In Reply Refer To:
Mail Stop 412 November 15, 2010

Office of Water Quality Technical Memorandum 2011.02

Subject: National Monitoring Network Water-Quality Sampling, Fiscal Year (FY) 2011

This memorandum contains a summary of the activities related to water-quality sampling as part of the National Monitoring Network (NMN) for FY 2011, including the stations being operated, constituents measured, and funding allocations. This is a continuation of sampling begun in FY 2008 with funds appropriated to support the National Monitoring Network component of the Ocean Action Plan. Information about the Ocean Action Plan can be found at http://www.whitehouse.gov/administration/eop/ceq/initiatives/oceans ; information about the NMN can be found at http://acwi.gov/monitoring/network/. Stations will continue to be operated in a manner identical to those being sampled for the National Stream Quality Accounting Network (i.e., same constituents and sampling strategy; sampling protocols are attached). The attachments to this memorandum provide specific information on sample collection and processing for FY 2011. Please ensure that all field personnel sampling at NMN stations have a copy of this memorandum and attachments.

This effort is being managed by the National Stream Quality Accounting Network (NASQAN) Coordinator. Questions on NMN operations can be addressed to Charlie Crawford (NASQAN Coordinator) or Dave Reutter (NASQAN support). Contact information is contained at the end of this memorandum.

NMN Stations

The highest priority rivers not included in the recently redesigned NASQAN network due to funding limitations will be sampled as part of the NMN. Five stations will be operated in FY 2011 as part of the USGS NMN. These include:

Apalachicola River near Sumatra, FL
Brazos River near Rosharon, TX
Delaware River at Trenton, NJ
Hudson River below Poughkeepsie, NY
Mississippi River above Vicksburg, MS

Attachment 1 provides a listing of the stations that will be sampled and the number of samples to be collected for NMN in FY 2011. The fund transfers, by Water Science Center (WSC) and by account number, are contained in Attachment 2 (if there are updates/corrections to the account numbers, please contact the NASQAN Coordinator). FY 2011 allocations are based on a two-percent increase over FY 2010. **Please note that the NMN budget allocations are only tentative because the FY 2011 NMN appropriation has not been finalized.** The USGS is likely to be operating on a continuing resolution for much of FY 2011. However, we anticipate being able to allocate the full amounts indicated in attachment 2 if the continuing resolution is at least at FY 2010 levels. If NMN funding is less than anticipated, we'll have to reduce the allocations and the number of samples collected in FY 2011 accordingly.

Sampling Frequency and Constituents Measured

All NMN stations will be sampled bi-monthly with a part of the year containing additional seasonally-weighted samples determined by the NASQAN coordinator. The seasonally-weighted samples are targeted at the period of the year when the greatest constituent transport typically occurs. The number of samples to be collected by month for each station is shown in the attached NMN SWPLAN spreadsheet. The sampling should be spaced two weeks apart for months during which two samples are collected. NMN sampling will not routinely be collecting samples based on hydrologic conditions ("event samples"). If you have questions or concerns about the information contained in the NMN SWPLAN, please contact the NASQAN Coordinator.

Constituents measured at NMN stations will be consistent across the network and are the same as those collected at NASQAN stations, although the laboratory schedules used may vary somewhat depending on local conditions. The constituents measured will include major ions, nutrients and carbon, pesticides (schedule 2033), suspended sediment, isotopes, and field parameters (including alkalinity). Collection of dissolved inorganic carbon (DIC) and ultraviolet absorbing organic constituents at 412 nm (UV412) will also continue to be collected. Analytical services (National Water Quality Laboratory (NWQL) and sediment laboratory for NMN samples will be charged to account GX11DD009U2LA00. (**Note: this account is only for NWQL or sediment laboratory costs.** Funds for shipping have been included in the WSC allocations and should not be charged to this account.)

Questions about NMN sampling procedures or how to implement the protocols contained in the attachments, in addition to any concerns about the information contained in the attachments or the protocols, should be addressed to the NASQAN Sampling Coordinator.

NMN Contact Information

For questions on the NMN Program, contact any of the following individuals:

NASQAN Coordinator: Charlie Crawford, Indiana WSC, cgcrawfo@usgs.gov, 317-290-3333 ext 176
NASQAN Support: Dave Reutter, Ohio WSC, dreutter@usgs.gov, 614-430-7732
NASQAN Sampling Coordinator: Richard Coupe, Mississippi WSC, rhcoupe@usgs.gov, 601-933-2982

Donna N. Myers
Chief, Office of Water Quality

This memorandum supersedes OWQ Technical Memorandum 2010.03.

Distribution: A, AO, B, NAWQA NLT, REX, WSC Directors, WSC and Regional Water-Quality Specialists

Attachments

List of Attachments

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Attachment 1. FY 2011 NMN Stations [See NMN SWPLAN spreadsheet for details on number of samples by constituent , number of QC samples, and number of samples to be collected per month.]

Station ID	Name	WSC	Fixed Samples
01372058	Hudson River below Poughkeepsie, NY	NY	12
01463500	Delaware River at Trenton, NJ	NJ	12
02359170	Apalachicola River near Sumatra, FL	FL	12
08116650	Brazos River near Rosharon, TX	TX	12
322023090544500	Mississippi River above Vicksburg, MS	MS	12

Attachment 2. NMN FY 2011 Funding Allocations by WSC and Account Number

WSC	Account	Amount
	Sample collection	
FL	GX11MC009U2MR00	$46,750
MS	GX11MG009LY1200	$47,910
NJ	GX11LJ009U2MR00	$54,540
NY	GX11LK009U2MR00	$57,790
TX	GX11SJ009U20400	$51,940
	Stream gaging	
MS	GX11MG009LY1200	$3,150

[1] Notify NASQAN Coordinator of corrections/updates to account numbers

Attachment 3. FY 2011 NMN Operations

Guidelines for NMN sampling procedures in FY 2011 are attached (Attachments 4, 5, and 6 which describe NASQAN protocols also used by the NMN). WSC personnel are responsible for data review, rerun requests, and timely responses to contamination problems and other data issues. Secondary review of NMN data has been integrated into the NAWQA Data Warehouse.

