

## Chapter 2 Quality Assurance

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## Chapter 2

### Quality Assurance

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#### 2.1 Introduction

This chapter provides the user with quality assurance requirements and procedures for conducting environmental measurement sampling episodes. In order to generate analytical data of known and defensible quality, adherence to established quality assurance protocol is necessary. Quality assurance measures coupled with a statistically based sampling plan will improve sample collection while maintaining the integrity of the samples prior to analysis. The NJDEP has established standard operating procedures to maintain consistency in sample collection and handling. Standard operating procedures may vary among the specific regulatory programs (i.e. CERCLA, RCRA, Drinking Water, Radiation and NJPDES).

Monitoring projects for the Clean Water Act, Safe Drinking Water Act Programs, RCRA and CERCLA are based on approved Quality Assurance Project Plans (QAPP) which ensure environmental monitoring data is of known quality. Plans are prepared by using a variety of standard references including, but not limited to, *EPA Requirements for QA Project Plans (QA/R-5)* and *Guidance on Quality Assurance Project Plans (G-5)*. These documents can be found on the USEPA Website at [http://www.epa.gov/quality/qa\\_docs.html](http://www.epa.gov/quality/qa_docs.html). Quality Requirements for non-EPA organizations are defined in the Code of Federal Regulations. The USEPA issues documents to provide information on satisfying the Federal Regulations. These documents contain policy statements (equivalent to EPA Order 5360) that identify and discuss mandatory elements of the USEPA Quality Systems.

Finally, this chapter highlights decontamination and QA/QC procedures for certain aspects, which may be required or encountered when conducting sampling episodes. Do not assume that all aspects or scenarios are discussed herein. The “site specific” nature of sampling makes it incumbent upon those responsible to indicate in the sampling plan 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.

##### 2.1.1 Laboratory Certification

The certification status of the laboratory must 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 *Regulations Governing the Certification of Laboratories and Environmental Measurements* N.J.A.C. 7:18 and/or voluntary certification under the National Environmental Laboratory Accreditation Program (NELAP). The Office of Quality Assurance offers certification in the following categories:

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

##### 2.1.2 Analyze Immediately – Environmental Laboratory Certification

N.J.A.C. 7:18 (Laboratory Certification) requires that any environmental laboratory\* submitting analytical data to the department, regardless of quality level, must be certified by the Office of

Quality Assurance. This includes certification for the “Analyze Immediately” parameters under the Safe Drinking Water, Water Pollution, and the Solid and Hazardous Water Programs. Environmental samples analyzed in the field under this heading include Chlorine Dioxide, Free Chlorine Residual, Ozone, pH, Temperature, Chlorine Total Residual (TRC), Oxygen Dissolved (probe), and Sulfite. Environmental laboratories measuring these parameters in the field must first be certified by the NJ Office of Quality Assurance before sample collection. \*Environmental laboratory is defined as any laboratory, facility, consulting firm, government or private agency, business entity or other person that the department has authorized.

### 2.1.3 Field and Laboratory Immunoassay Analysis Certification

Additionally, immunoassay methods that are considered laboratory or field methods require certification under the Solid and Hazardous Waste Program. Regardless of whether a company or organization is or is not a laboratory, certification must be obtained. This includes but is not limited to responsible parties, contractors and facilities. The New Jersey Office of Quality Assurance must be contacted to obtain additional information regarding the above laboratory, immunoassay and field instrument certification requirements.

## 2.2 Data Quality Levels

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For Site Remediation projects, selection and application of site-appropriate Data Quality Levels should be discussed in the Field Sampling Plan- Quality Assurance Project Plan (FSP-QAPP). The requirements of the FSP-QAPP are based on the, *EPA Requirements for QA Project Plans (QA/R-5)* and *Guidance on Quality Assurance Project Plans (G-5)*. To develop reliable site investigation data for NJDEP lead, publicly funded CERCLA (Superfund) or non Superfund publicly funded sites, the prime consultant/contractor awarded a term contract for the Remedial Investigation/Feasibility Study (RI/FS) or Engineering Design work has the responsibility to develop and implement a Field Sampling Plan-Quality Assurance Project Plan (FSP-QAPP). This document must present the organization, functional activities and specific Quality Assurance/Quality Control (QA/QC) activities needed to attain specific project goals and data quality objectives. Any sampling conducted by state contract vendors, including sampling associated with removal actions or operations and maintenance contracts, requires the development and implementation of a Quality Assurance Project Plan. The Department will approve these plans prior to implementation by a contractor. Requirements for these plans are generally specified in state contracts. Regardless of a document’s title or “deliverable” name (e.g. QAPP, FSP-QAPP), NJDEP and USEPA require these plans for all sampling events that are conducted in the state. It is recommended that these plans be contained in a stand-alone document.

For permit compliance sampling, a quality assurance program is often necessary to assure analytical accuracy sufficient to demonstrate compliance. Permits may require the permittee to achieve detection of pollutants if they are present at certain minimum concentrations, or to eliminate discharges if they exceed concentrations, which may not be detectable, unless proper quality assurance methods are implemented.

### 2.2.1 Quality Assurance Programs

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.

Sample collection, preservation and holding times for New Jersey certified parameters and methods are listed in the *Regulations Governing the Certification of Laboratories and Environmental Measurements* N.J.A.C 7:18. The criteria established in the Certification Regulations are required. However, as changes and additions are made by the USEPA, any changes published in the code of Federal Regulations for the samples being analyzed under the Safe Drinking Water Act and the Clean Water Act must follow the latest Code of Federal Regulations. Those requirements are published annually in 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. The USEPA Contract Laboratory Program (USEPA CLP) revises or issues a new Statement of Work (SOW), these requirements will supercede the NJ Certification Regulations. The current requirements are summarized in the Tables at the back of this section.

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 may be found on the USEPA Website at <http://www.epa.gov/epahome/cfr40.htm>

Current requirements 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 3<sup>rd</sup> Edition* issued 1996 and amended. They may be found on the USEPA Website at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

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 <http://www.epa.gov/oerrpage/superfund/programs/clp/index.htm> or at <http://www.epa.gov/superfund/programs/clp/index.htm>

The following quality assurance requirements have been established to maintain sample integrity. Their prime 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 must be a measure of control over all sample-handling procedures beginning with sample container cleaning procedures and ending with laboratory analysis. This chapter focuses on the first half of the control process including the procedures leading up to and ending with sample packaging and transport to the laboratory. Sample packaging and transport are discussed in Chapter 12.

### 2.2.2 Field Analytical Methods

Almost all projects require screening or semi-quantitative data during the field screening phase of the site investigation. For example, 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 of polychlorinated biphenyls (PCBs) or explosives during site characterization

The main advantage to engaging field analytical methods is they can allow for the performance of rapid characterization with only a few mobilizations via a dynamic sampling plan. Field analytical methods can provide data of sufficient quality to meet the predetermined data quality objectives providing that supporting QA/QC procedures are in place.

To be “effective,” the field data generated must be of sufficient quality, with respect to measurement precision or reproducibility, accuracy, sensitivity, and have good correlation with the standard laboratory methods to support the objective of the site investigation or cleanup and the DQO. Several factors to be considered before mobilization include the following (the factors are not intended to be all inclusive):

- The action levels for field decisions shall be established as part of the DQOs.
- The project objective shall permit screening and semi-quantitative data in addition to quantitative data to meet DQO.
- The percentage of samples to be analyzed in the field as well as sent off-site for laboratory confirmation shall be determined.
- The methodology to compare field and laboratory data shall be established, for example using duplicate (field duplicate samples) and/or performance evaluation samples in addition to initial and daily calibrations.
- For the field instrument or the analytical method, the measurement selectivity, sensitivity, precision, accuracy, representativeness and action levels shall be determined.
- The standard operating procedures and method detection limit studies are completed before mobilization to evaluate any matrix interference that might be associated with a particular field technology.
- If applicable, the field technician performing the analyses shall have proof of training by the manufacturer/vendor of the test method.
- If sample preservation is required, samples shall be preserved in the field immediately after collection according to the method specific table in Appendix 2 of this document.

The New Jersey Department of Environmental Protection (NJDEP) is committed to streamlining the site investigation and remediation process at contaminated sites. The site investigation shall follow the Technical Requirements for Site Remediation, N.J.A.C. 7:26E, <http://www.state.nj.us/dep/srp/regs/techrule> that places emphasis on laboratory analytical methods. However, field analytical methods may be employed if sufficient documentation can be provided to the NJDEP to support the proper application of the method. Persons wishing to use a field analytical method shall submit the proposal to the project team for approval (see Chapter 7, *Field Analysis*).

### 2.3 Sample Containers

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Prior to the collection of a sample, consideration must be given to the type of container that will be used to store and transport the sample. The party requesting the analysis is responsible for requesting the proper sample containers, or, providing the laboratory with an accurate description of the matrix being sampled in order that the laboratory can provide the proper 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 <http://www.state.nj.us/dep/oqa/labcert.html>.

#### 2.3.1 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 the potential exists for contaminants to adsorb to the surface of the plastic or for the plasticizers to 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 must 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. Prior to ordering bottles from the laboratory, the method requirements should always be reviewed with the laboratory

### 2.3.2 Volume of the Container

The analytical method and the sample matrix will dictate the volume of sample to be collected. The sampler must supply sufficient volume of matrix for the laboratory to perform the required analysis. In most cases, the methodology dictates the volume of sample material 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.

### 2.3.3 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 should be white or uncolored. 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 40ml vials for volatile organic aqueous analysis so that any air bubbles in the sample can be easily detected. These are acceptable for use.

### 2.3.4 Container Closures

Container closures may be specified by method. Container closures should form a leakproof seal (i.e., screw caps or ground glass stoppers). Closures must be constructed of a material that is inert with respect to the sampled material, such as PTFE (e.g., Teflon<sup>®</sup>) or as specified by the method. Alternately, the closure may be separated from the sample by a closure liner that is inert to the sample material such as PTFE liner or septum. No amendments must be added to ground glass stoppers to facilitate opening.

### 2.3.5 Decontamination of Sample Containers

Pre-cleaned certified sample bottles can be purchased from bottles supply companies. Pre-cleaned bottles must be accompanied by supplier's certificate indicating the certified use of the bottles. The certificates issued provide a bottle specific use on a compound basis. Sample containers can also be laboratory cleaned, preferably by the laboratory performing the analysis. The cleaning procedure is dictated by the specific analysis to be performed on the sample.

### 2.3.6 Chain of Custody

The sample bottles should be prepared for shipment accompanied by a chain of custody and the cooler or shuttle containing them should be custody sealed. The chain of custody must also accompany the bottles during transportation to the field, sample collection, and transportation

back to the lab, during analysis and to identify final disposal of the sample container. When collecting a sample, personnel should record the seal number associated with each sample shuttle or cooler and record whether the seal was intact upon arrival in the field. This assures that the sample containers were not tampered with in the time between their preparation and their arrival in the field. After sample collection, the bottles again should be sealed into the shuttle or cooler and the seal number should be recorded in the field logbook. Upon arrival at the lab, the person receiving the sample should note the number and condition of the custody seal. Refer to Chapter 11, Documentation, for additional information on Chain of Custody.

### 2.3.7 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 must be taken to avoid contamination. Sample shuttles, or coolers, and sample bottles themselves must be stored and transported in clean environments. Sample bottles and clean sampling equipment must never be stored near solvents, gasoline, or other equipment that is a potential source of contamination. When under chain of custody, sample bottles must be secured in locked vehicles, custody sealed in shuttles or in the presence of authorized personnel.

The analytical methods may specify maximum or minimum sample temperatures that are required to be met during transport of the samples during shipment and upon receipt of the samples at the laboratory. These temperatures are specified as part of the sample collection, preservation and holding times for New Jersey certified parameters and methods are listed in the *Regulations Governing the Certification of Laboratories and Environmental Measurements* N.J.A.C 7:18. The current requirements are summaries in the Tables at the back of this section.

## 2.4 Decontamination Procedures

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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 as may accumulate on the sampling equipment itself between uses.

The following four sampling equipment cleaning procedures form the basis of the standard NJDEP requirements. These four procedures cover decontamination of aqueous and non-aqueous equipment over a broad range of contaminant exposures for all programmatic needs.

Provided at the end of this section are general considerations intended to raise decontamination awareness when cleaning pumps, heavy equipment, equipment related to direct push technology, monitor well casings and screens, and selection of cleaning location. This is followed by discussion on the disposal of decontamination fluids and drill cuttings. Exception to the following procedures may be evaluated and approved by NJDEP on a case-by-case basis if justifications to do so, involving site specific issues or conditions, are presented and verified beforehand.

In most instances fixed laboratory decontamination serves as the preferred alternative to field decontamination. Advantages include: 1) decontamination takes place in a controlled environment; 2) reduced need to transport, handle or dispose cleaning solvents, acids or wash water; 3) more attention can be focused upon sampling with field decontamination labor reduced or eliminated; 4) reduced

probability of cross-contamination due to improperly field decontaminated equipment and; 5) laboratory documentation of cleaning procedures and materials used. Disadvantages may include: 1) relative cost to scope of sampling event; 2) constraints meeting demands in emergency situations and; 3) logistics.

While the option exists to use field decontamination procedures for almost all non-aqueous sampling and certain aqueous sampling equipment (e.g., foot check valves, filtering equipment, stainless steel/ Teflon<sup>®</sup> pumps, automatic wastewater composite samplers), field decontamination of bailers is not acceptable. Bailers are required to be laboratory cleaned, packaged and dedicated for exclusive use at one sample location for that day's sampling (see definition of "laboratory cleaned" in the glossary). Field decontamination of bailers elevates the potential of cross-contamination to unacceptable levels. The possibility of contaminating a clean well is also of concern when using improperly cleaned sampling devices.

In certain instances the use of "disposable" bailers presents an option to circumvent the logistics associated with decontamination of standard reusable bailers. To insure quality control over these devices, disposable bailers must be decontaminated at the source of manufacture and proof of decontamination must accompany their purchase. They must be sealed in a protective covering prior to shipment from the manufacturer. Since these bailers will be used on a one time only basis, inflexibility as to standard material of construction requirements may be waived. For example, in approved instances, disposable bailers constructed of polypropylene are acceptable when sampling for trace metal analysis.

Generally, sampling devices must be protected from ambient contact during storage and remain protected until used in the field. Non-aqueous equipment may be wrapped in aluminum foil when sampling for organics only and/or sealed in plastic bags or equivalent material when sampling for inorganics, then custody sealed for identification. Equipment should be handled as little as possible prior to use and disposable gloves must be worn at all times when handled. Sampling equipment must never be stored near solvents, gasoline, exhaust emissions, or other equipment and/or materials that may impact the integrity of prepared sampling instruments. A record should be kept of the date and time when cleaned and this information should be labeled on the sampling device.

Exhaust producing equipment must be situated in such a manner as to not compromise the decontamination process. The decontamination station must also be set up in such a way as to not adversely impact a clean environment.

Whenever sampling, regardless of how equipment has been cleaned, always start sampling in the area of the site with the lowest contaminant probability and proceed to the areas of highest known or suspected contamination. Following this procedure will add another measure of quality control keeping cross contamination interference to a minimum.

All equipment utilized for sampling must be decontaminated using distilled and deionized water. Through distillation, all ionized solids and a broad range of organic constituents will be removed, thus making it an ideal solvent for use when sampling for organic parameters. Deionized water is water that has been effectively freed from any existing ionic impurities. The use of distilled and deionized water, commonly available from commercial vendors, is acceptable provided that the lot number and the associated analysis are available upon request to the NJDEP, and, it meets ASTM Type II specifications.

