content. For example, three samples collected in 1970 contained hexachlorodibenzo-*p*-dioxin (0.5 to 37 ppmw) and heptachlorodibenzo-*p*-dioxin (90 to 135 ppmw). In 1969 more than 45 million pounds of pentachlorophenol was produced in the United States and more than 25 million pounds of this was used as a wood preservative (Fowler *et al.*, 1971). It would appear important, therefore, that investigation of the content and effects of chlorinated dibenzo-*p*-dioxins and chlorinated dibenzofurans in technical pentachlorophenol be continued.

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Comparison of Electron Capture and Electrolytic Conductivity Detectors for the Residue Analysis of *s*-Triazine Herbicides

Ranajit Purkayastha* and William P. Cochrane[1]

The capabilities of ^{63}Ni electron capture (ECD) and electrolytic conductivity detectors (CCD) have been compared for the analysis of *s*-triazine herbicides in water, soil, and corn samples by gas chromatography. Atrazine was selected for the residue study and other *s*-triazines were studied using various stationary phases only for their gas chromatographic characteristics. Quantitative recovery of atrazine from fortified water samples was obtained using dichloromethane. Extraction by acetonitrile, methanol, and acetone showed good recovery for soil samples. Acetonitrile extraction was found adequate for corn analysis. The cleanup procedures involved partitioning and column chromatography on deactivated alumina. Comparable sensitivities were obtained using both methods of detection and quantitation for the range studied (0.02 to 2.0 ppm). The CCD seemed to have wider application than the ECD, which required cleanup in all cases studied. The conductivity analysis of water and soil samples could be performed quantitatively without cleanup, whereas corn samples required cleanup.

The chemical analysis of triazine herbicides in soils has been reviewed by Mattson *et al.* (1970); they gave the detailed method for the analysis of atrazine in soil samples on a routine basis using acetonitrile–water–mixed solvent extraction, cleanup by alumina column, and microcoulometric gas chromatography.

The use of gas chromatography for the analysis of triazine herbicides in various substrates has also been reviewed recently (Cochrane and Purkayastha, 1972). In this review the different aspects such as extraction, cleanup, gas chromatographic separation, and detectors for the analysis of various herbicides have been presented.

These two reviews state that the following solvents have been used for the extraction of triazines: dichloromethane, chloroform, and diethyl ether for water; chloroform, dichloromethane, methanol, acetone, and acetonitrile–water for soil; and methanol(ammoniacal)–dichloromethane, methanol, chloroform, aqueous acetone, Skellysolve B, and acetonitrile for plant samples.

The water samples needed minimum or no cleanup. Partition and chromatography on alumina columns have been used for the removal of impurities from soil samples. But for plant samples, a further cleanup on a Florisil column has been found necessary in a few instances.

Recently the selectivity and sensitivity of the Coulson electrolytic conductivity detector (CCD) (Coulson, 1966) for nitrogen-containing pesticides have been shown to be satisfactory (Cochrane and Wilson, 1971; Eberle and Hørmann, 1971; Hormann and Eberle, 1971; Patchett, 1970; Ramsteiner *et al.*, 1971; Westlake *et al.*, 1970). Also, analysis of atrazine residues in a field soil and comparison of several extraction methods using an electron capture detector (ECD) have been reported (Purkayastha, 1971).

The purpose of the present work was to compare the capabilities of ECD and CCD for the residue analysis of *s*-triazine herbicides in water, soil, and corn samples by

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Table I. Gas Chromatographic Conditions Used

	CCD	ECD
Instrument	Microtek MT 220 fitted with Tracor Model C321 CCD	Pye Series 104 Model 74
Detector operation	N mode of Coulson Conductivity Detector	Pulse mode of ^{63}Ni (10 mCi) electron capture detector
Liquid phase	5% OV-17	3% OV-225
Support	Gas Chrom Q, 80/100	Gas Chrom Q, 80/100
Column	Glass, 6 ft × 6 mm	Glass, 5 ft × 6 mm
Flow rates		
Carrier gas	60 ml/min (He)	86 ml/min (N_2)
Sweep gas	60 ml/min (He)	None
Reducing gas	100 ml/min (H_2)	None
Temperatures		
Column	180, 195, or 225°	209°
Injection port	230°	
Transfer line	230°	
CCD Pyrolyzer	850°	
Detector	Room temperature	283°
Attenuation	1	2×10^{-10} A
Voltage	30 V (bridge)	>47 V (pulse amplitude) 0.75 μsec (pulse width) 15 μsec (pulse period)
Chart speed	0.5 in./min	0.5 in./min
Recorder	Varian A-25 (0.5 sec, 1 mV)	Honeywell (1 sec, 1 mV)
Sensitivity 50% f.s.d. given by	7 ng of atrazine (at 225°)	2.6 ng of atrazine (response varied with detector conditions)

