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

K. V. Krishnamurty
NYS Department of Health

M. M. Reddy
NYS Department of Health

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

SUMMARY OF PROCEDURES

PREPARED BY

DR. K. V. KRISHNAMURTY
DR. M. M. REDDY

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

5. COBALT
6. COPPER
7. IRON
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9. MAGNESIUM
10. MANGANESE
11. MERCURY
12. NICKEL
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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. Fuchs.

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-μm 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:

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tr>
<td>APHA</td>
<td>American Public Health Association</td>
</tr>
<tr>
<td>EPA</td>
<td>(United States) Environmental Protection Agency</td>
</tr>
<tr>
<td>NYSDH</td>
<td>New York State Department of Health</td>
</tr>
<tr>
<td>RSD</td>
<td>Relative Standard Deviation</td>
</tr>
<tr>
<td>USGS</td>
<td>United States Geological Survey</td>
</tr>
</tbody>
</table>
SEDIMENT ANALYSIS FLOW CHART

FIELD COLLECTION AND REFRIGERATION

SPLIT AND FROZEN

AIR DRIED room temp

WET SIEVED -2 mm

OVEN DRIED 105-110°C

SIEVED -100 mesh

EXTRACTION

TOTAL PHOSPHORUS

TOTAL PHOSPHORUS

METALS HNO₃-H₂O₂

NITROGEN

CARBON ANALYSIS

As Hg
S Se

NH₄OAc
(NH₄)₂C₂O₄
DIOTHIONITE CITRATE
NH₂OH

Mⁿ⁺ Fe Mn

PHOSPHORUS

NaOH
DIOTHIONITE CITRATE
HCl

C d Cr Cu Fe Pb Mn Ni Zn
Na K Ca Mg Si Al
Parameter: total particulate Kjeldahl (Micro) # 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-μm Millipore filter coated with Celite. Residue and Celite are resuspended in 10 ml of NH₃-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 Indophenol blue method: NH₄ is reacted with phenol and hypochlorite in alkaline medium to form a blue complex.

INSTRUMENTATION: Bausch and Lomb 400 Spectrophotometer with digital printout

RANGE: 30-600 μg N/liter

QUANTITY ANALYZED: 2 ml (Celite slurry)

PRECISION: Not available

INTERFERENCES: 20

STATUS: NYSDH, APHA, EPA

REFERENCES: 2, 8, 16, 18, 20

C. DATA REPORT:

UNITS: μg N/liter

MINIMUM REPORTABLE CONCENTRATION: 30 μg 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

<table>
<thead>
<tr>
<th>Effective date</th>
<th>3/1/75</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. SAMPLING:</td>
<td></td>
</tr>
<tr>
<td>COLLECTION:</td>
<td>3 liters using a depth-integrating sampler</td>
</tr>
<tr>
<td>CONTAINER:</td>
<td>Polyethylene bottle</td>
</tr>
<tr>
<td>PRETREATMENT:</td>
<td>100-ml aliquot filtered through a prewashed 0.45-μm Millipore filter coated with Celite</td>
</tr>
<tr>
<td>PRESERVATION:</td>
<td>Filtered aliquot frozen at site in dry-ice chest</td>
</tr>
<tr>
<td>TRANSIT TIME:</td>
<td>&lt; 2 days</td>
</tr>
<tr>
<td>B. METHOD:</td>
<td>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.</td>
</tr>
<tr>
<td>INSTRUMENTATION:</td>
<td>Bausch and Lomb 400 Spectrophotometer with digital printout</td>
</tr>
<tr>
<td>RANGE:</td>
<td>0.05-0.50 mg N/liter</td>
</tr>
<tr>
<td>QUANTITY ANALYZED:</td>
<td>25 ml</td>
</tr>
<tr>
<td>PRECISION:</td>
<td>RSD 35% at 0.26 mg N/liter</td>
</tr>
<tr>
<td>INTERFERENCES:</td>
<td>20</td>
</tr>
<tr>
<td>STATUS:</td>
<td>NYSDH, APHA, EPA</td>
</tr>
<tr>
<td>REFERENCES:</td>
<td>2, 6, 8, 16, 18, 20</td>
</tr>
<tr>
<td>C. DATA REPORT:</td>
<td></td>
</tr>
<tr>
<td>UNITS:</td>
<td>mg N/liter</td>
</tr>
<tr>
<td>MINIMUM REPORTABLE CONCENTRATION:</td>
<td>0.05 mg N/liter</td>
</tr>
<tr>
<td>SIGNIFICANCE THRESHOLD:</td>
<td>0.10 mg N/liter</td>
</tr>
<tr>
<td>FORMAT:</td>
<td>Computer Line Printer Output with Magnetic Tape Storage</td>
</tr>
<tr>
<td>REPORTED BY:</td>
<td>Environmental Health Center Division of Laboratories and Research New York State Department of Health</td>
</tr>
</tbody>
</table>
NITROGEN, total dissolved including NH₃, Kjeldahl

