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The Chemical Analysis of Water and Sediments in the Genesee River Watershed Study

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THE CHEMICAL ANALYSIS
OF WATER AND SEDIMENTS
IN THE GENESEE RIVER
WATERSHED STUDY



J. Makarewicz

SUMMARY OF PROCEDURES

PREPARED BY

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DECEMBER 1975

ENVIRONMENTAL HEALTH CENTER
DIVISION OF LABORATORIES AND RESEARCH
NEW YORK STATE DEPARTMENT OF HEALTH

This document describes the analytical procedures currently used at the Environmental Health Center, New York State Department of Health, for the chemical analysis of water and sediments in the Genesee River Watershed Study.

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II. SEDIMENT ANALYSIS (FLOW CHART)

A. NUTRIENT ELEMENTS

1. NITROGEN, Total N in dry solids
2. PHOSPHORUS, Total P in dry solids
3. CARBON, Total C in dry solids
4. CARBON, Total organic C in dry solids

B. METALS

1. ARSENIC
2. CALCIUM
3. CADMIUM
4. CHROMIUM
5. COPPER
6. IRON
7. LEAD
8. MAGNESIUM
9. MANGANESE

10. MERCURY
11. NICKEL
12. POTASSIUM
13. SODIUM
14. ZINC

BIBLIOGRAPHY

Water Column Analysis

The methods outlined in this manual for water column analysis have been used during the past seven years in the New York State Department of Health eutrophication research program directed by Dr. G. W. Fuhs.

A flow diagram of the sample handling and preservation techniques for nutrients and trace metals is presented in Fig. 1. The sample is split into several subsamples as required. If dissolved and particulate analyses are desired, a 300-ml subsample is filtered in the field through a 0.45- μ m Celite-coated Millipore filter. The filtrate and the resuspended residue are then analyzed for dissolved and particulate material respectively. Aliquots of the acidified subsample are used for trace metal analysis by flame atomic absorption spectrophotometry. Separate aliquots are used for the determination of arsenic and mercury.

The statistical information presented for each parameter was obtained in this laboratory during 1975.

The range reported refers to the actual working range used in this laboratory in routine analysis of large numbers of samples.

Minimum reportable concentration indicates the lowest result reported for an analytical determination. This value corresponds to an estimate of the result which is different from zero at the 95% confidence level. Results that are smaller than one-half the minimum reportable concentration are reported as "less than" values.

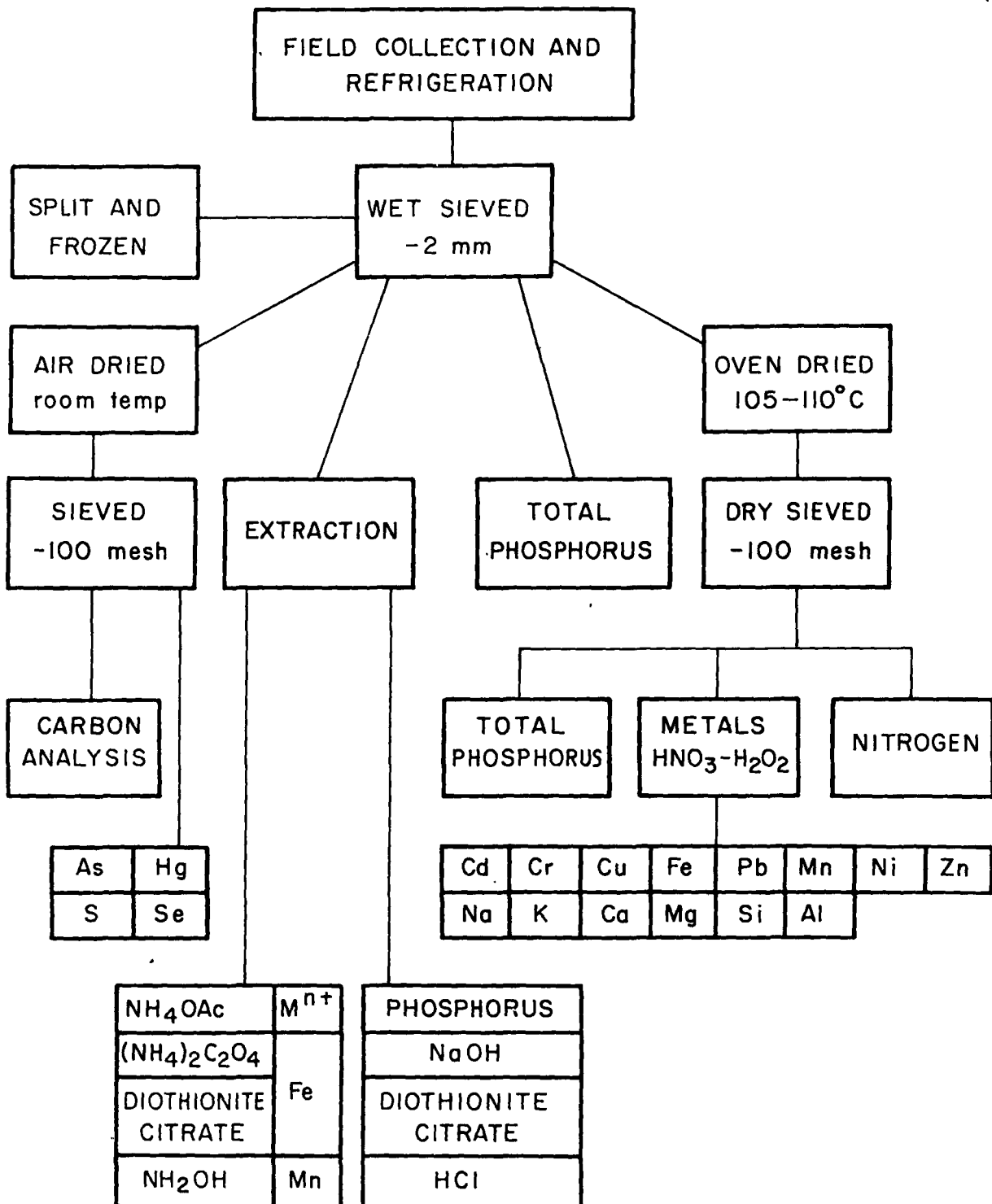
Significance threshold represents the smallest value reported with two significant figures.