There are four individual decontamination procedures from which to choose when preparing a sampling plan. Matrix, level of contamination and programmatic considerations drive selection. The Eight-Step, the Three-Step and a third, based upon US Army Corps of Engineers Cold Regions

Research and Engineering Laboratory studies, apply to aqueous and non-aqueous matrices for most levels of contamination encountered in New Jersey. The fourth, a synopsis of USGS procedures, applies specifically to the cleaning of ground and surface water sampling equipment when analysis for trace levels of inorganic, organic, biological or toxicity constituents and interference from extraneous sources of contamination must be highly controlled. This procedure, referred to here as “Ultra Clean,” is treated separately from the others in this section. There should be no crossover or mixing of procedures once an approval process is finalized. The application of these procedures has the concurrence of USEPA Region II Monitoring Management Branch.

### 2.4.1 Eight-Step Decontamination Procedure For Aqueous and Non-Aqueous Sampling Equipment – Laboratory Only

This procedure is based, in part, upon the American Society for Testing and Materials, *Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites*, number D 5088-90. The first step, a detergent and water wash, is to remove all visible particulate matter and residual oils and grease. This may be preceded by a steam or hot water, high pressure water wash to facilitate residual removal. A generous tap water rinse and a distilled and deionized water rinse to remove the detergent follow this. If aqueous sampling is to be performed, the following additional steps must be completed. An acid rinse, included if metals samples are to be collected, provides a low pH media for trace metals removal. It is followed by another distilled and deionized water rinse. If the sample is not to be analyzed for metals, the acid rinse and water rinse can be omitted. Next, a high purity solvent rinse is designated for trace organics removal. Acetone has been chosen because it is an excellent solvent, miscible in water and is not a targeted analyte in Priority Pollutant Analysis. If acetone is known to be a contaminant at a given site or Target Compound List analysis is to be performed, Methanol or another solvent may be substituted on a case by case basis with approval from NJDEP. Note, methanol can not be used when sampling gasoline and its by-products. The solvent must be allowed to evaporate and then a final distilled and deionized water rinse is performed. This rinse removes any residual traces of the solvent.

The field sampling equipment cleaning and decontamination procedures are as follows:

- Laboratory grade glassware detergent plus tap water wash
- Generous tap water rinse
- Distilled and deionized (ASTM Type II) water rinse
- 10% nitric acid rinse (trace metal or higher grade HNO<sub>3</sub> diluted with distilled and deionized (ASTM Type II) H<sub>2</sub>O)
- Distilled and deionized (ASTM Type II) water rinse\*
- Acetone (pesticide grade) rinse\*\*
- Total air dry or pure nitrogen blow out\*\*
- Distilled and deionized (ASTM Type II) water rinse\*\*

All sampling equipment decontaminated via this procedure must be laboratory cleaned, wrapped and/or sealed, and dedicated to a particular sampling point or location during a sampling episode. In instances where laboratory cleaning is not feasible, permission for field cleaning must be obtained from the NJDEP prior to the collection of any samples and be referenced in the approved quality assurance project plan. Sampling devices should be numbered in a manner that will not

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\*Only if sample is to be analyzed for metals.

\*\*Only if sample is to be analyzed for organics.

affect their integrity. Equipment should be custody sealed and information concerning decontamination methodology, date, time, and personnel should be recorded in the field logbook.

The use of distilled and deionized water commonly available from commercial vendors may be acceptable for sampling equipment decontamination. NJDEP may require specific lot numbers from containers or analytical verification that the distilled and deionized water meets ASTM Type II specifications.

Hexane is not a necessary solvent for dioxin, PCB, or other chlorinated organic sampling. The cleaning procedure outlined above is adequate for all sampling episodes. In those instances where acetone is a parameter of concern another solvent may be used. All substitutes must be approved by NJDEP.

In the field, decontamination should be carried out over a container and the material properly disposed off-site. Decontamination wastes must be disposed of properly.

#### 2.4.2 Three-Step Equipment Decontamination Procedure Non-Aqueous Matrix Only – Laboratory and Field

While it is preferred that all non-aqueous field sampling equipment be laboratory cleaned, wrapped, and dedicated to a particular sampling point or location during a sampling episode, field cleaning may be more practical. Refer to the general field decontamination considerations above. The first step, a detergent and water wash, is to remove all visible particulate matter and residual oils and grease. This may be preceded by a steam or high pressure water wash to facilitate residual removal. A generous tap water rinse and a distilled and deionized water rinse to remove the detergent follow this. If visual contamination persists, or gross contamination is suspected, the full eight-step decontamination procedure is required.

The field sampling equipment cleaning and decontamination procedures are as follows:

- Laboratory grade glassware detergent and tap water scrub to remove visual contamination
- Generous tap water rinse
- Distilled and deionized (ASTM Type II) water rinse

All sampling equipment decontaminated via this procedure must be wrapped and/or sealed during storage and prior to use. Wherever possible, sampling devices should be numbered in a manner that will not affect their integrity. Information concerning decontamination methodology, date, time, and personnel should be recorded in the field logbook.

The use of distilled and deionized water commonly available from commercial vendors may be acceptable for sampling equipment decontamination. NJDEP may require specific lot numbers from containers or analytical verification that the distilled and deionized water meets ASTM Type II specifications.

In the field, decontamination should be carried out over a container and the residual liquid material must be properly disposed. Decontamination wastes must be disposed in accordance with current NJDEP policy (see Chapter 2, Section 2.4.5.7, *Disposal of Development, Purge, Pump Test and Decontamination Water*).

When analysis for metals is required it may be necessary to use carbon steel split spoon sampling devices instead of stainless steel. If this is the case and it is necessary to utilize the acid rinse for removal of visible contamination, the nitric acid rinse may be lowered to a concentration of 1% instead of 10% so as to reduce the possibility of leaching metals from the spoon itself.

### 2.4.3 US Army Cold Regions Research and Engineering Laboratory Decontamination Procedures for Use Primarily on Water Sampling (or Ground-Water Sampling) Equipment – Laboratory and/or Field Exclusively for Organics Including Pesticides

Extensive study, by the US Army Corps of Engineer's Cold Regions Research and Engineering Laboratory in New Hampshire, has affirmed what many have suspected regarding certain aspects of solvent use during the decontamination process and sorptive/desorptive properties of commonly used materials during contaminant exposure. Paraphrasing, their conclusions indicate that if sampling equipment is not decontaminated, there will be significant carry over ( $\mu\text{g/L}$ ) of hydrophilic and hydrophobic organic contaminants for both permeable and non-permeable materials. They also found that organic contaminants (including pesticides) could be removed from non-permeable stainless steel and rigid PVC surfaces using a hot detergent wash and DI water rinse thereby eliminating the commonly practiced step of an acetone, methanol or hexane solvent rinse.

Other polymeric materials, such as other plastics or various fluoropolymers such as polytetrafluoroethylene (PTFE) or Teflon, were generally less readily decontaminated. Decontamination of polymers is a function of analyte; rigidity, porosity or sorptive nature of the material; and contact time for sorption and desorption. A hot water detergent wash and distilled (DI) water rinse removed organic contaminants from less sorptive rigid PVC however, more sorptive PTFE required additional oven drying to remove selected VOCs. Oven drying speeded diffusion of adsorbed contaminants out of the polymer.

Their findings strongly suggest that solvent rinsing for organic contaminant removal (use of acetone, methanol or hexane) may not be necessary for devices of stainless steel and rigid PVC construction. They did note that removal of pesticides from low-density polyethylene was aided somewhat by solvent use, however the hot water detergent wash procedure followed by hot air oven drying outperformed solvent use.

Hot air oven drying is a departure from currently accepted procedures and is offered here as a new alternative, if the following steps are performed without exception. Exposure of ground water sampling equipment to hot air drying must be conducted over a 24-hour period for most pieces of equipment. Temperatures must be maintained at  $110^{\circ}\text{C}$  (approx.  $230^{\circ}\text{F}$ ). This includes devices of polymer construction such as bailers and bladder pumps. In the field, an air-drying oven can be set up inside a trailer or building to facilitate this logistical consideration. For ground water sampling pumps, check with the manufacturer for heat tolerance of sealed internal electrical parts or size and shape distortion tolerances for bladder pumps constructed of permeable materials.

Sampling equipment constructed of polymers may be heat sensitive in terms of distortion tolerance (USACE observed warping in the oven although they did not observe any problems when rigid PVC was heated). When distortion or uneven heat distribution are of concern, the use of a hot-water ( $100^{\circ}\text{C}$ ) high-pressure washer may offer an alternative to hot water/heated drying. Hot-water ( $100^{\circ}\text{C}$ ) high-pressure cleaning may be applied to large dimensional sampling equipment constructed of stainless and/or carbon steel equipment typically associated with direct push sampling technology. Sampling equipment, whether rigid PVC, stainless steel, or other permeable plastic materials, exposed to neat compounds or contaminants at high concentrations pose limitations to the effectiveness of this, or any, decontamination technique. This specific procedure is considered most effective when contaminant concentrations are 100 parts per million or less. If this decontamination procedure is the chosen method in instances of equipment exposure to contaminant levels above 100 ppm, then the collection rate of quality control field (equipment) blanks must be increased. For rigid PVC or stainless steel sampling equipment, collect an additional field (equipment) blank if organic concentrations in the last sample collected exceeded 100 ppm.

These decontamination procedures are not applicable to any forms of tubing, as USACE has never demonstrated this technique as an effective means to decontaminate tubing of any construction material.

The field sampling equipment cleaning and decontamination procedures are as follows:

- For Permeable Polymeric Materials (Teflon<sup>®</sup>, Teflon<sup>®</sup>-lined PE, Polyethylene)
  - Laboratory grade glassware detergent and hot (approx. 100°C) DI water scrub to remove visual contamination from extruded or machine shaped pieces.
  - Generous DI water rinse for extruded or machine shaped pieces.
  - Exposure to hot air (117°C) drying for 24 hour period.
- For Rigid PVC and Stainless Steel
  - Laboratory grade glassware detergent and hot (approx. 100°C) DI water scrub to remove visual contamination.
  - Generous DI water rinse
  - Optional use of hot-water (100°C) high-pressure washing<sup>1</sup>

#### 2.4.4 Ultra Clean Sampling Equipment Decontamination – Laboratory or Field

In certain cases when contaminant and general chemistry levels are being measured at their respective lowest method detection levels and the end user requires analytical data that must be free from any conceivable sample equipment interference, this cleaning method may be considered. These procedures, currently used by the US Geological Society for cleaning most ground and surface water sampling equipment, are not typically used by the Department's Site Remediation Program. Most NJDEP site investigations document levels of contamination that are above the lowest detection levels and have data quality objective plans which assure sampling equipment interference can be quickly identified and rectified. "Ultra Clean" procedures are designed to address contaminants not normally associated with SRP investigations e.g., inorganic indicators of water quality like cobalt, copper, zinc, manganese and iron. Therefore, the most likely SRP-use scenarios would include measurement of those lowest of contaminant concentration investigations where long term trends of environmental and ambient sensitive constituents are being monitored, e.g. parameters associated with Monitored Natural Attenuation. However, the Department's Bureau of Freshwater and Biological Monitoring, whose main focus and objectives are more aligned with the USGS and the NJGS, does routinely use these cleaning procedures. Their investigations of surface water are geared to monitor long terms changes of inorganic, organic, biological and general chemistry constituents whose sensitive analytical nature dictate the use of such an intensive decontamination procedure. The cleaning procedures outlined and paraphrased below, are taken from the USGS *National Field Manual for the Collection of Water-Quality Data, Book 9, Chapter A3* and are not presented here in their 65 page entirety. For complete details visit the USGS Internet address: ([http://water.usgs.gov/owq/FieldManual/chapter3/Ch3\\_contents.html](http://water.usgs.gov/owq/FieldManual/chapter3/Ch3_contents.html)). If Internet access is not available, contact the USGS @ 412 National Center, 12201 Sunrise Valley Drive, Reston, VA 20192.

When sampling for inorganic analysis, do not use cleaning agents or items that might leach or sorb metals. Basins, brushes and other items used for cleaning should be constructed of a suitable

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<sup>1</sup>Hot water (100°C) high-pressure washing of large dimensional rigid PVC, stainless steel and direct push technology sampling equipment is acceptable.

nonmetallic material such as uncolored or white polypropylene, polyethylene or other plastic. When sampling for organic analysis do not use cleaning agents or items that might leach, sorb or leave residues of organic substances that could bias or interfere with the analysis.

All cleaning equipment is subject to specified cleaning procedures prior to use on sampling equipment. Simplified, wash basins and standpipes are filled with non-phosphate detergent solution whereupon wash bottles, scrub brushes and other small items are placed within and soaked for 30 minutes. All items are then subject to a tap and DI water rinse, followed by a 30 minute soak in 5% HCl. (and/or 10% HNO<sub>3</sub>). Acid solutions are neutralized before disposal and cleaning equipment given several final DI rinses before air-drying. Visit the above Internet address for more details.

Surface and ground water sampling equipment is subject to a very precise cleaning procedure. As stated above, contaminant exposure levels sampled by the USGS are often at the low end of the method detection level and the focus is research oriented. The procedures below, for the cleaning of submersible pumps and submersible pump tubing, are taken from Section 3.3.9.B of Book 9, Chapter 3 of the *USGS National Field Manual*. The procedures are divided into Office-laboratory cleaning and Field-site cleaning. For more discussion on cleaning other sampling devices visit the above Internet address.

### **Office-laboratory Cleaning**

Fluorocarbon-polymer tubing used to collect water containing large concentrations of volatile organic compounds (VOC) can be difficult to clean adequately.

- Collect additional blanks if VOC concentrations in last sample collected through the tubing were greater than 500 µg/L.
- Pump tubing should be replaced rather than cleaned if VOC concentrations in last sample exceeded about 700 µg/L.
- Most submersible pumps have a stainless steel casing and other metal parts and should not be acid rinsed.
- To clean pumps that are excessively contaminated, a dilute acid rinse followed by copious water rinsing can be used occasionally without damaging the pump.
- Repeated rinsing with dilute acid solution can pit or corrode the pump's stainless steel surface.
- If the surface appears dulled, the pump must not be used for collecting trace-metal samples.

Lubrication water inside water-lubricated pumps (for example, the Grundfos RediFlo2™) can become contaminated and cause contamination of subsequent samples. Replace the lubrication water with volatile organic blank water each time after sampling and when cleaning the pump. Follow the manufacturer's instructions.

