Chapter 2
Quality Assurance

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2.1 Introduction

This chapter provides the user with quality assurance (QA) requirements and procedures for conducting environmental measurements and sampling. In order to generate analytical data of known and defensible quality for its intended use, adherence to established quality assurance protocols is necessary. This document is designed to complement, not replace or supersede, program-specific technical guidance and regulatory requirements.

The foundation of an organization's quality program is typically documented in a Quality Management Plan (QMP). The QMP is viewed as the over-arching, 'umbrella' document which describes the quality policies, procedures, and applicable areas within the organization as well as the roles, responsibilities, and authorities of its members in planning, implementing, and assessing the degree to which the QMP is being followed. The QMP also includes the NJDEP QA management structure and contact information by relevant program for questions related to QA. Quality requirements for non- United States Environmental Protection Agency (USEPA) organizations that receive funds for work involving environmental data are specified in the Code of Federal Regulations for each type of extramural agreement. The USEPA issues documents to provide further information on satisfying the quality requirements. The current version of the USEPA QMP requirements is available at: https://www.epa.gov/quality/epa-qar-2-epa-requirements-quality-management-plans.

The New Jersey Department of Environmental Protection (NJDEP) prepares and maintains a QMP as required by the USEPA. The preparation and administration of the QMP is assigned to NJDEP's Office of Quality Assurance (OQA). The QMP is approved/signed by the NJDEP Commissioner, the NJDEP Quality Assurance Manager, all NJDEP Senior Staff, and the USEPA Region 2 Quality Assurance Manager. The QMP is valid for 5 years from the official date of publication. After this time period, USEPA requires the QMP be reissued or withdrawn from use by the NJDEP. The NJDEP's current QMP is available at: https://www.nj.gov/dep/enforcement/oqa/qap.html.

On a project level, Quality Assurance and Quality Control (QA/QC) measures applicable to sampling and analytical processes are documented in a Quality Assurance Project Plan (QAPP). Monitoring projects for the Clean Water Act Program, Safe Drinking Water Act Program, Resource Conservation Recovery Act (RCRA) Program, and Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Program, and state programs are based on the approved QAPP. Elements and details contained within a project's QAPP are based on a graded approach. That is, the level of detail will vary depending on the nature of the work being performed and the intended use of the data generated. Plans are prepared by using a variety of standard and guidance references including, but not limited to, USEPA's Quality Assurance Project Plan Standard and its related Guidance document. The most current version of these documents are available at: https://www.epa.gov/quality/epa-qar-5-epa-requirements-quality-assurance-project-plans and https://www.epa.gov/quality/guidance-quality-assurance-project-plans-epa-qag-5, respectively. Additional guidance documents may be found at the USEPA website: https://www.epa.gov/quality/agency-wide-quality-program-documents. Quality assurance measures coupled with a prescribed sampling plan will improve sample collection while maintaining the integrity of the samples prior to analysis. It is important to include the analytical laboratory staff in the QAPP development process to ensure appropriate laboratory QA/QC measures are implemented during analysis and the deliverables produced are documented and appropriate for the decision being made.

For site remediation projects, selection and application of site-appropriate data quality levels should be discussed in the Quality Assurance Project Plan (QAPP). This document presents the organization, functional activities, and specific QA/QC activities needed to attain specific project goals and data quality objectives. A

QAPP is required per the Technical Requirements for Site Remediation (N.J.A.C. 7:26E-2.2) for all remedial activities for which data are generated. To formulate a QAPP for a remediation case, see the Quality Assurance Project Plan Technical Guidance at: https://www.state.nj.us/dep/srp/guidance/#analytic_methods.

For permit compliance sampling, a quality control program is often necessary to assure analytical accuracy and sensitivity to demonstrate compliance. Permits may require a permittee to achieve specific reporting limits when monitoring for pollutants. In 2014, USEPA amended the Clean Water Act (CWA) regulations to codify that under the National Pollutant Discharge Elimination System (NPDES) program, permit applicants and holders of permits must use sufficiently sensitive analytical test methods when completing an NPDES permit application and when submitting permit compliance monitoring data. The final rule clarified existing EPA regulations, codified existing EPA guidance on the use of sufficiently sensitive analytical methods with respect to measurement of mercury, and extends the approach outlined in that guidance to the NPDES program more generally. Specifically, USEPA modified existing NPDES application, compliance monitoring, and analytical methods regulations.

Standard Operating Procedures (SOPs) for sample collection, handling, and analysis are examples of elements that must be included in the QAPP. SOP requirements are established to maintain consistency in performance of sample collection and handling activities.[1]

Finally, this chapter highlights additional elements required in a QAPP, such as sample containers, decontamination procedures, quality control samples, preservation and holding time requirements. All aspects or scenarios are not discussed herein. The "site specific" nature of sampling makes it incumbent upon investigators to document in the QAPP any known unique feature that may contribute or impart a bias to data quality and what steps, if any, will be taken to address those specific conditions.

The NJDEP Site Remediation and Waste Management Program maintains a library of guidance manuals on its website at https://www.nj.gov/dep/srp/guidance/. It is recommended that the reader access the website and review the guidance documents pertinent to the respective task. Additional guidance may also be found at websites of the USEPA and the American Society for Testing and Materials (ASTM). Examples of some of the relevant guidance manuals and webpages pertaining to this chapter are:

Soil SI/RI/RA Technical Guidance: https://www.nj.gov/dep/srp/guidance/#si_ri_ra_soils;

Ground Water SI/RI/RA Technical Guidance: https://www.nj.gov/dep/srp/guidance/#pa_si_ri_gw;

Ecological Evaluation Technical Guidance: https://www.nj.gov/dep/srp/guidance/#eco_eval;

Vapor Intrusion Technical Guidance: https://www.nj.gov/dep/srp/guidance/#vi; and

Occupational Safety and Health Administration (OSHA): https://www.osha.gov/shpguidelines/index.html.

The following guidance documents are available at: https://www.nj.gov/dep/srp/guidance/#analytic_methods.

- Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance
- Quality Assurance Project Plan Technical Guidance
- Data of Known Quality Protocols Technical Guidance
- Data Quality Assessment and Data Usability Evaluation Technical Guidance

[1] National Pollutant Discharge Elimination System (NPDES): Use of Sufficiently Sensitive Test Methods for Permit Applications and Reporting (40 CFR Parts 122 and 136) publ. in Federal Register /Vol. 79, No. 160 /Tuesday, August 19, 2014 https://www.gpo.gov/fdsys/pkg/FR-2014-08-19/pdf/2014-19265.pdf

2.2 Certifications

For permit compliance sampling, a quality control program is often necessary to assure analytic accuracy to demonstrate compliance. Permits may require a permittee to achieve specific reporting limits or use specific analytical methods when monitoring for pollutants. Pollutants may need to be reduced or eliminated if they exceed specific concentrations. Those concentration levels may not be achievable unless proper quality control methods are implemented. The use of sufficiently sensitive test methods is required to comply with permit limits in accordance with the National Pollutant Discharge Elimination System (NPDES).

Pursuant to *Regulations Governing the Certification of Laboratories and Environmental Measurements* N.J.A.C. 7:18 (Laboratory Certification Rule), available at: https://www.nj.gov/dep/rules/rules/njac7_18.pdf, the OQA requires that field environmental measurements and environmental laboratories submitting analytical data to the NJDEP, regardless of quality level, must be certified. Environmental laboratory is defined as any laboratory, facility, consulting firm, local government or private agency, business entity or other person that the NJDEP has authorized to generate analytical data.

The certification status of the laboratory should be determined prior to submitting environmental samples to a laboratory for analysis. Laboratories submitting analytical data to the State of New Jersey must hold current certification where applicable under the Laboratory Certification Rule, N.J.A.C. 7:18. The certification status of laboratories is available at: https://www.nj.gov/dep/enforcement/oqa/certlabs.htm.

Two options for laboratory certification are offered by the OQA, including:

1. Certification through the state program, referred to as the Environmental Laboratory Certification Program (ELCP).
2. The National Environmental Laboratory Accreditation Program (NELAP) which offers certification based upon nationwide criteria that use a quality program approach to ensure the integrity of analytical data.

OQA offers certification in the following categories:

- Drinking Water Program
- Water Pollution Program
- Radon/Radon Progeny in Air
- Solid and Hazardous Waste Programs
- CERCLA- Contract Laboratory Program (CLP) Programs
- Air Methods
- Radiological Parameters other than Air

2.2.1 Field Environmental Measurements Certifications

For measurements collected in the field and submitted to the NJDEP to support regulatory compliance decision-making, the OQA requires certification for "Analyze Immediately" and other field parameters through the Laboratory Certification Rule, N.J.A.C. 7:18, unless a specific NJDEP regulation allows for measurement by a non-certified entity. Refer to: https://www.nj.gov/dep/enforcement/oqa/labcert.html.

