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TITLE: ANALYSIS OF KRUMMRICH SAMPLES FOR PCB's, 2,3,7,8-TCDD, PHLNOLIC COMPOUNDS AND OTHER MAJOR ORGANICS

AUTHORS: Fred D. Hileman
B. Mason Hughes
Sue L. Mazer

ABSTRACT: Concrete and insulation samples were analyzed for 2,3,7,8-TCDD, PCBs, phenolic compounds, and other major organics. The results of these analyses are shown in this report.

Technical Approval: Joseph J. Brooks
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Approved By:
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Environmental Sciences Center
Service Project Manager

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Approval Date: 12-15-83

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TITLE: ANALYSIS OF KRUMMRICH SAMPLES FOR PCB's, 2,3,7,8-TCDD PHENOLIC COMPOUNDS AND OTHER MAJOR ORGANICS

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ACKNOWLEDGEMENT

Contributions of the following Dayton Laboratory personnel to this project are gratefully acknowledge:

Analyses: F. D. Hileman
David E. Kirk
B. M. Hughes
S. L. Mazer
D. McKenzie
C. Trang

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[redacted]

INTRODUCTION

Six insulation samples and two concrete samples were received for the analysis of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) polychlorinated biphenyls (PCBs), specific phenolic compounds including santophen (o-benzyl-p-chlorophenol) and generally for other high-level constituents which were present. All samples were assigned MRC Log Number 1-83-08-05-01.

SAMPLE PREPARATION FOR TCDD

Samples were prepared by weighing out an aliquot and if the sample was for quality control purposes the sample was spiked with 2,3,7,8-TCDD. All samples were spiked with $^{13}C_{12}$-2,3,7,8-TCDD to serve as a surrogate for quantitative analyses. The samples were extracted with petroleum ether. This ether extract was then decanted from the sample and partitioned against concentrated sulfuric acid and then against organic free water and then dried over anhydrous sodium sulfate.

The entire extract was then passed through a multiphase column consisting of (top to bottom) 2 grams of silica, 4 grams of 40% concentrated sulfuric acid on silica, one gram of silica, 2 grams of 36% KOH on silica and finally one gram of silica. This column was flushed with an additional 50 mL of hexane with the entire effluent passing onto a Woelm B alumina column which retained the 2,3,7,8-TCDD. The Woelm column was eluted with 10 mL of 2% CH_2Cl_2/hexane to elute PCBs and other potential interferents and this fraction was discarded. This column was then eluted with 15 mL of 50% CH_2Cl_2/hexane which was collected and concentrated to 50 µL for the insulation samples and submitted for GC/MS analyses.

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In certain instances the samples required further clean up (particularly several of the insulation samples). The additional cleanup step that used involved chromatographing the samples on a column of 10% Carbopak C on Celite. This column was eluted with 2 mL hexane, then 1 mL 50% CH_2Cl_2/cyclohexane, then 1 mL of 75:20:5 methylene chloride/methanol/benzene to remove interfering compounds with the Carbopak retaining the 2,3,7,8-TCDD. The TCDD was then eluted from the column with 2 mL of toluene and then again concentrated to 10 µL and resubmitted for GC/MS analyses.

ANALYSES FOR TCDD

The samples were analyzed on a Hewlett Packard 5985B GC/MS system using either a DB-5 or SP-2330 capillary column. The DB-5 column was used for the initial screening of samples since these columns are more durable. For those samples that had detectable levels of 2,3,7,8-TCDD or had serious interferences the samples were put through the additional clean up (the Carbopak C column) mentioned previously and then analyzed on the SP-2330 column. The SP-2330 column is considerably less durable than the DB-5 column but has the separating ability to isolate the 2,3,7,8-TCDD from the other 21 TCDDs.

RESULTS FOR TCDD

The results of the analyses are given in Table 1. The values for most samples are listed as none detected (ND) at a specified detection limit. In certain cases the 2,3,7,8-TCDD was detected and these results have been listed without the ND. Most notable among these is the "New Insulation, Dept 237". The chromatographic trace for this analysis is shown in Figure 1. Note that the 2,3,7,8-TCDD is only one of many TCDDs observed in this sample. Additional portions of this insulation are being sent for additional analyses. Contrast this to the analysis of the "Dirty

Insulation, Dept 239° shown in Figure 2. Here the 2,3,7,8-TCDD was undetectable but many additional TCDDs were also observed.

TABLE 1. ANALYSIS OF CONCRETE AND INSULATION FOR 2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN

Sample	ppb 2,3,7,8-TCDD found	ppb 2,3,7,8-TCDD added
Dirty Insulation Dept 237	ND (<.7)	-
Dirty Insulation Dept 237 (Replicate)	ND (<.7)	-
Residue Equipment Insulation Dept 237	ND (<.8)	-
Clean Insulation Dept 237	ND (<.7)	-
New Insulation Dept 237	30	-
Dirty Insulation (Soft) Dept 239	ND (<.7)	-
Dirty Insulation (Soft) Dept 239 (Spike)	1.0	0.6
Dirty Insulation (Soft) Dept 239 (Spike)	13	9
Dirty Insulation (Hard) Dept 239	2.1	-
Clean Insulation Dept 239	ND (<1.0)	-
Concrete MRC (Blank)	ND (<.5)	-
Concrete MRC (Spike)	2.4	1.9
Concrete MRC (Spike)	24	19
Concrete Dept 237	ND (<.5)	-
Concrete Dept 237 (Replicate)	ND (<.5)	-
Concrete Dept 239	ND (<.5)	-
Concrete Dept 239 (Replicate)	ND (<.7)	-

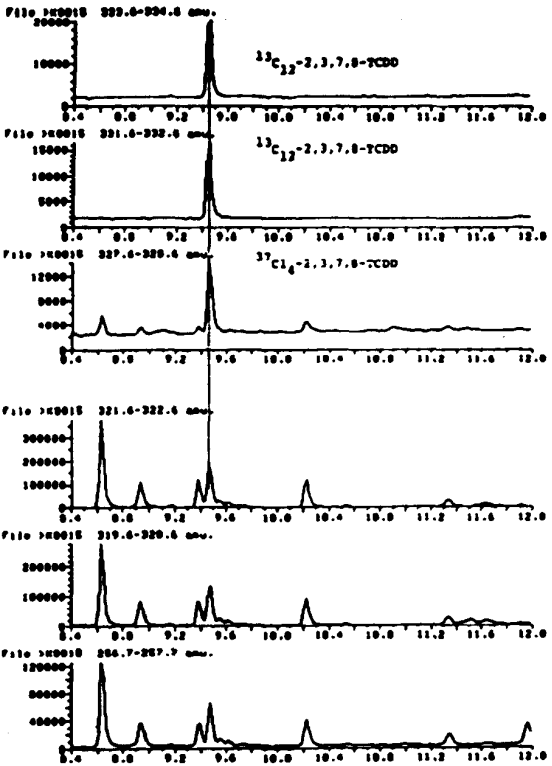

Figure 1. Analysis of "New Insulation, Dept 237" showing the chromatographic traces for the isotopically labeled 2,3,7,8-TCDDs on the top three traces and the chromatographic traces for the native 2,3,7,8-TCDD on the bottom three traces.

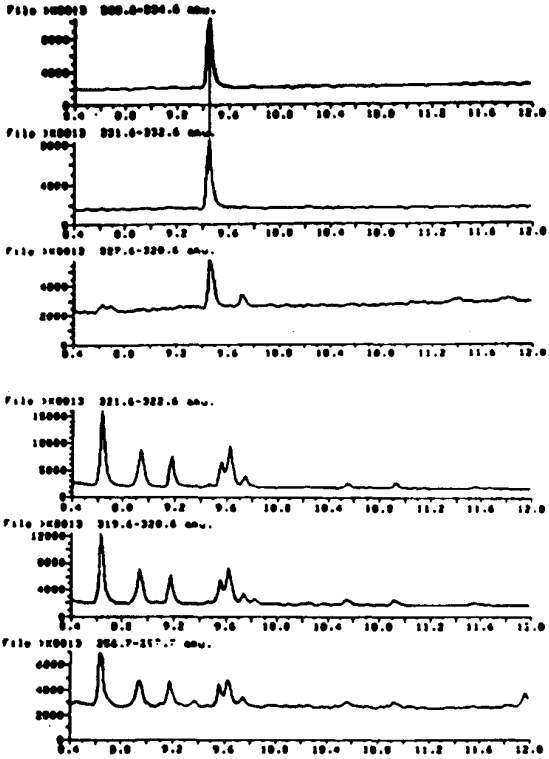

Figure 2. Analysis of "Dirty Insulation, Dept 239" showing the low level of 2,3,7,8-TCDD and the presence of other TCDDs.

