NRA-WATER QUALITY -73 Pesk:B/4 K. EKE

NRA

National Rivers Authority
National Laboratory Service

A REPORT ON THE INVESTIGATION INTO WATER CONTAMINATION AT HAUXTON, CAMBRIDGESHIRE.

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Date: 29th September 1995

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NAMAS TESTING No. 1284

NAMAS APPROVED LABORATORY

Pests: B/4

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INTRODUCTION

Following a request from the Anglian client, nine water samples were received by the laboratory at Reading for qualitative and quantitative organic analysis.
This report details the results from this investigation.

LAB. NUMBER	DATE SAMPLED	SAMPLING SITE	SAMPLE DESCRIPTION
95027941	22/08/95	ELLIOTS FARM, PIEZOMETER No.1, STATIC	SLIGHTLY OPAQUE WATER OF A GREYISH COLOUR. ALOT OF VERY FINE GREY SEDIMENT.SOME ODOUR.
95027942	22/08/95	ELLIOTS FARM, PIEZOMETER No.4, PUMPED	TRANSPARENT WATER OF A GREYISH COLOUR. ALOT OF VERY FINE GREY SEDIMENT.SLIGHT ODOUR.
95027943	22/08/95	ELLIOTS FARM, PIEZOMETER No.1, PUMPED	SLIGHTLY OPAQUE WATER OF A GREYISH COLOUR. ALOT OF VERY FINE GREY SEDIMENT.SOME ODOUR.
95027945	22/08/95	ELLIOTS FARM, PIEZOMETER No.5, PUMPED	TRANSPARENT WATER WITH A GREYISH COLOUR. ALOT OF VERY FINE GREY SEDIMENT.SLIGHT ODOUR.
95028126	23/08/95	RIVER RIDDY, 5m D/S AGREVO STW	SLIGHTLY YELLOW TRANSPARENT WATER. SOME BROWN SEDIMENT. SOME ODOUR.
95028127	23/08/95	SWD from A10 HAUXTON	YELLOW/GREY TRANSPARENT WATER. SOME BROWN SEDIMENT. SOME ODOUR.
95028128	23/08/95	ELLIOTS FARM, PIEZOMETER No.2 PUMPED	COLOURLESS TRANSPARENT WATER. ALOT OF VERY FINE GREY SEDIMENT.SOME ODOUR.
95028129	23/08/95	AGREVO, PIEZOMETER No.59, PUMPED	COLOURLESS TRANSPARENT WATER. SOME VERY FINE BROWN/GREY SEDIMENT.SLIGHT ODOUR.
95028130	23/08/95	GARFITTS LAND, PIEZOMETER No.G4 PUMPED	COLOURLESS,TRANSPARENT WATER. SOME VERY FINE GREY SEDIMENT. SLIGHT ODOUR.

Above table gives the description of the nine samples received by the laboratory.
Samples relating to lab.no. **95028126** and **95028127** had mass spectral scans requested.
The remaining samples in addition to mass spectral scans had selected phenols, volatile solvents, phenoxy-acid and triazine herbicides requested.

Methodologies used in the analysis

Mass scans:
The water was shaken with dichloromethane to extract organic compounds present. The extract was dried, reduced in volume and injected into a gas chromatograph with mass spectrometric detection. The mass spectrometer was in scan mode for this operation.

Phenols:
The phenolic compounds were simultaneously extracted from water into iso-octane and derivatised using pentafluorobenzoyl chloride. The product was separated from the water and injected into a gas chromatograph coupled with a mass spectrometric detector.

Phenoxy-acid herbicides:
The acidic herbicides were extracted into dichloromethane at a low pH.The extract was methylated using trimethylanilinium hydroxide and injected into a gas chromatograph coupled with a mass spectrometric detector.

Triazine herbicides:
The triazine herbicides were extracted from water into iso-hexane and dichloromethane. The extract was injected into a gas chromatograph coupled with a mass spectrometric detector

Volatile solvents:
The volatile solvents were extracted and analysed by purge and trap extraction coupled with mass spectrometric detection. This technique was used to identify and quantitate the low level components found. Higher levels were determined using headspace analysis with electron capture detection.

Results

All analysis requested was completed within 14 days. A preliminary report was communicated on the 25th August whilst the further investigation into these samples continued. Several compounds were identified that were not originally requested. All data pertaining to these samples has been tabulated. The most notable of the compounds identified from the mass scans were **HEMPA** and **SCHRADAN** (see appendix).
These compounds not routinely analysed by the laboratory were quantitated after suitable standard material was obtained and a method devised.
The scanning for volatile solvents was also undertaken but no unidentified peaks were detected.

HAUXTON SAMPLES - VOLATILE CHLORINATED SOLVENT ANALYSIS

ANALYSIS OF VOLATILE SOLVENTS BY HEADSPACE WITH ELECTRON CAPTURE DETECTION AND PURGE AND TRAP WITH MASS SPECTROMETRIC DETECTION.
ALL COMPOUNDS CONFIRMED BY MASS SPECTROMETRIC DETECTION.
DUE TO HIGH LEVELS OF SOME CHLORINATED SOLVENTS SAMPLES REQUIRED DILUTION HENCE ELEVATED MINIMUM REPORTING VALUES.

RESULTS IN MICROGRAMS PER LITRE

	95027941	95027942	95027943	95027945	95028128	95028129	95028130
1,1-DICHLOROETHENE	63.0	<1.0	75.3	<1.0	12.6	<1.0	<1.0
DICHLOROMETHANE	11.3	3.8	12.8	3.3	3.8	4.1	4.0
trans 1,2-DICHLOROETHENE	67.6	<1.0	91.2	<1.0	<1.0	<1.0	<1.0
cis 1,2-DICHLOROETHENE	207	<1.0	272	<1.0	14.2	<1.0	<1.0
CHLOROFORM	123	<0.2	220	<0.2	0.2	0.2	<0.2
1,1,1-TRICHLOROETHANE	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
CARBON TETRACHLORIDE	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
1,2-DICHLOROETHANE	1.2	<1.0	1.6	<1.0	<1.0	<1.0	<1.0
TRICHLOROETHENE	7350	6.4	10000	20.3	48.5	16.6	7.0
BROMODICHLOROMETHANE	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
1,1,2-TRICHLOROETHANE	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
TETRACHLOROETHENE	65.2	<0.2	70.0	<0.2	0.6	3.6	<0.2
DIBROMOCHLOROMETHANE	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
BROMOFORM	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
1,1,1,2-TETRACHLOROETHANE	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
1,1,2,2-TETRACHLOROETHANE	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0

HAUXTON SAMPLES - VOLATILE AROMATIC SOLVENT ANALYSIS

ANALYSIS OF VOLATILE SOLVENTS BY PURGE AND TRAP WITH MASS SPECTROMETRIC DETECTION.
DUE TO HIGH LEVELS OF SOME CHLORINATED SOLVENTS SAMPLES REQUIRED DILUTION HENCE ELEVATED MINIMUM REPORTING VALUES.

RESULTS IN MICROGRAMS PER LITRE

	95027941	95027942	95027943	95027945	95028128	95028129	95028130
BENZENE	3.0	<1.0	4.1	<1.0	<1.0	1.2	<1.0
TOLUENE	2.2	<1.0	2.5	1.0	<1.0	5.1	1.3
CHLOROBENZENE	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
ETHYLBENZENE	1.7	<1.0	1.1	<1.0	<1.0	<1.0	<1.0
TOTAL XYLENE	6.4	<3.0	8.3	<3.0	<3.0	3.8	<3.0
STYRENE	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
1,3-DICHLOROBENZENE	<1.0	<1.0	<1.0	<1.0	1.1	<1.0	<1.0
1,4-DICHLOROBENZENE	<1.0	<1.0	<1.0	<1.0	1.4	<1.0	<1.0
1,2-DICHLOROBENZENE	<1.0	<1.0	<1.0	<1.0	2.5	<1.0	<1.0

HAUXTON SAMPLES - ACID HERBICIDE ANALYSIS

ANALYSIS OF ACIDIC HERBICIDES BY EXTRACTION INTO DICHLOROMETHANE AT LOW pH FOLLOWED BY METHYLATION AND GAS CHROMATOGRAPHY-MASS SPECTROMETRY.