This requirement applies to both measurements that occur in a conventional laboratory environment as well as measurements that take place in the field. Organizations that collect environmental samples by performing low flow purging and sampling are required to have the applicable certification to analyze for the field parameters that are required as part of this procedure. These water quality indicator parameters

measured in the field include pH, specific conductance, turbidity, temperature, dissolved oxygen, and ORP/Eh.

Certification for field measurement of water quality indicator parameters pursuant to N.J.A.C. 7:18 is obtained through a process that includes an application submitted to NJDEP Office of Quality Assurance, proficiency test samples, reviews of demonstration of capability data and on-site assessments conducted by NJDEP OQA. Field measurement instrument maintenance and calibration records must be maintained by the certified company. Only a company that has successfully completed the process and that has been granted certification can perform environmental field measurements of water quality indicator parameters and provide analytical data for these parameters for the purpose of establishing compliance with any NJDEP program. Additionally, when performing environmental field measurements, a company shall use only those analytical methods for which it has been granted certification by NJDEP OQA.

A company not having required certifications and attempting to provide analytical data in response to a NJDEP program will result in enforcement actions that can include suspensions, revocations, and fines. Regardless of whether the equipment in question is rented or privately owned the requirement for certification cannot be ignored. All certification documentation must accompany the instrument into the field and accompany all water quality indicator parameter data submitted to the NJDEP. Field instrument calibration and data reporting forms are found in the NJDEP Low Flow Purging and Sampling Guidance at:

https://www.nj.gov/dep/srp/guidance/lowflow/

https://www.nj.gov/dep/srp/guidance/lowflow/lfcalibrate.pdf

https://www.nj.gov/dep/srp/guidance/lowflow/lfdatasheet.pdf

NJDEP defines an environmental laboratory as any laboratory, facility, consulting firm, government or private agency, business entity or other person that the NJDEP has authorized, pursuant to N.J.A.C. 7:18, to perform analysis in accordance with the procedures of a given analytical method using a particular technique as set forth in a certain methods reference document and to report the results from the analysis of environmental samples in compliance with a NJDEP regulatory program.

Additionally, immunoassay field methods require certification pursuant to N.J.A.C. 7:18 under the Solid and Hazardous Waste Program category. Regardless of whether a company or organization is considered a true laboratory, certification is required when reporting data for compliance purposes. This includes but is not limited to responsible parties, contractors, and facilities. For additional information regarding laboratory, immunoassay, and field instrument certification requirements see the OQA website at: https://www.nj.gov/dep/enforcement/oqa.html.

2.3 Data Quality Objectives

Selection and application of site-appropriate Data Quality Objectives (DQO) should be discussed in the QAPP. Refer to *Guidance on Systematic Planning using the Data Quality Objectives (DQO) Process* available at: https://www.epa.gov/quality/agency-wide-quality-program-documents. To develop reliable site investigation data for NJDEP publicly-funded CERCLA (Superfund), or non-Superfund publicly-funded sites, the primary consultant/contractor has the responsibility to develop and implement the QAPP. This document must present the organization, functional activities, and specific QA/QC activities needed to attain specific project goals and data quality objectives. DQOs, developed by the investigator, are qualitative and quantitative statements derived from the DQO planning process that clarify the purpose of the study, define the most appropriate type of information to collect, determine the most appropriate conditions from which to collect that information, and specify tolerable levels of potential decision errors and uncertainty.

Any sampling conducted by remediation professionals and state contract vendors, including sampling associated with removal actions or operations and maintenance contracts, requires the development and implementation of a QAPP. The NJDEP will approve these plans prior to implementation by a state contractor. Requirements for these plans are generally specified in state contracts and NJDEP program technical guidance. Regardless of a document's title or "deliverable" name (e.g., QAPP), NJDEP requires these plans for all sampling events that are conducted in the state, pursuant to N.J.A.C. 7:26E. It is recommended that these plans be contained in a stand-alone document.

For permit compliance sampling, a quality assurance program is necessary to assure analytical accuracy sufficient to demonstrate compliance.

Sample analytical data that undergo data validation by the NJDEP or the remediation professional or data validator must be generated as a laboratory full data deliverable, pursuant to N.J.A.C. 7:26E-2. Other programs may also have specific data deliverable requirements and subsequent evaluation and validation of these data. For example, drinking water compliance monitoring data submitted to the NJDEP Bureau of Safe Drinking Water must be uploaded in a specific electronic data deliverable (EDD) format to the NJ E2 platform. These data must be generated by the analytical laboratory using published EPA drinking water methods and reported in accordance with the NJDEP BSDW guidelines contained in the E2 Quick Reference Guide (which is updated periodically).

2.3.1 Laboratory Analytical Methods

The procedures established to control the collection and handling of samples are an integral part of the Quality Assurance Program operating within NJDEP. The importance of a controlled environmental sample collection process and analytical data protocol is demonstrated through integration of this information into the decision-making process. All phases of this process rely on the provision of accurate, precise, comparable, and complete analytical data.

Current requirements for samples collected under the Safe Drinking Water Act and the Clean Water Act (chemical, physical, and biological components of wastewater and other environmental samples) are published annually in 40 CFR Parts 136, 141, and 143, and are available from the Government Printing Office at: https://ecfr.io/Title-40/cfrv25#0. Methods for marine and freshwater biological monitoring that are not in 40 CFR are listed herein, refer to Appendices 2.4 and 2.5.

Current guidelines for samples to be analyzed in accordance with the USEPA SW846 Methods are published in the latest final update of the USEPA SW846 *Test Methods for Evaluating Solid Waste – Physical and Chemical Methods*. They may be found on USEPA's website at https://www.epa.gov/hw-sw846.

The analytical methods promulgated under Clean Water Act section 304(h) are sometimes referred to as the "304(h)" or "Part 136" methods. The methods measure chemical and biological pollutants in media, such as wastewater, ambient water, sediment, and biosolids (sewage sludge). In addition to 40 CFR Part 136 methods, some approved industry-specific methods are published or incorporated by reference at 40 CFR Parts 401 through 503. Approved methods can be found at: https://www.epa.gov/cwa-methods/approved-cwa-chemical-test-methods.

Current requirements for samples to be analyzed in accordance with the USEPA Contract Laboratory Program are published in the latest version of the USEPA CLP Statements of Work (SOWs). These documents are found on the USEPA website at https://www.epa.gov/clp.

The following quality assurance requirements have been established to maintain sample integrity. Their primary objectives are to maintain the physical form and chemical composition of the sample and to prevent contamination from other sources or changes in contaminant concentration. To meet these objectives, there should be a measure of control over all sample-handling procedures beginning with

sample container cleaning procedures and ending with laboratory analysis.

2.3.2 Field Screening Methods

Field screening methods allow for the performance of rapid characterization via a dynamic sampling plan. They can provide qualitative data to meet the predetermined DQO, providing that supporting QA/QC procedures are in place.

For projects utilizing screening or semi-quantitative data during the field screening investigations, tools such as headspace gas chromatography (GC) can be simple and fast for the analysis of VOCs in soil and water samples during underground storage tank removal or well installation and monitoring. Enzyme kits can provide rapid detection for a variety of parameters, such as polychlorinated biphenyls (PCBs) or explosives during site characterization.

The field screening data should be of known quality, with respect to measurement precision or reproducibility, accuracy, sensitivity, and correlate with the standard laboratory methods. Several factors to be considered before mobilization include, but not limited to:

- Action levels for field decisions as part of the DQOs
- Screening and semi-quantitative data in addition to quantitative data to meet DQO
- Percentage of samples to be analyzed in the field as well as sent off-site for laboratory confirmation
- Methodology to compare field and laboratory data, such as duplicates, proficiency testing samples, and calibration measurements
- Analytical method, the measurement selectivity, sensitivity, precision, accuracy, representativeness, and action levels for the field instrument
- Potential matrix interference that might be associated with a particular field technology
- The field technician performing the analyses should be trained and demonstrate competence

2.4 Sample Containers and Sample Preservation Requirements

Prior to the collection of a sample, consideration should be given to the type of container that will be used to store and transport the sample. The entity requesting the analysis is responsible for requesting the proper sample containers or providing the laboratory with an accurate description of the matrix being sampled to enable the laboratory to provide the correct quantity and type of sample container. Selection is based on the sample matrix, potential contaminants to be encountered, analytical methods requested, and the laboratory's internal quality assurance requirements. Selection of appropriate sample containers should also be based upon review of the criteria listed below, as well as the information provided in the analytical methods, the tables at the end of this chapter, and the NJ Laboratory Certification Regulations Subchapter 9. See: https://www.nj.gov/dep/enforcement/oqa/labcert.html.