### **Step 1. Preparation.**

- Wearing appropriate gloves, prepare several gallons of a laboratory-grade nonphosphate detergent solution (about 0.1 or 0.2 percent, v/v; use up to 2-percent solution for excessively contaminated pump systems).
- Preclean washbasins and standpipes.
- Place pump into sink or washbasin and scrub exterior surfaces with soft brush and detergent solution; rinse thoroughly with tap water.
- Disassemble the pump and place components into a detergent-solution washbasin.

| <b>Table 2.1 Ultra Clean Supplies for Water Sampling Device Cleaning</b> |   |
|--|---|
| <b>Item</b>  | <b>Description</b>  |
| Acid Solution  | Hydrochloric: American Chemical Society trace element grade (5 percent by volume in distilled/deionized water). Hydrochloric is required if analyzing for nitrogen species. Nitric: ACS trace element grade (10 percent by volume in distilled/deionized water).                        |
| Aluminum foil  | Organics only: Heavy duty, for work surfaces and equipment  |
| Bags, plastic or fluorocarbon<br>Polymer Noncolored plastic sheeting     | Sealable bags with uncolored closures strips Recyclable trash bags for large equipment storage. Clean sheeting used to provide a clean work surface.  |
| Brushes and sponges  | Uncolored; plastic components needed for inorganic work.  |
| Distilled/deionized water  | Maximum specific electrical conductance, 1 µS/cm.   |
| Detergent  | Nonphosphate laboratory soap.   |
| Gloves, disposable   | Powerless, noncolored vinyl, latex or nitrile (latex or nitrile for use with methanol).   |
| Inorganic grade blank water  | Blank water with certificate of analysis prepared and/or quality assured by the analyzing laboratory.   |
| Jerricans or carboys   | For waste solutions and as neutralization container. Neutralization container: 25- to 30-liter polyethylene, wide mouth, with layer of marble chips.  |
| Methanol   | ACS pesticide grade.  |
| Neutralization materials   | Marble landscape chips. (Agricultural limestone, soda ash, baking soda or crushed shells not recommended.)  |
| Pesticide-grade blank water,<br>Volatile-grade blank water               | Blank water prepared and/or quality assured by the analyzing laboratory; required for collecting blank samples for respective blank sample analysis.<br>Pesticide or volatile grade blank water can be used for Total Organic, Dissolved Organic and Suspended Organic Carbon analysis. |
| Standpipes for submersible pumps   | Plastic, glass or other suitable material; for example, Pipette jars or capped PVC casing; one standpipe labeled for blank water and one each for each cleaning solution. (Do not use PVC with methanol).   |
| Tapwater   | If quality is questionable, substitute distilled/deionized. Tapwater is more effective for initial and rapid removal of detergent residue.  |
| Tissues  | Laboratory grade, lint free.  |
| Washbasins   | One washbasin for each cleaning solution; white or uncolored plastic, nonleaching. (Stainless steel for methanol)   |
| Wash bottles   | Labeled to indicate contents.<br>Fluorocarbon polymer needed for methanol, and pesticide, volatile and inorganic blank water.   |

USGS, Handbooks for Water Resources Investigations, Book 9, *National Field Manual for the Collection of Water-Quality Data, Chapter A3.*

**Step 2. Detergent wash and tap water rinse pump components and tubing.**

- Soak pump components in the detergent solution for 30 minutes.
- Scrub pump components with soft sponge or brush.
- Rinse thoroughly with tap water.

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- Raise discharge end of tubing above the rest of the tubing. Using a peristaltic or valveless fluid-metering pump, fill the pump tubing with fresh detergent solution until solution rises to the end of the tubing. Plug the tubing end(s).
- After 30 minutes remove plug from discharge end of tubing and flush detergent solution from tubing by pumping copious amount of tap water through the tubing. Change gloves.

### Step 3. Check sampling requirements.

- If pump will be used for collecting samples for inorganic constituent analysis, reassemble the pump and go to Step 4.
- Complete Step 4 if pump will be used for collecting samples for analysis of both inorganic and organic analytes before proceeding to Step 5.
- If the pump will be used for collecting samples for organic compound analysis only, go to Step 5.

### Step 4. DIW rinse.

- Place pump components into washbasin and dispense DIW from a wash bottle to thoroughly rinse all pump components.
- Using a peristaltic pump and appropriate clean tubing, pump DIW through the sample tubing to rinse.
- Reassemble pump and connect pump tubing. Change gloves.
- If collecting equipment blanks to verify that the pump has been adequately cleaned.
- Rinse a clean standpipe dedicated to blank water with blank water.
- Insert pump into blank-water standpipe only after pump exterior has been rinsed with blank water or air-dried after the methanol rinse.
- Pour IBW into the standpipe and pump at least one tubing volume to waste before collecting the blank sample.

### Step 5. Rinse with blank water followed by a methanol rinse.

- Change to latex or nitrile gloves. Put pump components into solvent-resistant washbasin.
- Working under a fume hood, dispense methanol (or appropriate solvent) from a fluorocarbon-polymer wash bottle to rinse each pump component and the exterior pump casing. Collect the used solvent into a nonflammable container for storage until disposal.
- Do not reuse methanol or other solvents.
- Work under a fume hood, if possible, or in a well-ventilated area outside of the office laboratory, as methanol fumes can contaminate other equipment.
- Place methanol-rinsed components on a clean, aluminum foil surface and allow the pump components and casing to completely air dry before reassembling the pump.
- Using a valveless fluid metering pump and fluorocarbon-polymer tubing, pump about 2 L of methanol through sample tubing and to the methanol waste container.
- Reassemble the pump and connect the pump tubing. Change gloves and dispose of the methanol-contaminated gloves appropriately.
- Pour an organic-grade water (PBW or VBW) into a clean PBW/VBW standpipe. Insert pump and pass about two tubing volumes of organic grade blank waster (PBW or VBW) through the pump and tubing to waste.

**Field-site cleaning procedure for submersible pumps and pump tubing.****Step 1. Preparation.**

- Preclean the standpipes (one standpipe for each cleaning solution to be used). The standpipes need to be of sufficient height to supply necessary head for proper pump operation. Separate standpipes are designated for detergent solution and tap water rinse, DIW rinse, methanol rinse, and blank water. Double-bag each cleaned standpipe for transport to the field site.
- Estimate the volumes of cleaning solution and blank water that will be needed for the field effort. The volume of storage in tubing,  $V_s$ , of a set of pump-reel and extension tubing can be estimated as follows:

$$V_s = [(L_p \times C_p) + (L_e \times C_e) + V_{sp}] \times C_{sp}$$

Where,

$V_s$  is the volume of storage in tubing, in gallons.

$L_p$  is length of pump-tubing segment being cleaned, in feet

$L_e$  is length of extension tubing, in feet

$C_p$  (or  $C_e$ ) = 0.023 liter per foot for a 3/8-inch tubing inside-diameter

Or = 0.041 liter per foot for a 1/2-inch ID tubing

$V_{sp}$  is volume of solution needed to fill standpipe to minimum level required to operate pump, in liters

$C_{sp}$  = 0.264 gallon per liter.

- Prepare the volumes of cleaning solutions needed for the field effort, using appropriate bottles for short-term storage and transport.

**Step 2. Detergent wash and tap water rinse.**

- Put on disposable, powderless gloves (usually vinyl). Rest pump in a washbasin or pail partially filled with detergent solution and clean exterior of pump and tubing with a soft brush. Rinse thoroughly with tap water. (DIW can be substituted for tap water, but is less efficient in detergent removal and requires a greater volume of water than tap water.)
- Place pump into standpipe, add detergent solution to level above pump intake, and route intake and discharge end of pump tubing to the standpipe.
- Begin pumping:
  - Record the pumping rate.
  - Record the time it takes to fill the sample tubing.
  - Calculate the time it takes for a segment of solution to complete one cycle.
- Circulate detergent solution for about three cycles through the tubing and back to the standpipe. If possible, pump detergent solution through tubing at alternating high and low speed, and (or) introduce air segments between aliquots of the detergent solution to increase cleaning efficiency.
- Remove the discharge end of tubing from the standpipe and pump about two tubing volumes of detergent solution to waste, adding fresh solution to the standpipe as needed. Remove pump from standpipe.
- Rinse detergent from standpipe with tap water until sudsing stops.
- Rinse pump exterior with tap water. Place rinsed pump into standpipe; add tap water/DIW to level above pump intake. Begin pumping through sample tubing. Do not recirculate rinse water,

but add water as needed to maintain water level above pump intake. Continue for five or more tubing volumes. Direct rinse water to waste away from the vicinity of the wellhead and sampling area and (or) contain as required for disposal.

- Collect rinse water into a small bottle and stop the pump. Shake the bottle – if sudsing is observed in the rinse water, continue the rinse procedure until no suds appear in the rinse water. Change gloves.

### Step 3. Check sampling requirements.

- If pump will be used for collecting samples for inorganic constituent analysis, reassemble the pump and go to Step 4.
- Complete Step 4 if pump will be used for collecting samples for analysis of both inorganic and organic analytes before proceeding to Step 5.
- If the pump will be used for collecting samples for organic compounds analysis only, go to Step 5.

### Step 4. DIW rinse.

- A separate DIW rinse is not required if DIW was substituted for tap water.
- Use a clean DIW-dedicated standpipe, not the tap water standpipe, and rinse with DIW. Rinse pump exterior with DIW to remove any detergent residue. Place pump into the DIW standpipe and add DIW to level above pump intake. Change gloves.
- Start pumping DIW. Rinse DIW through sample tubing without recirculating, using about 3 tubing volumes of DIW. Keep the DIW level above pump intake.
- Collect DIW rinse water in a clean bottle, shake, and check for suds. Continue to DIW rinse until rinse water is free of suds.
- If collecting field blanks to verify that the pump has been adequately cleaned:
- Change gloves. Rinse clean blank-water standpipe with IBW. Rinse pump exterior with blank water.
- Place pump into the standpipe and add IBW to cover the pump intake.
- Turn on pump and displace any water residing in the pump and tubing. Continue pumping IBW for one tubing volume before collecting the blank sample.

### Step 5. Methanol rinse.

- Make certain that the pump or other nearby electrically powered equipment is grounded, the power cord is intact, and potential sources of sparks do not exist before rinsing pump with methanol.
- Change to latex or nitrile gloves. Wear safety glasses and apron. Work in a well-ventilated area outside of the field van and downwind of the sampling area.
- Place pump into a clean, dedicated, solvent-resistant standpipe and route discharge end of sample tubing to a methanol waste container. Add methanol solution to level above pump intake.
- Pump about 2 L of methanol through sample tubing into methanol waste container, keeping the level of solution above pump intake. The operator should stand back from the pump as a safety precaution in the event that an electrical spark ignites the methanol. Carefully put any unused

methanol from the bottom of standpipe into methanol waste container. Let methanol in the standpipe evaporate to dryness. Change gloves.

- Rinse pump exterior with organic-grade water and place pump into standpipe. Add organic-grade water to the standpipe to push the methanol out of the tubing and into the methanol waste container. Pump at least an additional 0.1 gallon (about 0.38 L) of organic-grade water through the system for every 10 ft. (about 3.05 m) of methanol-wetted tubing to the methanol waste container after used methanol is collected.
- Repeat the above with blank water (PWB or VBW) pumped from a blank-water standpipe if blank samples will be collected for analysis of organic compounds.
- Storage of the cleaned submersible pump and tubing:
- Place pump into two clean, noncontaminating storage bags and close bags.
- Cover the pump reel and tubing with doubled plastic bag or sheeting for transport to the next site.
- For long-term storage (longer than 3 days), the pump and exterior and interior of the tubing must be dry before being placed into plastic bags. Blowing filtered air or filtered (inert) gas through the tubing can dry tubing. If tubing cannot be dried, store chilled to prevent bacterial growth. If bacterial growth has occurred, reclean before use.

#### 2.4.5 General Decontamination Considerations

The following discussion is intended to assist personnel engaged in the decontamination of select equipment. Unless otherwise stated, use one of the above four decontamination procedures as it relates to the device's aqueous or non-aqueous nature and the sampling objectives.

##### 2.4.5.1 Decontamination of Pumps

###### 2.4.5.1.1 Purging Only

###### 2.4.5.1.1.1 Submersible

When submersible pumps (gear, reciprocating, progressive cavity or centrifugal) are only used to evacuate stagnant ground water in the well casing (volume-average sampling), they must be cleaned and flushed prior to and between each use. This cleaning process consists of an external laboratory grade glassware detergent wash and tap water rinse, or steam cleaning of pump casing and cables, followed by a 20

#### Technical Note:

Inspect the integrity of the seals and O-rings on the pump-motor/pump-body housing. Water inside the motor housing may indicate that methanol vapors could enter the motor. Direct-current motors inherently spark because of the commutator ring. AC motors might spark if the insulation is frayed or burnt on the motor windings or any associated wiring.

If flammable liquids are required for cleaning electrical pump systems, use extreme caution. Vapors from solvents such as methanol can ignite if a disruption in the motor lead-insulation system occurs in the vapor-enriched zone. (Ignition from a spark from an AC induction-type motor in good operating condition is not a concern if rated as using the National Electrical Code (NEC) at Class 1, Group 5.)

gallon flush of potable water through the pump. This flushing can be accomplished by the use of a clean plastic overpack drum or a plastic garbage can filled with potable water. This must be followed by a distilled and deionized rinse of the outside of the pump. For submersible pumps smaller than four inches in diameter, the recommended number of gallons required for flushing may be proportionately reduced (i.e. three-inch 15-gallons, two-inch 10-gallons). For Grundfos® Redi Flo 2 pumps, follow the manufacturer's *Installation and Operating Instruction* manual for cleaning the inside of the stator housing by completely removing the motor shaft and in order to achieve a *complete* replacement of motor fluid (distilled/deionized water). Pumps constructed of plastic parts or sealed inner workings are not an equipment option for consideration because of their limited ability to be decontaminated thoroughly and their demonstrated ability to sorb and desorb contaminants.

Exercise caution to avoid contact with the pump casing and water in the drum while the pump is running (do not use metal drums or garbage cans) to avoid electric shock. Always disconnect the pump from power source before handling. Surface pumps (centrifugal and diaphragm) used for well evacuation need not be cleaned between well locations if a check valve is used. New tubing should be used for each well and discarded after use. If the evacuation tubing is not disposed between locations, it must also be decontaminated in the same manner as the pump. The submersible pump and tubing should always be placed on clean polyethylene sheeting to avoid contact with the ground surface. All tubing must be rinsed/wiped with distilled and deionized water and paper towels to remove any residual material during installation. (Refer to ASTM D-5088-90, *Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites.*)

#### 2.4.5.1.1.2 Surface Centrifugal and Diaphragm Pumps

When surface centrifugal and/or diaphragm pumps are used for purging, there is no need for decontamination of the pump or diaphragm housings. It is, however, a good practice to flush the housing/diaphragms with potable water between wells in order to control the build up of silt or other debris inside the housing/diaphragm. This practice will prolong the life of the pumps and maintain operating efficiency by reducing the potential for excessive wear.

#### 2.4.5.1.2 Sampling

##### 2.4.5.1.2.1 Submersible (Low Flow Purging and Sampling Method)

The importance of proper pump decontamination for sampling can not be stressed enough because the pump and tubing form the sampling equipment. Proper decontamination is especially true for pumps that are rented and utilized on a well-to-well basis (typically variable speed submersible centrifugal). Never assume that rented pumps have been thoroughly cleaned. Pumps constructed of plastic parts or sealed inner workings are not an option for LFPS consideration because of their limited ability to be decontaminated thoroughly.

Almost all pumps have an individual aspect that requires attention to detail when it comes to decontamination. One such pump, the variable speed 2-inch submersible, is a popular choice for well-to-well sampling; however, close attention to decontamination is warranted. One manufacturer, Grundfos®, clearly states in their operational handbook that for thorough cleaning, the pump must be completely disassembled,

including removal of the motor shaft from the stator housing and all components within the impeller housing. Care must be taken upon reassembly to insure the cavity housing the motor shaft is *completely* refilled with distilled deionized water. Care must also be taken with this pump during periods of cold weather to avoid freezing of the coolant water. Proper decontamination not only helps to ensure more reliable data; it also prolongs the life of the pump.