RESULTS IN MICROGRAMS PER LITRE

	95027941	95027942	95027943	95027945	95028128	95028129	95028130
2,4,5-T	<0.6	<0.02	<0.2	<0.02	<0.2	<0.2	<0.2
FLUOROXYPYR	<0.2	<0.02	<0.2	<0.02	<0.2	<0.2	<0.2
2,3,6-TBA	232	<0.02	398	<0.02	<0.2	8.26	<0.2
2,4-D	0.44	<0.02	1.28	<0.02	<0.2	<0.2	<0.2
2,4-DB	<0.3	<0.02	<0.2	<0.02	<0.2	<0.2	<0.2
MECOPROP	<0.2	<0.02	<0.2	0.083	<0.2	<0.2	<0.2
MCPB	<0.2	<0.02	<0.2	<0.02	<0.2	<0.2	<0.2
MCPA	4.3	0.075	25.4	0.177	<0.2	<0.2	<0.2
DICHLORPROP	<0.2	<0.02	<0.2	<0.02	<0.2	<0.2	<0.2
DICAMBA	2.3	<0.02	4.5	<0.02	<0.2	<0.2	<0.2

HAUXTON SAMPLES - TRIAZINE HERBICIDES ANALYSIS

ANALYSIS OF TRIAZINE HERBICIDES BY EXTRACTION INTO ISO-HEXANE AND DICHLOROMETHANE FOLLOWED BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY.

RESULTS IN MICROGRAMS PER LITRE

	95027941	95027942	95027943	95027945	95028128	95028129	95028130
TRIETAZINE	0.208	<0.020	0.103	<0.020	<0.020	8.30	0.256
PROPAZINE	<0.020	<0.020	<0.020	<0.020	<0.020	0.309	<0.020
ETHOFUMESATE	0.135	<0.050	0.060	<0.050	<0.050	2.25	0.054
DESMETRYN	<0.020	<0.020	<0.02	<0.020	<0.020	<0.020	<0.020
PROMETRYN	<0.020	<0.020	<0.02	<0.020	<0.020	<0.020	<0.020
TERBUTRYN	<0.020	<0.020	<0.02	<0.020	<0.020	<0.020	0.054
ATRAZINE	0.066	<0.030	<0.030	<0.030	<0.030	0.329	0.087
SIMAZINE	0.123	<0.030	0.033	<0.030	0.034	13.1	1.30

HAUXTON SAMPLES - PHENOLS ANALYSIS

ANALYSIS OF PHENOLS BY SIMULTANEOUS EXTRACTION INTO ISO-OCTANE AND DERIVATISATION BY PENTAFLUOROBENZOYL CHLORIDE FOLLOWED BY GAS CHROMATOGRAPHY AND NEGATIVE ION MASS SPECTROSCOPY.

RESULTS IN MICROGRAMS PER LITRE

	95027941	95027942	95027943	95027945	95028128	95028129	95028130
p-CHLORO-o-CRESOL	1.35	<0.05	0.69	<0.05	*****	*****	*****
2,5-DICHLOROPHENOL	2.31	<0.05	1.62	<0.05	<0.100	<0.100	<0.100
PHENOL	2.09	0.175	1.24	0.06	0.133	0.594	0.207
2-CRESOL	2.28	<0.05	2.75	<0.05	<0.050	<0.050	<0.050
3-CRESOL	2.18	<0.05	2.50	<0.05	<0.050	0.069	<0.050
4-CRESOL	23.3	<0.05	5.04	<0.05	0.080	0.071	<0.050
2,3-XYLENOL	<0.05	<0.05	<0.05	<0.05	<0.050	<0.050	<0.050
2,4-XYLENOL	0.98	<0.05	0.98	<0.05	<0.050	<0.050	<0.050
2,5-XYLENOL	<0.05	<0.05	<0.05	<0.05	<0.100	<0.100	<0.100
2,6-XYLENOL	0.52	<0.05	0.52	<0.05	<0.050	<0.050	<0.050
3,4-XYLENOL	<0.05	<0.05	<0.05	<0.05	<0.050	<0.050	<0.050
3,5-XYLENOL	0.51	<0.05	0.37	<0.05	<0.050	<0.050	<0.050
2-CHLOROPHENOL	1.21	<0.05	1.73	<0.05	<0.100	<0.100	<0.100
3-CHLOROPHENOL	<0.05	<0.05	<0.05	<0.05	<0.050	<0.050	<0.050
4-CHLOROPHENOL	0.87	<0.05	6.46	<0.05	<0.050	<0.050	<0.050
2,4-DICHLOROPHENOL	4.48	<0.05	4.48	<0.05	<0.050	<0.050	<0.050
2,6-DICHLOROPHENOL	1.67	<0.05	1.67	<0.05	<0.050	<0.050	<0.050
2,4,6-TRICHLOROPHENOL	0.62	<0.05	0.78	<0.05	<0.100	<0.100	<0.100
2,4,5-TRICHLOROPHENOL	0.51	<0.05	0.93	<0.05	<0.050	<0.050	<0.050
PENTACHLOROPHENOL	<0.05	<0.05	0.068	<0.05	<0.100	<0.100	<0.100

ANALYSIS OF HAUXTON SAMPLES FOR PESTOX (SCHRADAN) AND HEMPA (HEXAMETHYL PHOSPHORIC TRIAMIDE)

Two toxic organophosphorous compounds Schradan and HEMPA were tentatively identified as being present in some sites as a result of GC-MS scan analysis. Reference standards were purchased and the presence of these compounds confirmed by retention time matching. All samples submitted for mass scan analysis from these sites were then analysed quantititavely for these compounds. The results are as follows.

RESULTS IN MICROGRAMS PER LITRE

LAB. NUMBER	DATE SAMPLED	SITE	SCHRADAN	HEMPA
95027941	22/08/95	ELLIOTS FARM, PIEZOMETER NO.1, STATIC	2,490	4,470
95027942	22/08/95	ELLIOTS FARM, PIEZOMETER NO.4, PUMPED	<1.0	<1.0
95027943	22/08/95	ELLIOTS FARM, PIEZOMETER NO.1, PUMPED	3,290	9,100
95027945	22/08/95	ELLIOTS FARM, PIEZOMETER NO.5, PUMPED	<1.0	<1.0
95028126	23/08/95	R. RIDDY 5m D/S AGREVO STW	22.8	3.97
95028127	23/08/95	SWD FROM A10 AT HAUXTON	<1.0	<1.0
95028128	23/08/95	ELLIOTS FARM PIEZOMETER NO.2, PUMPED	279	516
95028129	23/08/95	AGREVO, PIEZOMETER NO 59, PUMPED	15.3	16.8
95028130	23/08/95	GARFITTS LAND, PIEZOMETER NO.G4 PUMPED	<1.0	<1.0