Sample collection, preservation and holding times for New Jersey certified parameters and methods are listed in the Laboratory Certification Rule, N.J.A.C. 7:18. However, for samples being analyzed to achieve compliance with the Safe Drinking Water Act and the Clean Water Act, refer to the latest Code of Federal Regulations, specifically 40 CFR Parts 141 and 136 respectively. Changes to the USEPA SW846 Methods are issued by the USEPA Office of Solid Waste (OSW) and are not final until adopted by Federal Regulations.

Additional information may be found in Appendices 2.1 to 2.6 of this chapter.

2.4.1 Reactivity of Container Material with Sample

Choosing the proper composition of sample containers will help to ensure that the chemical and physical integrity of the sample is maintained. For sampling potentially hazardous material, glass is the recommended container type because it is chemically inert to most substances. Plastic containers are not recommended for most hazardous wastes because contaminants may adsorb to the surface of the plastic, solvents may degrade the plastic, or plasticizers may leach into the sample.

In some instances, the sample characteristics or analytes of interest may dictate that plastic containers be used instead of glass because some metals species will adhere to the sides of glass containers in an aqueous matrix. However, the methodology being used for the sample analysis should always be reviewed first to determine the required bottle type. For example, USEPA Method 1631 *Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry* requires the use of either fluoropolymer bottles with fluoropolymer or fluoropolymer-lined caps, or borosilicate glass bottles. Polyethylene bottles are prohibited under this method.

In the case of a strong alkali waste or hydrofluoric solution, plastic containers may be more suitable because glass containers may be etched by these compounds creating adsorptive sites on the container surface. Only plastic containers should be used when the determinations of boron and silica are critical. Prior to ordering bottles from the laboratory, the method requirements should always be reviewed with the laboratory.

2.4.2 Sample Volume

The analytical method and the sample matrix and in the case of drinking water compliance samples, the federal regulations for the parameter being analyzed, will dictate the volume of sample to be collected. The sampler should supply sufficient volume of sample matrix for the laboratory to perform the required analysis and to meet project reporting limits. In most cases, the methodology dictates the volume of sample material (including sets of containers) required to conduct the analysis. Individual labs may provide larger volume containers for various analytes to ensure sufficient quantities for replicates or other quality control checks. However, if the expected concentrations in the sample are significant, such as in waste samples, the sample volume required by the laboratory may be less, to minimize the hazardous waste disposal problems. Reduced volume size may also be allowable based on new technologies that allow for a smaller sample size.

2.4.3 Color of Container

The analytical method can dictate the color of the sample container. Whenever possible, amber glass containers should be used to prevent photodegradation of the sample, except when samples are being collected for metals analysis. Containers used for metals analysis can be opaque or colorless, unless the sample will be analyzed for Silver or any other element which may be affected by light. If amber containers are not available, containers should be protected from light at all times when practical during shipping and handling. Laboratories often provide clear glass 40 ml vials for volatile organic aqueous analysis so that any air bubbles in the sample can be easily detected.

2.4.4 Container Closures

Container closures may be specified by method. Container closures should form a leakproof seal (e.g., screw caps or ground glass stoppers). Closures should be constructed of a material that is inert with respect to the sampled material as specified by the method. Alternatively, the closure may be separated from the sample by a closure liner that is inert to the sample material. Nothing should be added to ground glass stoppers to facilitate opening.

2.4.5 Sample Container Quality

Single-use containers with certification documentation should be used for trace and low-level analytes. These new precleaned commercial containers are batch-certified with appropriate documentation. The container type and preparation are dictated by the specific analysis to be performed on the sample.

2.4.6 Chain of Custody

To prepare for transport and/or shipment, the sample bottles should be accompanied by a hard copy of the chain of custody. In addition, custody of the cooler should be maintained. The completed and signed chain of custody form should accompany the bottles onsite during sample collection, and transportation back to the laboratory. This assures that the sample containers were not tampered with between preparation and arrival in the field. After sample collection, the bottles should be placed into the sample shipment container or cooler. Once the sample set is ready for transfer back to the analytical laboratory, the chain of custody must be signed manually by each party who takes control of the sample set. Refer to Chapter 11, *Documentation*, for additional information on chain of custody. Contact the laboratory provider for questions about the documentation.

2.4.7 Sample Bottle Storage and Transport

No matter where the sample bottles are, whether at the lab waiting to be packed for shipment or in the field waiting to be filled with sample, care should be taken to avoid contamination. Sample shuttles or coolers, and sample bottles must be stored and transported in clean environments. Sample bottles and clean sampling equipment must never be stored near running vehicular exhaust pipes, solvents, gasoline, or other materials that are potential sources of contamination. When under chain of custody, sample bottles must be in a secured area or in the presence of authorized personnel. Sample bottle storage on site should be kept to a minimum to avoid extraneous contamination.

The analytical methods specify minimum or maximum sample temperatures that are required to be met during transport of the samples to the field, while onsite, during shipment to the lab, and upon receipt of the samples at the laboratory.

2.4.8 Sample Preservation Requirements

Certain analytical methodologies for specific analytes require chemical additives in order to stabilize and maintain sample integrity and may include field filtration. Generally, this is accomplished under three scenarios:

- Preservative may be added to the sampling bottles by the laboratory prior to shipment into the field. This is preferred.
- Preservatives may be added in the field immediately after the samples are collected (i.e., within 15 minutes).
- Preservatives may also be added to the sample bottles after sample collection and upon arrival at the laboratory, for example, for unfiltered samples collected for dissolved metals.

Many laboratories provide pre-preserved bottles as a matter of convenience and to ensure that samples are preserved immediately upon collection. A problem associated with this method arises if insufficient sample volume is collected, resulting in excess preservative in the sample. More commonly encountered problems with this method include the possibility of insufficient preservative provided to achieve the desired pH level or the need for additional preservation due to chemical reactions caused by the addition of sample liquids to pre-preserved bottles. The use of pre-preserved bottles is acceptable. It is recommended that field-sampling teams check the pH and be prepared to add additional preservatives to samples if necessary.

When samples are preserved after collection, special care should be taken. The transportation and handling of concentrated acids into the field requires additional preparation and adherence to appropriate preservation procedures. The analytical methods should be reviewed to determine the correct grade of acid that are required for preservation. Please refer to Chapter 6 for greater detail.

The following guidelines are recommended to achieve safe and accurate preservation and transportation of samples in the field:

- Sampling teams should be properly equipped to conduct sample processing/preservation activities in the field (away from vehicle or other machinery exhaust). To accomplish this task the following items are necessary:
  - Graduated/Auto pipettes
  - Pipette bulbs
  - Preservatives of known quality with their content, concentration, and expiration date clearly labeled
  - Limited range pH paper (important that the sampler notes the "use by" date and that the paper is properly stored and maintained)
  - Carrying case clearly labeled and constructed of appropriate material to facilitate safe transportation of preservatives in vehicles and in the field
- Sampling teams should also be properly equipped with appropriate health and safety equipment. Below are a few examples of items that should be onsite:
  - Protective goggles
  - Disposable gloves
  - Lab apron
  - First aid kit
  - Portable eye wash station or eye wash bottle
  - Tap water for immediate flushing if spillage occurs onto clothing
- A level surface area should be designated to conduct preservation activities. A clean sheet of plastic sheeting should be placed over the area and secured.
- Personnel assigned to conduct preservation activities should be familiar with specified preservation requirements and verify that the necessary pH level has been achieved.
- Preservation requirements are method and parameter specific. Additional information may be found in Appendices 2.1 and 2.2 of this chapter. These charts may indicate any additional preservation required upon arrival of samples at the laboratory as cited in the specific methodologies. Under the Laboratory Certification Rule, N.J.A.C. 7:18, the laboratory and the samplers are required to comply with any additional preservation requirements. The source of preservatives is also of concern. Preservatives may be provided in bulk by the laboratory performing the analysis or purchased from a commercial laboratory supply vendor. All preservative containers must be labeled with respect to contents, concentration, laboratory grade, and the date of purchase or preparation. Again, under no circumstances should the test sample aliquot be returned into the container retaining the sample for analysis.
- For total metals analysis, preservation should take place prior to analysis of the sample. For dissolved metals analysis, preservation is conducted after filtration. Note that filtered samples are not acceptable for comparison to the Ground Water Quality Standards (N.J.A.C 7:9C).
- In rare cases, a chemical reaction between the preservative and an aqueous sample may induce

effervescence. If this is observed during sample collection, immediately notify the laboratory before continuing. A decision will have to be rendered in the field regarding whether to continue sample collection. If expeditious shipping and laboratory analysis of an unpreserved sample can be negotiated (based on analytical method requirements), to maintain sample integrity, the sample should be discarded, the interior of the sample container rinsed at least two times with the sample source, and an unpreserved sample volume collected. Make note in the chain of custody if the sample is unpreserved and why it was unpreserved. The laboratory must be notified that an unpreserved sample is being submitted.