The following section reports on the analyses performed for possible PCB concentrations.

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ANALYSIS REPORT

DATE: October 27,1983

LOG NUMBER: 1-83-08-05-01 PROJECT NO: 483.24818

TO:
Ivory (Concrete Samples from E. Fay)

FROM:
Environmental Sciences Center
Monsanto Research Corporation
1515 Nicholas Road
Dayton, Ohio 45418

Phone No: (513) 268-3411

Title: Analysis of Concrete Samples for PCBs.

Standard Sampling Method No(s): N/A

Standard Extraction Method No(s): PCEX01

Standard Analysis Method No(s): ECAN02

Final Report File No(s): A24818

Table File No(s): B24818

Table 1 summarizes the results of analyses for Aroclor formulations in Concrete Samples submitted by E. Fay. Attached are also the ESC Extraction and Analysis Methods used in this study. Note that Dept No 23⁰ Concrete Sample contains more than 50 us/g Aroclor 1248 and thus must be treated as PCB containing Material).

ANALYST(S): B. M. Hughes, D. McKenzie and C. Trang

ANALYST APPROVAL: B. M. Hughes

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TABLE 1. Results of Capillary GC/EC Analyses of Unknown Drum Sample from the Queeny Plant.

ESC Project No: 483.24018 Date: October 17, 1983

ANALYTE	ESC ANALYTICAL METHOD	LOD	SAMPLE IDENTIFICATION: Dept No 239 Concrete Sampled by E. Fay	SAMPLE IDENTIFICATION: Dept No 237 Concrete Sampled by E. Fay	SAMPLE IDENTIFICATION: MRC Concrete Method Blank
			CONCENTRATION (ug/g)		
Aroclor 1248	ECAN02	1.0	71.6	ND	ND
Rng of Values	-	-	98.3	--	--
No of Values	-	-	27	--	--
Std Deviation	-	-	21.2	--	--
% Rel Std Dev	-	-	34.0	--	--
SURROGATE SPIKING COMPOUND		AMOUNT ADDED (ug/g)		RECOVERY (%)	
CL5-benzene	ECAN02	1.0	87	99	93
DOCUMENTATION					
ESC Extraction Method			PCEX01	PCEX01	PCEX01
Sample Amt extracted (g)			1.012	1.064	0
Extract Dilution			111	111	111
ESC Extract No.			PCB197	PCB198	PCB195
Process File Name			BP180	BP177	BP174

EXPLANATION OF ABBREVIATIONS:

LOD = limit of detection in ug/g.
ND = none detected with a detection limit given by the LOD value.
NA = not analyzed.
Rng of Values = Total range of Aroclor formulation concentrations measured from individual PCB isomers. The number of isomers used in this calculation is given by No. of Values
No of Values = Number of PCB isomers used to measure Aroclor concentration
% Rel Std Dev = [(Std Deviation)/(Concentration)] X 100%.

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The following pages describe Standard Extraction Methods and Standard Analysis Methods used for this project.

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ESC Standard Extraction Method (3EM)
TITLE: PCBs in Various Matrices

ESC Standard Extraction Method No: PCEX01

MATRI(X).(CES):	TYPICAL SAMPLE SIZE (g)	TYPICAL EXTRACT VOLUME (ml)
Still bottoms, soils, beef fat, beef serum, whole milk, beef feces	0.5 - 5.0	1

TYPICAL ANALYTES: Aroclors 1232, 1248, 1254, 1260, 1262, trichlorobiphenyl isomers through octachlorobiphenyl isomers

ESC Standard Analysis Method Nos: ECAN01, ECAN02

INTERFERENCES: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl

TYPICAL SURROGATES	TYPICAL RECOVERIES (%)
hexachlorobenzene	50 - 120
2,4,6-trichlorobiphenyl	50 - 120

APPARATUS AND MATERIALS:

Burdick and Jackson hexane, benzene and methylene chloride for sample extraction.
Concentrated sulfuric acid for sample digestion or sample clean-up.
Silica gel columns impregnated with potassium permanganate, sulfuric acid, and potassium hydroxide.
Activated basic alumina.
Disposable glassware.

LIMITATIONS: Method applies only to matrices which do not contain non-hydrolizable electron capturing species in the trichlorobiphenyl through octachlorobiphenyl retention time region.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATES OF METHOD CREATION AND UPDATES: 10/25/83

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ESC Standard Analysis Method (SAM) — ESC Standard Analysis Method No:
TITLE: Aroclor Formulations in Solvents — ECAN02

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5880 Capillary GC/EC interfaced to an HP-3356-LAS Laboratory Data System

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TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Aroclor formulations 1232, 1248, 1254, 1260 and 1262	0.1	30	30

INTERFERENCES AND LIMITATIONS: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl are potential interferences. Exact retention times of most likely PCB isomers can be obtained from the analysis of Aroclor 1248, Aroclor 1254 or Aroclor 1260 formulations.

CHROMATOGRAPHIC COLUMN: J&W DB-5 (0.25 uM) on fused silica wide bore (0.32mm)

COLUMN TEMPERATURE PROGRAM: 130(1)/4/300 — INJECTION TYPE: Splitless

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CALIBRATION AND STANDARDIZATION: Use internal standard quantitation technique and calibrate instrument response factors from authentic Aroclor formulation standards. Use a minimum of 10 PCB isomers for the determination of formulation identity and quantity.

CALCULATIONS: Formulation concentration = (isomer area)/(isomer RRF)

where: RRF is the relative response factor for the appropriate PCB isomer in a given formulation. Formulation concentration is obtained from the average of at least 10 such calculations.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATES OF METHOD CREATION AND METHOD UPDATES: 10/25/83

The folowing section reports on the analyses performed for the specific phenolic compounds requested.

EXTRACTION METHOD

The analysis method was similar to EPA Method 625 (1), and consisted of methylene chloride extraction followed by GC/MS. Samples were weighed into 40-mL septum-top vials, spiked with phenol-d_6, 2-chlorophenol-d_4, pyrene-d_{10}, 1,2-dichlorobenzene-d_4, and chrysene-d_{12} surrogate spike compounds, extracted by sonication for 0.5 hour with one 20 and two 10-mL portions of methylene chloride, and concentrated to an appropriate final volume. A sample weight of 10 g was desired to achieve low detection limits; however insufficient sample was present in many cases so various weights were used. The extracts were concentrated to 1-mL final volume unless high levels of organics were present. Additional extraction steps were performed in cases where the methylene chloride was easily absorbed into the sample matrix. Table 1 summarizes the particulars for the workup of each sample.

ANALYTICAL METHODS

The extracts were analyzed on a 5985A GC/MS system equipped with a 30 m fused silica capillary column coated with DB-5. Samples were co-injected in the splitless mode with anthracene-do internal standard and the column was programmed at 8°C/min from 50°C to 300°C. The mass spectrometer was scanned from 10 to 500 amu.

Two basic types of data reduction were performed. The first involved searching the data for major ions of the acid priority pollutants and Santophen. The acid priority pollutants were quantified using external standards, with internal standard normalization. The Santophen was quantified relative to the anthracene-d_{10} internal standard, since an external standard was not available. The second type of data reduction involved identification and semi-quantitation of the major components in each extract. The identification was first attempted using a computerized library search of over 30,000 spectra. The possibilities generated from the search were compared with the sample spectra to arrive at the best

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spectral match. In the many cases where the library search did not result in a spectral identification, a possible compound structure was postulated based on the principles of mass spectral fragmentation. These major components were semi-quantified relative to the anthracene-d_{10} internal standard.

RESULTS AND DISCUSSION

Results for the analysis of the concrete and insulation for acid priority pollutants and Santophen are given in Table 2. The priority pollutants were quantified using major ion areas of external standards, and are accurate to ±30% assuming 100% recovery. The Santophen, because a standard was not available, was quantified using the total ion area of the anthracene-d_{10} internal standard. This method provides results accurate to within a factor of two or three. The identification of Santophen was done by prediction of the major ions based on mass spectral fragmentation principles and then searching the data for a peak having the predicted mass spectrum. While we have confidence in this identification, it is not a positive one since a standard was not available for confirmation.

Recoveries of the deuterated spike compounds are provided in Table 3. These were calculated using major ion areas relative to a standard representing 100% recovery. In general, the recoveries fall into typical ranges for samples subjected to the Method 625 extraction protocol. However, the 2-chlorophenol-d_4 and phenol-d_5 recoveries were unusually low for both concrete samples, possibly reflecting some difficulty with this matrix. Unfortunately, insufficient sample was present to permit modified extraction protocols to be attempted. These low recoveries should be considered when the wide scan and priority pollutant data are evaluated. Since none of these data are corrected for recoveries, actual concentrations present may be much higher than the results reported.