For all procedures described here blanks and quality control check samples (either supplied by the National Bureau of Standards or secondary standards calibrated by this laboratory) are routinely analyzed. Periodic evaluation of procedures and computational methods is also done routinely.

Abbreviations used in this manual:

APHA	American Public Health Association
EPA	(United States) Environmental Protection Agency
NYSDH	New York State Department of Health
RSD	Relative Standard Deviation
USGS	United States Geological Survey

SEDIMENT ANALYSIS FLOW CHART



ANALYTES, total particulate
Kjeldahl (Micro)

PARAMETER
033409

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter coated with Celite.
Residue and Celite are resuspended in 10 ml
of NH_3 - free distilled water.

PRESERVATION: Resuspended residue frozen at site in dry-
ice chest

TRANSIT TIME: < 2 days

B. METHOD: A 2 ml slurry of the residue and Celite is digested
with acid. Nitrogen is determined by the Indo-
phenol blue method: NH_3 is reacted with phenol and
hypochlorite in alkaline medium to form a blue com-
plex.

INSTRUMENTATION: Bausch and Lomb 400 Spectrophotometer
with digital printout

RANGE: 30-600 μ g N/liter

QUANTITY ANALYZED: 2 ml (Celite slurry)

PRECISION: Not available

INTERFERENCES: 20

STATUS: NYS DH, APHA, EPA

REFERENCES: 2, 8, 16, 18, 20

C. DATA REPORT:

UNITS: μ g N/liter

MINIMUM REPORTABLE CONCENTRATION: 30 μ g N/liter

SIGNIFICANCE THRESHOLD: Not available

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

NITROGEN, total dissolved
including NH₃, Kjeldahl

PARAMETER
006401

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 100-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter coated with Celite

PRESERVATION: Filtered aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: A 25-ml aliquot of filtered water sample is digested with
acid. Nitrogen is determined by the Indophenol blue
method: NH₃ is reacted with phenol and hypochlorite in
alkaline medium to form a blue complex.

INSTRUMENTATION: Bausch and Lomb 400 Spectrophotometer with
digital printout

RANGE: 0.05-0.50 mg N/liter

QUANTITY ANALYZED: 25 ml

PRECISION: RSD 35% at 0.26 mg N/liter

INTERFERENCES: 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 2, 6, 8, 16, 18, 20

C. DATA REPORT:

UNITS: mg N/liter

MINIMUM REPORTABLE CONCENTRATION: 0.05 mg N/liter

SIGNIFICANCE THRESHOLD: 0.10 mg N/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

NITROGEN, total dissolved including NH ₃ , Kjeldahl	PARAMETER # 006401
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Effective date 3/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 100-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter coated with Celite

PRESERVATION: Filtered aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: A 25-ml aliquot of filtered water sample is digested with
acid. Nitrogen is determined by the Indophenol blue
method: NH₃ is reacted with phenol and hypochlorite in
alkaline medium to form a blue complex.

INSTRUMENTATION: Bausch and Lomb 400 Spectrophotometer with
digital printout

RANGE: 0.05-0.50 mg N/liter

QUANTITY ANALYZED: 25 ml

PRECISION: RSD 35% at 0.26 mg N/liter

INTERFERENCES: 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 2, 6, 8, 16, 18, 20

C. DATA REPORT:

UNITS: mg N/liter

MINIMUM REPORTABLE CONCENTRATION: 0.05 mg N/liter

SIGNIFICANCE THRESHOLD: 0.10 mg N/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

NITROGEN, ammonia as N in water

PARAMETER
000501

Effective date 4/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler
CONTAINER: Polyethylene bottle
PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter

PRESERVATION: Filtered aliquot frozen at site in dry-ice
chest

TRANSIT TIME: < 2 days

B. METHOD: Indophenol blue: NH_3 is reacted with phenol and
hypochlorite in alkaline medium to form a blue
complex. Nitroprusside is used as a catalyst
to facilitate color development at 37.5°C.

INSTRUMENTATION: Technicon AutoAnalyzer

RANGE: 0.05-0.50 mg N/liter

QUANTITY ANALYZED: 4 ml

PRECISION: RSD 18% at 0.10 mg N/liter
5.7% at 0.46 mg N/liter

INTERFERENCES: 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 4, 8, 18, 19, 20

C. DATA REPORT:

UNITS: mg N/liter

MINIMUM REPORTABLE CONCENTRATION: 0.05 mg N/liter

SIGNIFICANCE THRESHOLD: 0.10 mg N/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

NITROGEN, ammonia as N in water	PARAMETER # 000501
---------------------------------	-----------------------

Effective date 8/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler
CONTAINER: Polyethylene bottle
PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter

PRESERVATION: Filtered aliquot frozen at site in dry-ice
chest

TRANSIT TIME: < 2 days

B. METHOD:

Indophenol blue: NH_3 is reacted with phenol and hypochlorite in alkaline medium to form a blue complex. Nitroprusside is used as a catalyst to facilitate color development at 37.5°C.