- If a soil sample reacts with a required preservative, a new sample bottle or sampling device is required, an unpreserved sample must be submitted to the laboratory, and the laboratory must be notified that an unpreserved field sample is being submitted. For example, some methods, such as USEPA Method 5035, specify that if an unpreserved sample is submitted, it must be analyzed within 7 days of sample collection. Therefore, it is important that field personnel document the preservation status of samples submitted for analysis on the chain of custody.
- Samples should be placed into a cooler and maintained at 4°C immediately upon collection and preservation. Samples shall never be frozen unless specifically allowed by the analytical method.

Note: Certain methods, such as USEPA Method 1631 and 1630 (methyl mercury), allow samples to be optionally preserved at the laboratory, provided they are received at the laboratory within 48 hours of sample collection.

Information on required holding times can be found at the following USEPA websites:

- Requirements for samples collected under the Safe Drinking Water Act and the Clean Water Act are published annually in 40 CFR Parts 136 and 141 and may be found on the USEPA https://ecfr.io/Title-40/cfrv25#0.
- Guidelines for samples to be analyzed in accordance with the USEPA SW846 Methods are published in the latest final update of the USEPA SW846 *Test Methods for Evaluating Solid Waste - Physical and Chemical Methods 3rd Edition* issued 1996 and amended and may be found on the USEPA website at: https://www.epa.gov/hw-sw846.
- Requirements for samples to be analyzed in accordance with the USEPA Contract Laboratory Program are published in the latest version of the USEPA CLP SOWs. These documents may be found on the USEPA website at: https://www.epa.gov/clp.

2.5 Decontamination Procedures

An important aspect of quality control is the decontamination of field sampling equipment. Improperly cleaned and prepared sampling equipment can lead to misinterpretation of environmental data due to interference caused by cross-contamination.

In addition, sampling equipment left in-situ for purposes of obtaining multiple samples over a period of time (e.g., periodic sampling for permit compliance) will often need to be cleared of accumulated contaminants, silt, soot, dust, etc. This will assure that the samples are free of such material which may have accumulated on the sampling equipment between uses. See Chapter 5 for more information on equipment decontamination procedures.

2.6 Quality Control Samples

Quality Control (QC) samples are intended to provide control over the collection of environmental measurements and subsequent validation, review, and interpretation of generated analytical data. The various types of blank samples are designed to address QC concerns related to sample bottle and equipment preparation, packaging, handling, preservative purity, and sample collection techniques. Common terms used to reference various QC blank samples include trip blanks, field blanks, field rinsate blanks, equipment blanks, and field reagent blanks. Some of these terms may be interchanged based on definitions found in specific analytical methods. Clear definitions of these terms should be provided in the site specific QAPP.

For this manual, the definitions of trip blanks and field blanks are described below. Additional quality control samples are described in Appendix 2.6 of this chapter. A detailed description of the various types of quality control blanks is also available in a USEPA Fact Sheet at: https://www.epa.gov/sites/default/files/2015-06/documents/blanks.pdf.

Trip blanks are prepared by the laboratory and are used to measure possible cross-contamination of samples during shipping to and from the site. The analysis is typically for volatile organics and only when environmental samples are of an aqueous matrix. For aqueous sampling, the trip blank water should be from the same source as the method blank water used in the laboratory during analysis. Trip blanks are never opened outside of the analytical laboratory and travel to and from the site with the empty or full sample bottles to simulate sample-handling conditions. Contaminated trip blanks could also indicate inadequate bottle cleaning or blank water of questionable quality.

The primary purpose of the trip blank is to detect additional sources of contamination that might potentially influence contaminant values reported in actual samples both quantitatively and qualitatively. Potential sources of contamination include:

- Laboratory reagent water
- Sample containers
- Cross contamination in shipment, bottle handling and storage
- Ambient air or contact with analytical instrumentation during preparation and analysis at the laboratory
- Laboratory reagents used in analytical procedures

The purpose of a field blank is to place a mechanism of control on sample equipment and its related handling, preparation, storage, and shipment. The field blank water travels and is stored with the sample bottles, therefore, it is also representative of bottle shipment effects on sample quality. The field blank water should be from the same source as the method blank water used in the laboratory. By being opened in the field and transferred over a decontaminated sampling device (when applicable), the field blank is indicative of ambient conditions and/or equipment conditions that may potentially affect the quality of the associated samples.

A field blank also provides an additional check on possible sources of contamination beyond that which is intended for trip blanks. This includes potential contamination from ambient air as well as from sampling instruments used to collect and transfer samples from point of collection into sample containers.

The following is a breakdown by matrix of blank samples.

2.6.1 Aqueous Matrix

2.6.1.1 Field Blanks

2.6.1.1.1 Description

The performance requirement for field blank collection begins with two sets of identical bottles (method dependent). One set of bottles is filled with demonstrated analyte free water provided by the laboratory performing the sample analysis, while the second set of bottles is empty. The bottles should also be identical to those provided for aqueous sample collection. Field blanks must be analyzed for all the same parameters that the collected samples will be analyzed for. At the field location, in an area suspected to be contaminated, the water is passed from the full set of bottles through the dedicated or field decontaminated sampling device(s) and into the empty set of bottles. This will constitute identical bottle to bottle transfer. Field blanks must be preserved in the same manner as samples. On a site-specific basis, field QC sample requirements may be amended as documented in the project plans. Field blanks are generally not required for potable well sampling events or when a sample is collected directly from a source into a sampling container. However, field blanks may be required to detect cross contamination from ambient air during potable sampling events if known sources of contamination are in proximity or monitoring instruments indicate the presence of contamination above background levels.

2.6.1.1.2 Frequency

Field blanks for the aqueous matrix should be performed at a minimum rate of 5% per analytical parameter per sampling procedure. For sampling events lasting more than one day, field blanks associated with an aqueous matrix should be collected at a minimum of one per day. On a site-specific basis, QA field QC frequency requirements may be amended.

2.6.1.2 Trip Blanks

2.6.1.2.1 Description

Trip blanks are only required for aqueous sampling events, for volatile organic parameters, pursuant to the specific analytic method. Trip blanks are prepared by the laboratory, accompany sample bottles into the field, and are returned to the laboratory along with the collected samples for analysis. These bottles are never opened outside of the analytical laboratory. Trip blanks must be returned to the laboratory with the same set of bottles they accompanied to the field.

The trip blank is primarily used to measure possible cross-contamination of samples during shipping to and from the site. The QAPP should discuss the collection of trip blanks.

The primary purpose of this type of blank is to detect additional sources of contamination that might potentially influence contaminant values reported in actual samples both quantitatively and qualitatively. Potential sources of contamination may include:

- Laboratory reagent water
- Sample containers
- Cross contamination in shipment, bottle handling and storage
- Ambient air or contact with analytical instrumentation during preparation and analysis at the laboratory
- Laboratory reagents used in analytical procedures

2.6.1.2.2 Frequency

When sampling for volatile organic compounds, it is recommended trip blanks be included at a rate of one per sample shipment. However, USEPA has issued analytical methods that require additional trip blanks for each batch of twenty samples submitted to the CLP laboratory. Therefore, the analytical methods and data quality objectives should be evaluated to determine the required number and frequency of trip blanks. This information should be documented in a site-specific QAPP.

2.6.2 Non-Aqueous Matrix

2.6.2.1 Field Blanks

2.6.2.1.1 Description

The performance requirement for field blank collection begins with two sets of identical bottles (method dependent). One set of bottles is filled with demonstrated analyte free water provided by the laboratory performing the sample analysis, while the second set of bottles is empty. The bottles should also be identical to those provided for aqueous sample collection. For soil preserved in methanol, see Chapter 6 *Sample Collection*, Section 6.2.7.4 *Closed-System Vials, Chemical Preservation – Methanol* for more discussion on methanol preserved soil collection. At the field location, in an area suspected to be contaminated, the water is passed from the full set of bottles through the dedicated or field decontaminated sampling device(s) and into the empty set of bottles. This will constitute identical bottle to bottle transfer. Field blanks must be preserved in the same manner as samples. On a site-specific basis, field QC sample requirements may be amended as documented in the project plans.

2.6.2.1.2 Frequency

Field blanks for the non-aqueous matrix should be performed at a rate of one per day. For sampling events lasting more than one day, field blanks associated with a non-aqueous matrix should be collected at a minimum of one per day. On a site-specific basis, field QC frequency requirements may be amended.

