# DEPARTMENT OF ENVIRONMENTAL PROTECTION

**Laboratory Accreditation Program**

**Document Number**: #

**Title**: Sample Collection, Receipt, and Handling Guidance

**Effective Date**: Date

**Authority**: 27 Pa. C.S. §§ 4101 – 4113 (relating to environmental laboratory accreditation),

Environmental Laboratory Accreditation Regulations, 25 Pa. Code Chapter 252

**Policy**: It is the policy of the Department of Environmental Protection (DEP) to provide laboratory management personnel with the information necessary to either obtain or maintain accreditation to perform and report environmental analyses in Pennsylvania.

**Purpose**: This technical guidance will provide laboratory management with the tools to develop sample collection, receiving, handling, and reporting procedures that meet the requirements for laboratory accreditation.

**Applicability**: The guidance will apply to all laboratories desiring to obtain and/or maintain accreditation under the 25 Pa. Code Chapter 252 and/or the Pennsylvania NELAP program.

**Disclaimer:** The policies and procedures outlined in this guidance document are intended to supplement existing requirements. Nothing in the policies or procedures will affect regulatory requirements.

The policies and procedures herein are not an adjudication or a regulation. There is no intent on the part of the Department to give these rules that weight or deference. This document establishes the framework within which DEP will exercise its administrative discretion in the future. DEP reserves the discretion to deviate from this policy statement if circumstances warrant.

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# Sample Collection, Receipt, and Handling Guidance

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## 1.0 Introduction

In the Commonwealth of Pennsylvania, numerous decisions are made daily by the public, the regulated community and the government that are based upon data generated by environmental laboratories. Valid and accurate data is essential to determine compliance with State and Federal laws. The production of valid and accurate data begins the moment the sample is collected.

In order to ensure that laboratories are continuing to report the most accurate information, The Department of Environmental Protection (Department or DEP) has expanded upon the language previously published in 25 Pa. Code Chapter 252 to further clarify: 1) the procedures environmental laboratories must develop and maintain in order to ensure they are following applicable State and Federal sample collection, preservation, and transportation requirements, 2) the necessary documentation a laboratory must maintain in order to show the laboratory's sample collection personnel have the training for sample collection as well as training in ethical and legal responsibilities, 3) the specific circumstances under which pH and the residual chlorine content of a sample must be checked and documented, and 4) the records the laboratory must maintain in order to document that all sample collection, preservation, and transportation requirements have been met for a sample in order to report valid and accurate data.

## 2.0 Background

Various analytes that are important to ensuring the health of the public and environment are affected by conditions such as pH, residual chlorine, and temperature. State and Federal regulations have been established to specify how to collect and preserve samples to ensure the most valid and accurate data is produced. Environmental laboratories accredited under Chapter 252 and/or the TNI Standard must follow the regulations published by State and Federal organizations to provide the data necessary for the Department to make decisions related to keeping the public and environment safe.

Recently, Chapter 252 was revised to clarify the sample collection, preservation, and transportation training and documentation that laboratories must adhere to in order to maintain accreditation and provide valid and accurate data. The technical guidance included in this document further explains and clarifies the language in Chapter 252 and provides suggestions on how the laboratory can comply with the regulations.

## 3.0 Sample Collection, Preservation, and Transport Instructions: § 252.307

All accredited environmental laboratories are required to maintain instructions for sample collection and preservation that meet the requirements of § 252.307(f) and (g). Section 252.307(f) requires that "when an environmental laboratory collects a sample to be analyzed, the sample collection method required by applicable State and Federal laws, regulations or permit conditions shall be followed." Subsection (g) states that, "an environmental laboratory shall follow the sample container, preservation procedures, and holding times required by State and Federal regulations. If the sample container, preservation procedures, and holding times are not required by State or Federal regulations, an environmental laboratory shall follow the sample container, sample preservation procedures, and holding time established in the method." For the purposes of this document the term "sample collection and handling" includes but is not limited to any technique or requirement related to sample collection, preservation, transport, storage, holding time, etc.. For the purposes of this document the

instructions for sample collection, preservation, and transport will be referred to as the "Sample Collection Instructions." The laboratory may develop its own Sample Collection Instructions or it may use sample collection and preservation instructions developed by another entity or organization, provided that the instructions meet all of the requirements of 25 Pa. Code Chapter 252.

All accredited laboratories are subject to the requirements of § 252.307 and therefore must maintain one or more Sample Collection Instructions that clearly and accurately describe the sample collection, preservation, and transport requirements for the various types of samples and analytical testing for which any or all of the following apply:

- The laboratory provides bottles to customers,
- A laboratory employee collects the samples,
- A laboratory employee picks-up the samples and transports them to the laboratory,
- The laboratory receives the samples for analysis,
- The laboratory receives samples to be shipped to another laboratory for analysis.

