# Appendix C: Quality Assurance Project Plan for the Upper Salem River Watershed Surface Water Quality Monitoring Program (June 5, 2007)

# QUALITY ASSURANCE PROJECT PLAN (QAPP)

## *RP07-024* UPPER SALEM RIVER WATERSHED RESTORATION PLAN

**Rutgers Cooperative Extension Water Resources Program**

**May 17, 2006**

**Revised & Resubmitted March 30, 2007**
**Revised & Resubmitted May 1, 2007**
***Revised & Resubmitted June 5, 2007***

# QUALITY ASSURANCE PROJECT PLAN (QAPP)

## *RP07-024* UPPER SALEM RIVER WATERSHED RESTORATION PLAN

### Rutgers Cooperative Extension Water Resources Program


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_____

Signature Date

1. Project Name: Upper Salem River
Watershed Restoration Plan

Requested By: Mike Haberland
New Jersey Department of Environmental Protection (NJDEP)

2. This project has been initiated by the NJDEP to collect data needed to prepare a comprehensive watershed restoration plan for the Upper Salem River.

3. Date Project Requested: December 2005

4. Date Project Initiated: June 2007

5. Project Officer: Christopher C. Obropta, Ph.D., P.E.
Rutgers Cooperative Extension
Water Resources Program

6. QA Officers: Katie Buckley
Rutgers Cooperative Extension
Water Resources Program

7. Project Description:

A. Objective and Scope

Based upon numerous monitoring sources including the NJDEP/USGS water quality monitoring network, the Upper Salem River is impaired for phosphorus and aquatic life, and listed on Sublist 5 of the *New Jersey 2004 Integrated Water Quality Monitoring and Assessment Report*. Additionally, a Total Maximum Daily Load (TMDL) for fecal coliform has been proposed for 17.9 miles of the Upper Salem River. This TMDL requires 84% reductions in fecal coliform from medium/high density residential, low density/rural residential, commercial, industrial, mixed urban/other urban, forest, and agricultural lands. The goal of this project is to improve the water quality of the Salem River by developing a Watershed Restoration Plan that achieves the required TMDL reductions. The study area is 14.6 square miles.

B. Data Usage

The data collected in accordance with this quality assurance project plan will help us describe both dry weather and wet weather water quality conditions. These data will provide the information needed to identify and quantify sources of pollution so that appropriate management practices can be implemented to minimize these sources.

C. Monitoring Network Design and Rationale

**Sampling Locations:** The proposed sampling locations are shown in Attachment A. Ten sampling stations have been proposed throughout the watershed; their state plane coordinates are listed in the following table.

| Site ID | Site Description | Northing | Easting |
|---|---|---|---|
| S1 | Salem River below the Salem River Reservoir | 4,383,925.72 | 478,967.60 |
| S2 | Salem River below Daretown Lake | 4,384,809.95 | 477,666.51 |
| S3 | Salem River and Tributary1 confluence at Commissioner's Pike | 4,385,590.11 | 476,979.31 |
| S4 | Salem River Tributary 1 at Route 40 | 4,386,261.59 | 478,347.62 |
| S5 | Salem River Tributary 2 at Davis Road | 4,384,726.75 | 475,213.75 |
| S6 | Salem River Tributary 2 at County 615 | 4,386,160.75 | 474,997.11 |
| S7 | Salem River Tributary 3 at Route 40 | 4,387,527.47 | 476,323.61 |
| S8 | Salem River below Avis Mill Pond | 4,387,349.52 | 474,804.46 |
| S9 | Salem River below East Lake | 4,388,138.02 | 472,911.20 |
| S10 | Salem River at Woodstown Station, 01482500 | 4,388,045.10 | 471,635.57 |

A WAAS-enabled Garmin Rino 120 GPS (global positioning system) unit will be used to locate and identify the sampling locations. Sampling locations will be marked with stakes and surveying tape.

**Temporal and Spatial Aspects:**

*Biweekly Surface Water Sampling*

Surface water quality samples will be collected from all sampling locations twice a month, independent of weather, in June 2007, July 2007, August 2007, September 2007, October 2007, and November 2007 (12 events). Three additional surface water quality samples will be collected from all sampling locations in June 2007, July 2007 and August 2007 for fecal coliform and *E. coli* analyses (Nine additional sampling events). These nine additional sampling events will be independent of precipitation and will allow for a total of five fecal coliform and five *E. coli* analyses at all sampling locations within a 30 day period during the warmer summer months.

All scheduling is subject to the natural occurrence of appropriate stream flow conditions (i.e., non-flooding conditions). In accordance with the Field Sampling Procedures Manual (See Section 6.8.1.1, Chapter 6D – page 59 of 188), field personnel will not wade into flowing water when the product of depth (in feet) and velocity (in feet per second) equals ten or greater to ensure the health and safety of all field personnel. If the stream flow conditions preclude entry into the stream, samples will be collected from the closest bridge crossing to that location or from the stream bank. Flow will have to be estimated or calculated based on the recorded flow at the closest USGS gaging station and the drainage area.

Bacteriology samples will be collected directly into a bacteriological sample container in accordance with the methods outlined in section 6.8.2.2.7 of the Field Sampling Procedures

Manual (See Chapter 6D - page 67 of 188). Composite samples will not be collected for bacteriology samples.

For the most part, the Salem River and its tributaries are uniformly mixed which warrants grab sampling (See Section 6.8.2.2.3, Chapter 6D-Page 66 of 188 of the Field Sampling Procedures Manual). A single grab sample will be collected at all locations where the stream width is six feet or less. At stream locations with a width greater than six feet, a minimum of three subsurface grab samples (i.e., quarter points) will be collected at equidistant points across the stream. The number of individual samples in a composite varies with the width of the stream being sampled. Horizontal intervals will be at least one foot wide (See Section 6.8.2.2.2, Chapter 6D – Page 64 of 188 of the Field Sampling Procedures Manual). These grab samples then will be composited in a larger volume container from which the desired volume will be transferred to the sample bottles. A dedicated large volume container will be assigned to each sample location.

Field equipment used for surface water quality sample collection (i.e., bottles and buckets) will be decontaminated/cleaned in the laboratory prior to each sampling event. A dedicated large volume container will be assigned to each sample location. Prior to each sampling event, the large volume containers will be decontaminated in the laboratory using the following procedures in accordance with the Field Sampling Procedures Manual (See Chapter 2A – Page 10 of 61): 1) laboratory grade glassware detergent plus tap water wash, 2) generous tap water rinse, 3) distilled/deionized water rinse, 4) 10% nitric acid rinse, 5) distilled/deionized water rinse. Note that the samples collected will not be analyzed for metals or organics. Also, field equipment decontamination water will be disposed of in accordance with the laboratory's Standard Operating Procedures and Quality Assurance Manual.

*Wet Weather Surface Water Sampling*

Three wet weather sampling events, at a minimum, will be conducted between June 2007 and November 2007 at each station. The wet weather samples for this plan will be in addition to the 12 biweekly surface water samples. Collection of stormwater samples will begin at the onset of the storm (i.e., a storm predicted to produce a minimum of ½ inch of precipitation), and an attempt will be made to span the course of the event. By using this method of sampling, the samples should accurately reflect loading for the entire event. A priority will be to acquire first flush samples. Again, flow will be measured along with concentrations to quantify loading for selected parameters. A total of three samples will be obtained between the onset of the storm and the time when the flow reaches the pre-storm level, unless impractical, at each station during each storm event. At each station, the samples obtained for the entire event will be flow-weight composited to provide one sample from each station, with the exception of fecal coliform and *E. coli*, which will require analysis of each individual grab sample. Samples will be collected via manual grab sampling and not with a composite sampler.

Rainfall data will be collected from a weather monitoring station (Campbell Scientific) installed under a previous 319(h) project in the Upper Cohansey Watershed. The monitoring station is located on Cake Road in Upper Deerfield Township. Using a log in and protected password, the Rutgers Cooperative Extension Water Resources Program will be able to access weather data online and in several formats. Data is recorded every ten minutes and can be

downloaded as hourly and daily. Temperature, relative humidity, rainfall, windspeed, solar radiation, and evapotranspiration can be recorded from this weather monitoring station.

If three ½ inch storm events are not captured between June - November 2007, the Water Resources Program, after consultation with the Department, may have to defer the Wet Weather Surface Water Sampling portion of the Upper Salem study to June – November 2008. Attempts will be made to conduct this portion of the study as early on in the study period as possible.

*Biological Sampling*

Samples of the benthic macroinvertebrate community will be collected in accordance with the Rapid Bioassessment Protocol (RBP) procedure used by the NJDEP Bureau of Freshwater and Biological Monitoring, which is based on USEPA's *Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers* (EPA 841-B-99-002 Nov. 1999). A multihabitat sampling approach, concentrating on the most productive habitats of the stream plus coarse particulate organic matter (CPOM) or leaf litter, will be used. Benthic macroinvertebrates will be collected from four locations, S1, S3, S6, and S10 (See Attachment A), once in the late summer as described in Attachment B. AMNET samples in this watershed have been collected in the late summer, therefore, for data comparability purposes, samples will be collected in the late summer as part of this study.

**Basis for Sampling Locations:**

Surface water quality sampling will be conducted to assess the loading inputs of nutrients, total suspended solids (TSS) and bacteria to Upper Salem River, as well as the movement of nutrients, TSS and bacteria from basin to basin to identify and quantify the sources of pollution under dry weather and wet weather conditions. Biological sampling will be conducted so that the benthic macroinvertebrate community can be better characterized, compared, and evaluated for biological integrity.