Quality-Control

The specific types of QC samples to be collected at each NMN station are described in the NMN SWPLAN provided by the NASQAN Coordinator. Guidelines for collection, processing, and coding of QC samples are provided in Attachment 6. Please carefully follow the coding instructions for all QC samples. Consistency across the network is necessary for efficient analysis of the QC data. Standard WSC quality-assurance procedures (for example, periodic review of field operations and data quality by the WSC Water Quality Specialist) should be done for NMN operations.

Laboratory Information

Account Number

Account **GX11DD009U2LA00** has been established for analysis of NMN chemistry and suspended-sediment samples and should be used on the Analytical Service Request (ASR) and sediment analysis request for all NMN samples. This account is only for NWQL and sediment analyses. Shipping charges are included in WSC allocations and should not be charged to the laboratory account.

Laboratory Schedules

Laboratory schedules for analysis of NMN samples at the National Water Quality Laboratory (NWQL) are listed below. There are several choices for routine analyses of both nutrient and major ions, depending upon expected concentrations. **The appropriate schedule for each station is to be selected to avoid nondetects.** If you have questions about this issue please contact the NASQAN Sampling Coordinator.

Schedules and Lab Codes

997 Nutrients, particulate carbon, nitrogen, organic carbon
1010 Nutrients (low-level ortho-P), particulate carbon, nitrogen, organic carbon
1069 Nutrients (low-level P and N), particulate carbon, nitrogen, organic carbon
998 Major ions and miscellaneous trace elements (conductance < 2000)
1201 Major ions and miscellaneous trace elements (conductance > 2000)
2033 Pesticides in filtered water, extracted by NWQL (also used for pesticide blanks and spikes)
Lab Code 8096 Ultraviolet absorbing organic constituents, 412 nm
Lab Code 8097 Carbon, inorganic, filtered (DIC)
452 Nutrient blank
1675 Organic carbon blank
1674 Routine major ion blank

Updated information from the NWQL on sample requirements for these schedules can be obtained from the NWQL web page (http://nwql.cr.usgs.gov/usgs/catalog/index.cfm).

Sediment Laboratories

Suspended-sediment samples should be submitted to the Iowa WSC sediment laboratory. However, NAWQA stations also included in the NMN network may continue to use the sediment laboratory they have been using. A single sample will be submitted for each station on each sampling date. Samples may be composited in the field before being sent to the sediment laboratory. **Samples should be analyzed for concentration and sand-fine break.**

Isotope samples
Field crews at all NMN stations should collect aliquots from the composite water sample for analysis of stable isotopes of water. The 60-mL glass bottles for this sample are available from the Reston Stable Isotope Laboratory; contact Ty Coplen by e-mail at tbcoplen@usgs.gov to obtain the bottles. No filtering is required but a filtered aliquot may be sent. Fill the 60-mL bottle 2/3 full of water and put on the Polyseal cap. No preservation is needed. Label the sample bottle with station id and the date and time of sample collection. Samples can be stored for 4 to 12 months before shipping so that multiple samples may be mailed together to reduce shipping and handling costs. Package samples securely and ship to:

U.S. Geological Survey
Isotope Laboratory
431 National Center
12201 Sunrise Valley Dr
Reston, VA 20192

More information on collection of stable isotope samples is available at http://isotopes.usgs.gov/ and http://isotopes.usgs.gov/lab/instructions.html.

Late samples
No NMN samples are scheduled to be collected in September; therefore, all NMN should be submitted to the NWQL before closeout. All suspended-sediment samples collected in a given fiscal year should be submitted to the laboratory such that they can be charged to the current fiscal year NMN account.

Attachment 4. NMN Water-Quality Sampling Protocols

Introduction

This document provides an overview of standard methods for collecting and processing water-column samples for use in large rivers as part of the NMN program. The methods are intended to provide water-quality data oriented to meet the specific goals of NMN, and conform to established methods of the U.S. Geological Survey as described in detail in the *National Field Manual for the Collection of Water-Quality Data* (http://water.usgs.gov/owq/FieldManual/index.html). Additional information on the rationale for and complete description of these sampling and processing procedures are also contained in Edwards and Glysson (1999), as well as selected Technical Memoranda issued by the Office of Surface Water (OSW) (Technical Memorandum 94.05; http://water.usgs.gov/admin/memo/SW/sw94.05.html) and Office of Water Quality (OWQ) [Technical Memorandum 97.06 (http://water.usgs.gov/admin/memo/QW/qw97.06.html).

Constituents routinely measured by the NMN program include field parameters, suspended sediment, major ions, nutrients, dissolved/particulate carbon, and pesticides. Additional special samples are collected, as described below:

1. All stations
    - Stable isotopes of water (for Ty Coplen)

Specific information regarding the analytical method for particulate carbon/nitrogen is available as follows: OWQ Technical Memorandum 2000.05 (http://water.usgs.gov/admin/memo/QW/qw00.05.html) and OWQ Technical Memorandum 2000.08 (http://water.usgs.gov/admin/memo/QW/qw00.08.html).

Sampling equipment

To the greatest extent possible, isokinetic sampling techniques that provide samples representative of stream conditions are required by the NMN program. Until recently, the range of available samplers for use in large rivers was limited and the ideal sampling scenario was not always possible. Large-river sites traditionally used the US D-77 bag and frame-type bag samplers, which require field calibration each time they are used. Some sites where excessive depth is not an issue have used the US D-77 rigid-bottle sampler, although it is known to be difficult to use correctly because of the very small range in which it samples isokinetically and because it becomes unstable at relatively low velocities. The DH-81 sampler has been used at wadeable sites.

In 2002, OWQ, in concurrence with OSW, recommended that the US D-77 rigid-bottle and bag samplers and the frame-type bag sampler be phased out for collection of water-quality and sediment samples. According to OWQ Technical Memorandum 2002.09 (http://water.usgs.gov/admin/memo/QW/qw02.09.html), the US DH-2, US DH-95, US D-95, and US D-96 isokinetic samplers have been approved by the Interagency Technical Committee of the Subcommittee on Sedimentation, and provide the field with a well-tested and documented set of samplers that work in most sampling conditions. Accordingly, the D-77 rigid-bottle and bag samplers should have been replaced with the newer samplers. Please contact the NASQAN Coordinator (Charlie Crawford) if one of the discontinued samplers still is being used.