gas chromatography. Atrazine, the most widely used *s*-triazine, was selected for the residue study and the experiments were conducted with fortified samples, mostly in the range of the 0.02 to 2.0-ppm level. The other *s*-triazines were studied only for their gas chromatographic characteristics.

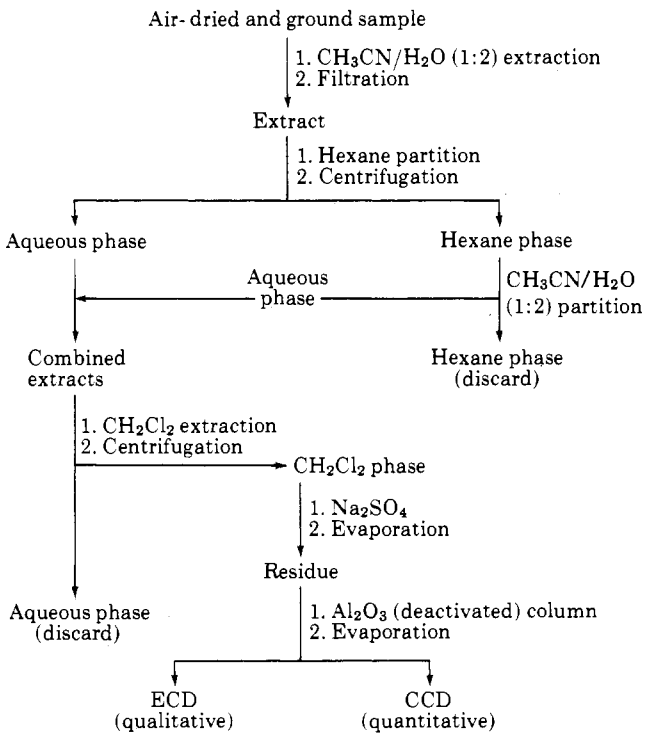


Figure 1. Analysis of atrazine residues in corn.

The residue methods developed for atrazine are believed to be applicable, with some modifications, to other *s*-triazines.

EXPERIMENTAL SECTION

The gas chromatographic conditions used in the present investigation for the Coulson Electrolytic Conductivity Detector (CCD) and Electron Capture Detector (ECD) are shown in Table I.

In addition to the OV-17 and OV-225 columns, 3% Carbowax 20M and 5% Reoplex-400 were also studied in the present work.

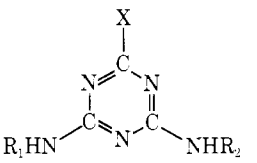
The CCD responses and relative retention times (parathion = 1) of the triazines on these latter columns have been included in Table II. For the relative retention times and half-scale deflection values of these compounds on OV-1 and OV-17 columns using both ECD and CCD, the readers are referred to the article by Cochrane and Wilson (1971).

ANALYTICAL PROCEDURES

Water Samples. The water samples (100–500 ml) were extracted by shaking with methylene chloride (100 ml × 2) in a separatory funnel. The extracts were dried (Na_2SO_4), concentrated to small volume on a rotary film evaporator, and analyzed with and without cleanup. The cleanup was done by chromatography on a deactivated (13% H_2O) alumina column (25 g). The column was first eluted with 2% diethyl ether in carbon tetrachloride (100 ml) which was discarded, and then with 6% diethyl ether in carbon tetrachloride (200 ml); the second eluate contained atrazine.