INSTRUMENTATION: Technicon AutoAnalyzer

RANGE: 0.005 - 0.1 mg N/liter

QUANTITY ANALYZED: 4 ml

PRECISION: RSD 19% at 0.047 mg N/liter
10% at 0.090 mg N/liter

INTERFERENCES: 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 4, 8, 18, 19, 20

C. DATA REPORT:

UNITS: mg N/liter

MINIMUM REPORTABLE CONCENTRATION: 0.005 mg N/liter

SIGNIFICANCE THRESHOLD: 0.01 mg N/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

nitrate as N in water

000801

Receiving date 3/1/75

SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45 μ m Millipore filter

PRESERVATION: Filtered aliquot frozen at site in dry-ice
chest

TRANSIT TIME: < 2 days

METHOD: Nitrate passed through a 'Cd-Cu Reductor' is reduced
to nitrite which is reacted with sulfanilamide. The
diazocompound is coupled with 1-naphthylethylene dia-
mine to yield a highly colored azo dye. Its color
intensity is measured spectrophotometrically.

INSTRUMENTATION: Technicon AutoAnalyzer

RANGE: 0.2-2.5 mg N/liter

QUANTITY ANALYZED: 4 ml

PRECISION: RSD 3.1% at 0.65 mg N/liter
2.7% at 1.9 mg N/liter

INTERFERENCES: 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 4, 8, 20, 21

DATA REPORT:

UNITS: mg N/liter

MINIMUM REPORTABLE CONCENTRATION: 0.2 mg N/liter

SIGNIFICANCE THRESHOLD: 1.0 mg N/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

NITROGEN, nitrate as N in water

PARAMETER
000801

Effective date 8/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler
CONTAINER: Polyethylene bottle
PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45 μ m Millipore filter

PRESERVATION: Filtered aliquot frozen at site in dry-ice
chest

TRANSIT TIME: < 2 days

B. METHOD: Nitrate passed through a 'Cd-Cu Reductor' is reduced to nitrite which is reacted with sulfanilamide. The diazo compound is coupled with 1-naphthylethylene diamine to yield a highly colored azo dye. Its color intensity is measured spectrophotometrically.

INSTRUMENTATION: Technicon AutoAnalyzer

RANGE: 0.03-0.7 mg N/liter

QUANTITY ANALYZED: 4 ml

PRECISION: RSD 18% at 0.067 mg N/liter
8.3% at 0.25 mg N/liter

INTERFERENCES: 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 4, 8, 20, 21

C. DATA REPORT:

UNITS: mg N/liter

MINIMUM REPORTABLE CONCENTRATION: 0.03 mg N/liter

SIGNIFICANCE THRESHOLD: 0.1 mg N/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

PHOSPHORUS, total dissolved
as P in water

PARAMETER
107201

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler
CONTAINER: Polyethylene bottle
PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter coated with Celite
PRESERVATION: Filtered aliquot frozen at site in dry-ice chest
TRANSIT TIME: < 2 days

B. METHOD: $K_2S_2O_8$ in acid medium oxidizes organic phosphorus to orthophosphate. The orthophosphate is converted to phosphomolybdate and reduced to molybdenum blue by adding ascorbic acid. The intensity of blue color is measured spectrophotometrically.

INSTRUMENTATION: Bausch and Lomb Spectrophotometer with
digital printout

RANGE: 0.002-0.100 mg P/liter

QUANTITY ANALYZED: 50 ml

PRECISION: RSD 9.3% at 0.021 mg/l

INTERFERENCES 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 7, 8, 17, 20

C. DATA REPORT:

UNITS: mg P/liter

MINIMUM REPORTABLE CONCENTRATION: 0.002 mg P/liter

SIGNIFICANCE THRESHOLD: 0.01 mg P/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

PHOSPHORUS, total particulate
as P

PARAMETER
107001

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter coated with Celite.
Residue and Celite are resuspended in 10 ml of
phosphate-free distilled water.

PRESERVATION: Resuspended residue frozen at site in dry-ice
chest

TRANSIT TIME: < 2 days

B. METHOD: A 4-ml slurry of the residue and Celite is digested
with alkaline $K_2S_2O_8$. The orthophosphate is converted
to phosphomolybdate which is reduced to molybdenum
blue by ascorbic acid. The intensity of blue color is
measured spectrophotometrically.

INSTRUMENTATION: Bausch and Lomb Spectrophotometer with Digital
Printout

RANGE: 0.002-0.10 mg P/liter

QUANTITY ANALYZED: 4 ml

PRECISION: RSD 14.9% at 0.20 mg P/liter

INTERFERENCES: 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 7, 8, 9, 17, 20

C. DATA REPORT:

UNITS: mg P/liter

MINIMUM REPORTABLE CONCENTRATION: 0.002 mg P/liter

SIGNIFICANCE THRESHOLD: 0.01 mg P/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

PHOSPHORUS, orthophosphates as P in water	PARAMETER # 106901
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Effective date 3/1/75

A. SAMPLING:

- COLLECTION: 3 liters using a depth-integrating sampler
- CONTAINER: Polyethylene bottle
- PRETREATMENT: 300-ml aliquot filtered through a prewashed 0.45- μ m Millipore filter
- PRESERVATION: Filtered aliquot frozen at site in dry-ice chest
- TRANSIT TIME: < 2 days

B. METHOD: Orthophosphate is converted to phosphomolybdate and molybdate reduced to molybdenum blue with ascorbic acid. The intensity of blue color is measured spectrophotometrically.

INSTRUMENTATION: Bausch and Lomb Spectrophotometer with Digital Printout

RANGE: 0.002-0.100 mg P/liter

QUANTITY ANALYZED: 5 ml (Celite slurry)

PRECISION: RSD 4.8% at 0.025 mg P/liter
 14.6% at 0.013 mg P/liter

INTERFERENCES: 20

STATUS: NYS DH, APHA, EPA

REFERENCES: 8, 17, 20

C. DATA REPORT:

UNITS: mg P/liter

MINIMUM REPORTABLE CONCENTRATION: 0.002 mg P/liter

SIGNIFICANCE THRESHOLD: 0.010 mg P/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
 Division of Laboratories and Research
 New York State Department of Health

CARBON, dissolved organic

PARAMETER

030501

Effective date 3/24/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter coated with Celite

PRESERVATION: Filtered aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Organic carbon in the filtered sample is oxidized with $K_2S_2O_8$ at 175°C and 8 Kg/cm² pressure. The CO₂ produced is determined by infrared measurement.