The decision on which sampler to use is based upon the hydrologic conditions in the stream, the limits of the samplers, and informed judgment in the field, all of which are essential for collecting a representative sample. Further information is available for the D-96 sampler

(http://fisp.wes.army.mil/Instructions%20US%20D-96%20Instructions%20020709.pdf), the DH-2 sampler (http://fisp.wes.army.mil/Instructions%20050720%20US%20DH-2.pdf), and the DH-81 sampler (http://fisp.wes.army.mil/Instructions%20US_DH-81_010612.pdf). For information on USGS guidelines for equipment selection, consult Chapter 2 in the National Field Manual and USGS Open-File Report 2005-1087 – A Guide to the Proper Selection and Use of Federally-Approved Sediment and Water-Quality Samplers.

A brief overview of the guidelines for the use of each sampler type is provided below.

1. D-99
    - must be coated with Plasti-Dip to prevent trace-element contamination---if plastic coating becomes scratched, reapply Plasti-Dip (spray plastic paint, available locally or online-www.usplastic.com is a source)
    - collects a maximum of nearly 6 L sample per vertical
    - approved for flow velocities between 3 to 15 ft/s
    - nozzle sizes are 3/16, 1/4, and 5/16 inch and give the sampler the capability to sample to depths of 220, 120, and 78 feet respectively
    - Teflon® is required for all components that are in contact with the water sample—nozzle holder, and nozzles.
    - Use disposable poly bags
2. D-96
    - must be coated with Plasti-Dip to prevent trace-element contamination---if plastic coating becomes scratched, reapply Plasti-Dip (spray plastic paint, available locally or online-www.usplastic.com is a source)
    - collects a maximum of nearly 3 L sample per vertical
    - approved for flow velocities between 2 to 12.5 ft/s
    - nozzle sizes are 3/16, 1/4, and 5/16 inch and give the sampler the capability to sample to depths of 110, 60, and 39 feet respectively
    - Teflon® is required for all components that are in contact with the water sample—nozzle holder, nozzles.
    - Use disposable poly bags

3. D-96- A1
    - must be coated with Plasti-Dip to prevent trace-element contamination---if plastic coating becomes scratched, reapply Plasti-Dip (spray plastic paint, available locally or online-www.usplastic.com is a source)
    - collects a maximum of nearly 3 L sample per vertical
    - approved for flow velocities between 2 to 6 ft/s
    - nozzle sizes are 3/16, 1/4, and 5/16 inch and give the sampler the capability to sample to depths of 110, 60, and 39 feet respectively
    - Teflon® is required for all components that are in contact with the water sample—nozzle holder, nozzles.
    - Use disposable poly bags

4. DH-2
    - must be coated with Plasti-Dip to prevent trace-element contamination---if plastic coating becomes scratched, reapply Plasti-Dip (spray plastic paint, available locally or online-www.usplastic.com is a source)
    - collects a maximum of 1 L sample per vertical
    - approved for flow velocities between 2 to 6 ft/s
    - nozzle sizes are 3/16, 1/4, and 5/16 inch and give the sampler the capability to sample to depths of 37, 20, and 13 feet, respectively
    - Teflon® is required for all components that are in contact with the water sample—nozzle holder, and nozzles.
    - Use disposable poly bags
5. DH-81
    - suspended from a rod--used for wadeable streams
    - uses standard D-77 cap and accessories, 1-L bottle
    - collects a maximum of 800 mL sample per vertical
    - approved for flow velocities between 2 and 7.6 ft/s
    - nozzle sizes are 3/16, 1/4, and 5/16 inch and give the sampler the capability to sample to depths up to 12 ft
    - Teflon® is required for all components that are in contact with the water sample—bottle, cap, and nozzles
6. Support equipment
    - when using metal support equipment, such as cranes and reels, or sampling from bridges or cable cars, always use the clean hands/dirty hands technique--GREAT CARE IS NEEDED TO AVOID CONTAMINATING THESE SAMPLES
    - to prevent contamination during sample processing, especially of low-level trace element samples, field vehicles will ideally be dedicated to water-quality work
    - when a dedicated WQ vehicle is not available, the processing area in the vehicle must be free of contaminants including metallic objects, dirt, and oil residue
    - separate storage areas for the sampling equipment must be available
    - prepare areas separately for processing inorganic and organic samples; if necessary, cover metal cabinets and shelves with plastic sheeting before processing the inorganic samples and cover t g and preservation chambers must be used for processing inorganic constituents; use portable he counter space with aluminum foil before processing organic compounds
    - processing chambers for multi-use vehicles
    - field equipment blanks must be collected regularly to document contaminant-free samples

Processing equipment

The inorganic protocol requires the use of protected environments for processing and preservation of water-quality samples in order to reduce the possibility of contamination. They can be easily constructed with several plastic bags and a plastic milk crate, or a frame built with PVC tubing.

- Two types of compositing vessels will be used in general, depending upon the constituent-
    1. churn splitter for major ions and alkalinity, nutrients, carbon, and isotopes
        - to reduce the potential for contamination from atmospheric inputs, the churn splitter must be modified with a plastic (polyethylene or polypropylene) funnel in the lid capable of being capped (already modified in newer churn splitters)

        - this funnel is usually made by cutting off the bottom section of a 1-liter sample bottle and inserting the top into a 1-inch hole drilled into the lid of the churn splitter; the bottom is used for the funnel cap
        - as a further precaution against contamination, the churn splitter should be placed inside two large, clear plastic bags which are kept closed except when adding sample to the churn
        - the churn, inside the plastic bags, is then placed inside a large, covered plastic container (i.e. garbage can) which serves as the churn carrier
    2. glass carboy for pesticides (note: 8-L or 14-L Teflon® churn splitter may alternatively be used for pesticides)
        - wrap the cork with aluminum foil

- **Since the churn splitter has been approved for processing whole-water samples only when concentrations of suspended sediment do not greatly exceed 1,000 mg/L, the cone splitter is recommended for use at a small number of stations where these sediment concentrations are observed**.
    - if logistical concerns preclude the use of the cone splitter at a particular site, however, the rationale for the decision must be documented and the churn may be used
    - for those stations where the cone splitter is used, it will probably be necessary for the entire sample to be collected first and later poured into the cone for splitting; the individual vertical samples must be saved in containers composed of Teflon®, either Teflon® bags or 3-liter Teflon® bottles
    - due to the large volume of water which is collected, 2- or 3-liter bottles may be required under the ports of the cone splitter during the first split
    - careful precautions against atmospheric contamination must also be taken when using the cone splitter, preferably involving the use of the splitter only within an enclosed laboratory vehicle
    - space limitations may dictate that the splitter be used outdoors; if this is the case, a splitting container is recommended to shield the bottles from atmospheric inputs
    - consult National Field Manual Ch. 5.1.1.B for further details on the use of the cone splitter
    - glass or plastic bottles will be used to collect the sample for suspended sediment.