## 3.1 Collection and Preservation Requirements

3.1.1 The laboratory must evaluate the purpose for which the provided bottles, collected samples, or received samples will be tested/analyzed. The sample collection and handling requirements vary for different types of samples and for different compliance purposes. The locations of the requirements for sample bottles, collection techniques, preservation, transport, and holding times vary depending the on the purpose of the testing. Section 252.307(j) states, "An environmental laboratory shall maintain instructions for sample collection and preservation that meet the requirements of subsections (f) and (g)." Subsections (f) and (g) specifically require that the sample collection methods required by applicable State and Federal regulations, permit conditions, or test methods be followed.

3.1.2 The laboratory should first determine if any Federal or State regulations exist that specify any sample collection and handling requirements. Where they exist, they must be followed. Where the Federal or State regulations do not list the specific sample collection and handling requirements, the laboratory must evaluate the specific methods for which the testing will occur to determine the collection and handling requirements for these contaminants.

3.1.3 Federal Requirements – Various Federal regulations exist that outline the sample collection and handling requirements. The specific purpose of the sampling will dictate which Federal regulation applies. Some examples of Federal regulations for sample collection, preservation, and holding time requirements are listed below.

3.1.3.1 40 CFR 136.3(e) Table II lists the container type, thermal and chemical preservation requirements, and holding times that a laboratory must follow to meet the Federal regulations for the analysis of pollutants under the Clean Water Act. Meanwhile, the table in 40 CFR 141.23(l)(2) lists the chemical preservatives, container type, and holding times that a laboratory must follow to meet the Federal regulations for some of the regulated drinking water contaminants under the Safe Drinking Water Act. However, not all of the regulated contaminants are listed in these tables. Where Federal requirements are not listed, the laboratory would refer to the permit or method specifications. Specifically, collection, preservation,

holding times, and transport requirements for most organic contaminants for SDWA compliance are not included in 40 CFR Part 141.

3.1.3.2 40 CFR 141.23(l)(2) outlines the specific collection, preservation, and holding time requirements for the SDWA inorganic non-metals contaminants.

3.1.3.2.1 The footnote to the table in this section of the CFR provides that samples collected for the analysis of Fluoride must be collected in plastic or glass, no chemical preservative is required, and the holding time is 28 days. The sample does not need to be shipped or stored with or under thermal preservation.

3.1.3.2.2 The footnote to the table also includes requirements for Cyanide. Cyanide must be collected in plastic or glass, preserved at the time of collection with sodium hydroxide to pH of 12, shipped and stored at a temperature at or below 4°C, and the holding time of 14 days.

3.1.3.3 40 CFR136.3(e), Table II, is a list of required containers, preservation techniques, and holding times for microbiology, organic, inorganic non-metals, trace metals, radiochemical, and protozoan contaminants regulated under the Clean Water Act.

3.1.3.3.1 Table II lists some general footnotes that apply to all analytes, such as Footnote 1 that explains the abbreviations for container types. Footnote 2 explains that grab samples must be preserved within 15 minutes of collection and also gives specific information regarding composite samples. Footnote 3 further explains some transport requirements and definitions of hazardous materials. Footnote 4 explains how to interpret holding times.

3.1.3.3.2 Table II also uses a combination of analyte/compound names and short-hand numbering to identify the contaminants listed in various tables within Part 136. Within each specific analyte/compound there may be additional analyte/compound-specific footnotes.

3.1.3.3.3 Specifically, Acrolein and Acrylonitrile are identified as compounds numbered 3 and 4 from Table 1C—Organic Tests. Table II indicates that Acrolein and Acrylonitrile may be collected in glass FP-lined septum containers, the samples must be cooled to ≤6°C, preserved with 0.008% sodium thiosulfate, and acid preserved with hydrochloric acid to pH between 4-5, and the holding time is 14 days. There are several footnotes that give additional specifics, such as Footnote 18 that requires that aqueous samples must be thermally preserved to ≤6°C, and should not be frozen. Footnote 18 also states that if samples are analyzed within 15 minutes of collection then thermal preservation is not required. Footnote 10 also explains that pH adjustment is not required if acrolein will not be measured and samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

3.1.4 Federal and Method Requirements – Some Federal or State regulations include references to additional sources for sample collection and handling requirements.

3.1.4.1 40 CFR 141.23 outlines the specific collection, preservation, and holding time requirements for the SDWA trace metals contaminants. This example pertains

specifically to Arsenic. The table requires that samples collected for analysis of Arsenic be collected in plastic or glass bottles, preserved with concentrated $HNO_3$ to a pH<2, and that this acidification is encouraged and allowed at the laboratory rather than at the time of sampling provided the shipping time and other instructions specified in Section 8.3 of EPA 200.7, 200.8, or 200.9 are followed.[1] EPA 200.7, Section 8.3 states that samples are NOT filtered, acidified with 1:1 nitric acid to pH<2. The methods also state that the preservation may occur at the time of collection or at the laboratory within two weeks of collection. EPA 200.7 also states that samples that are not acidified at the time of collection must be held for at least 16 hours after acidification, checked for proper preservation to pH<2, and then they may be analyzed.