INSTRUMENTATION: Carbon Analyzer - Oceanography International Corp.

RANGE: 1-40 mg C/liter

QUANTITY ANALYZED: .5 ml

PRECISION: RSD 6% at 7.9 mg/liter

INTERFERENCES: 10

STATUS: EPA

REFERENCES: 5, 8, 10

C. DATA REPORT:

UNITS: mg C/liter

MINIMUM REPORTABLE CONCENTRATION: 1 mg C/liter

SIGNIFICANCE THRESHOLD: 1 mg C/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CARBON, particulate organic

PARAMETER
030601

Effective date 3/24/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polypropylene bottle

PRETREATMENT: 300-ml aliquot filtered through a prewashed
0.45- μ m Millipore filter coated with Celite.
Residue and Celite are resuspended in 10 ml of
CO₂-free distilled water.

PRESERVATION: Resuspended residue frozen at site in dry-ice
chest

TRANSIT TIME: < 2 days

B. METHOD: A 1-ml slurry of the residue and Celite is oxidized
with K₂S₂O₈ at 175°C and 8 Kg/cm² pressure. This is
followed^{2 8} by infrared determination of the CO₂
produced.

INSTRUMENTATION: Carbon Analyzer - Oceanography International
Corp.

RANGE: 0.13-6.0 mg C/liter

QUANTITY ANALYZED: 1 ml (Celite slurry)

PRECISION: RSD 14% at 1.2 mg C/liter
27% at 0.9 mg C/liter

INTERFERENCES: 10

STATUS: EPA

REFERENCES: 5, 8, 10

C. DATA REPORT:

UNITS: mg C/liter

MINIMUM REPORTABLE CONCENTRATION: 0.13 mg C/liter

SIGNIFICANCE THRESHOLD: Not available

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CHLORIDE in water

PARAMETER
001001

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 300-ml aliquot filtered through
0.45- μ m Millipore filter

PRESERVATION: Filtered aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Thiocyanate (SCN^-) ion is liberated from mercuric thiocyanate through sequestration of mercury by the chloride ion to form un-ionized HgCl_2 . In presence of ferric ion, the liberated SCN^- forms a deep red complex in concentration proportional to the original Cl^- concentration.

INSTRUMENTATION: Technicon AutoAnalyzer

RANGE: 3-50 mg Cl^- /liter

QUANTITY ANALYZED: 4 ml

PRECISION: RSD \ 2.3% at 8.3 mg Cl^- /liter
2.9% at 38 mg Cl^- /liter

INTERFERENCES: 8

STATUS: EPA

REFERENCES: 1, 4, 8

C. DATA REPORT:

UNITS: . mg Cl^- /liter

MINIMUM REPORTABLE CONCENTRATION: -- 3 mg Cl^- /liter

SIGNIFICANCE THRESHOLD: 10 mg Cl^- /liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

SILICON as SiO₂

PARAMETER
013801

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: None

PRESERVATION: 100-ml aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: SiO₂ reacts with ammonium molybdate at pH 1.2 to form yellow molybdosilicic acid. This is reduced by amino-naphthylsulfonic acid to heteropoly blue which is measured spectrophotometrically.

INSTRUMENTATION: Bausch and Lomb 400 Spectrophotometer

RANGE: 0.2 - 2 mg SiO₂/liter

QUANTITY ANALYZED: 10 ml

PRECISION: RSD 73% at 0.3 mg SiO₂/liter
40% at 0.6 mg SiO₂/liter

INTERFERENCES: 20

STATUS: APHA, EPA

REFERENCES: 8, 20

C. DATA REPORT:

UNITS: mg SiO₂/liter

MINIMUM REPORTABLE CONCENTRATION: 0.2 mg SiO₂/liter

SIGNIFICANCE THRESHOLD: 1.0 mg SiO₂/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

SULFATE in water

PARAMETER
002401

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: None

PRESERVATION: 100-ml aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

- B. METHOD: The reagent is equimolar BaCl_2 and MTB (methyl thymol blue). By pH-control the Ba^{2+} -indicator chelate is prevented from forming at first. After sufficient time is allowed for the precipitation of BaSO_4 the solution is made basic and the uncombined MTB is determined spectrophotometrically.

INSTRUMENTATION: Technicon AutoAnalyzer

RANGE: 2-30 mg SO_4^{2-} /liter

QUANTITY ANALYZED: 4 ml

PRECISION: RSD 3% at 30 mg SO_4^{2-} /liter
8% at 7.6 mg SO_4^{2-} /liter

INTERFERENCES: 14

STATUS: Experimental

REFERENCES: 14

C. DATA REPORT:

UNITS: mg SO_4^{2-} /liter

MINIMUM REPORTABLE CONCENTRATION: 2 mg SO_4^{2-} /liter

SIGNIFICANCE THRESHOLD: 10 mg SO_4^{2-} /liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

ARSENIC in water

PARAMETER
009301

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Arsenic is reduced to AsH₃ by zinc and absorbed in a pyridine solution of Ag-diethyldithiocarbamate to yield a red complex. Its color intensity is measured spectrophotometrically. Predigestion is required if water sample is turbid or preserved by HNO₃.