Overview of filtration systems:

- for inorganics and pesticides--a variable-speed pump fitted with a peristaltic pump head that forces the sample through either C-Flex or Teflon® tubing into a filter assembly
    - for inorganic constituents --a GWV capsule filter system (effective pore size of 0.45 µm) (available from the National Field Supply Service through One-Stop Shopping as item Q398FLD).
    - for pesticide samples--a 142-mm-diameter plate filter with a glass fiber filter (pore size of 0.7 µm) which has been baked to remove any organic residue
- for particulate carbon, dissolved organic carbon, dissolved inorganic carbon, UV, and nitrogen samples—a GWV capsule filter system) (available from the National Field Supply Service through One-Stop Shopping as item Q398FLD) or a Teflon® pressure-filtration assembly that holds a 25-mm glass fiber filter (nominal pore size of 0.7 µm) (available from the National Field Supply Service through One-Stop Shopping as item Q441FLD).

Equipment cleaning

Cleaning procedures are described in detail in Chapter 3 in the National Field Manual. Briefly, they include the following--

- use of gloves, which are changed between each step
- 30-minute soak in Liqui-Nox (0.1% solution), or other phosphate-free detergent, followed by scrubbing with a nonmetallic, noncolored brush **(except for the organic carbon filtration unit and Teflon equipment, unless deemed necessary because of exposure to high concentrations of organic carbon)**
- thorough rinsing with hot tap water
- final rinses:
    1. sampling equipment (and cone splitter, if used)
        - 30-minute soak in a solution of 5% hydrochloric acid (trace-element grade)
        - three rinses with DIW
        - rinse with a small amount of methanol (pesticide grade)
        - final rinse with organic-free water
    2. churn splitter and associated tubing
        - 30-minute soak in a solution of 5% hydrochloric acid (trace-element grade)
        - three rinses with DIW
    3. glass carboy (or Teflon® churn splitter) and Teflon® tubing for pesticide filtration
        - rinse with DIW and then rinse with a small amount of methanol (pesticide grade)
- for the organic carbon filtration unit and sampling equipment--thorough rinse with organic-free water
- allow everything to air dry completely
- protect areas of the sampling equipment and Teflon® tubing that will contact the sample with a Teflon® bag and place in another sealable double plastic bag or other container for storage and transport
- wrap filtration units for pesticides and organic carbon with aluminum foil or place in a Teflon® bag and store in a sealable container
- rinse all inorganic sample bottles three times with DIW, then half-fill with DIW for transport to the field

Field analyses

Complete descriptions of standard protocols for field measurements are provided in Chapter 6 of the National Field Manual.

Specific guidelines for the NMN program include the following:

- Obtain a cross-section stream profile of field measurements at least twice/year
    - record observations from at least 3 depths in the sampling vertical in the cross section
    - repeat during the year under different flow conditions.
    - code these measurements as follows
        - medium= WSQ (QC sample)
        - stype=B (other QA)
- Routine measurements
    - water temperature and dissolved oxygen should be measured directly in the stream (see National Field Manual Ch. 6.1 and 6.2)
    - specific conductance and pH can be measured in the stream or from an unfiltered sample from the churn splitter (see National Field Manual Ch. 6.3)

- alkalinity, bicarbonate, and carbonate concentrations are measured using a filtered sample as soon as possible after sample collection is complete, using the incremental method of alkalinity determination with the digital titrator (see National Field Manual Ch. 6.6)

Collection methods

A complete description of sample collection guidelines is provided in Chapter 4 of the National Field Manual.

- Overview of multi-vertical, depth-integrated sampling for water-quality constituents:
  - EDI method is suitable for rivers with well-defined and relatively stable discharge patterns and streambeds
  - EWI method is preferable for wadeable streams or large rivers with unstable streambeds
  - rinse all sampling and processing equipment thoroughly with river water prior to collecting the first sample
    - **IMPORTANT NOTE!!** copious field rinsing of sample equipment prior to collecting the sample is necessary to remove the methanol residue which may remain from the cleaning procedures and contaminate the DOC/PC sample
    - **THREE vigorous field rinses with at least 3-4 liters each time is REQUIRED**
  - if possible, collect samples at the same cross section throughout the period of record (may not be possible if station is wadeable during part of the year, and sampled from a bridge or cableway during the rest of the year)
  - the number of verticals should be based primarily on the requirement to collect a representative sample of the cross section, and secondarily to obtain the required sample volume
  - the vertical transit rate and operational depth for the DH-81 is a function of the stream velocity, sample-container volume, and nozzle size--

Sampler	Nozzle diameter (inches)	Ratio	Depth (feet)
DH-81	3/16	0.1	wadeable
	1/4	0.2	wadeable
	5/16	0.4	wadeable

Stream velocity x ratio = maximum vertical transit rate.

The D-96 is not subject to the same transit rate limitations of rigid bottle samplers. The minimum transit rate is one at which the sample volume does not exceed 3 L. The sampling time for the three diameter nozzles at varying stream velocities is given in USGS OFR 2005-1087 (Davis and FISP, 2005) that describes the D-96 sampler. The minimum transit rate can be calculated using the sample time from the table and the total distance to be transited.

Complete depth- and width-integrated samples are collected for suspended sediment and percent fines as part of the routine NMN sample trip. Samples may be composited in the field. (NAWQA stations also in the NMN network may use existing protocols for collecting suspended-sediment samples although percent fines should be requested as part of the analysis.)