D. Monitoring Parameters

Surface water quality sample collection, as well as *in situ* measurements of stream width, stream depth, and stream velocity, will be conducted by the Rutgers Cooperative Extension Water Resources Program. Stream width, stream depth, and stream velocity will be measured in accordance with the methods outlined in Attachment C. *In situ* measurements of pH, temperature, and dissolved oxygen will be measured by the Rutgers EcoComplex Laboratory, NJDEP Certified Laboratory #03019. Surface water quality samples will be analyzed for fecal coliform, ammonia-nitrogen, nitrate-nitrogen, nitrite-nitrogen, total Kjeldahl nitrogen, total phosphorus, dissolved orthophosphate phosphorus, and TSS by NJDEP Certified Laboratory #PA166, QC Laboratories. *E. coli* analyses will be conducted by NJDEP Certified Laboratory #PA001, QC Laboratories, Wind Gap Division. The Vineland Division of QC Inc. (NJDEP Certified Laboratory #06005) is currently seeking certification for *E. coli* analyses. Certification is anticipated by June 2007. Once certification is obtained at the Vineland Division, *E. coli* analyses for the project will be conducted there.

Biological sampling will include benthic macroinvertebrate grab/jab type sampling, along with the collection of CPOM. Physicochemical measurements will include *in situ* pH,

temperature, dissolved oxygen, stream width, stream depth, and stream velocity. Benthic macroinvertebrate sampling and identification will be conducted by Rutgers Cooperative Extension Water Resources Program in accordance with the Rapid Bioassessment Protocol (RBP) procedure used by the NJDEP Bureau of Freshwater and Biological Monitoring, which is based on USEPA's *Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers* (EPA 841-B-99-002 Nov. 1999). The Water Resources Program will make stream width, stream depth, and stream velocity determinations in accordance with the procedures specified in Attachment C. *In situ* measurements of pH, temperature, and dissolved oxygen will be measured by the Rutgers EcoComplex Laboratory, NJDEP Certified Laboratory #03019.

E. Parameter Table

Measurements of the sampled parameters will be performed in accordance with Table 1A – List of Approved Biological Methods and Table 1B – List of Approved Inorganic Test Procedures (40 CFR Part 136.3) of Attachment D. Sample containers, preservation techniques, and holding times will be in accordance with Table II (40 CFR Part 136.3) of Attachment E. QC Laboratories will provide appropriate containers for all analyses. Any deviations from the test procedures and/or preservation methods and holding times will be reported to the NJDEP Office of Quality Assurance and will be noted in the final report from the laboratory.

8. Schedule*

| Task | Date |
|---|---|
| Submit quality assurance work plan | May 2006 |
| Conduct biweekly water quality sampling | June 2007 – November 2007 |
| Conduct wet weather water quality sampling | June 2007 - November 2007 |
| Conduct biological sampling | Late Summer 2007 (i.e., August 2007) |
| Submit data and summary report to NJDEP | February 2008 |

* All scheduling is subject to the natural occurrence of appropriate stream flow conditions (i.e., non-flooding conditions).

9. Project Organization and Responsibility:

| | | |
|---|---|---|
| Laboratory Operations: | (QA Director) | Phil Worby<br>QC Laboratories<br>Lisa Galloway Evrard<br>Rutgers EcoComplex<br>Laboratory |
| | (Lab Director) | Thomas J. Hines<br>QC Laboratories<br>Lisa Galloway Evrard<br>Rutgers EcoComplex<br>Laboratory |
| | (NJDEP Representative) | Marc Ferko |
| Sampling Operations: | (QA Officer) | Katie Buckley<br>Rutgers Cooperative Extension<br>Water Resources Program |
| | (NJDEP Representative) | Marc Ferko |
| Data Processing/<br>Data Quality Review: | (QA Officer/<br>Project Officer) | Katie Buckley/<br>Christopher C. Obropta<br>Rutgers Cooperative Extension<br>Water Resources Program |
| | (NJDEP Representative) | Beth Torpey |
| Overall QA: | (QA Officer) | Katie Buckley |
| Overall Coordination: | (Project Officer) | Christopher C. Obropta |

10. Organizational Chart:

| |
|---|
| Overall Coordination:<br>Christopher C. Obropta<br>(Rutgers Cooperative Extension<br>Water Resources Program)<br><br>Overall QA:<br>Katie Buckley<br>(Rutgers Cooperative Extension<br>Water Resources Program) |
| Data Quality Review/Data Processing:<br>Christopher C. Obropta/<br>Katie Buckley<br>Beth Torpey (NJDEP) |
| Sampling QC/Sampling Operations:<br>Katie Buckley<br>Marc Ferko (NJDEP) |
| Laboratory QC/Laboratory Director:<br>Phil Worby/Thomas J. Hines<br>QC Laboratories<br>Lisa Galloway Evrard<br>Rutgers EcoComplex Laboratory<br>Marc Ferko |

11. Sampling Procedures:

All sampling procedures will be in conformance with the NJDEP 2005 Field Sampling Procedures Manual or with prior written approval.

- Bacteriology samples will be collected in accordance with the methods outlined in section 6.8.2.2.7 of the Field Sampling Procedures Manual (See Chapter 6D - page 67 of 188).
- Manual composite sampling for wider portions of the streams will be conducted in accordance with the methods outlined in section 6.8.2.2.2 of the Field Sampling Procedures Manual (See Chapter 6D – page 64 of 188).

- Grab sampling where the natural stream conditions make compositing unnecessary will be conducted in accordance with the methods outlined in section 6.8.2.2.3 of the Field Sampling Procedures Manual (See Chapter 6D – page 66 of 188).

  In addition, instrumentation used for the collection of field data will be properly calibrated, in conformance with the manufacturer's instructions, laboratory SOPs and QA Manuals, and the NJDEP Field Sampling Procedures Manual.

12. Chain of Custody Procedures:

    Chain of Custody procedures will be followed for all samples collected for this monitoring program. A sample chain of custody form is provided in Attachment F. A sample is in someone's "custody" if 1) it is in one's actual physical possession, 2) it is in one's view, after being in one's physical possession, 3) it is in one's physical possession and then locked up so that no one can tamper with it, and 4) it is kept in a secured area, restricted to authorized personnel only.

13. Calibration Procedures and Preventative Maintenance:

    Calibration and preventative maintenance of laboratory and field equipment will be in accordance with the manufacturer's instructions, NJDEP Field Sampling Procedures Manual, NJAC 7:18 and 40 CFR Part 136.

14. Documentation, Data Reduction, and Reporting:

    The QA Officer, for a minimum of five years, will keep all data on file, and all applicable data will be included in the summary report to NJDEP.

15. Quality Assurance and Quality Control:

    NJAC 7:18 and 40 CFR Part 136 will be followed for all quality assurance and quality control (QA/QC) practices, including detection limits, quantitation limits, precision, and accuracy. Tables of parameter detection limits, quantitation limits, accuracy, and precision applicable to this study are provided in Attachment G. QC Laboratories, Rutgers EcoComplex Laboratory, and Rutgers Cooperative Extension Water Resources Program will perform data validation.

    With regard to the benthic macroinvertebrate samples, a ***single*** reference collection from the project will be sent to:

    Marion McClary, Jr., Ph.D.
    Associate Professor of Biological Sciences
    Associate Director of Biological Sciences
    School of Natural Sciences
    Fairleigh Dickinson University

***once*** to confirm the identifications done by the Rutgers Cooperative Extension Water Resources Program.

16. Performance and Systems Audits:

All NJDEP certified laboratories participate ***annually in a NJDEP mandated Performance Testing program.*** The NJDEP Office of Quality Assurance conducts a performance audit of each laboratory that is certified. The NJDEP Office of Quality Assurance also periodically conducts on-site technical systems audits of each certified laboratory. The findings of these audits, together with the ***NJDEP mandated Performance Testing program***, are used to update each laboratory's certification status.

The NJDEP Office of Quality Assurance periodically conducts field audits of project sampling operations. The Office of Quality Assurance will be contacted during the project to schedule a possible field audit.

