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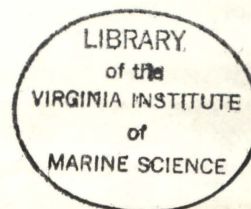
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Analyses of Sediment and Elutriate  
Samples from the James River, Virginia

by

R. J. Huggett

Virginia Institute of Marine Science



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July, 1975

Corps of Engineers Contract No.  
DACW-65-75-M-2262

\* Note: Samples and sample numbers were supplied by the  
Corps of Engineers, Norfolk District.



## Methods and Procedures

### Sediment Samples

#### Total and Volatile Solids (TS & VS)

The sediment samples were dried in an oven at 103°C to constant weight, (dry weight/wet weight x 100 = % total solids). The dried samples were placed in a muffle furnace for one hour at 550-600°C. The decrease in weight after ashing was reported as volatile solids.

#### Chemical Oxygen Demand (COD)

This parameter was determined by the dichromate reflux method. The oxidizable substances were oxidized by a standard solution of potassium dichromate in sulfuric acid. The excess dichromate was titrated with standard ferrous ammonium sulfate. Silver sulfate was used as a catalyst; mercuric sulfate was used to eliminate the interference of chloride ions.

#### Total Kjeldahl Nitrogen (TKN)

The samples were digested with a solution containing sulfuric acid, potassium sulfate, and mercuric sulfate converting organic nitrogen to ammonium sulfate. The digested samples were steam-distilled into a saturated boric acid solution and titrated with standard hydrochloric acid.

#### Total Phosphorus (TP)

The samples were digested in concentrated  $\text{HNO}_3$  and evaporated to dryness; concentrated  $\text{H}_2\text{SO}_4$  was added and heated until the solution cleared. Water was added and the samples were filtered through a glass filter. The filtrates were analyzed for total phosphorus by the single solution method, using ascorbic acid as the reducing agent. The developed samples were read on a Klett-Summerson Photoelectric colorimeter, model 900-3.

#### Metals (Cd, Zn and Pb)

One gram of sample was heated to fuming with ten milliliters of concentrated  $\text{HNO}_3$  acid. After cooling, ten additional milliliters of acid were added, heated and cooled. The samples were centrifuged and the supernatants measured for volume and analyzed on a Varian Atomic Absorption Spectrophotometer, model AA-5.

#### Mercury

The samples were digested with concentrated  $\text{H}_2\text{SO}_4$  overnight. The digested samples were oxidized with 5%  $\text{KMnO}_4$  and transferred to 300 ml BOD bottles. After the addition of reductant\* solution the BOD bottles were immediately attached to the aeration apparatus of a Coleman Mercury Analyzer MAS-50. Mercury concentrations were determined from standard curves.



## Mercury (cont'd.)

\* Composition of reductant solution:

H <sub>2</sub> O	600 ml
H <sub>2</sub> SO <sub>4</sub>	100 ml
NaCl	5 grams
(NH <sub>2</sub> OH) <sub>2</sub> SO <sub>4</sub>	10 grams
SnSO <sub>4</sub>	20 grams
q.s. to 1 liter	

## Oil and Grease (O & G)

The sediment samples were dried with magnesium sulfate monohydrate, then soxhlet-extracted with hexane (Standard Methods for the Examinations of Water and Wastewater, 12th Ed., APHA, Inc., N.Y., 1965; 531-532). The hexane was then evaporated to dryness. The weight of solid residue from the solvent evaporation yields oil and grease.

## Elutriate and Site Water Samples

### Elutriate Preparation

A 500 gram portion of the homogenized sediment was mixed with 2 liters of "site" water in a one-gallon wide-mouth jar and agitated on a Burrell shaker for 30 minutes. The agitated mix was allowed to settle for 1½ hours then centrifuged for ten minutes at  $10 \times 10^3$  rpm (9800 x gravity) and filtered through 0.45u millipore filters.

## Oil and Grease (O & G)

One liter of the centrifuged supernatant was acidified with 5 ml of concentrated H<sub>2</sub>SO<sub>4</sub>, put into a separatory funnel with 40 ml of petroleum ether and shaken for two minutes. The petroleum ether was removed and placed in a distilling flask.

The procedure was repeated with an additional 40 ml of petroleum ether.

The separatory funnel was rinsed with 20 ml of petroleum ether. The combined petroleum ether extracts were evaporated on a water bath to 10 ml which was then put into a tared weighing bottle. The sample was taken to dryness with a stream of N<sub>2</sub>. The bottle was put into a desicator for 30 minutes before weighing.

