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TITLE: ANALYSIS OF DECATUR WATER SAMPLE FOR PRIORITY POLLUTANTS

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ABSTRACT: A water sample from the Decatur Plant was analyzed for the U.S. EPA designated priority pollutants, and a number of pollutants as designated by the NPDES Consolidated Permits as Group B pollutants, Results of these analyses are shown in this report.

Approved by: Robert F. Ivory

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1. INTRODUCTION

A water sample collected at the Decatur Plant was submitted to the Dayton Laboratory for priority pollutant screening analyses. The analyses were conducted in accordance with the U. S. Environmental priority pollutants. [1].

2. SUMMARY

All analyses were conducted according to the procedures developed and recommended by the U.S. EPA [1-5]. The organic priority pollutants found in the Decatur Plant water sample are shown in Table 1 and Table 2 shows the applicable quality control data. Although all the organic priority pollutants listed in Table 3 were investigated through GC/MS screening analysis, only those pollutants observed in the samples are reported. None of the Aroclors or pesticides listed in Table 4 were detected above their screening levels. The analytical results for the priority and nonpriority pollutant metals, as determined by atomic absorption (AA) and inductively coupled plasma (ICP) spectroscopy, are shown in Table 5. Table 6 shows the quality control data for the metals analyses. Table 7 shows the detection limits for the metals concentrations. Table 8 shows the analytical results for the total phenol and cyanide concentrations in the water samples, and the applicable quality control data. Table 9 shows the results of analyses for a number of the Group B pollutants as designated by the NPDES consolidated permit. The Quality Control data for these analyses are shown in Table 10.

3. SAMPLE COLLECTION

Samples were collected by Decatur Plant personnel. Samples were received at the Dayton Laboratory in the designated EPA-approved sampling containers.

4. PRIORITY POLLUTANT ANALYTICAL PROCEDURES

Analytical procedures developed and recommended by EPA [1] were used throughout this project. It is important to realize that the analysis scheme for analyzing for organic priority pollutants is a screening procedure, and is therefore a qualitative semi-quantitative analysis. Some of the organic priority pollutant analytical methods are still under development and require further verification and validation. Therefore, the data presented for organic species serve to identify which of the organic pollutants were present and to indicate their general concentration ranges. The accuracy of these data may vary by a factor of 2 to 10, depending on the overall composition and concentration of the materials present.

Two of the 129 priority pollutants were not determined in the project: 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) and asbestos. The EPA Environmental Monitoring and Support Laboratory (EMSL) recommends that TCDD be omitted because of its extreme toxicity and the potential health hazard involved in preparing standard solutions in the laboratory from the pure compound [1], and therefore, the compound is not included as part of our routine screening procedures. There is presently no EPA-approved method for the determination of asbestos fibers in wastewater.

The analytical procedures used [1] divides the 129 priority pollutants into six basic categories: volatile organics, extractable organics, metals, cyanide, total phenol and asbestos. The following sections outline the analytical procedures and the EPA-approved Dayton Laboratory's modifications for analyzing each group of priority pollutants.

4.1 Extractable Organics

The 129 priority pollutants contain 81 solvent-extractable organic compounds that can be separated by gas chromatographic methods. Extractable organics are divided into three groups for analysis: base/neutral organics, acid organics (phenols), pesticides and Aroclors. Compounds classified as extractable organics are listed by these groups in Table 3.