INSTRUMENTATION: Bausch and Lomb Spectrophotometer with Digital Printout

RANGE: 0.02-0.15 mg As/liter

QUANTITY ANALYZED: 100 ml

PRECISION: RSD 16.9% at 0.16 mg As/liter

INTERFERENCES: 20

STATUS: APHA, EPA, USGS

REFERENCES: 8, 11, 20, 22

C. DATA REPORT:

UNITS: mg As/liter

MINIMUM REPORTABLE CONCENTRATION: 0.02 mg As/liter

SIGNIFICANCE THRESHOLD: 0.1 mg As/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CADMIUM in water

PARAMETER
009701

Effective date 6/11/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (228.8 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.02-0.5 mg Cd/liter

QUANTITY ANALYZED: 5 ml

PRECISION: Not available

INTERFERENCES: 23

STATUS: USGS, EPA

REFERENCES: 23

C. DATA REPORT:

UNITS: mg Cd/liter

MINIMUM REPORTABLE CONCENTRATION: 0.02 mg Cd/liter

SIGNIFICANCE THRESHOLD: 0.1 mg Cd/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CALCIUM in water

PARAMETER
011401

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO_3 added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (422.7 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.5-30 mg Ca/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 12% at 30 mg Ca/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Ca/liter

MINIMUM REPORTABLE CONCENTRATION: 0.5 mg Ca/liter

SIGNIFICANCE THRESHOLD: 1.0 mg Ca/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CHROMIUM in water

PARAMETER
009801

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (357.9 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.1-1.0 mg Cr/liter

QUANTITY ANALYZED: 5 ml

PRECISION: Not available

INTERFERENCES: 8

STATUS: USGS

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Cr/liter

MINIMUM REPORTABLE CONCENTRATION: 0.1 mg Cr/liter

SIGNIFICANCE THRESHOLD: 1.0 mg Cr/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

COBALT in water

PARAMETER
011601

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (240.7 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.1-1.0 mg Co/liter

QUANTITY ANALYZED: 5 ml

PRECISION:

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Co/liter

MINIMUM REPORTABLE CONCENTRATION: 0.1 mg Co/liter

SIGNIFICANCE THRESHOLD: 1.0 mg Co/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

COPPER in water

PARAMETER
009901

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (324.7 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.05-5.0 mg Cu/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 11.9% at 0.22 mg Cu/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Cu/liter

MINIMUM REPORTABLE CONCENTRATION: 0.05 mg Cu/liter

SIGNIFICANCE THRESHOLD: 0.1 mg Cu/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

IRON in water

PARAMETER
010001

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (248.3 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.05-1.5 mg Fe/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 18.4% at 0.14 mg Fe/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Fe/liter

MINIMUM REPORTABLE CONCENTRATION: 0.05 mg Fe/liter

SIGNIFICANCE THRESHOLD: 0.1 mg Fe/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

LEAD in water

PARAMETER
010101

Effective date 6/11/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic Absorption (217.0 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.1-2.5 mg Pb/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 73% at 0.2 mg Pb/liter

INTERFERENCES: 23

STATUS: USGS, EPA

REFERENCES: 23

C. DATA REPORT:

UNITS: mg Pb/liter

MINIMUM REPORTABLE CONCENTRATION: 0.1 mg Pb/liter

SIGNIFICANCE THRESHOLD: 1.0 mg Pb/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

MAGNESIUM in water

PARAMETER
012601

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (285.2 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.1-10 mg Mg/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 4.8% at 6.7 mg Mg/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Mg/liter

MINIMUM REPORTABLE CONCENTRATION: 0.1 mg Mg/liter

SIGNIFICANCE THRESHOLD: 1.0 mg Mg/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

MANGANESE in water

PARAMETER
010201

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic Absorption (279.5 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.02-2.5 mg Mn/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 12.5% at 0.11 mg Mn/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Mn/liter

MINIMUM REPORTABLE CONCENTRATION: 0.02 mg Mn/liter

SIGNIFICANCE THRESHOLD: 0.10 mg Mn/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

MERCURY in water

PARAMETER
010301

Effective date 9/4/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (253.7 nm)

INSTRUMENTATION: Varian AA-4 atomic absorption spectrophotometer

RANGE: 0.0004-0.0036 mg Hg/liter

QUANTITY ANALYZED: 50 ml

PRECISION: RSD 6.6% at 0.0017 mg Hg/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Hg/liter

MINIMUM REPORTABLE CONCENTRATION: 0.0004 mg Hg/liter

SIGNIFICANCE THRESHOLD: 0.001 mg Hg/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

NICKEL in water

PARAMETER
012801

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (232.0 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.05-1.5 mg Ni/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 17.5% at 0.22 mg Ni/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Ni/liter

MINIMUM REPORTABLE CONCENTRATION: 0.05 mg Ni/liter

SIGNIFICANCE THRESHOLD: 0.1 mg Ni/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

POTASSIUM in water

PARAMETER
010401

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (766.5 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.1-5.0 mg K/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 18.3% at 0.9 mg K/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg K/liter

MINIMUM REPORTABLE CONCENTRATION: 0.1 mg K/liter

SIGNIFICANCE THRESHOLD: 1.0 mg K/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage.

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

SODIUM in water

PARAMETER
010701

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (589.0 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.5-100 mg Na/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 9.5% at 52 mg Na/liter

INTERFERENCES: 8

STATUS: USGS, EPA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Na/liter

MINIMUM REPORTABLE CONCENTRATION: 0.5 mg Na/liter

SIGNIFICANCE THRESHOLD: 1.0 mg Na/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

ZINC in water

PARAMETER
010901

Effective date 3/1/75

A. SAMPLING:

COLLECTION: 1-4 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRETREATMENT: 5 ml conc HNO₃ added per liter of sample

PRESERVATION: Aliquot frozen at site in dry-ice chest

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (213.9 nm)

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 0.05-1.5 mg Zn/liter

QUANTITY ANALYZED: 5 ml

PRECISION: RSD 8.7% at 0.23 mg Zn/liter

INTERFERENCES: 8

STATUS: EPA, USGA

REFERENCES: 8, 22, 23

C. DATA REPORT:

UNITS: mg Zn/liter

MINIMUM REPORTABLE CONCENTRATION: 0.05 mg Zn/liter

SIGNIFICANCE THRESHOLD: 0.10 mg Zn/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

Sediment Analysis

Although several sediment analysis schemes are available in the literature, the following flow chart has been specially developed for fluvial sediments.

