control (0)	ND	—
3	Control (0)	ND	—
4	0.020	0.025	125
5	0.020	0.020	100
6	0.020	0.025	125
7	0.050	0.05	100
8	0.050	0.05	100
9	0.050	0.05	100
10	0.25	0.23	92
11	0.25	0.22	88
12	0.25	0.23	92
531	5.00	5.25	105
532	5.00	4.73	95
533	5.00	4.32	86
534	50.0	40.6	81
535	50.0	43.0	86
536	50.0	45.8	92
mean			93
%RSD			7.7

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table II. Recovery test results for folpan in tomatoes

Folpan			
Lab ID	ppm spike	ppm found	% recovery
217	Control (0)	ND	—
218	Control (0)	ND	—
219	Control (0)	ND	—
220	0.020	0.012	60
221	0.020	0.014	70
222	0.020	0.015	75
223	0.050	0.04	80
224	0.050	0.04	80
225	0.050	0.04	80
226	0.25	0.28	112
227	0.25	0.29	116
228	0.25	0.29	116
229	2.50	2.50	100
230	2.50	2.64	106
231	2.50	2.67	107
mean			100
% RSD			16

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at, or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table III. Recovery test results for folpan in melons

Folpan			
Lab ID	ppm spike	ppm found	% recovery
112	Control (0)	ND	—
113	Control (0)	ND	—
114	Control (0)	ND	—
115	0.020	0.025	125
116	0.020	0.027	135
117	0.020	0.027	135
118	0.050	0.04	80
119	0.050	0.03	60
120	0.050	0.04	80
121	0.25	0.29	116
122	0.25	0.25	100
123	0.25	0.28	112
124	2.50	2.54	102
125	2.50	2.43	97
126	2.50	2.71	108
mean			95
% RSD			19

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table IV. Recovery test results for folpan in apples

Folpan			
Lab ID	ppm spike	ppm found	% recovery
157	Control (0)	ND	—
158	Control (0)	ND	—
159	Control (0)	ND	—
160	0.020	0.020	100
161	0.020	0.021	105
162	0.020	0.021	105
163	0.050	0.05	100
164	0.050	0.05	100
165	0.050	0.05	100
166	0.25	0.25	100
167	0.25	0.23	92
168	0.25	0.25	100
169	2.50	2.49	100
170	2.50	2.39	96
171	2.50	2.47	99
mean			99
% RSD			2.8

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table V. Recovery test results for folpan in squash

Folpan			
Lab ID	ppm spike	ppm found	% recovery
232	Control (0)	ND	—
233	Control (0)	ND	—
234	Control (0)	ND	—
235	0.020	0.024	120
236	0.020	0.017	85
237	0.020	0.022	110
238	0.050	0.04	80
239	0.050	0.04	80
240	0.050	0.03	60
241	0.25	0.29	116
242	0.25	0.25	100
243	0.25	0.23	92
244	2.50	2.15	86
245	2.50	2.38	95
246	2.50	2.31	92
mean			89
%RSD			17

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table VI. Recovery test results for folpan in potatoes

Folpan			
Lab ID	ppm spike	ppm found	% recovery
97	Control (0)	ND	—
98	Control (0)	ND	—
99	Control (0)	ND	—
100	0.020	0.024	120
101	0.020	0.025	125
102	0.020	0.019	95
103	0.050	0.05	100
104	0.050	0.05	100
105	0.050	0.05	100
106	0.25	0.19	76
107	0.25	0.19	76
108	0.25	0.19	76
109	2.50	2.46	98
110	2.50	2.55	102
111	2.50	2.43	97
mean			92
% RSD			13

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table VII. Recovery test results for folpan in grapes

Folpan			
Lab ID	ppm spike	ppm found	% recovery
247	Control (0)	ND	—
248	Control (0)	ND	—
249	Control (0)	ND	—
250	0.020	0.019	95
251	0.020	0.017	85
252	0.020	0.018	90
253	0.050	0.05	100
254	0.050	0.05	100
255	0.050	*	*
256	0.25	0.22	88
257	0.25	0.22	88
258	0.25	0.20	80
259	5.00	4.64	93
260	5.00	5.36	108
261	5.00	4.92	98
mean			94
% RSD			9.4

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

* Test failure

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table VIII. Recovery test results for folpan in strawberries

Folpan			
Lab ID	ppm spike	ppm found	% recovery
556	Control (0)	ND	—
557	Control (0)	ND	—
558	Control (0)	ND	—
559	0.020	0.025	125
560	0.020	0.035	175
561	0.020	0.026	130
562	0.050	0.05	100
563	0.050	0.04	80
564	0.050	0.04	80
565	0.25	0.22	88
566	0.25	0.24	96
567	0.25	0.28	112
568	2.50	2.53	101
569	2.50	2.66	106
570	2.50	2.37	95
mean			95
% RSD			12

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

6.1.2. Linearity

The linearity was tested by injecting a six (6) point calibration curve covering the range of 0.025 to 0.5 ppm. These concentrations when calculated to ng folpan/per injection shows a linearity between 0.025-0.5 ng, with a mean correlation coefficient of 0.995. The calibration curves data and results are given in tables IX and X and standard curve no. 1.

Table IX. Folpan standard curves

Standard curve date	Peak area						
	ppm 0.025	ppm 0.050	ppm 0.10	ppm 0.20	ppm 0.30	ppm 0.40	ppm 0.50
12.08.91	3549	8475	20466	38506	65195	85883	101373
02.09.91	2700	6007	12682	29161	47850	69985	87782
04.11.91	2194	4967	9968	25707	43191	64237	79853
14.11.91	5447	11663	22250	48574	77609	100808	108057
01.12.91	2246	6610	16134	40157	76023	100071	115102
10.12.91	1920	5345	11827	30388	51310	81612	106833

Table X. Linearity results for folpan

Standard curve data	Correlation coefficient (r)	Slope $x10^4$	Intercept $x10^3$
12.08.91	0.998	21.2	−1.4
02.09.91	0.998	18.1	−4.2
04.11.91	0.997	16.7	−4.8
14.11.91	0.991	23.2	1.4
01.12.91	0.995	25.3	−6.0
10.12.91	0.993	22.1	−8.4
mean	0.995	21.1	
% RSD	0.3	15	

6.1.3. Specificity

Control and spiked samples were analyzed as described in section 5. The control chromatograms did not show any significant peaks at the retention time for folpan. In addition, 25 pesticides that were included for regular field pest control have been tested. The results as shown in chromatogram 21 indicate that none of the 25 pesticides interfered with the analytical method.

The list of the pesticides is included.

- methamidophos
- dimethoate
- chlorpyrifos
- fenthion
- carbaryl
- pirimicarb
- deltamethrin
- cyhalothrin
- fluvalinate
- permethrin
- cypermethrin
- endosulfan
- buprofenzin
- clofentizine
- amitraz
- mancozeb
- chlorothalonil
- triforine
- carbendazim
- hexaconazole
- myclobutanil
- triadimenol
- penconazole
- iprodione
- vinclozolin

6.1.4. Precision

The day by day precision was calculated from the recovery test results run during the study period.

The results are expressed as the % relative standard deviation (RSD) of the recoveries at each level of spiking.

The precision results are as follows:

- 23% RSD for the limit of detection level
- 13% RSD for the limit of quantitation level
- 11% RSD for higher concentrations

6.1.5. Sensitivity

For all crops analyzed, the detection limit is 0.02 ppm (mg/kg) and the limit of quantitation is 0.05 ppm (mg/kg). These parameters were calculated from spiked and control samples analyzed as described in section 5. See chromatogram no. 23.

6.1.6. Stability under frozen conditions

The treated crops were stored at –18°C until they were analyzed. In order to determine the stability of folpan under storage conditions, representative crops were spiked with 1.0 ppm folpan and kept at –18°C for several months.

Test method

A stock solution of folpan in acetone containing 100 ppm (0.1 mg/ml) was prepared. 250µl of the folpan stock solution were added on top of 25g crop sample (5x50µl) spiking the sample with 1.0 ppm folpan. The acetone was dried at room temperature and the spiked samples were transferred into plastic bags for storage at –18°C.

At the end of the storage period, the samples (all 25 g) were analyzed according to the analytical procedure described in section 5.

The results as given in Table XI show a mean recovery of 77% after 7 months of storage at –18°C.

Table XI. Folpan storage stability results

Crop spiked	Storage time (months)	Replicate No.	Recovery %
lettuce	6	1	91
lettuce	7	2	87
lettuce	7	3	100
lettuce	7	4	112
potato	6	1	92
potato	7	2	58
potato	7	3	59
potato	7	4	76
tomato	6	1	68
tomato	7	2	78
tomato	7	3	63
tomato	7	4	81
melon	5	1	64
melon	6	2	68
melon	6	3	58
		mean	77
		% relative standard deviation	22

6.2. Phthalimide

6.2.1. Recovery

The recovery test was carried out by spiking clean control samples with known amounts of phthalimide. Samples were spiked at several levels of phthalimide including: limit of detection, limit of quantitation, and three more levels covering the phthalimide residues. The results as shown in tables no. XII-XIX give an average recovery of 85%.

Table XII. Recovery test results for phthalimide in lettuce

Phthalimide			
Lab ID	ppm spike	ppm found	% recovery
590	Control (0)	ND	—
591	Control (0)	ND	—
592	Control (0)	ND	—
593	0.10	0.10	100
594	0.10	0.13	130
595	0.10	0.11	110
596	0.20	0.24	120
597	0.20	0.21	105
597	0.20	0.19	95
599	1.00	0.94	94
600	1.00	*	*
601	1.00	90	90
602	2.00	1.59	80
603	2.00	1.76	88
604	2.00	1.72	86
mean			95
%RSD			13.3

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

* Test failure

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table XIII. Recovery test results for phthalimide in tomatoes

Phthalimide			
Lab ID	ppm spike	ppm found	% recovery
187	Control (0)	ND	—
188	Control (0)	ND	—
189	Control (0)	ND	—
190	0.10	0.072	72
191	0.10	0.073	73
192	0.10	0.070	70
193	0.20	0.15	75
194	0.20	0.14	70
195	0.20	0.17	85
196	1.00	0.66	66
197	1.00	0.73	73
198	1.00	0.77	77
199	2.00	1.53	77
200	2.00	1.52	76
201	2.00	1.50	75
mean			75
% RSD			7.0

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at, or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table XIV. Recovery test results for phthalimide in melons

Phthalimide			
Lab ID	ppm spike	ppm found	% recovery
127	Control (0)	ND	—
128	Control (0)	ND	—
129	Control (0)	ND	—
130	0.10	0.086	86
131	0.10	0.085	85
132	0.10	0.077	77
133	0.20	0.19	95
134	0.20	0.17	85
135	0.20	0.16	80
136	1.00	0.75	75
137	1.00	0.78	78
138	1.00	0.74	74
139	2.00	1.29	65
140	2.00	1.21	61
141	2.00	1.37	69
mean			76
% RSD			14

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table XV. Recovery test results for phthalimide in apples

Phthalimide			
Lab ID	ppm spike	ppm found	% recovery
142	Control (0)	ND	—
143	Control (0)	ND	—
144	Control (0)	ND	—
145	0.10	0.092	92
146	0.10	0.104	104
147	0.10	0.113	113
148	0.20	0.22	110
149	0.20	0.22	110
150	0.20	0.22	110
151	1.00	0.94	94
152	1.00	0.95	95
153	1.00	0.87	87
154	2.00	1.78	89
155	2.00	1.70	85
156	2.00	1.59	80
mean			96
% RSD			12

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table XVI. Recovery test results for phthalimide in squash

Phthalimide			
Lab ID	ppm spike	ppm found	% recovery
202	Control (0)	ND	—
203	Control (0)	ND	—
204	Control (0)	ND	—
205	0.10	0.107	107
206	0.10	*	*
207	0.10	0.108	108
208	0.20	0.18	90
209	0.20	0.18	90
210	0.20	0.22	110
211	1.00	0.84	84
212	1.00	0.84	84
213	1.00	0.84	84
214	2.00	1.68	84
215	2.00	1.78	89
216	2.00	1.94	97
mean			90
%RSD			9.5

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

* Test failure

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table XVII. Recovery test results for phthalimide in potatoes

Phthalimide			
Lab ID	ppm spike	ppm found	% recovery
82	Control (0)	ND	—
83	Control (0)	ND	—
84	Control (0)	ND	—
85	0.10	0.090	90
86	0.10	0.085	85
87	0.10	0.088	88
88	0.20	0.19	95
89	0.20	0.16	80
90	0.20	0.20	100
91	1.00	0.76	76
92	1.00	0.88	88
93	1.00	0.70	70
94	2.00	1.64	82
95	2.00	1.37	69
96	2.00	1.45	73
mean			81
% RSD			13

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table XVIII. Recovery test results for phthalimide in grapes

Phthalimide			
Lab ID	ppm spike	ppm found	% recovery
172	Control (0)	ND	—
173	Control (0)	ND	—
174	Control (0)	ND	—
175	0.10	0.107	107
176	0.10	0.102	102
177	0.10	0.118	118
178	0.20	0.26	130
179	0.20	0.19	95
180	0.20	0.17	85
181	1.00	0.78	78
182	1.00	0.89	89
183	1.00	0.79	79
184	2.00	1.55	78
185	2.00	1.61	81
186	2.00	1.58	79
mean			88
% RSD			19

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

Table XIX. Recovery test results for phthalimide in strawberries

Phthalimide			
Lab ID	ppm spike	ppm found	% recovery
541	Control (0)	ND	—
542	Control (0)	ND	—
543	Control (0)	ND	—
544	0.10	0.067	67
545	0.10	0.052	52
546	0.10	0.060	60
547	0.20	0.17	85
548	0.20	0.14	70
549	0.20	0.14	70
550	1.00	0.78	78
551	1.00	0.77	77
552	1.00	0.80	80
553	2.00	1.56	78
554	2.00	1.30	65
555	2.00	1.41	71
mean			75
% RSD			8.4

ND — Not detected: <0.02 ppm folpan, <0.1 ppm phthalimide.

Mean and % Relative Standard Deviation (% RSD) are calculated using samples spiked at or above the level of limit of quantitation (≥0.05 ppm for folpan and ≥0.2 ppm for phthalimide).

6.2.2. Linearity

The linearity was tested by injecting a six (6) point calibration curve covering the range of 0.25 to 3.0 ppm.

This concentration, when calculated to ng phthalimide per injection, shows a linearity between 0.25-3.0 ng, with a mean correlation coefficient of 0.998.

The calibration curves data and results are given in tables no. XX and XXI and standard curve no. 2.

Table XX. Phthalimide standard curves

Standard curve date	Varian Model 3700 (Peak height)					
	ppm 0.25	ppm 0.50	ppm 1.0	ppm 1.5	ppm 2.0	ppm 3.0
30.07.91	38	71	129	167	217	384
14.08.91	36	58	135	155	220	330
20.08.91	28	47	98	130	181	290
Standard curve date	**HP Model 5890 (Peak area)**					
	ppm 0.25	ppm 0.50	ppm 1.0	ppm 1.5	ppm 2.0	ppm 3.0
14.11.91	1019	2002	3826	5808	7723	11128
16.11.91	1054	1631	3239	5699	7422	10556
19.11.91	820	1414	2872	4351	5544	8526
01.12.91	916	1805	3625	5443	7599	10266
16.12.91	621	1377	2635	3995	5432	8146
22.12.91	731	1521	3234	4739	5834	8895

Table XXI. Linearity results for phthalimide

Standard curve data	Correlation coefficient (r)	Slope $x10^2$	Intercept
Varian Model 3700			
30.07.91	0.990	1.20	2.4
14.08.91	0.995	1.06	10.4
20.08.91	0.997	0.94	−0.4
mean	0.994	1.07	
% RSD	0.4	12	
HP Model 5890			
14.11.91	0.999	37.0	165
16.11.91	0.997	35.8	13
19.11.91	0.999	28.0	67
01.12.91	0.997	34.8	162
16.12.91	1.000	27.3	−51
22.12.91	0.999	29.4	121
mean	0.999	32.1	
% relative standard deviation	0.1	13	

6.2.3. Specificity

Control and spiked samples were analyzed as described in section 5. The control chromatograms did not show any significant peaks at the retention time for phthalimide. In addition, 25 pesticides that were included for regular field pest control have been tested. The results as shown in chromatogram 22 indicate that none of the 25 pesticides interfered with the analytical method.

The list of pesticides is given in section 6.1.3.

6.2.4. Precision

The day by day precision was calculated from the recovery test results run during the study period.

The results are expressed as the % relative standard deviation (RSD) of the recoveries at each level of spiking.

The precision results are as follows:

- 22% RSD for the limit of detection level
- 17% RSD for the limit of quantitation level
- 11% RSD for higher concentrations

6.2.5. Sensitivity

For all crops analyzed, the detection limit is 0.1 ppm (mg/kg) and the limit of quantitation is 0.2 ppm (mg/kg). These parameters were calculated from spiked and control samples analyzed as described in section 5.

6.2.6. Stability under frozen conditions

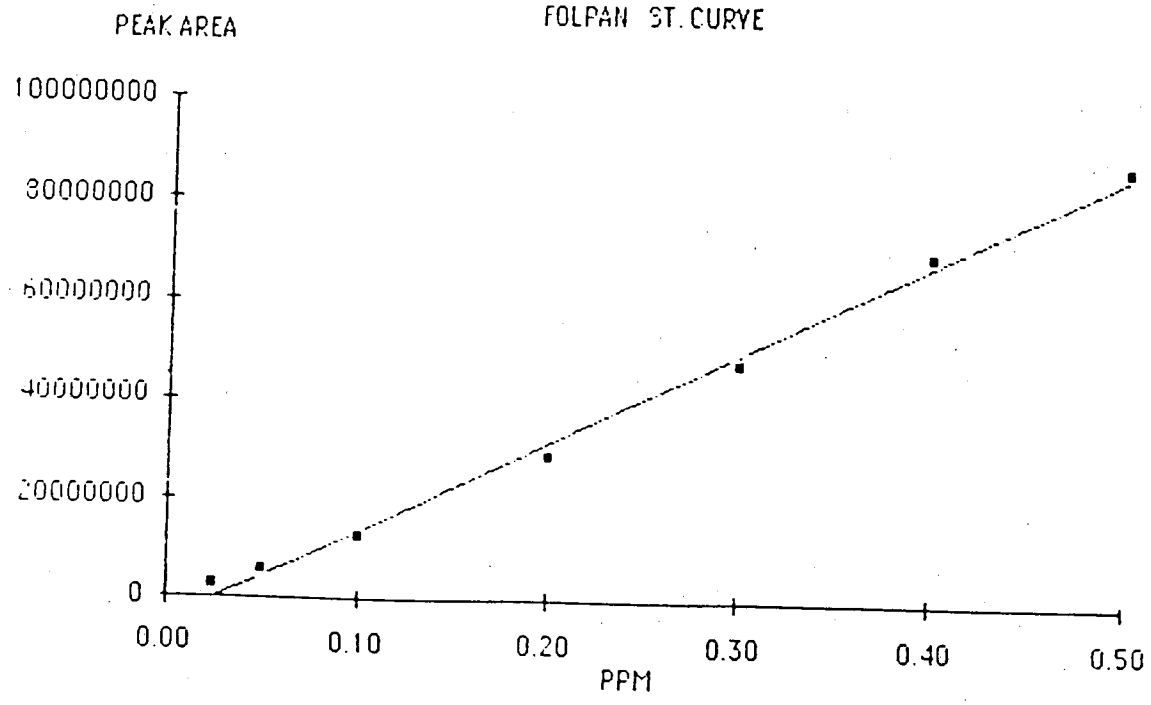
The treated crops were stored at –18°C until they were analyzed. In order to determine the stability of phthalimide under storage conditions, representing crops were spiked with 1.0 ppm folpan and kept at –18°C for several months.