2.6.2.2 Trip Blanks

Trip blanks are not required for the non-aqueous matrix unless specifically requested for by Special Analytical Services (SAS) consideration or when specifically required by the analytical method.

2.6.3 Air Matrix

Quality control blank sample collection and handling utilized during air sampling events should follow the specifications of the analytical method and/or applicable guidance document. Additional information on quality assurance/quality control of air emissions measurements is available at:

Stack Emissions: https://www.nj.gov/dep/bts/index.html

Vapor Intrusion guidance: https://www.nj.gov/dep/srp/guidance/#vi

Bureau of Stationary Sources: https://www.state.nj.us/dep/aqpp/

2.6.4 Blank Water Quality

Demonstrated analyte free water used in the field and trip blanks must originate from one common source and physical location within the laboratory and must be the same as the method blank water used by the

laboratory performing the specific analysis. The use of commercially prepared water or water not originating from the laboratory analyzing the samples is generally not permitted. An exception to this requirement is allowable if:

- It is the same water used for method blank analysis;
- The laboratory has analyzed that water and generated data from a specific batch/lot of containers; or,
- The blank sample is drawn from an unopened container from the same batch/lot thus documenting the water is free of contaminants (demonstrated analyte free).

For some regulatory programs, the source of field blank water may be different from the source of the trip blank water, however, it must also be demonstrated as analyte free water.

Laboratory certification requirements for the source of blank/method water can be found in *Regulations Governing the Certification of Laboratories and Environmental Measurements* at N.J.A.C. 7:18-3.3. Pursuant to N.J.A.C. 7:18, a source of water which meets the required standards of quality for each type of testing shall be available for use in the preparation of reagents, standards and for glassware rinsing. The laboratory shall maintain testing documentation on analyte-free water.

2.6.5 QC Sample Management and Holding Times

2.6.5.1 Sample Management

Blanks and other QC samples should be maintained in a similar manner as the environmental sample containers during shipment, storage on site, and return to the laboratory. Blanks and QC sample storage on-site should be kept to a minimum to avoid extraneous contamination and to ensure holding times are met. The Site Remediation and Waste Management Program recommends that quality control samples not be held on site in excess of 4 days.

An exception applies to storm water sampling events. The spontaneity of storm conditions precludes any possibility for preplanning sample shipment. Therefore, due to logistical constraints, trip and field blanks are not normally required. While this exception is understandable, the storage of these samples must be carefully controlled to prevent the possibility of cross contamination or degradation.

2.6.5.2 Holding Times

Holding times for individual parameters are dictated by the federal regulations and specified in the analytical method being used or under the regulated program.

Current requirements for samples collected under the Safe Drinking Water Act and the Clean Water Act are published annually in 40 CFR Parts 136 and 141 and are available from the Government Printing Office at: https://ecfr.io/Title-40/cfrv25#0.

Samples to be analyzed in accordance with the USEPA SW846 Methods are published in the latest final update of the USEPA SW846 *Test Methods for Evaluating Solid Waste – Physical and Chemical Methods* and amended and may be found on the USEPA Website at: https://www.epa.gov/hw-sw846.

Current requirements for samples to be analyzed in accordance with the USEPA Contract Laboratory Program are published in the latest version of the USEPA CLP SOWs. These documents may be found on the USEPA website at: https://www.epa.gov/clp. The clock governing laboratory contract holding times for samples and blanks analyzed by Contract Laboratory Program (CLP) methodologies begins when the sample is received in the laboratory as documented on the laboratory's external chain of custody form. This is known as the Verified Time of Sample Receipt (VTSR). Please refer to the tables at the end of this chapter for additional information.

The holding time clock for all other certified methods and parameters begins at the time of sample collection in the field.

Sample hold times include 2 components, the first is the preparation of the sample (i.e., extractable organics) and the second is the instrument analysis of the prepared sample (i.e., the extract).

2.6.6 Specialty Methods

Specialty methods refer to non-standard methods, which are methods that are not routinely used or not published by USEPA. Additional QC samples may be required for specialty methods. These QC samples should be defined and documented in the QAPP.

These methods are used for:

1. Emerging contaminants (e.g., Per- and polyfluoroalkyl substances (PFAS));
2. New Jersey defined methods, such as Extractable Petroleum Hydrocarbon and NJ Low Level TO-15; and
3. Modification to approved methods.

It is important to note that these specialty methods may include lab and field quality control provisions. The project manager may also determine the need to implement additional types of QA/QC blanks when initial sampling episodes produce blank contamination that cause the generated data to become suspect.

Examples may include additional blank samples prepared at the same time and in the same manner as the trip and field blanks, which are designated for placement in laboratory storage areas, sample preparation areas or perhaps at ambient air ventilators or other field locations. These additional blanks are then subject to the same analysis as the samples to determine if location specific cross-contamination during handling/storage may be occurring. Specialty methods may also include the use of alternative analytical methodologies for unique, site specific parameters of concern.

Many methods have additional quality control requirements that have evolved to monitor both storage and analytical procedures. All parties, including the laboratory, should be aware of these changes as new or revised methods are issued by USEPA or other governmental agencies in response to changes in regulation, contractual requirements, and instrumentation.

2.6.7 Additional QC Samples

Additional QC samples may be required in specific cases.

2.6.7.1 Duplicate Samples Obtained in the Field (Field Duplicates)

The collection of duplicate samples provides for the evaluation of the laboratory's and field sampling team's performance by comparing analytical results of two samples from the same location.

Duplicate samples, when required by the analytical method, are to be included for each matrix at a maximum rate of one for every twenty samples. If less than twenty samples are collected during a sampling episode, one duplicate should be performed. Duplicate requirements may be waived or expanded depending on the regulatory program or remedial phase. Keep in mind that various USEPA methods require a higher frequency of field duplicate samples. Therefore, the analytical methods should be reviewed to determine the appropriate number of field duplicates.

2.6.7.1.1 Aqueous Matrix Duplicates

Duplicate water samples (potable well, monitoring well, surface water) should be obtained by the collection of one sample and splitting it into separate sample containers. Note that sufficient

sample volume should be collected at one time in order to fill all of the necessary sample containers for each parameter. For example, samples for volatile organics analysis from monitoring wells should be filled from the same bailer full of water whenever possible and be the first set of containers filled. When other sampling devices are used, the vials for volatile organics should be alternately filled.

2.6.7.1.2 Non-Aqueous Matrix Duplicates

The collection of non-aqueous matrix duplicate samples for volatile organic samples should be taken from discrete locations or intervals, without using compositing or mixing (as described below for non-volatile organic analyses). This practice is necessary to prevent loss of volatile constituents and to preserve, to the extent practicable, the integrity of the volatile fraction (see Chapter 6 *Sample Collection*, Section 6.2.7 *VOC Sample Collection for Soils*, for further information).

Prior to the duplicate sample collection for non-volatile organic analysis, the soil or sediment sample aliquot requires homogenization. This is necessary in order to generate two equally representative samples. Note that sufficient sample volume should be collected at one time to fill all of the necessary sample containers. It may be necessary to co-locate or depth-integrate collection, so enough sample volume is available. A description of this process should be provided in the sampling plan. Moisture content, particle size, and adsorption properties of various soils, sediments, and waste materials may inhibit the ability to achieve complete mixing prior to filling sample containers.

Homogenization should be accomplished by placing the sample in a properly decontaminated tray or bowl and mixing it with a decontaminated instrument. The extent of mixing required will depend on the nature of the sample and should be done to achieve a consistent physical appearance and texture prior to filling sample containers. Exclusion of extraneous material is recommended.

Once mixing is completed the sample should be divided in half and sample material should be scooped alternately from each half to fill containers. Several laboratory methodologies for compositing samples published by the American Society for Testing and Materials (ASTM) and USGS have been suggested for use in the field; however, they were not specifically designed for homogenization of known or suspected hazardous materials and often must be "modified" to be useful. They tend to assume a uniform sample exists to begin with and their intent may be to calculate average grain size, predict weight to volume ratios, or to reduce the size of a sample to one more convenient for handling and analysis. They also tend to assume a much larger volume of material will be subject to the methodology. Therefore, these methods are not recommended for generating duplicate samples in the field.

2.6.7.2 Splitting Samples with Interested Parties

When various sites are under investigation, property owners and other interested parties may desire to obtain samples for analysis which are duplicates of those obtained by NJDEP personnel or contractors. If this becomes necessary, procedures for obtaining duplicate samples described above should be followed.