Soil Samples. The soil samples (10–30 g) were extracted for 90 min in a mechanical shaker with acetonitrile-water (9:1) (100 ml) at room temperature. Aqueous acetonitrile (35%) was also studied. Solvents such as ace-

Table II. Relative Retention Times and CCD Detector Response of Some s-Triazines



s-Triazine	Structure X	Structure R_1	Structure R_2	5% Reoplex-400 ½ f.s.d. (ng)	5% Reoplex-400 R_p[a]	3% Carbowax 20M ½ f.s.d. (ng)	3% Carbowax 20M R_p[b]
Prometone	OMe	*i*-Pr	*i*-Pr	20	0.35	10	0.35
Atratone	OMe	Et	*i*-Pr	20	0.42	15	0.43
Propazine	Cl	*i*-Pr	*i*-Pr	20	0.51	10	0.48
Atrazine	Cl	Et	*i*-Pr	20	0.60	12	0.63
Prometryne	SMe	*i*-Pr	*i*-Pr	30	0.67	15	0.71
Simazine	Cl	Et	Et	25	0.70	15	0.82
Ametryne	SMe	Et	*i*-Pr	25	0.78	20	0.91
Bladex	Cl	Et	C_3H_6CN	140[c]	0.94	140[c]	1.04
Outfox	Cl	*i*-Pr		50	1.06	50	1.21
Sencor (BAY 94337) 4-Amino-3-methylthio-6-*tert*-butyl-1,2,4-triazin-5-one				70	1.30	50	1.57

R_p = retention time relative to parathion.

[a]Retention time of parathion was 14.5 min at column temp 195°. [b]Retention time of parathion was 9.8 min at column temp 195°. [c]Large amounts for ½ f.s.d. due to long retention time at 195° (50 ng for ½ f.s.d. at 225°), and on-column decomposition to an unknown product, R_p = 4.6 (5% Reoplex-400); R_p = 3.4 (3% Carbowax 20M). Ratio of peak areas of Bladex to breakdown product = 5.22.

tone and methanol were also found to be good extractants. With these two latter solvents a Goldfisch extractor (Fischer Scientific Co.) was used. Where acetonitrile extraction was used, the mixture was filtered under suction through a Hyflo Super-Cel bed and the filter bed was washed with 35% aqueous acetonitrile (100 ml). The extract was partitioned with methylene chloride (100 ml × 2). The phases were allowed to separate and the methylene chloride layer was collected and dried with anhydrous sodium sulfate. The solvent was removed (rotary film evaporator) after filtration and made up to a small volume (1 ml) and analyzed by CCD without cleanup or analyzed by ECD after cleanup. The cleanup was performed as follows. The residue left after the evaporation of methylene chloride was dissolved in carbon tetrachloride (10 ml) and the solution was transferred to a basic alumina column (Aluminum oxide W200 basic, Woelm, equilibrated to contain 13% H_2O, and previously washed with hexane, 25 g, 23 mm × 70 mm). The column was first eluted with 2% diethyl ether in carbon tetrachloride (100 ml) and then with 6% diethyl ether in carbon tetrachloride (250 ml). The first eluate was discarded, while the second eluate was evaporated to dryness and taken up in diethyl ether (30 ml). The ether solution was evaporated to dryness (air current) and dissolved in hexane (10 ml), and the volume of solution was reduced to 300 μl (in a centrifuge tube by air current). Aliquot of this solution (1 μl or more) was injected into the gas chromatograph for ECD determination.

Where a solvent other than acetonitrile was used for extraction, the extract was evaporated to dryness (rotary film evaporator) and the residue was dissolved in 35% aqueous acetonitrile, and further processing was done as described earlier.

Corn Samples. The experimental details for the corn samples are very similar to those described for soil samples. The analysis of atrazine residues in corn is given in Figure 1.

The air-dried and ground sample (5 g) was extracted with 35% aqueous acetonitrile with mechanical shaking for 90 min and then filtered under suction through a Hyflo Super-Cel bed. The filtrate was partitioned with hexane and the aqueous phase was collected. The hexane phase was re-partitioned with aqueous acetonitrile. (Corn samples were also analyzed by CCD successfully without the hexane partition step.) All the aqueous phases were combined and extracted with methylene chloride. The organic phase was dried (Na_2SO_4), concentrated, and cleaned up by chromatography on deactivated (13% H_2O) alumina column. The CCD determination can be made on this solution. But this solution is not free from interferences for ECD work. Only qualitative results have been obtained with ECD even after an additional tlc clean-up step.