The results as given in Table XXII show a mean recovery of 65% after 7 months of storage at –18°C.

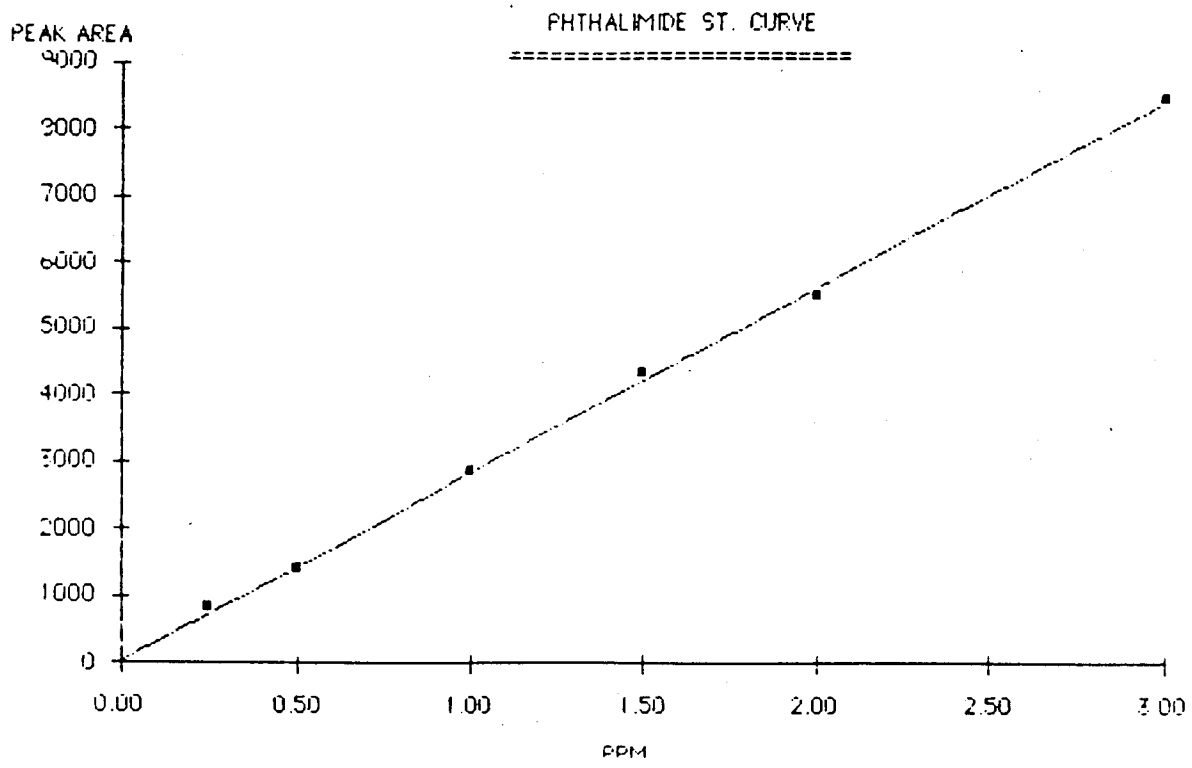
Table XXII. Phthalimide storage stability results for phthalimide

Crop spiked	Storage time (months)	Replicate No.	Recovery %
lettuce	6	1	76
lettuce	7	2	73
lettuce	7	3	71
lettuce	7	4	74
potato	6	1	57
potato	7	2	53
potato	7	3	49
tomato	6	1	67
tomato	7	2	61
tomato	7	3	63
melon	6	1	57
melon	7	2	67
melon	7	3	75
		mean	65
		% relative standard deviation	14

1. Folpan standard calibration curve containing 0.025-0.5 ppm (0.025-0.5 ng)



2. Phthalimide standard calibration curve containing 0.25-3.0 ppm (0.25-3 ng)



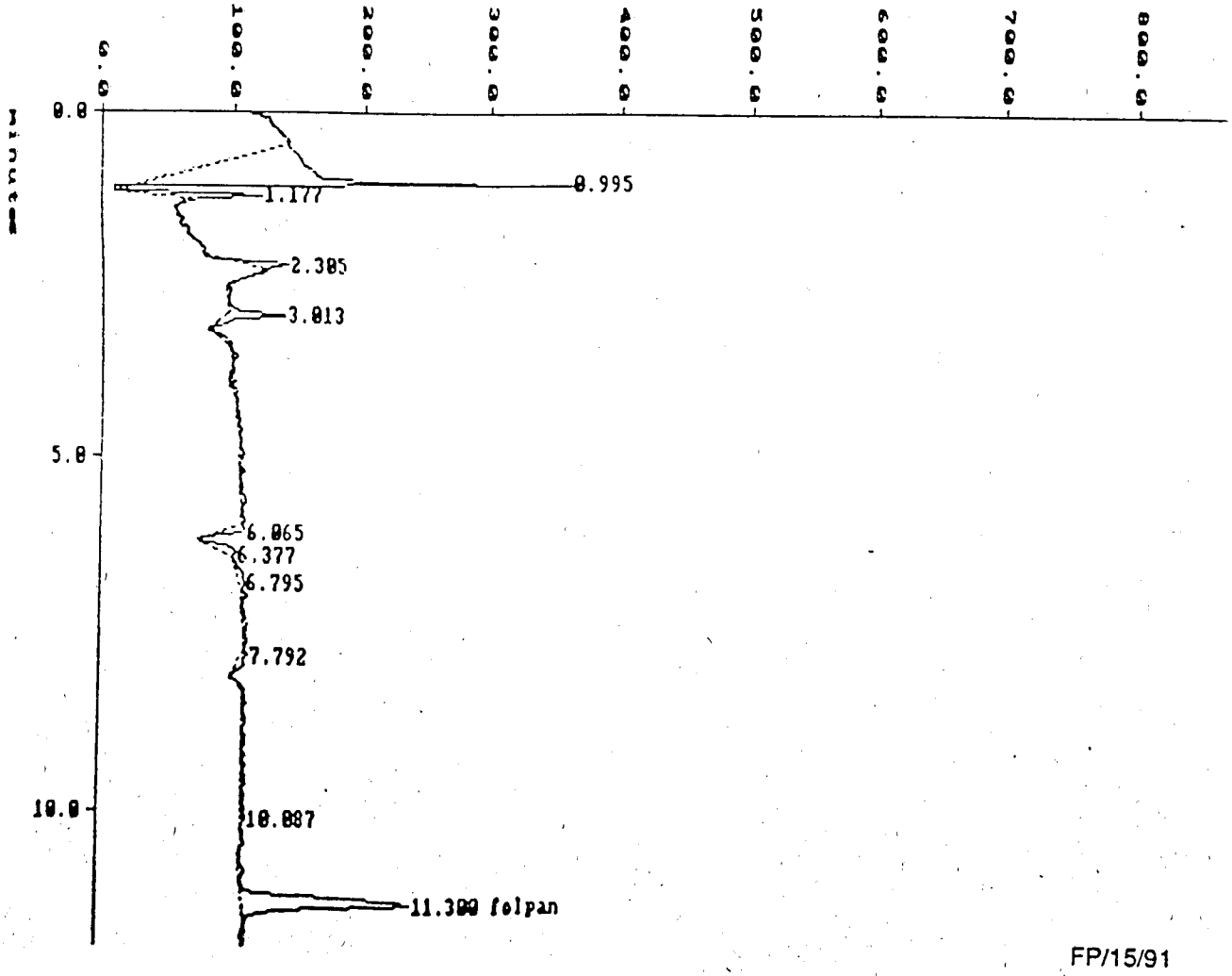
3. A standard solution containing 0.1 ppm folpan

File : FOLPA055.DB1 F 5T01
Run : 06 Type : Sample
Path : C:\CHROM
Collection : 11:02:24 Sep 02 1991 Method : YEHUDA [09:50:00 Aug 28 1991]
Integration: 11:02:24 Sep 02 1991 Method : YEHUDA [09:50:00 Aug 28 1991]
Report : 11:15:09 Sep 02 1991 Method : YEHUDA [09:50:00 Aug 28 1991]

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

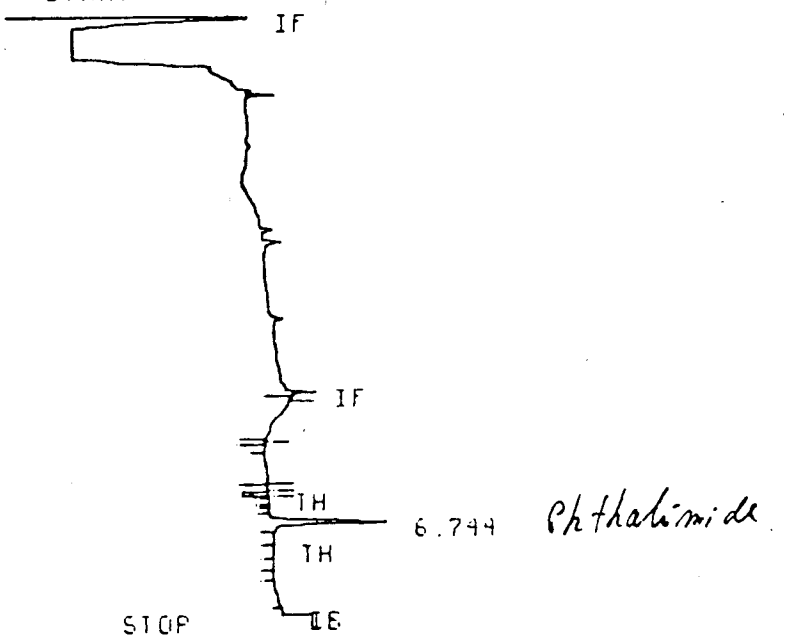
K'	Pk #	RT	Area	Height	ExpRT	Name
	10	11.300	12681500	131.300	11.200	folpan



4. A standard solution containing 1.0 ppm phthalimide

SE 1

RUN # 34 NOV 18, 1991 18:40:25
START



RUN# 34 NOV 18, 1991 18:40:25

SAMPLE# 26

AREA%

RT	AREA	TYPE	WIDTH	AREA%
6.744	2872	PB	.056	100.00000

TOTAL AREA= 2872
MUL FACTOR=1.0000E+00

5. Lettuce control for folpan analysis

Collection : 16:46:43 Sep 02 1991 Method : YEHUDA [09:50:00 Aug 28 1991]
Integration: 16:46:43 Sep 02 1991 Method : YEHUDA [09:50:00 Aug 28 1991]
Report : 16:59:29 Sep 02 1991 Method : YEHUDA [09:50:00 Aug 28 1991]

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

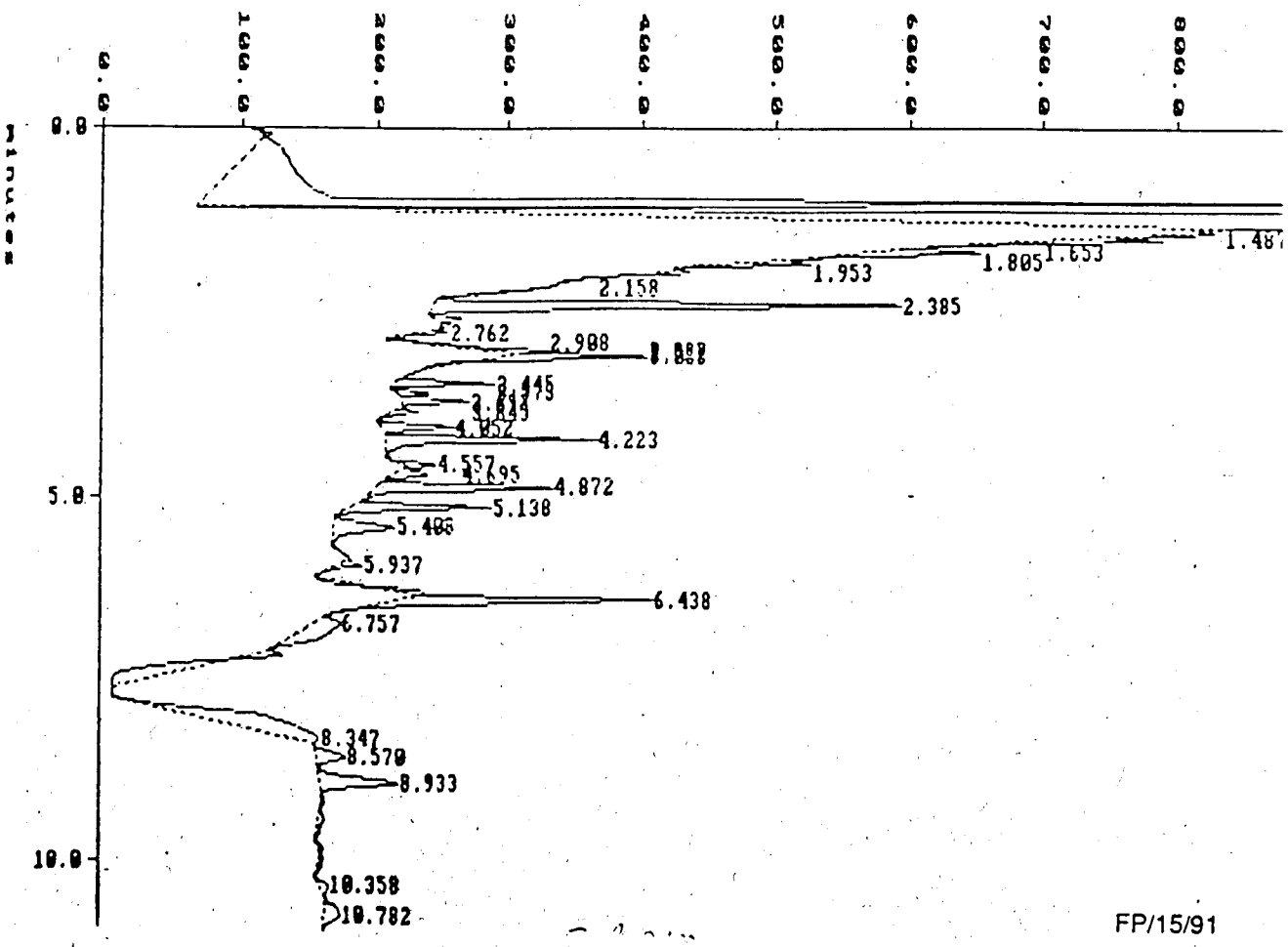
K'	Pk #	RT	Area	Height	ExpRT	Name
					11.200	folpan

0 Matched Components 0.00 % of Total Area
34 Unknown Peaks 100.0 % of Total Area

34 Peaks > Area Reject 2.55882e+0 Total Area

(FOLPA069.D01) mV

3



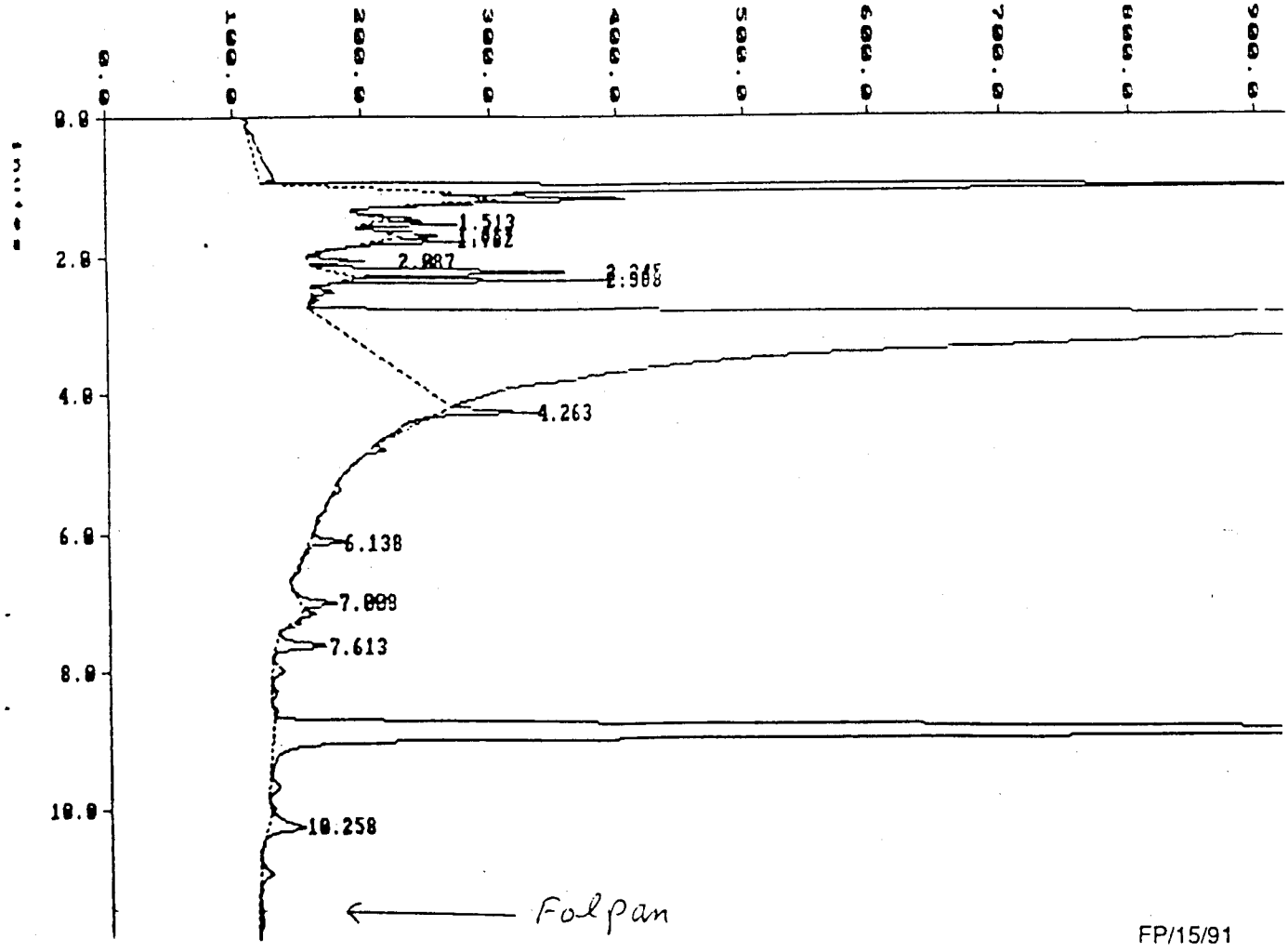
6. Grape control for folpan analysis

File : K____68.D01 524 247 HAIM
Run : 04 Type : Sample
Path : C:\CHROM
Collection : 17:29:45 Dec 17 1991 Method : YEHUDA [09:53:40 Dec 15 1991]
Integration: 17:29:45 Dec 17 1991 Method : YEHUDA [09:53:40 Dec 15 1991]
Report : 17:42:54 Dec 17 1991 Method : YEHUDA [09:53:40 Dec 15 1991]