Using a regular sediment sampler or plastic shovel, bottom materials are collected and placed in a plastic pail. After wet-sieving with a 2-mm plastic sieve and discarding material $> 2\text{mm}$, the sample is split into several subsamples. Some are used wet, some air-dried, some oven-dried ($105\text{-}110^{\circ}\text{C}$), and some frozen for preservation. The wet-sieved ($< 2\text{ mm}$) sample is used directly for extractable nutrients and for trace metals and other ions. The dried samples are ground, homogenized, sieved through a 100-mesh plastic sieve, and stored for further analysis. The air-dried sample is used to analyze for carbon and such volatile elements as sulfur, selenium, arsenic, and mercury. The oven-dried sample is used for the analysis of nitrogen, phosphorus, and metals.

Metals are analyzed for by first preparing a $\text{HNO}_3\text{-H}_2\text{O}_2$ -digested extract of an oven-dried, sieved aliquot. A 50-ml stock solution is prepared from the digestate and analyzed directly or diluted to bring the solution concentration to the correct range for analysis.

The statistical information presented for each parameter was obtained in this laboratory during 1975.

The range reported refers to the actual working range used in this laboratory in routine analysis of large numbers of samples.

Minimum reportable concentration indicates the lowest result reported for an analytical determination. This value corresponds to an estimate of the result which is different from zero at the 95% confidence level. Results that are smaller than one-half the minimum

reportable concentration are reported as "less than" values.

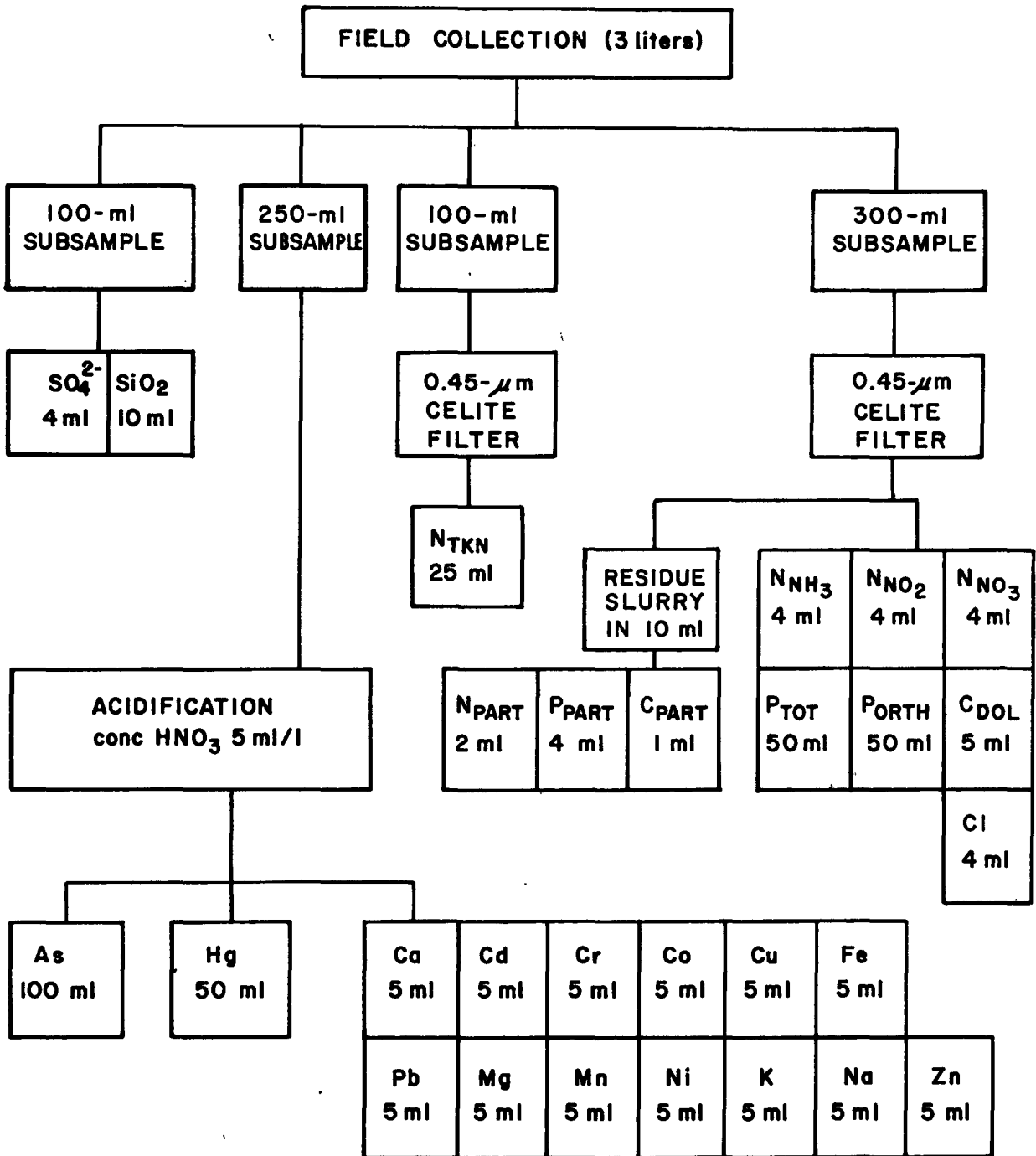
Significance threshold represents the smallest value reported with two significant figures.

For all procedures described here blanks and quality control check samples (either supplied by the National Bureau of Standards or secondary standards calibrated by this laboratory) are routinely analyzed. Periodic evaluation of procedures and computational methods is also done routinely.