17. Corrective Action:

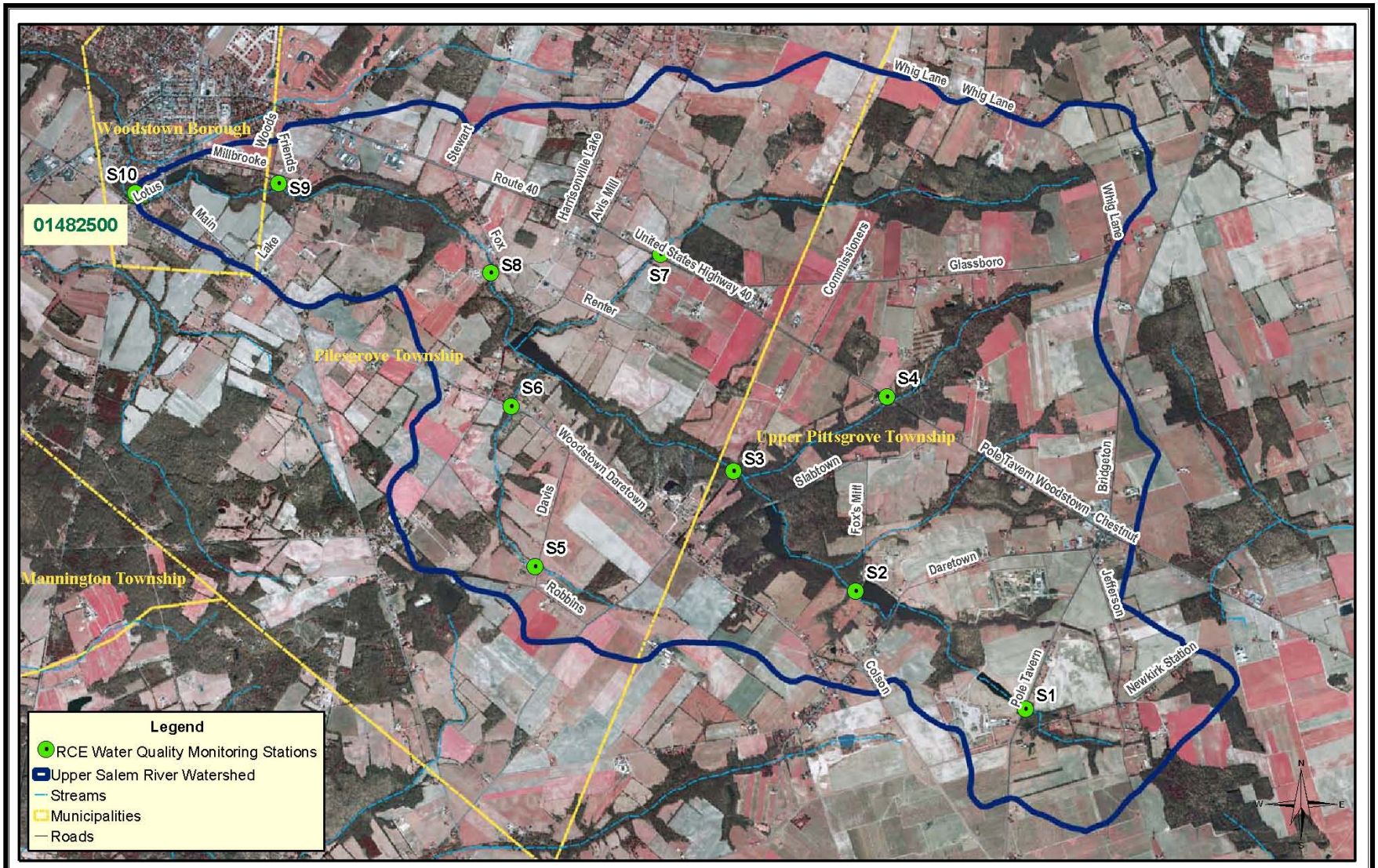
All NJDEP certified laboratories must have a written corrective action procedure which they adhere to in the event that calibration standards, performance evaluation results, blanks, duplicates, spikes, etc. are out of the acceptable range or control limits. If the acceptable results cannot be obtained for the above-mentioned QA/QC samples during any given day, sample analysis must be repeated for that day with the acceptable QA/QC results. NJDEP will be notified if there are any deviations from the approved work plan.

18. Reports:

The summary report will include at a minimum an Introduction, Purpose and Scope, Results and Discussion, Conclusions and Recommendations, and an Appendix with Data Tables.

# ATTACHMENT A

## Sampling Locations
## Upper Salem River Watershed





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Date Produced: May 2006, Revised Jan 2007

# SAMPLING LOCATIONS
# UPPER SALEM RIVER WATERSHED

Data Source: NJDEP 1996 GIS Data; 2002 Aerial Orthophotography; RCE Water Resources Program Proposed Sampling Locations

1" = 4,000 feet

0 2,200 4,400 Feet

# ATTACHMENT B

## Biological Sampling Procedures and Analysis

# Biological Sampling Procedures and Analysis

**These sampling and data analysis procedures are in accordance with the Rapid Bioassessment Protocol procedures used by the NJDEP Bureau of Freshwater and Biological Monitoring, which is based on USEPA's Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers (EPA 841-B-99-002 Nov. 1999).**

*Sampling Procedures:*

Samples will be collected using a multi-habitat sampling approach, concentrating on the most productive habitat of the stream (i.e., the riffle/run areas), plus coarse particulate organic matter (CPOM) or leaf litter. This sampling method minimizes habitat or substrate variation between sampling sites and includes all likely functional feeding groups of macroinvertebrates in the stream. Given the nature of the substrate at the sampling sites, either a Surber Square Foot Bottom Sampler will be used to collect three grab type samples at each site, or samples will be collected by jabbing a standard aquatic D-frame dip net in habitats thought to be productive and stable a total of 20 times at each sampling location. These samples will be sorted in the field, composited (i.e., the contents from the three grab samples or 20 jabs from each site will be combined into a single container), and preserved in 80% ethanol for later subsampling, identification and enumeration.

A composite collection of a variety of CPOM forms (e.g., leaves, needles, twigs, bark, or fragments of these) will be collected. It is difficult to quantify the amount of CPOM to be collected in terms of weight or volume, given the variability of its composition. Collection of several handfuls of material is usually adequate, and the material is typically found in depositional areas, such as in pools and along snags and undercut banks. The CPOM sample will be processed using a U.S. Standard No. 30 sieve, and added to the composite of the grab samples for each site.

A 100-organism subsample of the benthic macroinvertebrate composite sample from each sampling site will be taken in the laboratory according to the methods outlined in the Rapid Bioassessment Protocol used by the NJDEP Bureau of Freshwater and Biological Monitoring. With the exception of chironomids and oligochaetes, benthic macroinvertebrates will be identified to genus. Chironomids will be identified to subfamily as a minimum, and oligochaetes will be identified to family as a minimum.

A habitat assessment will be conducted concurrent with the benthic macroinvertebrate sampling in accordance with the methods used by the NJDEP Bureau of Freshwater and Biological Monitoring. The measurement of physicochemical parameters will also be conducted concurrent with the benthic macroinvertebrate sampling. Surface water sampling for the measurement of pH, temperature, and dissolved oxygen will be conducted on a representative cross section of the steam. At least four subsurface grab samples will be collected across an established transect. These grab samples will be composited, and an appropriate volume will be transferred to sample bottles for *in situ* measurement of pH, temperature, and dissolved oxygen. Stream width, stream depth, and stream velocity will be measured in accordance with the methods outlined in Attachment C.

## Biological Sampling Procedures and Analysis (continued)

*Data Analysis:*

The NJDEP Bureau of Freshwater and Biological Monitoring uses several community measures of biometrics adapted from the Rapid Bioassessment Protocols to evaluate the biological condition of sampling sites within the Ambient Biomonitoring Network in New Jersey. These community measures include taxa richness, EPT index, %EPT, %CDF, and Modified Family Biotic Index. This analysis integrates several community parameters into one easily comprehended evaluation of biological integrity referred to as the New Jersey Impairment Score (NJIS). The NJIS has been established for three categories of water quality bioassessment for New Jersey streams: non-impaired, moderately impaired, and severely impaired, and is based on comparisons with reference streams and a historical database consisting of 200 benthic macroinvertebrate samples collected from New Jersey streams.

If the above metrics are not utilized, or if different metrics or indices are used, these changes will be discussed with NJDEP for approval. For example, to determine the similarity among the sampling sites with respect to species composition, the Percentage Similarity Index may be calculated for all pair wise comparisons of the sampling sites. Also, the benthic macroinvertebrates may be separated into the four broad functional feeding groups to evaluate community structure. In addition, the Shannon diversity index may be calculated to evaluate community structure. In addition, the findings from the habitat assessment will be used to interpret survey results and identify obvious constraints on the attainable biological potential of the site.

The final report will include a characterization of the aquatic biota, in particular the benthic macroinvertebrate community.

# ATTACHMENT C

## Stream Flow Measurement Procedure

## Stream Flow Measurement Procedure

Stream width, depth, velocity, and flow determinations will be made in conformance with the following procedures:

1. A measuring tape is extended across the stream, from bank to bank, perpendicular to flow. Meter calibration is checked.

2. Using a Marsh-McBirney, Inc. Model 2000 Flo-Mate Portable Water Flow meter, velocity and depth measurements are made at points along the tape. Normally depth is measured using a rod calibrated in tenths of a foot. In shallow streams, a yardstick may be used to measure depth. Velocities are measured at approximately 0.6 depth (from the surface) where depths are less than 2.5 feet and at 0.2 and 0.8 depth (from the surface) in areas where the depth exceeds 2.5 feet.

3. The stream cross section is divided into segments with depth and velocity measurements made at equal intervals along the cross section. The number of measurements will vary with site conditions and uniformity of stream cross section. Each cross section is divided into equal parts depending upon the total width and uniformity of the section. At a minimum, velocities are taken at quarter points for very narrow sections. In general, velocity and depth measurements are taken every one to five feet. A minimum of ten velocity locations is used whenever possible. The velocity is determined by direct readout from the Marsh-McBirney meter set for 5 second velocity averaging.