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Pk #	RT	Area	Height	ExpRT	Name
					10.600	captan
					11.300	FOLPAN



7. Apple control for folpan analysis

File : W____23.281 F HAIM

Run : 23 158 Type : Sample

Path : C:\CHROM

Collection : 19:19:12 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]

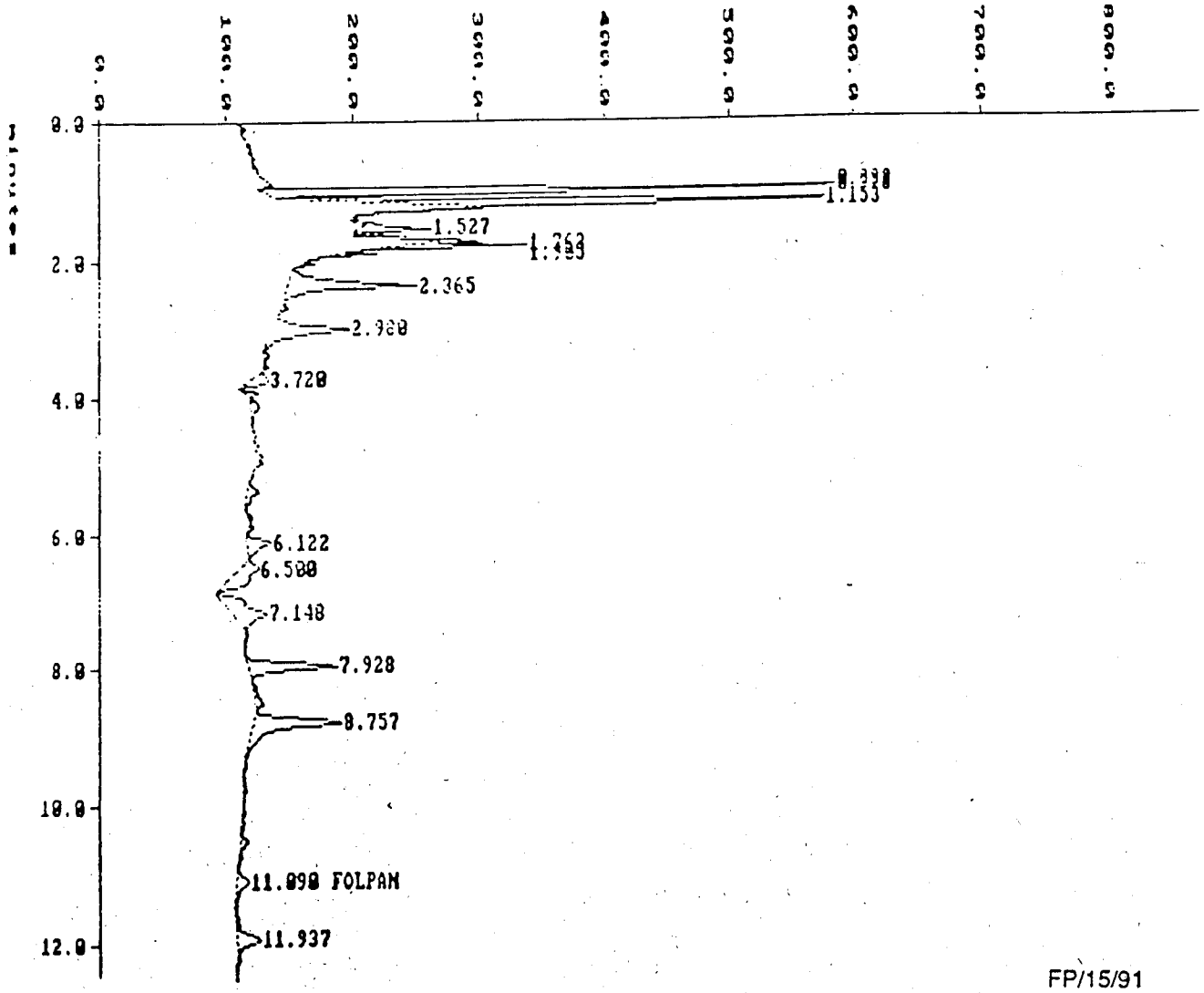
Integration: 19:19:12 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]

Report : 19:32:28 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Pk #	RT	Area	Height	ExpRT	Name
					10.630	CAPTAN
	15	11.090	1257638	10.474	11.200	FOLPAN



8. Squash control for folpan analysis

Collection : 23:19:37 Nov 19 1991 Method : YEHUDA [14:42:12 Nov 19 1991]
Integration: 23:19:37 Nov 19 1991 Method : YEHUDA [14:42:12 Nov 19 1991]
Report : 23:32:47 Nov 19 1991 Method : YEHUDA [14:42:12 Nov 19 1991]

Sample Wt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

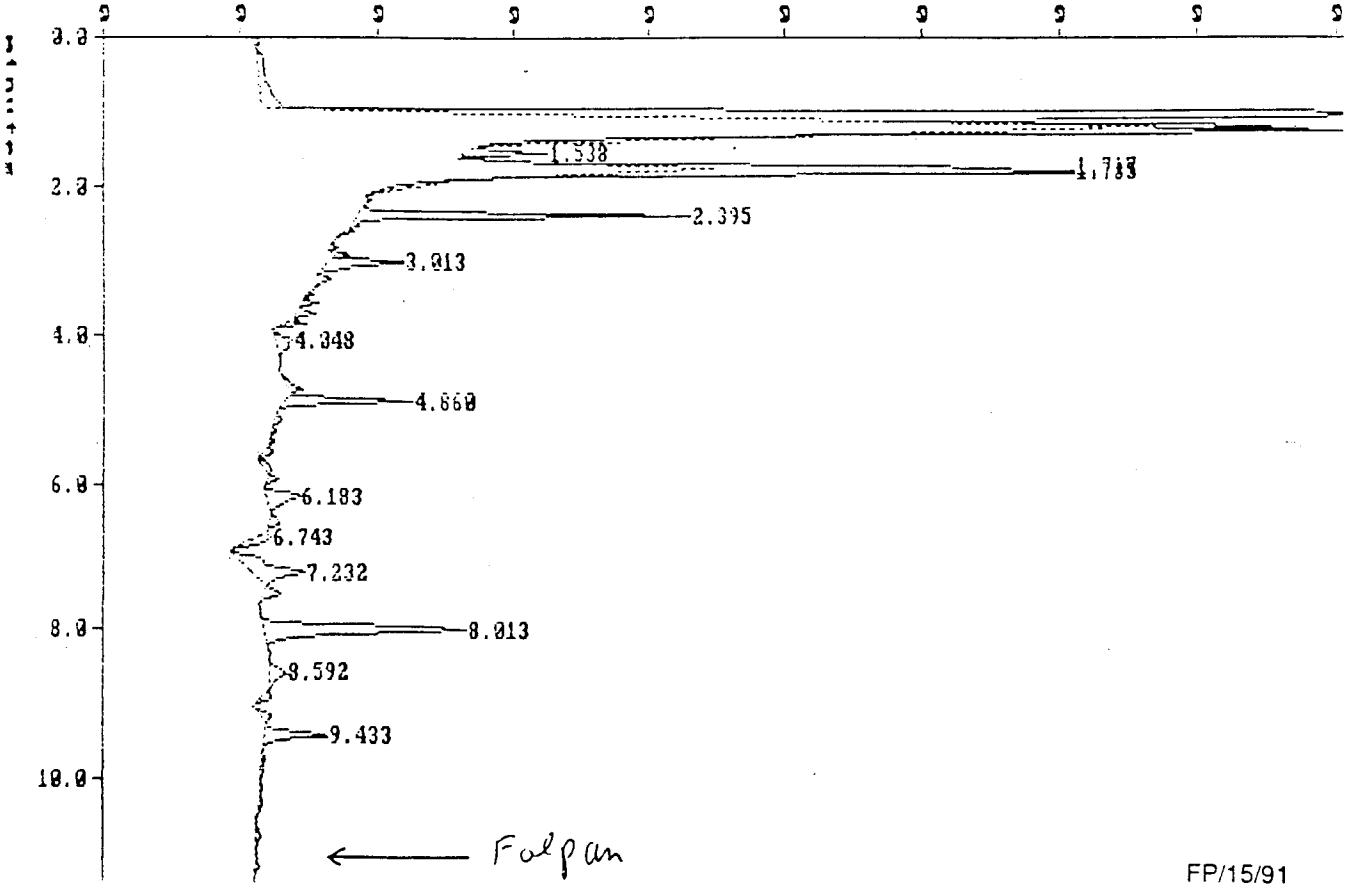
K'	Pk #	RT	Area	Height	ExpRT	Name
					10.600	CAPTAN
					11.200	FOLPAN

0 Matched Components 0.00 % of Total Area
17 Unknown Peaks 100.0 % of Total Area

17 Peaks : Area Reject 1.12735e+3 Total Area

(W____91.001) mV

233



9. Tomato control for folpan analysis

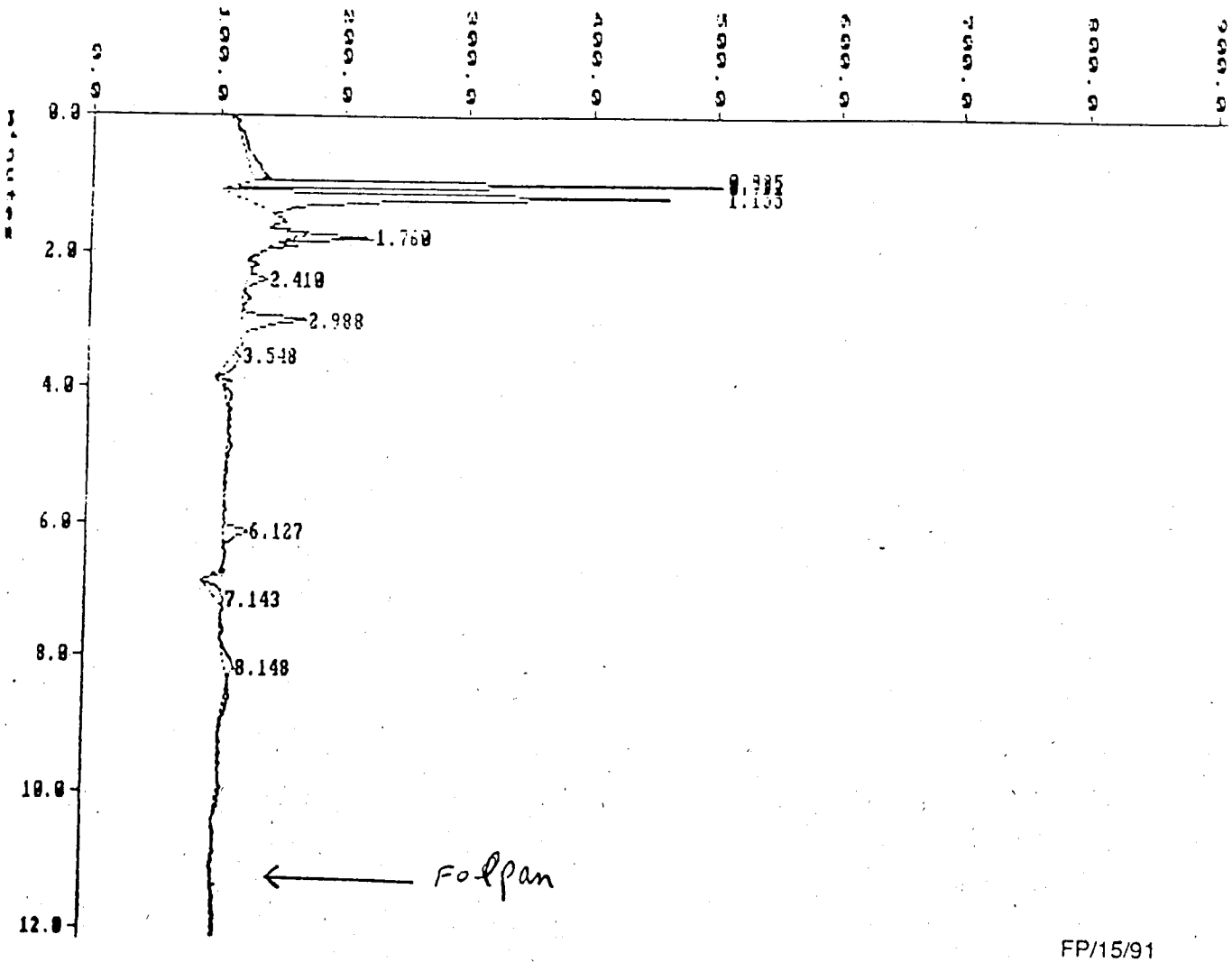
File : W____25.001 F 217 HAIM
Run : 25 Type : Sample
Path : C:\CHROM
Collection : 19:52:42 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]
Integration: 19:52:42 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]
Report : 20:05:59 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]

Sample Wt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

X'	Pk #	RT	Area	Height	ExpRT	Name
					12.520	CAPTAN
					11.200	FOLPAN

0



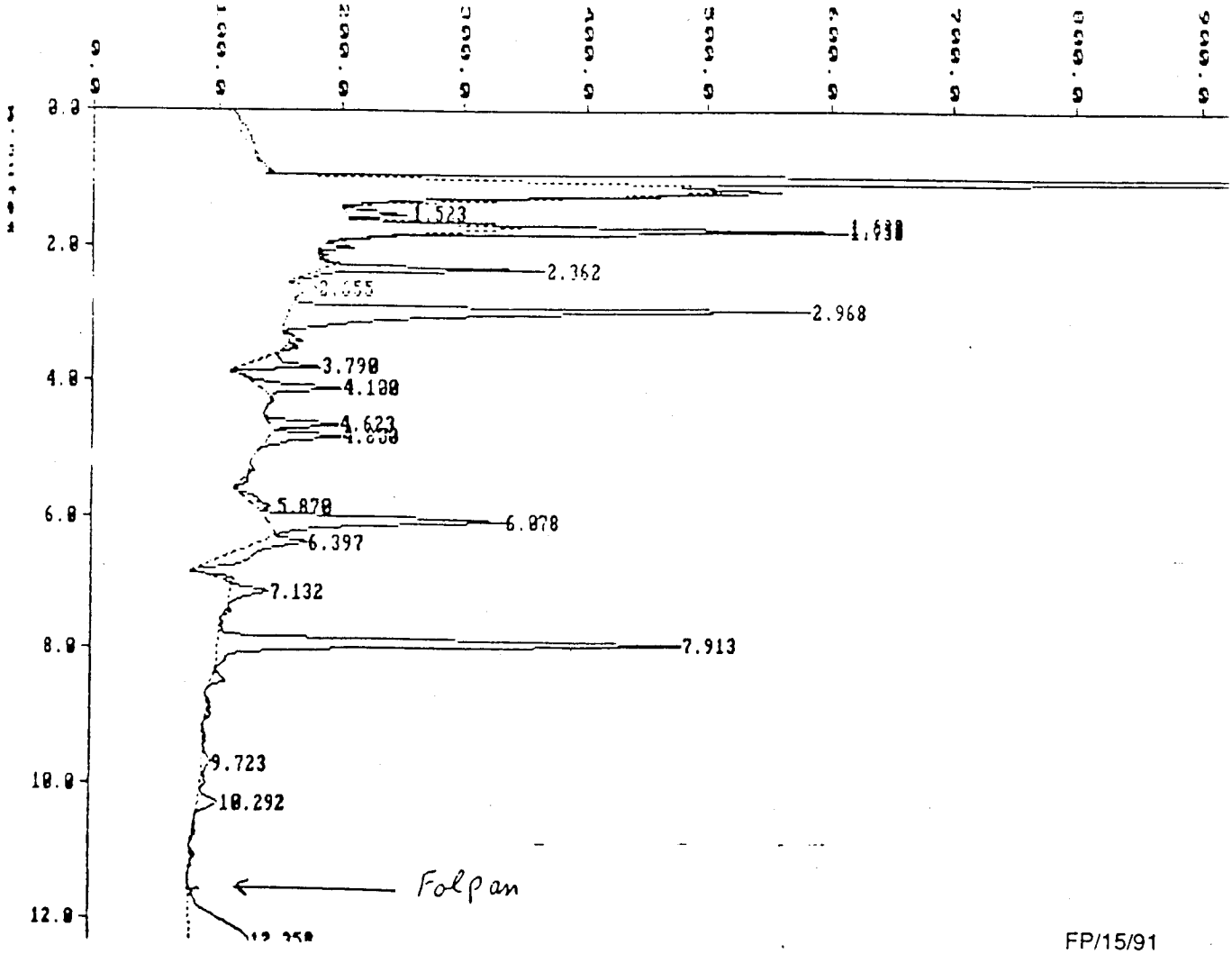
10. Potato control for folpan analysis

File : V____46.D01 st2ppm+3183 HAIM
Run : 46 99 Type : Sample
Path : C:\CHROM
Collection : 11:58:10 Nov 14 1991 Method : YEHUDA [11:16:30 Nov 10 1991]
Integration: 11:58:10 Nov 14 1991 Method : YEHUDA [11:16:30 Nov 10 1991]
Report : 12:11:19 Nov 14 1991 Method : YEHUDA [11:16:30 Nov 10 1991]