MASS SCAN ANALYSIS

SAMPLE No.:- 95027941

SITE:- HAUXTON - Piezometer No.1 - STATIC

DATE SAMPLED :- 22/8/95

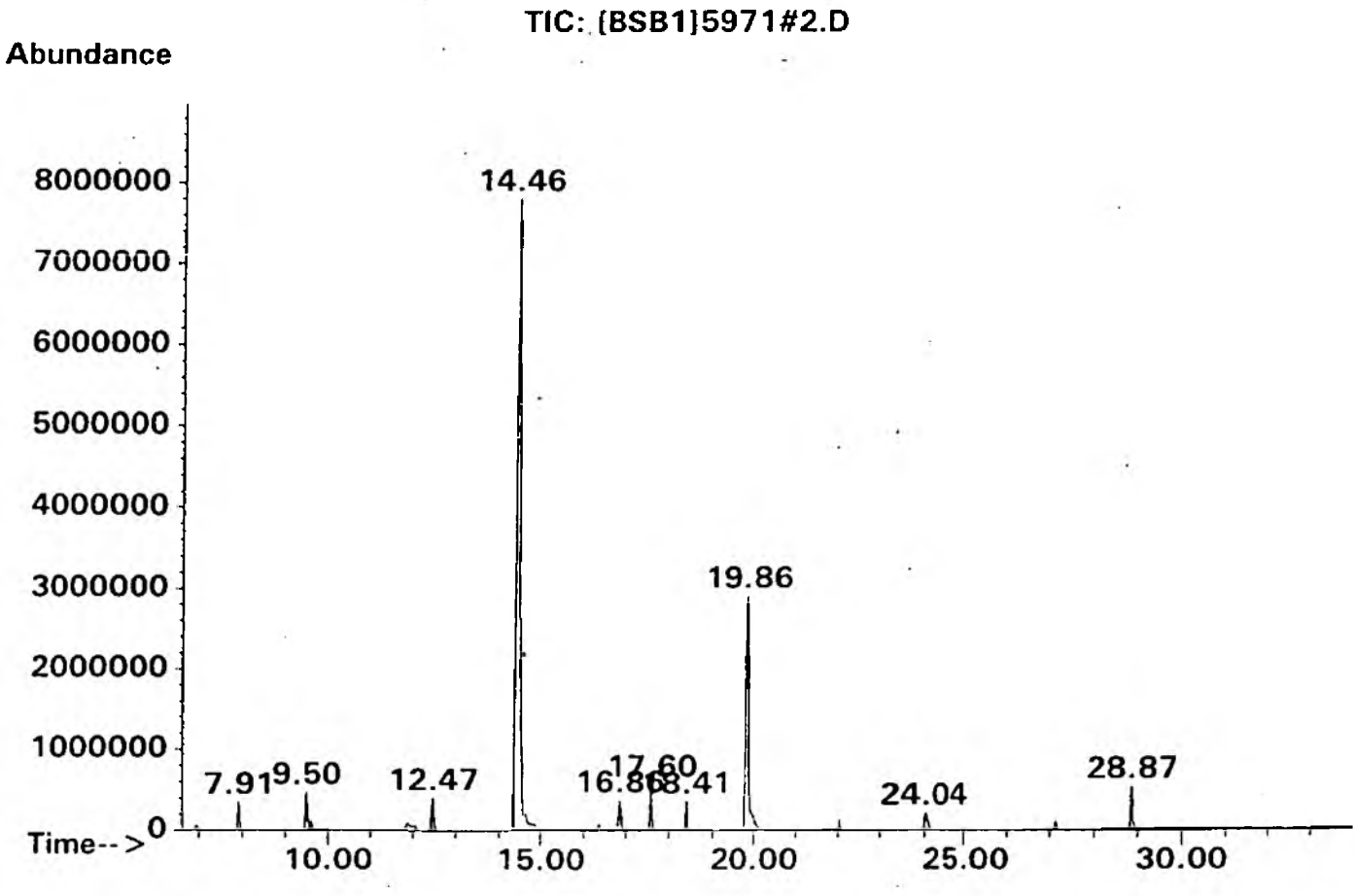
PHYSICAL APPEARANCE:- Slightly opaque water of a greyish colour. Alot of sediment. Some odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions. The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME R.T	LIBRARY ID.	CAS#	%MATCH	CONF. BY
7.91	1,4-Oxathiane	15980-15-1	91	
9.50	Ethane, 1,1-Oxybis [2-chloroethane	111-44-4	91	
9.61	Urea, tetramethyl	632-22-4	72	
11.88	Phenol, 2-Chloro-6-methyl-	87-64-9	91	
12.47	UNIDENTIFIED			
14.46	HEMPA (Hexamethyl phosphoric triamide)	680-31-9	90	YES
16.36	1,4-Benzenedicarboxylic acid ester	120-61-6	53	
16.86	Pentamethylformylphosphoramide	000-00-0	47	
17.60	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.41	UNIDENTIFIED			
19.86	PESTOX (Schradan)	152-16-9	89	YES
24.04	Bisphenol A.	80-05-7	95	
28.87	Peak present in blank			

COMMENTS

The two largest peaks in the chromatogram are at 14.46 and 19.86 minutes. These peaks gave good mass spectral matches for the toxic organophosphorous compounds HEMPA and SCHRADAN respectively. Subsequently reference standards were purchased and the presence of these compounds was confirmed by retention time matching. Further work determined the levels of HEMPA and Schradan to be 4,470 and 2,490 micrograms per litre respectively.

MASS SCAN ANALYSIS

SAMPLE No.:- 95027942

SITE:- HAUXTON - Piezometer No.4 - PUMPED

DATE SAMPLED :- 22/8/95

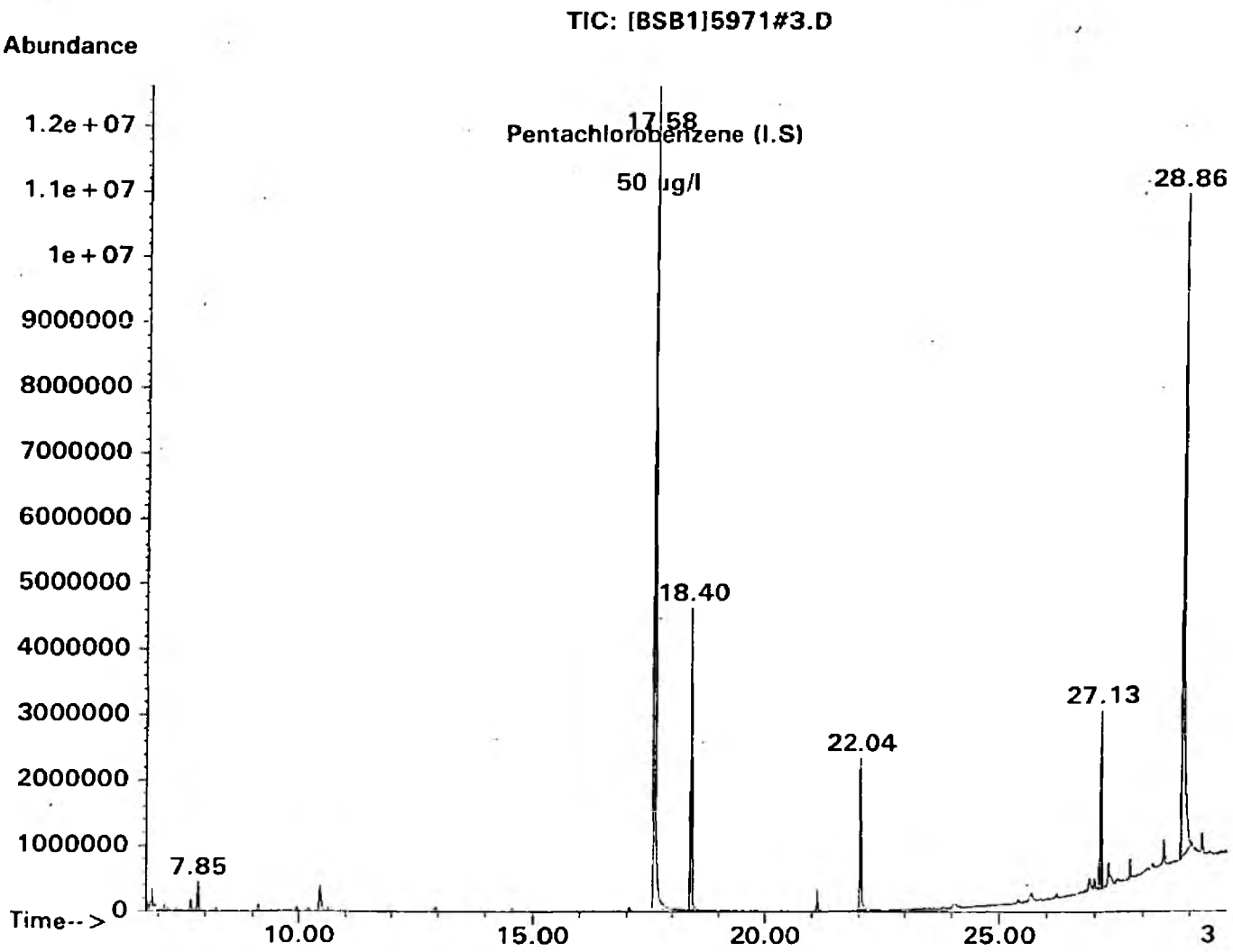
PHYSICAL APPEARANCE:- Transparent water of a greyish colour. Alot of sediment. Slight odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions.The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME	LIBRARY ID.	CAS#	%MATCH	CONF. BY R.T
7.85	Peak present in blank			
17.58	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.40	Peak present in blank			
22.04	Peak present in blank			
27.13	Peak present in blank			
28.87	Peak present in blank			

COMMENTS

The sample did not contain significant quantities of any chemical extractable into dichloromethane and amenable to gas chromatography.