The analytical procedure for extractable organics is described in Reference 1. Figure 1 depicts the sample processing scheme for

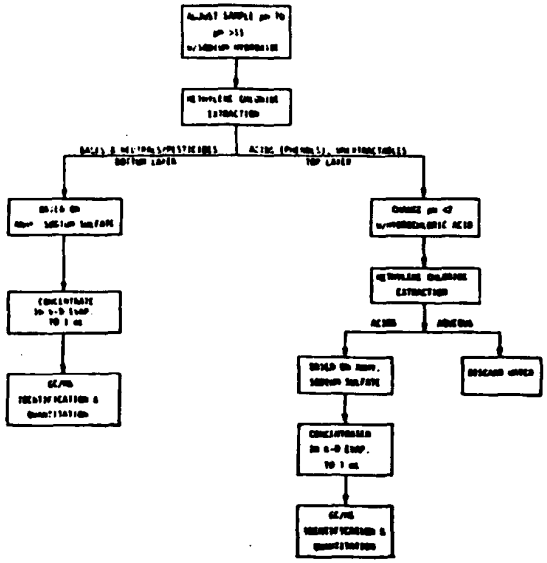

Figure 1. Sample processing scheme for the analysis of the base/ neutral and acid fractions of extractable organics.

the base/neutral and acid fractions. A 1-liter aliquot of the extractable organics was made alkaline (pH >11) with sodium hydroxide, and the organic base/neutral compounds extracted with methylene chloride. The residual aqueous phase was acidified (pH <2) with hydrochloric acid, and the organic acids were then extracted with methylene chloride.

The methylene chloride extracts, one containing the base/neutral organics and the other the acidic organics, were dried on columns of anhydrous sodium sulfate. These dried extracts were concentrated to 1.0 mL in Kuderna-Danish (K-D) evaporative concentrators. The concentrates were sealed in septum-capped vials and stored at 4°C until analysis. Gas chromatographic/mass spectrometric (GC/MS) analysis was conducted using SP-2250-DB and SP-1240-DA GC columns for the base/neutral and acid extracts, respectively. Each sample was spiked with deuterated anthracene (an internal standard) immediately prior to analysis at the GC/MS.

The sample processing scheme used for pesticides and Aroclors is shown in Figure 2. A 1-liter aliquot of the original extractable organics sample was adjusted to a pH of 6.5 to 7.5 using either 50% NaOH or concentrated HCl, and the pesticides and Aroclors were extracted into a 15% methylene chloride/hexane solution. The organic phase was separated and the sample cleaned up by acetonitrile partition. Following the cleanup procedure, the samples were screened by gas chromatography on SP-2250/SP-2401 and 3% OV-101 columns, utilizing an electron capture detector.

4.2 Volatile Organics

Table 3 lists those priority pollutants classified as volatile organics. The recommended method for volatile organic analysis was designed by EPA for determining those chemical species which

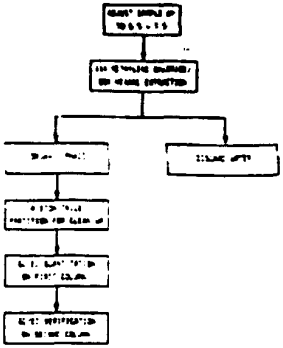

Figure 2. Sample processing scheme for pesticides and Aroclor analysis.

are amenable to the Bellar purge and trap method [1]. Hermetically sealed 40 mL glass vials collected from each of the sampling points were composited in the laboratory for one analysis of each sampling point. Two laboratory composite solutions were prepared, one for analysis and one as a backup sample. Figure 3 is a simplified diagram of the analytical scheme used for volatile organics analysis. An internal standard, 1,4-dichlorobutane, was added to 5 mL of the composited sample, and this spiked sample was sparged with helium onto a Tenax-GC silica-packed sample tube.

Analyses were completed using a Hewlett-Packard 5981 GC-mass spectrophotometer with a 5937 data system. Sample tubes were heated to 180°C over a 1-minute period and held at that temperature for 4 minutes to desorb the compounds onto a Carbowax 1500 column held at -40°C. For compounds with boiling points below room temperature, cryogenic trapping at -40°C (liquid nitrogen cooling) generally provides better reproducibility of retention time than does the suggested protocol temperature of 30°C. After desorption, the GC column temperature was raised at 8°C/minute to 170°C.