Composite samples are collected for splitting into sub-samples for all other constituents, as described below:

- Compositing with the churn splitter
    - in order to obtain a sample for DOC/PC which is representative and comparable to other constituents, **assuming ongoing acceptable results are obtained from laboratory blank samples for DOC,** this sample will be collected using the same sampler and collection method as the other water-quality samples and composited into the churn splitter
    - A total of two to four passes are necessary at each vertical in the cross section, collected into different sets of containers. The order of sample collection for specific analytes should be consistent; the constituent groups most affected are DOC/PC and suspended sediment/chemistry (when both are collected). **It is necessary to flush the sampling equipment with the maximum volume of ambient water prior to collection of the DOC/DIC/PC sample to avoid potential contamination with methanol.** Additionally, to minimize temporal variability between the suspended sediment concentration and chemistry samples, it is necessary that they be collected as close in time as possible. For these reasons, the order of sample collection should be as follows:
        1. bottles for suspended-sediment concentration
        2. churn splitter (inorganics and DOC/PC)
        3. glass carboy (or Teflon® churn splitter) for pesticides
- Compositing with the cone splitter
    - two to three passes are necessary at each vertical in the cross section, collected into different sets of containers as follows--
        1. bottles for suspended-sediment concentration
        2. Teflon® containers for pesticides, inorganics and DOC/PC
    - **copious field rinsing of the Teflon® containers and the cone splitter is necessary to remove any residue of methanol that may remain from cleaning and contaminate the DOC/PC sample; THREE vigorous rinses with native water, at least 3-4 liters each time, is required**

Sample processing

Continue to collect DIC and UV 412nm samples

Note: Information on these analytes is currently not available in the NWQL catalog. Contact Dave Reutter or Richard Coupe if additional information is needed.

- **Dissolved Inorganic Carbon (DIC)** – Filter sample using either a 25mm glass-fiber filter or a capsule filter into a 125-ml amber glass bottle baked at 450 degree C (same bottle type used for DOC samples). **Do not rinse bottles but rinse filter with organic-free water. Do not add sulfuric acid preservative, do not leave headspace.** Chill and maintain at 4 degree C, ship immediately. If using a glass-fiber filter, it may be retained for Total Particulate Carbon or Particulate Inorganic Carbon analysis. Include 'LC 8097 DIC' on the bottle label.
- **UV 412nm** – no additional sample processing is required for this constituent as an aliquot for analysis of this constituent will be taken from the bottle used for analysis of UV254 and UV280. However, a slightly larger sample volume is required and the bottle label needs to be changed to reflect the additional constituent. Label the UAS bottle 'UAS' 'UV-254, UV-280, and UV-412'.

- **The ASR must include** – '8097' and '8096' in the Lab Code list and **'CL08013'** in the NWQL Proposal Number section. A description of bottle types required for the remaining NMN schedules can be found in the NWQL catalog located at http://nwql.cr.usgs.gov/usgs/catalog/index.cfm.

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Complete descriptions of sample processing details are provided in Chapter 5 of the National Field Manual.

- Overview of filtration guidelines:
    - **Withdraw samples for whole-water constituents first,** including nutrients and particulate carbon
    - Specifics on procedures for particulates--
        - **Particulate carbon and nitrogen**
            - a total of three filters are required to be sent to the NWQL for analysis of particulate carbon and nitrogen
            - the pressure must be regulated to less than **15** lb/in^2
            - each filter, after drying as much as possible, is carefully removed, folded in half with the sediments inside, and placed on a 6-inch square of aluminum foil; after the volume of sample that passed through the filter is recorded on the foil and in comments to NWQL on ASR, it is folded and double bagged into sealable bags
            - samples for the NWQL should be shipped as soon as possible
    - **Inorganic constituents, dissolved organic and inorganic carbon, and UV absorbance**
        - Capsule filters are used for the filtration of dissolved nutrients, major ions, and alkalinity samples as well as DOC, DIC, and UV if so desired.
        - the filter must be preconditioned (with 100 mL DIW (deionized water) if using the low-capacity capsule (Aquaprep) or the 25mm filter, or with 2 liters DIW if using the large-capacity filter), using the pump to drain the water remaining in the filter as much as possible
        - samples should be filtered according to the following order:
            - no more than 100 mL should be filtered for use in rinsing before filling bottles for major ions, dissolved nutrient analysis and alkalinity
            - **Use the Teflon® pressure-filtration assembly that holds a 25-mm glass-fiber filter for the DOC sample or use approved low or large capacity capsule-filter type. (see Office of Water Quality-Information Notes http://water.usgs.gov/usgs/owq/WaQI/index.html), particularly Note 2009.10. Do not rinse the sample bottle, filter 100 mL for DOC analysis.**
            - **Also note: if the DOC samples are filtered with the 25-mm glass-fiber filters, the lab code for the DOC sample must be changed. The lab code has changed from LC2612 to LC2613. Please delete LC2612 and add LC2613 to the lab code list in the Analytical Work Request Section of the ASR.**
            - Use the same filtration options as above for DOC. Do not rinse the sample bottle, filter 125 mL for DIC analysis. Do not add sulfuric acid preservative, do not leave headspace. Add 'LC8097' to the lab code list and ''CL08013' in the NWQL Proposal Number section. (LC8096 is for the UV-412 sample discussed below.) (an additional lab code to

differentiate between samples filtered with the 25mm filter or the capsule filter will be added at a later date.)

Because the DOC sample requires acidification (see below), a separate bottle must be filtered for UV absorption (see NWQL Policy Memo 2000.04)

- **60 mL** is sufficient volume, filtered into a baked-glass amber DOC bottle
- Label bottle "UV-254, UV-280, and UV-412". Add 'LC8096' to the lab code list.