#### 2.4.5.1.2.2 Bladder Pumps

Most bladder pumps can not be easily decontaminated in the field due to their unique construction. For that reason bladder pumps **are not** employed for sampling on a well-to-well basis **unless** they are constructed with easy to clean parts and *disposable* bladders. Bladder pumps with non-disposable bladders are best suited for dedicated (permanently installed) scenarios. If they are constructed with disposable bladders, proper decontamination should include exchanging the used bladder and a thorough eight-step decon procedure.

#### 2.4.5.2 Decontamination of Heavy Equipment

Heavy equipment associated with a sampling episode must be cleaned prior to usage. Items such as drill rigs, well casing, auger flights, and backhoes all present potential sources of interference to environmental samples. These items may come in contact with the materials adjacent to the matrix being sampled or may be attached to actual sampling equipment that has been cleaned in accordance with procedures set forth above. Heavy equipment may potentially retain contaminants from other sources such as roadways, storage areas or from previous job sites and not have been removed. In addition to initial on-site cleaning, these items must be cleaned between use at each sample location. (Refer to ASTM D-5088-90).

Two options are available to accomplish cleaning of heavy equipment: steam cleaning and manual scrubbing. The use of a steam generator can remove visible debris and has several advantages. Steam generators using potable water provide a heated and high-pressure medium that is very effective for residuals removal. They are also efficient in terms of ease of handling and generate low volumes of wash solutions. Potential disadvantages include the need for a fixed or portable power source and they may not be cost effective for use on small pieces of equipment or for one day sampling events.

A second option involves manual scrubbing of equipment using a solution of laboratory grade glassware detergent followed by a thorough water rinse. This procedure can be as effective as steam cleaning or preferred in situations where steam cleaning fails to remove visible materials. The disadvantages to manual scrubbing include intensive labor and generation/disposal of wash and rinse solutions.

The above requirements for cleaning heavy equipment should be incorporated into Field Sampling – Quality Assurance Project Plans where applicable.

#### 2.4.5.3 Decontamination of Direct Push Equipment

Direct push technology can be applied to the collection of samples from aqueous and non-aqueous matrices. This versatility can be extended to samples collected for either fixed laboratory analysis or field analytical methods. Regardless of the sampling objectives, decontamination of the equipment can not be taken lightly since this equipment contacts the sample directly. At a minimum, to effectively clean the type of heavy equipment associated with the technology, a hot-water high-pressure system must be utilized after a pre-soap and water wash to clean all

equipment. Logistically, this will require additional support equipment to be on-site, typically a trailer with a “steam jenny” or equivalent and water tank capable of holding several tens of gallons of potable water. As with general heavy on-site equipment, all sampling equipment must be initially cleaned upon arrival at the site and again between each sample location. If vertical delineation is driving the investigation, each interval must be sampled with decontaminated equipment.

This decontamination process follows closely the US Army Corps of Engineers Cold Regions cleaning procedure. If the required sampling involves collection strictly from a non-aqueous source, the decontamination procedure may be abbreviated to the Three Step procedure discussed in Section 2.4.2 of this chapter. If however, heavy organics are visibly encountered and a hot-water high-pressure system is not on-site, then incorporation of solvents (e.g. acetone) must be included into the decontamination procedure. For large heavy equipment this will require large amount of the solvent to be on-site and consideration for drying time and disposal must also be factored. In addition, if the Three-Step procedure is chosen over the USACE method, additional field (equipment) blanks beyond the normal QA/QC requirement should be considered.

All decontamination should take place in an area removed from close proximity to all sample locations. Consideration for disposal of spent decontamination fluids must be made prior to site activity. In most instances use of hot-water high-pressure systems generates limited volumes of decontamination fluids and if those fluids can be controlled from leaving the site or from creating an erosion issue, then adsorption back into the soil is generally acceptable. Only in cases where contamination may threaten to leave the site or when creation of a possible erosion issue is unavoidable should containerization of fluids be considered.

#### 2.4.5.4 Decontamination of Monitor Well Casing and Screen

Before installation, field cleaning of well casing must consist of a manual scrubbing to remove foreign material and steam cleaning, inside and out, until all traces of oil and grease are removed. Special attention to threaded joints may be necessary to remove cutting oil or weld burn residues. The casing should then be handled and stored in such a manner so as to prevent cross contamination prior to installation.

#### 2.4.5.5 Cleaning Location

It is preferred, given site-specific conditions, that cleaning of all equipment take place in one central location on-site. A designated area or decontamination pad should be established to conduct all cleaning. All equipment such as drill rigs, backhoes, and other mobile equipment should receive an initial cleaning *prior* to use at a site. The frequency of subsequent cleanings while on-site will depend on how the equipment is actually used in relation to taking environmental samples. Unless otherwise specified and approved, all wash/rinse solutions should be collected and contained on-site. The actual fate of this material will be determined after review of analytical data generated from samples and on site discharge impacts have been evaluated.

#### 2.4.5.6 Disposal of Drill Cuttings

During the routine course of site investigation, where materials are known (via field instrumentation or visual observation) or suspected (historic information) to be contaminated, sampling activity (i.e., soil boring or installation of monitoring wells) will produce waste intrinsic to the site. The disposition of this material must be carried out in a manner such as not to contribute further environmental degradation or pose a threat to public health or safety.

Contaminated material may be disposed of on-site provided:

- that the soils are deemed to be non-hazardous;
- the disposed soil/water will not erode/flow either off-site or on-site onto uncontaminated areas;
- no potential to contaminate an uncontaminated aquifer exists, and;
- the potential to create a health hazard to adjoining property owners through airborne exposure is non-existent.

If any of the above conditions cannot be met on-site, the materials must be placed in containers (drums, rolloffs, etc.) and stored in a secure area of the site (fenced or access by unauthorized persons prevented) or transported to a central, secured location. The need to perform analyses of the secured material will be determined by NJDEP. The material will be retained for remediation or disposal in accordance with regulations as part of the selected site remedy.

When test pits are utilized for investigation, the excavated material may be returned to the hole. If the test pit is excavated below the water table, contaminated soils may not be returned to the test pit excavation below the water table; certified clean fill must first be used to bring the bottom of the test pit above the level of the water table. Holes produced from soil borings are to be grouted in accordance with the “Subsurface and Percolating Waters Act,” N.J.S.A. 58:4A-4.1 et seq., their implementing regulations (N.J.A.C. 7:9D-1.1 et seq.) and any NJDEP-approved changes to these specifications including repeals, new rules and amendments. Holes less than 25 feet in depth may be filled with sufficient quantities of uncontaminated soil material to make up for the amount of soil sampled and account for settling, thus allowing the hole to return to natural grade.

When materials of a noncontaminated nature are to be disposed of on-site, the following guidelines must be considered:

- disposed cuttings, soil or water will not erode or flow off-site;
- disposed water will not flow through an area of contamination and thereby spread it to a clean area; or
- NJDEP approves the disposal procedures.

Finally, at off-site (i.e., background) locations where no contamination is expected, the primary consideration is the wish of the property owner. If acceptable to the property owner, drill cuttings and mud from well installation may be raked into adjacent soils. If the property owner requests the uncontaminated material be removed from the site, it is to be properly contained and removed to the site under investigation and disposed of or stored per decision of the NJDEP. If drill cuttings and/or development water are expected to be contaminated, they are to

be removed from the off-site location to a secure on-site location and retained for remediation or disposed of per applicable regulations.

#### 2.4.5.7 Disposal of Installation, Development, Purge, Pump Test and Decontamination Waters

Similar to drill cuttings, an initial determination as to whether these wastewaters should be considered contaminated should be made by evaluating field instrumentation readings or by previous analytical information. Additional field-tests to assist in that determination (e.g., pH, color, other physical or chemical characterizations) must be utilized to the maximum extent possible.

Essentially, water generated that is not considered to be contaminated may be re-applied directly to the ground surface and permitted to percolate back into the ground water system. Care should be taken, however, to avoid nuisance situations where the discharge may cause undue concern on the part of property owners or the community. In such cases, it is advisable to dispose of the water into a local stormwater or sanitary sewer system, or collect and discharge the water slowly to avoid such a condition. Please note that all discharges of pollutants to surface water and/or the sanitary sewer are subject to the permit requirements contained in the NJPDES regulations.

Where the water is considered to be contaminated, the water generated may be re-applied to the ground surface provided all the following conditions are met:

- The water is not permitted to migrate off-site.
- There is no potential for contaminating a previously uncontaminated aquifer (for example, the discharge will not be permissible if a lower aquifer is being tested and is contaminated while the upper aquifer is not).
- The discharge will not cause an increase to ground surface soil contamination.
- If the above conditions cannot be met, the water shall be collected and secured at a single location (preferably the primary site under investigation).
- Collected water may be subsequently re-applied to ground surface only if, based on analytical results, there are indications that the above conditions can be met. If not, arrangements for proper disposal must be accomplished prior to the event.

In addition to the above considerations, the requirements of the New Jersey Pollutant Discharge Elimination System (NJPDES) must be followed for all discharges of pollutants to ground water and stormwater. The NJPDES Regulations requires the issuance of either an individual or general permit, or a permit-by-rule authorization (see N.J.A.C. 7:14A-7.5), for these discharges. If an individual NJPDES Discharge to Ground Water permit has already been issued for the facility, all discharges from the development and sampling of monitoring wells, done in accordance with the permit, are deemed to have a permit-by-rule without any additional written approval required [see N.J.A.C. 7:14A-7.5(a)4]. A NJPDES DGW permit-by-rule may also be available at other facilities for on-site disposal of development, purge, pump test and decontamination waters generated during the course of a site remediation. The most current NJPDES regulations at N.J.A.C. 7:14A-7.5 must be consulted. An unofficial version of the NJPDES regulations can be accessed via the NJDEP web site at: <http://www.nj.gov/dep/dwq/714a.htm>, however it may not include the most recent changes. Department staff familiar with the most recently promulgated regulations should be consulted.

It is preferable to complete discharges of development, purge, and decon waters at a single, known contaminated area on-site. This area will be selected by the NJDEP. In cases where such

an area cannot be located, as with contaminated well field projects, discharges will occur as close to the well or sampling location as reasonably possible.

## 2.5 Procedures For Quality Assurance and Quality Control (QA/QC)

QA/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 currently required by the NJDEP are designed to address QA/QC concerns related to sample bottle and equipment preparation, packaging, handling, and sample collection technique.

The trip blank (field reagent blank) is primarily 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. However, various USEPA Drinking Water Methods require the collection of field reagent blanks for non-volatile methods. Additionally, non-aqueous samples collected utilizing methanol preservation techniques may require trip blank analysis. Anticipated trip blank collection should be stated in the QAPP to avoid confusion in the field and possible rejection of data. For aqueous sampling, the trip blank water should be from the same source as the method blank water used in the laboratory during analysis. Regardless of whether the trip blank is associated with aqueous or non-aqueous samples, it is never opened and travels to and from the site with the empty or full sample bottles in an effort to simulate sample-handling conditions. Contaminated trip blanks may also indicate inadequate bottle cleaning or blank water of questionable quality.

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. The following have been identified as potential sources of contamination.

- 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. Since the field blank travels and is stored with the sample bottles, and 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 cleaned sampling device (where applicable), the field blank is indicative of ambient conditions and/or equipment conditions that may potentially affect the quality of the associated samples.

The primary purpose of this type of blank is to provide an additional check on possible sources of contamination beyond that which is intended for trip blanks. A field blank serves a similar purpose as a trip blank regarding water quality and sample bottle preparation. However, it is primarily used to indicate potential contamination from ambient air as well as from sampling instruments used to collect and transfer samples from point of collection into sample containers (it may also be referred to in the literature as a field rinsate blank).

The following is a breakdown by matrix of blank sample requirements.

### 2.5.1 Non-Aqueous Matrix

#### 2.5.1.1 Field Blanks

##### 2.5.1.1.1 Description

The performance requirement for field blank collection begins with two (2) sets of identical bottles (method dependent); one set filled with demonstrated analyte free water provided by the laboratory performing the sample analysis, and one empty set of bottles. The bottles should also be identical to those provided for aqueous sample collection. Note: Since field blanks are aqueous; the lab must provide water for volatile analysis in 40ml septum vials. For soil preserved in methanol see Chapter 6 Section 6.2.7.4.5, *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 and only need to be collected and analyzed for volatile organics when volatile organics constitute a parameter being investigated. On a site-specific basis, QA parameter requirements may be amended at the discretion of NJDEP. Note: for logistical purposes it is recommended that the lab provide at least one extra full 40ml vial to perform the field blank.

Various USEPA Methods such as, USEPA Method 1631 *Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry* requires the use of additional field blanks as well as equipment blanks as part of the method requirements. Therefore, the analytical methods should be reviewed to determine method requirements.

##### 2.5.1.1.2 Frequency

For sampling events lasting more than one day, field blanks associated with a non-aqueous matrix should be performed at a rate of 10% of the total number of samples collected throughout the event. If, for example, 40 samples were to be collected over a six-day period, then only four field blanks would be required. For one-day sampling events, with the total number of samples collected being less than 10, it is required that one field blank be collected. On a site-specific basis, QA frequency requirements may be amended at the discretion of NJDEP. However, it is not necessary to collect more than one field blank per day.

##### 2.5.1.2 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.

### 2.5.2 Aqueous Matrix

#### 2.5.2.1 Field Blanks

A description of field blanks for the aqueous matrix is the same as 2.5.1.1.1 above with one exception: Field blanks must be analyzed for all the same parameters that the samples collected will be analyzed.

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.

Field Blanks may be required to detect cross contamination from ambient air during potable sampling events if known sources of contamination are within close proximity or monitoring instruments indicate the presence of contamination above background levels.

Frequency – Field blanks for the aqueous matrix must be performed at a rate of one per day.

Various USEPA Methods such as, USEPA Method 1631 *Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry* requires the use of additional field blanks as well as equipment blanks as part of the method requirements. Therefore, the analytical methods should be reviewed to determine the method requirements

### 2.5.2.2 Trip Blanks (Field Reagent Blanks)

#### 2.5.2.2.1 Description

Trip blanks are required for aqueous sampling events. They consist of a set of sample bottles filled at the laboratory with laboratory demonstrated analyte free water. Trip blanks accompany sample bottles into the field and are returned to the laboratory along with the collected samples for analysis. These bottles are never opened. Trip blanks must return to the lab with the same set of bottles they accompanied to the field. At a minimum, trip blanks must be analyzed for volatile organic parameters. The inclusion of additional parameters or amendments to the requirements for trip blanks is at the discretion of NJDEP. Trip blanks and associated samples shall not be held on-site for more than two calendar days unless prior agreement to extend the sampling handling time has been granted by the oversight program.

#### 2.5.2.2.2 Frequency

Trip blanks must be included at a rate of one per sample shipment (not to exceed two (2) consecutive field days). However, USEPA has issued analytical methods that require additional Trip Blanks or each batch of twenty samples submitted to the laboratory. Therefore, the analytical methods must be reviewed prior to determining the required number of Trip Blanks.

### 2.5.3 Air Matrix

Trip and field blank procedures and frequencies for the various air sampling methods available should follow the specifications of the individual analytical method utilized. QA sample requirements may be amended at the discretion of NJDEP.

### 2.5.4 Blank Water Quality

The 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,

- 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).