Sample Wgt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

Pk #	RT	Area	Height	ExpRT	Name
				10.500	CAPTAN
				11.200	FOLPAN

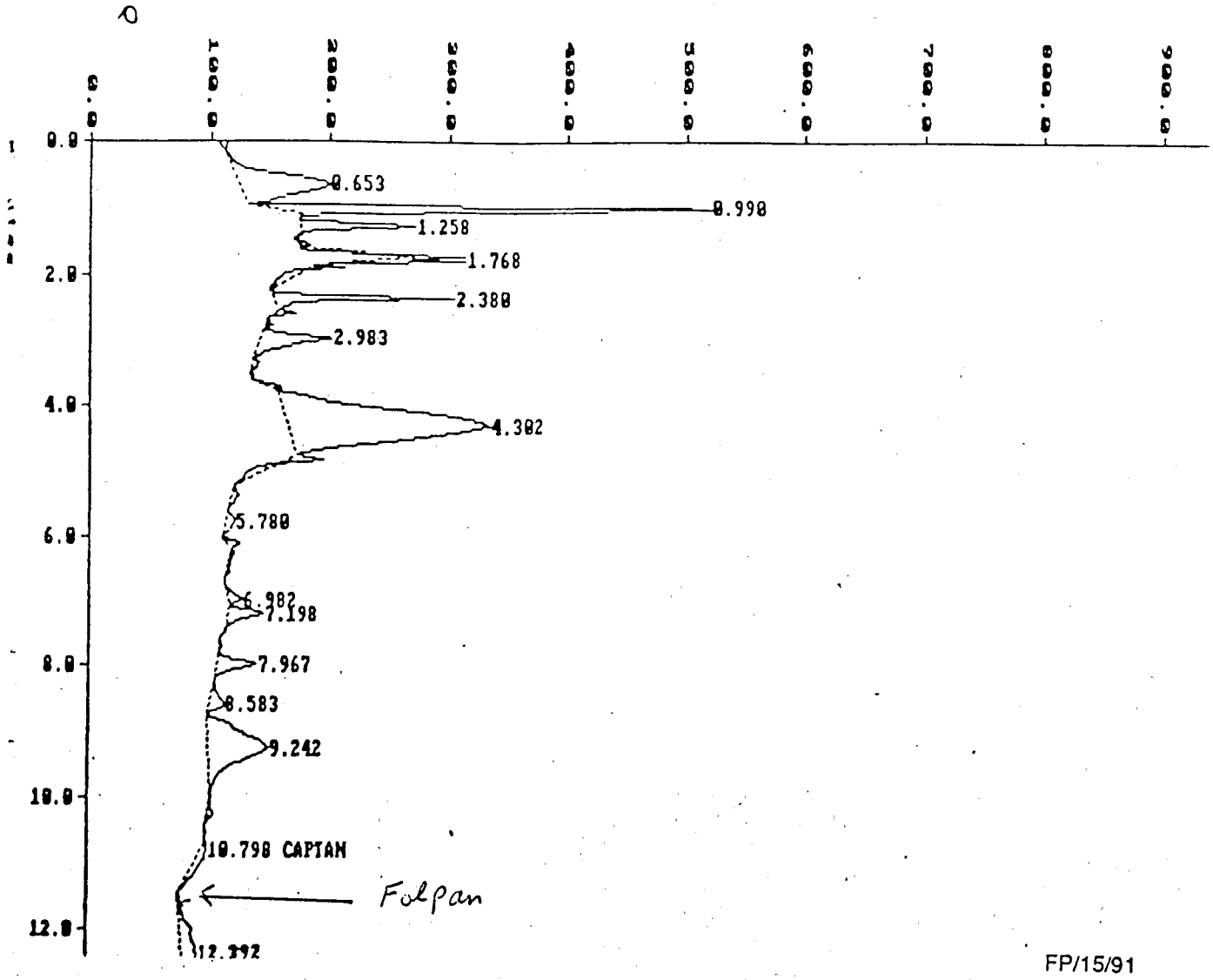


ile : F____11.DB1 F 114 HAIM
un : 11 Type : Sample
ath : C:\CHROM
ollection : 13:33:13 Dec 09 1991 Method : YEHUDA [10:46:18 Dec 09 1991]
ntegration: 13:33:13 Dec 09 1991 Method : YEHUDA [10:46:18 Dec 09 1991]
eport : 13:46:20 Dec 09 1991 Method : YEHUDA [10:46:18 Dec 09 1991]

ample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Pk #	RT	Area	Height	ExpRT	Name
	14	10.798	2146756	4.857	10.600	CAPTAN
					11.300	FOLPAN



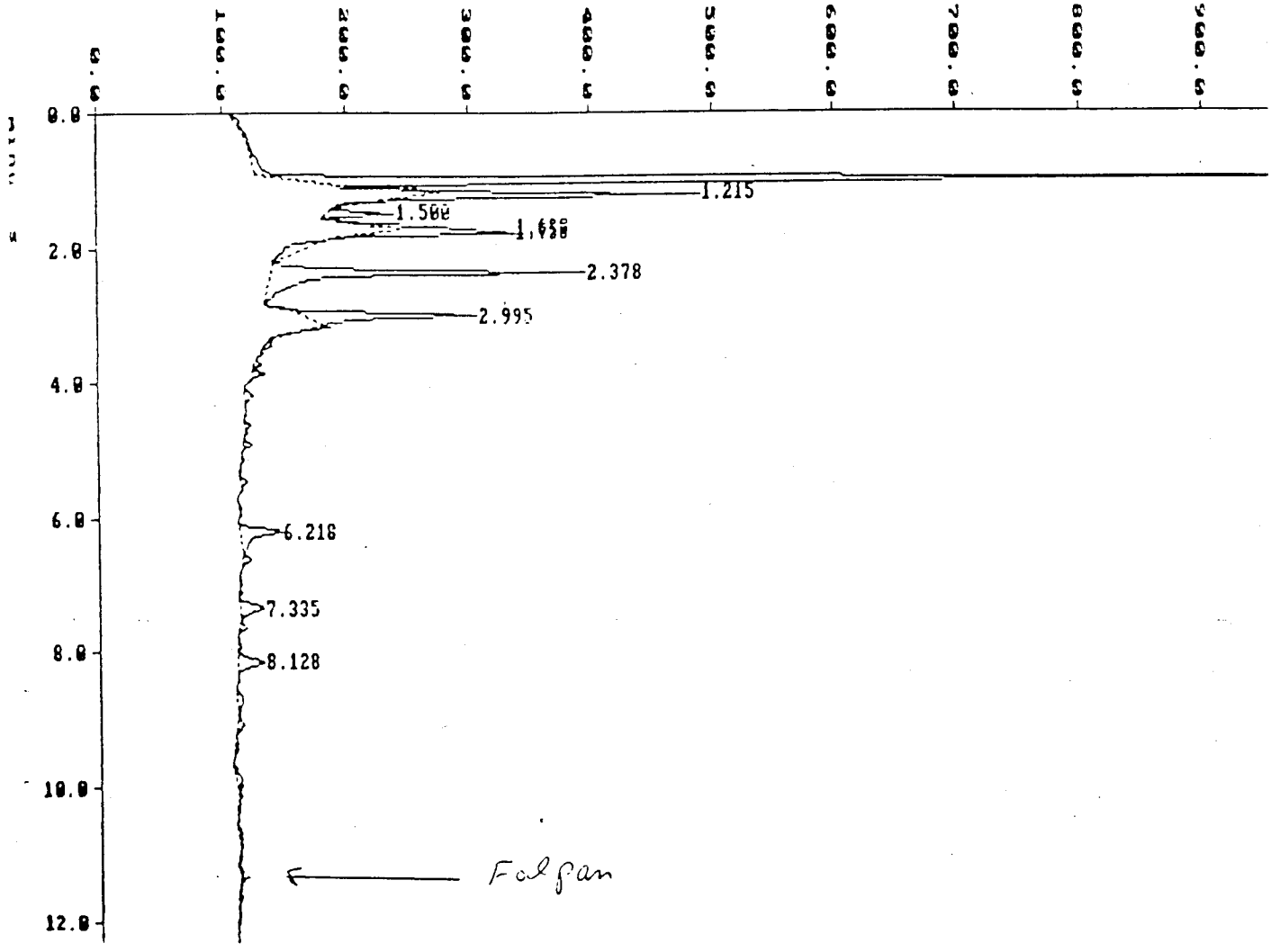
12. Strawberry control for folpan analysis

ile : E____46.D01 F 408 HAIM
un : 29 Type : Sample
ath : C:\CHROM
ollection : 01:23:54 Dec 06 1991 Method : YEHUDA [09:54:38 Dec 05 1991]
ntegration: 01:23:54 Dec 06 1991 Method : YEHUDA [09:54:38 Dec 05 1991]
eport : 01:37:04 Dec 06 1991 Method : YEHUDA [09:54:38 Dec 05 1991]

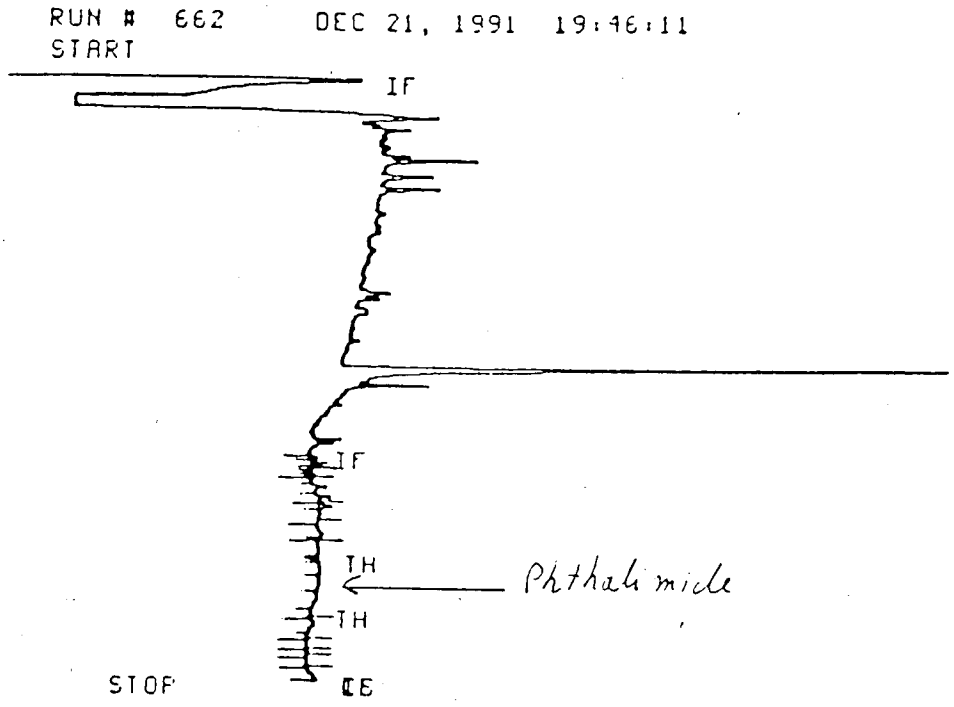
Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

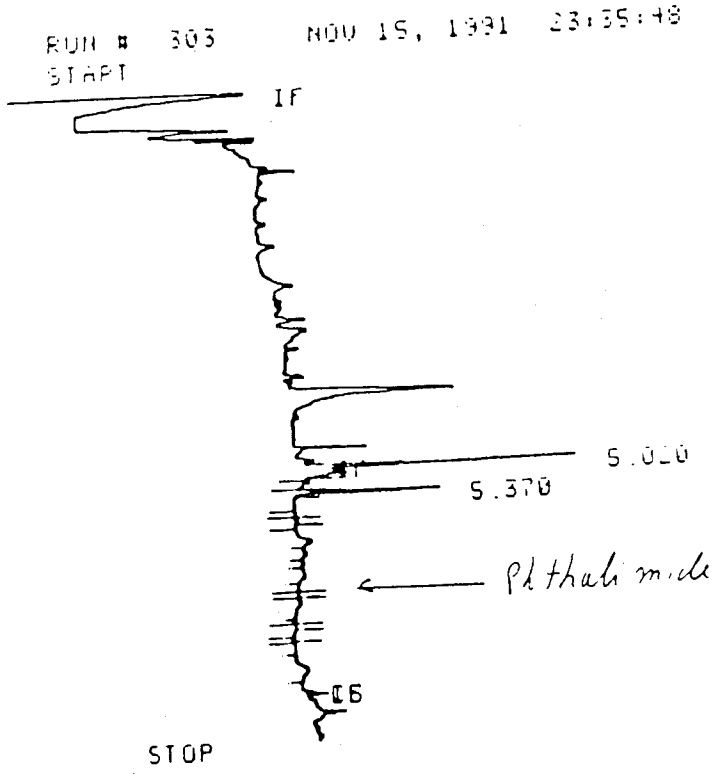
K'	Pk #	RT	Area	Height	ExpRT	Name
					10.600	CAPTAN
					11.302	FOLPAN



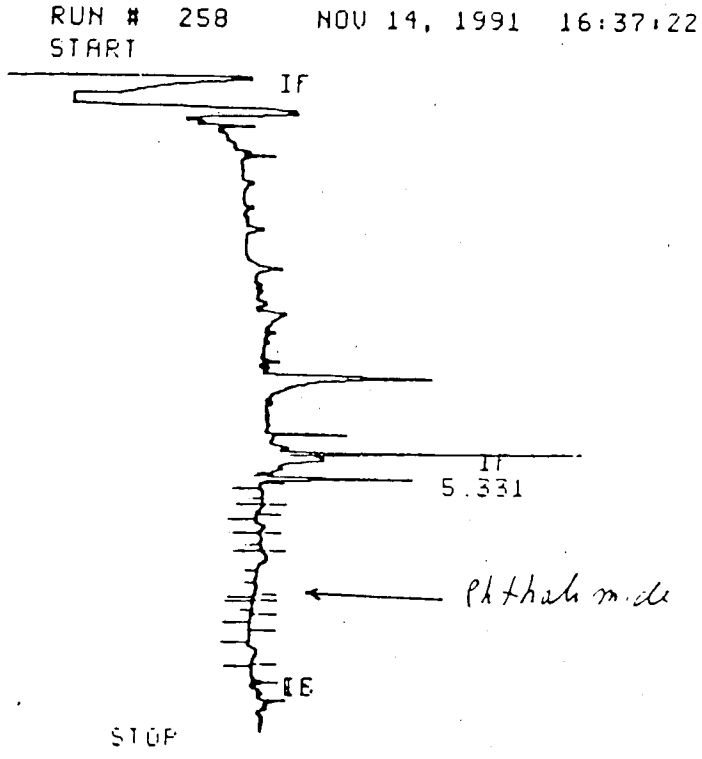
13. Lettuce control for phthalimide analysis



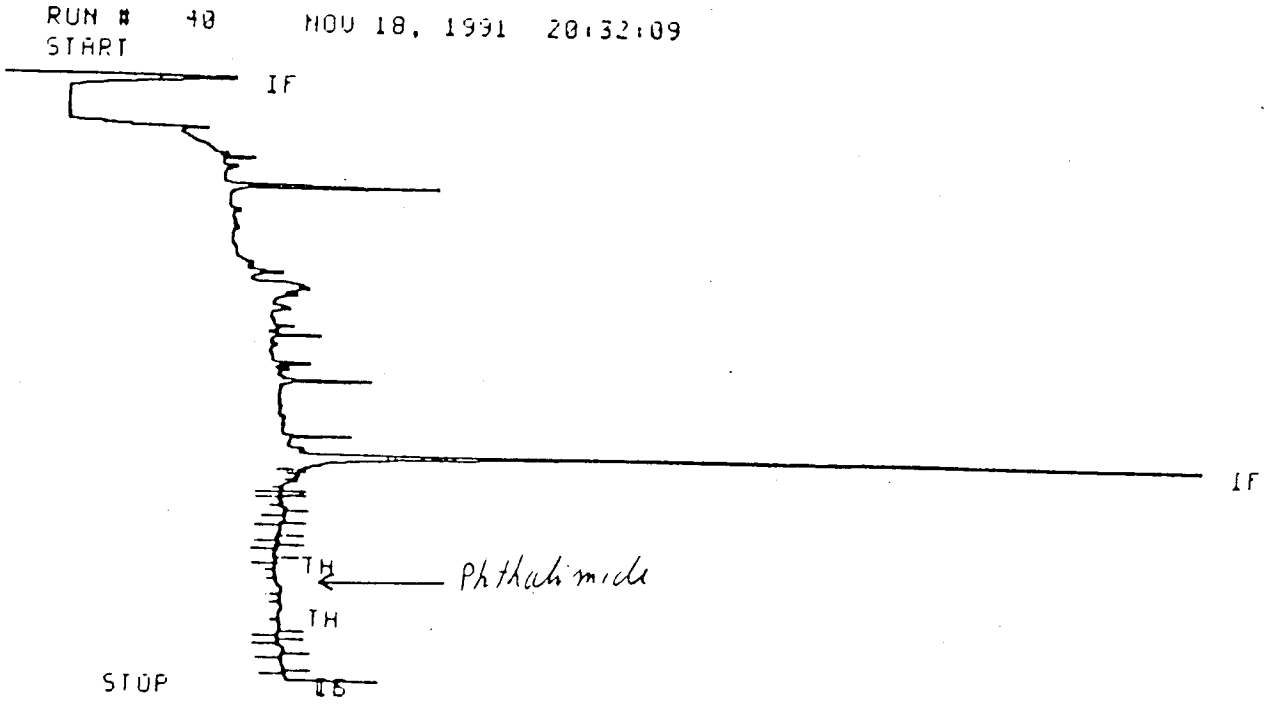
14. Grape control for phthalimide analysis