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Results of the wide scan analyses are given on a per sample basis in Tables 4-13. The major organics reported in these tables were quantified relative to the anthracene-d_{10} internal standard, and are accurate to within a factor of 2 or 3, assuming 100% recovery in the extraction process. Some of the compounds listed in this table (e.g. phenol and 2,4,-dichlorophenol) were analyzed more accurately in the priority pollutant analysis (Table 2). Peak numbers in the tables correspond with peak numbers on Figures 1-10, which depict total ion chromatograms for these samples.

Examination of structures of some of the organics listed in Tables 4-13 led to the suspicion that polychlorinated biphenyls might also be present in some of the samples. The mass spectral data was searched for the major ions of the mono-through octa-chlorobiphenyls, and their presence was detected in the Concrete 239 sample. While no PCB's were detected in the other samples, detection limits ranged from about 0.5 to 50 µg/g per PCB isomer, depending upon sample weight and final extract volume.

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REFERENCE

1. EPA Guidelines Establishing Test Procedures for the Analysis of Pollutants, Proposed Regulations, Federal Register, Monday, December 3, 1979.

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TABLE 1. SUMMARY OF VARIABLES IN WORKUP OF CONCRETE AND INSULATION SAMPLES FOR GC/MS WIDE SCAN AND PHENOL ANALYSIS

Sample Identification	Sample wt. (g)	Approximate spike level (µg/g)	Final Extract Volume (mL)	Number of Extractions
MRC Concrete Blank	10.1	4	1	3
Method (glassware) Blank	0	4[a]	1	3
Concrete 237	10.1	4	1	3
Concrete 239	3.87	10	1	3
"New" Insulation 237	3.89	10	1	5
"Clean" Insulation 237	2.99	13	1	5
"Dirty Insulation 237	6.61	6	200[b]	5
"Clean" Insulation 239	1.25	32	1	3
"Dirty" Insulation 239	3.71	10	100[c]	5
Residue Equip Insulation 237	2.38	17	1	5

[a]Assumes 10 g sample size.

[b]Extract was actually concentrated to 20 mL, but required 1:10 dilution prior to analysis.

[c]Extract was actually concentrated to 10 mL, but required 1:10 dilution prior to analysis.

TABLE 7 ANALYSIS OF GRANDRICH CONCRETE AND INSULATION SAMPLES FOR ACID PRIORITY POLLUTANTS AND SURROGATES

PAGE 1

Item (Item)	Analyte	Method of Quantification	GMC Concrete Blank 1 2 3 Conc (ug/kg)	Method Blank (Gleason) Conc (ug/kg)	Concrete 737 Conc (ug/kg)	Concrete 739 Conc (ug/kg)	Concrete 740 Conc (ug/kg)	Spike Level (ug/kg)	%
7 8	2-Chlorophenol	MI/E	0.2	ND[4]	0.2	ND	0.2	0.57	85
10 9	2-Nitrophenol	MI/E	0.3	ND	0.3	ND	0.3	ND	ND
7 7	Phenol,	MI/E	0.2	ND	0.2	ND	0.2	5.5	55
11 2	2,4-Dimethylphenol	MI/E	0.3	ND	0.3	ND	0.3	ND	ND
11 7	2,4-Dichlorophenol	MI/E	0.3	ND	0.3	ND	0.3	4.8	ND
15 1	2,4,6-Trichlorophenol	MI/E	0.4	ND	0.4	ND	0.4	1.9	ND
14 0	4-Chloro-m-cresol	MI/E	0.3	ND	0.3	ND	0.3	4.0	ND
17 9	2,4-Dinitrophenol	MI/E	2.0	ND	2.0	ND	2.0	5.0	ND
19 3	4,6-Dinitro-o-cresol	MI/E	1.0	ND	1.0	ND	1.0	2.5	ND
21 5	Pentachlorophenol	MI/E	0.5	ND	0.5	ND	0.5	1.2	ND
10 1	4-Nitrophenol	MI/E	0.6	ND	0.6	ND	0.6	1.5	ND
23 5	p-Benzyl-p-chlorophenol	TI/I	0.05	ND	0.05	ND	0.1	Void	0.1

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TABLE 7 ANALYSIS OF GROUNDWATER FROM MW-20 AND TRIPLICATE SAMPLES FOR ACID PRIORITY POLLUTANTS AND SURROGATES[a]
PAGE 2

Ref. Time (Min)	Analyte	Method of Quantitation	Client Sample ID: MW-20 Conc. (µg/L)	Conc. (µg/Kg)	Spike Sample ID: MW-20 Conc. (µg/L)	Conc. (µg/Kg)	Client Sample ID: MW-20 Conc. (µg/L)	Conc. (µg/Kg)	Spike Sample ID: MW-20 Conc. (µg/L)	Conc. (µg/Kg)	Det. Limit[b] (µg/L)	Det. Limit[b] (µg/Kg)
7.0	2-Chlorophenol	BE/E	0.6	ND	50	2.0x10⁴	1.6	ND	50	ND	0.6	ND
10.9	2-Nitrophenol	BE/E	0.4	ND	50	ND	2.4	ND	60	ND	2.2	ND
7.7	Phenol	BE/E	0.6	ND	50	2.0x10⁴	1.6	ND	50	ND	0.8	ND
11.2	2,4-Dimethylphenol	BE/E	0.9	ND	50	ND	2.4	ND	60	ND	1.2	ND
11.7	2,4-Dichlorophenol	BE/E	0.9	2.9	50	2.0x10⁴	2.4	2.6	60	2.0x10⁴	1.2	ND
15.1	2,4,6-Trichlorophenol	BE/E	1.2	ND	120	ND	3.2	ND	100	1.0x10⁴	1.6	8
14.0	4-Chloro-m-cresol	BE/E	0.9	ND	50	ND	2.4	ND	60	ND	1.2	ND
17.9	2,4-Dinitrophenol	BE/E	4.0	ND	400	ND	16	ND	500	ND	8.0	ND
19.2	4,6-Dinitro-o-cresol	BE/E	3.0	ND	300	ND	8.0	ND	250	ND	4.0	ND
21.5	Pentachlorophenol	BE/E	1.5	ND	150	ND	4.0	ND	130	ND	2.0	ND
10.1	4-Nitrophenol	BE/E	1.0	ND	100	ND	4.0	ND	150	ND	2.0	ND
23.5	p-Benzyl-p-chlorophenol	TI/E	0.1	5.0	7.5	1.5x10⁴	0.2	86	7.0	1.4x10	0.1	0.04

a • Results were not corrected for recoveries of deuterated spike compounds (see Table 1)

b BE/E • Compound was quantified relative to major ions areas of external standards
TI/E • Compound was quantified relative to total ion area of internal standard

c • Limit of detection in µg/g Because varying sample weights and extract volumes were used, detection limits were calculated specifically for each sample

d ND • Not detected, with a detection limit given by the L.O.D. for that compound in the sample
e • Results were not corrected for recoveries of deuterated spike compounds (see Table 1)

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TABLE [illegible] - RECOVERIES OF DEUTERATED SPIKE COMPOUNDS IN [illegible] CONCRETE AND INSULATION SAMPLES

Det. Time (min)	Spike Compound	Method Blank	NBC Concrete Blank	% Recovery Concrete 23%	Concrete 23%	"New" Insulation 2%
7.4	Phenol-d_6	67.0	51.0	4.30	10.0	[illegible]
7.5	2-Chlorophenol-d_4	59.9	59.5	ND*	4.54	[illegible]
8.6	1,2-Dichlorobenzene-d_4	8[illegible].0	79.9	80.[illegible]	4.2	[illegible]
26.1	Pyrene-d_{10}	108.9	102.9	92.2	[illegible]	66.1
30.0	Chrysene-d_{12}	97.9	109.1	110.1	[illegible]	82.9

Det. Time (min)	Spike Compound	"Clean" insulation 23%	"Dirty" insulation 23%	% Recovery "Clean" insulation 23%	"Dirty" insulation 23%	Residue Equipment insulation 2%
7.4	Phenol-d_6	101.6	99.5	80.9	82.3	21.4
7.5	2-Chlorophenol-d_4	88.5	51.2	61.5	89.3	
8.6	1,2-Dichlorobenzene-d_4	50.5	67.3	14.4	71.2	[illegible]
26.1	Pyrene-d_{10}	56.9	35.0	82.5	61.0	78.0
30.0	Chrysene-d_{12}	97.9	37.3	127.2	62.1	97.5

*ND = not detected with a detection limit of 2% recovery

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TABLE 4. ORGANICS DETECTED IN GC/MS ANALYSIS OF NBC CONCRETE BLANK[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	24.23	Unknown phthalate (149, 150, 205, 223)	0.34
		TOTAL	0.34 μg/g

a = Results were not corrected for spike recoveries (see Table 3).

b = Identifications are tentative ones based upon library searches and spectral comparisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 0.3 μg/g.