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-μm 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

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-µm Millipore filter

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

TRANSIT TIME: < 2 days

B. METHOD: Indophenol.blue: NH₃ 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

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

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-μm Millipore filter

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

**TRANSIT TIME:** < 2 days

B. **METHOD:**

Indophenol blue: NH₃ 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

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 μm 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 diazo compound is coupled with 1-naphthyléthylene diamine 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

Effective date 8/1/75

A. SAMPLING:

COLLECTION: 3 liters using a depth-integrating sampler

CONTAINER: Polyethylene bottle

PRÉTREATMENT: 300-ml aliquot filtered through a prewashed 0.45 μm 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
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-μm Millipore filter coated with Celite
PRESERVATION: Filtered aliquot frozen at site in dry-ice chest
TRANSIT TIME: < 2 days

B. METHOD: K₂S₂O₉ 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-μm 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 K₂S₂O₈. The orthophosphate is converted to phosphomolybdic acid 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**

**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-µm 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:** NYSDH, 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

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-μm Millipore filter coated with Celite

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

TRANSIT TIME: < 2 days

METHOD: Organic carbon in the filtered sample is oxidized with K₂S₂O₈ 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

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-µm 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 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
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-μm 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 HgCl₂. 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
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-naphthsulfonic 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
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 BaCl₂ and MTB (methyl thymol blue)
   By pH-control the Ba²⁺-indicator chelate is prevented from forming at first. After sufficient time is allowed for
   the precipitation of BaSO₄ the solution is made basic and the uncombined MTB is determined spectrophotometrically.

INSTRUMENTATION: Technicon AutoAnalyzer

RANGE: 2-30 mg SO₄²⁻/liter
QUANTITY ANALYZED: 4 ml
PRECISION: RSD 3% at 30 mg SO₄²⁻/liter
           8% at 7.6 mg SO₄²⁻/liter
INTERFERENCES: 14
STATUS: Experimental
REFERENCES: 14

C. DATA REPORT:

UNITS: mg SO₄²⁻/liter
MINIMUM REPORTABLE CONCENTRATION: 2 mg SO₄²⁻/liter
SIGNIFICANCE THRESHOLD: 10 mg SO₄²⁻/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

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 (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

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

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  

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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

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

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

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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
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
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

PREPARATION: 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
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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
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

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 > 2mm, the sample is split into several subsamples. Some are used wet, some air-dried, some oven-dried (105-110°C), and some frozen for preservation. The wet-sieved (-2 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 HNO₃-H₂O₂-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
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

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$_2$S$_2$O$_8$ 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 Supplement

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

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. Approximately 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: NYSDH

   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. H₂SO₄ and H₂O₂ 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

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 HNO₃-H₂O₂ and the digestate made to 50 ml stock solution with distilled water.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 50-3000 µg Ca/g

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 12% at 3000 µg Ca/g

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: µg Ca/g dry sample

MINIMUM REPORTABLE CONCENTRATION: 50 µg Ca/g

SIGNIFICANCE THRESHOLD: 100 µg 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
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 HNO₃-H₂O₂ and the digestate made to 50 ml stock solution.

    INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer
    RANGE: (sediments) 3-21 µg Cd/g
    QUANTITY ANALYZED: 0.5 g 5 ml digestate
    PRECISION: RSD 5% at 19.7 µg Cd/g
    INTERFERENCES: 23
    STATUS: Experimental
    REFERENCES: 13, 23

C. DATA REPORT:
    UNITS: µg Cd/g dry sample
    MINIMUM REPORTABLE CONCENTRATION: 2 µg Cd/g
    SIGNIFICANCE THRESHOLD: 10 µg 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

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.