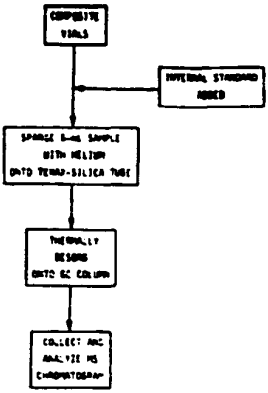

Figure 3. Analytical scheme for volatile organic analysis.

Qualitative identification of a compound was made using the three criteria listed in the protocol [1]: 1) the characteristic ions must be found to maximize in the same spectrum, 2) retention time must coincide with known retention times, and 3) ratios of the three characteristic masses must stand in the proper known proportions. Quantification of volatile organics was made using response ratios to the 1,4-dichlorobutane standard.

4.3 Metals

In addition to extractable and volatile organics, the 129 priority pollutants include 13 metals which are to be analyzed for under the protocol. Four metals (arsenic, mercury, selenium, and thallium) are analyzed by atomic absorption (AA) spectroscopy. All other metals were determined by an inductively coupled plasma (ICP) excitation technique.

4.4 Total Cyanide

Total cyanide was determined according to the conventional wet chemistry technique described in Reference 3. Independent standards were included with the plant samples for quality assurance.

4.5 Total Phenol

In addition to specific phenolic compounds and phenol measured by GC/MS, total phenol was also measured by typical wet chemistry techniques [1-3]. Independent phenol standards were also analyzed for quality assurance.

4.6 Non-Priority Pollutant Compounds

A number of pollutants as designated by the NPDES Consolidated Permit as Group B pollutants were analyzed in accordance with approved EPA methods for specified compounds. [2,3].

5. REFERENCES

1. Draft Final Report: Sampling and Analysis Procedures for Screening of Industrial effluents for Priority Pollutants. U.S. Environmental Protection Agency, Cincinnati, Ohio. April 1977. 145 pp.

2. Manual of Methods for Chemical Analysis of Water and Wastes. EPA-60014-79-020. U.S. Environmental Protection Agency, EMSL, Cincinnati, Ohio, March 1979.

3. Standard Methods for Examination of Water and Wastewater, Fourteenth Edition. American Public Health Association, Washington, D.C., 1976. 1193 pp.

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

5. U.S., Federal Register, Book 3, Vol. 45, No. 98, Monday, May 1, 1980.

TABLE 1. ANALYTICAL RESULTS FOR ORGANIC PRIORITY POLLUTANTS

	µgL (ppb) Sample ID	
Analyte	Method Blank	CCO, 2/27/82
Extractable organics		
Bis-(2-ethylheyl)-phthalate	190	<10[a]
Fluoranthene	<10	<10
Pyrene	<10	ND[b]
Chrysene	<10	ND
Phenol	ND	<25
Volatile Organics		
Methylene Chloride	ND	<10
Chloroform	ND	<10
Trichloroethylene	10	<10
Benzene	<10	<10
Acrylonitrile	ND	800
Toluene	<10	<10

[a] <=Compound was observed, but below screening level

[b] ND=Compound not detected

TABLE 2. ORGANIC ANALYSES QUALITY CONTROL DATA

Analyte	Amount of Spike, ug/L	Percent Recovery in sample: Method Blank	Percent Recovery in sample: CCO, 2/27/82
D_4-Dichlorobenzene	100	36	24
D_5-Phenol	100	27	22
D_{10}-Pyrene	100	61	42
D_{12}-Chrysene	100	58	42