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TABLE 5. ORGANICS DETECTED IN GC/MS ANALYSIS OF METHOD (GLASSWARE) BLANK[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (µg/g)[c]
1	24.23	Unknown phthalate (149, 150, 205, 223)	0.32
		TOTAL	0.32 µg/g

a = Results were not corrected for spike recoveries (see Table 3).

b = Identifications are tentative ones based upon library searches and spectral comparisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this samples was 0.3 µg/g. A 10 g sample weight was assumed.

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TABLE 6. ORGANICS DETECTED IN GC/MS ANALYSIS OF CONCRETE 237[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	3.92	Unknown (101, 59, 43)	3.4
2	12.23	Chlorophenol (isomer)	1.6
3	30.78	Unknown phthalate (149, 167, 150)	0.6
		TOTAL	5.6 μg/g

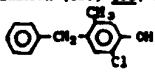
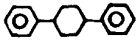
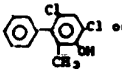
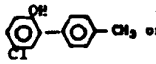
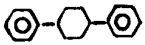
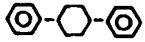
a = Results were not corrected for spike recoveries (see Table 3), nor were any background contaminants subtracted out (see Tables 4 and 5).

b = Identifications are tentative ones based upon library searches and spectral comparisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 0.5 μg/g.

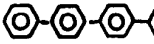
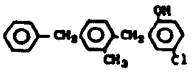
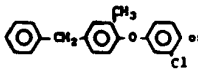
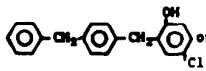
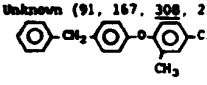
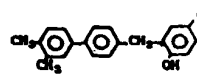
MONS 022700

TABLE 7. ORGANICS DETECTED IN GC/MS ANALYSIS OF CONCRETE 230[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	22.50	unknown (231, 232, 155, 105, possibly 	90
2	23.38	Terphenyl (isomer)	11
3	23.55	o-Benzyl-p-chlorophenol (Santophen)	7.0×10^2
4	23.77	Unknown (104, 91, 117, 236), possibly 	8.3
5	24.03	Unknown (174, 176, 252, 217), possibly 	6.6
6	24.13	Unknown (183, 218, 165), possibly 	12
7	24.70	Phenylbicyclohexyl (isomer)	85
8	24.87	Phenylbicyclohexyl (isomer)	33
9	25.15	Unknown (91, 104, 145, 236, possibly 	44
10	25.28	Phenylbicyclohexyl (isomer)	66
11	25.37	Unknown (91, 104, 117, 236), possibly 	73
12	25.78	Unknown (236, 167, 178, 180)	160
13	26.28	Unknown (236, 193, 178, 167)	110

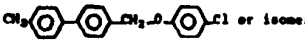
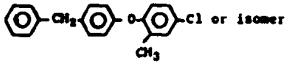
MONS 022701

TABLE 7. (continued)

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
14	26.57	Terphenyl (isomer)	22
15	27.03	Terphenyl (isomer)	13
16	30.75	Unknown, (312, 229, 228, 313), possibly  or isomer	5.7
17	30.92	Unknown (322, 91, 321, 152), possibly  or isomer	7.7
18	31.27	Unknown (229, 308, 91, 195), possibly  or isomer	51
19	31.57	Unknown (180, 91, 167, 308), possibly  or isomer	43
20	31.85	Unknown (91, 167, 308, 217), possibly  or isomer	8.6
21	32.15	Unknown (322, 321, 105, 77), possibly  or isomer	6.9

MONS 022702

TABLE 7. ORGANICS DETECTED IN GC/MS ANALYSIS OF CONCRETE 239[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
22	32.38	Unknown (91, 300, 195, 273), possibly 	7.0
23	32.77	Unknown (91, 167, 168, 217, 300), possibly 	14
		TOTAL	1.5×10^3 μg/g

a = Results were not corrected for spike recoveries (see Table 3), nor were any background contaminants subtracted out (see Table 4 and 5).

b = Identifications are tentative ones based upon library searches and spectral comparisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 5 μg/g.

MONS 022703

TABLE 8. ORGANICS DETECTED IN GC/MS ANALYSIS OF "NEW" INSULATION 237[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	24.23	Unknown phthalate (149,150, 205, 223)	1.8
		TOTAL	1.8 μg/g

a = Results were not corrected for spike recoveries (see Table 3), nor were any background contaminants subtracted out (see Tables 4 and 5).

b = Identifications are tentative ones based upon library searches and spectral comparisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 1.5 μg/g.

MONS 022704

TABLE 9. ORGANICS DETECTED IN GC/MS ANALYSIS OF "CLEAN" INSULATION 237[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	12.23	Chlorophenol (isomer)	2.8
2	23.42	o-Benzyl-p-chlorophenol (Santophen)	5.4
3	24.23	Unknown phthalate (149, 150, 205, 223)	1.2
		TOTAL	9.4 μg/g

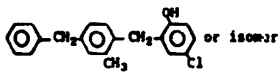
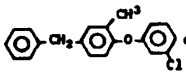
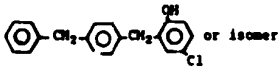
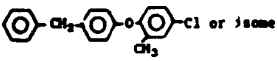
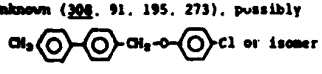
a = Results were not corrected for spike recoveries (see Table 3), nor were any background contaminants subtracted out (see Tables 4 and 5).

b = Identifications are tentative ones based upon library searches and spetral comperisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identificaions by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 1.0 μg/g.

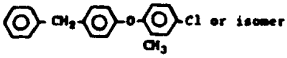
MONS 022705

TABLE 10. ORGANICS DETECTED IN GC/MS ANALYSIS OF "DIRTY" INSULATION 237[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	7.50	Phenol	8.5×10^3
2	7.55	Chlorophenol (isomer)	6.9×10^3
3	11.57	Dichlorophenol (isomer)	3.2×10^3
4	12.27	Chlorophenol (isomer)	6.0×10^3
5	23.42	o-Benzyl-p-chlorophenol (Santophen)	1.5×10^3
6	30.92	Unknown (322, 91, 321, 152), possibly  or isomer	1.8×10^3
7	31.37	Unknown (229, 308, 91, 195), possibly  or isomer	3.7×10^4
8	31.58	Unknown (180, 91, 167, 308), possibly  or isomer	9.7×10^3
9	31.88	Unknown (91, 167, 308, 217), possibly  or isomer	2.1×10^3
10	32.38	Unknown (308, 91, 195, 273), possibly  or isomer	2.5×10^3

MONS 022706

TABLE 10. (continued)

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
11	32.78	Unknown (91, 167, 168, 217, 308), possibly 	6.0×10^3
		TOTAL	8.5×10^4 μg/g

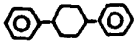
a = Results were not corrected for spike recoveries (see Table 3), nor were any background contaminants subtracted out (see Tables 4 and 5).

b = Identifications are tentative ones based upon library searches and spectral comparison or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 1.5×10^3 μg/g.

MONS 022707

TABLE 11. ORGANICS DETECTED IN GC/MS ANALYSIS OF "CLEAN" INSULATION 239[a]

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	12.23	Chlorophenol (isomer)	35
2	20.42	Phenylmethylphenol (isomer)	6.5
3	23.35	Terphenyl (isomer)	8.4
4	23.43	o-Benzyl-p-chlorophenol (Santophen)	88
5	24.27	Unknown phthalate (149, 150)	180
6	24.63	Phenylbicyclohexyl (isomer)	8.0
7	24.9	Phenylbicyclohexyl (isomer)	4.8
8	25.1	Unknown (91, 104, 145, 236), possibly  or isomer	5.6
9	25.22	Phenylbicyclohexyl (isomer)	8.8
10	25.68	Unknown (236, 178, 167, 193, 263)	13
11	26.20	Unknown (236, 193, 167, 178)	11
		TOTAL	3.7×10^2 μg/g

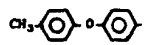
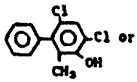
a = Results were not corrected for spike recoveries (see Table 3), nor were any background contaminants subtracted out (see Tables 4 and 5).

b = Identifications are tentative ones based upon library searches and spectral comparisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 5.5 μg/g.