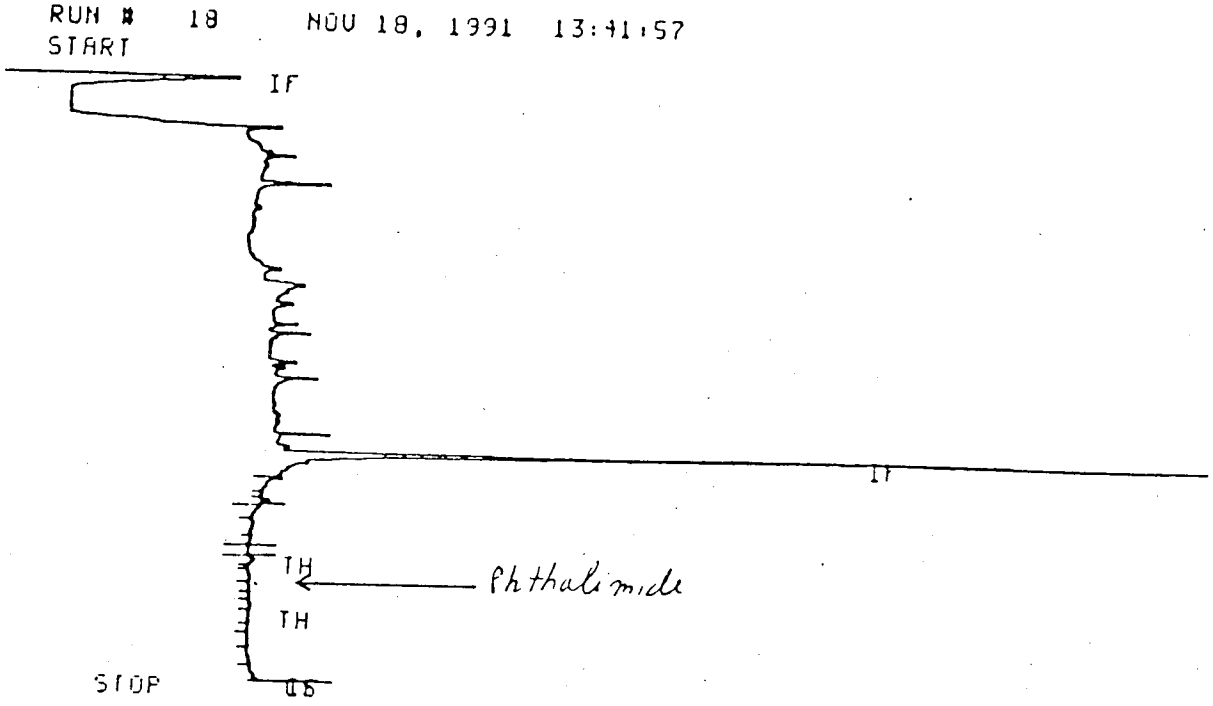
15. Apple control for phthalimide analysis



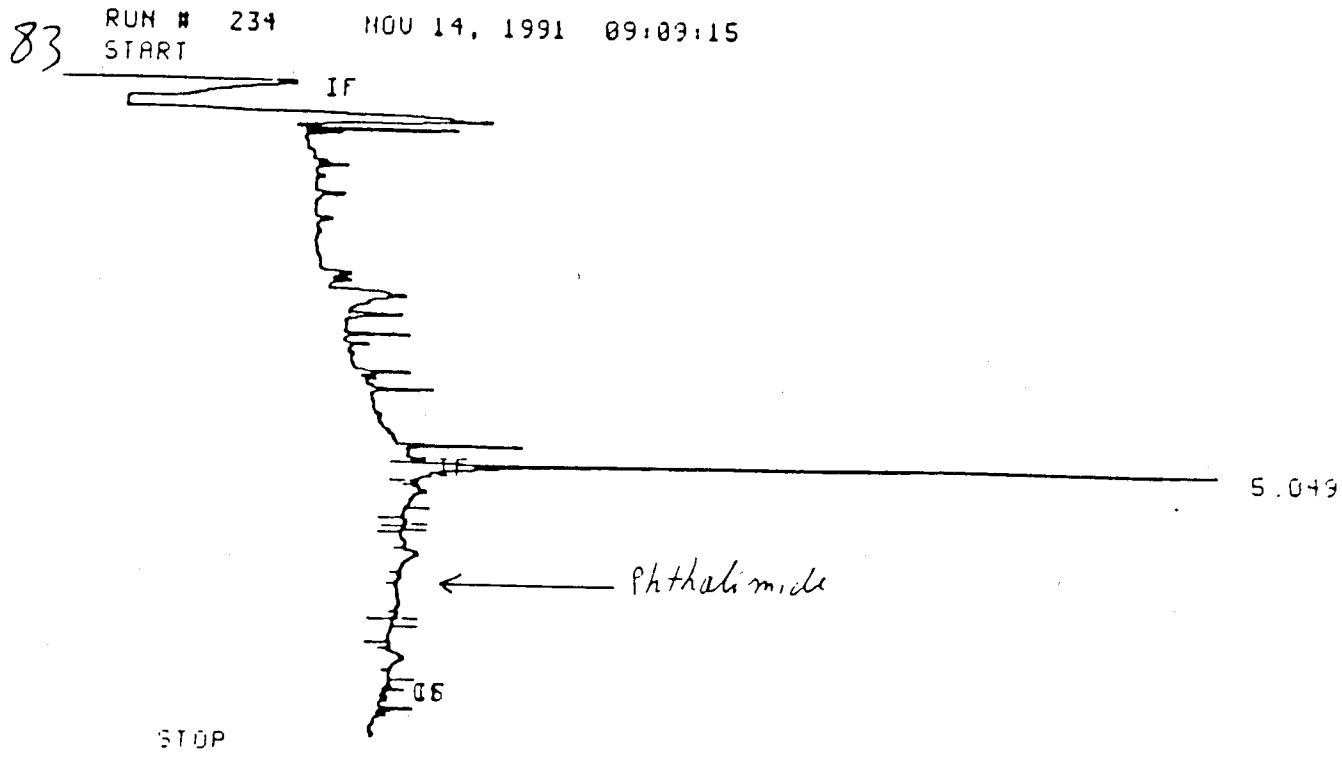
16. Squash control for phthalimide analysis



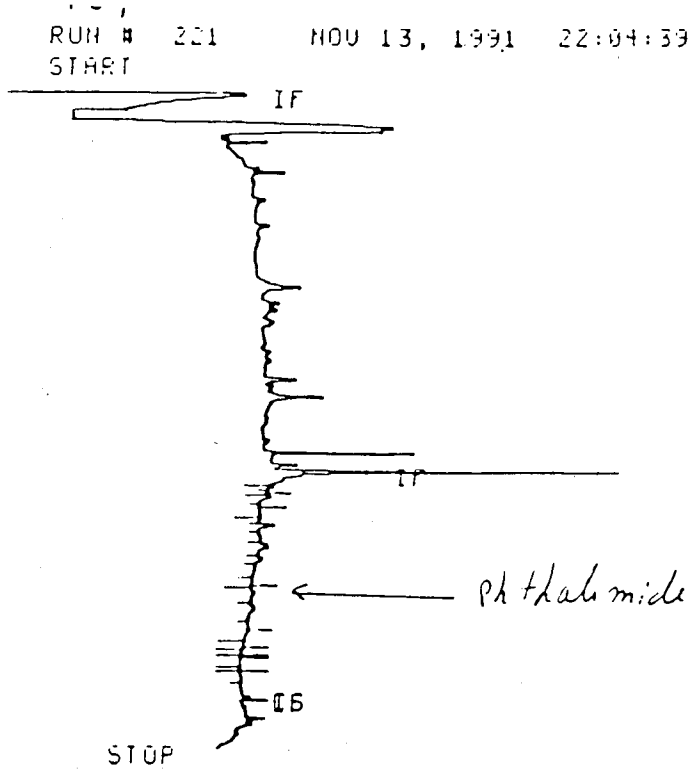
17. Tomato control for phthalimide analysis



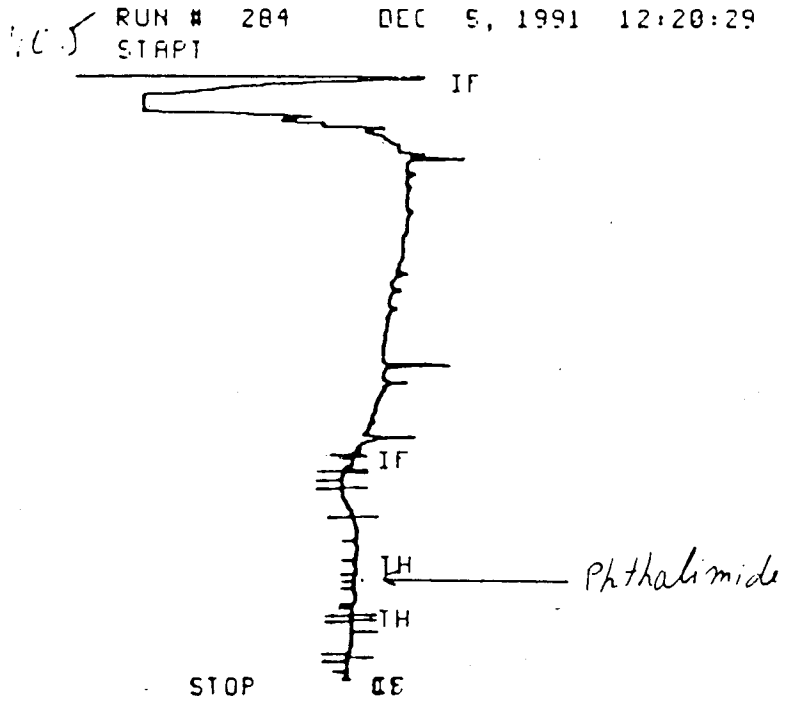
18. Potato control for phthalimide analysis



19. Melon control for phthalimide analysis



20. Strawberry control for phthalimide analysis



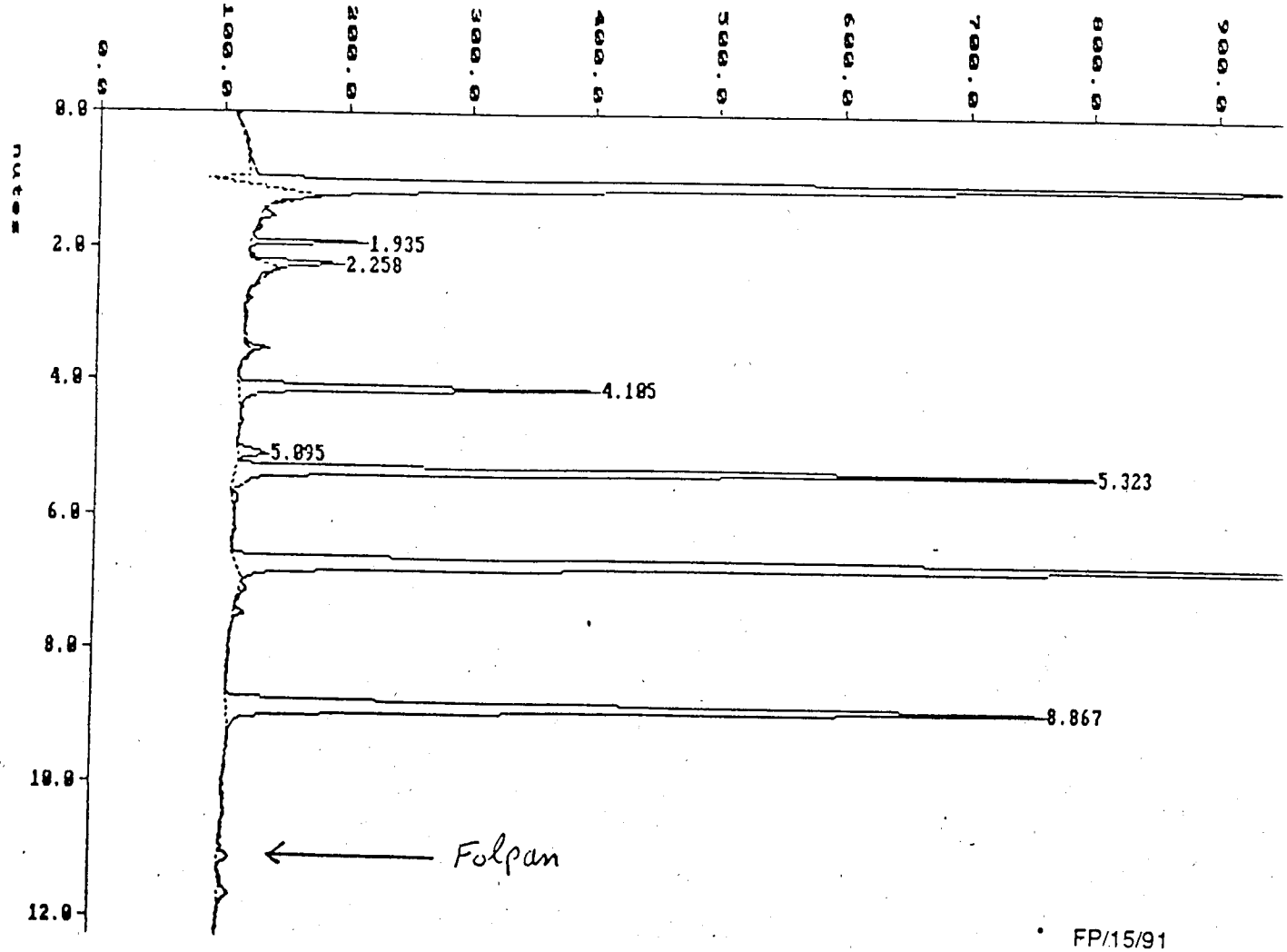
21. A pesticide mixture (0.1 ppm in hexane) for interfering peaks with the folpan analysis

File : K____72.D01 524 HAIM
Run : 08 Type : Sample
Path : C:\CHROM
Collection : 16:00:51 Dec 18 1991 Method : YEHUDA [09:53:40 Dec 15 1991]
Integration: 16:00:51 Dec 18 1991 Method : YEHUDA [09:53:40 Dec 15 1991]
Report : 16:14:01 Dec 18 1991 Method : YEHUDA [09:53:40 Dec 15 1991]

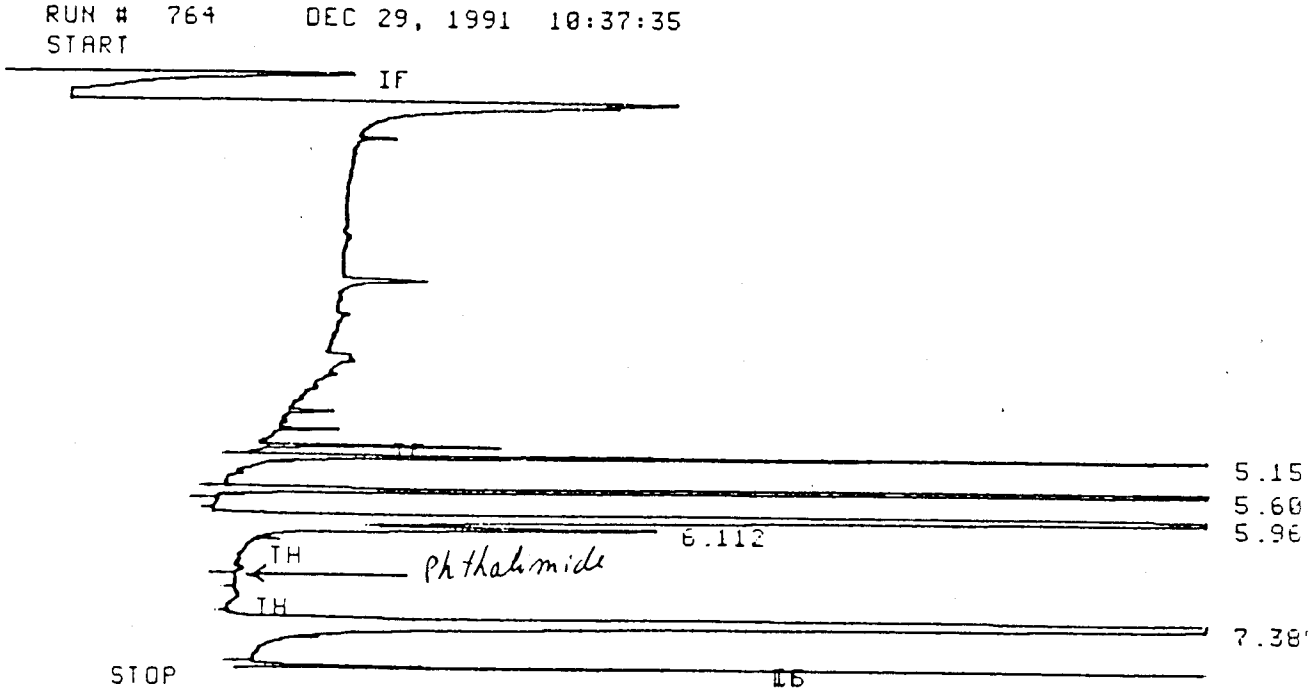
Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Pk #	RT	Area	Height	ExpRT	Name
					10.600	captan
					11.300	FOLPAN



22. A pesticide mixture (1.0 ppm in hexane) for interfering peaks with the phthalimide analysis



RUN# 764 DEC 29, 1991 10:37:35

SAMPLE# 7

AREA%

RT	AREA	TYPE	WIDTH	AREA%
5.155	7308	PB	.028	6.11630
5.600	19613	BB	.038	16.41475
5.965	42053	PB	.051	35.19550
6.112	2732	BB	.039	2.28650
7.389	47778	PB	.089	39.98694

