

## Cover Sheet for

# ENVIRONMENTAL CHEMISTRY METHOD

***Pesticide Name:*** Fenitrothion

***MRID #:*** 409345-01

***Matrix:*** Soil

***Analysis:*** GC/NPD

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## INTRODUCTION

The present report details the quantitative determination of Sumithion and Sumioxon residues in samples of Illinois and Florida soil treated with Sumithion at 2.5 lb./acre. Samples were taken in triplicate at various intervals after treatment and shipped frozen (dry ice) to our laboratory. Samples were maintained frozen (-20°C) until time of analysis.

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## MATERIALS AND METHODS

### Chemicals

Authentic reference standards of Sumithion and Sumioxon were supplied by Sumitomo Chemical Company, LTD. at the following specified purities: Sumithion (Lot No. 40926) - 96.6%; Sumioxon was supplied at unspecified purity; Chlorfenvinphos (Lot No. S 502) was obtained from the EPA Analytical Reference Standard Repository (Research Triangle Park, NC 27711) at 91.6% purity, for use as an internal standard. Toluene (HPLC grade) was obtained from Fisher Scientific.

### Samples and Sample Storage

Soil samples were received frozen on dry ice from the two field study sites:

1. Watson Consulting Services & Enterprises, Inc.  
Field Research Station  
21 W042 Finley Road  
Downers Grove, IL 60515
2. Wayne L. Curry  
Weed Systems, Inc.  
Route 3 Box 3115  
Melrose, FL 32666

Dissipation study reports from the Illinois and Florida studies are attached as Appendix I and II (Soil and Turf dissipation) and Appendix III (Turf dissipation), respectively. Upon receipt, samples were identified and stored frozen (-20°C) until the time of analysis.

### Fortification of Soil Samples with Sumithion, Sumioxon, and Chlorfenvinphos

Control soil samples from both study sites were fortified with authentic reference standards of Sumithion, Sumioxon, and Chlorfenvinphos at various concentrations to facilitate the determination of standard curves covering the ranges of each compound's concentration in the actual test samples. The concentration of each standard added to control samples are given in Table I. Authentic standards were initially dissolved in toluene and volumetrically added to control soil samples.

### Extraction of Soil Samples

Soil samples were thoroughly mixed and 5.0 or 10.0 gm ( $\pm 0.01$  gm) subsamples were removed and placed into 20 ml glass scintillation vials. To each sample was added 200  $\mu$ l of internal standard working solution (50  $\mu$ g Chlorfenvinphos/ml) followed by 10 ml toluene and extracted on a wrist-action shaker for 40 minutes. The solvent was then removed and centrifuged at 200 x g for 10 minutes to remove particulates. Aliquots (2 to 5  $\mu$ l) of the supernatant were then injected into the GLC.

### Gas-Liquid Chromatography

All sample extracts were analyzed using a Varian 3400 gas-liquid chromatograph equipped with a Model 800 Autosampler, nitrogen-phosphorous selective detector and a Hewlett-Packard 3392 A Integrator. Analyses utilized a 10 meter HP-17 crosslinked 50% Ph Me Silicone column (0.54 mm i.d.; 2.0  $\mu$ m film thickness). GLC analysis conditions were as follows.

Column Flow - 3.0 ml/min N<sub>2</sub>

Injector temperature - 250°C

Detector temperature - 300°C

#### Temperature Program

initial temp	200°C	held 0 min
to	250°C	rate 50°/min
to	275°C	rate 250°/min

### Analysis of Chromatographic Data

Standard Curves. Standard curves were determined concurrently with each extracted series of samples (i.e., samples included in a single chromatographic analysis set, consisting of 15 to 45 samples). A representative standard curve is presented in Figure 1.

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Test Samples. Concentrations of Sumithion and Sumioxon were calculated using regression analysis comparing concentrations of standards to the relative response of compound/internal standard (chlorfenvinphos).