4. Using the field data collected, total flow, average velocity, and average depth can be computed. Individual partial cross-sectional areas are computed for each depth and velocity measurement. The mean velocity of flow in each partial area is computed and multiplied by the partial cross-sectional area to produce an incremental flow. Incremental flows are summed to calculate the total flow. The average velocity for the stream can be computed by dividing the total flow by the sum of the partial cross-sectional areas. The average depth for the stream can be computed by dividing the sum of the partial cross-sectional areas by the total width of the stream. The accuracy of this method depends upon a number of factors, which include the uniformity of the steam bottom, total width, and the uniformity of the velocity profile.

- Flow measurements will be collected for all sampling events. However, in accordance with the Field Sampling Procedures Manual (See Section 6.8.1.1, Chapter 6D – page 59 of 188), field personnel will not wade into flowing water when the product of depth (in feet) and velocity (in feet per second) equals ten or greater. All scheduling is subject to the natural occurrence of appropriate stream flow conditions (i.e., non-flooding conditions) to ensure the health and safety of all field personnel. If the stream flow conditions preclude entry into the stream, flow will have to be estimated or calculated based on the recorded flow at the closest USGS gaging station and the drainage area.

**ATTACHMENT D**

**Table 1A – List of Approved Biological Methods**
**&**
**Table 1B – List of Approved Inorganic Test Procedures**
**40 CFR Part 136.3**
**July 1, 2005**

## TABLE IA—LIST OF APPROVED BIOLOGICAL METHODS

| Parameter and units | Method[1] | EPA | Standard methods 18th, 19th, 20th Ed. | ASTM | AOAC | USGS | Other |
|---|---|---|---|---|---|---|---|
| Bacteria: | | | | | | | |
| 1. Coliform (fecal), number per 100 mL. | Most Probable Number (MPN), 5 tube 3 dilution, or | p. 132[3] | 9221C E[4] | | | | |
| | Membrane filter (MF)[2], single step. | p. 124[3] | 9222D[4] | | | B–0050–85[5] | |
| 2. Coliform (fecal) in presence of chlorine, number per 100 mL. | MPN, 5 tube, 3 dilution, or | p. 132[3] | 9221C E[4] | | | | |
| | MF, single step[6] | p. 124[3] | 9222D[4] | | | | |
| 3. Coliform (total), number per 100 mL. | MPN, 5 tube, 3 dilution, or | p. 114[3] | 9221B[4] | | | | |
| | MF[2], single step or two step | p. 108[3] | 9222B[4] | | | B–0025–85[5] | |
| 4. Coliform (total), in presence of chlorine, number per 100 mL. | MPN, 5 tube, 3 dilution, or | p. 114[3] | 9221B[4] | | | | |
| | MF[2] with enrichment | p. 111[3] | 9222(B+B.5c)[4] | | | | |
| 5. *E. coli*, number per 100 mL[28]. | MPN[7,9,15], multiple tube, | | 9221B.1/9221F[4,12,14] | | | | |
| | multiple tube/multiple well, | | 9223B[4,13] | | 991.15[11] | | Colilert®[13,17] Colilert-18®[13,16,17] |
| | MF[2,6,7,8,9] two step, or | 1103.1[20] | 9222B/9222G[4,19] 9213D[4] | D5392–93[10] | | | |
| | single step | 1603[21] 1604[22] | | | | | mColiBue 24[18] |
| 6. Fecal streptococci, number per 100 mL. | MPN, 5 tube, 3 dilution, | p. 139[3] | 9230B[4], 9230C[4] | | | | |
| | MF[2], or | p. 136[3] | | | B–0055–85[5] | | |
| | Plate count | p. 143[4] | | | | | |
| 7. Enterococci, number per 100 mL. | MPN[7,9] multiple tube | | 9230B[4] | | | | |
| | multiple tube/multiple well | | | D6503–99[10] | | | Enterolert®[13,23] |
| | MF[2,6,7,8,9] two step | 1106.1[24] | 9230C[4] | D5259–92[10] | | | |
| | single step, or | 1600[25] | | | | | |
| | Plate count | p. 143[3] | | | | | |
| Protozoa: | | | | | | | |
| 8. *Cryptosporidium*[26] | Filtration/IMS/FA | 1622[26] 1623[27] | | | | | |
| 9. *Giardia*[28] | Filtration/IMS/FA | 1623[27] | | | | | |
| Aquatic Toxicity: | | | | | | | |
| 10. Toxicity, acute, fresh water organisms, LC50, percent effluent. | *Ceriodaphnia dubia* acute | 2002.0[29] | | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | Sea urchin, *Arbacia punctulata*, fertilization. | 1008.0 [31] | | | | | |

Notes to Table IA:

[1] The method must be specified when results are reported.

[2] A 0.45 μm membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

[3] USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/8–78/017.

[4] APHA. 1998, 1995, 1992. Standard Methods for the Examination of Water and Wastewater. American Public Health Association. 20th, 19th, and 18th Editions. Amer. Publ. Hlth. Assoc., Washington, D.C.

[5] USGS. 1989. U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of Interior, Reston, Virginia.

[6] Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

[7] Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

[8] When the MF method has not been used previously to test ambient waters with high turbidity, large number of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

[9] To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

[10] ASTM. 2000, 1999, 1996. Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. American Society for Testing and Materials. 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[11] AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. Association of Official Analytical Chemists International. 481 North Frederick Avenue, Suite 500, Gaithersburg, Maryland 20877–2417.

[12] The multiple-tube fermentation test is used in 9221B.1. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.

[13] These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β–glucuronidase produced by *E. coli*.

[14] After prior enrichment in a presumptive medium for total coliform using 9221B.1, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 μg/mL of MUG may be used.

[15] Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple-well procedures, Quanti-Tray® or Quanti-Tray® 2000, and the MPN calculated from the table provided by the manufacturer.

[16] Colilert-18® is an optimized formulation of the Colilert® for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 °C rather than the 24 h required for the Colilert® test and is recommended for marine water samples.

[17] Descriptions of the Colilert®, Colilert-18®, Quanti-Tray®, and Quanti-Tray®/2000 may be obtained from IDEXX Laboratories, Inc., One IDEXX Drive, Westbrook, Maine 04092.

[18] A description of the mColiBlue24" test, Total Coliforms and *E. coli*, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.

[19] Subject total coliform positive samples determined by 9222B or other membrane filter procedure to 9222G using NA-MUG media.

[20] USEPA. 2002. Method 1103.1: *Escherichia coli* (*E. coli*) In Water By Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington D.C. EPA–821–R–02–020.

[21] USEPA. 2002. Method 1603: *Escherichia coli* (*E. coli*) In Water By Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar ( modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington D.C. EPA–821–R–02–023.

[22] Preparation and use of MI agar with a standard membrane filter procedure is set forth in the article, Brenner *et al.* 1993. "New Medium for the Simultaneous Detection of Total Coliform and *Escherichia coli* in Water." Appl. Environ. Microbiol. 59:3534–3544 and in USEPA. 2002. Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA 821–R–02–024.

[23] A description of the Enterolert® test may be obtained from IDEXX Laboratories, Inc., One IDEXX Drive, Westbrook, Maine 04092.

[24] USEPA. 2002. Method 1106.1: Enterococci In Water By Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE-EIA). U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA–821–R–02–021.

[25] USEPA. 2002. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC. EPA–821–R–02–022.

[26] Method 1622 uses filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of *Cryptosporidium*. USEPA. 2001. Method 1622: Cryptosporidium in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA–821–R–01–026.

[27] Method 1623 uses filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts. USEPA. 2001. Method 1623. Cryptosporidium and Giardia in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA–821–R–01–025.

[28] Recommended for enumeration of target organism in ambient water only.

[29] USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA/821/R–02/012.
[30] USEPA. October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Fourth Edition. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA/821/R–02/013.
[31] USEPA. October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Third Edition. U.S. Environmental Protection Agency, Office of Water, Washington DC. EPA/821/R–02/014.

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES

| Parameter, units and method | Reference (method number or page) | | | | |
|---|---|---|---|---|---|
| | EPA [1,35] | *Standard Methods* [Edition(s)] | ASTM | USGS [2] | Other |
| 1. Acidity, as $CaCO_3$, mg/L: | | | | | |
| Electrometric endpoint or phenolphthalein endpoint. | 305.1 | 2310 B(4a) [18th, 19th, 20th]. | D1067–92 | I–1020–85<br>I–2030–85 | |
| 2. Alkalinity, as $CaCO_3$, mg/L: | | | | | |
| Electrometric of Colorimetric titration to pH 4.5, manual or automatic. | 310.1<br>310.2. | 2320 B [18th, 19th, 20th] | D1067–92 | I–1030–85<br>I–2030–85 | 973.43 [3] |
| 3. Aluminium—Total,[4] mg/L; Digestion [4] followed by: | | | | | |
| AA direct aspiration [36] | 202.1 | 3111 D [18th, 19th] | | I–3051–85 | |
| AA furnace | 202.2 | 3113 B [18th, 19th]. | | | |
| Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES) [36]. | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [60] | |
| Direct Current Plasma (DCP) [36]. | | | D4190–94 | | Note 34. |
| Colorimetric (Eriochrome cyanine R). | | 3500–Al B [20th] and 3500–Al D [18th, 19th]. | | | |
| 4. Ammonia (as N), mg/L: | | | | | |
| Manual, distillation (at pH 9.5) [6] followed by. | 350.2 | 4500–$NH_3$ B [18th, 19th, 20th]. | | | 973.49 [3] |
| Nesslerization | 350.2 | 4500–$NH_3$ C [18th] | D1426–98(A) | I–3520–85 | 973.49 [3] |
| Titration | 350.2 | 4500–$NH_3$ C [19th, 20th] and 4500–$NH_3$ E [18th]. | | | |
| Electrode | 350.3 | 4500–$NH_3$ D or E [19th, 20th] and 4500–$NH_3$ F or G [18th]. | D1426–98(B). | | |
| Automated phenate, or | 350.1 | 4500–$NH_3$ G [19th, 20th] and 4500–$NH_3$ H [18th]. | | I–4523–85 | |
| Automated electrode | | | | | Note 7. |
| 5. Antimony-Total,[4] mg/L; Digestion [4] followed by: | | | | | |
| AA direct aspiration [36] | 204.1 | 3111 B [18th, 19th] | | | |
| AA furnace | 204.2 | 3113 B [18th, 19th] | | | |
| ICP/AES [36] | 200.7 [5] | 3120 B [18th, 19th, 20th] | | | |
| 6. Arsenic-Total[4] mg/L: | | | | | |