Abbreviations used in this manual:

APHA	American Public Health Association
EPA	(United States) Environmental Protection Agency
NYSDH	New York State Department of Health
RSD	Relative Standard Deviation
USGS	United States Geological Survey

WATER COLUMN ANALYSIS



NITROGEN, total N in dry solids	PARAMETER # 36008.
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Effective date 11/1/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet sieved; material > 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Air-dried, homogenized and sieved (-100 mesh). Sample is directly used for analysis in the P & E 240 CHN Analyzer. Approximately 5-500 mg samples are used.

INSTRUMENTATION: Perkin-Elmer 240 Elemental Analyzer

RANGE: 0.010-20%

QUANTITY ANALYZED: 5-500 mg

PRECISION: 23% at 0.057% N

INTERFERENCES: 15

STATUS: NYSDH

REFERENCES: 15

C. DATA REPORT:

UNITS: Percent

MINIMUM REPORTABLE CONCENTRATION: 0.01%

SIGNIFICANCE THRESHOLD: 0.10%

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

PHOSPHORUS, total P in dry
solids

PARAMETER
036108

Effective date 3/1/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Alkaline $K_2S_2O_8$ digestion of the homogenized (-100 mesh)
oven-dried sample results in orthophosphate formation.
Determined spectrophotometrically by molybdenum blue
method. Approximately 0.2-g samples are used.

INSTRUMENTATION: Bausch and Lomb 400 Spectrophotometer with
digital printout

RANGE: 0.002 - 0.100 mg P/liter

QUANTITY ANALYZED: 0.2 g

PRECISION: RSD 14.9% at 0.19 mg P/liter

INTERFERENCES: 20

STATUS: APHA, EPA

REFERENCES: 7, 8, 9, 17, 20

C. DATA REPORT:

UNITS: Percent

MINIMUM REPORTABLE CONCENTRATION: 0.002 mg P/liter

SIGNIFICANCE THRESHOLD: 0.010 mg P/liter

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CARBON, total in dry solids

PARAMETER

#136208

Effective date 1/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet sieved; material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Air-dried, homogenized and sieved (-100 mesh).
Sample is directly used for analysis in the Perkin-
Elmer 240 Elemental Analyzer. Approximately 5-500 mg
samples are used.

INSTRUMENTATION: Perkin-Elmer 240 Elemental Analyzer

RANGE: 0.01 - 60%

QUANTITY ANALYZED: 5-500 mg

PRECISION: 20% at 0.5% C

INTERFERENCES: 15

STATUS: NYSDH

REFERENCES: 15

C. DATA REPORT:

UNITS: Percent

MINIMUM REPORTABLE CONCENTRATION: 0.01%

SIGNIFICANCE THRESHOLD: 0.10%

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CARBON, total organic in dry
solids

PARAMETER
036208

Effective date: 11/1/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Air-dried, homogenized, and sieved (-100 mesh) sample
is directly used for analysis in the Perkin-Elmer
240 CHN Analyzer. Sample is treated with phosphoric
acid before combustion to decompose carbonates. Ap-
proximately 5-500 mg samples are used.

INSTRUMENTATION: Perkin-Elmer 240 Elemental Analyzer

RANGE: 0.1 - 10%

QUANTITY ANALYZED: 2 mg

PRECISION: RSD 6% at 2.2% C

INTERFERENCES: 15

STATUS: NYS DH

REFERENCES: 15

C. DATA REPORT:

UNITS: Percent

MINIMUM REPORTABLE CONCENTRATION: 0.10%

SIGNIFICANCE THRESHOLD: 0.10%

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

ARSENIC, extractable in
sediment

PARAMETER
009303

Effective date 7/1/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Air-dried, homogenized and sieved (-100 mesh) sample
is digested with conc H_2SO_4 and H_2O_2 and the extract
is diluted. The silver diethyl dithiocarbamate method
is used to determine As. Approximately 1-g samples are
used and the volume of the extract is 100 ml.

INSTRUMENTATION: Bausch and Lomb 400 Spectrophotometer with
digital printout

RANGE: 2-15 μg As/g

QUANTITY ANALYZED: 1 g

PRECISION: Not available

INTERFERENCES: 11, 20

STATUS: APHA, USGS

REFERENCES: 11, 20

C. DATA REPORT:

UNITS: μg As/g dry solid

MINIMUM REPORTABLE CONCENTRATION: 2 μg As/g

SIGNIFICANCE THRESHOLD: Not available

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CALCIUM, extractable in
sediment

PARAMETER

011403

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (422.7 nm)
Approximately 0.5-g samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$
and the digestate made to 50 ml stock solution with
distilled water.

INSTRUMENTATION: Varián AA-5 atomic absorption spectrophotometer

RANGE: 50-3000 $\mu\text{g Ca/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 12% at 3000 $\mu\text{g Ca/g}$

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Ca/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 50 $\mu\text{g Ca/g}$

SIGNIFICANCE THRESHOLD: 100 $\mu\text{g Ca/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CADMIUM, extractable in
sediment

PARAMETER
009703

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (228.8 nm) Approximately 0.5-g
samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$ and the
digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 3-21 $\mu\text{g Cd/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 5% at 19.7 $\mu\text{g Cd/g}$

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Cd/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 2 $\mu\text{g Cd/g}$

SIGNIFICANCE THRESHOLD: 10 $\mu\text{g Cd/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

CHROMIUM, extractable in
sediment

PARAMETER
009803

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (357.9 nm)
Approximately 0.5-g samples are digested with
 $\text{HNO}_3\text{-H}_2\text{O}_2$ and the digestate made to 50 ml stock
solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 12-850 $\mu\text{g Cr/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 35% at 23 $\mu\text{g Cr/g}$
16% at 730 $\mu\text{g Cr/g}$