MASS SCAN ANALYSIS

SAMPLE No.:- 95027943

SITE:- HAUXTON - Piezometer No.1 - PUMPED

DATE SAMPLED :- 22/8/95

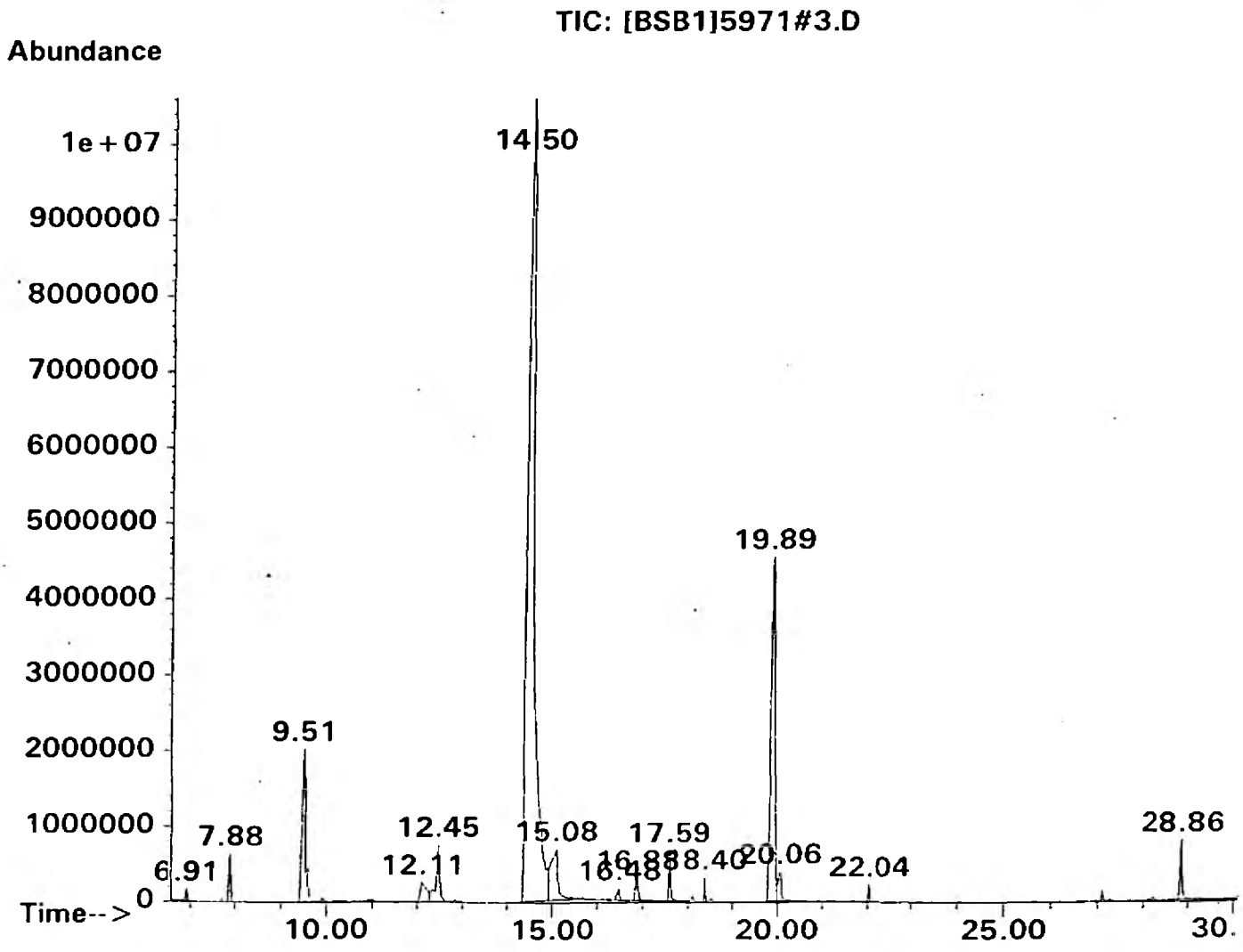
PHYSICAL APPEARANCE:- Slightly opaque water of a greyish colour. Alot of sediment. Some odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions. The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME	LIBRARY ID.	CAS#	%MATCH	CONF. BY R.T
6.91	Tetrachloroethene	127-18-4	97	
7.88	1,4-Oxathiane	15980-15-1	87	
9.51	Ethane, 1,1-Oxybis [2-chloroethane	111-44-4	97	
9.59	Urea, tetramethyl	632-22-4	80	
9.91	Benzofuran	271-89-6	90	
12.11	Phenol, 2-Chloro-6-methyl-	87-64-9	94	
12.45	UNIDENTIFIED			
14.50	HEMPA (Hexamethyl phosphoric triamide)	680-31-9	95	YES
15.08	UNIDENTIFIED			
16.48	Benzaldehyde, 4-methyl-,oxime	3235-02-7	35	
16.88	Pentamethylformylphosphoramide	000-00-0	38	
17.58	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.12	Phenol, 2,5-bis(1-methylpropyl)	54932-77-3	87	
18.40	Peak present in blank			
18.54	Phenol, 2,6-bis(1-methylpropyl)	5510-99-6	64	
19.89	PESTOX (Schradan)	152-16-9	90	YES
22.04	Peak present in blank			
28.22	Phenol, 4,4'-butylidenebis[2-(1,1-	85-60-9	93	
28.86	Peak present in blank			

COMMENTS

The two largest peaks in the chromatogram are at 14.50 and 19.89 minutes. These peaks gave good mass spectral matches for the toxic organophosphorous compounds HEMPA and SCHRADAN respectively. Subsequently reference standards were purchased and the presence of these compounds was confirmed by retention time matching. Further work determined the levels of HEMPA and Schradan to be 9,100 and 3,290 micrograms per litre respectively.

MASS SCAN ANALYSIS

SAMPLE No.:- 95027945

SITE:- HAUXTON - Piezometer No.5 - PUMPED

DATE SAMPLED :- 22/8/95

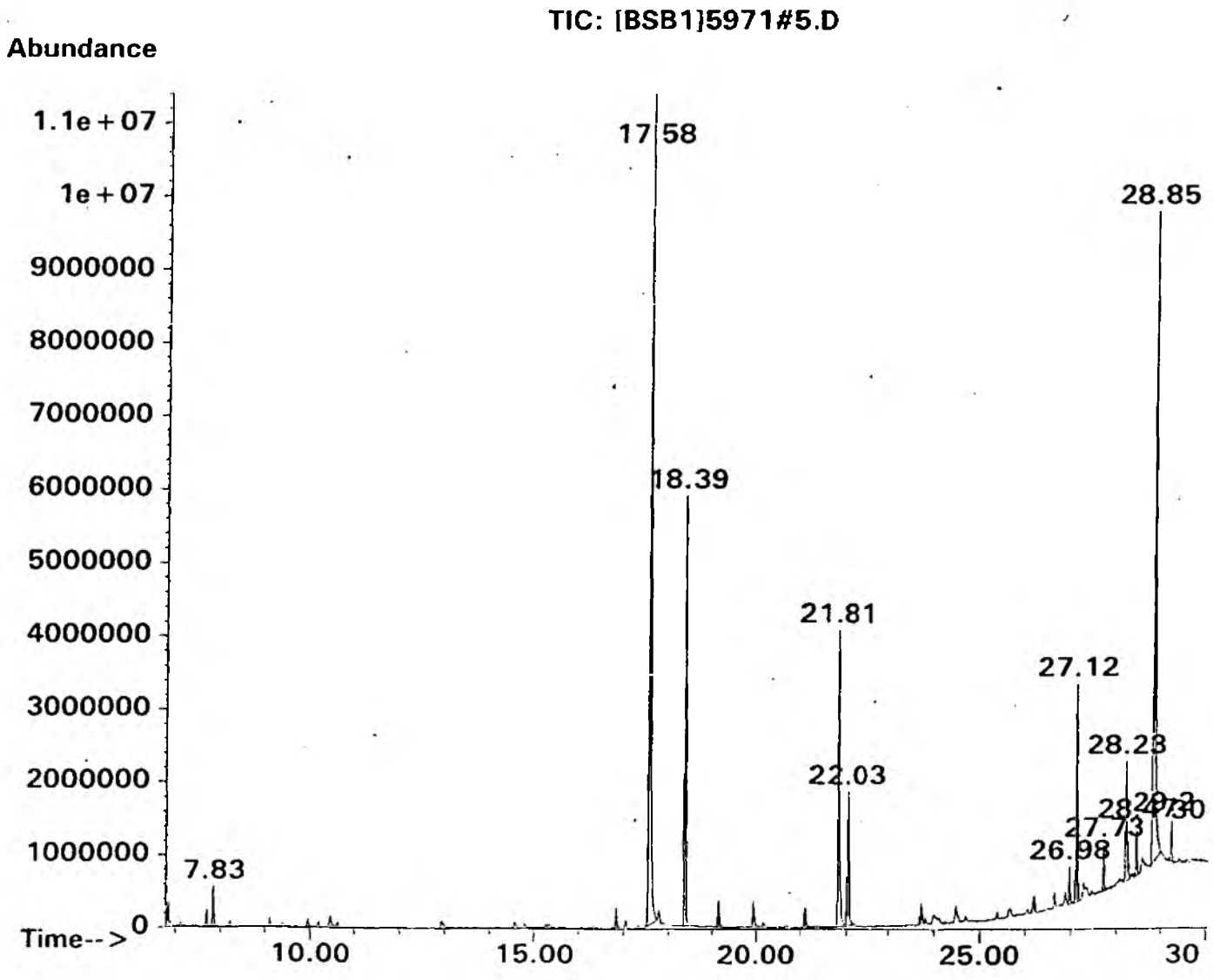
PHYSICAL APPEARANCE:- Transparent water of a greyish colour. Alot of sediment. Slight odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions. The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME	LIBRARY ID.	CAS#	%MATCH	CONF. BY R.T
7.83	Peak present in blank			
17.58	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.39	Peak present in blank			
21.81	Carbanochloridic acid, 2-chloro-1-	817-80-1	64	
22.03	Peak present in blank			
26.98	Eicosane	112-95-8	87	
27.12	Peak present in blank			
27.74	Hexadecane, 2,6,10,14-tetramethyl	638-36-8	80	
28.23	Phenol, 4,4'-butylidenebis[2-(1,1-	85-60-9	86	
28.47	Heptacosane	593-49-7	86	
28.85	Peak present in blank			
29.25	Octacosane	630-02-4	64	
30.12	Tricosane	638-67-5	80	

COMMENTS

The sample did not contain significant quantities of any chemical extractable into dichloromethane and amenable to gas chromatography.