TOTAL AREA= 119484
MUL FACTOR=1.0000E+00

23. Control lettuce sample spiked with 0.02 ppm folpan

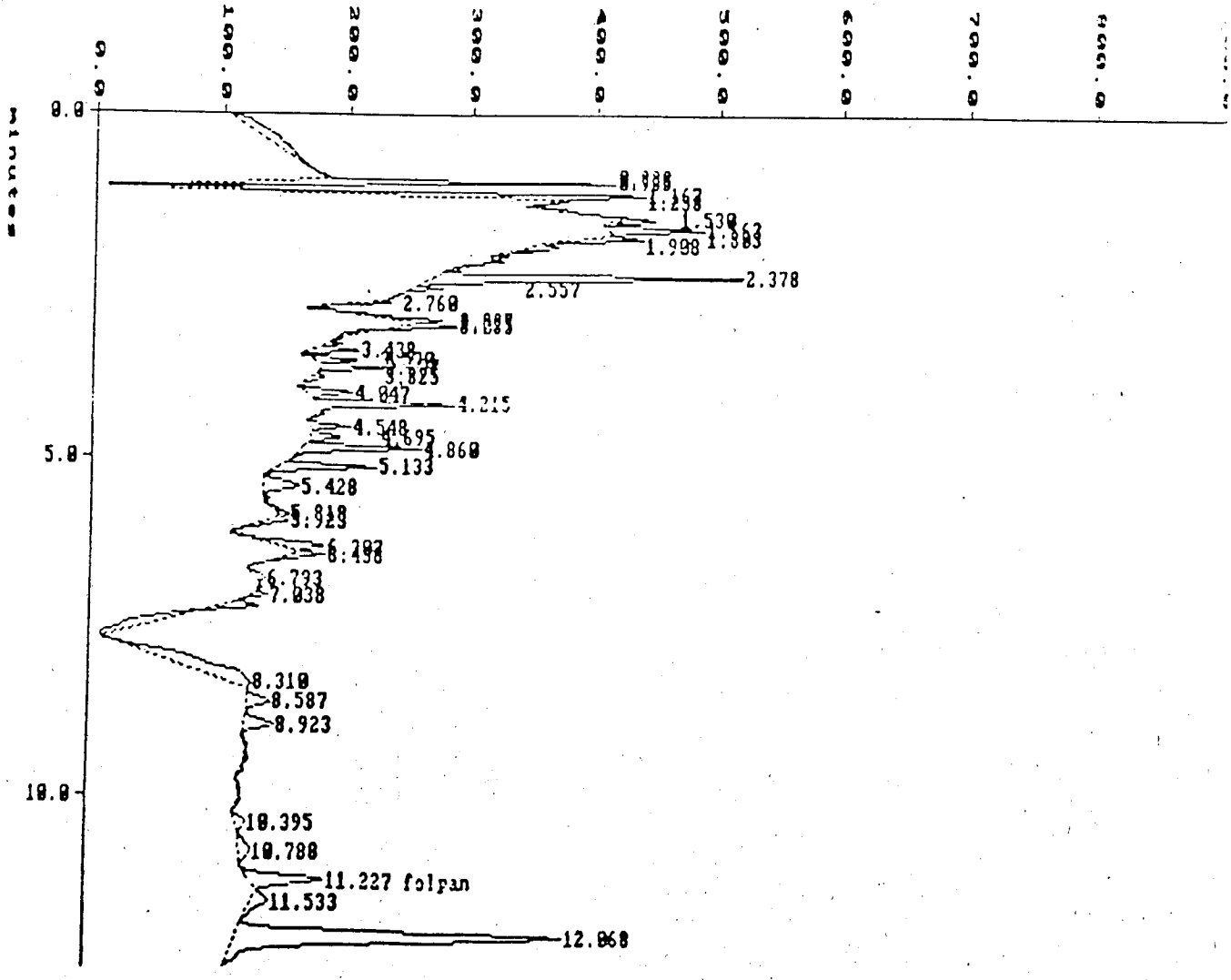
File : FOLPA062.D01 F
Run : 13
Path : C:\CHROM
Type : Sample

Collection : 13:41:35 Sep 02 1991 Method : YEHUDA [09:58:00 Aug 28 1991]
Integration: 13:41:35 Sep 02 1991 Method : YEHUDA [09:58:00 Aug 28 1991]
Report : 13:54:21 Sep 02 1991 Method : YEHUDA [09:58:00 Aug 28 1991]

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Pk #	RT	Area	Height	ExpRT	Name
	36	11.226	4971176	58.358	11.200	folpan



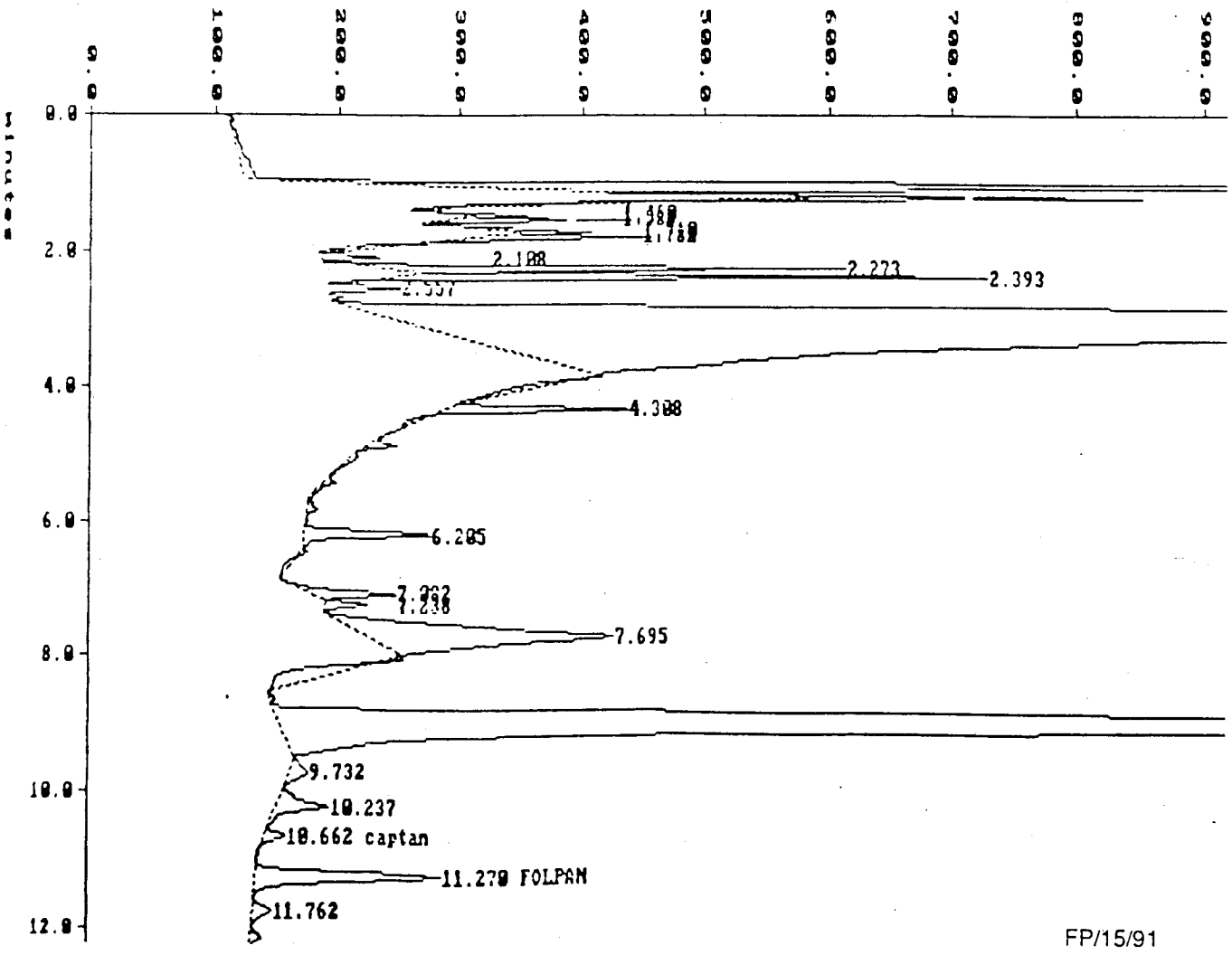
24. Control grape sample spiked with 0.05 ppm folpan

File : K____46.D01 522-10 254 HAIM
Run : 41 Type : Sample
Path : C:\CHROM
Collection : 00:47:16 Dec 17 1991 Method : YEHUDA [09:53:40 Dec 15 1991]
Integration: 00:47:16 Dec 17 1991 Method : YEHUDA [09:53:40 Dec 15 1991]
Report : 01:00:30 Dec 17 1991 Method : YEHUDA [09:53:40 Dec 15 1991]

Sample Amt : 1.02200e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Pk #	RT	Area	Height	ExpRT	Name
	21	10.662	1276352	18.239	10.600	captan
	22	11.270	13825224	153.386	11.300	FOLPAN



FP/15/91

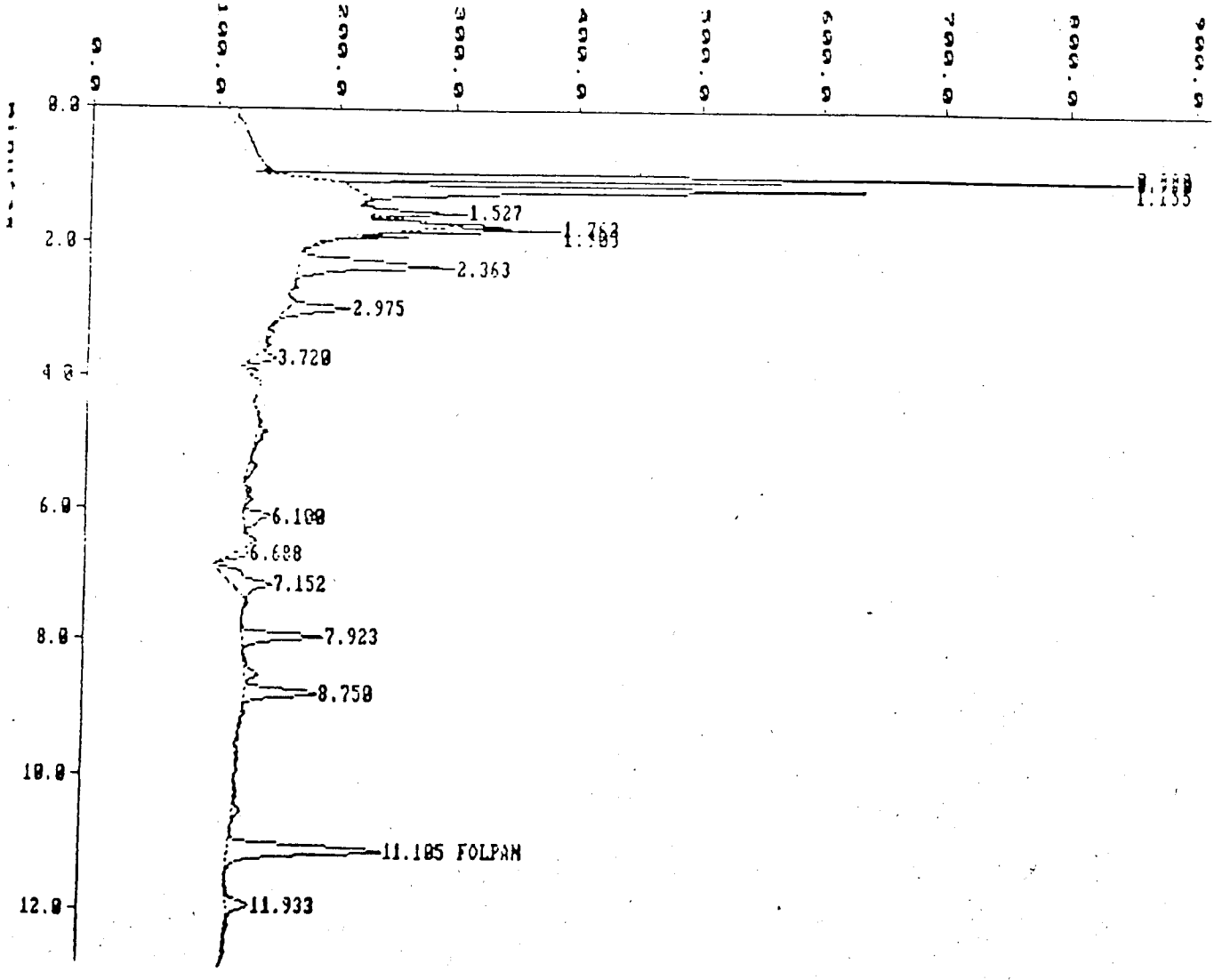
25. Control apple sample spiked with 0.05 ppm folpan

File : W____17.001 F 164 HAIM
Run : 17 Type : Sample
Path : C:\CHROM
Collection : 17:39:44 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]
Integration: 17:39:44 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]
Report : 17:53:02 Nov 18 1991 Method : YEHUDA [13:07:47 Nov 18 1991]

Sample Wt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Pk #	RT	Area	Height	ExpRT	Name
					10.500	CAPTAN
	15	11.105	12391296	127.574	11.200	FOLPAN



26. Control squash sample spiked with 0.05 ppm folpan

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

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EXTERNAL STANDARD (AREA)

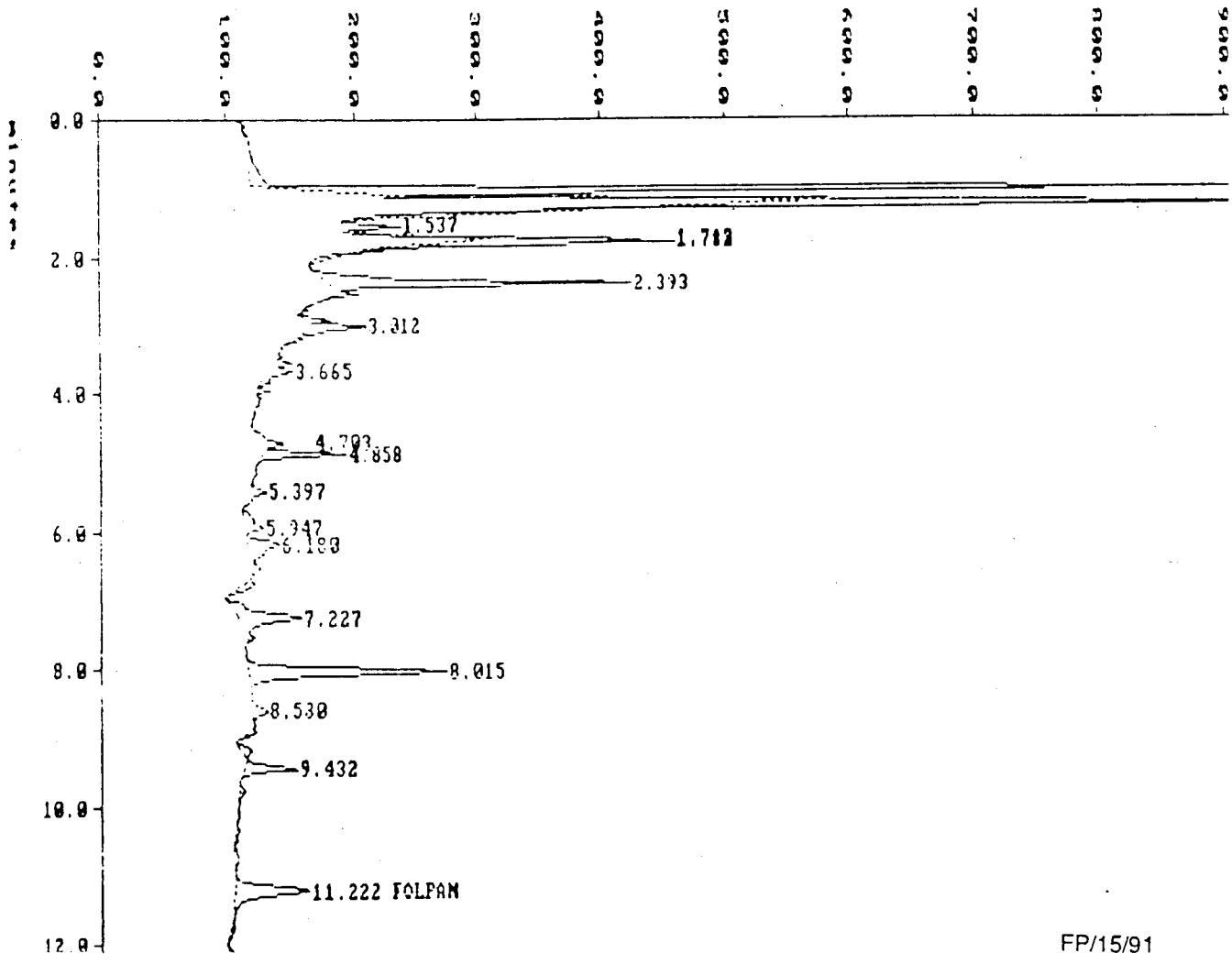
K'	Pk #	RT	Area	Height	ExpRT	Name
					10.600	CAPTAN
	20	11.222	5919912	59.553	11.200	FOLPAN

1 Matched Components 5.56 % of Total Area

19 Unknown Peaks 94.44 % of Total Area

20 Peaks > Area Reject 1.06522e+8 Total Area

(W____85.DQ1) mV



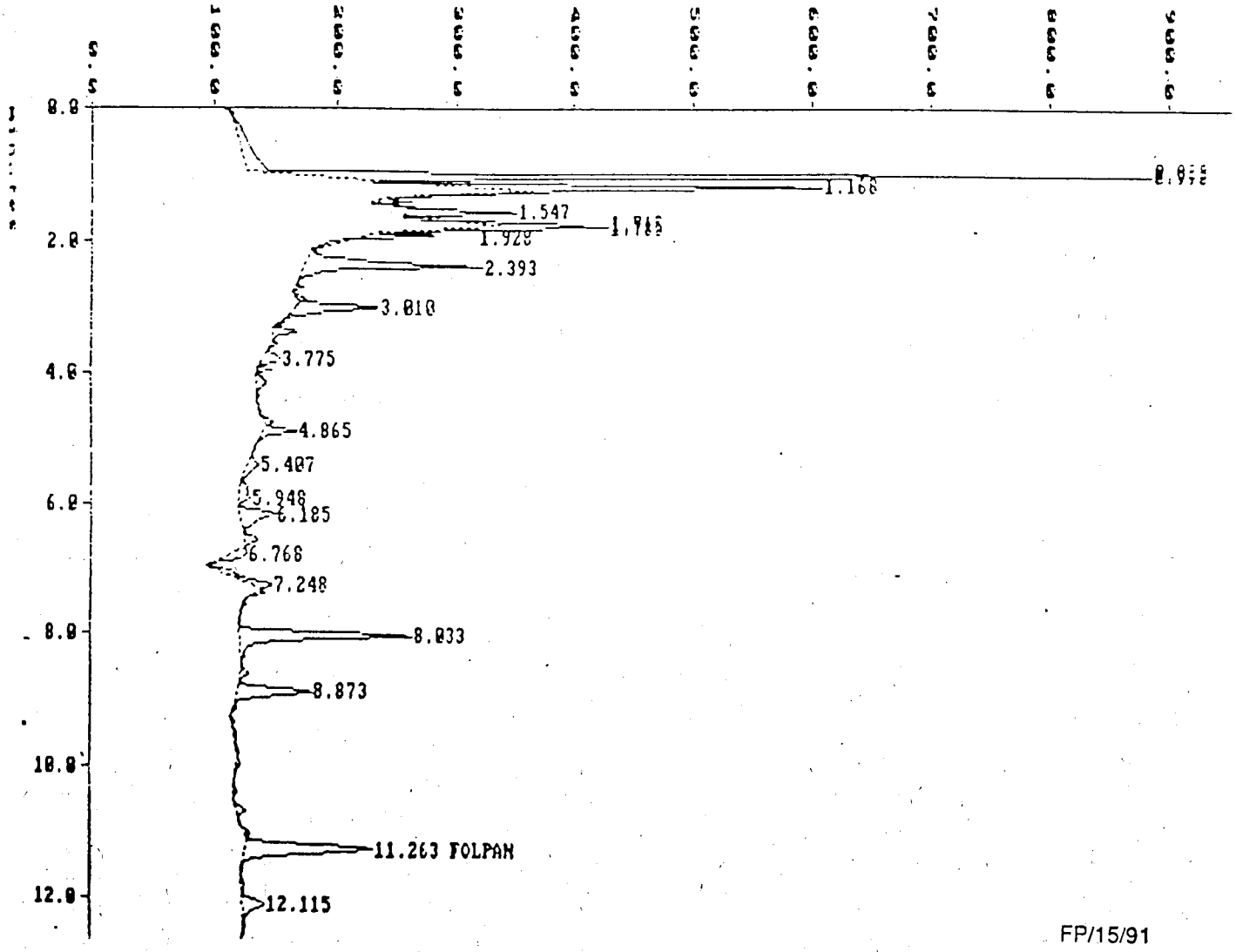
27. Control tomato sample spiked with 0.05 ppm folpan