Lab certification requirements for the source of blank/method water can be found in Section 7:28-3.3-9 of the *Regulations Governing the Certification of Laboratories and Environmental Measurements* N.J.A.C. 7:18. Basically, it states that 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. If the water of the required quality is not produced in the environmental laboratory, it shall be purchased from commercial suppliers. The environmental laboratory shall maintain a field of the required analysis for each lot of water. A source of purified water is not necessary for radon/radon progeny-in-air analyses.

The laboratory performing the analysis may be required to provide documentation that trip and field blank water was demonstrated analyte free if contamination is detected in blanks, or at NJDEP's discretion. This would be verified by analytical results of method blanks run by the laboratory on the day of trip and field blank preparation and shipment. This does not, however, change requirements for the analysis of method blanks on the day of sample analysis at the laboratory.

A method blank is carried through the entire sample preparation procedure and analysis at the laboratory. It is utilized as a check on laboratory procedures as well as possible contamination from laboratory equipment (i.e. reagents, glassware, etc.).

### 2.5.5 Sample Handling and Holding Times

#### 2.5.5.1 Handling Time

Field and trip blank samples must travel with sample containers and must arrive on-site within one day of their preparation in the lab. Blanks and their associated samples may be held on-site for no longer than two calendar days, and must arrive back in the lab within one day of shipment from the field. This constitutes the maximum 4-day handling time. Exceptions to this NJDEP QA/QC imposed requirement may be granted by managers overseeing a project when legal holidays or weekend sampling conflict with laboratory shipment practices. Without exception, blanks and all samples must be maintained at 4°C while stored on-site and during shipment. Sample bottles and blanks must be handled in the same manner prior to their return to the laboratory.

The only standing exception, which requires no prior approval to handling time requirements, is when sampling storm water runoff. The spontaneity of storm conditions precludes any possibility for preplanning sample bottle shipment. Therefore, due to these obvious logistical constraints, trip and field blanks are not normally required.

While the exception is understandable, the storage of these sample bottles must be carefully controlled to ensure the possibility of cross contamination is kept to an absolute minimum.

#### 2.5.5.2 Maximum Holding Time

The clock governing 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). Holding times for individual parameters are dictated by the specific analytical method being used. The holding time clock for all other certified methods and parameters begins at the time of sample collection in the field. Please refer to the Tables at the end of this chapter for additional information.

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 may be found on the USEPA Website at <http://www.epa.gov/epahome/cfr40.htm>.

Current Requirements 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 3<sup>rd</sup> Edition* issued 1996 and amended and may be found on the USEPA Website at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

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 <http://www.epa.gov/oerrpage/superfund/programs/clp/index.htm>

#### 2.5.6 Special Analytical Services (SAS)

It is important to note that both trip and field blanks are only capable of determining that contamination of samples may have occurred from additional sources other than the actual environmental matrix being investigated. They cannot identify, but may suggest, possible sources of additional contaminant contribution to the reported analytical values. NJDEP may also implement additional types of QA/QC blanks (Special Analytical Services – SAS) when initial sampling episodes produce blank contamination that cause the generated data to become suspect. SAS has been developed to help identify the specific source(s) of blank contamination in a particular analytical fraction to aid in the assessment of reported analytical values. To date its primary purpose has been to confirm or eliminate laboratory contaminant sources in blank samples.

An example of special analytical services may be the inclusion of additional blank samples that are prepared at the same time and in the same manner as the trip and field blanks, but 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. SAS may also include the use of alternate analytical methodologies for unique, site specific parameters of concern.

It is important to note that many methods have additional quality control requirements that have evolved to monitor both storage and analytical procedures. All parties, including the laboratory, must 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.

Two recent changes to consider are as follows

- The USEPA CLP Program in the Low/Medium Level Organics and the Low Concentration Organics Statement of Works require the analysis of instrument blanks and storage blanks. The purpose of instrument blanks is to determine the level of contamination associated with the instrumental analysis itself, particularly with regard to the carry over of analytes from standards or highly contaminated samples into other analyses. The storage blank indicates whether contamination may have occurred during storage of samples. A storage blank is used for volatile organics, and upon receipt of the first sample in a Sample Delivery Group or a batch, two 40 mL screw-cap volatile vials with a PTFE- faced silicone septum are filled with reagent water. The vials are stored with the samples in the Sample Delivery Group under the same conditions. After all samples in the Sample Delivery Group have been analyzed, the storage blank is analyzed.
- The USEPA Method 1631 requires the preparation and analysis various blanks not normally found in other method. Three of the blanks are described below of bubbler blanks, bottle blanks,

blanks and reagent blanks. A bubbler blank is used to demonstrate freedom from system contamination. A least three bubbler blanks must be run per analytical batch by placing a clean gold trap on the bubbler immediately following analysis of a sample, and analyzing the sample a second time. Filling a sample bottle with reagent water acidified to pH <2, capping the bottle, allowing the bottle to stand for a minimum of 24 hours and analyzing the water generates a bottle blank. A reagent blank is generated by adding aliquots of BrCl,  $\text{HN}_2\text{OH}_2$ , and  $\text{SNCL}_2$  to previously purged reagent water in the bubbler and analyzing the reagents water. Reagents blanks are used to identify contamination from the reagents.

#### 2.5.7 Additional QA/QC Samples

Additional parameter blanks may be required in specific cases. NJDEP may make such a determination during review of the site-specific sampling portion of a project plan.

##### 2.5.7.1 Duplicate Samples Obtained in the Field (Field Duplicates)

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 are to be included for each matrix at a minimum rate of one for every twenty samples (5% of total) and be submitted to the lab as "blind" samples. If less than twenty samples are collected during a particular sampling episode, one duplicate should be performed. Duplicate requirements may be waived or expanded depending on the particular regulatory program or remedial phase involved. Keep in mind that various USEPA Methods require a higher frequency of Field Duplicate Samples. Therefore, the analytical methods must be reviewed to determine the appropriate number of Field Duplicates.

###### 2.5.7.1.1 Aqueous Matrix Duplicates

Duplicates of water samples (potable well, monitor well, surface water) should be obtained by alternately filling sample containers from the same sampling device for each parameter. Samples for volatile organics analysis from monitor 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 re-used, the vials for volatile organics should be alternately filled. If heterogeneity is suspected, separate samples of each phase should be collected as the nature of phased liquids precludes homogenization. It is generally not necessary to homogenize ground water or surface water samples.

###### 2.5.7.1.2 Non-Aqueous Matrix Duplicates

Obtaining duplicate samples in a soil or sediment matrix requires homogenization of the sample aliquot prior to filling sample containers. Regardless, volatile organic samples must always be taken from discrete locations or intervals without compositing or mixing. This practice is necessary to prevent loss of volatile constituents and to preserve, to the extent practicable, the physical integrity of the volatile fraction (see Chapter 6. *Sample Collection*, Section 6.2.7, *VOC Sample Collection for Soils*, for further information). Homogenization of the sample for remaining parameters is necessary to generate two equally representative samples. Note that enough sample must be collected at one time in order to fill all 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 filling a properly decontaminated stainless steel tray or bowl with the sample and mixing it with a decontaminated stainless steel or Teflon<sup>®</sup> instrument. The extent of mixing required will depend on the nature of the sample and should be done to achieve a consistent physical appearance prior to filling sample containers.

Once mixing is completed the sample should be divided in half and scooping sample material alternately from each half should fill containers. Several laboratory methodologies for compositing samples published by the American Society for Testing and Materials (ASTM) 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 particular methodology. Therefore, these methods are not recommended for generating duplicate samples in the field.

#### 2.5.7.2 Splitting Samples with Responsible 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.

In order to maintain the integrity of any sample “split” between interested parties, the following procedures shall be followed:

- Personnel authorized by NJDEP (e.g. contractors or treatment facilities) using approved NJDEP sampling methods shall be permitted to obtain all sample aliquots.
- Other interested parties must provide their own sample containers, blank samples, preservatives, sample shuttles, chain of custody forms, etc.
- NJDEP personnel shall witness the sampling procedures to verify consistent handling and packaging of each set of samples.
- Duplicate samples, trip blanks and field blanks must be included as part of those samples, which are split between the two or more parties, involved.
- All interested parties desiring to obtain split samples during planned sampling episodes must provide the Department with a minimum of two weeks notice. This is essential for planning purposes and to avoid confusion or delays in the field.
- Use of the same analytical methods must be conducted between all parties in order to allow for comparability of data. Choice of analytical methodologies must be agreed upon prior to the sampling event.

#### 2.5.7.3 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.

## **2.6 Sample Preservation Requirements**

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Certain analytical methodologies for specific analytes require chemical additives in order to stabilize and maintain sample integrity. Generally this is accomplished under two scenarios:

- Preservative may be added to the sampling bottles by the laboratory prior to shipment into the field or,
- Preservatives are added in the field immediately after the samples are collected.

Many laboratories provide pre-preserved bottles as a matter of convenience and to help ensure that samples will be preserved immediately upon collection. A problem associated with this method arises if not enough sample is collected, resulting in too much 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. NJDEP approves the use of pre-preserved bottles. However, field-sampling teams must always check the pH level and be prepared to add additional preservatives to samples if necessary.

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

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

- Sampling teams must be properly equipped to conduct preservation of samples in the field. To accomplish this task the following items are necessary:
  - Graduated pipettes
  - Pipette bulbs
  - Preservatives in glass containers with their content and concentration clearly labeled
  - Limited range pH paper (important that the sampler note 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 must also be properly equipped with appropriate health and safety equipment. Use of and immediate access to the following items are strongly recommended:
  - Protective goggles
  - Disposal gloves
  - Lab apron
  - First aid kit
  - Portable eye wash station
  - Containerized 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 must be familiar with specified preservation requirements and verify that the necessary pH level has been achieved. To accomplish this task, a small amount of the preserved sample aliquot should be placed into a separate clean beaker or the container lid. The liquid should then be checked with pH paper so as to indicate that the desired pH level has been achieved. Under no circumstances should the test sample aliquot be returned into the container retaining the sample for analysis.
- Preservation requirements are method and parameter specific. Additional information may be found in Appendix 2-1 following this chapter. These charts may indicate any additional preservation required upon arrival of samples at the laboratory as cited in the specific methodologies. The laboratory and the samplers are required under the certification regulations to know the additional preservation requirements. The source of preservatives is also of concern. They 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.
- Preservation must take place immediately upon sample collection except when samples are to be filtered. Samples requiring filtration must be processed immediately after collection. Filtered samples are then preserved immediately following the filtration process.
- In rare cases a chemical reaction between the preservative and an aqueous sample may induce effervescence. Should this be observed during sample collection, immediately notify both the laboratory and the oversight program before continuing. A decision will have to be rendered in the field regarding whether or not to continue sample collection. If expeditious shipping and laboratory analysis of an unpreserved sample can be negotiated (based on analytical method requirements) in order to maintain sample integrity, the sample should be discarded, the interior of the sample container rinsed with the sample source and an unpreserved sample volume collected. The fact the sample is unpreserved must be noted on the chain of custody 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 notified that an unpreserved field sample is being submitted. Some methods, such as USEPA Method 624, specifies that if an unpreserved sample is submitted, it must be analyzed with 7 days of sample collection. Therefore, it is important to the sampler and the laboratory to be knowledgeable about the analytical methods
- Samples must be placed into a cooler and maintained at 4°C immediately upon collection and preservation.

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

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

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 may be found on the USEPA Website at: <http://www.epa.gov/epahome/cfr40.htm>.

Current Requirements 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* -

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*Physical and Chemical Methods 3rd Edition* issued 1996 and amended and may be found on the USEPA Website at: <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

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: <http://www.epa.gov/oerrpage/superfund/programs/clp/index.htm>

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- <http://www.epa.gov/oerrpage/superfund/programs/clp/index.htm>
- <http://www.state.nj.us/dep/srp/regs/techrule>
- [http://water.usgs.gov/owq/FieldManual/chapter3/Ch3\\_contents.html](http://water.usgs.gov/owq/FieldManual/chapter3/Ch3_contents.html)
- <http://water.usgs.gov/owq/FieldManual/mastererrata.html#Chapter3>

## **Appendix 2.1 Tables of Analytical Methods**

The tables in this section are similar to those found in the New Jersey Regulations Governing the Certification of Laboratories and Environmental Measurements N.J.A.C. 7:18. The tables were updated to reflect the current methodology changes and new methods that have been added since the Regulations have been written. These tables are provided for guidance only if there is a conflict between the Tables and the information provided by the Office of Quality Assurance or their regulations, the Office of Quality Assurance information or decision always takes precedent over the tables. Note: Throughout these tables “P or G” in the Container column means “Plastic or Glass, either soft or hard” respectively with the exception of Fluoride which is polyethylene only. Footnotes appear on the last page of this Appendix.

**Table 2.2 Required Preservation, Container, and Maximum Holding Times for Drinking Water Samples, Except Radiochemical Parameters**

| Parameter   | Preservation   | Container                            | Maximum Holding Time                                      |
|---|--|--------------------------------------|---|
| Total Coliform Finished Drinking Water            | Cool 4°C, 0.008% sodium thiosulfate (Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> ) | P or G                               | 30 hours  |
| Heterotrophic Plate Count Finished Drinking Water | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>                       | P or G                               | 8 hours   |
| Total Coliform Source Water                       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>                       | P or G                               | 8 hours   |
| Fecal Coliform Source Water                       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>                       | P or G                               | 8 hours   |
| Cryptosporidium                                   | Cool 0-8°C, No Freezing  | LPDE Cubitainer                      | Elution must begin within 96hrs of sampling <sup>14</sup> |
| Giardi cysts                                      | Cool 0-8°C, No Freezing  | LPDE Cubitainer                      | Elution must begin within 96hrs of sampling <sup>14</sup> |
| Alkalinity  | Cool 4°C   | P or G                               | 14 days   |
| Antimony  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                               | 6 months  |
| Arsenic   | Conc. HNO <sub>3</sub> to pH < 2   | P or G                               | 6 months  |
| Asbestos  | Cool 4°C   | P or G                               | Filter within 48 hours                                    |
| Barium  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                               | 6 months  |
| Beryllium   | Conc. HNO <sub>3</sub> to pH < 2   | P or G                               | 6 months  |
| Bromate   | 50 mg/L Ethylenediamine (EDA) solution   | P or G                               | 28 days   |
| Bromide   | None   | P or G                               | 28 days   |
| Cadmium   | Conc. HNO <sub>3</sub> to pH < 2   | P or G                               | 6 months  |
| Calcium   | Conc. HNO <sub>3</sub> to pH < 2   | P or G                               | 6 months  |
| Chlorate  | 50 mg/L Ethylenediamine (EDA) solution   | P or G                               | 28 days   |
| Chloride  | None   | P or G                               | 28 days   |
| Chlorite  | 50 mg/L Ethylenediamine (EDA) solution Cool 4°C                                      | P or G                               | 14 days   |
| Chlorinated Hydrocarbons                          | Refrigerate at 4°C. After collection, Ascorbic acid                                  | Glass with foil or Teflon®-lined cap | 14 days until extraction; 40 days after extraction        |