To maintain the integrity of any sample "split" between interested parties, the following procedures are recommended:

- A second set of sample containers, blank samples, preservatives, sample shuttles, chain of custody forms, etc., should be provided.
- Duplicate samples, trip blanks, and field blanks should be included for all interested parties.
- All interested parties desiring to obtain split samples during planned sampling episodes

should provide each other with sufficient notice. This is essential for planning purposes and to avoid confusion or delays in the field.

- All parties should use the same analytical methods to allow for comparability of data. Choice of analytical methodologies should be agreed upon prior to the sampling event.

2.6.7.3 Background Samples

When background samples are required for comparison of site conditions to the surrounding environment they should be collected and handled in the same manner as all other samples. Requirements for inclusion of background samples are determined on a program specific and/or case-by-case basis.

2.7 Quality Assurance for Emerging Contaminants

Quality assurance requirements related to emerging contaminants may require special attention during project planning and execution. It is critical for the investigator to work with a certified analytical laboratory to ensure that samples are collected in the appropriate containers, shipped to the laboratory within the required hold times, and analyzed with a method that will allow for the sufficient evaluation of analytical results. All of this should be considered and included as part of pre-sampling planning and site characterization.

New analytical methods are periodically developed by USEPA to detect and quantify unregulated or emerging contaminants. Selection of analytical approaches should be based on project data quality objectives. Special considerations may include different holding times and avoiding the use of materials such as certain clothing, gloves, sunscreen, field notebooks, sample containers, preservatives. Guidelines for sample collection and handling may vary by parameter, method, and analytical laboratory.

Timely information is available at: http://www.nj.gov/dep/srp/emerging-contaminants/.

For technical fact sheets on other emerging contaminants, please visit: https://www.epa.gov/fedfac/emerging-contaminants-and-federal-facility-contaminants-concern.

References

American Society for Testing Materials, *Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites*, ASTM D-5088-90.

Fink, Michael J., Boyajian, Ralph T., *Decontamination Procedures for Ground Water Sampling Equipment*, Proceedings of the Third National Outdoor Action Conference on Aquifer Restoration, Ground Water Monitoring and Geophysical Methods. May 1989.

Mackiewicz, Michael C., *A Simple 11 Step Procedure to Document the Accuracy, Precision and Significance of Measurements by Field Instrumentation*, Proceedings of the Fourth National Outdoor Action Conference on Aquifer Restoration, Ground Water Monitoring and Geophysical Methods, Ground Water Management, 1990.

Parker, Louise V., *The Effects of Ground Water Sampling Devices on Water Quality: A Literature Review,*

Ground Water Monitoring Review, Spring 1994.

Parker, Louise V., and Ranney, Thomas A., *Decontamination Materials Used in Ground Water Sampling Devices: Organic Contaminants*, Ground Water Monitoring Review, Winter 2000.

Yeskis, Douglas, and Zavala, B., *Ground Water Sampling Guidelines for Superfund and RCRA Project Managers*, USEPA Report 542-S-02-001, May 2002.

USGS Handbooks for Water Resources Investigations, Book 9, *National Field Manual for the Collection of Water-Quality Data*, Chapters 1-9, 2003.

USEPA, *Immunoassay Guidelines for Planning Environmental Projects, Office* of Environmental Measurement and Evaluation, October 1996.

USEPA, *Field Analytical and Site Characterization Technologies Summary of Applications,* EPA-542-R- 97-011. November 1997.

URLs

https://www.epa.gov/quality

https://www.nj.gov/dep/enforcement/oqa/labcert.html

https://ecfr.io/Title-40/cfrv25#0

https://www.epa.gov/hw-sw846

https://www.epa.gov/hw-sw846/sw-846-compendium

https://www.epa.gov/clp

https://www.nj.gov/dep/srp/regs/

http://water.usgs.gov/owq/FieldManual/chapter3/Ch3_contents.html

Appendix 2.1
Sample Preservation, Container, and Holding Times
for
Drinking Water, Wastewater, Ground Water, and Surface Water
(Except Radiological Parameters)

Potable water samples shall be handled and preserved in accordance with the requirements in the Code of Federal Regulations (CFR) Title 40 Part 141. In addition, the *Regulations Governing the Certification of Laboratories and Environmental Measurements* at N.J.A.C. 7:18 shall be reviewed to ensure that requirements for sample handling and preservation for specific parameters are also incorporated when necessary.

Non-potable water samples shall be handled and preserved in accordance with the requirements in the Code of Federal Regulations (CFR) Title 40 Part 136.3 Table II- Required Containers, Preservation Techniques, and Holding Times and the corresponding Table II Notes at the bottom of the table. In addition, the *Regulations Governing the Certification of Laboratories and Environmental Measurements* at N.J.A.C. 7:18 shall be reviewed to ensure that, requirements for sample handling and preservation for specific parameters are also incorporated when necessary.

The regulation is available at: https://www.nj.gov/dep/rules/rules/njac7_18.pdf.

CFR Title 40 Part 141 is available at:
https://www.ecfr.gov/cgi-bin/text-idx?SID=bdceb25a993c314c50a0c73d852db2e3&mc=true&tpl=/ecfrbrowse/Title40/40cfr141_main_02.tpl

CFR Title 40 Part 136 is available at:
https://www.ecfr.gov/cgi-bin/text-idx?SID=05e16b47f48c318f64b0abfcaa2caf23&mc=true&tpl=/ecfrbrowse/Title40/40cfr136_main_02.tpl

Appendix 2.2
Sample Preservation, Container, and Holding Times
for
Radiological Parameters

In accordance with 7:26C-2.3(a)(3)(iii), remediation of radiologically impacted sites may not be conducted without prior NJDEP approval. Documents relating to remediation of contaminated sites shall be submitted for NJDEP review and will be referred to the Bureau of Environmental Radiation for review. This would include Quality Assurance Project Plans (QAPP) which a Licensed Site Remediation Professional (LSRP) should develop with input from applicable regulations, selected analytical methods, the contracted NJ OQA-certified laboratory, and other relevant guidance. For more information, refer to FSPM Chapter 12 which addresses radiological assessment in more detail.
https://www.nj.gov/dep/srp/guidance/fspm/pdf/chapter12.pdf

Regulations Governing the Certification of Laboratories and Environmental Measurements at: N.J.A.C. 7:18
https://www.nj.gov/dep/rules/rules/njac7_18.pdf

The Office of Quality Assurance provides a list of matrix specific parameters and methods which a laboratory can receive certification. https://www.nj.gov/dep/enforcement/oqa/docs/part3.pdf

Remediation Standards for Radioactive Materials may be found at N.J.A.C. 7:28-12:
https://www.state.nj.us/dep/rpp/rms/rad_cleanups.htm

The Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM) provides detailed guidance on how to demonstrate that a site is in compliance with a radiation dose- or risk-based regulation.
https://www.epa.gov/radiation/download-marssim-manual-and-resources

The Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP) provides guidance for the planning, implementation and assessment phases of projects that require laboratory analysis of radionuclides. https://www.epa.gov/radiation/multi-agency-radiological-laboratory-analytical-protocols-manual-marlap

EPA Manual for the Certification of Laboratories Analyzing Drinking Water includes radiochemistry sample preservation. https://www.epa.gov/dwlabcert/laboratory-certification-manual-drinking-water

Potable and Non-Potable Water Samples

Parameter	Preservation	Container	Holding Times
Gross alpha	Conc. HCl or HNO_3 to pH 2*	P or G	180 days
48-Hour Rapid Gross Alpha*	Conc. HCl or HNO_3 to pH 2*	P or G	48-hours**
Gross beta	Conc. HCl or HNO_3 to pH 2*	P or G	180 days
Strontium-89	Conc. HCl or HNO_3 to pH 2	P or G	180 days
Strontium-90	Conc. HCl or HNO_3 to pH 2	P or G	180 days
Radium (total)	Conc. HCl or HNO_3 to pH 2	P or G	180 days
Radium-224	Conc. HCl or HNO_3 to pH 2	P or G	4 days (recommended)
Radium-226	Conc. HCl or HNO_3 to pH 2	P or G	180 days
Radium-228	Conc. HCl or HNO_3 to pH 2	P or G	180 days
Cesium-134/137	Conc. HCl or HNO_3 to pH 2	P or G	180 days

Iodine-131	None	P or G	8 days
Tritium	None	G	180 days
Uranium	Conc. HCl or HNO_3 to pH 2	P or G	180 days
Plutonium	Conc. HCl or HNO_3 to pH 2	P or G	180 days
Photon emitters (including Cobalt-60, Zinc-65, Ruthenium-106, and Barium-133)	Conc. HCl or HNO_3 to pH 2	P or G	180 days
Radon-222***	Cool 4°C	G	4 days (recommended)

Drinking water samples subject to radiological measurements shall be handled and preserved in accordance with the requirements presented in the table above and the requirements listed below. This table includes requirements from the USEPA's Manual for the Certification of Laboratories Analyzing Drinking Water, USEPA-815-B-97-001. If there is any conflict between the table and the USEPA publication (including any amendments or supplements) on which any part of the table is based, the USEPA rule or publication shall control, except in reference to 48-Hour Rapid Gross Alpha and Radium-224 Methods.