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271 271 15

125 012 100

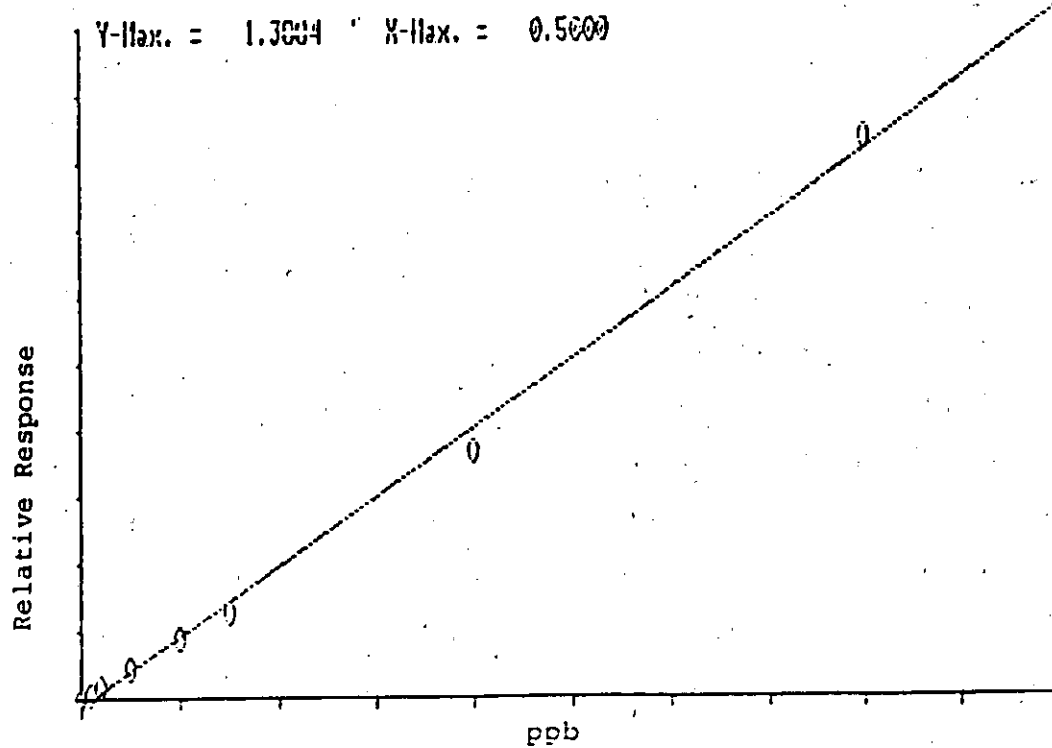
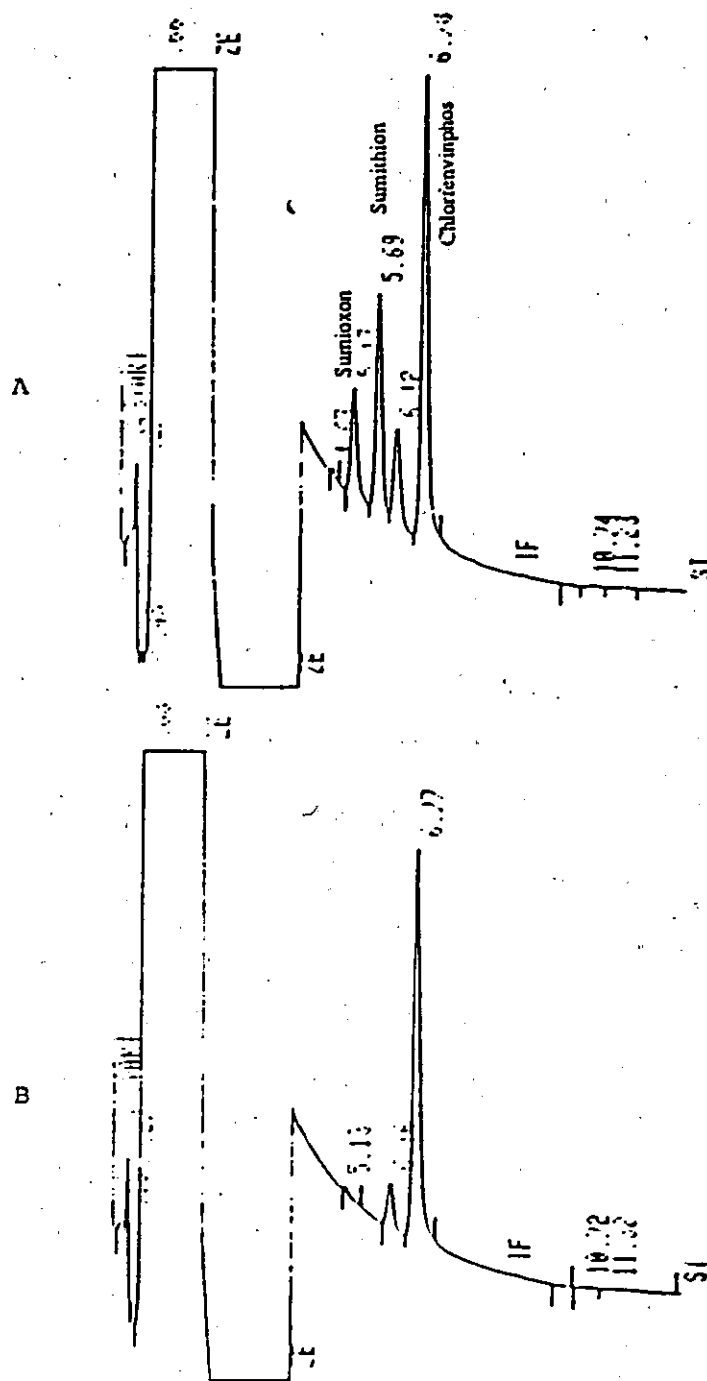


Figure 1. Representative Standard Curve Used For Analysis of Soil Samples.

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Figure 2. Gas-Liquid Chromatograms of Sumithion, Sumioxon and Chlorfenvinphos (Internal Standard). (A) Authentic Reference Standards. (B) Illinois Soil Sample.

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Table 1. Concentrations of Authentic Reference Standards in Fortified Control Soil Sample Used For Representative Standard Curve.