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Cr/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 10 $\mu\text{g Cr/g}$

SIGNIFICANCE THRESHOLD: 100 $\mu\text{g Cr/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

COPPER, extractable in
sediment

PARAMETER
009903

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (324.7 nm)
Approximately 0.5-g samples are digested with
HNO₃-H₂O₂ and the digestate made to 50 ml stock
solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 13-1010 µg Cu/g

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 14% at 15 µg Cu/g
6% at 920 µg Cu/g

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: µg Cu/g dry sample

MINIMUM REPORTABLE CONCENTRATION: .5 µg Cu/g

SIGNIFICANCE THRESHOLD: 10 µg C/g

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

IRON, extractable in sediment	PARAMETER # 010003
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Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material > .2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (248,3 nm)
Approximately 0.5-g samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$ and the digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 9×10^3 - 7.2×10^4 $\mu\text{g Fe/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 19% at 1.1×10^4 $\mu\text{g Fe/g}$
14% at 6.1×10^4 $\mu\text{g Fe/g}$

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Fe/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: .5 $\mu\text{g Fe/g}$

SIGNIFICANCE THRESHOLD: 10 $\mu\text{g Fe/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

LEAD, extractable in sediment

PARAMETER
010103

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (217.0 nm)
Approximately 0.5-g samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$
and the digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 10-770 $\mu\text{g Pb/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 40% at 21 $\mu\text{g Pb/g}$
2% at 750 $\mu\text{g Pb/g}$

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Pb/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 10 $\mu\text{g Pb/g}$

SIGNIFICANCE THRESHOLD: 100 $\mu\text{g Pb/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

MAGNESIUM, extractable in sediment	PARAMETER # 012603
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Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (285.2 nm) Approximately 0.5-g
samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$ and the digestate
made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 10 - 1000 $\mu\text{g Mg/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 4.8% at 660 $\mu\text{g Mg/g}$

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Mg/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 10 $\mu\text{g Mg/g}$

SIGNIFICANCE THRESHOLD: 100 $\mu\text{g Mg/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

MANGANESE, extractable in
sediment

PARAMETER
010203

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (279.5 nm) Approximately 0.5-g
samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$ and the digestate
made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 120-1800 $\mu\text{g Mn/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 14% at 150 $\mu\text{g Mn/g}$
10% at 570 $\mu\text{g Mn/g}$

INTERFERENCES: Not available

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Mn/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 2 $\mu\text{g Mn/g}$

SIGNIFICANCE THRESHOLD: 10 $\mu\text{g Mn/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

MERCURY in sediment

PARAMETER
010303

Effective date 3/1/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (253.7 nm) Air-dried, sieved
(-100 mesh) sample is digested with H_2SO_4 and $KMnO_4$
and the extract diluted. Approximately 1-g samples
are used.

INSTRUMENTATION: Varian AA-4 atomic absorption spectrophotometer

RANGE: Not available

QUANTITY ANALYZED: 1 g

PRECISION: Not available

INTERFERENCES: 23

STATUS: USGS

REFERENCES: 23

C. DATA REPORT:

UNITS: μg Hg/g dry sample

MINIMUM REPORTABLE CONCENTRATION: Not available

SIGNIFICANCE THRESHOLD: Not available

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

NICKEL, extractable in sediment

PARAMETER

012803

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (232.0 nm) Approximately 0.5-g
samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$ and the
digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 5 - 80 $\mu\text{g Ni/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 15% at 30 $\mu\text{g Ni/g}$
10% at 73 $\mu\text{g Ni/g}$

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Ni/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 5 $\mu\text{g Ni/g}$

SIGNIFICANCE THRESHOLD: 10 $\mu\text{g Ni/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

POTASSIUM, extractable in
sediment

PARAMETER
010403

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved; material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (766.5 nm) Approximately 0.5-g
samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$ and the
digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 10 - 500 $\mu\text{g K/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 18% at 90 $\mu\text{g K/g}$

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g K/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 10 $\mu\text{g K/g}$

SIGNIFICANCE THRESHOLD: 100 $\mu\text{g K/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

SODIUM, extractable in sediment

PARAMETER
010703

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (589.0 nm) Approximately 0.5-g
samples are digested with HNO₃-H₂O₂ and the digestate
made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 50 - 10,000 µg Na/g

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 9.5% at 5200 µg Na/g

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: µg Na/g dry sample

MINIMUM REPORTABLE CONCENTRATION: 50 µg Na/g

SIGNIFICANCE THRESHOLD: 100 µg Na/g

FORMAT: Computer Line Printer Output with Magnetic Tape Storage

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

ZINC, extractable in sediment

PARAMETER

010903

Effective date 6/11/75

A. SAMPLING: See sediment analysis flow chart

COLLECTION: Bottom sampler or plastic shovel

CONTAINER: Plastic pail

PRETREATMENT: Bed material is wet-sieved, and material
> 2 mm is discarded.

PRESERVATION: Several split samples frozen and stored

TRANSIT TIME: < 2 days

B. METHOD: Atomic absorption (213.9 nm) Approximately 0.5-g
samples are digested with $\text{HNO}_3\text{-H}_2\text{O}_2$ and the digestate
Made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 13-1400 $\mu\text{g Zn/g}$

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 29% at 18 $\mu\text{g Zn/g}$
28% at 1130 $\mu\text{g Zn/g}$

INTERFERENCES: 23

STATUS: experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: $\mu\text{g Zn/g}$ dry sample

MINIMUM REPORTABLE CONCENTRATION: 5 $\mu\text{g Zn/g}$

SIGNIFICANCE THRESHOLD: 10 $\mu\text{g Zn/g}$

FORMAT: Computer Line Printer Output with Magnetic Tape

REPORTED BY: Environmental Health Center
Division of Laboratories and Research
New York State Department of Health

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