MASS SCAN ANALYSIS

SAMPLE No.:- 95028126

SITE:- HAUXTON - River Riddy, 5m D/S of S/W O/F from Agrevo car park.

DATE SAMPLED :- 23/8/95

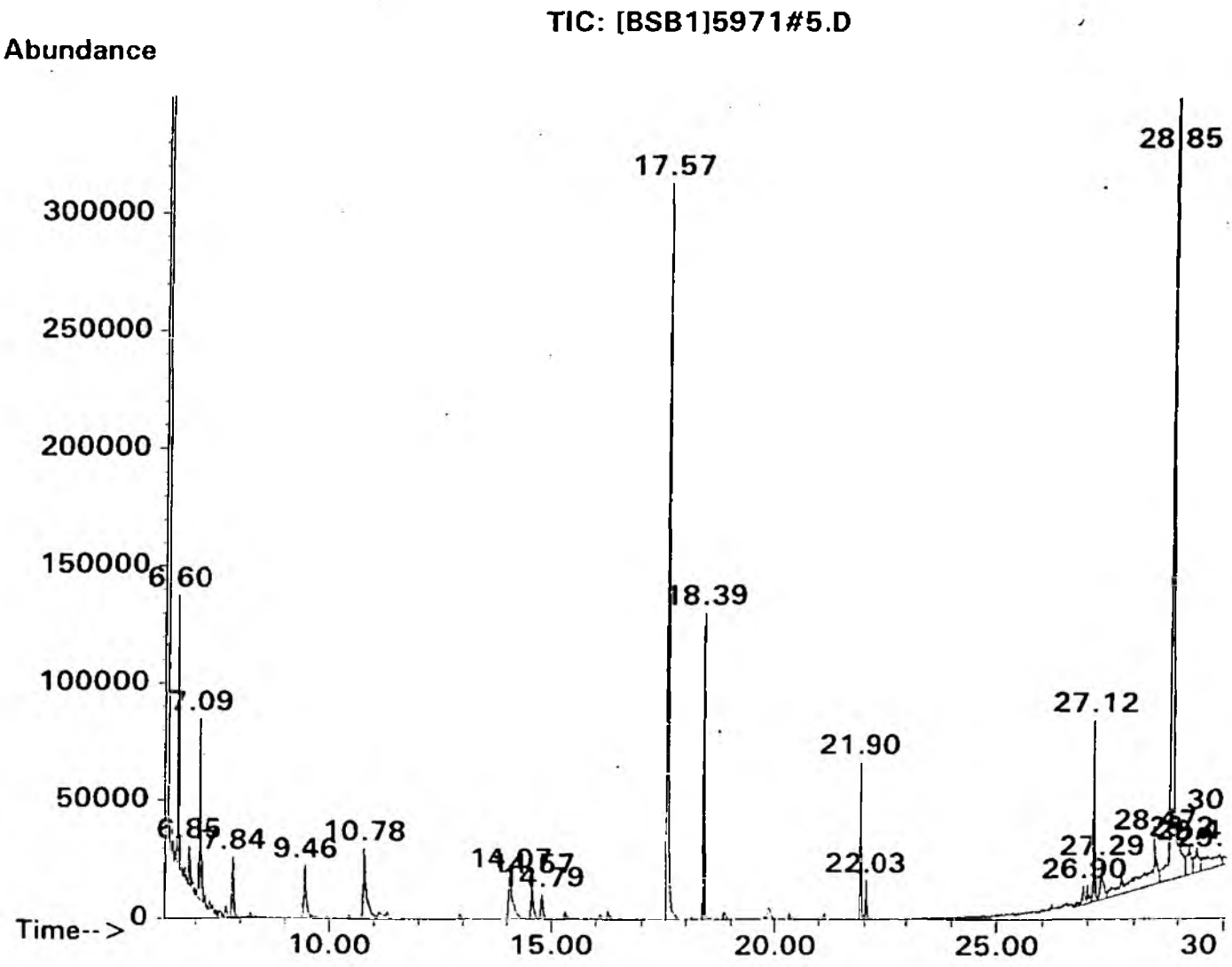
PHYSICAL APPEARANCE:- Slightly yellow transparent water. Some brown sediment. Some odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions.The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME	LIBRARY ID.	CAS#	%MATCH	CONF. BY R.T
6.60	Isoxazole	288-14-2	28	
6.85	Heptane, 2,3-dimethyl	3074-71-3	40	
7.09	UNIDENTIFIED			
7.84	Ethylbenzene	100-41-4	45	
9.46	Ethane, 1,1'-oxybis[2-chloro-	111-44-4	64	
10.78	Phenol, 2-methyl- (o-Cresol)	95-48-7	91	
14.07	Phenol, 4-chloro-3-methyl	59-50-7	91	
14.57	Naphthalene, 1-methyl	90-12-0	90	
14.79	Naphthalene, 2-methyl	91-57-6	90	
17.57	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.39	Peak present in blank			
21.90	Ethofumesate	26225-79-6	95	YES
22.03	Peak present in blank			
26.90	Phosphine oxide, triphenyl-	791-28-6	45	
27.12	Peak present in blank			
27.29	UNIDENTIFIED			
28.47	UNIDENTIFIED			
28.85	Peak present in blank			

COMMENTS

The sample was found to contain a number of organic compounds including phenols, PAH's and the herbicide Ethofumesate the presence of which was confirmed by retention time matching.

MASS SCAN ANALYSIS

SAMPLE No.:- 95028127

SITE:- HAUXTON - Surface water discharge from A10 road.