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

| Parameter, units and method | Reference (method number or page) | | | | |
|---|---|---|---|---|---|
| | EPA [1,35] | *Standard Methods* [Edition(s)] | ASTM | USGS [2] | Other |
| Titrimetric (EDTA), or Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration (See Parameters 13 and 33). | 130.2 | 2340 B or C [18th, 19th, 20th]. | D1126–86(92) | I–1338–85 | 973.52B [3] |
| (28) Hydrogen ion (pH), pH units: | | | | | |
| Electrometric measurement, or. | 150.1 | 4500–$H^+$ B [18th, 19th, 20th]. | D1293–84 (90)(A or B) | I–1586–85 | 973.41 [3] |
| Automated electrode | | | | I–2587–85 | Note 21. |
| 29. Iridium—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration or | 235.1 | 3111 B [18th, 19th] | | | |
| AA furnace | 235.2 | | | | |
| 30. Iron—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration [36] | 236.1 | 3111 B or C [18th, 19th] | D1068–96(A or B) | I–3381–85 | 974.27 [3] |
| AA furnace | 236.2 | 3113 B [18th, 19th] | D1068–96(C) | | |
| ICP/AES [36] | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [50] | |
| DCP [36] or | | | D4190–94 | | Note 34. |
| Colorimetric (Phenanthroline). | | 3500–Fe B [20th] and 3500–Fe D [18th, 19th]. | D1068–96(D) | | Note 22. |
| (31) Kjeldahl Nitrogen—Total, (as N), mg/L: | | | | | |
| Digestion and distillation followed by. | 351.3 | 4500–$N_{org}$ B or C and 4500–$NH_3$ B [18th, 19th, 20th]. | D3590–89(A) | | |
| Titration | 351.3 | | D3590–89(A) | | 973.48 [3] |
| Nesslerization | 351.3 | 4500–$NH_3$ C [18th] | D3590–89(A) | | |
| Electrode | 351.3 | 4500–$NH_3$ C [19th, 20th] and 4500–$NH_3$ E [18th]. | | | |
| Automated phenate colorimetric | 351.1 | | | I–4551–78 [8] | |
| Semi-automated block digestor colorimetric. | 351.2 | | D3590–89(B) | I–4515–91 [45] | |
| Manual or block digestor potentiometric. | 351.4 | | D3590–89(A) | | |
| Block digester, followed by Auto distillation and Titration, or. | | | | | Note 39. |
| Nesslerization, or | | | | | Note 40. |
| Flow injection gas diffusion | | | | | Note 41. |
| 32. Lead—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration [36] | 239.1 | 3111 B or C [18th, 19th] | D3559–96(A or B) | I–3399–85 | 974.27 [3] |

| | | | | | |
|---|---|---|---|---|---|
| AA furnace | 239.2 | 3113 B [18th, 19th] | D3559–96(D) | I–4403–89 [51] | |
| ICP/AES [36] | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [50] | |
| DCP [36] | | | D4190–94 | | Note 34. |
| Voltametry [11] or | | | D3559–96(C) | | |
| Colorimetric (Dithizone) | | 3500–Pb B [ 20th] and 3500–Pb D [18th, 19th]. | | | |
| 33. Magnesium—Total,[4] mg/L; Digestion [4] followed by: | | | | | |
| AA direct aspiration | 242.1 | 3111 B [18th, 19th] | D511–93(B) | I–3447–85 | 974.27 [3] |
| ICP/AES | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [50] | |
| DCP or | | | | | Note 34. |
| Gravimetric | | 3500–Mg D [18th, 19th] | | | |
| 34. Manganese-Total,[4] mg/L; Digestion [4] followed by: | | | | | |
| AA direct aspiration [35] | 243.1 | 3111 B [18th, 19th] | D858–95(A or B) | I–3454–85 | 974.27 [3] |
| AA furnace | 243.2 | 3113 B [18th, 19th] | D858–95(C) | | |
| ICP/AES [36] | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [50] | |
| DCP [36], or | | | D4190–94 | | Note 34 |
| Colorimetric (Persulfate), or | | 3500–Mn B [20th] and 3500–Mn D [18th, 19th]. | | | 920.203 [3] |
| (Periodate) | | | | | Note 23. |
| 35. Mercury—Total,[4] mg/L: | | | | | |
| Cold vapor, manual or | 245.1 | 3112 B [18th, 19th] | D3223–91 | I–3462–85 | 977.22 [3] |
| Automated | 245.2 | | | | |
| Oxidation, purge and trap, and cold vapor atomic fluorescence spectrometry (ng/L). | 1631E [43] | | | | |
| 36. Molybdenum—Total [4], mg/L; Digestion [4] followed by: | | | | | |
| AA direct aspiration | 246.1 | 3111 D [18th, 19th] | | I–3490–85 | |
| AA furnace | 246.2 | 3113 B [18th, 19th] | | I–3492–96 [47] | |
| ICP/AES | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [50] | |
| DCP | | | | | Note 34. |
| 37. Nickel—Total,[4] mg/L; Digestion [4] followed by: | | | | | |
| AA direct aspiration [35] | 249.1 | 3111 B or C [18th, 19th] | D1886–90(A or B) | I–3499–85. | |
| AA furnace | 249.2 | 3113 B [18th, 19th] | D1886–90(C) | I–4503–89 [51]. | |
| ICP/AES [36] | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [50]. | |
| DCP [36], or | | | D4190–94 | | Note 34. |
| Colorimetric (heptoxime) | | 3500–Ni D [17th]. | | | |
| 38. Nitrate (as N), mg/L: | | | | | |
| Colorimetric (Brucine sulfate), or Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40). | 352.1 | | | | 973.50,[3] 419D,[17] p. 28 [9] |
| 39. Nitrate-nitrite (as N), mg/L: | | | | | |
| Cadmium reduction, Manual or. | 353.3 | 4500–$NO_3^-$ E [18th, 19th, 20th]. | D3867–99(B). | | |

* Nitrate (as N), mg/L, Ion Chromatography EPA 300.00

TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

| Parameter, units and method | Reference (method number or page) | | | | |
|---|---|---|---|---|---|
| | EPA [1,35] | *Standard Methods* [Edition(s)] | ASTM | USGS [2] | Other |
| Automated, or | 353.2 | 4500–$NO_3^-$ F [18th, 19th, 20th]. | D3867–99(A) | I–4545–85. | |
| Automated hydrazine | 353.1 | 4500–$NO_3^-$ H [18th, 19th, 20th]. | | | |
| 40. Nitrite (as N), mg/L; Spectrophotometric: | | | | | |
| Manual or | 354.1 | 4500–$NO_2^-$ B [18th, 19th, 20th]. | | | Note 25. |
| Automated (Diazotization) | | | | I–4540–85. | |
| 41. Oil and grease—Total recoverable, mg/L: | | | | | |
| Gravimetric (extraction) | 413.1 | 5520B [18th, 19th, 20th] [38]. | | | |
| Oil and grease and nonpolar material, mg/L: Hexane extractable material (HEM): n-Hexane extraction and gravimetry. | 1664A [42] | 5520B [18th, 19th, 20th] [38]. | | | |
| Silica gel treated HEM (SGT-HEM): Silica gel treatment and gravimetry. | 1664A [42]. | | | | |
| 42. Organic carbon—Total (TOC), mg/L: | | | | | |
| Combustion or oxidation | 415.1 | 5310 B, C, or D [18th, 19th, 20th]. | D2579–93 (A or B) | | 973.47,[3] p. 14 [24] |
| 43. Organic nitrogen (as N), mg/L: Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4). | | | | | |
| 44. Orthophosphate (as P), mg/L; Ascorbic acid method: | | | | | |
| Automated, or | 365.1 | 4500–P F [18th, 19th, 20th] | | I–4601–85 | 973.56 [3] |
| Manual single reagent | 365.2 | 4500–P E [18th, 19th, 20th] | D515–88(A) | | 973.55 [3] |
| Manual two reagent | 365.3. | | | | |
| 45. Osmium—Total[4], mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration, or | 252.1 | 3111 D [18th, 19th]. | | | |
| AA furnace | 252.2. | | | | |
| 46. Oxygen, dissolved, mg/L: | | | | | |
| Winkler (Azide modification), or. | 360.2 | 4500–O C [18th, 19th, 20th] | D888–92(A) | I–1575–78 [6] | 973.45B [3] |
| Electrode | 360.1 | 4500–O G [18th, 19th, 20th]. | D888–92(B) | I–1576–78 [6]. | |