  The UV absorption samples require a different lab code when using the 25mm glass-fiber filters. (A similar problem we have with the DOC samples).
  Note: The UV absorbing organic compounds samples at 254nm and at 280 nm are part of schedules 997/1010/1069

  If using the 25mm GFF for processing the UV samples

- On the ASR - delete LC2616 (UV 254nm) and LC2617 (UV 280nm) then add LC2614 (UV 254nm) and LC2615 (UV 280nm)

  - Stable isotopes of water
    - Fill the 60-mL bottle 2/3 full of water and put on the Polyseal cap. No preservation is needed.
    - Samples should be packaged securely and sent directly to Ty Coplen for analysis.
  - Pesticides
    - pre-rinse the filter with at least 100 mL of native water
    - collect approximately 1 L of filtered sample, without prerinsing, leaving about a 2-cm headspace in the bottle

- Preservation
  - the FA bottle for schedule 998/1201/1674 is preserved with 2 mL of 7.5-7.7 Normal Ultrex-grade nitric acid, provided in polypropylene vials (see National Field Manual Ch. 5.4 for more information). The pH of the preserved sample must be <2.
  - samples for whole-water nutrients and dissolved organic carbon are preserved with 1 mL of 4.5 normal sulfuric acid (see and NWQL Policy Memo 2000.04 for more information
  - all nutrient and organic samples are stored at 4°C
  - Record all expiration dates and lot numbers for acids.
- Shipping
  - A detailed description of shipping requirements is provided in NWQL Technical Memorandum 02.04 [http://nwql.usgs.gov/Public/tech_memos/nwql.02-04.html]
  - All sample bottles must be clearly labeled with pre-printed labels or a waterproof marker with the following minimum information--site identification number, date, time, and sample designation code
  - Particulate carbon and nitrogen 25 mm filters can be placed in a sealable bag in with ASRs as an added protection from melted ice.
  - Include a NWQL Analytical Services Request (ASR) Form for each sample sent to the laboratory, retaining a copy; place the ASR form inside a sealable plastic bag and tape the bag to the inside of the cooler lid

References

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National Water Quality Technical Memorandum 02.04, 2002, Requirements for the Proper Shipping of Samples to the National Water Quality Laboratory

Office of Surface Water Technical Memorandum 94.05, 1994, Maximum sampling depths and transit rates for suspended sediment and water-quality samplers [http://water.usgs.gov/admin/memo/SW/sw94.05.html]

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Office of Water Quality Technical Memorandum 00.05, 2000, Particulate Organic Carbon

Office of Water Quality Technical Memorandum 00.08, 2000, New Method for Particulate Carbon and Particulate Nitrogen

Office of Water Quality Technical Memorandum 02.09, 2002, Water-Quality Field Methods phase out of US D-77 and frame-type samplers

U.S. Geological Survey, variously dated, National field manual for the collection of water-quality data: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chaps. A1-A9, available online at http://pubs.water.usgs.gov/twri9A

Attachment 5. Sample Coding Guidance

Environmental Samples

Time Time of collection rounded to 10-minute interval
Sample medium WS (surface water)

If no associated QC sample:
Sample type 9 (regular)

71999 (sample purpose)	25 (NMN)
50280 (purpose of site visit)	1001 (Fixed frequency)
99111 (QC sample associated)	1 (No associated QA data)
	10 (Blank)
	30 (Replicate sample)
	40 (Spike)
	100 (More than one type of QA sample)

Also include sampler type (parameter code 84164), sampling method (parameter code 82398) and splitter type (parameter code 84171) when logging in samples. Coding for these parameters are found at: http://waterdata.usgs.gov/usa/nwis/qwdata?codes_table26_help

Blanks

(1) Analytical Services Request Forms

	Environmental	Field blank
Time	Time of collection rounded to nearest 10-minute interval	8 minutes after environmental sample time
Sample medium	WS (surface water)	OAQ (QA, artificial)
Sample type	9 (regular)	2 (blank)

Login comment: Field blank prepared before sampling; equipment last used at station ID on date; blank water lot number ____

(2) QWDATA

Parameter	Environmental sample
99111 (QA data associated w/sample)	10 (blank)
71999 (sample purpose)	25 (NMN)

	Blank sample
71999 (sample purpose)	20 (NMN)
50280 (purpose of site visit)	1098 (QC sample)
99100 (type of blank solution)	40 (Pesticide/DOC grade organic-free water)

10 (IBW/inorganic grade water)
110 (both IBW & Pest/DOC grade OFW) **Note: Please avoid collecting the inorganic blank and the organic blank samples during the same sampling trip.**

99101 (source of blank solution)	10 (NWQL)
99102 (type of blank sample)	100 (field) 150 (lab) 1 (source) 40 (sampler) 50 (splitter/tubing)

Coding of Blank Water Lot Numbers
99200 (Inorganic Grade) see list of IBW lot numbers at
http://privusgs2.er.usgs.gov/display/nawqadatasynth/complete+descriptive+version

or
99202 (Pesticide Grade) see list of OBW lot numbers at
http://privusgs2.er.usgs.gov/display/nawqadatasynth/complete+descriptive+version

Replicates (NWQL and Suspended Sediment Samples)

(1) Analytical Services Request Forms

	Primary	Secondary
Time	Time of collection rounded to nearest 10-minute interval	Ten minutes later than primary sample
Sample medium	WS (surface water)	WSQ (QA, surface water)
Sample type	7 (replicate)	7 (replicate)

(2) QWDATA

Parameter	Primary	Secondary
99111 (QA data associated w/sample)	30 (replicate)	--
99105 (replicate type)	10 (concurrent) or 30 (split)	10 (concurrent) or 30 (split)
71999 (sample purpose)	25 (NMN)	25 (NMN)

Pesticide field spikes

(1) Analytical Services Request Forms

	Environmental	Spike
Time	Time of collection rounded to nearest 10-minute interval	Three minutes after environmental sample time
Sample medium	WS (surface water)	WSQ (QA, surface water)
Sample type	9 (regular)	1 (spike)

(2) QWDATA

Parameter	Primary sample
99111 (QA data associated w/sample)	40 (spike)
99106 (spike type)	10 (field)
71999 (sample purpose)	25 (NM)

	Secondary sample
99106 (spike type)	10 (field)
99104 (lot number)	see list of spike lot numbers at http://privusgs2.er.usgs.gov/display/nawqadatasynth/complete+descriptive+version
99107 (spike source)	10 (NWQL)
99108 (spike volume)	0.1 (mL)
71999 (sample purpose)	25 (NMN)

Login comment: Field spike for S2033 for station ID on date/time;
spike lot number is _____.

Note: Normally spikes are collected during the non-growing season. If taken during the growing season, a higher spike volume may need to be added to sample.

Attachment 6. Guidelines for Collection of NMN QC Samples

The collection and evaluation of QC samples are essential elements of the NMN program, which contains a strong interpretive component. Without QC information, the quality of the environmental data cannot be evaluated or qualified, limiting the interpretive value of the data.