TABLE 3. PRIORITY POLLUTANT SCREENING LEVELS GC/MS ANALYSIS OF ORGANICS IN INDUSTRIAL EFFLUENTS

Compound	Screening level, µg/L	Compound	Screening level, µg/L
Acids		**Base/Neutrals:**	
2-Chlorophenol	25	1,3-Dichlorobenzene	10
2-Nitrophenol	25	1,4-Dichlorobenzene	10
Phenol	25	Hexachloroethane	10
2,4-Dimethylphenol	25	1,2-Dichlorobenzene	10
2,4-Dichlorophenol	25	Bis(2-chloroisopropyl) ether	10
2,4,6-Trichlorophenol	25	Hexachlorobutadiene	10
4,6-Dinitro-o-cresol	250	1,2,4-Trichlorobenzene	10
2,4-Dinitrophenol	250	Naphthalene	10
p-Chloro-m-cresol	25	Bis(2-chloroethyl) ether	10
Pentachlorophenol	25	Hexachlorocyclopentadiene	10
4-Nitrophenol	25	Nitrobenzene	10
		Bis(2-chloroethoxy) methane	10
Volatiles:		2-Chloronaphthalene	10
Chloromethane	10	Acenaphthylene	10
Dichlorodifluoromethane	10	Acenaphthene	10
Bromomethane	10	Isophorone	10
Vinyl chloride	10	Fluorene	10
Chloroethane	10	2,6-Dinitrotoluene	10
Methylene chloride	10	1,2-Diphenylhydrazine	10
Trichlorofluoromethane	10	2,4-Dinitrotoluene	10
1,1-Dichloroethylene	10	N-nitrosodiphenylamine	10
1,1-Dichloroethane	10	Hexachlorobenzene	10
trans-1,2-Dichloroethylene	10	4-Bromophenyl phenyl ether	10
Chloroform	10	Phenanthrene/anthracene	10
1,2-Dichloroethane	10	Dimethyl phthalate	10
1,1,1-Trichloroethane	10	Diethyl phthalate	10
Carbon tetrachloride	10	Fluoranthene	10
Bromodichloromethane	10	Pyrene	10
Bis(chloromethyl) ether	10	Di-n-butyl phthalate	10
1,2-Dichloropropane	10	Benzidine	10
trans-1,3-dichloropropene	10	Butyl benzyl phthalate	10
Trichloroethylene	10	Chrysene	10
Dibromochloromethane	10	Bis(2-ethylhexyl) phthalate	10
cis-1,3-dichloropropene	10	Benz(a)anthracene	10
1,1,2-Trichloroethane	10	Benzo(b)fluoranthene	10
Benzene	10	Benzo(k)fluoranthene	10
2-Chloroethyl vinyl ether	10	Benzo(a)pyrene	10
Bromoform	10	Indeno(1,2,3-cd)pyrene	25
Tetrachloroethylene	10	Dibenzo(a,h)anthracene	25
1,1,2,2-Tetrachloroethane	10	Benzo(g,h,i)perylene	25
Toluene	10	N-Nitrosodimethylamine	10
Chlorobenzene	10	N-nitrosodi-n-propylamine	10
Ethylbenzene	10	4-Chlorophenyl phenyl ether	10
		3,3'-Dichlorobenzidine	10
Parent Injectables:		Di-n-octyl phthalate	10
Acrolein	200		
Acrylonitrile	100	**Pesticides and Aroclors**	10

TABLE 4. PRIORITY POLLUTANT PESTICIDES AND AROCLORS

Compound	GC/MS Screen, µg/L	GC/EC, µg/L
β-Endosulfan	10	1
α-BHC	10	1
γ-BHC	10	1
β-BHC	10	1
Aldrin	10	1
Heptachlor	10	1
Heptachlor epoxide	10	1
α-Endosulfan	10	1
Dieldrin	10	1
4,4'-DDE	10	1
4,4'-DDD	10	1
4,4'-DDT	10	1
Endrin	10	1
Endosulfan sulfate	10	1
δ-BHC	10	1
Chlordane	10	1
Toxaphene	10	1
Endrin aldehyde	10	1
Methoxychlor	10	1
Aroclor 1242	10	1
Aroclor 1254	10	1
Aroclor 1221	10	1
Aroclor 1232	10	1
Aroclor 1248	10	1
Aroclor 1260	10	1
Aroclor 1016	10	1

TABLE 5. ANALYTICAL RESULTS FOR PRIORITY AND NONPRIORITY METAL POLLUTANTS, µg/L (ppb)