DATE SAMPLED :- 23/8/95

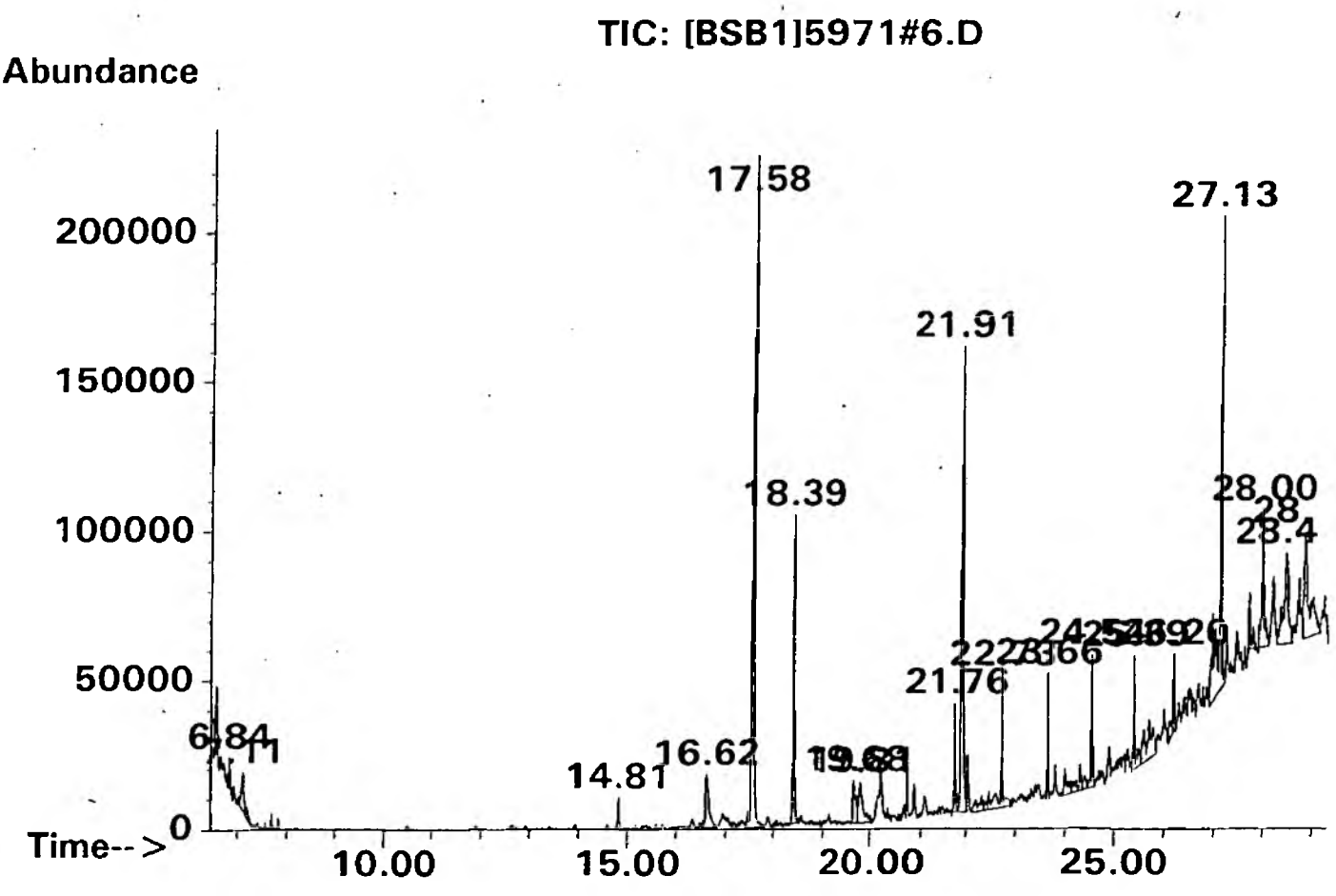
PHYSICAL APPEARANCE:- Yellow/grey transparent water. Some brown sediment. Some odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions. The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME R.T	LIBRARY ID.	CAS#	%MATCH	CONF. BY
6.84	UNIDENTIFIED			
7.11	UNIDENTIFIED			
14.81	Triacetin	000-00-0	36	
16.62	Ethanol, 2-[2-(butoxyethoxy)etho	143-22-6	64	
17.57	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.39	Peak present in blank			
19.68	Simazine	122-34-9	84	YES
19.80	UNIDENTIFIED			
20.23	Trietazine	1912-26-1	94	YES
21.76	Tricosane	638-67-5	78	
21.90	Ethofumesate	26225-79-6	95	YES
22.03	Peak present in blank			
22.73	Eicosane	112-95-8	95	
23.66	Hexadecane, 2,6,10,14-tetramethyl	638-36-8	86	
24.54	Pentacosane	629-99-2	86	
25.39	Tridecane	629-50-5	64	
26.21	UNIDENTIFIED			
27.13	Peak present in blank			
28.00	UNIDENTIFIED			
28.48	UNIDENTIFIED			
28.86	Peak present in blank			

COMMENTS

The presence of the herbicides simazine, trietazine and ethofumesate in this sample was confirmed by retention time matching. There were also quantities of hydrocarbons suggesting low-level oil contamination.

MASS SCAN ANALYSIS

SAMPLE No.:- 95028128

SITE:- HAUXTON - Elliots farm piezometer No.2 - PUMPED

DATE SAMPLED :- 23/8/95

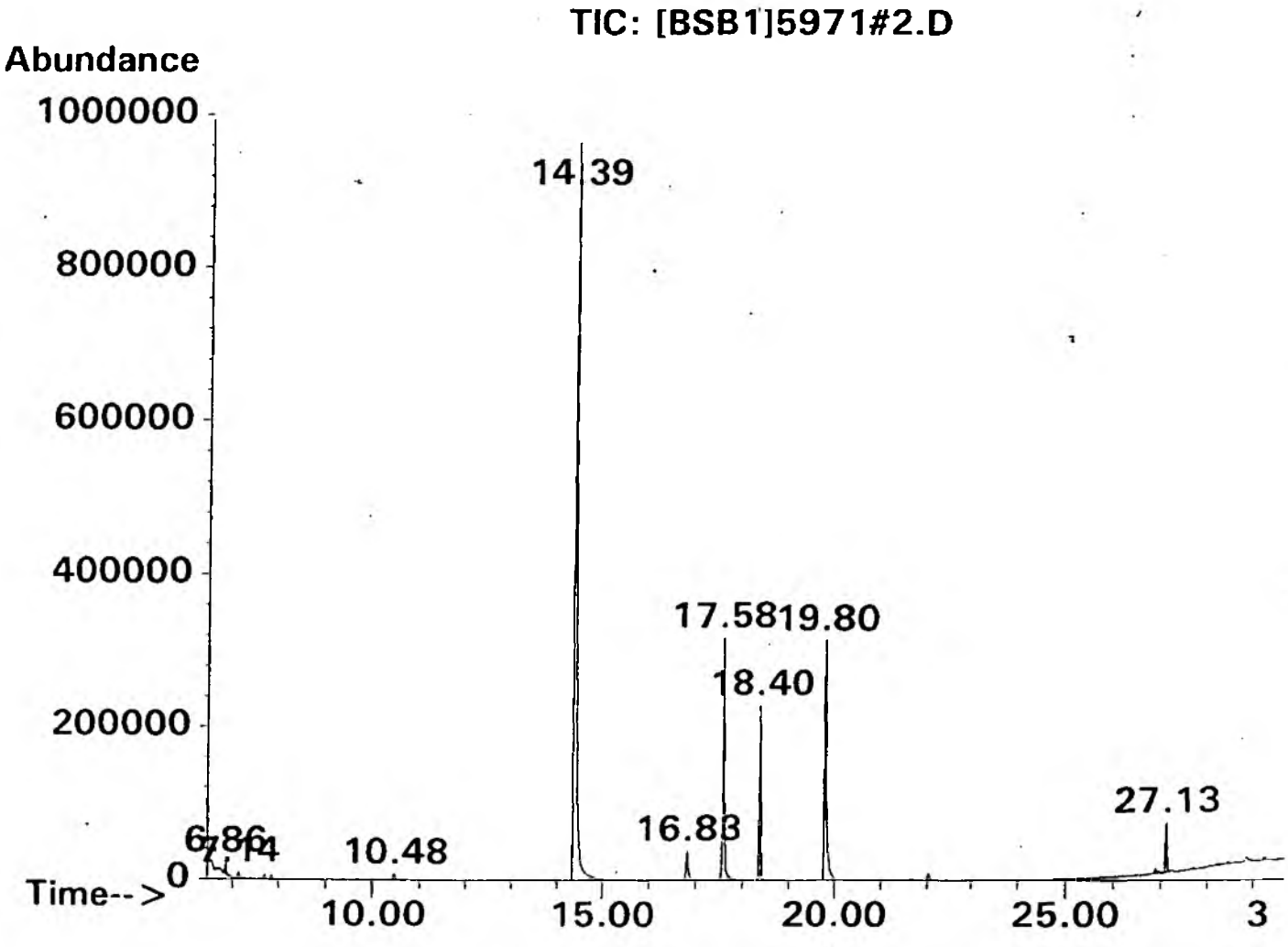
PHYSICAL APPEARANCE:- Colourless transparent water. Alot of very fine grey sediment.Some odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions.The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME R.T	LIBRARY ID.	CAS#	%MATCH	CONF. BY
6.86	UNIDENTIFIED			
7.14	UNIDENTIFIED			
10.48	UNIDENTIFIED			
14.39	Phosphoric triamide, hexamethyl HEMPA	680-31-9	91	YES
16.83	Pentamethyl phosphoramide	000-00-0	25	
17.57	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.40	Peak present in blank			
19.80	PESTOX (Schradan)	152-16-9	86	YES
27.13	Peak present in blank			

COMMENTS

The sample was found to contain quantities of the organophosphorous compounds HEMPA and SCHRADAN previously identified in samples 95027941 & 95027943. Further work established the amounts of these chemicals as 516 and 279 micrograms per litre respectively.