RESULTS AND DISCUSSION

From Table II it can be observed that most of the compounds give a 50% f.s.d. (full-scale deflection) in the 10–30 ng range, with the exception of Bladex (140 ng), Outfox (50 ng), and Sencor (70 ng). The high value for

Table III. Detection of Atrazine Residues in Water[a] by Electron Capture Detector with Cleanup[b]

Fortification level, ppm	Atrazine added, μg	Atrazine found, μg	% recovery
2.00	200.0	200.0	100
2.00	200.0	185.0	93
2.00	200.0	195.0	98
2.00	200.0	200.0	100
0.05	5.0	4.7	94[c]
0.05	5.0	4.3	86[c]
0.05	5.0	4.3	86[c]
0.05	5.0	4.7	94[c]
0.02	2.0	1.9	95[c]
0.02	2.0	1.8	90[c]
0.02	2.0	1.7	85[c]

[a]100 ml of water was extracted. [b]Samples were extracted with dichloromethane and cleaned up by alumina column. [c]Interfering peak present.

Table IV. Determination of Atrazine Residues in Water[a] by Electron Capture Detector and Coulson Conductivity Detector without Cleanup

Fortification level, ppm	% recovery	
	ECD	CCD
0.00[b]	0	0
0.01[b]	46[c]	101
0.01[b]	82[c]	103
0.01[d]	54[c]	94
0.01[d]	64[c]	106
0.00[e]	0	0
0.01[e]	ND[f]	95

[a]Samples (515 ml) were extracted with dichloromethane. [b]Tap water. [c]Interfering peak present. [d]Distilled water. [e]Ottawa River water. [f]ND, not determined.

Table V. Determination of Atrazine Residues in Soil without Cleanup by Coulson Conductivity Detector

Fortification level, ppm	Soil, g	Atrazine added, μg	Atrazine found, μg	% recovery	
				Apparent	Corrected
0.00	10.0	0.00	0.00	0	0
0.00	13.0	0.00	0.06	0	0
0.02	13.0	0.26	0.36	138	115
0.52	10.0	5.15	5.30	103	103

Samples were extracted with aqueous acetonitrile.

Bladex is probably due to on-column decomposition. It can be further observed that higher sensitivities have been obtained with the Carbowax column in comparison to the Reoplex column for most of the compounds.

In their study on OV-17 and OV-1 columns, Cochrane and Wilson (1971) found the chlorine-containing triazines (simazine, atrazine, propazine, Bladex, and Outfox) to have the 50% f.s.d. figures in the range of 1–2.5 ng using ECD, while these values were 7–15 ng using CCD. On the two OV columns used they did not observe any decomposition of Bladex.

In Table III the results on the recovery study of fortified water samples by electron capture gas chromatography have been summarized. At the 2-ppm level the recovery was quantitative and no interfering peaks could be observed, but at the lower levels although the recovery was good, some peak-overlapping interference was experienced even with cleanup.

For example, Ottawa River water fortified with 0.01 ppm of atrazine was analyzed by ECD after cleanup and it showed a good detectable peak for atrazine.

Ottawa River water, distilled water, and laboratory tap water were analyzed and compared without cleanup by both ECD and CCD. All these water samples were fortified at the 0.01-ppm level with atrazine and the results are given in Table IV. Variable recoveries (46–82%) were obtained with ECD, whereas CCD consistently gave quantitative recoveries.

Table VI. Determination of Atrazine Residues in Soil after Cleanup by Coulson Conductivity (CCD) and Electron Capture (ECD) Detectors

Soil, g	Atrazine added, μg	Atrazine found, μg		% recovery	
		CCD	ECD	CCD	ECD
10.0	2.30	2.32	2.05	101	89
10.0	2.30	2.00	2.02	87	88
10.0	2.30	2.46	1.77	107	77
10.0	2.30	2.07	2.02	90	88
10.0	2.30	2.32	2.07	101	90
20.0	0.44	0.52		119	
20.0	0.44	0.42		95	

Samples were extracted with acetone in a Goldfisch apparatus and cleaned up by chromatography on deactivated basic alumina column.