* If HCl is used to acidify samples that are to be analyzed for gross alpha or gross beta activities, the acid salts shall be converted to nitrate salts before transfer of the samples to planchets.

**48-hour Rapid Gross Alpha Method applies to (Community Water System (CWS) compliance monitoring, as well as testing under Private Well Testing Act (PWTA). Initial counting of the plancheted sample shall be initiated no sooner than 36 hours from sample collection and must be completed by 48 hours after sample collection (N.J.A.C 7:18-6.4(a)3ii).

*** The method for sampling described in EPA/600/2-87/082-1989 "Two Test Procedures for Radon in Drinking Water" shall be followed.

Sample shall be acidified at the time of collection, in accordance with the requirements listed under "Preservation" in the table above. A minimum of 16 hours shall elapse between acidification and analysis. If suspended solids activity is to be measured, then a second unpreserved sample shall be taken for this measurement; and if the sample is shipped in its original container to a certified environmental laboratory or storage area, acidification of the sample (in its original container) may be delayed for a period not to exceed five days.

Appendix 2.3
Sample Preservation, Container, and Holding Times
for
Solid/Hazardous Waste Samples, Aqueous, Soils, Liquids, Sediments, Sludges, and Air

Information listed in the table below was based on the following references. For the most current requirements go to the link provided below.

SW-846 Method Compendium:

https://www.epa.gov/hw-sw846/sw-846-compendium

NOTE: Sample hold times include 2 components, the first is the preparation of the sample (i.e., extractable organics) and the second is the instrument analysis of the prepared sample (i.e., the extract).

Parameter	Preservation	Container	Holding Times
Volatile Organics for soil/ sediment, sludge	Cool to 0-6° C	Glass Teflon®-lined cap (septum lid)	14 days until analysis
VOAs for Soil by 5035	Refer to Container details, Cool to 0-6° C	Encore™ or equivalent field coring device (3 x 5-grams) plus total solids jar (1 x 2-oz)	14 days until analysis
VOAs in Soil by 5035	Refer to Container details, Cool to 0-6° C	Terracore or equivalent field preserved 40-ml vials (2 x 40 ml vials with MeOH; plus 1 x 40-ml DI water vial; plus 2-oz total solids jar; plus plastic 5-gram coring device)	14 days until analysis
VOAs in non-potable water by 8260 See entries below	HCL, pH<2, Cool to 0-6° C	Amber Glass, Teflon Lined, 3 x 40-ml Vials with HCl, pH<2	14 days until analysis
VOAs by 624 in non-potable water	Na2S2O3, Cool to 0-6° C	Amber Glass, Teflon Lined cap, 3 x 40 ml Vials	7 days until analysis
Acrolein and Acrylonitrile in liquid samples	Adjust to pH 4-5, Cool to 0-6° C These compounds are highly reactive and should be analyzed ASAP per SW846 Methods 5021, 5030, 5031, 5032.	Glass, Teflon®-lined cap 3 x 40-ml vials with PTFE-lined septum caps	7 days until analysis

Appendix 2.3 (continued) Required Preservation, Container and Holding Times for Solid/Hazardous Waste Samples (Soils, Liquids, Sediments, Sludges, and Air)

Parameter	Preservation	Container	Holding Times
SVOC by 8270, Organochlorine Pesticides by 8081, PCBs by 8082, and Herbicides by 8151 for soil, sediment, sludge, solid waste	Cool to 0-6° C	Amber Glass, Teflon®-lined cap, 1 x 8-oz wide mouth jar	14 days until extraction
SVOC by 8270s, Organochlorine Pesticides by 8081, PCBs by 8082, and Herbicides by 8151 for non-potable water	Cool to 0-6 C	Amber Glass, Teflon®-lined cap, 2 x 1,000-ml wide mouth	7 days until extraction
SVOCs by 625; OC-Pesticides and PCBs by 608 in non-potable water	Na2S2O3, Cool to 0-6° C	Amber Glass, Teflon Lined cap, 2 x 1,000 ml	7 days to extraction
NJ-EPH in non-potable water	HCl, pH<2, Cool to 0-6° C	Amber Glass, Teflon Lined cap, 2 x 1,000 ml, HCl, pH<2	14 days to extraction
NJ-EPH and TPH-DRO by 8015M in soil, sediment, solid waste	Cool to 0-6° C	Amber Glass, Teflon Lined cap, 1 x 4-oz jar	14 days to extraction
TPH-GRO by 8015M in soil, sediment, solid	MeOH, Cool to 0-6° C	Amber Glass, Teflon Lined, 15 grams placed in 40 mL Vial, MeOH, 4° C	14 days to extraction
TPH-GRO by 8015M in non-potable water	HCl, pH<2, 0-6° C	Amber Glass, Teflon lined cap, 2 x 40 ml VOA Vials	14 days to extraction
TPH-DRO by 8015M in non-potable water	Cool to 0-6° C	Amber Glass, Teflon Lined cap, 2 x 1,000 ml	7 days to extraction
Metals, Total by 200.7, 200.8, 6010, 6020 (except Cr VI and Hg) in non-potable water	HNO_3 to pH < 2	P, G, 1 x 500 ml	180 days
Metals, Dissolved by 6010, 6020 (except Cr VI and Hg) in non-potable water (field filtered)	Filter on-site, HNO_3 to pH < 2	P, G, 1 x 500 ml	180 days
Metals, Dissolved by 6010, 6020 (except Cr VI and Hg) in non-potable water (lab filtered)	None (Filtered and preserved by lab upon receipt)	P, G, 1 x 500 ml	180 days
Metals, Total by 6010, 6020; in Soil, sediment, solid samples	None	Amber Glass, Teflon Lined, 1 x 4-oz. jar	180 days

Appendix 2.3 (continued) Required Preservation, Container and Holding Times for Solid/Hazardous Waste Samples (Soils, Liquids, Sediments, Sludges, and Air)

Parameter	Preservation	Container	Holding Times
Chromium VI by 7196A in Soil, Sediment, Solid samples	Cool to 0-6° C	Amber Glass, Teflon Lined, 1 x 4-oz.	30 days to digestion; analysis 168 hours (7 days) after digestion
Chromium VI by 7196A, SM3500Cr-D in non-potable water samples	Cool to 0-6° C	Plastic, 1 x 500 ml	24 hours to digestion
Mercury, Total by 245.1, 7470A, 7474 in non-potable water samples	HNO_3 to pH < 2	P, G	28 days
Mercury, Dissolved by 245.1, 7470A, 7474 in non-potable water	Filter on-site, HNO_3 to pH < 2	P, G	28 days
Mercury, Total by 7471B in Soil, sediment, Solid samples	Cool to 0-6° C	Amber Glass, Teflon Lined Lid, 1 x 4-oz.	28 days
Ambient Air Analysis			
TO-15 VOAs in Air by Canister with Pressure Gage and Regulator Flow Controller	None	Canisters (1-L, 2.7-L, 6-L	30 days from sample collection to analysis
NJDEP-SRP TO-15 Low Level Method for VOAs in Air by Canister with Pressure Gage and Regulator Flow Controller	None	Canisters (1-L, 2.7-L, 6-L)	30 days from sample collection to analysis
TO-13, TO-17 for SVOCs and PAHs in Air by Active Sampling (Pump) onto Sorbent Tubes (PUF-XAD)	Cool to 0-6° C after sample collection and in refrigeration unless samples are analyzed the same day of collection. The samples must be stored in an organic solvent free environment. Small packages of activated charcoal/silica gel must be with each shipment container of multiple tubes.	Sorbent Tubes (PUF-XAD)	7 days to extraction
Mercury in Air by NIOSH 6009 Modified	No ice, room temperature (25° C), and keep dry	Hopcalite Sorbent Tube	21 days to analysis
Metals (Arsenic, Lead, Nickel) in Air by 6010, 6020	None	Filter	180 days to analysis

Appendix 2.4
Sample Preservation, Container, and Holding Times for the Analysis of BIOLOGICAL Samples for Freshwater, Estuarine and Marine Samples