Objectives of the QC sample analysis include the following--

- to determine the extent of contamination introduced from the equipment and methods used for sampling and processing
- to provide information about the variability and bias of measured concentrations
- to evaluate the effect of the natural-water matrix on the bias and recovery of measured concentrations for selected analytes

QC samples for NMN include field-equipment blanks, replicates, and field-matrix spikes. Consult the NMN SWPLAN for information on the QC samples required of the individual NMN sites in FY 2010.

NMN data-check reviews

We have established a data-review process as part of the migration from the old NASQAN data management system to the NAWQA data warehouse. Data-check reviews in an Excel format will continue to be sent periodically to the WSC's NMN project chief . The NMN project chiefs will review the results flagged for their sites, make any needed changes, and document the changes in the data-check spreadsheet. Once the data-reviews have been completed, the WSC reviewer should email the reviewed data-check spreadsheet to Dave Reutter. Contact David Reutter at (614) 430-7732 or email at dreutter@usgs.gov if additional information is needed.

Field blanks

A field blank is a blank solution that is generated under actual field conditions and is subjected to the same aspects of sample collection, field processing, preservation, transportation, and laboratory handling as the environmental samples.

Field blanks should be prepared immediately before collecting and processing a native-water sample at a selected site.

- equipment should already be pre-cleaned in preparation for the routine environmental sampling
- it is not necessary to clean the equipment after sampling and processing of the field blank before using it to collect and process the environmental sample
- field blanks will be required for all constituents except suspended sediment
- use the following schedules--

Schedule	Analysis	Source solution
452	Nutrients	IBW
1674	Major ions	IBW
1675	PC/PIC/POC/DOC	OBW
2033	Pesticides	OBW

Lab Code		
8097	DIC	IBW
8096	UV 412nm	OBW

The bottle requirements for these schedules can be found from the links in the NMN Schedule Description document.

Approximately two to three gallons of blank water (either inorganic- and organic-free) are required for adequate rinsing and processing.

- because of the potential for contamination, a field blank should be prepared using either inorganic- or organic-free blank water, **but not both during the same site visit**.
- field blanks (see Chapter 4.3.1.B of the National Field Manual) for inorganic constituents (schedules 1674 and 452) should be prepared as follows:
    - collect two initial samples of the source solution one for each schedule if necessary
    - after the initial rinsing with blank water, fill the sampler bottle or bag and pour through the nozzle into a sample bottle for the sampler blank
    - pour the remainder of the blank water from the sampler into the churn splitter; refill the sampler and repeat until the churn contains about 5 liters of water; pump an aliquot of blank water from the churn splitter, using the routine pumping system, into a sample bottle for the pump blank
    - pump an aliquot of blank water from the churn splitter through the preconditioned filtration system (with 100 mL IBW if using the low-capacity capsule filter (Aquaprep) or the 25mm filter, if using the large-capacity filter 1L DIW (deionized water) followed by 1L of IBW) into a sample bottle for the equipment blanks
    - preserve all samples as required; submit only the final field equipment blank samples (filtered nutrients) to the laboratory and store the remainder of the samples for later analysis, if necessary
- field blanks for organic constituents (schedules 1675 and 2033) should be prepared as follows:
    - collect two initial samples (1-L and 125-mL) of the source solution, one for each schedule if necessary
    - **rinsing of the sampler should simulate as closely as possible the field rinsing that occurs prior to collection of the environmental sample**
    - after the initial rinsing, fill the sampler bottle or bag with blank water and pour through the nozzle into the glass carboy or Teflon® churn splitter; refill the sampler and repeat until the carboy/churn contains at least 2 to 3 liters of water; pump an aliquot of blank water from the glass carboy/churn through the preconditioned pesticide filtration system for the pesticide equipment blank
    - refill the sampler and pour another 2 to 3 liters of blank water into the churn splitter; collect three aliquots through the churn spigot and pump through the preconditioned PC filtration system for PC analysis
    - after conditioning the capsule filter (with 50 mL OBW if using the low-capacity capsule filter (Aquaprep) or the 25mm filter; if using the large-capacity filter 1L DIW followed by 1L of OBW), filter an additional 100 mL from the churn splitter and submit the filtrate for the DOC field blank

If the data from the laboratory fall within the QC requirements (described below), the field blank is acceptable and the other samples may be discarded.

- If all or some of the data exceed the acceptable levels, further investigation will be required to determine the source of the contamination.

Quality-Control Requirements

Acceptable concentrations for the equipment blank for inorganic constituents and pesticides are either less than or no higher than the MDL (minimum detection limit) + 100 percent. Concentrations higher than this must be evaluated in context with environmental data, and may still be considered acceptable.

Blanks for organic carbon are considered acceptable if concentrations are either less than or no higher than twice the MRL (0.15 mg/L). Concentrations higher than this must be evaluated in context with environmental data, and may still be considered acceptable.

Consult the sample coding guidelines document for completing ASR's and entering data into QWDATA.

Replicates and Splits

Concurrent replicates are two or more samples that are collected as closely as possible in time and space, but processed, handled, and analyzed separately; these samples provide information about the total variability inherent in the observed concentrations, including sampling, processing, and laboratory variability.

- collection of concurrent replicate samples requires two separate passes for the particular suite of constituents at each vertical in the cross section, composited in separate compositing vessels

Splits are two or more separate subsamples split from a single sample; these samples provide information about the variability inherent in the observed concentrations due to sample processing and laboratory analysis

- collection of split samples requires a single pass for the particular suite of constituents, composited in one compositing vessel; separate samples are dispensed from the compositing vessel, following the routine procedures

Consult the sample coding guidelines document for completing ASR's and entering data into QWDATA.

Field-matrix spikes

Environmental sample fortified in the field with known concentrations of all, or a representative selection of, the analytes for a particular method; this sample must be submitted along with a split sample which is not spiked

- field matrix spikes (supplied by NWQL) will be collected only for pesticides (Schedule 2033).

Detailed instructions for using the pesticide spike mixture for S2033 are provided with the spike mixture (500 microliters in a 1 mL ampule), along with 100 microliter micropipets and a Teflon® squeeze bottle

- the ampule should be kept in the freezer if long-term storage is required

Consult the sample coding guidelines document for completing ASR's and entering data into QWDATA.

The contacts for NMN regarding questions about QA/QC sampling and review of QC data are Richard Coupe (rhcoupe, 601-933-2982) or Dave Reutter (dreutter, 614-430-7732).