Table VII. Determination of Atrazine Residues in Corn[a] by Coulson Conductivity Detector

Fortification level, ppm	Corn, g	Atrazine added, μg	Atrazine found, μg	% recovery
0.00	5.0	0.00	0.00	0
0.00	5.0	0.00	0.00	0
0.02	6.0	0.13	0.10	75
0.02	6.0	0.13	0.13	100
0.05	5.0	0.26	0.21	80
0.05	5.0	0.26	0.19	72
1.03	5.0	5.15	3.23	63
1.03	5.0	5.15	3.46	67
2.06	5.0	10.30	10.90	106
2.06	1.3	2.58	1.80	70[b]

Samples were extracted with aqueous acetonitrile and cleaned up by chromatography on alumina column.

[a]Air-dried corn plant was powdered in a mill (dry matter = 94.5%). [b]Cleaned up by silica gel column.

The recovery of atrazine was very good (97%) from a soil sample containing 0.52 ppm of atrazine by ECD. In this experiment the acetonitrile–water (9:1) extraction was used, followed by methylene chloride partition and column chromatography on basic alumina. The background interference for the same soil, as ascertained by CCD, was 0.2 μg per 10-g sample (*i.e.*, 20 ppb). Quantitative recovery of atrazine at the fortification levels of 0.02 and 0.52 ppm was obtained by CCD without cleanup (Table V). The recovery value of 138% has been corrected to 115% by subtracting the interference level observed in the blank soil.

Good recovery (115%) for a soil sample was also obtained at the 0.02 ppm of atrazine concentration by CCD after the usual cleanup using aqueous acetonitrile extrac-

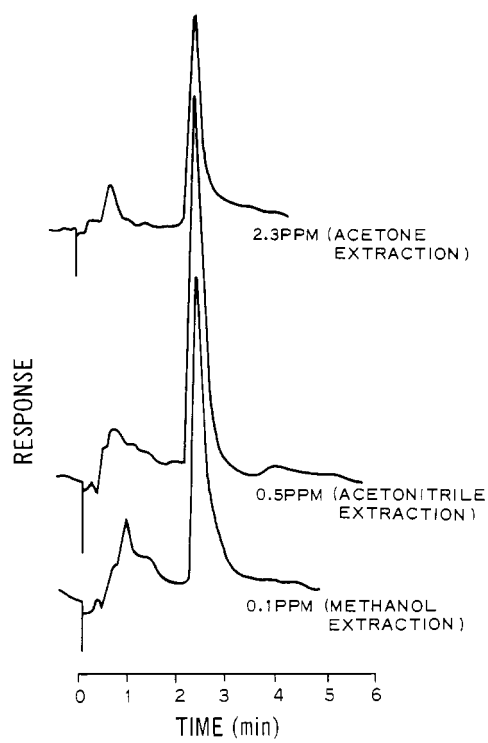


Figure 2. Chromatograms of fortified soil extracts containing atrazine as detected at 225° by CCD after cleanup.

Table VIII. Status of ECD and CCD for the glc Analysis of Atrazine Residues

Substrate	Without cleanup		With cleanup	
	ECD	CCD	ECD	CCD
Water	Semiquantitative	Quantitative	Quantitative	Quantitative
Soil	Not detectable	Quantitative	Quantitative	Quantitative
Corn	Not detectable	i. Not detectable[a] ii. Semiquantitative[b]	Qualitative	Quantitative

[a]Acetonitrile extraction. [b]Hexane extraction.