* Nitrite (as N), mg/L, Ion Chromatography EPA 300.00

| | | | | | |
|---|---|---|---|---|---|
| 47. Palladium—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration, or | 253.1 | 3111 B [18th, 19th] | | | p. S27 [10] |
| AA furnace | 253.2 | | | | p. S28 [10] |
| DCP | | | | | Note 34. |
| 48. Phenols, mg/L: | | | | | |
| Manual distillation [26] | 420.1 | | | | Note 27. |
| Followed by:. | | | | | |
| Colorimetric (4AAP) manual, or. | 420.1 | | | | Note 27. |
| Automated [19] | 420.2. | | | | |
| 49. Phosphorus (elemental), mg/L: | | | | | |
| Gas-liquid chromatography | | | | | Note 28. |
| 50. Phosphorus—Total, mg/L: | | | | | |
| Persulfate digestion followed by. | 365.2 | 4500–P B, 5 [18th, 19th, 20th]. | | | 973.55 [3] |
| Manual or | 365.2 or 365.3 | 4500–P E [18th, 19th, 20th] | D515–88(A) | | |
| Automated ascorbic acid reduction. | 365.1 | 4500–P F [18th, 19th, 20th] | | I–4600–85 | 973.56 [3] |
| Semi-automated block digestor. | 365.4 | | D515–88(B) | I–4610–91 [45]. | |
| 51. Platinum—Total,[4] mg/L: Digestion[4] followed by: | | | | | |
| AA direct aspiration | 255.1 | 3111 B [18th, 19th]. | | | |
| AA furnace | 255.2. | | | | |
| DCP | | | | | Note 34 |
| 52. Potassium—Total,[4] mg/L: Digestion[4] followed by: | | | | | |
| AA direct aspiration | 258.1 | 3111 B [18th, 19th] | | I–3630–85 | 973.53 [3] |
| ICP/AES | 200.7 [5] | 3120 B [18th, 19th, 20th]. | | | |
| Flame photometric, or | | 3500–K B [20th] and 3500–K D [18th, 19th]. | | | |
| Colorimetric | | | | | 317 B [17] |
| 53. Residue—Total, mg/L: | | | | | |
| Gravimetric, 103–105° | 160.3 | 2540 B [18th, 19th, 20th] | | I–3750–85. | |
| 54. Residue—filterable, mg/L: | | | | | |
| Gravimetric, 180° | 160.1 | 2540 C [18th, 19th, 20th] | | I–1750–85. | |
| 55. Residue—nonfilterable (TSS), mg/L: | | | | | |
| Gravimetric, 103–105° post washing of residue. | 160.2 | 2540 D [18th, 19th, 20th] | | I–3765–85. | |
| 56. Residue—settleable, mg/L: | | | | | |
| Volumetric, (Imhoff cone), or gravimetric. | 160.5 | 2540 F [18th, 19th, 20th]. | | | |
| 57. Residue—Volatile, mg/L: | | | | | |
| Gravimetric, 550° | 160.4 | | | I–3753–85. | |
| 58. Rhodium-Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration, or | 265.1 | 3111 B [18th, 19th]. | | | |

| | | | | | |
|---|---|---|---|---|---|
| Colorimetric (methylene blue). | 376.2 | 4500–$S^{-2}$D [18th, 19th, 20th]. | | | |
| 67. Sulfite (as $SO_3$), mg/L: | | | | | |
| Titrimetric (iodine-iodate) | 377.1 | 4500–$SO_3^{-2}$B [18th, 19th, 20th]. | | | |
| 68. Surfactants, mg/L: | | | | | |
| Colorimetric (methylene blue). | 425.1 | 5540 C [18th, 19th, 20th] | D2330–88. | | |
| 69. Temperature, °C: | | | | | |
| Thermometric | 170.1 | 2550 B [18th, 19th, 20th] | | | Note 32. |
| 70. Thallium—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration | 279.1 | 3111 B [18th, 19th]. | | | |
| AA furnace | 279.2. | | | | |
| ICP/AES | 200.7 [5] | 3120 B [18th, 19th, 20th]. | | | |
| 71. Tin—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration | 282.1 | 3111 B [18th, 19th] | | I–3850–78 [6]. | |
| AA furnace, or | 282.2 | 3113 B [18th, 19th]. | | | |
| ICP/AES | 200.7 [5]. | | | | |
| 72. Titanium—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration | 283.1 | 3111 D [18th, 19th]. | | | |
| AA furnace | 283.2. | | | | |
| DCP | | | | | Note 34. |
| 73. Turbidity, NTU: | | | | | |
| Nephelometric | 180.1 | 2130 B [18th, 19th, 20th] | D1889–94(A) | I–3860–85. | |
| 74. Vanadium—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration | 286.1 | 3111 D [18th, 19th]. | | | |
| AA furnace | 286.2 | | D3373–93. | | |
| ICP/AES | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [50]. | |
| DCP, or | | | D4190–94 | | Note 34. |
| Colorimetric (Gallic Acid) | | 3500–V B [20th] and 3500–V D [18th, 19th]. | | | |
| 75. Zinc—Total,[4] mg/L; Digestion[4] followed by: | | | | | |
| AA direct aspiration [36] | 289.1 | 3111 B or C [18th, 19th] | D1691–95(A or B) | I–3900–85 | 974.27,[3] p. 37 [9] |
| AA furnace | 289.2. | | | | |
| ICP/AES [36] | 200.7 [5] | 3120 B [18th, 19th, 20th] | | I–4471–97 [50]. | |
| DCP,[36] or | | | D4190–94 | | Note 34. |
| Colorimetric (Dithizone) or | | 3500–Zn E [18th, 19th]. | | | |
| (Zincon) | | 3500–Zn B [20th] and 3500–Zn F [18th, 19th]. | | | Note 33. |

Table 1B Notes:

[1] "Methods for Chemical Analysis of Water and Wastes," Environmental Protection Agency, Environmental Monitoring Systems Laboratory—Cincinnati (EMSL-CI), EPA–600/4–79–020, Revised March 1983 and 1979 where applicable.

[2] Fishman, M.J., *et al.* "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments, "U.S. Department of the Interior, Techniques of Water-Resource Investigations of the U.S. Geological Survey, Denver, CO, Revised 1989, unless otherwise stated.

[3] "Official Methods of Analysis of the Association of Official Analytical Chemists," methods manual, 15th ed. (1990).

[4] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979 and 1983". One (Section 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (Section 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all samples types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials may also benefit by this vigorous digestion, however, vigorous digestion with concentrated nitric acid will convert antimony and tin to insoluble oxides and render them unavailable for analysis. Use of ICP/AES as well as determinations for certain elements such as antimony, arsenic, the noble metals, mercury, selenium, silver, tin, and titanium require a modified sample digestion procedure and in all cases the method write-up should be consulted for specific instructions and/or cautions.

NOTE TO TABLE 1B NOTE 4: If the digestion procedure for direct aspiration AA included in one of the other approved references is different than the above, the EPA procedure must be used. Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals (or digestion of the original sample solution for total metals) may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses, provided the sample solution to be analyzed meets the following criteria:

a. has a low COD (<20)

b. is visibly transparent with a turbidity measurement of 1 NTU or less

c. is colorless with no perceptible odor, and

d. is of one liquid phase and free of particulate or suspended matter following acidification.

[5] The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 136.

[6] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary: however, manual distillation will be required to resolve any controversies.

[7] Ammonia, Automated Electrode Method, Industrial Method Number 379–75 WE, dated February 19, 1976, Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.

[8] The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979).

[9] American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 25 West 43rd Street, New York, NY 10036.

[10] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

[11] The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[12] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional $BOD_5$ test method which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional $BOD_5$ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data using a nitrification inhibitor.

[13] OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, PO Box 2980, College Station, TX 77840.

[14] Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, PO Box 389, Loveland, CO 80537.

[15] The back titration method will be used to resolve controversy.

[16] Orion Research Instruction Manual, Residual Chlorine Electrode Model 97–70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138. The calibration graph for the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 mL 0.00281 N potassium iodate/100 mL solution, respectively.

[17] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976.

[18] National Council of the Paper Industry for Air and Stream Improvement, Inc. Technical Bulletin 253, December 1971.

[19] Copper, Biocinchoinate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, PO Box 389, Loveland, CO 80537.

[20] After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

[21] Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378–75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II. Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.

[22] Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, PO Box 389, Loveland, CO 80537.

[23] Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2–113 and 2–117, Hach Chemical Company, Loveland, CO 80537.

[24] Wershaw, R.L., *et al*, "Methods for Analysis of Organic Substances in Water," Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14.

[25] Nitrogen, Nitrite, Method 8507, Hach Chemical Company, PO Box 389, Loveland, CO 80537.

[26] Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.

[27] The approved method is cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0±0.2. The approved methods are given on pp 576–81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrometric procedure.

[28] R.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, Vol. 47, No. 3, pp. 421–426, 1970.

[29] Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $Na_2S_2O_3$ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[30] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition.