3.1.4.2 40 CFR 136.3(e), Table II, includes requirements for Chromium VI, numbered 18 from Table IB—Metals. The Main identifier of "Table IB—Metals" includes Footnote 7, which includes specifications for filtration for dissolved metals from grab and composites from automated samplers. Specifically, Chromium VI samples must be collected in plastic, fluoropolymer, or glass; cooled, stored at ≤6°C, and preserved to a pH of 9.3-9.7; and the holding time is 28 days. The pH preservation range includes Footnote 20, which states that the laboratory must meet the ammonium sulfate buffer solution specifications in EPA Method 218.6 in order to achieve a 28-day holding time and if the requirements of EPA 218.6 are not met then the holding times listed in the testing methods must be followed. EPA 218.6, Section 8.2 states that dissolved Chromium VI must be filtered through a 0.45um filter and the pH adjusted to between 9-9.5 with buffer.

3.1.5 Method Requirements – Where Federal or State regulations do not include any specific sample collection and handling requirements, the laboratory must consult the requirements of the approved method for which testing will be conducted.

3.1.5.1 40 CFR Part 141 does not list the sample collection and handling requirements for regulated volatile organic compounds (VOCs). The laboratory must consult the promulgated method. For this example, the laboratory would consult the appropriate EPA-approved analysis method, either EPA 524.2, EPA 524.3, EPA 551 or EPA 502. EPA 524.2, Sections 8.1 and 8.2 include very detailed sampling instructions for collection, preservation, dechlorination, and storage. These instructions require a sample to be collected in duplicate, dechlorinated, then acid preserved to pH<2. The method also outlines the specific flow and pipe-flushing requirements to obtain a valid sample. Samples must be collected with zero head-space, shipped to the laboratory on ice, and stored at ≤ 4° C. Finally, Section 8.3 outlines the requirements for collection of duplicate field reagent blanks.

3.1.5.2 Some methods specifically require collection of field or trip blanks. These blanks must be prepared, stored, and handled according to method criteria. For example, VOCs by EPA 524.2 and low-level mercury by EPA 1631 and EPA 245.7 both include procedures for the use of field blanks.

[1] EPA followed by a number (e.g. EPA 218) refers to the EPA Method relating to that number. Thus EPA 218 means EPA Method 218. EPA is the United States Environmental Protection Agency

3.1.6 Program/Permit-Specific Requirements – Occasionally, program-specific requirements exist that are different from one program to another. In other instances, the specific sampling requirements of the permit or program will dictate sample collection and handling requirements.

3.1.6.1 The laboratory must ensure that it follows the Program-specific sample preservation requirements of the applicable Federal regulations. For example: Total Cyanide is listed under both 40 CFR Parts 136 and 141. However, Part 136 specifies that Total Cyanide samples must be preserved with sodium hydroxide to a pH>10, while Part 141 specifies that Total Cyanide samples must be preserved with sodium hydroxide to a pH>12.

3.1.6.2 The EPA and DEP have established specific requirements for the sampling and analysis of Lead and Copper for compliance with the Safe Drinking Water Act. 40 CFR Part 141, § 141.86(b)(2) and 25 Pa. Code Chapter 109 require that a public water system (PWS) must establish "targeted sampling sites" from which paired samples for both Lead and Copper are collected as 1-Liter "first-draw" samples. "First-draw" is defined as a sample that is taken after the line has stood motionless in the plumbing system for at least 6 hours. The sample is collected in a 1 Liter plastic or glass container. Samples must be acid preserved with nitric acid within 14 days of collection to pH<2. The "paired" sample requirement means that both the lead and copper result must be obtained from the same 1L sample container.

3.1.6.3 Permit-specific requirements include sampling location or the requirement to collect grab or composite samples or even a specific timeframe or window for when the samples must be collected. Examples include SDWA TTHM/HAA5 samples, where the sampling plan is established and the samples must be collected within a specific time period each quarter.

## 3.2 Sample Collection Instructions

The laboratory may use current laboratory instructions or standard operating procedures (SOPs) if the laboratory's instructions meet the requirements of the regulations, methods, and/or permits. The laboratory may choose to or be required to develop new instructions to meet the sample collection and handling requirements if the laboratory's instructions are insufficient or incorrect. Alternatively, the laboratory may choose to use the instructions developed by another entity. At a minimum, the laboratory should critically evaluate all instructions before use, control these documents in accordance with the document review, approval, and control requirements of the applicable accreditation standards (Chapter 252 and/or NELAP), and ensure that only those instructions that meet all applicable requirements are used by laboratory personnel and provided to customers or clients.