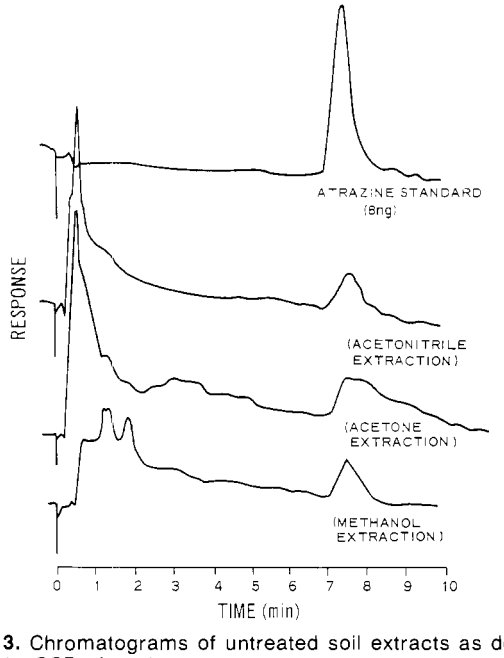


Figure 3. Chromatograms of untreated soil extracts as detected at 185° by CCD after cleanup.

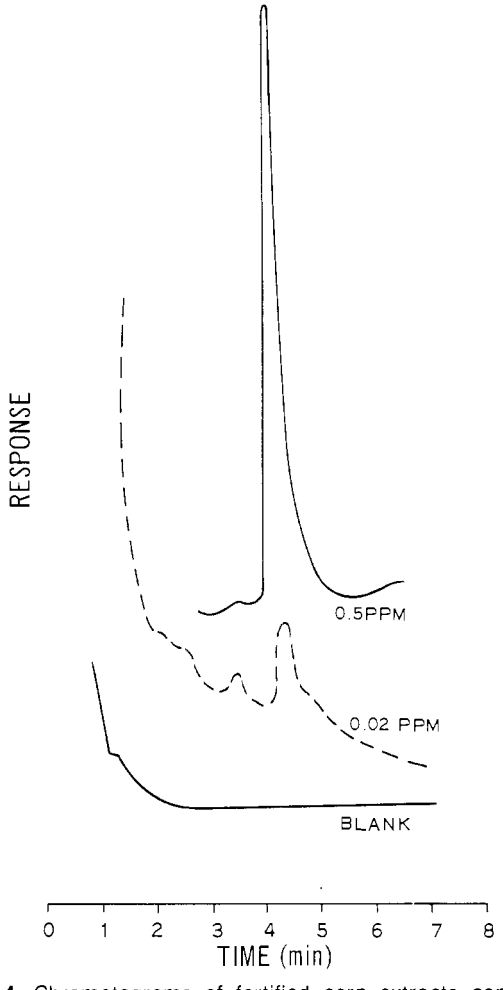


Figure 4. Chromatograms of fortified corn extracts containing atrazine as detected at 200° by CCD after cleanup.

tion. The gas chromatographic separation was done on a 5% Reoplex-400 on Chromosorb W column. The soil interference was found to be in the range of 5 to 30 ppb.

Figure 2 shows the chromatograms of cleaned-up soil extracts by CCD at 2.3, 0.5, and 0.1-ppm fortification levels extracted by acetone, acetonitrile, and methanol, respectively.

We have compared the recovery data obtained with CCD and ECD on cleaned-up soil extracts (Table VI). At the 0.23-ppm level, the recoveries with the two detectors were comparable and showed good reproducibility, but at the 0.02-ppm level, while the CCD results were good, we could not perform ECD analysis due to large interferences from the soil.

Figure 3 shows the chromatograms of the untreated soil extracts obtained by CCD. Three solvents (methanol, acetone, and acetonitrile) were used and the extracts were cleaned up. The field soil used in this study showed an interference level of about 20 ppb by the CCD.

With corn samples the CCD recovery results (Table VII) were satisfactory at the 0.02, 0.05, 1.03, and 2.06-ppm level. Cleanup by silica gel (Davison, 60–200 mesh, activated) column was also found adequate. The eluting solvent was hexane–acetone (2:1). Figure 4 shows typical CCD chromatograms of corn samples obtained at various fortification levels (blank, 0.02 and 0.5 ppm).

In Table VIII an attempt has been made, on the basis of the present study, to evaluate the status of ECD and CCD for the analysis of atrazine residues in water, soil, and corn. Quantitative determination of atrazine is possible with CCD for water and soil samples with or without cleanup.

Water and soil samples can be analyzed quantitatively by ECD only after cleanup. Quantitative analyses of corn samples are possible only by CCD after cleanup.

It can be concluded, therefore, that the CCD has a wider application in the analysis of triazines than the ECD. The ECD required cleanup in all cases studied, whereas CCD analysis could be made without cleanup in the case of water and soil samples.