MASS SCAN ANALYSIS

SAMPLE No.:- 95028129

SITE:- HAUXTON - Agrevo - Piezometer No. 59 - PUMPED

DATE SAMPLED :- 23/8/95

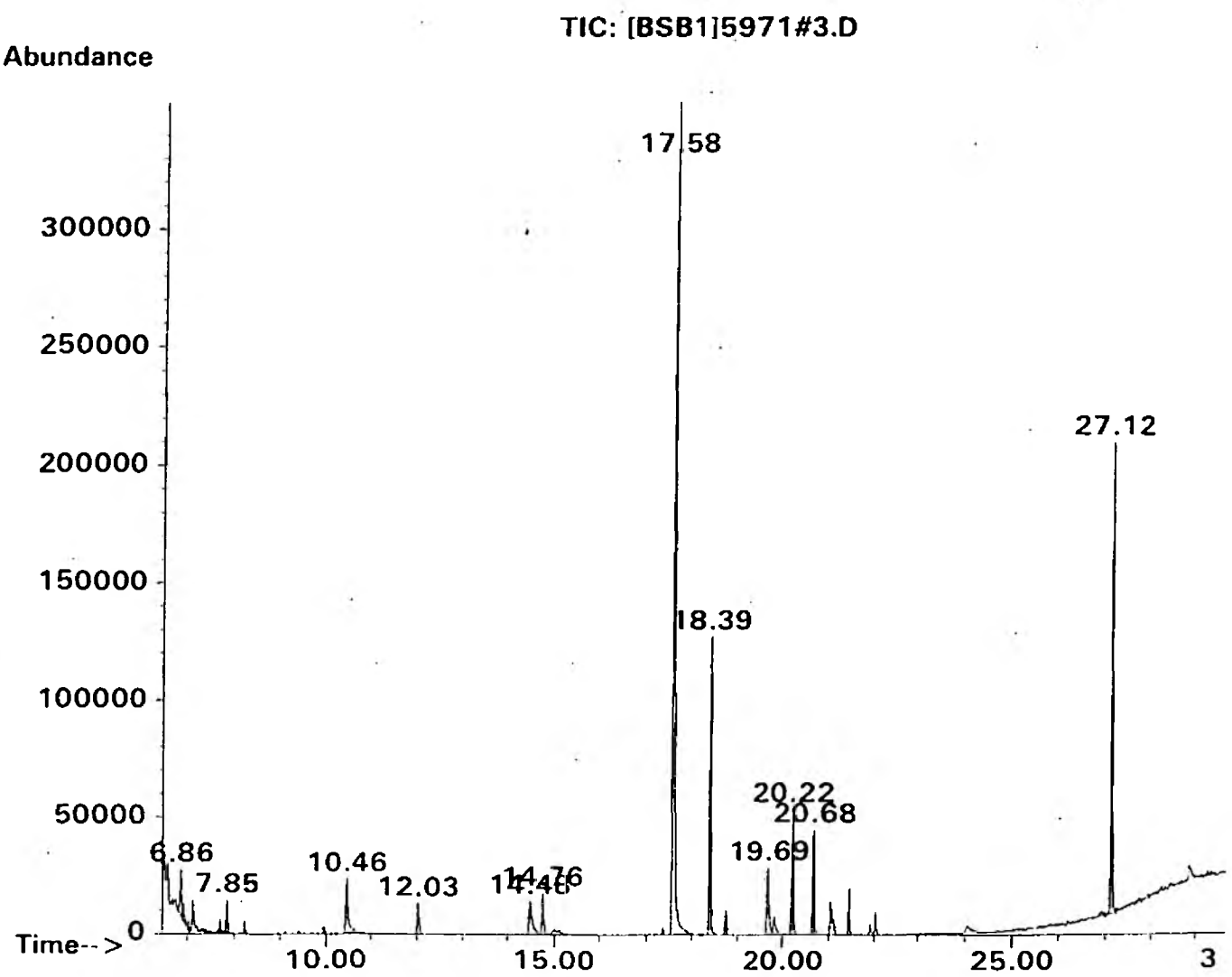
PHYSICAL APPEARANCE:- Colourless transparent water. Some brown/grey very fine sediment. Slight odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions.The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME R.T	LIBRARY ID.	CAS#	%MATCH	CONF. BY
7.85	Ethylbenzene	100-41-4	64	
10.46	1-Hexanol, 2-ethyl	104-76-7	83	
12.03	Benzene, 1-chloro-2-(chloromethyl)	611-19-8	83	
14.48	UNIDENTIFIED			
14.76	2,4,5-Trichlortoluene	000-00-0	91	
17.57	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.39	Peak present in blank			
18.74	UNIDENTIFIED			
19.68	Simazine	122-34-9	93	YES
20.22	Trietazine	1912-26-1	98	YES
20.68	6-Ethoxy-2,2-dimethoxy-2-3-dihydro	68365-05-9	40	
27.12	Peak present in blank			

COMMENTS

The triazine herbicides simazine and trietazine were positively identified as being present in the sample.

MASS SCAN ANALYSIS

SAMPLE No.:- 95028130

SITE:- HAUXTON - Garfitts land. Piezometer No. G4 - PUMPED

DATE SAMPLED :- 23/8/95

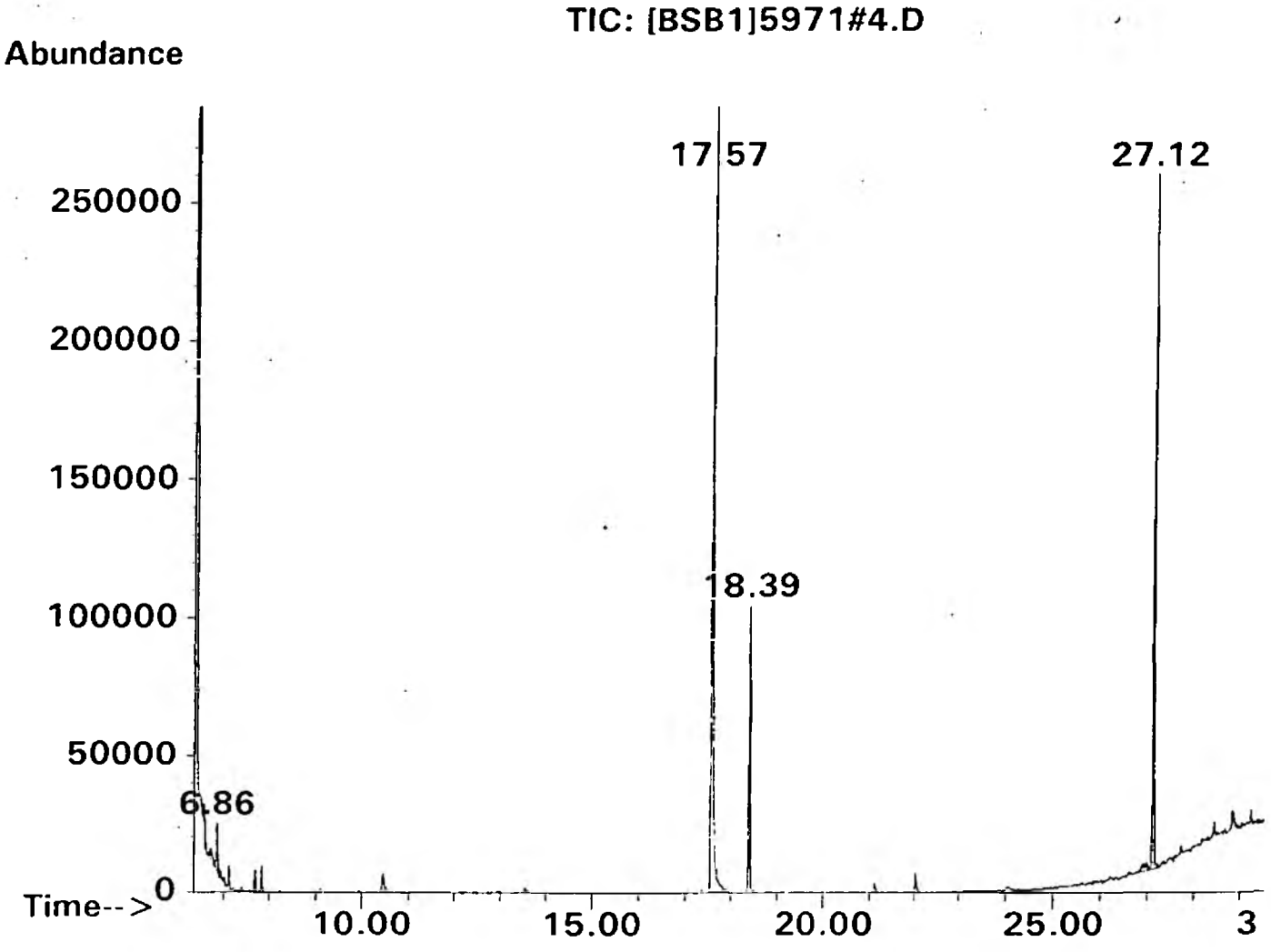
PHYSICAL APPEARANCE:- Colourless transparent water. Some very fine sediment. Slight odour.