Analyte	Sample ID CCO, 2/27/82
Priority metal pollutants	
Arsenic[a]	3.3
Mercury[a]	<0.8
Selenium[a]	4.9
Thallium[a]	<63
Antimony	<730
Beryllium	<23
Cadmium	<30
Chromium	<36
Copper	<52
Lead	<880
Nickel	102
Silver	<80
Zinc	432
Nonpriority metal pollutants	
Aluminum	740
Barium	16
Boron	8.4×10^2
Calcium	1.3×10^5
Cobalt	<99
Iron	2.3×10^3
Magnesium	1.2×10^3
Manganese	35
Molybdenum	<130
Potassium	2.5×10^4
Sodium	4.2×10^5
Strontium	31
Tin	$<5.3 \times 10^3$
Titanium	16
Vanadium	<150

[a]Analyses by AA; all others by ICP

NOTE: ICP values are an average of duplicate analyses

TABLE 6. QUALITY CONTROL FOR METALS ANALYSES
µg/L (PPB)

Analyte	Reference Standard	Observed Value	True Value	% Recovery
Aluminum	ICP	969	1,000	97
Barium	ICP	1,060	1,000	106
Beryllium	ICP	1,090	1,000	109
Calcium	ICP	1,040	1,000	104
Cadmium	ICP	1,020	1,000	10
Cobalt	ICP	1,100	1,000	110
Chromium	ICP	1,100	1,000	110
Copper	ICP	1,020	1,000	10.
Iron	ICP	1,050	1,000	105
Manganese	ICP	1,090	1,100	109
Nickel	ICP	1,110	1,100	111
Lead	ICP	1,050	1,100	105
Antimony	ICP	1,100	1,100	100
Titanium	ICP	1,070	1,100	107
Vanadium	ICP	1,090	1,100	109
Zinc	ICP	1,050	1,100	105
Arsenic[a]	SRMA	80	77	104
Selenium[a]	SRMA	12	11	111
Mercury[a]	Tm-CL	19	20	93
Thallium[a]	Spex A	981	1,100	98

[a]Analyses by AA: all others by ICP

TABLE 7. LOQ[a] Values for ICP METALS as of 3/23/82

Analyte	LOQ Value, µg/L (ppb)
Aluminum	480
Antimony	730
Barium	13
Beryllium	23
Boron	580
Cadmium	30
Calcium	1.8×10^4
Chromium	36
Cobalt	99
Copper	23
Iron	17
Lead	880
Magnesium	63
Manganese	5
Molybdenum	130
Nickel	30
Silver	80
Tin	5.3×10^3
Titanium	6
Vanadium	150
Zinc	7

[a]Lowest observable quantity

TABLE 8. TOTAL PHENOL AND CYANIDE CONCENTRATIONS

µg/L (ppb)

Sample ID	Phenol	Cyanide
CCO, 2/27/82	11	50

QUALITY CONTROL DATA

Reference Standard	Observed Value, µg/L	True Value, µg/L	% Recovery
Total Phenol	222	250	89
Cyanide	110	160	69

TABLE 9. ANALYTICAL RESULTS-FOR NON-PRIORITY POLLUTANTS IN DECATUR SAMPLE

Analyte	Sample ID CCO, 2/27/82, mg/L
Total organic nitrogen	38.6
Surfactants	0.04
Oil and grease	2
Nitrate/Nitrile as N	0.8
Sulfate	974
Sulfite	150
Sulfide	1.0
Total Phosphorous	26.2

TABLE 10. QUALITY CONTROL DATA FOR NON-PRIORITY POLLUTANTS

Analyte Standard	Observed Value mg/L	True Value mg/L	% Recovery
Oil & Grease	16	19	84
Oil & Grease	25	26	96
Total Organic Nitrogen	5.4	5.4	100
Sulfate (ERA 3253)	221	221	100
Nitrate (ERA 3253)	4.5	4.0	111
Total Phoaphate (ERA yellow)	3.4	3.3	103