[31] EPA Methods 335.2 and 335.3 require the NaOH absorber solution final concentration to be adjusted to 0.25 N before colorimetric determination of total cyanide.
[32] Stevens, H.H., Ficke, J.F., and Smoot, G.F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation," Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975.
[33] Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2–231 and 2–333, Hach Chemical Company, Loveland, CO 80537.
[34] "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986—Revised 1991, Thermo Jarrell Ash Corporation, 27 Forge Parkway, Franklin, MA 02038.
[35] Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, "Precision and Recovery Statements for Methods for Measuring Metals".
[36] "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, PO Box 200, Matthews, NC 28106–0200, April 16, 1992. Available from the CEM Corporation.
[37] When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.
[38] Only use Trichlorotrifluorethane (1,1,2-trichloro-1,2,2-trifluoroethane; CFC–113) extraction solvent when determining Total Recoverable Oil and Grease (analogous to EPA Method 413.1). Only use *n*-hexane extraction solvent when determining Hexane Extractable Material (analogous to EPA Method 1664A). Use of other extraction solvents is strictly prohibited.
[39] Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, PO Box 9010, College Station, TX 77842.
[40] Nitrogen, Total Kjeldahl, Method PAI-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, PO Box 9010, College Station, TX 77842.
[41] Nitrogen, Total Kjeldahl, Method PAI-DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, OI Analytical/ALPKEM, PO Box 9010, College Station, TX 77842.
[42] Method 1664, Revision A "n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry" EPA–821–R–98–002, February 1999. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.
[43] USEPA. 2002. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry." September 2002. Office of Water, U.S. Environmental Protection Agency (EPA–821–R–02–019). The application of clean techniques described in EPA's draft Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels (EPA–821–R–96–011) are recommended to preclude contamination at low-level, trace metal determinations.
[44] Available Cyanide, Method OIA–1677 (Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry), ALPKEM, A Division of OI Analytical, PO Box 9010, College Station, TX 77842–9010.
[45] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonia Plus Organic Nitrogen by a Kjeldahl Digestion Method", Open File Report (OFR) 00–170.
[46] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry", Open File Report (OFR) 93–449.
[47] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry", Open File Report (OFR) 97–198.
[48] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis" Open File Report (OFR) 92–146.
[49] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace-Atomic Absorption Spectrometry" Open File Report (OFR) 98–639.
[50] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry", Open File Report (OFR) 98–165.
[51] "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediment", Open File Report (OFR) 93–125.

TABLE IC—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

| Parameter[1] | EPA method number[2,7] | | | Other approved methods | | |
|---|---|---|---|---|---|---|
| | GC | GC/MS | HPLC | *Standard Methods* [Edition(s)] | ASTM | Other |
| 1. Acenaphthene | 610 | 625, 1625B | 610 | 6440 B [18th, 19th, 20th]. | D4657–92 | Note 9, p.27. |
| 2. Acenaphthylene | 610 | 625, 1625B | 610 | 6440 B, 6410 B [18th, 19th, 20th]. | D4657–92 | Note 9, p.27. |
| 3. Acrolein | 603 | 624[4], 1624B | | | | |
| 4. Acrylonitrile | 603 | 624[4], 1624B | | | | |
| 5. Anthracene | 610 | 625, 1625B | 610 | 6410 B, 6440 B [18th, 19th, 20th]. | D4657–92 | Note 9, p. 27. |

**ATTACHMENT E**

**Table II - Required Containers, Preservation Techniques, and Holding Times**
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3544. Available from the American Society for Microbiology, 1752 N Street NW., Washington, DC 20036. Table IA, Note 22.

(58) USEPA. 2002. Method 1604: Total Coliforms and *Escherichia coli (E. coli)* in Water by Membrane Filtration using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington D.C. September 2002, EPA 821–R–02–024. Available from NTIS, PB2003–100129. Table IA, Note 22.

(59) USEPA. 2002. Method 1600: Enterococci in Water by Membrane Filtration using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington D.C. September 2002, EPA–821–R–02–022. Available from NTIS, PB2003–100127. Table IA, Note 25.

(60) USEPA. 2001. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC April 2001, EPA–821–R–01–026.

Available from NTIS, PB2002–108709. Table IA, Note 26.

(61) USEPA. 2001. Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC April 2001, EPA–821–R–01–025. Available from NTIS, PB2002–108710. Table IA, Note 27.

(62) AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. AOAC International. 481 North Frederick Avenue, Suite 500, Gaithersburg, Maryland 20877–2417. Table IA, Note 11.

(c) Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director upon the recommendation of the Director of the Environmental Monitoring Systems Laboratory—Cincinnati.

(d) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring Systems Laboratory—Cincinnati, additional alternate test procedures for nationwide use.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters cited in Tables IA, IB, IC, ID, and IE are prescribed in Table II. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded, by the Regional Administrator, to the Director of the Environmental Monitoring Systems Laboratory—Cincinnati, Ohio for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Director of the Environmental Monitoring Systems Laboratory, the Regional Administrator may grant a variance applicable to the specific charge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

| Parameter No./name | Container [1] | Preservation [2,3] | Maximum holding time [4] |
|---|---|---|---|
| Table IA—Bacteria Tests: | | | |
| 1–5 Coliform, total, fecal, and *E. coli* | PP, G | Cool, <10 °C, 0.0008% $Na_2S_2O_3$[5]. | 6 hours. |
| 6 Fecal *streptococci* | PP, G | Cool, <10° 0.0008% $Na_2S_2O_3$[5] | 6 hours. |
| 7 *Enterococci* | PP, G | Cool, <10° 0.0008% $Na_2S_2O_3$[5] | 6 hours. |
| Table IA—Protozoa Tests: | | | |
| 8 *Cryptosporidium* | LDPE | 0–8 °C | 96 hours. [17] |
| 9 *Giardia* | LDPE | 0–8 °C | 96 hours. [17] |
| Table IA—Aquatic Toxicity Tests: | | | |
| 6–10 Toxicity, acute and chronic | P,G | Cool, 4 °C [16] | 36 hours. |

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

| Parameter No./name | Container [1] | Preservation [2,3] | Maximum holding time [4] |
|---|---|---|---|
| Table IB—Inorganic Tests: | | | |
| 1. Acidity | P, G | Cool, 4°C | 14 days. |
| 2. Alkalinity | P, G | do | Do. |
| 4. Ammonia | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 9. Biochemical oxygen demand | P, G | Cool, 4°C | 48 hours. |
| 10. Boron | P, PFTE, or Quartz. | $HNO_3$ TO pH<2 | 6 months. |
| 11. Bromide | P, G | None required | 28 days. |
| 14. Biochemical oxygen demand, carbonaceous | P, G | Cool, 4°C | 48 hours. |
| 15. Chemical oxygen demand | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 16. Chloride | P, G | None required | Do. |
| 17. Chlorine, total residual | P, G | do | Analyze immediately. |
| 21. Color | P, G | Cool, 4°C | 48 hours. |
| 23–24. Cyanide, total and amenable to chlorination. | P, G | Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid [5]. | 14 days.[6] |
| 25. Fluoride | P | None required | 28 days. |
| 27. Hardness | P, G | $HNO_3$ to pH<2, $H_2SO_4$ to pH<2 | 6 months. |
| 28. Hydrogen ion (pH) | P, G | None required | Analyze immediately. |
| 31, 43. Kjeldahl and organic nitrogen | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| Metals:[7] | | | |
| 18. Chromium VI [7] | P, G | Cool, 4 °C | 24 hours. |
| 35. Mercury [17] | P, G | $HNO_3$ to pH<2 | 28 days. |
| 3, 5–8, 12,13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals except boron, chromium VI and mercury [7]. | P, G | do | 6 months. |
| 38. Nitrate | P, G | Cool, 4°C | 48 hours. |
| 39. Nitrate-nitrite | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 40. Nitrite | P, G | Cool, 4°C | 48 hours. |
| 41. Oil and grease | G | Cool to 4°C, HCl or $H_2SO_4$ to pH<2. | 28 days. |
| 42. Organic Carbon | P, G | Cool to 4 °C HC1 or $H_2SO4$ or $H_3PO4$, to pH<2. | 28 days. |
| 44. Orthophosphate | P, G | Filter immediately, Cool, 4°C | 48 hours. |
| 46. Oxygen, Dissolved Probe | G Bottle and top. | None required | Analyze immediately. |
| 47. Winkler | do | Fix on site and store in dark | 8 hours. |
| 48. Phenols | G only | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 49. Phosphorus (elemental) | G | Cool, 4°C | 48 hours. |
| 50. Phosphorus, total | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 53. Residue, total | P, G | Cool, 4°C | 7 days. |
| 54. Residue, Filterable | P, G | do | 7 days. |
| 55. Residue, Nonfilterable (TSS) | P, G | do | 7 days. |
| 56. Residue, Settleable | P, G | do | 48 hours. |
| 57. Residue, volatile | P, G | do | 7 days. |
| 61. Silica | P, PFTE, or Quartz. | Cool, 4 °C | 28 days. |
| 64. Specific conductance | P, G | do | Do. |
| 65. Sulfate | P, G | do | Do. |
| 66. Sulfide | P, G | Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9. | 7 days. |
| 67. Sulfite | P, G | None required | Analyze immediately. |
| 68. Surfactants | P, G | Cool, 4°C | 48 hours. |
| 69. Temperature | P, G | None required | Analyze. |
| 73. Turbidity | P, G | Cool, 4°C | 48 hours. |
| Table IC—Organic Tests [8] | | | |
| 13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons. | G, Teflon-lined septum. | Cool, 4 °C, 0.008% $Na_2S_2O_3$ [5]. | 14 days. |
| 6, 57, 106. Purgeable aromatic hydrocarbons | do | Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] HCl to pH2[9]. | Do. |
| 3, 4. Acrolein and acrylonitrile | do | Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] adjust pH to 4–5[10]. | Do. |
| 23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols [11]. | G, Teflon-lined cap.. | Cool, 4 °C, 0.008% $Na_2S_2O_3$ [5] | 7 days until extraction; 40 days after extraction. |
| 7, 38. Benzidines [11] | do | do | 7 days until extraction.[13] |
| 14, 17, 48, 50–52. Phthalate esters [11] | do | Cool, 4 °C | 7 days until extraction; 40 days after extraction. |