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Determination of D-048 [1-(2-Butynyl)-1-(*p-tert*-butylphenoxy)-2-butyl Sulfite] in Cottonseed

James M. Devine[1]

A gas chromatographic method is described for the determination of D-048 [1-(2-butynyl)-1-(*p-tert*-butylphenoxy)-2-butyl sulfite] residues in cottonseed, cottonseed meal, and cottonseed oil. After extraction and cleanup by acetonitrile partition and Florisil column chromatography, D-048 is identified using a flame photometric detector in the sulfur mode. Recovery of D-048 averaged 91 ± 9% from the various cottonseed samples fortified at levels ranging from 0.1 to 1.0 ppm. The method is sensitive to 0.2 ppm for cottonseed oil and 0.1 ppm for cottonseed and cottonseed meal.

D-048 [1-(2-butynyl)-1-(*p-tert*-butylphenoxy)-2-butyl sulfite] is a new insecticide being developed for use on cotton. It is a nonsystemic, selective acaricide, effective against motile stages of phytophagus mites (Uniroyal Bulletin, 1970). To obtain necessary residue data, an analytical method was needed to determine D-048 in cottonseed, cottonseed meal, and cottonseed oil. The method, employing a flame photometric detector, is described in this report.

MATERIALS

Apparatus. A Tracor MT-220 gas chromatograph, equipped with a flame photometric detector in the sulfur mode (394-nm filter), was employed for the analysis. The gas chromatographic column was 4 ft × 3 mm i.d., glass, packed with 11% DC-200 (2.5 MCS) on 60–80 mesh Gas Chrom Q previously coated with 0.01% Versamid 900.

Reagents. All solvents were reagent grade. No additional purification was necessary. Sodium sulfate was reagent grade, anhydrous. Florisil (Fisher F-100), 60–100 mesh, was heated overnight at 130° and cooled in a desiccator before use. Addition of 2% moisture was needed to deactivate the Florisil for proper elution.

ANALYTICAL PROCEDURE

Sample Preparation and Extraction. Cottonseed and cottonseed meal were chopped in a Wiley Mill equipped with a 2-mm sieve. No preparation was necessary for the oil.

A 50-g sample of ground cottonseed was extracted with 200 ml of hexane and 50 g of sodium sulfate for 3 min in a Waring blender. Due to a greater amount of lint, some cottonseed samples may need a larger volume of solvent for adequate extraction. The homogenate was filtered through a coarse porosity fritted Buchner funnel using vacuum. The volume of recovered solvent was measured and the extract was quantitatively transferred to a 300-ml round-bottomed flask. The hexane was removed on a rotary vacuum evaporator at 40° and the oil was transferred to a 500-ml separatory funnel with a total of 40 ml of hexane. The extract was then processed through the acetonitrile partition and Florisil cleanup steps.

A 10-g sample of cottonseed oil was transferred to a 500-ml separatory funnel with a total of 40 ml of hexane. The mixture was then processed through the acetonitrile partition and Florisil cleanup steps.

A 50-g subsample of cottonseed meal was blended with 200 ml of acetonitrile for 3 min in a Waring blender. The homogenate was filtered through a coarse porosity fritted Buchner funnel using vacuum. A 100-ml aliquot of the filtrate was passed through 50 g of sodium sulfate. The sodium sulfate was then rinsed with two 20-ml aliquots of acetonitrile. The acetonitrile was removed on a rotary vacuum evaporator at 40°. The residue was taken up in acetone for analysis.

Acetonitrile Partition Step. The hexane–oil mixture was extracted with two 100-ml portions of acetonitrile (previously saturated with hexane), shaking for 1 min each time. The combined acetonitrile extracts were evaporated just to dryness on a rotary vacuum evaporator at 40°. The residue was taken up in 10 ml of benzene and processed through the Florisil column.

Table I. Summary of D-048 Recoveries from Cottonseed and Products

Fortification, ppm	% Recovery		
	Cottonseed	Oil	Meal
1.0	94	76	99
0.5	80	86	98
0.2	85	100	90
	100	108	80
	85	96	
0.1	85		96
Average:	88	93	93

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