PREERVATION: 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 HNO₃-H₂O₂ and the digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 12-850 µg Cr/g

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 35% at 23 µg Cr/g
16% at 730 µg Cr/g

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: µg Cr/g dry sample

MINIMUM REPORTABLE CONCENTRATION: 10 µg Cr/g

SIGNIFICANCE THRESHOLD: 100 µg 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

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

Effective date: 6/11/75

A. SAMPLING:

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 HNO₃-H₂O₂ and the digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: (sediments) 9 x 10³ - 7.2 x 10⁴ µg Fe/g
QUANTITY ANALYZED: 0.5 g 5 ml digestate
PRECISION: RSD 19% at 1.1 x 10⁴ µg Fe/g
14% at 6.1 x 10⁴ µg Fe/g
INTERFERENCES: 23
STATUS: Experimental
REFERENCES: 13, 23

C. DATA REPORT:

UNITS: µg Fe/g dry sample
MINIMUM REPORTABLE CONCENTRATION: .5 µg Fe/g
SIGNIFICANCE THRESHOLD: 10. µg 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
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 HNO₃-H₂O₂
   and the digestate made to 50 ml stock solution.

   INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

   RANGE: (sediments) 10-770 μg Pb/g

   QUANTITY ANALYZED: 0.5 g 5 ml digestate

   PRECISION: RSD 40% at 21 μg Pb/g
              2% at 750 μg Pb/g

   INTERFERENCES: 23

   STATUS: Experimental

   REFERENCES: 13, 23

C. DATA REPORT:

   UNITS: μg Pb/g dry sample

   MINIMUM REPORTABLE CONCENTRATION: 10 μg Pb/g

   SIGNIFICANCE THRESHOLD: 100 μg 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
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 HNO₃-H₂O₂ and the digestate made to 50 ml stock solution.
   
   **INSTRUMENTATION:** Varian AA-5 atomic absorption spectrophotometer
   
   **RANGE:** 10 - 1000 µg Mg/g
   
   **QUANTITY ANALYZED:** 0.5 g 5 ml digestate
   
   **PRECISION:** RSD 4.8% at 660 µg Mg/g
   
   **INTERFERENCES:** 23
   
   **STATUS:** Experimental
   
   **REFERENCES:** 13, 23

C. **DATA REPORT:**
   
   **UNITS:** µg Mg/g dry sample
   
   **MINIMUM REPORTABLE CONCENTRATION:** 10 µg Mg/g
   
   **SIGNIFICANCE THRESHOLD:** 100 µg 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

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 HNO₃-H₂O₂ and the digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 120-1800 µg Mn/g

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 14% at 150 µg Mn/g
             10% at 570 µg Mn/g

INTERFERENCES: Not available

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: µg Mn/g dry sample

MINIMUM REPORTABLE CONCENTRATION: 2 µg Mn/g

SIGNIFICANCE THRESHOLD: 10 µg 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
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$_2$SO$_4$ 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

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 HNO₃-H₂O₂ and the digestate made to 50 ml stock solution.

   INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

   RANGE: (sediments) 5 - 80 µg Ni/g

   QUANTITY ANALYZED: 0.5 g 5 ml digestate

   PRECISION: RSD 15% at 30 µg Ni/g
               10% at 73 µg Ni/g

   INTERFERENCES: 23

   STATUS: Experimental

   REFERENCES: 13, 23

C. DATA REPORT:

   UNITS: µg Ni/g dry sample

   MINIMUM REPORTABLE CONCENTRATION: 5 µg Ni/g

   SIGNIFICANCE THRESHOLD: 10 µg 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

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 HNO₃-H₂O₂ and the digestate made to 50 ml stock solution.

INSTRUMENTATION: Varian AA-5 atomic absorption spectrophotometer

RANGE: 10 - 500 μg K/g
QUANTITY ANALYZED: 0.5 g 5 ml digestate
PRECISION: RSD 18% at 90 μg K/g
INTERFERENCES: 23
STATUS: Experimental
REFERENCES: 13, 23

C. DATA REPORT:

UNITS: μg K/g dry sample

MINIMUM REPORTABLE CONCENTRATION: 10 μg K/g

SIGNIFICANCE THRESHOLD: 100 μg 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
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
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

3. METHOD: Atomic absorption (213.9 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) 13-1400 μg Zn/g

QUANTITY ANALYZED: 0.5 g 5 ml digestate

PRECISION: RSD 29% at 18 μg Zn/g
28% at 1130 μg Zn/g

INTERFERENCES: 23

STATUS: Experimental

REFERENCES: 13, 23

C. DATA REPORT:

UNITS: μg Zn/g dry sample

MINIMUM REPORTABLE CONCENTRATION: 5 μg Zn/g

SIGNIFICANCE THRESHOLD: 10 μg Zn/g

FORMAT: Computer Line Printer Output with Magnetic Tape Tabulation

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


10. Instructions and Procedures Manual for the Total Carbon System Model 0524B, Oceanography International Corp., College Station, TX.