SAMPLE PREPARATION:- 100mls of sample was spiked with Pentachlorobenzene to a concentration of 50ug/l and triple extracted with Dichloromethane under neutral, acidic and basic conditions.The extract was dried with sodium sulphate, reduced to 500ul in an evaporator and transferred to a GC vial for analysis.

ANALYSIS 1ul was injected onto a GC-MS system operating in scan mode.

RESULTS

CHROMATOGRAM



PEAK IDENTIFICATION

RET TIME R.T	LIBRARY ID.	CAS#	%MATCH	CONF. BY
17.57	PENTACHLOROBENZENE (I.S) ADDED AT 50 ug/l			
18.39	Peak present in blank			
27.13	Peak present in blank			

COMMENTS

The sample did not contain significant quantities of any chemical extractable into dichloromethane and amenable to gas chromatography.

RESULTS SUMMARY - COMPOUNDS PRESENT AT >1.0ug/L

ALL QUANTITATIVE RESULTS ARE IN MICROGRAMS PER LITRE

95027941

Quantitative Results:-

Trichloroethene	7,350	MCPA	4.30
HEMPA	4,470	Benzene	3.00
Schradan	2,490	2,5-Dichlorophenol	2.31
2,3,6-TBA	232	Dicamba	2.30
cis-1,2-Dichloroethene	207	2-Cresol	2.28
Chloroform	123	Toluene	2.20
trans-1,2-Dichloroethene	67.6	3-Cresol	2.18
Tetrachloroethene	65.2	Phenol	2.09
1,1-Dichloroethene	63.0	Ethylbenzene	1.70
4-Cresol	23.3	2,6-Dichlorophenol	1.67
Dichloromethane	11.3	p-chloro-o-Cresol	1.35
Xylene	6.40	2-Chlorophenol	1.21
2,4-Dichlorophenol	4.48	1,2-Dichloroethane	1.20

Qualitative Results:-

Confirmed:-	Tentatative:-	%Match
None	1,4-Oxathiane	91
	Ethane, 1,1-Oxybis [2-chloroethane	91
	Bisphenol A.	95
	Phenol, 2-chloro-6-methyl-	91
	Urea, tetramethyl	72
	1,4-Benzenedicarboxylic acid ester	53
	Pentamethylformylphosphoramide	47

95027942

Quantitative Results:-

Trichloroethene	6.40
Dichloromethane	3.80

Qualitative Results:-

Confirmed:-	Tentatative:-
None	None

95027943

Quantitative Results:-

Trichloroethene	10,000	Dicamba	4.50
HEMPA	9,100	Xylene	8.30
Schradan	3,290	Benzene	4.10
2,3,6-TBA	398	2-Cresol	2.75
cis-1,2-Dichloroethene	272	3-Cresol	2.50
Chloroform	220	Toluene	2.50
trans-1,2-Dichloroethene	91.2	2-Chlorophenol	1.73
1,1-Dichloroethene	75.3	2,6-Dichlorophenol	1.67
Tetrachloroethene	70.0	2,5-Dichlorophenol	1.62
MCPA	25.4	1,2-Dichloroethane	1.60
Dichloromethane	12.8	2,4-D	1.28
4-Chlorophenol	6.46	Phenol	1.24
4-Cresol	5.04	Ethylbenzene	1.10
2,4-Dichlorophenol	4.48		

Qualitative Results:-

Confirmed:-	Tentatative:-	%Match
None	Ethane, 1,1-Oxybis[2-chloroethane	97
	Phenol, 2-Chloro-6-methyl	94
	Phenol, 4,4'-butylidenebis[2-(1,1-	93
	Benzofuran	90
	1,4-Oxathiane	87
	Phenol, 2,5-bis(1-methylpropyl)	87
	Urea, tetramethyl	80
	Phenol, 2,6-bis(1-methylpropyl)	64
	Pentamethylphosphoramide	38
	Benzaldehyde, 4-methyl-,oxime	35

95027945

Quantitative Results:-

Trichloroethene	20.3
Dichloromethane	3.30
Toluene	1.00

Qualitative Results:-

Confirmed:-	Tentatative:-	%Match
None	Eicosane	87
	Heptacosane	86
	Phenol, 4,4'-butylidenebis[2-(1,1-	86
	Hexadecane, 2,6,10,14-tetramethyl	80
	Tricosane	80
	Octacosane	64
	Carbanochloridic acid, 2-chloro-1-	64

95028126

Quantitative Results:-

Schradan	22.8
HEMPA	3.97

Qualitative Results:-

Confirmed:-	Tentatative:-	%Match
Ethofumesate	2-Cresol	91
	Phenol, 4-chloro-3-methyl	91
	Naphthalene, 1-methyl	90
	Naphthalene, 2-methyl	90
	Ethane, 1,1'-Oxybis[2-chloro-	64
	Ethylbenzene	45
	Phosphine oxide, triphenyl-	45
	Heptane, 2,3-dimethyl	40

95028127

Quantitative Results:-

None

Qualitative Results:-

Confirmed:-	Tentatative:-	%Match
Simazine	Eicosane	95
Trietazine	Pentacosane	86
Ethofumesate	Hexadecane, 2,6,10,14-tetramethyl	86
	Tricosane	78
	Tridecane	64
	Ethanol, 2-[2-(butoxyethoxy)etho	64
	Triacetin	36

95028128

Quantitative Results:-

HEMPA	516	Dichloromethane	3.8
Schradan	279	1,2-Dichlorobenzene	2.5
Trichloroethene	48.5	1,4-Dichlorobenzene	1.4
cis-1,2-Dichloroethene	14.2	1,3-Dichlorobenzene	1.1
1,1-Dichloroethene	12.6		

Qualitative Results:-

Confirmed:-	Tentatative:-
None	None

95028129

Quantitative Results:-

HEMPA	16.8	Toluene	5.10
Trichloroethene	16.6	Dichloromethane	4.10
Schradan	15.3	Xylene	3.80
Simazine	13.1	Tetrachloroethene	3.60
Trietazine	8.30	Ethiofumesate	2.25
2,3,6-TBA	8.26	Benzene	1.20

Qualitative Results:-

Confirmed:-	Tentatative:-	%Match
None	2,4,5-Trichlortoluene	91
	1-Hexanol, 2-ethyl	83
	Benzene, 1-chloro-2-(chloromethyl)	83
	Ethylbenzene	64
	6-Ethoxy-2,2-dimethoxy-2-3dihydro	40

95028130

Quantitative Results:-

Trichloroethene	7.00
Dichloromethane	4.00
Simazine	1.30
Toluene	1.30

Qualitative Results:-

Confirmed:-	Tentatative:-
None	None

APPENDIX

PHYSIOCHEMICAL PROPERTIES OF SCHRADAN AND HEMPA

SCHRADAN

CAS NUMBER	152-16-9
CHEMICAL NAME	Octamethyldiphosphoramide
SYNONYMS	OMPA; PESTOX; Octamethylpyrophoshporamide; octamethylpyrophosphoric tetramide.
FORMULA	$C_8H_{24}N_4O_3P_2$
USES	Schradan is a systemic insecticide effective against sap-feeding insects and mites but is relatively inert as a contact insecticide. It is non-phytotoxic at insecticidal concentrations. It has now been withdrawn from use.
PHYSICAL PROPERTIES	**M.Pt** 14-20°C; **B.Pt$_{0.5}$** 120-125°C; **Specific gravity d^{20}** 1.137; **Volatility** v.p 1 x 10^{-3}mmHg at 25°C. It is miscible with water and most organic solvents, is slightly soluble in petroleum oils and is readily extracted from aqueous solution by chloroform. It is stable to water and alkali, but hydrolysed under acid conditions to dimethylamine and orthophosphoric acid.
SUPPLY CLASSIFICATION	Very toxic.
RISK PHRASES	Very toxic in contact with skin and if swallowed.
TOXICITY	LD_{50} oral rat 5 mg/kg 96h LC50 Guppy 22mg/l 48h LC50 Fathead minnow 144mg/l

HEMPA

CAS NUMBER	680-31-9
CHEMICAL NAME	Hexamethylphosphoramide
SYNONYMS	Hexametapol; Hexamethylphosphoric triamide; Tris(dimethylamino) phosphine oxide.
FORMULA	$C_6H_{18}N_3OP$
USES	Aprotic solvent; de-icing additive for jet fuels; Chemosterilant for insect pests; Chemical mutagen.
PHYSICAL PROPERTIES	**M.Pt** 7.2°C; **B.Pt** 235°C; Colourless liquid completely miscible with water.
SUPPLY CLASSIFICATION	Toxic
RISK PHRASES	Toxic by inhalation, in contact with skin and if swallowed. May cause cancer. May cause heritable genetic damage.
TOXICITY	LD_{50} oral rat 2,650 mg/kg