3.2.1 The Department recommends that the laboratory develop/maintain different documents for different uses or purposes. If the laboratory employs personnel to collect and transport samples for analysis, these instructions might require additional detail or information for the specific employee or procedure utilized by the laboratory. However, a laboratory that provides bottles to customers for their own collection might choose to have separate instructions, different than those utilized by the laboratory's trained employees.

3.2.2 The Department recommends that the laboratory's sample collection instructions include a table that shows the sample container type and number of required containers, thermal preservation requirements, necessary chemical preservatives for that analysis, and holding times. A table will improve readability, understanding, and compliance. The Department also recommends that instructions provided to clients or customers be simple and specific.

3.2.3 The instructions must meet any applicable Federal, State, method, or permit requirements. This means that the laboratory must be aware of and ensure that the sample collection instructions meet the specific sample analysis and compliance purposes for which they are to be collected. The laboratory might choose to have different sample collection instructions for SDWA compliance, CWA compliance, underground storage tanks, monitoring wells, etc.

3.2.4 The instructions must include the sample collection and handling requirements for all compliance testing that is listed on the laboratory's scope of accreditation AND any compliance samples that are received by the laboratory to be subcontracted to another laboratory for analysis. A laboratory that provides sample bottles to customers for their own collection, or sends staff out to collect the samples, must ensure that the sample collection instructions are applicable to the analysis types to be collected. The instructions should answer the following questions:

3.2.4.1 How many bottles are needed, what type of bottle is required, what size container is required, etc.? For example: Regulated VOCs by EPA 524.2 require that 40 mLs of sample be collected in duplicate in glass vials. The method also requires a field reagent blank. Therefore, the instructions provided to the sample collector for EPA 524.2 should state that at least three 40mL VOA vials should be returned to the laboratory; two collected with sample, and one is a field reagent blank.

3.2.4.2 What volume or how much sample is required to complete the analysis of the sample and any batch quality control specified in the regulations and/or method? For example: A sample collected for Total Phosphorous by SM 4500-P E. The method requires a laboratory fortified matrix (LFM) be performed per batch of 20 samples, and 25 Pa. Code Chapter 252 requires the analysis of one duplicate per batch. The laboratory uses the Persulfate Digestion Method, which requires at least 50 mL of sample for digestion. For the laboratory to meet the regulation and method requirements, the laboratory's instructions should inform the sample collector that a minimum of 150 mL of sample needs to be collected.

3.2.4.3 Are there any specific collection requirements, such as "zero headspace" or "first draw"? The laboratory should provide definitions for these terms and instructions that explain collection procedures to meet these terms. For example: A laboratory's definition of zero headspace could be defined as any sample with an air bubble that does not exceed 6 mm, as defined in SW-846 Chapter 4, Rev. 4, section 4.1.2. The USEPA has defined "first-draw" as it relates to collection of Lead and Copper samples for compliance with 40 CFR 141.86.

3.2.4.4 How much chemical preservative needs to be added to the sample in order meet the chemical preservation requirements for that sample size and matrix or specific

endpoint (such as a final pH)? Such as: 1 mL of 1:1 hydrochloric acid must be added to 50 mL of sample to reduce the pH to <2.

3.2.4.5 What type(s) of preservative must be added to the sample (i.e. sulfuric acid, sodium hydroxide, sodium thiosulfate, ammonium chloride, etc.)?

3.2.4.6 How are the preservatives added to the sample? Is there a specific order in which the preservatives must be added? Such as: Does the bottle already contain the preservative? Does the sample require dechlorination, to be shaken, and then acidified? Are there any specific instructions on what to do with the preservation, such as "do not rinse the bottle", etc.?

3.2.4.7 Are there thermal preservation requirements? Some examples of thermal preservation requirements include, but are not limited to: the temperature specific requirements (≤6°C, ≤10°C, frozen, none, etc.), received on ice, etc.

3.2.4.8 Are there any method specific packing requirements for the samples? Or does the laboratory have specific packing instructions to eliminate or reduce contamination? Some examples of method specific packing could include: microbiology sample bottles are bagged in zip-top bags before placed in the cooler with ice. How much ice? What type of ice (dry ice, lose packed ice, tight packed ice, etc.)? What type of cooler should be used to transport the samples?

3.2.4.9 Are there any alternative preservation routines for the sample? For example: EPA 1631 states trace level mercury samples can be collected without preservation but must be preserved within 48 hours of sample collection. The option exists to preserve the sample at the time of collection or preserve the sample upon receipt in the laboratory.

3.2.4.10 Are there any specific transport requirements? Such as how much time do you have to transport the sample to the laboratory to ensure holding times are met? Does the method require the collection and analysis of field reagent blanks or trip blanks?