MONS 022708

TABLE 12. ORGANICS DETECTED IN GC/MS ANALYSIS OF "DIRTY" INSULATION 239

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	11.57	Dichlorophenol (isomer)	8.8×10^2
2	12.32	Chlorophenol (isomer)	1.4×10^4
3	15.13	Trichlorophenol (isomer)	8.7×10^2
4	20.45	Phenylmethylphenol (isomer)	2.9×10^3
5	21.77	Unknown (91, 218, 65), possibly  or isomer	9.8×10^2
6	23.52	o-Benzyl-p-chlorophenol (Santophen)	1.8×10^3
7	23.98	Unknown (174, 176, 252, 217), possibly  or isomer	2.6×10^2
8	24.08	Unknown aromatic containing 1 or 2 chlorines (183, 218, 165, 254)	1.5×10^2
9	24.23	Unknown phthalate (149, 205, 223)	2.2×10^2
		TOTAL	2.3×10^4 μg/g

a = Results were not corrected for spike recoveries (see Table 3), nor were any background contaminants subtracted out (see Tables 4 and 5).

b = Identifications are tentative ones based upon library searches and spectral comparisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 1.5×10^2 μg/g.

MONS 022709

TABLE 13. ORGANICS DETECTED IN GC/MS ANALYSIS OF RESIDUE EQUIP INSULATION 237

Peak No.	Retention Time (min.)	Organics Detected[b]	Amount Detected (μg/g)[c]
1	12.25	Chlorophenol (isomer)	9.8
2	24.23	Unknown phthalate (149, 150, 205, 223)	3.7
3	30.78	Unknown phthalate (149, 167)	6.8
		TOTAL	20.3 μg/g

a = Results were not corrected for spike recoveries (see Table 3), nor were any background contaminants subtracted out (see Tables 4 and 5).

b = Identifications are tentative ones based upon library searches and spectral comparisons or manual interpretation of mass spectra. Except for the acid priority pollutants, no attempt was made to confirm identifications by analysis of actual standards. Major ions of unknowns are listed in parentheses, with possible molecular ions being underlined.

c = Concentrations were calculated relative to the anthracene-d_{10} internal standard and are semiquantitative only. The detection limit for this sample was 3.5 μg/g.

MONS 022710

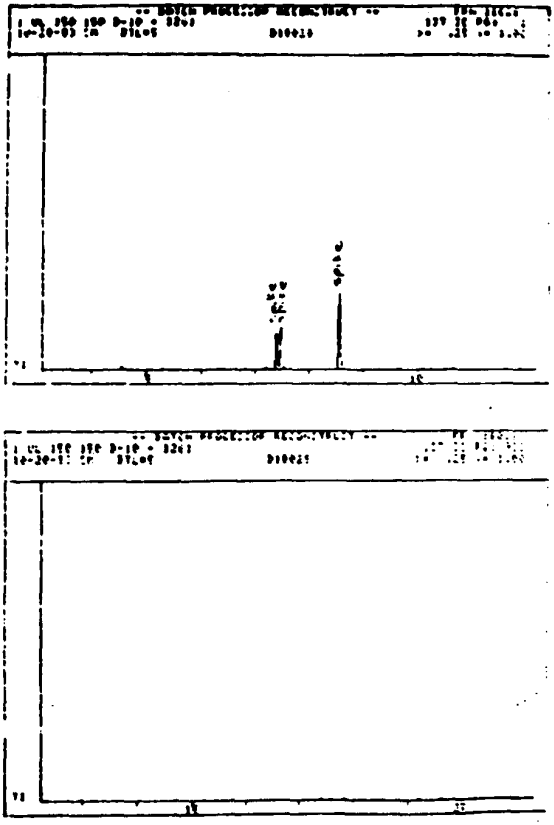


Figure 1. Total ion chromatogam of extract of MRC Concrete Blank.

MONS 022711

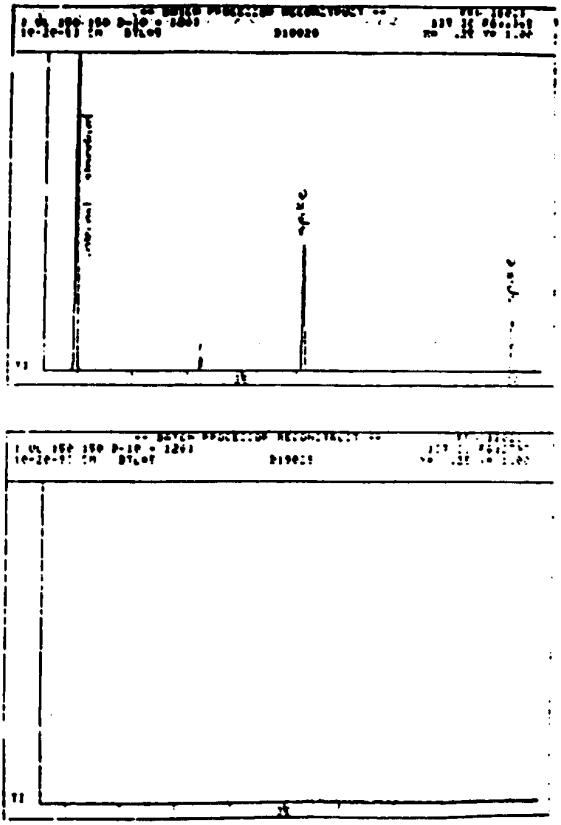


Figure 1. (continued).

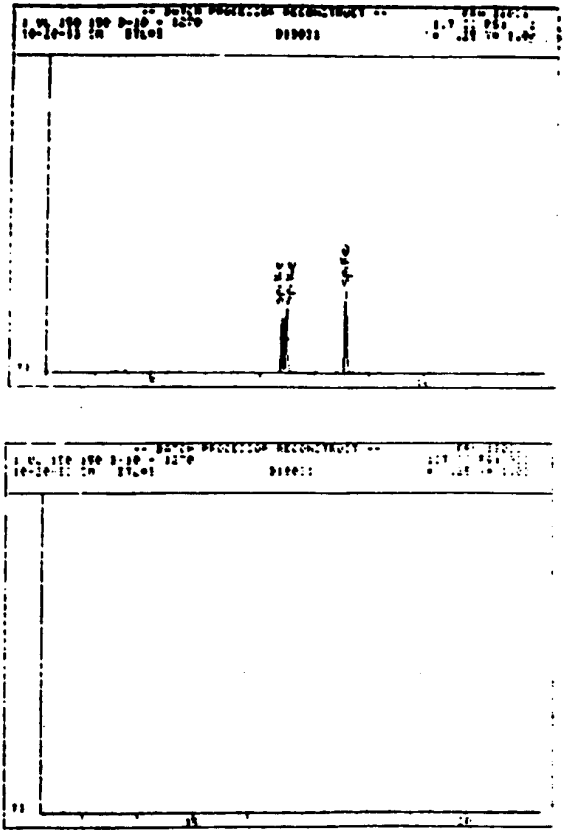


Figure 2. Total ion chromatogram of extract of Method (Glassware) Blank for Krummrich.

MONS 022713

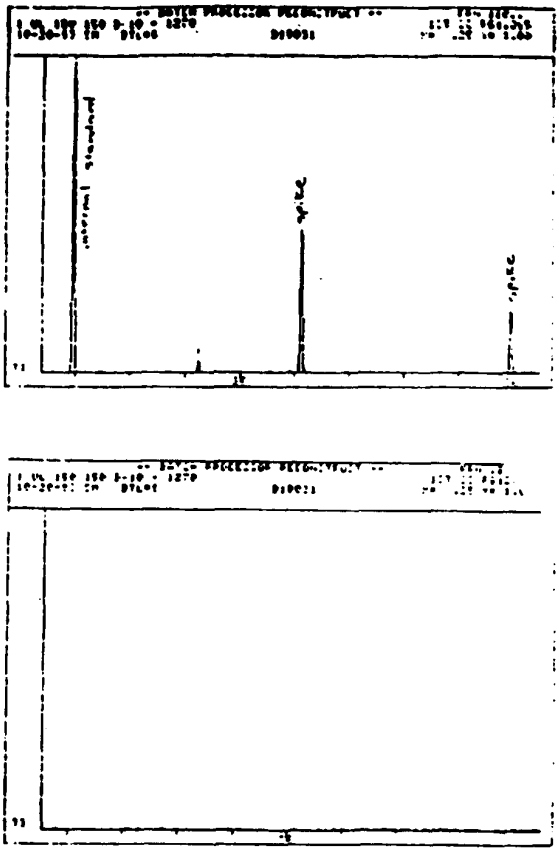


Figure 2. (continued).