Table 2.2 (continued) Required Preservation, Container, and Maximum Holding Times for Drinking Water Samples, Except Radiochemical Parameters

| <b>Parameter</b>                                | <b>Preservation</b>  | <b>Container</b>                       | <b>Maximum Holding Time</b>                        |
|---|--|--|--|
| Chlorinated Pesticides                          | 80mg/L Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual chlorine (Cl <sub>2</sub> ) is present, Cool 4°C            | Glass with Teflon®-lined septum        | 7 days until extraction; 14 days after extraction  |
| Chlorinated Phenoxy Acids                       | 80mg/L Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual chlorine (Cl <sub>2</sub> ) is present, Cool 4°C            | Glass with Teflon®-lined septum        | 14 days until extraction; 28 days after extraction |
| Chlorine Dioxide                                | None   | P or G                                 | Analyze Immediately                                |
| Chlorinated Acids                               | Refrigerate at 4°C after collection, Ascorbic acid   | Glass with foil or Teflon®-lined cap   | 7 days until extraction; 30 days after extraction  |
| Chromium  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                                 | 6 months   |
| Copper  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                                 | 6 months   |
| Cyanide   | NaOH to pH > 12, Cool 4°C, 0.6 g Ascorbic acid   | P or G                                 | 14 days  |
| EDB/DBCP/1,2,3-TCP                              | Cool 4°C, 0.08% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>  | Glass with Teflon®-lined septum        | extract: 14 days; 24 hours to analysis             |
| Fluoride  | None   | Polyethylene only                      | 28 days  |
| Free Chlorine Residual                          | None   | P or G                                 | Analyze Immediately                                |
| Lead  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                                 | 6 months   |
| Mercury   | Conc. HNO <sub>3</sub> to pH < 2   | P or G                                 | 28 days  |
| N-Methyl-Carbamoyloximes<br>N-Methyl-Carbamates | Monochloroacetic acid to pH 3, 80mg/L Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , Cool 4°C until storage, Store at -10°C | Glass with Teflon®-lined septum        | 28 days at -10°C                                   |
| Nickel  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                                 | 6 months   |
| Nitrate-Nitrate                                 | Conc. H <sub>2</sub> SO <sub>4</sub> to pH < 2; Cool 4°C   | P or G                                 | 28 days  |
| Nitrate-N                                       | Cool 4°C   | P or G                                 | 48 hours   |
| Nitrite-N                                       | Cool 4°C   | P or G                                 | 48 hours   |
| Nitrogen- and Phosphorus-Containing Pesticides  | 80mg/L Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> (if residual Cl <sub>2</sub> ) Cool 4°C                                 | Glass (dark) with Teflon®-lined septum | 14 days until extraction; 14 days after extraction |
| o-Phosphate                                     | Cool 4°C   | P or G                                 | 48 hours   |
| Perchlorates                                    | None Required  | P or G                                 | 28 days  |

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Table 2.2 (continued) Required Preservation, Container, and Maximum Holding Times for Drinking Water Samples, Except Radiochemical Parameters

| Parameter   | Preservation   | Container                       | Maximum Holding Time  |
|---|--|---------------------------------|---|
| Odor  | Cool 4°C   | P or G                          | 24 hours  |
| Organic Compounds   | If residual Cl <sub>2</sub> 40-50 mg sodium arsenite or sodium thiosulfate; if unchlorinated, 6 N HCl to pH < 2                            | Glass with Teflon®-lined septum | 7 days until extraction; 30 days after extraction   |
| Organohalide Pesticides and Commercial PCB Products (Arochlors) | 3mg Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> or 7uL Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> (0.04g/mL), Cool 4°C until analyzed | Glass with Teflon®-lined septum | If Heptachlor, 7 days until extraction; 40 days after extraction. If no extraction, analysis within 14 days |
| Ozone   | None   | G                               | Analyze Immediately   |
| pH  | None   | P or G                          | Analyze Immediately   |
| Selenium  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                          | 6 months  |
| Silver  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                          | 6 months  |
| Sodium  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                          | 6 months  |
| Sulfate   | Cool 4°C   | P or G                          | 28 days   |
| Temperature   | None   | P or G                          | Analyze Immediately   |
| Thallium  | Conc. HNO <sub>3</sub> to pH < 2   | P or G                          | 6 months  |
| TTHMs   | Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual Cl <sub>2</sub> and 6N HCl   | Glass with Teflon®-lined septum | 14 days   |
| Total Dissolved Solids  | Cool 4°C   | P or G                          | 7 days  |
| Turbidity   | Cool 4°C   | P or G                          | 48 hours  |
| Volatile Aromatic and Unsaturated Organic Compounds             | 1:1 HCl to pH < 2 Cool, 4°C until analysis, Ascorbic acid  | Glass with Teflon®-lined septum | 14 days   |
| Volatile Halogenated Organic Compounds                          | 1:1 HCl to pH < 2 Cool, 4°C until analysis, Ascorbic acid  | Glass with Teflon®-lined septum | 14 days   |
| Volatile Organic Compounds                                      | 1:1 HCl to pH < 2 Cool, 4°C until analysis, Ascorbic acid  | Glass with Teflon®-lined septum | 14 days   |

**Table 2.3 Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters**

| Parameter                              | Preservation  | Container      | Maximum Holding Time   |
|--|---|----------------|------------------------|
| <b>Bacterial Tests</b>                 |   |                |                        |
| Coliform (fecal)                       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup> | P, G           | 6 hours                |
| Coliform (total)                       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup> | P, G Cool      | 6 hours                |
| Fecal streptococci                     | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup> | P, G           | 6 hours                |
| Enterococci                            | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup> | P, G (sterile) | 6 hours                |
| Escherichia coli                       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup> | P, G (sterile) | 6 hours                |
| Heterotrophic Plate Count              | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup> | P, G (sterile) | 8 hours                |
| Salmonella sp. Bacteria                | Cool 4°C  | P, G (sterile) | 24 hours               |
| Helminth Ova                           | Cool 4°C  | P, G (sterile) | 24 hours               |
| Enteric Viruses <sup>13</sup>          | Cool 4°C  | P, G (sterile) | 8 hours                |
| <b>Toxicity Test</b>                   |   |                |                        |
| Acute or Chronic Toxicity              | Cool 4°C  | P, G           | 36 hours <sup>12</sup> |
| <b>Inorganic Tests</b>                 |   |                |                        |
| Acidity, as CaCO <sub>3</sub>          | Cool 4°C  | P, G           | 14 days                |
| Alkalinity as CaCO <sub>3</sub>        | Cool 4°C  | P, G           | 14 days                |
| Aluminum-total <sup>3</sup>            | HNO <sub>3</sub> to pH < 2  | P, G           | 6 months               |
| Ammonia (as N)                         | Cool 4°C H <sub>2</sub> SO <sub>4</sub> to pH < 2                           | P, G           | 28 days                |
| Antimony-total <sup>3</sup>            | HNO <sub>3</sub> to pH < 2  | P, G           | 6 months               |
| Arsenic-total <sup>3</sup>             | HNO <sub>3</sub> to pH < 2  | P, G           | 6 months               |
| Barium-total <sup>3</sup>              | HNO <sub>3</sub> to pH < 2  | P, G           | 6 months               |
| Beryllium-total <sup>3</sup>           | HNO <sub>3</sub> to pH < 2  | P, G           | 6 months               |
| Biochemical Oxygen Demand              | Cool 4°C  | P, G           | 48 hours               |
| Boron-total <sup>3</sup>               | HNO <sub>3</sub> to pH < 2  | P, G           | 6 months               |
| Bromide <sup>3</sup>                   | None required   | P, G           | 28 days                |
| Cadmium-total <sup>3</sup>             | HNO <sub>3</sub> to pH < 2  | P, G           | 6 months               |
| Calcium-total <sup>3</sup>             | HNO <sub>3</sub> to pH < 2  | P, G           | 6 months               |
| Carbonaceous Biochemical Oxygen Demand | Cool 4°C  | P, G           | 48 Hours               |

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Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| Parameter   | Preservation   | Container   | Maximum Holding Time                                    |
|---|--|---|---|
| Chemical Oxygen Demand (COD)                                      | Cool 4°C H <sub>2</sub> SO <sub>4</sub> to pH < 2                    | P, G  | 28 days   |
| Chloride  | None required  | P, G  | 28 days   |
| Chlorine total residual (TRC)                                     | None required  | P, G  | Analyze Immediately                                     |
| Chromium VI (dissolved)   | Cool 4°C   | P, G  | 24 hours  |
| Chromium-total <sup>3</sup>                                       | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Cobalt-total <sup>3</sup>   | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Color   | Cool 4°C   | P, G  | 48 hours  |
| Copper-total <sup>3</sup>   | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Cyanide-total <sup>3</sup>  | Cool 4°C, NaOH to pH > 12, 0.6g ascorbic acid                        | P, G  | 14 days (24 hours when sulfide is present) <sup>2</sup> |
| Cyanide amenable to chlorination <sup>3</sup>                     | Cool 4°C, NaOH to pH > 12, 0.6g ascorbic acid                        | P, G  | 14 days (24 hours when sulfide is present) <sup>2</sup> |
| Fluoride  | None required  | Polyethylene only   | 28 days   |
| Gold-total <sup>3</sup>   | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Hardness-total as CaCO <sub>3</sub>                               | HNO <sub>3</sub> to pH < 2 H <sub>2</sub> SO <sub>4</sub> to pH < 2, | P, G  | 6 months  |
| Hydrogen ion (pH)   | None required  | P, G  | Analyze Immediately                                     |
| Iridium-total <sup>3</sup>  | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Iron-total <sup>3</sup>   | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Kjeldahl & Organic Nitrogen                                       | Cool 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2                   | P, G  | 28 days   |
| Lead-total <sup>3</sup>   | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Magnesium-total <sup>3</sup>                                      | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Mercury-dissolved <sup>11</sup> (does not include methyl mercury) | 5mL/L of 12 N HCl or 5mL/L of 12 N BrCl<br>Cool 4°C                  | Fluoropolymer with fluoropolymer or fluoropolymer lined cap | 28 days   |
| Mercury-dissolved <sup>11</sup> (includes methyl mercury)         | 5mL/L of 12 N HCl<br>Cool 4°C  | Fluoropolymer with fluoropolymer or fluoropolymer lined cap | 28 days   |
| Mercury-total <sup>3</sup>  | HNO <sub>3</sub> to pH < 2   | P, G  | 28 days   |
| Mercury-total <sup>11</sup> (does not include methylmercury)      | 5mL/L of 12 N HCl or 5 mL/L of 12 N BrCl                             | Fluoropolymer with fluoropolymer or                         | 28 days   |

Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| <b>Parameter</b>                                     | <b>Preservation</b>  | <b>Container</b>  | <b>Maximum Holding Time</b> |
|--|--|---|-----------------------------|
|  | Cool 4°C   | fluoropolymer lined cap                                     |                             |
| Mercury-total <sup>11</sup> (includes methylmercury) | 5mL/L of 12 N HCl<br>Cool 4°C  | Fluoropolymer with fluoropolymer or fluoropolymer lined cap | 28 days                     |
| Molybdenum-total <sup>3</sup>                        | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months                    |
| Nickel-total <sup>3</sup>                            | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months                    |
| Nitrate (as N)                                       | Cool 4°C   | P, G  | 48 hours                    |
| Nitrate-Nitrite(as N)                                | Cool 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2                           | P, G  | 28 days                     |
| Nitrite (as N)                                       | Cool 4°C   | P, G  | 48 hours                    |
| Oil and grease                                       | Cool 4°C HCl or H <sub>2</sub> SO <sub>4</sub> to pH < 2                     | G   | 28 days                     |
| Organic carbon-total (TOC)                           | Cool 4°C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH < 2 or phosphoric acid | P, G  | 28 days                     |
| Orthophosphate (as P)                                | Filter Immediately, Cool 4°C   | P, G  | 48 hours                    |
| Osmium-total <sup>3</sup>                            | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months                    |
| Oxygen dissolved (probe)                             | None Required  | Glass bottle and top  | Analyze Immediately         |
| Oxygen dissolved (Winkler)                           | Fix on site and store in dark  | Glass bottle and top  | 8 hours                     |
| Palladium-total <sup>3</sup>                         | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months                    |
| Petroleum Hydrocarbons                               | HCl to pH 2  | G   | 7 days                      |
| Phenols  | Cool 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2                           | G only  | 28 days                     |
| Phosphorus (elemental)                               | Cool 4°C   | G   | 48 hours                    |
| Phosphorus-total                                     | Cool 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2                           | P, G  | 28 days                     |
| Platinum-total <sup>3</sup>                          | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months                    |
| Potassium-total <sup>3</sup>                         | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months                    |
| Residue-total  | Cool 4°C   | P, G  | 7 days                      |
| Residue-filterable (TDS)                             | Cool 4°C   | P, G  | 7 days                      |
| Residue-nonfilterable (TSS)                          | Cool 4°C   | P, G  | 7 days                      |
| Residue-settleable                                   | Cool 4°C   | P, G  | 48 hours                    |
| Residue-volatile                                     | Cool to 4°C  | P, G  | 7 days                      |
| Rhodium-total <sup>3</sup>                           | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months                    |
| Ruthenium-total <sup>3</sup>                         | HNO <sub>3</sub> to pH < 2   | P, G  | 6 months                    |

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Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| Parameter                        | Preservation  | Container  | Maximum Holding Time                              |
|----------------------------------|---|--|---|
| Salinity                         | Cool 4°C  | G  | 28 days   |
| Selenium-total <sup>3</sup>      | HNO <sub>3</sub> to pH < 2  | P, G   | 6 months  |
| Silica-dissolved                 | Cool 4°C  | P  | 28 days   |
| Silver-total <sup>3</sup>        | HNO <sub>3</sub> to pH < 2  | P, G   | 6 months  |
| Sodium-total <sup>3</sup>        | HNO <sub>3</sub> to pH < 2  | P, G   | 6 months  |
| Specific conductance             | Cool 4°C  | P, G   | 28 days   |
| Sulfate                          | Cool 4°C  | P, G   | 28 days   |
| Sulfide                          | Cool 4°C, add zinc acetate & NaOH to pH > 9   | P, G   | 7 days  |
| Sulfite                          | None required   | P, G   | Analyze Immediately                               |
| Surfactants                      | Cool 4°C  | P, G   | 48 hours  |
| Temperature                      | None required   | P, G   | Analyze Immediately                               |
| Thallium-total <sup>3</sup>      | HNO <sub>3</sub> to pH < 2  | P, G   | 6 months  |
| Tin-total <sup>3</sup>           | HNO <sub>3</sub> to pH < 2  | P, G   | 6 months  |
| Titanium-total <sup>3</sup>      | HNO <sub>3</sub> to pH < 2  | P, G   | 6 months  |
| Turbidity                        | Cool 4°C  | P, G   | 48 hours  |
| Vanadium-total <sup>3</sup>      | HNO <sub>3</sub> to pH < 2  | P, G   | 6 months  |
| Zinc-total <sup>3</sup>          | HNO <sub>3</sub> to pH < 2  | P, G   | 6 months  |
| <b>Organic Tests<sup>4</sup></b> |   |  |   |
| Acenaphthene <sup>7</sup>        | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> Store in dark                    | Glass, Teflon <sup>®</sup> -lined cap                  | 7 days until extraction; 40 days after extraction |
| Acenaphthylene <sup>7</sup>      | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>                                  | Glass, Teflon <sup>®</sup> -lined cap<br>Store in dark | 7 days until extraction; 40 days after extraction |
| Acrolein                         | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub><br>Adjust pH to 4-5 <sup>6</sup> | Glass, Teflon <sup>®</sup> -lined septum               | 14 days   |
| Acrylonitrile                    | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub><br>Adjust pH to 4-5 <sup>6</sup> | Glass, Teflon <sup>®</sup> -lined septum               | 14 days <sup>6</sup>                              |
| Anthracene <sup>7</sup>          | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>                                  | Glass, Teflon <sup>®</sup> -lined cap                  | 7 days until extraction; 40 days after extraction |
| Benzene                          | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub><br>HCl to pH 2                   | Glass, Teflon <sup>®</sup> -lined septum               | 14 days   |
| Benzidine <sup>7</sup>           | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>                                  | Glass, Teflon <sup>®</sup> -lined cap                  | 7 days until extraction <sup>8</sup>              |

Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| <b>Parameter</b>                         | <b>Preservation</b>   | <b>Container</b>            | <b>Maximum Holding Time</b>                          |
|--|---|-----------------------------|--|
| Benzo(a) anthracene <sup>7</sup>         | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub><br>Store in dark                         | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Benzo(a)pyrene <sup>7</sup>              | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Benzo(b) fluoranthene <sup>7</sup>       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Benzo(g,h,i) perylene <sup>7</sup>       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Benzo(k) fluoranthene <sup>7</sup>       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Benzyl chloride                          | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined septum | 14 days  |
| Benzyl butyl phthalate <sup>7</sup>      | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Bis(2-chloroethoxy) methane <sup>7</sup> | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Bis(2-chloroethyl) ether <sup>7</sup>    | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Bis(2-ethylhexyl) phthalate <sup>7</sup> | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Bromodichloro-methane                    | Cool 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup> , HCl to pH 2 <sup>5</sup>   | Glass, Teflon®-lined septum | 14 days  |
| Bromoform                                | Cool 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup>  | Glass, Teflon®-lined septum | 14 days  |
| Bromomethane                             | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| Carbon tetrachloride                     | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 4-Chloro-3-methylphenol <sup>7</sup>     | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Chlorobenzene                            | Cool 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup>  | Glass, Teflon®-lined septum | 14 days  |
| Chloroethane                             | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 2-Chloroethylvinyl ether                 | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| Chloroform                               | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |

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Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| Parameter                               | Preservation  | Container                   | Maximum Holding Time                                 |
|---|---|-----------------------------|--|
| Chloromethane                           | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 2-Chloronaphthalene <sup>7</sup>        | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| 2-Chlorophenol <sup>7</sup>             | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| 4-Chlorophenylphenyl ether <sup>7</sup> | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Chrysene <sup>7</sup>                   | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Dibenzo (a,h)anthracene <sup>7</sup>    | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Dibromochloro-methane                   | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 1,2-Dichloro-benzene <sup>7</sup>       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 1,3-Dichloro-benzene <sup>7</sup>       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 1,4-Dichloro-benzene <sup>7</sup>       | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 3,3'-Dichloro-benzidine <sup>7</sup>    | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined septum | 14 days  |
| Dichlorodifluoro-methane                | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined septum | 14 days  |
| 1,1-Dichloroethane                      | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 1,2-Dichloroethane                      | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 1,1-Dichloroethene                      | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| trans-1,2-Dichloro-ethene               | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| 2,4-Dichlorophenol <sup>7</sup>         | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| 1,2-Dichloropropane                     | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| cis-1,3-Dichloro-propene                | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |

Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| <b>Parameter</b>                       | <b>Preservation</b>   | <b>Container</b>            | <b>Maximum Holding Time</b>                          |
|--|---|-----------------------------|--|
| trans-1,3-Dichloro-propene             | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| Diethyl phthalate <sup>7</sup>         | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| 2,4-Dimethylphenol <sup>7</sup>        | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Dimethyl phthalate                     | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Di-n-butyl phthalate <sup>7</sup>      | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Di-n-octyl phthalate <sup>7</sup>      | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| 2,3-Dinitrophenol <sup>7</sup>         | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| 2,4-Dinitrotoluene <sup>7</sup>        | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| 2,6-Dinitrotoluene <sup>7</sup>        | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Epichlorohydrin                        | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined septum | 14 days  |
| Ethylbenzene                           | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined septum | 14 days  |
| Fluoranthene <sup>7</sup>              | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Fluorene <sup>7</sup>                  | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Hexachlorobenzene <sup>7</sup>         | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Hexachlorobutadiene <sup>7</sup>       | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Hexachlorocyclopentadiene <sup>7</sup> | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Hexachloroethane <sup>7</sup>          | Cool 4°C  | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Ideno(1,2,3-cd)pyrene <sup>7</sup>     | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |
| Isophorone <sup>7</sup>                | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap    | 7 days until extraction;<br>40 days after extraction |

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Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| Parameter                                  | Preservation  | Container                 | Maximum Holding Time                                 |
|--|---|---------------------------|--|
| Methylene chloride                         | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon®-lined cap  | 14 days  |
| 2-Methyl-4,6-dinitrophenol <sup>7</sup>    | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| Naphthalene <sup>7</sup>                   | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| Nitrobenzene <sup>7</sup>                  | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| 2-Nitrophenol <sup>7</sup>                 | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| 4-Nitrophenol <sup>7</sup>                 | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| N-Nitrosodimethylamine <sup>7, 10</sup>    | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| N-Nitrosodi-n-propylamine <sup>7, 10</sup> | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| N-Nitrosodiphenylamine <sup>7, 10</sup>    | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| 2,2'-Oxybis(1-chloropropane)               | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| PCB-1016 <sup>7</sup>                      | Cool 4°C  | Glass, Teflon®-lined cap, | 7 days until extraction;<br>40 days after extraction |
| PCB-1221 <sup>7</sup>                      | Cool 4°C  | Glass, Teflon®-lined cap, | 7 days until extraction;<br>40 days after extraction |
| PCB-1232 <sup>7</sup>                      | Cool 4°C  | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| PCB-1242 <sup>7</sup>                      | Cool 4°C  | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| PCB-1248 <sup>7</sup>                      | Cool 4°C  | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| PCB-1254 <sup>7</sup>                      | Cool 4°C  | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| PCB-1260 <sup>7</sup>                      | Cool 4°C  | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| Pentachlorophenol                          | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |
| Phenanthrene <sup>7</sup>                  | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon®-lined cap  | 7 days until extraction;<br>40 days after extraction |

Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| Parameter  | Preservation  | Container                                | Maximum Holding Time                                 |
|--|---|--|--|
| Phenol <sup>7</sup>                                | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| Pyrene <sup>7</sup>                                | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>Store in dark            | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| 2,3,7,8-Tetra-chlorodi-benzo-p-dioxin <sup>7</sup> | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| 1,1,2,2-Tetrachloro-ethane                         | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon <sup>®</sup> -lined septum | 14 days  |
| Tetrachloroethene                                  | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon <sup>®</sup> -lined septum | 14 days  |
| Toluene  | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon <sup>®</sup> -lined septum | 14 days  |
| 1,2,4-Trichloro-benzene <sup>7</sup>               | Cool 4°C  | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| 1,1,1-Trichloroethane                              | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon <sup>®</sup> -lined septum | 14 days  |
| 1,1,2-Trichloroethane                              | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon <sup>®</sup> -lined septum | 14 days  |
| Trichloroethene                                    | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon <sup>®</sup> -lined septum | 14 days  |
| Trichlorofluoro-Methane                            | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2 <sup>5</sup> | Glass, Teflon <sup>®</sup> -lined septum | 14 days  |
| 2,4,6-Trichloro-phenol <sup>7</sup>                | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup>                             | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| Vinyl chloride                                     | Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1</sup><br>HCl to pH 2              | Glass, Teflon <sup>®</sup> -lined septum | 14 days <sup>5</sup>                                 |
| <b>Pesticides Tests<sup>7</sup></b>                |   |  |  |
| Aldrin   | Cool 4°C, pH 5-9 <sup>10</sup>  | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| Ametryn  | Cool 4°C, pH 5-9 <sup>10</sup>  | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| Aminocarb  | Cool 4°C pH 5-9 <sup>10</sup>   | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| Atraton  | Cool 4°C, pH 5-9 <sup>10</sup>  | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |
| Atrazine   | Cool 4°C, pH 5-9 <sup>10</sup>  | Glass, Teflon <sup>®</sup> -lined cap    | 7 days until extraction;<br>40 days after extraction |

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Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| <b>Parameter</b>    | <b>Preservation</b>            | <b>Container</b>                      | <b>Maximum Holding Time</b>                          |
|---------------------|--------------------------------|---------------------------------------|--|
| Azinphos methyl     | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Barban              | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| alpha-BHC           | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| beta-BHC            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| delta-BHC           | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Gamma-BHC (Lindane) | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Captan              | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Carbaryl            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Carbophenothion     | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Chlordane           | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Chlorpropham        | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| 2,4-D               | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| 4,4'-DDD            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| 4,4'-DDE            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| 4,4'-DDT            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Demeton-O           | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Dementon-S          | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Diazinon            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Dicamba             | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |

Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| <b>Parameter</b>   | <b>Preservation</b>            | <b>Container</b>                      | <b>Maximum Holding Time</b>                          |
|--------------------|--------------------------------|---------------------------------------|--|
| Dichlofenthion     | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Dichloran          | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Dicofol            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Dieldrin           | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Dioxathion         | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Disulfoton         | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Diuron             | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Endosulfan I       | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Endosulfan II      | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Endosulfan Sulfate | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Endrin             | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Endrin aldehyde    | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Ethion             | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Fenuron            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Fenuron-TCA        | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Heptachlor         | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Heptachlor epoxide | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Isodrin            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Linuron            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |

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Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| <b>Parameter</b> | <b>Preservation</b>            | <b>Container</b>         | <b>Maximum Holding Time</b>                          |
|------------------|--------------------------------|--------------------------|--|
| Malathion        | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Methiocarb       | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Methoxychlor     | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Mexacarbate      | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Mirex            | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Monuron          | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Monuron-TCA      | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Nuburon          | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Parathion methyl | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Parathion ethyl  | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| PCNB             | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Perthane         | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Prometron        | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Prometryn        | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Propazine        | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Propham          | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Propoxur         | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Secbumeton       | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |
| Siduron          | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon®-lined cap | 7 days until extraction;<br>40 days after extraction |

Table 2.3 (continued) Required Preservation, Container, and Maximum Holding Times for Wastewater Samples and Solid/Hazardous Waste Samples (Aqueous Matrices), Except Radiochemical Parameters

| <b>Parameter</b>  | <b>Preservation</b>            | <b>Container</b>                      | <b>Maximum Holding Time</b>                          |
|-------------------|--------------------------------|---------------------------------------|--|
| Simazine          | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Strobane          | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Swep              | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| 2,4,5-T           | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| 2,4,5-TP (Silvex) | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Terbutylazine     | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Toxaphene         | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |
| Trifluralin       | Cool 4°C, pH 5-9 <sup>10</sup> | Glass, Teflon <sup>®</sup> -lined cap | 7 days until extraction;<br>40 days after extraction |

**Table 2.4 Required Preservation, Container and Maximum Holding Times for Radiochemical Measurements in Drinking Water and Wastewater Samples**

| <b>Parameter</b>  | <b>Preservation</b>                    | <b>Container</b> | <b>Maximum Holding Time</b> |
|---|--|------------------|-----------------------------|
| Gross alpha   | Conc. HCl or HNO <sub>3</sub> to pH 2* | P or G           | 6 months                    |
| 48-Hour Rapid Gross Alpha*  | Conc. HCl or HNO <sub>3</sub> to pH 2* | P or G           | 48-hours**                  |
| Gross beta  | Conc. HCl or HNO <sub>3</sub> to pH 2* | P or G           | 6 months                    |
| Strontium-89  | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 6 months                    |
| Strontium-90  | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 6 months                    |
| Radium (total )   | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 6 months                    |
| Radium-224  | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 4 days (recommended)        |
| Radium-226  | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 6 months                    |
| Radium-228  | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 6 months                    |
| Cesium-134/137  | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 6 months                    |
| Iodine-131  | None                                   | P or G           | 8 days                      |
| Tritium   | None                                   | G                | 6 months                    |
| Uranium   | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 6 months                    |
| Plutonium   | Conc. HCl or HNO <sub>3</sub> To pH 2  | P or G           | 6 months                    |
| Photon emitters (including Cobalt-60, Zinc-65, Ruthenium-106, and Barium-133) | Conc. HCl or HNO <sub>3</sub> to pH 2  | P or G           | 6 months                    |
| Radon-222***  | Cool 4°C                               | G                | 4 days (recommended)        |

Drinking water samples that are to be subject to radiochemical measurements shall be handled and preserved in accordance with the requirements of Table 2.4 and the requirements listed below. Table 2.4 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 Table 2.4 and the USEPA publication (including any amendments or supplements) on which any part of Table 2.4 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 CWS compliance monitoring, as well as testing for radium under private well testing Act (PWTA). Maximum holding time to initial counting of the plancheted sample shall be 48 hours from 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 Table 2.4. 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.

**Table 2.5 Required Preservation, Container and Maximum Holding Times for Radiochemical Measurements in Solid/Hazardous Waste Samples (Soils, Liquids, Sediments, and Sludges)**

| <b>Parameter</b>                                     | <b>Preservation</b> | <b>Container Volume</b> | <b>Maximum Holding Time</b> |
|--|---------------------|-------------------------|-----------------------------|
| Gross Alpha-Beta                                     | Cool to 4 ° C       | 1 liter                 | 6 months                    |
| Radium-Total   | Cool to 4 ° C       | 1 liter                 | 6 months                    |
| Radium-226   | Cool to 4 ° C       | 1 liter                 | 6 months                    |
| Radium-228   | Cool to 4 ° C       | 1 liter                 | 6 months                    |
| Photon Emitters: Co-60,<br>Zn-65, Cs-134/137, Ba-133 | Cool to 4 ° C       | 1 liter                 | 6 months                    |
| Strontium-89, 90                                     | Cool to 4 ° C       | 1 liter                 | 6 months                    |
| Uranium  | Cool to 4 ° C       | 1 liter                 | 6 months                    |
| Thorium  | Cool to 4 ° C       | 1 liter                 | 6 months                    |

Solid/hazardous waste samples (non-aqueous) shall be handled and preserved in accordance with requirements of Table 2.5. Table 2.5 incorporates requirements from SW-846. If there is any conflict between Table 2.5 and SW-846 (including any amendments or supplements), SW-846 shall prevail.