3.2.5 Before the sample collection instructions can be used by laboratory employees or distributed to customers and clients, the laboratory should:

- Evaluate the purpose of the sampling.
- Answer the questions outlined in section 3.2.4 for all samples collected or received by the laboratory.
- Evaluate the current or to be developed sample collection instructions and ensure that they meet all of the regulatory, method, and permit requirements.
- Ensure that all documents are properly reviewed, approved, and controlled.

## 3.3 Distribution and Control of Sample Collection Instructions

3.3.1 Documentation Control – Laboratories are required to have a system for the maintenance and control of all documents. Section 252.401(c) states, "Environmental laboratories are required to have a document control system that provides procedures for control and maintenance of all documents. The document control system must

ensure that SOPs, methods, manuals, or documents clearly indicate the time period during which the procedure or document was in force." Sample collection instructions are included in this requirement.

3.3.1.1 The sample collection instructions are part of the laboratory's SOPs, manuals, or other documents that must include a date on which the document was effective. The laboratory should keep a log of all documents and their effective dates and dates of retire.

3.3.1.2 The laboratory must ensure that only the approved and effective versions of the sample collection instructions are available for use and distribution.

3.3.1.3 The sample collection instructions that are used by laboratory employees must be controlled in such a manner that they can be retrieved when they are to be retired, thus ensuring that they can no longer be used.

3.3.2 Availability of Documents to Non-Laboratory Personnel – § 252.307(j)(2) states, "The environmental laboratory shall make the sample collection instructions available to all laboratory sample collection personnel and to customers and clients that collect samples."

3.3.2.1 Sample collection instructions that are made available to customers, clients, or other non-laboratory employees are not required to be controlled in a manner that allows for their return upon retire. However, the laboratory must ensure that the sample collection instructions provided to customers or clients meet the requirements of 252.401(c).

3.3.2.2 The laboratory should provide the sample collection instructions to customers each time they receive bottles. The laboratory should provide instructions that are limited to those sample types to be collected, however, this is not a requirement.

3.3.2.3 The laboratory could choose to provide the instructions in hard-copy or electronically, such as available on their website.

3.3.2.4 The laboratory is not required to provide specific training to non-laboratory personnel related to sample collection and handling. However, the sample collection instructions should be detailed and provide enough information that a lay person can properly collect a compliance sample. The Department recommends that the laboratory provide assistance as needed or when requested.

## 4.0 Training and Documentation Requirements for Laboratory Employees that Collect Samples

The laboratory management must define the minimal level of qualification, experience, and skills necessary for all laboratory employees, including sample collection personnel. The laboratory management must ensure and document that each employee has read, understood, and is using the latest version of the standard operating procedures (SOP) and quality manual (QM) as it relates to the employee's job duties. For the purposes of this section "sample collection personnel" refer to employees of the laboratory, not customers or clients that collect their own samples.

4.1 The laboratory's sample collection instructions, or SOPs, to be utilized by laboratory personnel when collecting compliance samples are part of the documentation that must be read, understood, and used. The laboratory management must ensure that all sample collection personnel are appropriately trained and that they have demonstrated capability in their job duties. The laboratory management may choose to require a signature of the employee to document understanding or the laboratory management may choose to document compliance and understanding in other ways.

4.2 The laboratory management must ensure and document that the sample collection personnel have demonstrated capability and have received training on the proper sample collection and preservation requirements for the specific sampling undertaken. Some examples of this training and demonstration of capability may include:

4.2.1 The sample collector trainee goes out in the field with a trained sample collector to observe the sample collection, preservation, and transportation procedures. Then the trainee practices collection, preservation, and transportation techniques under the supervision or guidance of the trained sample collector.

4.2.2 A demonstration of capability can be performed by allowing the sample collector trainee to collect samples without interference but under the supervision/observation of a trained sample collector. The trained employee would observe the trainee to determine: Is the proper sample bottle used? Was the sample collected properly (i.e. first draw)? Were the proper preservatives added? Were the preservatives added in the correct order? Were the samples packed for transportation properly? Was the record of the sample collection recorded accurately and properly? If all of these activities are performed acceptably then the trainee would have demonstrated capability for sample collection for the particular samples taken.

4.3 Sample collection personnel must participate in ethics training and the potential liability for improper, unethical or illegal actions as it relates to the employee's job duties. They must also read, understand, and acknowledge their personal, ethical, and legal responsibilities, including any punishments and penalties. Training in ethical and legal responsibilities must occur within 2 months of employment and at least once every 14 months thereafter.

4.4 Laboratory management can choose to provide training that covers both sample collection and analytical duties to all personnel in the laboratory, or the laboratory management can choose to separate the training based on job duties. Regardless of how the laboratory decides to train its sample collection personnel and/or analysts, the laboratory must document the procedure for this training, the completion of the training, and that the demonstration of capability performed by the employee is acceptable.