Standard (ng added to sample before extraction) <sup>1</sup>		
Matrix	Sumithion	Sumioxon
SOIL	0	0
	20	20
	40	40
	75	75
	125	125
	250	250
	375	375
	1000	1000

<sup>1</sup> Soil samples weighed 5.00 gm  $\pm$  0.1 or 10.0 gm  $\pm$  0.1.

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# A5. Precision and Accuracy Analysis.

- Sumioxon

ppb Standard	Calculated Concentrations (ppb)	N	Mean	S.D.	Coefficient of Variation (%)	Mean Recovery (%)
1260.0	1305, 1324, 1264, 1388	4	1320.25	51.64	3.91	104.8
630.0	659, 548, 519, 631, 395, 626, 640, 651, 633, 630	10	593.20	83.09	14.01	94.2
236.25	185, 212, 184, 207, 178, 243, 247, 214, 195, 236, 250, 254	12	217.08	27.95	12.88	91.9
157.5	124, 158, 154, 168, 137, 141, 158, 146, 137, 143, 142, 131	12	144.92	12.59	8.69	97.8
100.0	96, 95, 102, 105, 94, 93, 95, 97, 103, 95, 100, 99	12	97.83	3.90	3.99	97.8
78.75	40, 85, 87, 70, 89, 92, 80, 87, 74, 84, 67, 73	12	77.33	14.25	18.42	98.2
37.0	42, 44, 33, 31, 47, 49, 32, 38, 29, 44, 49, 38	12	39.67	7.19	18.13	107.2
31.5	83, 47, 63, 34, 76, 36, 27, 41, 40, 36, 34, 42	12	46.58	17.79	38.19	147.9
25.0	34, 21, 20, 19, 38, 36, 60, 27, 25, 41, 16, 19	12	29.67	12.75	42.98	118.7
15.75	50, 35, 55, 16, 68, 13, 13, 24, 31, 14, 21, 13	12	29.42	18.89	64.23	186.8
12.5	22, 21, 9, 12, 10, 14, 10, 10, 14, 13, 14, 11	12	13.33	4.21	31.55	106.6
7.5	6.11, 3.39, 8.34, 10.03, 1.27, 6.04, 0.0, 4.57, 7.28, 4.01, 6.16, 6.23	12	5.29	2.84	53.67	70.5
4.0	1.83, 2.67, 5.36, 5.55, 0.0, 0.0, 0.0, 3.07, 5.12, 0.0, 2.86, 5.66	12	2.68	2.33	87.16	67.0
2.0	1.23, 0.60, 4.40, 3.06, 0.0, 0.81, 0.0, 2.75, 1.65, 0.0, 2.01, 3.09	12	1.63	1.45	88.75	81.5

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## Precision and Accuracy Analysis.

Summation

ppb Standard	Calculated Concentrations (ppb)	N	Mean	S.D.	Coefficient of Variation (%)	Mean Recovery (%)
1336.0	1357, 1367, 1364, 1339, 1388, 1334, 1366, 1331, 1363	9	1357	18.55	1.37	101.6
668.0	674, 647, 613, 623, 672, 572, 681, 618, 675, 677, 682, 624	12	647	35.86	5.54	96.9
250.5	229, 225, 229, 223, 225, 229, 253, 233, 239, 245, 254, 237	12	235	10.71	4.56	93.8
167.0	157, 118, 164, 140, 168, 161, 124, 144, 155, 140, 147, 147	12	147.1	15.28	10.39	89.2
100.0	96, 95, 101, 103, 97, 92, 97, 94, 98, 97, 99, 99	12	97.3	2.99	3.07	97.3
83.5	128, 70, 88, 112, 79, 85, 78, 88, 81, 80, 81, 83	12	87.8	16.17	18.42	103.3
37.0	44, 41, 35, 35, 42, 47, 41, 41, 41, 44, 44, 35	12	40.8	3.95	9.68	110.3
33.4	19, 71, 45, 48, 37, 49, 43, 48, 37, 38, 33, 43	12	42.6	12.23	28.71	127.5
25.0	33, 27, 21, 21, 32, 37, 34, 32, 29, 32, 24, 23	12	28.8	5.43	18.85	115.2
16.7	7.8, 42.0, 29.1, 36.3, 19.5, 38.1, 26.0, 31.7, 19.1, 24.3, 16.8, 33.4	12	27.0	10.0	37.07	161.7
12.5	18.8, 20.4, 11.4, 12.3, 11.1, 15.2, 10.8, 11.6, 11.8, 11.6, 11.8, 11.4	12	13.18	3.21	24.36	105.4
7.5	3.17, 3.84, 7.92, 7.97, 4.33, 7.16, 4.53, 3.71, 5.09, 4.89, 5.85, 6.53	12	5.42	1.65	30.49	72.3
4.0	0.05, 1.91, 5.51, 5.68, 2.08, 5.77, 2.01, 2.89, 3.57, 2.02, 4.10, 5.04	12	3.39	1.85	54.65	84.8
2.0	0.0, 0.39, 3.56, 4.84, 1.42, 0.0, 0.73, 1.93, 1.83, 0.54, 1.7, 3.46	12	1.70	1.55	91.04	85.0

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