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

| Parameter No./name | Container [1] | Preservation [2, 3] | Maximum holding time [4] |
|---|---|---|---|
| 82–84. Nitrosamines [11, 14] | .....do ..... | Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] store in dark. | Do. |
| 88–94. PCBs [11] | .....do ..... | Cool, 4 °C | Do. |
| 54, 55, 75, 79. Nitroaromatics and isophorone [11]. | .....do ..... | Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] store in dark. | Do. |
| 1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons [11]. | .....do ..... | .....do | Do. |
| 15, 16, 21, 31, 87. Haloethers [11] | .....do ..... | Cool, 4 °C, 0.008% $Na_2S_2O_3$[5] | Do. |
| 29, 35–37, 63–65, 73, 107. Chlorinated hydrocarbons [11]. | .....do ..... | Cool, 4 °C | Do. |
| 60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs [11]. | | | |
| aqueous: field and lab preservation. | G | Cool, 0–4 °C, pH<9, 0.008% $Na_2S_2O_3$ [5]. | 1 year. |
| Solids, mixed phase, and tissue: field preservation.. | .....do ..... | Cool, <4 °C | 7 days. |
| Solids, mixed phase, and tissue: lab preservation. | .....do ..... | Freeze, <−10 °C | 1 year. |
| Table ID—Pesticides Tests: | | | |
| 1–70. Pesticides [11] | .....do ..... | Cool, 4°C, pH 5–9 [15] | Do. |
| Table IE—Radiological Tests: | | | |
| 1–5. Alpha, beta and radium | P, G | $HNO_3$ to pH<2 | 6 months. |

Table II Notes

[1] Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic).

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[3] When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid ($HNO_3$) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid ($H_2SO_4$) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See §136.3(e) for details. The term "analyze immediately" usually means within 15 minutes or less of sample collection.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[16] Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the 4C temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature can not be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

[17] Samples collected for the determination of trace level mercury (100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipment, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that have been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

# ATTACHMENT F

## Sample Chain of Custody Form

# QC Laboratories

1205 Industrial Blvd.
Southampton, PA 18966-0514
Phone: 215-355-3900
Fax: 215-355-7231

# CHAIN OF CUSTODY

Page _____ of _____

Lab LIMS No:

Bill to/Report to: (if different)

Client/Acct. No.

Address

Sampling Site Address: (if different)

City/State/Zip

Phone/Fax

P.O. No.

Client Contact

QC Contact

**LAB USE ONLY:**

# ____ Ascorbic/HCl Vials # ___ HCl Vials
# ____ $Na_2S_2O_3$
# ____ Na OH/Zn acetate pH
# ____ $HNO_3$ pH
# ____ $H_2SO_4$ pH
# ____ NaOH pH
# ____ Unpreserved
# ____ Hcl pH
# ____ Temp control _____ ID# _____

**MATRIX CODES**

DW: DRINKING WATER
GW: GROUND WATER
WW: WASTEWATER
SO: SOIL
SL: SLUDGE
OIL: OIL
SOL: NON SOIL SOLID
MI: MISCELLANEOUS
X: OTHER

LAB USE ONLY

| PROJECT / FIELD ID | Collection: Date | Collection: Military Time | GRAB | COMP | Matrix Code | Number of Containers: Total | $H_2SO_4$ | HCl | Vials | $HNO_3$ | NaOH | ZnAc | Unpres | Bact | ANALYSIS REQUESTED | Field pH, Temp (C or F), DO, $Cl_2$, S. Cond. etc. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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SAMPLED BY: (Name/Company)

Verbal/fax data due: ____/____/____

Hardcopy due: ____/____/____

Report Format: ☐ Standard ☐ Forms
☐ Standard + QC ☐ NJ Reduced ☐ Disk

*Please call for pricing and availability on rush (<14-21 day) turnaround and on all but standard format.*

Field Parameters Analyzed By:

Sig: Date/Time:

**SAMPLE CUSTODY EXCHANGES MUST BE DOCUMENTED BELOW. USE FULL LEGAL SIGNATURE, DATE AND MILITARY TIME (24 HOUR CLOCK, I.E. 8AM IS 0800, 4 PM IS 1600)**

| RELINQUISHED BY SAMPLER | DATE | TIME | RECEIVED BY | DATE | TIME |
|---|---|---|---|---|---|
| 1 | | | 1 | | |
| RELINQUISHED BY | DATE | TIME | RECEIVED BY | DATE | TIME |
| 2 | | | 2 | | |
| RELINQUISHED BY | DATE | TIME | RECEIVED BY | DATE | TIME |
| 3 | | | 3 | | |
| RELINQUISHED BY | DATE | TIME | RECEIVED BY | DATE | TIME |
| 4 | | | 4 | | |
| RELINQUISHED BY | DATE | TIME | RECEIVED BY | DATE | TIME |
| 5 | | | 5 | | |

DELIVERY METHOD: ☐ QC COURIER ☐ CLIENT ☐ UPS ☐ FEDEX ☐ OTHER_____

Custody Seal Number

COMMENTS:

Hazardous: yes / no

***For example to aid completion, see reverse side.***

FINAL REPORT

# ATTACHMENT G

## Tables of Parameter Detection Limits, Accuracy, and Precision

| Parameter: | Dissolved Ortho-Phosphate (as P) | Total Phosphorus (as P) | Ammonia-Nitrogen | Nitrate-Nitrogen | Nitrite-Nitrogen | Total Kjeldahl Nitrogen | Total Suspended Solids | Fecal Coliform | *E. coli*[*] |
|---|---|---|---|---|---|---|---|---|---|
| **Referenced Methodology – (NJDEP Certified Methodology)** | EPA 365.2 | EPA 365.2 | EPA 350.2 350.3 | EPA 300.0 | EPA 300.0 | EPA 351.2 351.4 | Standard Methods 2540D | Standard Methods 9222D | EPA 1103.1 |
| **Method Detection Limit (ppm)-Calculated** | 0.0024 | 0.016 | 0.008 | 0.02 | 0.04 | 0.047 | NA | <10 | <10 |
| **Instrument Detection Limit (ppm)** | NA | NA | NA | 0.02 | 0.04 | NA | NA | NA | NA |
| **Project Detection Limit (ppm)** | ***0.01*** | ***0.02*** | ***0.05*** | ***0.02*** | ***0.04*** | ***0.05*** | NA | <10 | <10 |
| **Quantitation Limit (ppm)** | 0.01 | 0.02 | 0.05 | 0.02 | 0.04 | 0.05 | 0.5 | NA | NA |
| **Accuracy (mean % recovery)** | 99.50 | 97.08 | 98.89 | 92.18 | 95.84 | 97.66 | NA | NA | NA |
| **Precision-% (mean – RPD)** | 1.33 | 4.19 | 5.14 | 0.70 | 0.96 | 7.39 | 7.44 | 10% RPD | 10% RPD |
| **Accuracy Protocol (% recovery for LCL/UCL)** | 92.38/ 106.62 | 86.71/ 107.44 | 85.13/ 112.65 | 71.17/ 113.19 | 78.80/ 112.88 | 72.39/ 122.92 | NA | NA | NA |
| **Precision Protocol-% (maximum RPD)** | 2.90 | 8.62 | 14.74 | 1.99 | 3.14 | 16.20 | 22.80 | 10% | 10% |

*RPD – Relative % Difference; NA – Not Applicable*

**Laboratory: QC Laboratories (#PA166 & #PA001[*])**

| Parameter: | pH (SU) | Temperature (°C) | Dissolved Oxygen (mg/L) |
|---|---|---|---|
| **Referenced Methodology – (NJDEP Certified Methodology)** | Standard Methods 4500-$H^+$ B | Standard Methods 2550 B | Standard Methods 4500-O G |
| **Method Detection Limit (ppm)** | NA | NA | NA |
| **Instrument Detection Limit (ppm)** | 0.00-14.00 S.U. | 0.0 to 100.0 °C | 0 – 20 mg/L |
| **Project Detection Limit (ppm)** | 0.00-14.00 S.U. | 0.0 to 100.0 °C | 0 - 20 mg/L |
| **Quantitation Limit (ppm)** | NA | NA | NA |
| **Accuracy (mean % recovery)** | NA | NA | NA |
| **Precision (mean – RPD)** | ±0.01 S.U. | ±0.3 °C | ±0.3 mg/L |
| **Accuracy Protocol (% recovery for LCL/UCL)** | NA | NA | NA |
| **Precision Protocol (maximum RPD)** | ±0.01 S.U. | ±0.3 °C | ±0.3 mg/L |

*RPD – Relative % Difference; NA – Not Applicable*

**Laboratory: Rutgers EcoComplex Laboratory (#03019)**