References

Leahy, Patrick P., 1993a, QA/QC plan for Intensive Fixed Sites, unpublished memorandum, July 8, 1993

Leahy, Patrick P., 1993b, QA/QC objectives and procedures for NAWQA SW Basic Fixed Sites, unpublished memorandum, August 17, 1993

U.S. Geological Survey, September 2006, Collection of water samples (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A4, accessed October 6, 2009 at http://pubs.water.usgs.gov/twri9A4

Zaugg, A.D., Sandstrom M.W., Smith, S.G., and Fehlberg, K.M., 1995, Methods of analysis by the USGS NWQL--determination of pesticides in water by C-18 solid-phase extraction and capillary-column gas chromatography-mass spectrometry with selected-ion monitoring: USGS Open-File Report 95-181, 49 pp.

Attachment 7. NMN Parameter Codes

Schedule	Analyte	Parameter Code
Field parameter	Temperature	00010
Field parameter	Specific conductance	00095
Field parameter	Dissolved oxygen	00300
Field parameter	pH	00400
Field parameter	Alkalinity, filtered, field	39086
998/1201	Calcium	00915
998/1201	Magnesium	00925
998/1201	Sodium	00930
998/1201	Potassium	00935
998/1201	Chloride	00940
998/1201	Sulfate	00945
998/1201	Fluoride	00950
998/1201	Silica	00955
998/1201	Arsenic	01000
998/1201	Boron	01020
998/1201	Iron	01046
998/1201	Strontium	01080
998/1201	Vanadium	01085
998/1201	Lithium	01130
998/1201	Selenium	01145
998/1201	Turbidity	63676
998/1201	Residue, 180 degrees Celsius (TDS)	70300
997/1010/1069	Nitrogen, ammonia, filtered	00608
997/1010/1069	Nitrogen, nitrite, filtered	00613
997/1010/1069	Nitrogen, ammonia + organic (Kjeldahl), filtered	00623
997/1010/1069	Nitrogen, ammonia + organic (Kjeldahl), unfiltered	00625
997/1010/1069	Nitrogen, nitrite + nitrate, filtered	00631
997/1010/1069	Phosphorus, unfiltered, total as phosphorus	00665
997/1010/1069	Phosphorus, filtered	00666
997/1010/1069	Phosphorus, phosphate, ortho, filtered	00671
997/1010/1069	Carbon, organic, filtered, recoverable (DOC)	00681
997/1010/1069	Carbon, inorganic, sediment, suspended (PIC)	00688
997/1010/1069	Carbon, organic, sediment, suspended, recoverable (POC)	00689
997/1010/1069	Carbon, inorganic + organic, sediment, suspended (PC)	00694
997/1010/1069	Total nitrogen	49570
997/1010/1069	Ultraviolet absorbing organic constituents - 254 nm	50624
997/1010/1069	Ultraviolet absorbing organic constituents - 280nm	61726
LC 8096	Ultraviolet absorbing organic constituents - 412nm	66700
LC 8097	Carbon, inorganic, filtered (DIC)	00691
SusSed	Suspended sediment, percent finer than 62 microns	70331
SusSed	Suspended sediment	80154

Schedule	Analyte	Parameter Code
2033	Terbuthylazine	04022
2033	Hexazinone	04025
2033	Simazine	04035
2033	Prometryn	04036
2033	Prometon	04037
2033	2-Chloro-4-isopropylamino-6-amino-s-triazine {CIAT}	04040
2033	Cyanazine	04041
2033	Fonofos	04095
2033	alpha-Endosulfan	34362
2033	Dicrotophos	38454
2033	Dichlorvos	38775
2033	Chlorpyrifos	38933
2033	Dieldrin	39381
2033	Metolachlor	39415
2033	Malathion	39532
2033	Diazinon	39572
2033	Atrazine	39632
2033	Alachlor	46342
2033	Acetochlor	49260
2033	1-Naphthol	49295
2033	Cyfluthrin	61585
2033	Cypermethrin	61586
2033	Endosulfan sulfate	61590
2033	Fenamiphos	61591
2033	Iprodione	61593
2033	Isofenphos	61594
2033	lambda-Cyhalothrin	61595
2033	Metalaxyl	61596
2033	Methidathion	61598
2033	Myclobutanil	61599
2033	Oxyfluorfen	61600
2033	Phosmet	61601
2033	Tefluthrin	61606
2033	Tribufos	61610
2033	2-Chloro-2,6-diethylacetanilide	61618
2033	2-Ethyl-6-methylaniline	61620
2033	3,4-Dichloroaniline	61625
2033	3,5-Dichloroaniline	61627
2033	4-Chloro-2-methylphenol	61633
2033	Azinphos-methyl-oxon	61635
2033	Chlorpyrifos, oxygen analog	61636
2033	Diazinon, oxygen analog	61638
2033	Disulfoton sulfone	61640
2033	Ethion monoxon	61644
2033	Fenamiphos sulfone	61645
2033	Fenamiphos sulfoxide	61646
2033	Malaoxon	61652
2033	Paraoxon-methyl	61664
2033	Phorate oxygen analog	61666
2033	Phosmet oxon	61668

Schedule	Analyte	Parameter Code
2033	Terbufos oxygen analog sulfone	61674
2033	Fipronil	62166
2033	Fipronil sulfide	62167
2033	Fipronil sulfone	62168
2033	Desulfinylfipronil amide	62169
2033	Desulfinylfipronil	62170
2033	Tebuconazole	62852
2033	cis-Propiconazole	79846
2033	trans-Propiconazole	79847
2033	Ethion	82346
2033	Metribuzin	82630
2033	2,6-Diethylaniline	82660
2033	Trifluralin	82661
2033	Dimethoate	82662
2033	Phorate	82664
2033	Parathion-methyl	82667
2033	EPTC	82668
2033	Tebuthiuron	82670
2033	Molinate	82671
2033	Ethoprophos	82672
2033	Benfluralin	82673
2033	Carbofuran	82674
2033	Terbufos	82675
2033	Propyzamide	82676
2033	Disulfoton	82677
2033	Propanil	82679
2033	Carbaryl	82680
2033	Thiobencarb	82681
2033	Dacthal	82682
2033	Pendimethalin	82683
2033	Propargite	82685
2033	Azinphos-methyl	82686
2033	cis-Permethrin	82687