MONS 022714

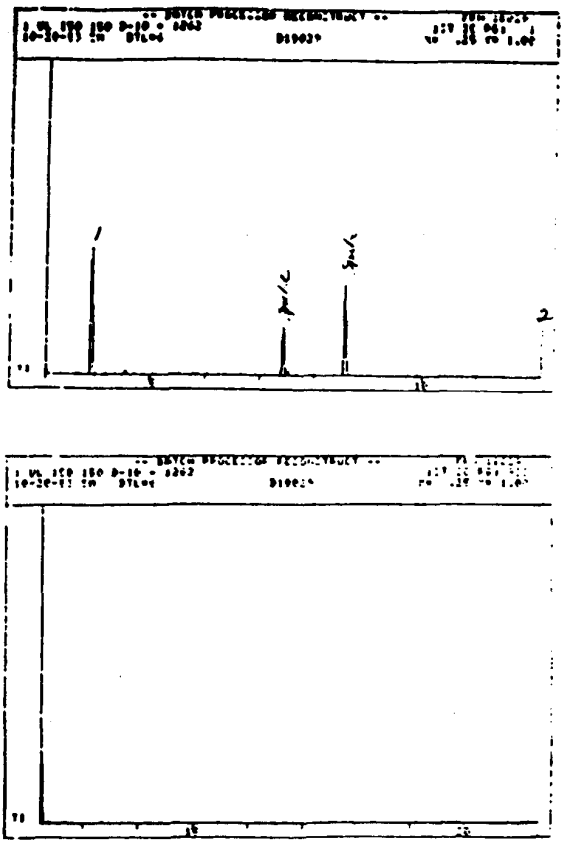


Figure 3. Total ion chromatogram of extract of Concrete 237.

MONS 022715

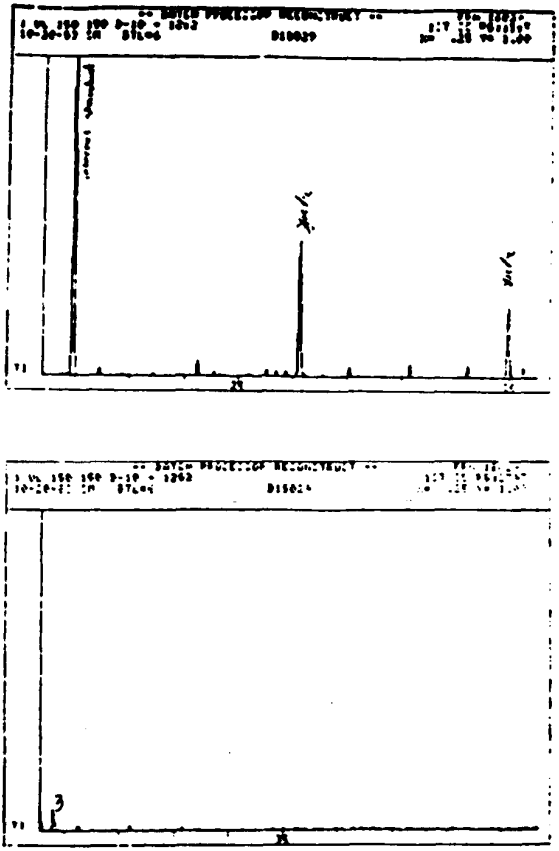


Figure 3. (continued).

MONS 022716

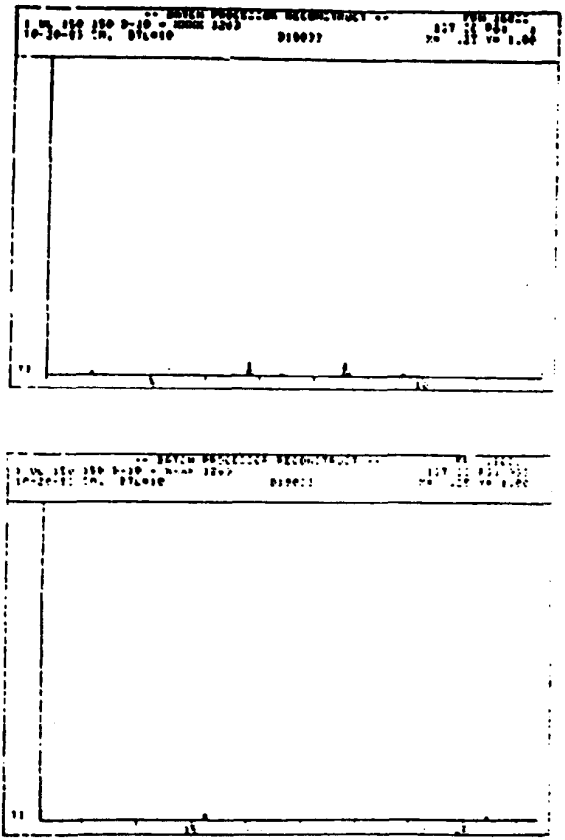


Figure 4. Total ion chromatogram of extract of Concrete 239.

MONS 022717

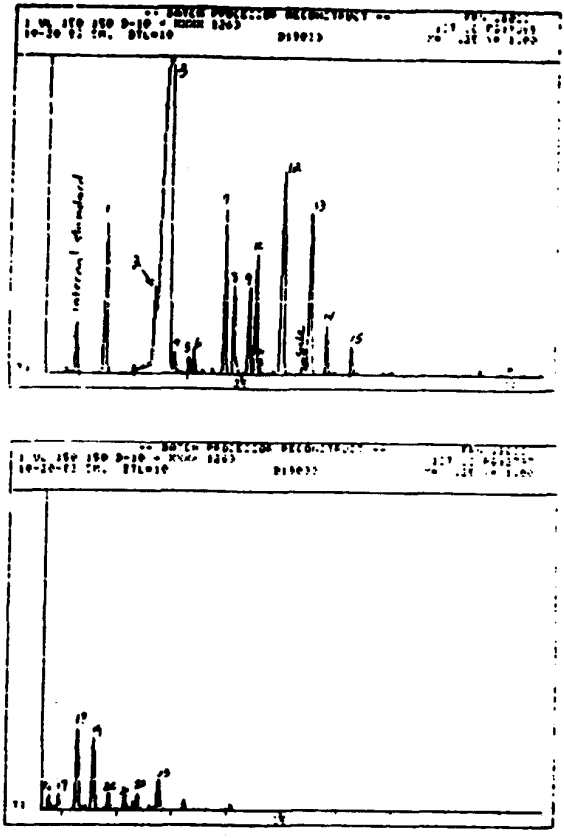


Figure 4. (continued).

MONS 022718

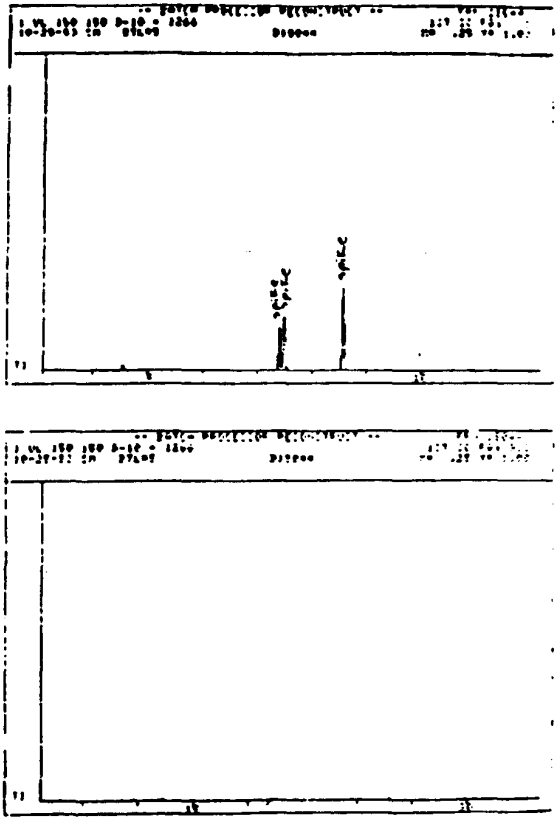


Figure 5. Total ion chromatogram of extract of "New" Insulation 237.