Parameter	Sample Container	Container Volume	Preservation[1]	Holding Times	Analytical Methodology	Sample Container Cleaning
PHYTOPLANKTON FRESHWATER						
Live samples; including harmful algae identification	P, G	250 ml	Refer to individual method	24 hours	SM 10200	Warm detergent solution wash, thorough rinse in tap and distilled water
Preserved samples; including harmful algae identification	P, G	1000 ml	50 ml neutralized formalin store/transport in dark, cool container	1 month	SM 10200	Warm detergent solution wash, thorough rinse in tap and distilled water
Chlorophyll a/ Pigment analysis	P, G	500 ml	Refer to individual method store/transport in dark	24 hours	SM 10200H EPA 445.0	Warm detergent solution wash, thorough rinse in tap and distilled water
Cyanotoxins	G (amber) Plastic (PET or HDPE only)	250 ml	Refer to individual method store/transport in dark	24 hours	EPA 546, 544, 545.	
MARINE AND ESTUARINE						
Live samples	P, G	250 ml	Refer to individual method	24 hours	SM 10200H	Warm detergent solution wash, thorough rinse in tap and distilled water
Preserved samples	P, G	1000 ml	10 ml or more Lugol's solution to maintain weak tea color. Store/transport in dark, cool container.	48 hours	SM 10200H	Warm detergent solution wash, thorough rinse in tap and distilled water

Appendix 2.4 (continued) Sample Preservation, Holding Times, and Methodologies for the Analysis of BIOLOGICAL Samples for Freshwater, Estuarine and Marine Samples

Parameter	Sample Container	Container Volume	Preservation[1]	Holding Time	Analytical Methodology	Sample Container Cleaning
PHYTOPLANKTON MARINE AND ESTUARINE						
Chlorophyll a	P, G amber or foil-covered	250 ml	Refer to individual method store/transport in dark	48 hours	SM 10200H	Warm detergent solution wash, thorough rinse in tap and distilled water
ZOOPLANKTON						
Freshwater	P, G	6,000 ml	300 ml neutralized formalin. Store in cool container	1 month	SM 10200	Warm detergent solution wash, thorough rinse in tap and distilled water
Marine & Estuary	P, G	6,000 ml	5% formalin (5 ml neutralized formalin/100 ml tap water), store and transport in cool container	1 month	SM 10200	Warm detergent solution wash, thorough rinse in tap and distilled water
PERIPHYTON DIATOMETER SLIDES AND ROCK SCRAPINGS						
Species Composition	125ml jar polyseal cap	N/A	Lugol's solution (4% buffered formalin, "M3" fixative, or, 2 % glutaraldehyde), store and transport in iced container in the dark	1 month	SM 10300+ EPA99 Periphyton.6	Warm detergent solution wash, thorough rinse in tap and distilled water
DIATOMS (Sediment Core) Species Composition	sealed plastic bag	N/A	Refer to individual method Store and transport in dark container	N/A		
PERIPHYTON						
Chlorophyll a	P, G	30 ml	90% neutralized acetone, Refer to individual method For cooling, store and transport in dark container	48 hours	SM 10300 EPA Method 445.0	Warm detergent solution wash, thorough rinse in tap and distilled water

Appendix 2.4 (continued) Sample Preservation, Holding Times, and Methodologies for the Analysis of BIOLOGICAL Samples for Freshwater, Estuarine and Marine Samples

Parameter	Sample Container	Container Volume	Preservation[1]	Holding Time	Analytical Methodology	Sample Container Cleaning
MACROINVERTEBRATES						
Species composition	P, G	N/A	5% neutralized formalin (5 ml neutralized, formalin/100 ml sample water [95% ethanol, 70% isopropyl alcohol])	N/A	SM 10500 EPA99: Macroinvert-ebrates 7	Warm detergent solution wash, thorough rinse in tap and distilled water

[1] Neutralized formalin = 100 % neutralized formalin with sodium tetraborate to pH 7.0 – 7.3

Appendix 2.5
Sample Preservation, Container, and Holding Times for Estuarine and Marine Samples

Parameter	Sample Container	Container Volume	Preservation[1]	Holding Times	Analytical Methodology[2]
Ammonia	Polypropylene	50mL	2mL 3.5% phenol added to 40mL, Cool to 0-6° C	14 days	EPA 350.1 Modified
Nitrate + Nitrite	Polypropylene	50mL	Freeze	28 days	EPA 353.4
Total Nitrogen	Polypropylene	50mL	Freeze	28 days	USGS I-4650-03
Orthophosphate	Polypropylene	50mL	Freeze	28 days	EPA 365.5
Total Phosphorous	Polypropylene	50mL	Freeze	28 days	USGS I-4650-03
Biogenic Silica	Polypropylene	50mL	Cool to 0-6° C	28 days	EPA 366.0 Modified
TOC	P,G	125ml	Sulfuric Acid to a pH < 2, Cool to 0-6° C	28 days	SM 5310B

[1] All samples are placed in a cooler filled with ice for transportation to the lab
[2] Methods recommended by the NJDEP Bureau of Marine Water Monitoring

Appendix 2.6
Table of Quality Control Samples

This table provides descriptive information about the various types of quality control samples often used during sampling events which support a variety of programs. This is not meant to be an all-inclusive list or description of the various types of quality control samples. Consult with the NJDEP program or analytical method for applicable requirements. A detailed description of the various types of quality control blanks is also available in a USEPA Fact Sheet at: https://www.epa.gov/sites/default/files/2015-06/documents/blanks.pdf.

Type	Description	Frequency	Purpose	Comments
Temperature Blank	Temperature reading measured from a bottle of water inside sample cooler upon receipt by laboratory.	One temperature blank per cooler measured and recorded upon receipt of sample coolers at laboratory.	To evaluate whether samples were adequately cooled during shipment.	Lab-provided, unopened in field
Trip Blank (TB)	VOC blank (unopened) used to evaluate contamination introduced during sampling, storage and transport.	Minimum one per shipment. Refer to analytical method for any additional trip blank requirements.	To evaluate contamination introduced during shipment.	Lab-provided, unopened in field
Field Blank (FB)	Collected as analyte-free water poured through sampling equipment prior to sample collection. If no equipment requiring decontamination is used in sampling, then analyte-free water is poured from bottle to bottle. This type of blank is sometimes referred to as an “equipment blank” or “rinsate blank”.	Minimum 5% (one per twenty) per analytical parameter per matrix per sampling procedure. Minimum of one per day. *	To assess contamination from field conditions during sampling and to evaluate the adequacy of the decontamination process if decontaminated equipment is used.	Lab-provided DI water and containers or reagent water of known quality
Field Reagent Blank (FRB)	Collected as analyte-free water poured from bottle to bottle as specified in certain drinking water methods.	See specific method.	To evaluate contamination introduced during sample collection, storage, and transport.	Lab-provided DI water and containers or reagent water of known quality

Type	Description	Frequency	Purpose	Comments
Field Duplicate (DUP)	A field duplicate is a sample collected at the same time and location in the field as the original sample by the same sampling team and submitted to the same lab for analysis. They are taken through all steps of the analytical procedure in an identical manner. These samples are used to assess precision of the total method, including sampling, analysis, and site heterogeneity. It can be collected as a sub-sample (in which one sample is collected and then split into two or more portions) or a co-located sample (in which two different samples are collected from the same location).	Minimum 5% (one per twenty) per analytical group per matrix per sampling procedure per sampling team. One per ≤ 20 field samples, if a site-specific MS/MSD was not collected. *	To evaluate errors introduced during sampling and other field activities, to measure precision.	Lab-provided containers
Field Split (Split)	Collected (as needed) as one field sample split (sub-sampled) into two samples shipped to separate laboratories.	As needed.	To evaluate interlaboratory comparability.	Lab-provided containers
Matrix Spike / Matrix Spike Duplicate (MS/MSD)	Matrix spike. A sample prepared by adding a known concentration of a target analyte to an aliquot of a specific homogenized environmental sample for which an independent estimate of the target analyte concentration is available. The matrix spike is accompanied by an independent analysis of the unspiked aliquot of the environmental sample. Spiked samples are used to determine the effect of the matrix on a method's recovery efficiency.	One per twenty per sample analytical group per sample matrix. As needed. *	To evaluate laboratory sample preparatory and analytical bias and precision for measurement of specific target analyte compounds in specific sample matrices.	Lab-provided containers

Notes:

* 5% or one per twenty, whichever is more frequent

Practitioners are advised to clearly identify both field samples and field QC samples when placing a project bottle order with the laboratory, and to collect sufficient sample volume to support the requested analytical parameters for each of the individual field samples and field QC samples. The laboratory will provide sample containers (with preservatives) and analyte-free water for the collection of field blanks, as well as containers (with preservatives) for site-specific MS/MSDs, DUPs, Splits, FBs, and TBs. Refer to NJDEP Analytical Methods QA/QC Technical; Guidance (2014) at: https://www.nj.gov/dep/srp/guidance/ .