## Metals

Lead, Cadmium and Zinc were determined by the solvent extracting technique on a Varian Aerograph AA-5 Atomic Absorption Spectrophotometer. Ammonium Pyrolodine Dithiocarbamate (APDC) was used as a chelating agent and the metals were extracted with Methyl Isobutyl Ketone (MIBK), (Methods for Chemical Analysis of Water and Wastes, Environmental Protection Agency, 1971).



## Metals (cont'd.)

Mercury determinations were made by wet digestion and flameless atomic absorption spectrophotometry. Five ml of  $H_2SO_4$  was added to 100 ml of sample and the mixture was digested for 30 minutes. One ml of 5%  $KMnO_4$  was added to the digested sample and let stand for 3 hours before analysis.

The digested and oxidized sample was transferred to a 300 ml BOD bottle. After the addition of 10 ml of reductant solution, the BOD bottle was immediately attached to the aeration apparatus of a Coleman Mercury Analyzer MAS-50.

## Chemical Oxygen Demand (COD)

This parameter was determined by the dichromate reflux method. The oxidizable substances were oxidized by a standard solution of potassium dichromate in sulfuric acid. The excess dichromate was titrated with standard ferrous ammonium sulfate. Silver sulfate was used as a catalyst; mercuric sulfate was used to eliminate the interference of chloride ions (Methods for Chemical Analysis of Water and Wastes, Environmental Protection Agency, 1971).

## Total Kjeldahl Nitrogen (TKN)

The sample (50 ml) was digested with a solution containing sulfuric acid, potassium sulfate, and mercuric sulfate converting organic nitrogen to ammonium sulfate. The digested sample was steam-distilled into a saturated boric acid solution and titrated with standard hydrochloric acid.

## Total Phosphorus (TP)

Samples (50 ml) were digested by the persulfate oxidation technique and run by the single solution method, using ascorbic acid as the reducing agent. The developed samples were read on a Klett-Summerson Photoelectric Colorimeter, model 900-3.

# Site Water for the Elutriate Samples

Site Water Sample	COD <u>ppm</u>	TKN <u>ppm</u>	TP <u>ppb</u>	Zn <u>ppb</u>	Cd <u>ppb</u>	Pb <u>ppb</u>	Hg <u>ppb</u>	O&G <u>ppm</u>
1	7.7	0.55	33.8	2.61	<0.2	<1.0	0.10	6.3
5	5.7	0.34	35.7	0.52	<0.2	<1.0	0.05	1.0
9	1.5	0.55	46.2	1.16	<0.2	<1.0	0.15	2.4

# Elutriate Samples

Elutriate Sample	COD <u>ppm</u>	TKN <u>ppm</u>	TP <u>ppb</u>	Zn <u>ppb</u>	Cd <u>ppb</u>	Pb <u>ppb</u>	Hg <u>ppb</u>	O&G <u>ppm</u>
1A	1.9	2.22	37.5	0.48	<0.2	<1.0	0.14	4.2
B	1.9	1.90	39.4	0.32	<0.2	<1.0	0.02	3.0
5A	0.8	2.26	32.6	0.72	<0.2	<1.0	0.02	0.5
B	1.5	1.99	36.3	0.88	<0.2	<1.0	0.02	0.3
9A	2.3	2.45	31.6	1.08	<0.2	<1.0	0.03	0.5
B	1.9	2.86	33.5	1.44	<0.2	<1.0	0.01	0.8



## James River Sediment Samples

Sample	TS %	VS %	COD %	TKN mg/kg	TP ppm	Zn ppm	Cd ppm	Pb ppm	Hg ppm	O&G mg/kg
1	36.87	9.49	8.08	2160.	425.	134.	0.81	36.1	0.33	1170.
2	69.66	1.99	1.98	470.	180.	38.2	0.10	15.4	0.19	165.
3	30.64	12.50	10.2	2680.	584.	156.	0.65	50.6	0.46	1175.
4	47.37	6.84	5.91	1625.	312.	105.	0.27	28.1	0.30	430.
5	30.04	10.59	10.45	3125.	489.	175.	1.66	47.6	0.23	1100.
6	42.18	9.66	7.80	2320.	320.	138.	1.19	37.2	0.76	690.
7	30.59	10.73	9.45	2750.	555.	160.	0.88	54.3	0.46	590.
8	41.27	9.49	8.29	2540.	504.	137.	0.80	39.5	0.44	460.
9	33.94	10.29	9.81	2920.	507	177.	1.41	53.3	0.47	1160.
10	43.30	8.50	8.71	1940.	321.	139.	1.43	40.4	0.21	740.
11	41.93	8.57	8.94	1860.	289.	118.	0.69	38.2	0.17	535.
12	41.67	9.51	8.42	1920.	329.	148.	1.06	44.9	0.46	900.