MONS 022719

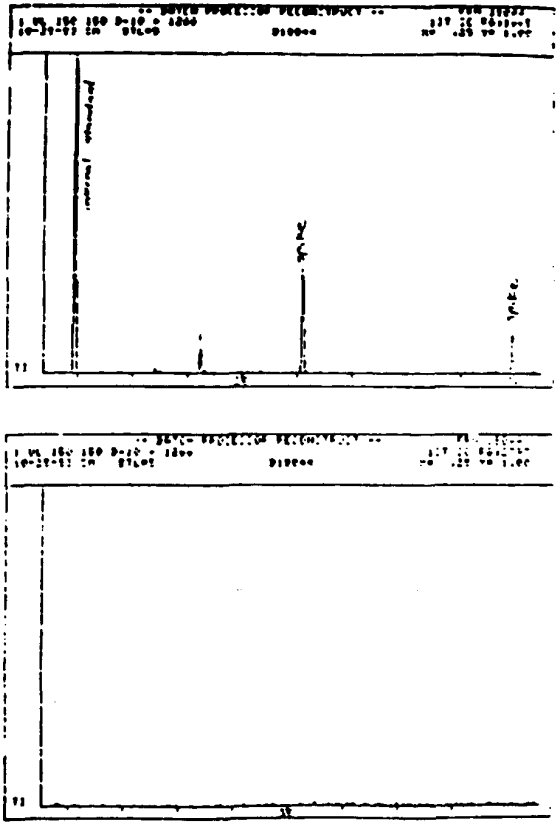


Figure 5. (continued).

MONS 022720

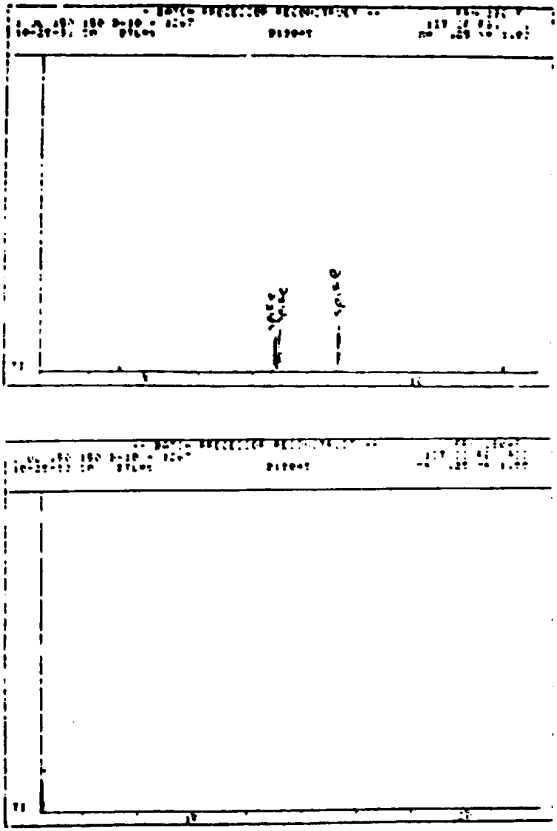


Figure 6. Total ion chromatogram of extract of "Clean" Insulation 237.

MONS 022721

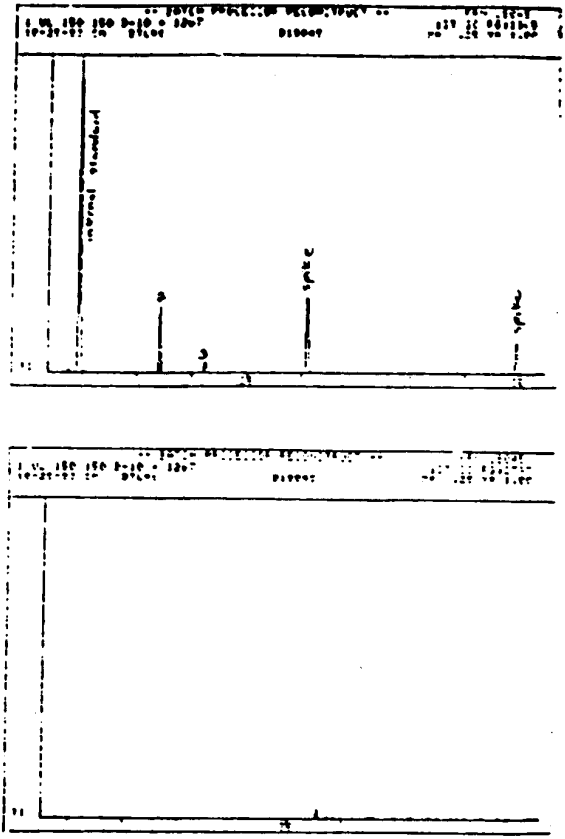


Figure 6. (continued).

MONS 022722

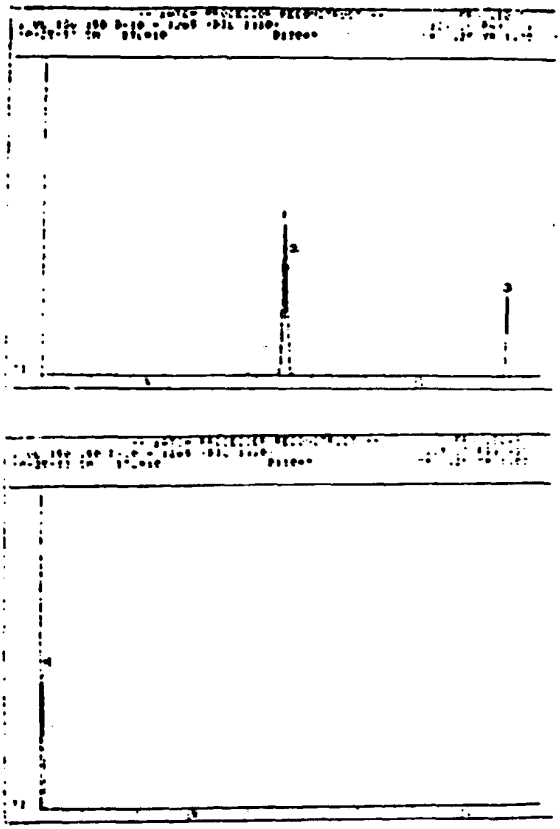


Figure 7. Total ion chromatogram of extract of "Dirty" Insulation 237 (diluted 1:10).

MONS 022723

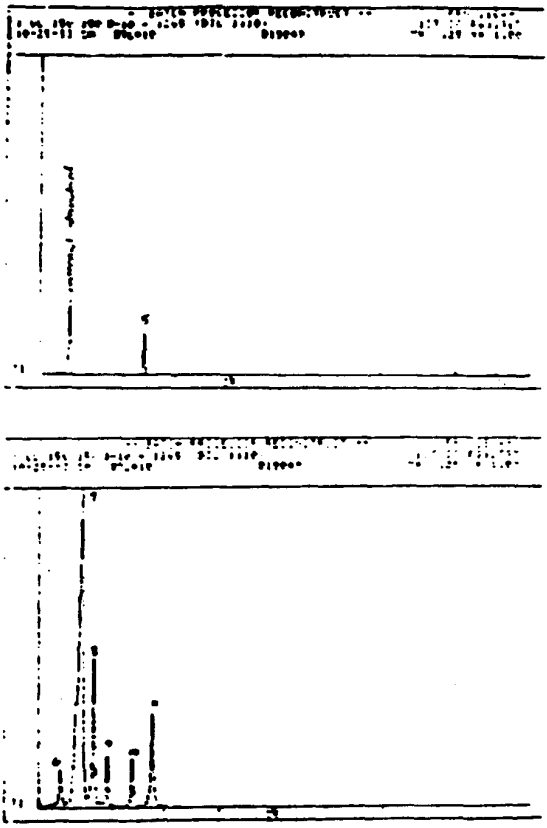


Figure 7. (continued).

MONS 022724

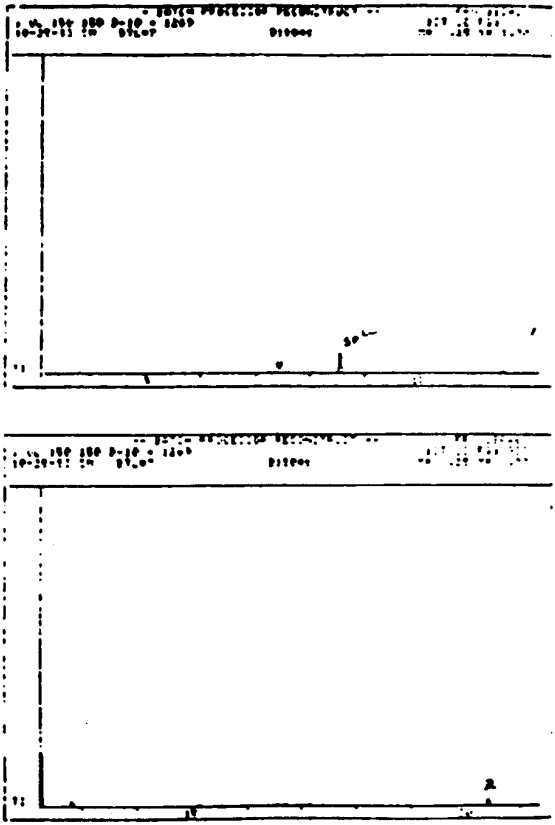


Figure 8. Total ion chromatogram of extract of "Clean" Insulation 239.