**Table 2.6 Required Preservation, Container and Maximum Holding Times for Solid/Hazardous Waste Samples (Soils, Liquids, Sediments, Sludges, and Ambient Air)**

| Parameter  | Preservation  | Container   | Maximum Holding Time  |
|--|---|---|---|
| Volatile Organics for soil/ sediment, and sludge   | Cool 4°C  | Glass Teflon®-lined cap   | 14 days   |
| Volatile Organics (Non-Aqueous sample)   | Cool 4°C, dark  | Encore™ or equivalent field core sampling/ storage containers & 60 ml septum sealed glass vial  | Transfer immediately upon receipt to methanol and sodium bisulfate solution, analyze within 14 days |
| Volatile Organics (Non-Aqueous sample)   | Cool 4°C, dark  | Field preserved vials methanol & sodium bisulfate Glass, 40 ml vial stir bar [sodium bisulfate only], septum sealed glass vial & 60 ml septum sealed glass vial | 14 days   |
| Volatile organics in liquid samples  | Cool 4°C, if residual Cl <sub>2</sub> add Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> and HCl to pH < 2 | Glass, Teflon®-lined cap  | 14 days   |
| Acrolein and Acrylonitrile in liquid samples   | Cool 4°C Adjust to pH 4-5   | Glass, Teflon®-lined cap  | 14 days   |
| Semivolatile organics/ organochlorine pesticides/ PCBs and herbicides for soil/sediment, and sludge  | Cool 4°C  | Glass, Teflon®-lined cap  | 14 days until extraction; 40 days after extraction  |
| Semivolatile organics/ organochlorine pesticides/ PCBs and herbicides for concentrated waste samples | Cool 4°C  | Glass, Teflon®-lined cap  | 14 days until extraction; 40 days after extraction  |
| Metals except Cr VI and Hg (total) for liquid samples  | Cool 4°C, HNO <sub>3</sub> to pH < 2  | P, G  | 6 months  |
| Metals except Cr VI and Hg (dissolved) for liquid samples  | Cool 4°C, Filter on-site HNO <sub>3</sub> to pH < 2   | P, G  | 6 months  |
| Metals except Cr VI and Hg (suspended) for liquid samples  | Cool 4°C Filter on-site   | P, G  | 6 months  |

Table 2.6 (continued) Required Preservation, Container and Maximum Holding Times for Solid/Hazardous Waste Samples (Soils, Liquids, Sediments, Sludges, and Ambient Air)

| <b>Parameter</b>  | <b>Preservation</b>   | <b>Container</b>             | <b>Maximum Holding Time</b>  |
|---|---|------------------------------|--|
| Metals except Cr VI and Hg for solid samples                                    | Cool 4°C  | P, G                         | 6 months   |
| Chromium VI for solid samples   | Cool 4°C  | P, G                         | 30 days to digestion; analysis 168 hours after digestion   |
| Chromium VI for liquid samples  | Cool 4°C  | P, G                         | 24 hours   |
| Mercury (total) for liquid samples  | HNO <sub>3</sub> to pH < 2  | P, G                         | 28 days  |
| Mercury (dissolved) for liquid samples  | Filter on-site HNO <sub>3</sub> to pH < 2   | P, G                         | 28 days  |
| Mercury (total) for solid samples   | Cool 4°C  | P, G                         | 28 days  |
| <b>Ambient Air Analysis</b>   |   |                              |  |
| TO-15 Volatile Organics in Specially Prepared Canisters – GC/MS                 | None  | Specially prepared canisters | 30 days from sample collection   |
| TO-17 Volatile Organics in Ambient Air using Active Sampling onto Sorbent Tubes | Cool ≤4°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                | 30 days from sample collection; except 7days if limonene, carene, labile sulfur, bischloromethylether or nitrogen containing volatiles |

**Table 2.7 Required Preservation, Container and Maximum Holding Times From VTSR for CERCLA-CLP Aqueous and Non-Aqueous Samples**

| <b>Parameter</b>   | <b>Preservation</b>  | <b>Container</b>  | <b>Maximum Holding Time From Validated Time of Sample Receipt (VTSR)</b>  |
|--|--|---|---|
| Volatile Organics (Aqueous)  | Cool 4°C, dark 0.08% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual Cl <sub>2</sub> | Glass, white polypropylene or black phenolic plastic screw Teflon®-lined septum   | 10 days   |
| Volatile Organics (Non-Aqueous)  | Cool 4°C, dark   | Glass, polypropylene cap, white Teflon® liner   | 10 days   |
| Volatile Organics (Non-Aqueous)  | Cool 4°C, dark   | Encore™ or equivalent field core sampling/ storage container & 60 ml septum sealed glass vial   | Transfer immediately upon receipt to methanol and sodium bisulfate solution analyze within 10 days                                      |
| Volatile Organics (Non-Aqueous)  | Cool 4°C, dark   | Field preserved vials methanol & sodium bisulfate glass, 40 ml vial stir bar [sodium bisulfate only], septum sealed glass vial & 60 ml septum sealed glass vial | 10 days   |
| Pesticide/PCBs   | Cool 4°C, dark   | Amber Glass, white polypropylene or black phenolic, baked polyethylene cap  | Extraction Aqueous: continuous liquid-liquid extraction must be started within 5 days, Non-Aqueous: 10 days analysis, 40 days from VTSR |
| Polychlorinated Dibenzop-Dioxins (PCDDs) and Dibenzofurans (PCDFs) (Non Aqueous) | Cool 10°C, dark  | Amber Glass, white polypropylene or black phenolic, baked polyethylene cap  | Extraction: 30 days from VTSR, analysis 45 days from extraction   |

Table 2.7 (continued) Required Preservation, Container and Maximum Holding Times From VTSR for CERCLA-CLP Aqueous and Non-Aqueous Samples

| <b>Parameter</b>   | <b>Preservation</b>  | <b>Container</b>   | <b>Maximum Holding Time From Validated Time of Sample Receipt (VTSR)</b>                                       |
|--|--|--|--|
| Polychlorinated Dibenzop-Dioxins (PCDDs) and Dibenzofurans (PCDFs) (Aqueous)                 | Cool 4°C, dark   | Amber Glass, white polypropylene or black phenolic, baked polyethylene cap | Extraction: 30days from VTSR, analysis: 45 days from extraction  |
| Polychlorinated Dibenzop-Dioxins (PCDDs) and Dibenzofurans (PCDFs) (Fish and Tissue Samples) | Cool 4°C, dark until prepared then-10°C until analysis   | Wrapped in aluminum foil in field  | Extraction: 1 year from VTSR. Once thawed, must be analyzed within 24 hours. Analysis: 45 days from extraction |
| Cyanide, total amenable to chlorination  | Aqueous - 0.6g ascorbic acid if residual Cl <sub>2</sub> , NaOH to pH>12, cool 4°C, CaCO <sub>3</sub> in presence of sulfide           | Plastic bottle, plastic cap, plastic liner                                 | 14 days  |
| Metals except Hg (Aqueous)   | HNO <sub>3</sub> to pH<2, cool 4°C, until analyzed   | Plastic bottle, plastic cap, plastic liner                                 | 180 days   |
| Metals – Dissolved except Hg (Aqueous)   | Field filter 0.45 µm pore diameter filter, rinse bottle with sample then immediately HNO <sub>3</sub> to pH<2, cool 4°C until analyzed | Plastic bottle, plastic cap, plastic liner                                 | 180 days   |
| Metals except Hg (Non-Aqueous)   | Cool 4°C until analyzed  | Flint glass bottle, black phenolic cap, polyethylene liner                 | 180 days   |
| Hg (Aqueous)   | HNO <sub>3</sub> to pH<2, Cool, 4°C until analyzed   | Plastic bottle, plastic cap, plastic liner                                 | 26 days  |
| Hg – Dissolved (Aqueous)   | Field filter 0.45 µm pore diameter filter, rinse bottle with sample immediately, HNO <sub>3</sub> to pH<2, Cool, 4°C until analyzed    | Plastic bottle, plastic cap, plastic liner                                 | 26 days  |
| Hg (Non-Aqueous)   | HNO <sub>3</sub> to pH<2, Cool, 4°C until analyzed   | Flint glass bottle, black phenolic cap, polyethylene liner                 | 28 days  |

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Table 2.7 (continued) Required Preservation, Container and Maximum Holding Time From VTSR for CERCLA-CLP Aqueous and Non-Aqueous Samples

| <b>Parameter</b>                   | <b>Preservation</b>   | <b>Container</b>   | <b>Maximum Holding Time From Validated Time of Sample Receipt (VTSR)</b>  |
|------------------------------------|---|--|---|
| Cyanide (Aqueous)                  | 0.6g ascorbic acid if residual $\text{Cl}_2$ NaOH to pH>12, cool 4°C until analyzed | Plastic bottle, plastic cap, plastic liner                                   | 14 days   |
| Cyanide (Non-Aqueous)              | Cool 4°C, until analyzed  | Plastic bottle, plastic cap, plastic liner                                   | 14 days   |
| Low Level Volatile Organics        | Cool 4°C, dark, 0.008% $\text{Na}_2\text{S}_2\text{O}_3$                            | Glass, black phenolic or white polypropylene screw cap, Teflon®-lined septum | 7 days  |
| Low Level Semi-volatile Organics   | Cool 4°C, dark  | White polypropylene or black phenolic, baked polyethylene cap                | Extraction: continuous extraction must be started within 5 days of VTSR. Analysis: 40 days from start of extraction |
| Low Level Pesticides/PCBs Organics | Cool 4°C, dark  | Amber glass, white polypropylene or black phenolic, baked polyethylene cap   | Extraction: continuous extraction must be started within 5 days of VTSR. Analysis: 40 days from start of extraction |

## Footnotes

- <sup>1</sup> Use only in the presence of residual chlorine.
- <sup>2</sup> Optionally, all samples may be tested with lead acetate paper before pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then the NaOH is added to pH 12.
- <sup>3</sup> Filter samples immediately on-site before adding preservatives for dissolved metals.
- <sup>4</sup> Applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
- <sup>5</sup> Sample receiving no pH adjustment shall be analyzed within seven days of sampling.
- <sup>6</sup> The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment shall be analyzed within three days of sampling.
- <sup>7</sup> When the extractable analytes of concern fall within a single chemical Category, the specified preservative and maximum holding times shall be observed for optimum safe guard of sample integrity. When the analyses of concern fall within two or more chemical categories, the sample may be preserved by cooling to four (4) degrees Celsius, reducing residual chlorine with 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$ , storing in the dark and, for pesticides only, adjusting the pH to 6-9. Samples preserved in this manner may be held for seven days before extraction and 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in reference 1 (regarding the requirement for thiosulfate reduction of residual chlorine), and references 8 and 9 (regarding the analysis of benzidine).
- <sup>8</sup> Extracts may be stored up to seven days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- <sup>9</sup> For the analysis of diphenylnitrosamine, add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$  and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- <sup>10</sup> The pH adjustment may be performed upon receipt at the environmental laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$ .
- <sup>11</sup> Method 1631 Revision B: Mercury in Water by Oxidation, Purge and Trap and Cold Vapor Atomic Fluorescence Spectrometry is required. Samples may be shipped to laboratory unpreserved if collected in fluoropolymer bottles, filled to top with no headspace, capped tightly, and maintained at 4°C from time of collection until preservation. The samples must be acid preserved within 48 hours after sampling.
- <sup>12</sup> First use of samples shall begin within 36 hours of collection. For storm water discharges, first use of the sample shall begin within 48 hours of collection.
- <sup>13</sup> Once collected if the assay can not begin within 8 hours then the sample must be frozen. Once defrosted, the sample can be held at 4°C until the assay begins. The assay must then be done the day that the sample is defrosted.
- <sup>14</sup> Elution, concentration and the application of the purified sample to the slide must be completed in one work day. The sample must be stained within 72 hours of the application of the purified sample to the slide. Up to 7 days are permitted between sample staining and examination.

**Table 2.8 Analysis of BIOLOGICAL Samples Using NJDEP Methodologies for Freshwater, Estuarine And Marine Samples**

| Parameter                             | Sample Container                | Container Volume | Preservation <sup>(1)</sup>   | Maximum Holding Time | Analytical Methodology                | Sample Container Cleaning |
|---------------------------------------|---------------------------------|------------------|---|----------------------|---------------------------------------|---------------------------|
| <b>PHYTOPLANKTON</b>                  |                                 |                  |   |                      |                                       |                           |
| <b>FRESHWATER</b>                     |                                 |                  |   |                      |                                       |                           |
| Species Composition<br>(live samples) | P,G                             | 250 ml           | Cool, 4° C  | 24 hours             | SM17:10200<br>EPA73:<br>Plankton 3,4  | (2)                       |
| (preserved)                           | P,G                             | 1000 ml          | 50 ml<br>neutralized<br>formalin<br>store/transport in<br>dark, cool container  | 1 month              | As Above                              | As Above                  |
| Chlorophyll a                         | P,G<br>amber or<br>foil-covered | 250 ml           | Cool, 4° C<br>store/transport<br>in dark  | 48 hours             | SM17:10200H<br>EPA73:<br>Plankton 5.2 | As Above                  |
| <b>MARINE AND ESTUARINE</b>           |                                 |                  |   |                      |                                       |                           |
| Species Composition<br>(live samples) | P,G                             | 250 ml           | Cool, 4° C  | 24 hours             | SM17:10200<br>EPA73:<br>Plankton 3,4  | (2)                       |
| (preserved)                           | P,G                             | 1000 ml          | 10 ml or more<br>Lugol’s solution<br>to maintain weak<br>tea color.<br>Store/transport<br>in dark, cool<br>container. | 48 hours             | As Above                              | As Above                  |
| <b>PHYTOPLANKTON</b>                  |                                 |                  |   |                      |                                       |                           |
| <b>MARINE AND ESTUARINE</b>           |                                 |                  |   |                      |                                       |                           |
| Chlorophyll a                         | P,G<br>amber or<br>foil-covered | 250 ml           | Cool, 4° C<br>store/transport<br>in dark  | 48 hours             | SM17:10200H<br>EPA73:<br>Plankton 5.2 | As Above                  |
| <b>ZOOPLANKTON</b>                    |                                 |                  |   |                      |                                       |                           |
| Freshwater                            | P,G                             | 6,000 ml         | 300 ml<br>neutralized<br>formalin.<br>Store in cool<br>container  | 1 month              | SM17: 10200<br>EPA73:<br>Plankton 3,4 | (2)                       |

Table 2.8 (continued) Analysis of BIOLOGICAL Samples Using NJDEP Methodologies for Freshwater, Estuarine And Marine Samples

| <b>Parameter</b>                            | <b>Sample Container</b> | <b>Container Volume</b> | <b>Preservation<sup>(1)</sup></b>   | <b>Maximum Holding Time</b> | <b>Analytical Methodology</b>          | <b>Sample Container Cleaning</b> |
|---|-------------------------|-------------------------|---|-----------------------------|--|----------------------------------|
| Marine & Estuary                            | P,G                     | As Above                | 5% formalin (5 ml neutralized formalin/100 ml tap water), store and transport in cool container                                   | As Above                    | As Above                               | As Above                         |
| <b>PERIPHYTON</b>                           |                         |                         |   |                             |  |                                  |
| <b>DIATOMETER SLIDES AND ROCK SCRAPINGS</b> |                         |                         |   |                             |  |                                  |
| 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                     | SM17: 10300 EPA99 Periphyton.6         | As Above                         |
| <b>PERIPHYTON</b>                           |                         |                         |   |                             |  |                                  |
| Chlorophyll a                               | P,G                     | 30 ml                   | 90% neutralized acetone, cool 0-4° C, store and transport in dark container   | 48 hours                    | SM17: 10300 EPA73: Periphyton 3.2      | (2)                              |
| Ash Free Weight                             | 120 ml jar polyseal cap | 30 ml                   | 90 % neutralized acetone, cool 0-4° C, store and transport in dark container  | N/A                         | SM17:10300 EPA73: Plankton 5.1         | As Above                         |
| <b>MACROINVERTEBRATES</b>                   |                         |                         |   |                             |  |                                  |
| Species composition                         | P,G                     | N/A                     | 5% neutralized formalin (5 ml neutralized, formalin/100 ml sample water [95% ethanol, isopropyl alcohol])                         | N/A                         | SM17:10500 EPA99: Macroinvertebrates 7 | As Above                         |

(1) Neutralized formalin = 100 % neutralized formalin with sodium tetraborate to pH 7.0 – 7.3

(2) Warm detergent solution wash, thorough rinse in tap and distilled water.