4.5 Laboratory management must maintain records of all sample collection personnel, including dates of employment, signatures, initials, training, and demonstrations of capability.

## 5.0 Sample Receipt and Handling Procedures § 252.401(f) and (g)

The laboratory must have documented procedures that outline the laboratory's practices, procedures, and policies related to sample receiving and acceptance. The personnel

responsible for sample receiving (aka: sample receiving personnel/staff) must be appropriately trained and this training must be documented. DEP cautions all laboratories to establish documented procedures that can accommodate all sample types and analysis types, for example short holding times or specialized samples that require special or unique handling protocols. The laboratory cannot bypass its normal/documented sample acceptance and receiving protocols. If short holding time samples are handled differently, then those procedures and practices must be documented, validated, and ensure compliance with all applicable requirements.

§ 252.401(f) states, "An environmental laboratory shall establish procedures for handling environmental samples. (1) An environmental laboratory shall establish procedures for checking and verifying the condition of the sample. The results of these checks shall be recorded." The condition of the sample that must be checked and verified. This includes verifying and documenting the sample container, chemical preservation, and thermal preservation. The laboratory's procedures must establish when, where, and how these checks are performed and who is responsible for the check. The laboratory must establish procedures for documenting these checks. Some of these checks must occur upon receipt, or shortly thereafter, others may be performed after receipt and during regular sample processing/analytical testing in the laboratory, or others must occur after sample analysis to avoid compromising the integrity of the sample.

5.1 Visual Observations – The laboratory must check and document the physical condition of the sample upon receipt. The laboratory must also compare the received samples to those listed on the sample receiving documentation, such as a chain-of-custody ("COC").

5.1.1 The laboratory must check and document the physical condition of the sample upon receipt and specifically note any anomalies or questions. The laboratory's documentation should strive to answer the following question: "What does the sample receiving personnel/sample custodian see when he/she removes the sample from the transport container," or "what does the person receiving the sample observe when the customer drops off the samples at the laboratory?"

5.1.2 Some observations that the laboratory must make include: Does the sample container have legal seals? Are the containers leaking, cracked, or broken? Was the sample received on ice? What type and size of container was received?

5.1.3 The laboratory must evaluate the samples based on the observations made at receipt compared to the information provided on the COC. The laboratory is required to ensure that specific information is obtained regarding the samples. This information includes the requirements of § 252.401(f)(2): client/project name, date, time and location of sample collection, name of collector, and field identification code. The laboratory should ask questions such as: Are any samples missing that are included on the chain of custody? Are any of the required items from § 252.401(f)(2) missing from the COC? Was the sample received within hold time? Is this a SDWA compliance sample? What is the PWSID# and contact information?

5.1.4 The laboratory's sample receiving procedures must include instructions for what to do when the visual observations do not match those from the COC or other documentation provided with the sample. Questions that should be answered include: Should the sample receiving personnel contact a project manager or the collector directly? How are anomalies documented? How are corrections documented?

5.2 Temperature/Thermal Preservation – The laboratory must check and document the temperature of samples upon receipt. The temperature check must occur at sample receipt to ensure proper temperature measurements. Samples whose temperatures cannot be taken at receipt should remain in their transport containers. Sample temperatures should be taken when samples are removed from coolers so that they are not allowed to warm. The laboratory must establish procedures for how and when sample temperatures are taken. Laboratories must establish how to choose which sample bottle(s) will be checked.

5.2.1 The laboratory is required to take and document the temperature of all samples received at the laboratory. The laboratory must also document if the cooling process has begun by documenting if the samples were "received on ice" or "not received on ice."

5.2.2 The laboratory may choose to check and document the temperature of every container, or a subset of the containers based on relevant sample collection information. Some considerations for development of the laboratory's procedures for checking and documenting temperature could include the following:

5.2.2.1 What is the type of sample (aqueous, solid, oil, etc.)? Different sample types might yield different temperatures even if the samples were taken at the same location; therefore, the laboratory's procedures might include checking each sample type for each location.

5.2.2.2 What time were the samples collected? Were they collected at the same time or different times? Different sample sources taken from the same client or customer site within a short time period, with the same contact time with the ice, should have the same or similar sample temperature and might not require individual sample container temperatures. In this case, a temperature of one sample could be representative of all the samples received in that cooler. The Department recommends that samples within the same container, collected within the same hour could be considered representative and a single temperature could be taken. However, samples collected between 8am and 4pm from various sample sites are not representative, and a temperature of the container sampled at 8am would not be the same as the temperature of the sample collected at 4pm.

5.2.2.3 What date were the samples collected? Samples that are not received by the laboratory on the same day as collection but are all contained within the same cooler will probably have the same temperature, so a temperature of each sample location may not be necessary.