File : W____55.D01 F 223 HAIM
Run : 01 Type : Sample
Path : C:\CHROM
Collection : 11:35:57 Nov 19 1991 Method : YEHUDA [13:07:47 Nov 18 1991]
Integration: 11:35:57 Nov 19 1991 Method : YEHUDA [13:07:47 Nov 18 1991]
Report : 11:49:15 Nov 19 1991 Method : YEHUDA [13:07:47 Nov 18 1991]

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Fi #	RT	Area	Height	ExpST	Name
					10.000	CAPTAN
	15	11.263	9387124	103.549	11.000	FOLPAN



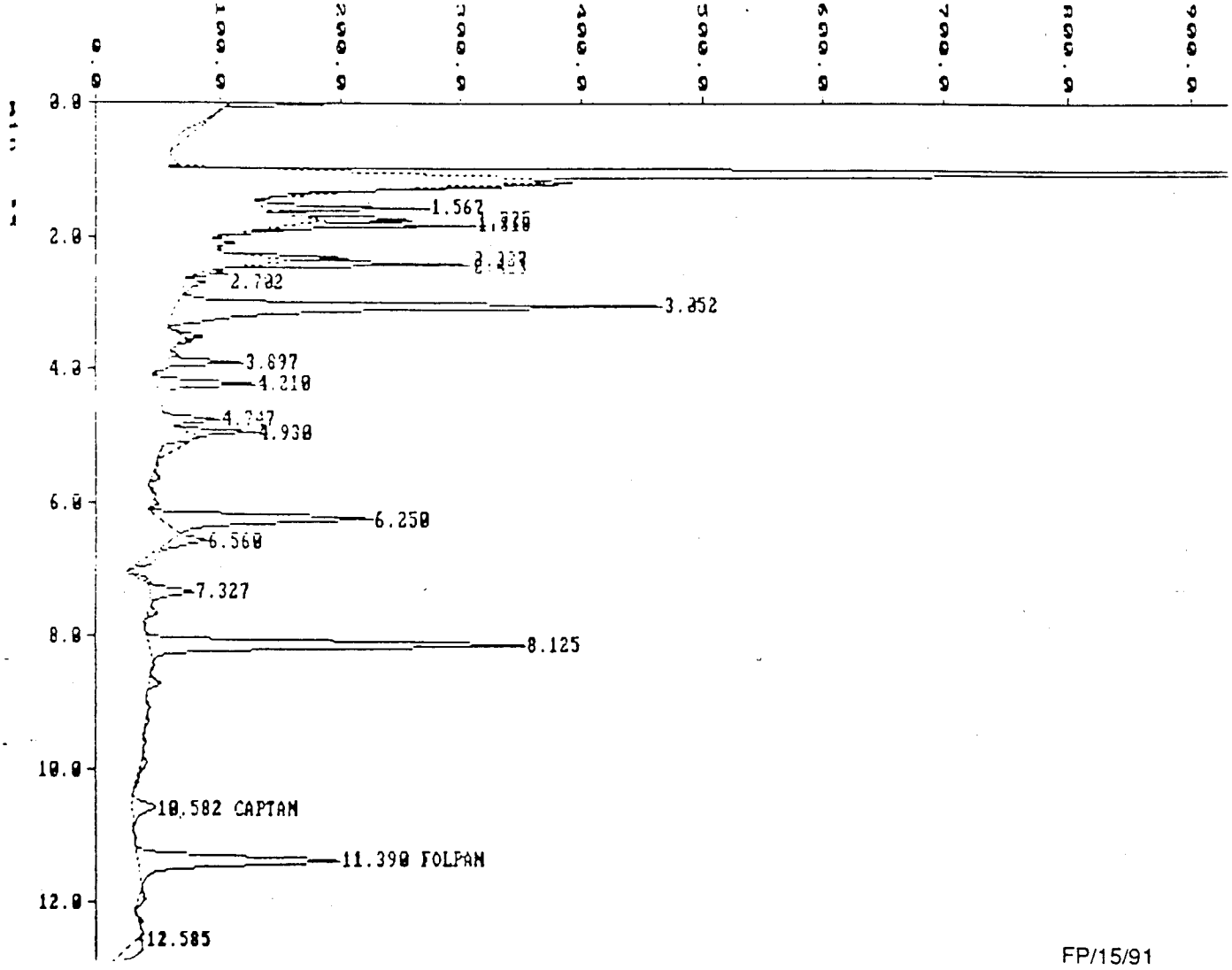
28. Control potato sample spiked with 0.05 ppm folpan

File : V____18.001 st2ppm+3183 HAIM
Run : 18 103 Type : Sample
Path : C:\CHROM
Collection : 22:57:14 Nov 13 1991 Method : YEHUDA [11:16:30 Nov 18 1991]
Integration: 22:57:14 Nov 13 1991 Method : YEHUDA [11:16:30 Nov 18 1991]
Report : 23:10:25 Nov 13 1991 Method : YEHUDA [11:16:30 Nov 18 1991]

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K	Pk #	RT	Area	Height	ExpRT	Name
	17	10.582	2203575	18.763	10.500	CAPTAN
	18	11.390	16773115	167.194	11.200	FOLPAN



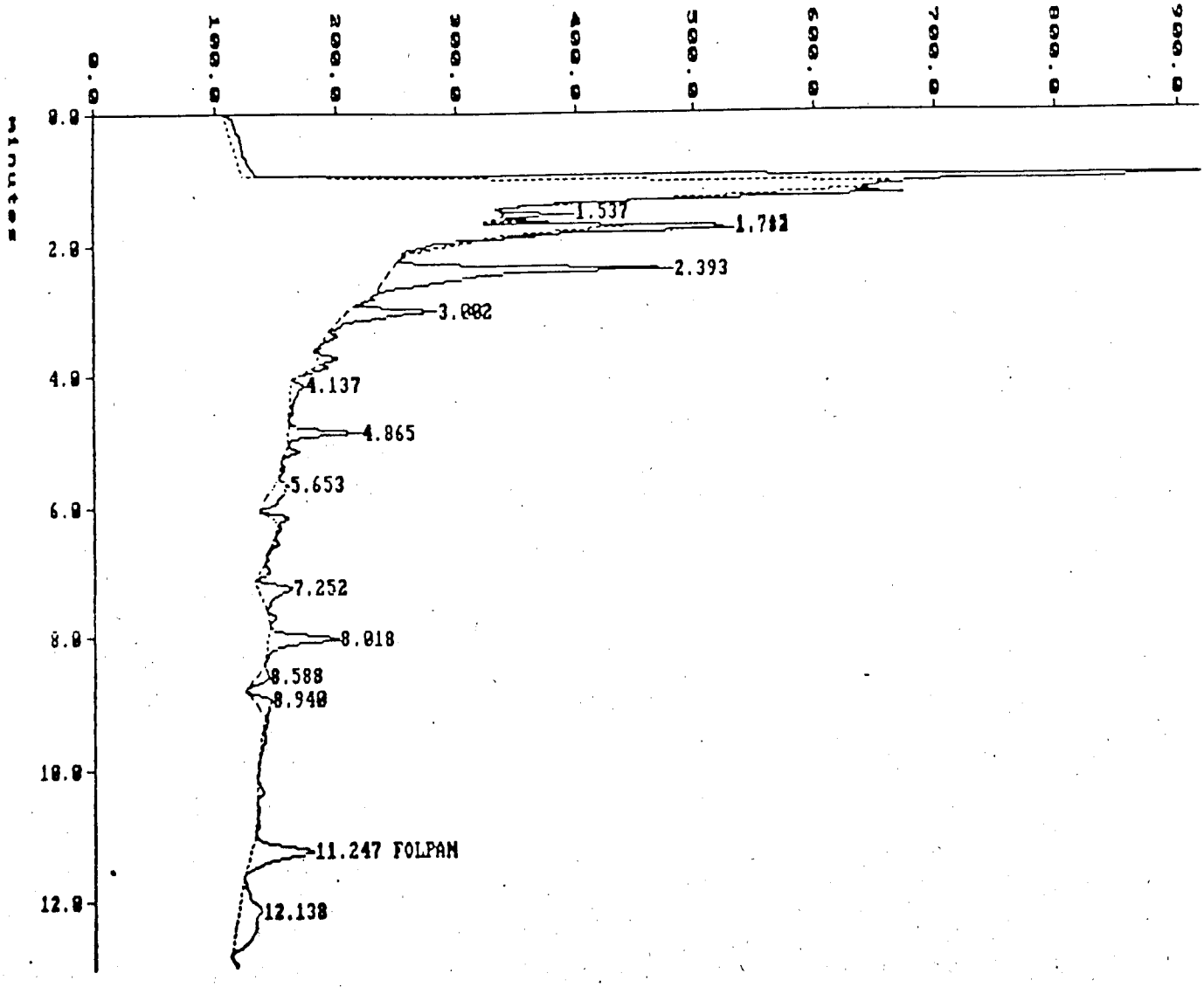
29. Control melon sample spiked with 0.05 ppm folpan

Report : 11:34:49 Dec 09 1991 Method : YEHUDA [10:46:10 Dec 09 1991]

Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

K'	Pk #	RT	Area	Height	ExpRT	Name
					10.600	CAPTAN
	15	11.247	7386000	53.410	11.300	FOLPAN



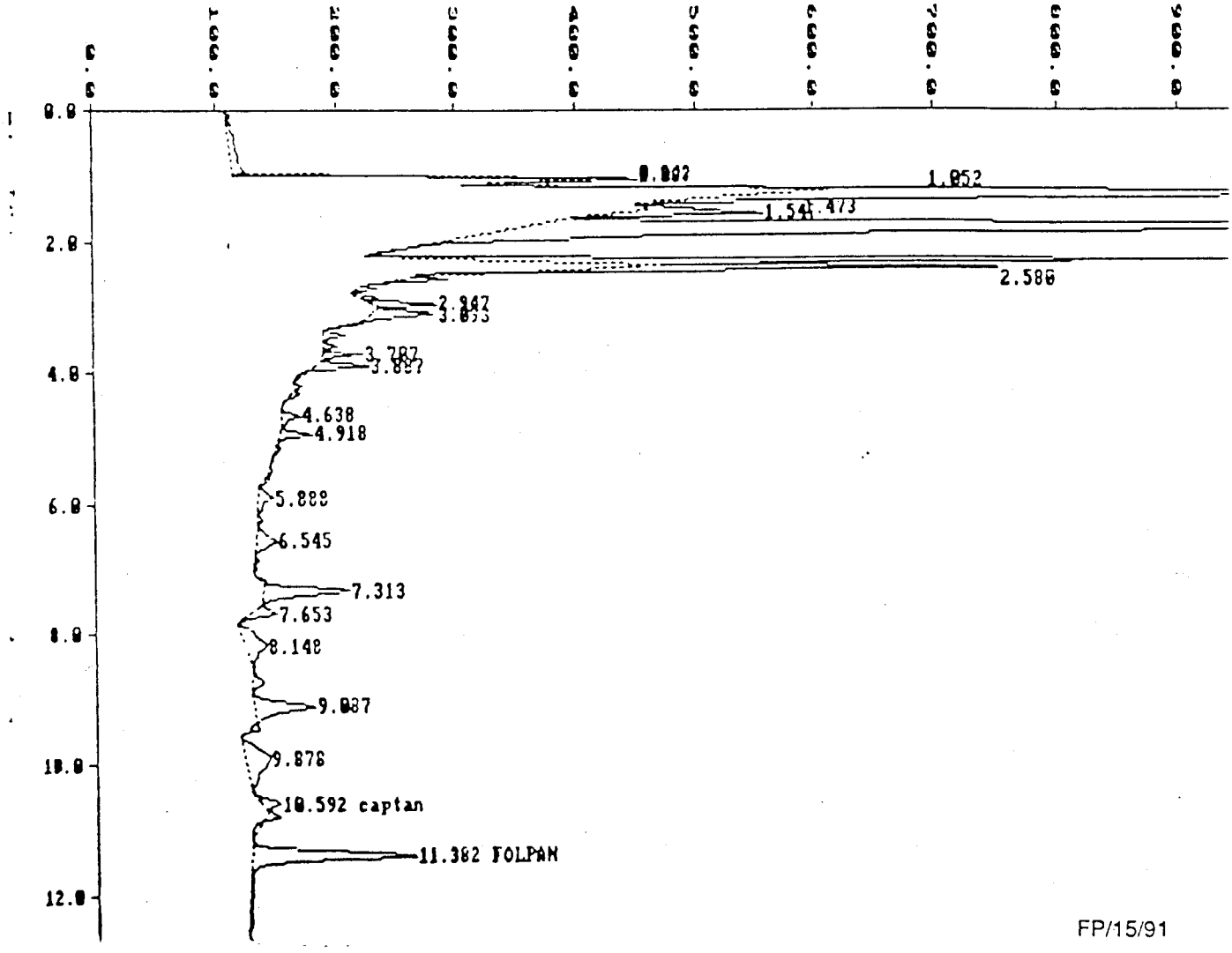
30. Control strawberry sample spiked with 0.05 ppm folpan

File : L____15.D01 o 562 HAIM
Run : 15 Type : Sample
Path : C:\CHROM
Collection : 01:34:28 Dec 19 1991 Method : YEHUDA [18:10:57 Dec 18 1991]
Integration: 01:34:28 Dec 19 1991 Method : YEHUDA [18:10:57 Dec 18 1991]
Report : 01:47:31 Dec 19 1991 Method : YEHUDA [18:10:57 Dec 18 1991]

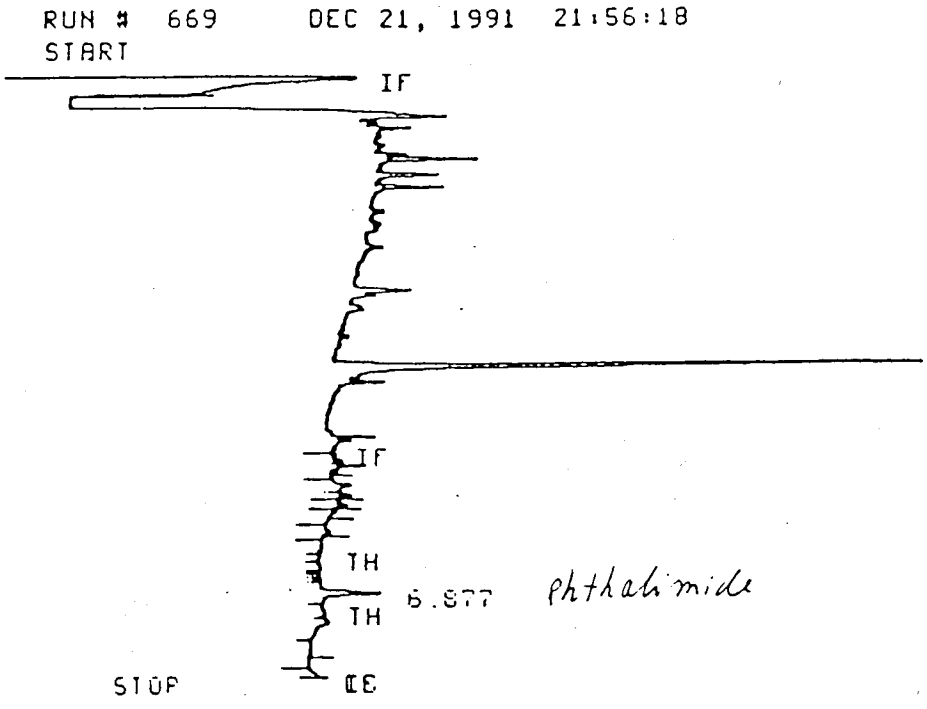
Sample Amt : 1.00000e+0 Dilution: 1.00000e+0

EXTERNAL STANDARD (AREA)