MONS 022725

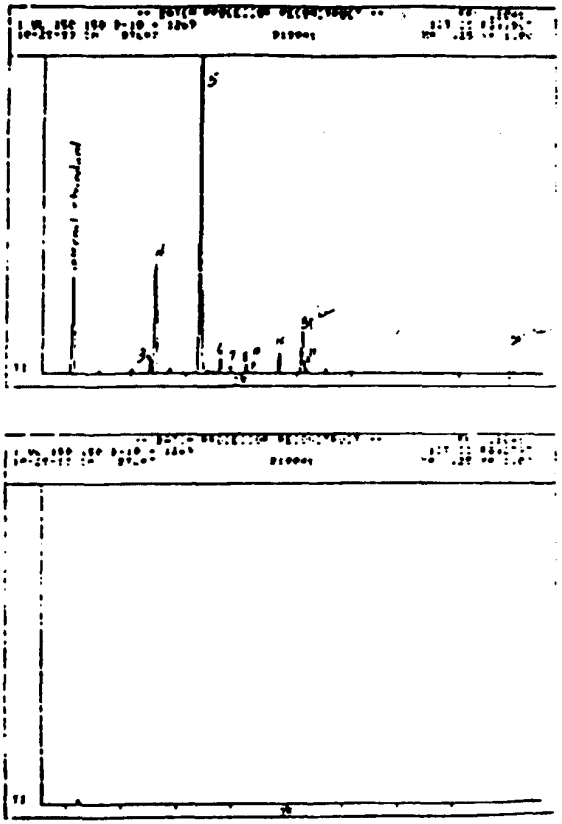


Figure 8. (continued).

MONS 022726

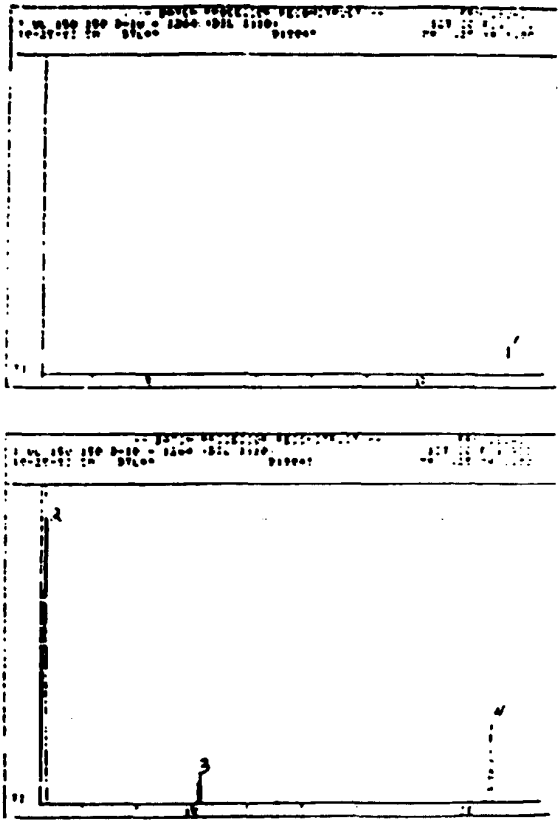


Figure 9. Total ion chromatogram of extract of "Dirty" Insulation 239 (diluted 1:10).

MONS 022727

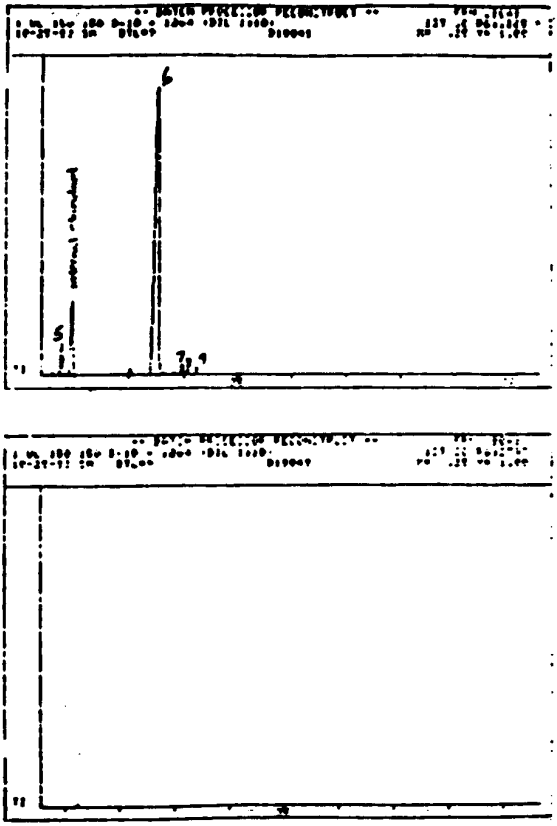


Figure 9. (continued).

MONS 022728

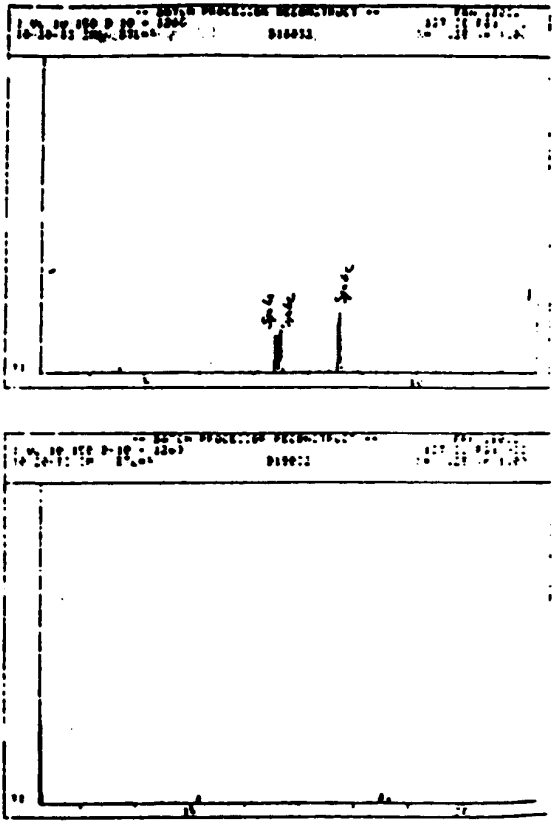


Figure 10. Total ion chromatogram of extract of Residue Equip Insulation 237.

MONS 022729

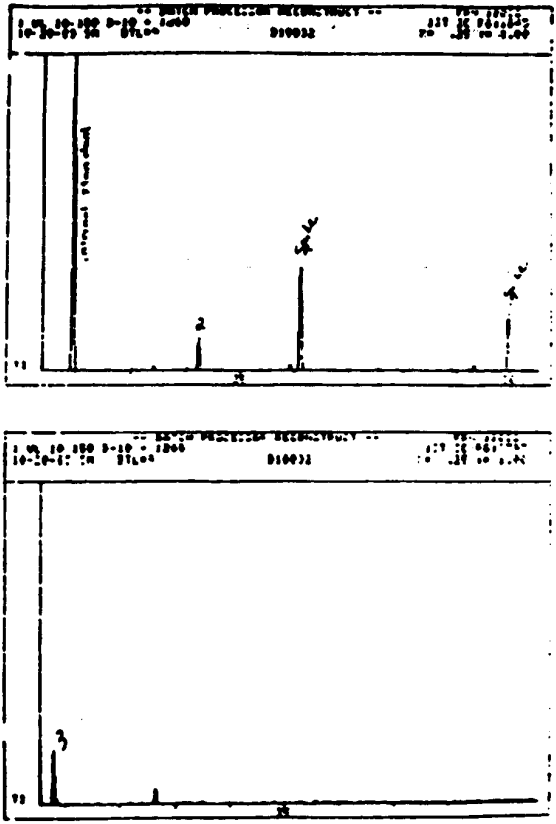


Figure 10. (continued).

MONS 022730