5.2.2.4 Is it acceptable to use a temperature blank to measure a representative temperature for all the samples in a cooler? Temperature blanks are not appropriate in most cases. Temperature blanks might be acceptable for samples that have been previously stored in a refrigerator, later removed from the refrigerator, packed at a laboratory, and shipped to another location under the exact same conditions.

- Temperature blanks may be acceptable when a laboratory subcontracts samples to another laboratory for analysis.

- Temperature blanks would not be appropriate for when multiple samples are collected from different sites and in contact with ice for a different amount of time. Temperature blanks are not representative of the conditions under which all the samples were held. For example, a collector visits multiple collection sites throughout the day and goes about collecting samples from 8AM until 3PM that day. At 8AM that day, the collector places a temperature blank in the cooler with ice. As samples are collected, they are placed in to the cooler with ice. The temperature blank is not representative of the temperature of all the samples in the cooler because the temperature blank has had more contact time with the ice than the samples collected later in the day.

5.2.2.5 The laboratory's sample receiving procedures must include instructions relating to what to do when the sample temperature does not match expectations. For example, the sample collection documentation states that the samples were collected from a stream in December at 8AM, where outside temperatures are below freezing. The samples are received at the laboratory at 9AM the same day and the sample temperature is measured to be 25°C. The temperature measurement is inconsistent with the time and date of sampling or the sample location or the sample type. The laboratory must have documented procedures to handle situations such as these.

5.2.3 The laboratory must ensure that the record appropriately documents compliance with the temperature preservation requirement. The actual observation must be recorded in a manner that allows the data user and laboratory to determine compliance, non-compliance, and usability of the data. The Department strongly recommends that the laboratory measure and document the actual temperature measurement of each sample container. It is not acceptable to record "All temps ok," as this does not include a record of the actual observation. The laboratory may choose to record "between freezing and 6°C" for samples that require thermal preservation above freezing and 6°C. If the temperature measurement demonstrates that the samples are not compliant, then the actual temperature must be documented for full and complete interpretation of the validity of the sample.

5.3 Chemical Preservation/pH Check – The laboratory must verify and document the chemical preservation of the sample. The laboratory may choose to perform this check at receipt, by sample receiving personnel, or in the laboratory, by analysts who are responsible for performing the particular test.

5.3.1 The laboratory must verify that the samples were properly chemically preserved based on the regulatory, method, and permit requirements for sample collection. For those samples that require chemical preservation, the laboratory must verify that the sample was appropriately preserved. Chemical preservation could include acid or alkali preservation to a specific pH, dechlorination with a particular chemical (NOTE: dechlorination is more fully explained in Section 5.4 of this document), addition of a buffer, etc.

5.3.2 Laboratories that perform analysis of Safe Drinking Water Act ("SDWA") compliance samples are required by § 252.401(f)(1)(ii) to check and document the pH of every sample container collected for chemistry analyses. The pH check is not required for unpreserved samples if the sample was collected by trained/qualified sample collection

personnel employed by the laboratory that performs the sample analysis. The following are examples of a laboratory's compliance requirements with this subsection:

5.3.2.1 A laboratory employee trained in sample collection collects two samples for SDWA compliance. One sample is for Cyanide analysis, and the second sample is for Alkalinity. The employee returns the two containers to the laboratory for receipt and log-in. The laboratory must check and document the pH of the Cyanide sample, to ensure that it is pH>12 because Cyanide has a pH preservation requirement and the laboratory must check and document the preservation of all chemically preserved samples. The laboratory is not required to check and document the pH of the sample for Alkalinity because there is not a pH preservation requirement for Alkalinity and because trained/qualified laboratory staff of the testing laboratory collected the sample.

5.3.2.2 A sample is received by the laboratory for Nitrite analysis for compliance with the SDWA. The sample was not collected by the laboratory's sample collection personnel. The sample was collected by the Public Water Supplier. Even though the requested analysis does not include a pH preservation requirement, the laboratory must check and document the pH of the sample to ensure it was not improperly preserved.

5.3.2.3 A sample was subcontracted by Lab ABC to Lab XYZ for analysis of Anions by Ion Chromatography ("IC") for compliance with SDWA. The originating laboratory (Lab ABC) collected the sample using trained laboratory employees (sample collection personnel). Because the testing laboratory, Lab XYZ, did not perform the sample collection, Lab XYZ must check and verify the pH of the samples. Also note that "sister" or "satellite" laboratories are separate laboratory locations in accordance with § 252.201(c).

5.3.3 The laboratory's procedures must specify where, when, and who performs and documents the chemical preservation and pH checks. Such as: Is the pH checked at sample receipt or in the laboratory? Who is responsible for checking the pH? When is the pH of the sample checked? Is the pH checked after analysis (i.e. VOCs)?, etc. Laboratories can establish procedures within their own laboratory to meet this requirement. Some samples require specific and specialized pH verification procedures that the laboratory might not want to occur in sample receiving. For example:

5.3.3.1 Oil and Grease samples to be analyzed by EPA 1664A require that the sample pH be checked by dipping a glass stir-rod into the sample, touched to the pH paper, and then rinsed back into the sample container with n-Hexane. The laboratory management might determine that this type of pH check is to be performed by the laboratory-trained analyst for Oil and Grease analysis but all other chemical preservations are checked in sample receiving.