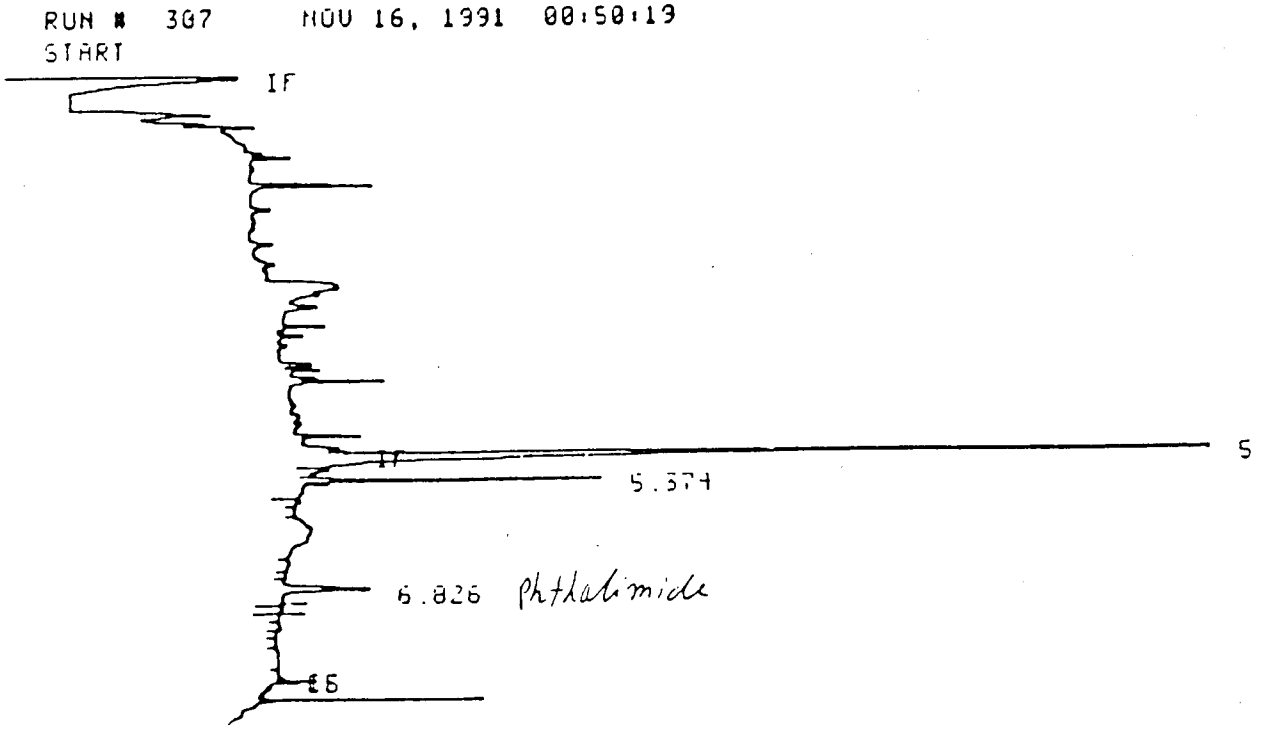
X'	Pk #	RT	Area	Height	ExpRT	Name
	25	10.591	1226964	15.604	10.600	captan
	26	11.382	12457548	136.933	11.380	FOLPAN



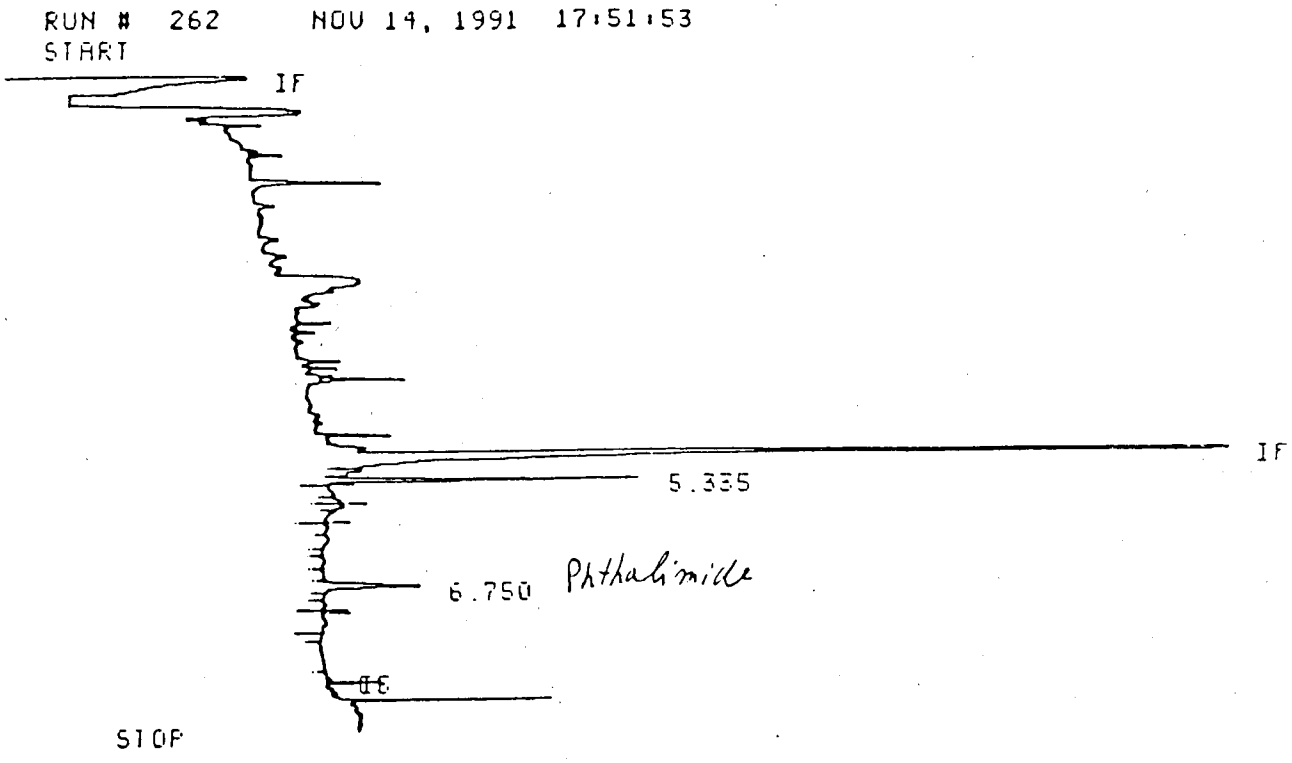
31. Control lettuce sample spiked with 0.2 ppm phthalimide



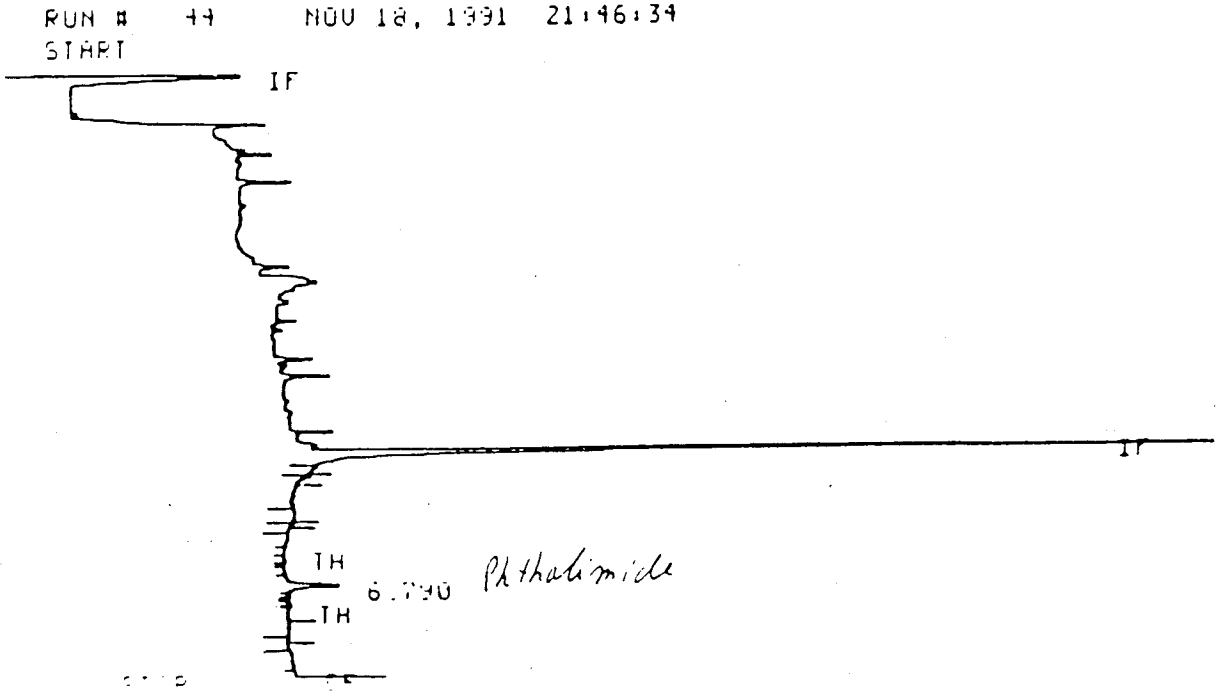
32. Control grape sample spiked with 0.2 ppm phthalimide



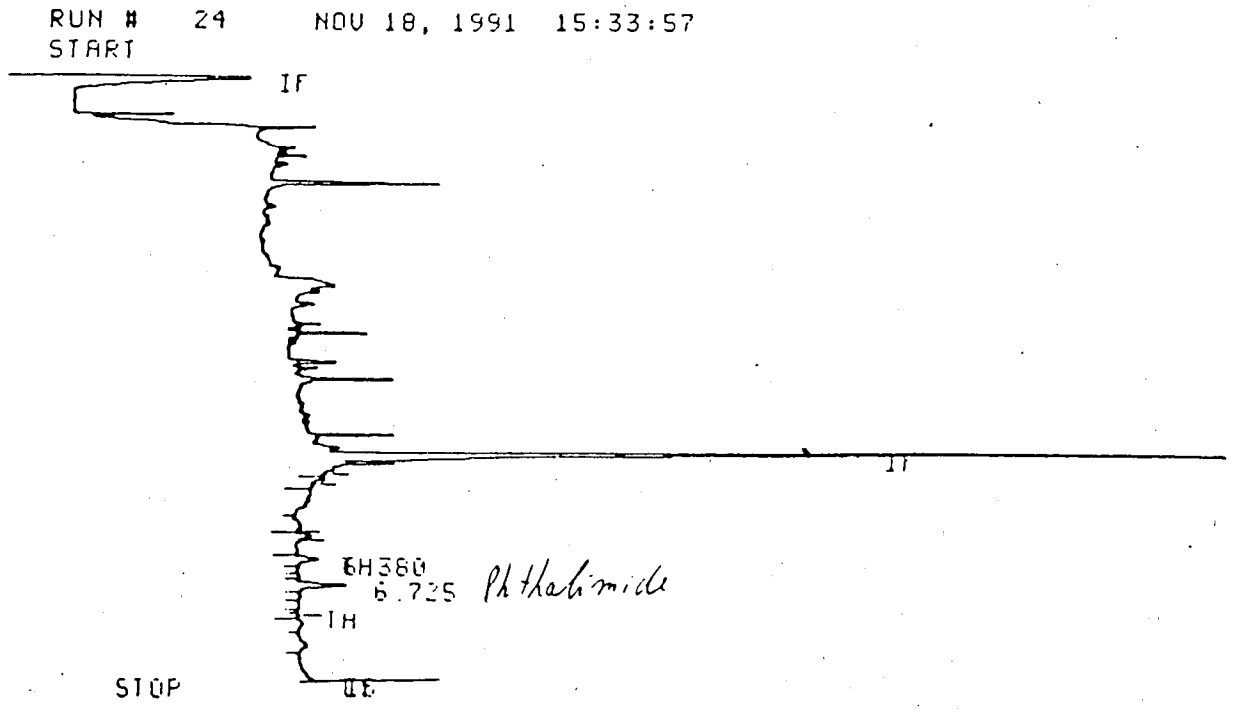
33. Control apple sample spiked with 0.2 ppm phthalimide



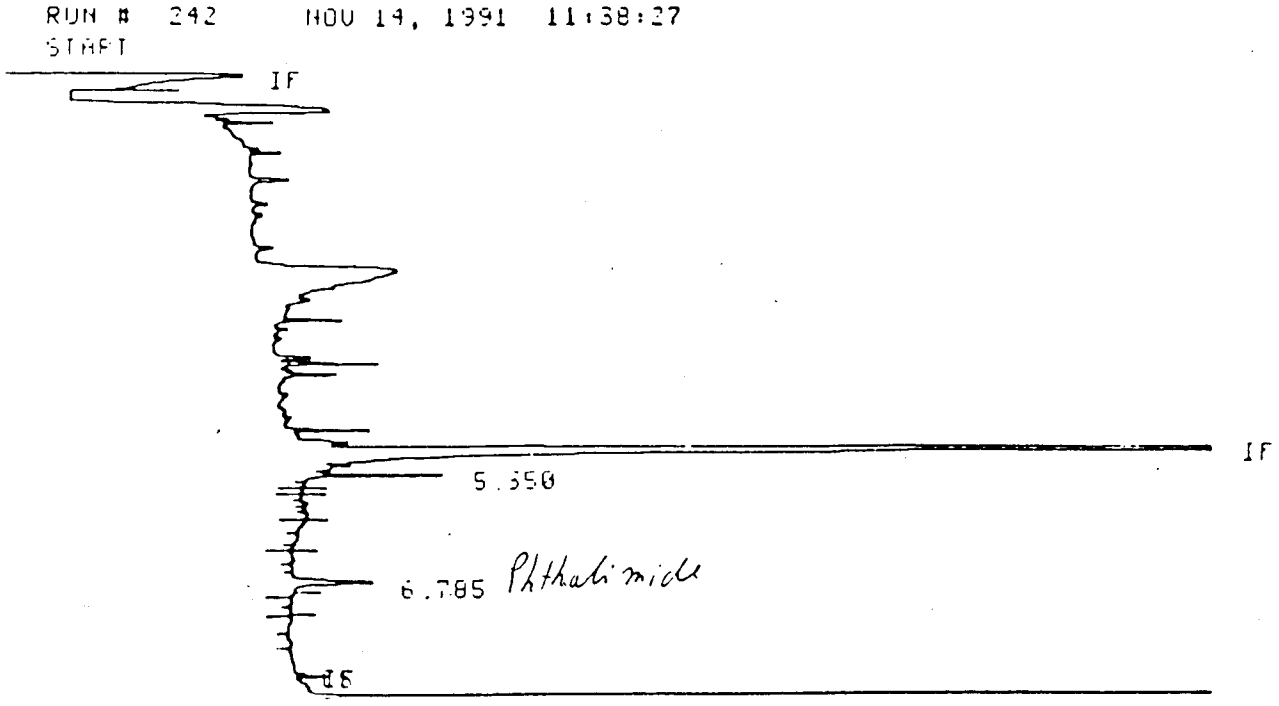
34. Control squash sample spiked with 0.2 ppm phthalimide



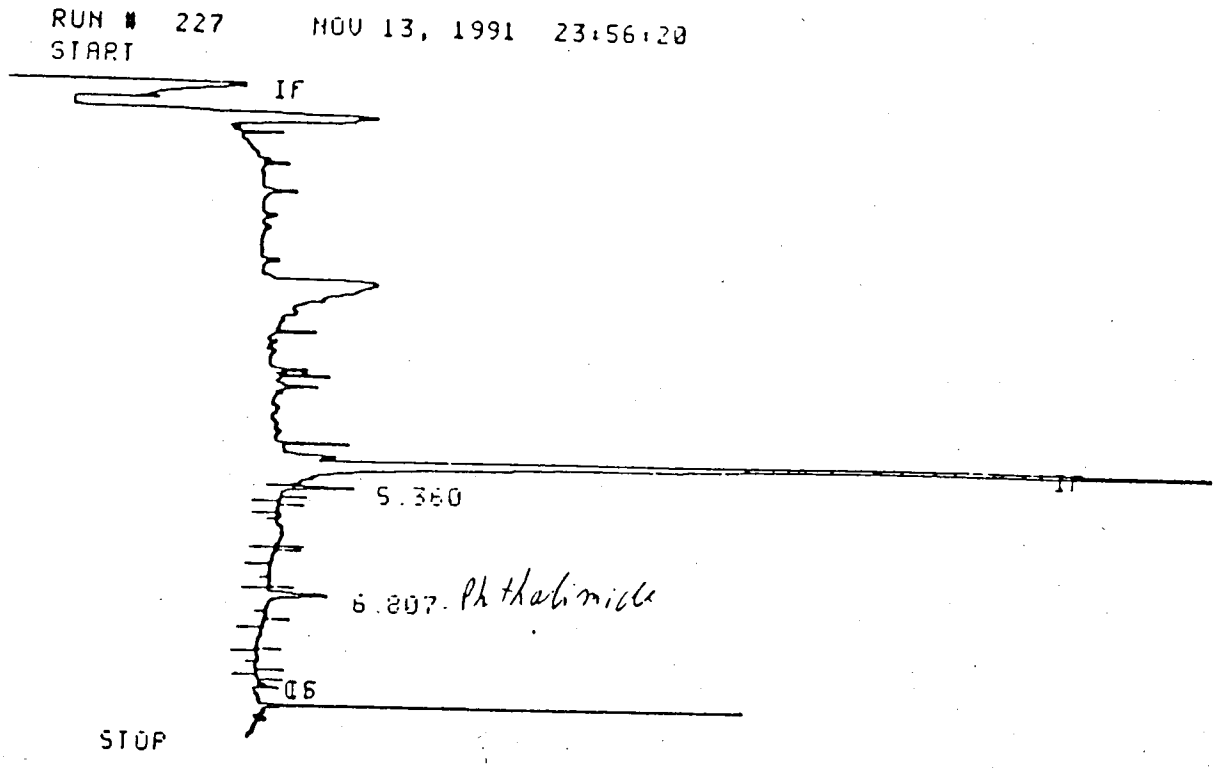
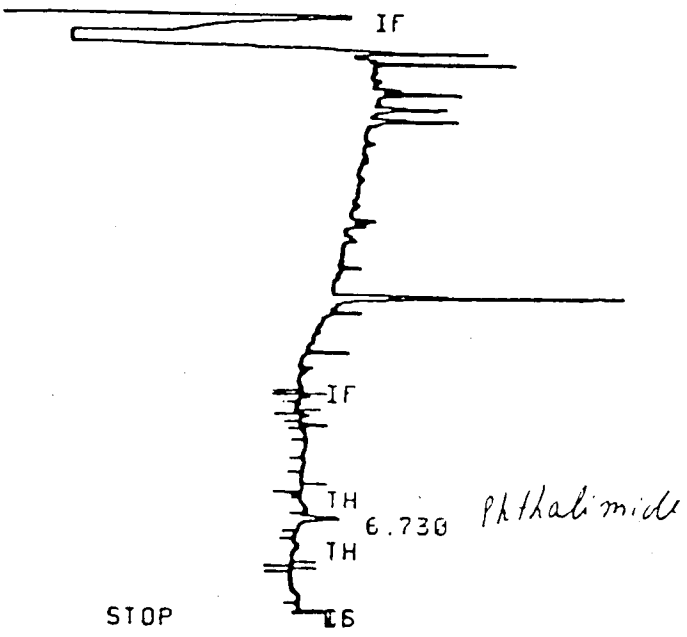
35. Control tomato sample spiked with 0.2 ppm phthalimide



36. Control potato sample spiked with 0.2 ppm phthalimide



37. Control melon sample spiked with 0.2 ppm phthalimide



38. Control strawberry sample spiked with 0.2 ppm phthalimide