5.3.3.2 VOCs analysis for water samples requires that the sample remain sealed until analysis. Verification of the pH preservation of these samples could occur after sample analysis to avoid compromising the samples.

5.3.4 The procedure must also specify how the pH is checked, to what tolerance, and what documentation/record is required.

5.3.4.1 The laboratory must ensure that the pH is checked to the tolerance of the method. If the method specifies that the sample pH must be between 12.5 and 13, then the laboratory must employ an appropriate procedure to check pH within this range. The laboratory cannot use wide-range pH paper. However, narrow-range paper might be acceptable depending on the range of the particular pH paper. The laboratory may choose to or need to use a pH meter that is appropriately calibrated.

5.3.4.2 The laboratory must ensure that the record appropriately documents compliance with the chemical preservation check requirement. The actual observation must be recorded. It is not acceptable to record "All pH ok," as this does not include a record of the actual observation. The laboratory may choose to record "pH<2" for trace metals samples that require preservation to pH <2. The laboratory may choose to record "pH between 6.5 and 7.5" if the laboratory is using pH paper that measures pH between 6.5 and 7.5.

5.3.5 The Department's regulations do not establish a specific pH range for samples that do not require acid or alkali preservation. The laboratory must determine the procedures to be used to evaluate the acceptance and rejection of samples based on pH measurements. The laboratory must document the procedure to be used to handle samples when the sample does not fall within the laboratory identified or expected pH range.

5.4 Dechlorination/Presence of Residual Chlorine – The laboratory must verify and document the absence or appropriate removal of residual chlorine from the sample when the sample collection requirements specify its removal or when its presence would compromise the validity of the test. The laboratory may choose to perform this check at sample receipt or in the laboratory.

5.4.1 The laboratory must verify that residual chlorine was properly removed when the regulatory, method, and permit requirements specify its removal at the time of sample collection. The laboratory must also ensure that the correct dechlorinating agent is used. Section 252.401(f)(1)(iii) also requires that samples must be checked for the presence of residual chlorine when its presence will compromise the validity of the test.

5.4.1.1 Microbiology samples are required to be dechlorinated upon collection. Microbiology samples can easily be contaminated when handled inappropriately or carelessly. The laboratory might choose to have only trained microbiology analysts perform the check of residual chlorine for microbiology samples.

5.4.1.2 Many organic contaminants require dechlorination with ascorbic acid or sodium thiosulfate at the time of collection. The laboratory may choose to have all chemistry samples that require dechlorination to be checked in sample receiving or by the analyst that prepares and/or analyzes the sample.

5.4.2 Laboratories must establish a procedure for determining if the presence of residual chlorine will compromise or interfere the validity of the test. The laboratory must

establish which tests and samples require a residual chlorine check. Some considerations would be:

- Does the method require a check for the presence of residual chlorine?
- Does the method require chemical treatment of samples for residual chlorine?
- Do the sample preservation requirements indicate that residual chlorine changes the validity of the test or affect the measurement of the analyte of interest?

5.4.2.1 40 CFR 141.23(2) includes the collection, preservation, container, and maximum holding times for Nitrate and Nitrite for SDWA compliance. Nitrate and Nitrite for SDWA compliance include very specific chemical preservation requirements that will determine what analytical result will be obtained, Nitrate, Nitrite, or Total Nitrate+Nitrite.

5.4.2.2 Analysis of Nitrate and Nitrite individually must occur within 48 hours on a sample that is not chemically preserved. The 48-hour analysis time for Nitrate includes a Footnote 5 which states, "If the sample is chlorinated, the holding time for an unacidified sample kept at 4°C is extended to 14 days." A chemical reaction occurs when a sample containing Nitrite is chlorinated; the Nitrite is destroyed and only Nitrate remains. A chlorinated sample collected for Nitrate is stable for up to 14 days instead of 48 hours. Thus, the presence of chlorine will directly impact the validity of the test for Nitrite, Nitrate, and Total Nitrate+Nitrite.

5.4.2.3 A sample that is taken for Nitrite compliance that tests positive for the presence of chlorine does not necessarily invalidate the sample. For example, a sample that is to be tested for Nitrite from a PWS that uses chlorine as an approved treatment technique should contain residual chlorine, and its presence is expected and acceptable. However, a PWS that does not use chlorine as a treatment technique or where the concentration of residual chlorine is above the acceptable treatment level will indicate an improper sample and the laboratory must note this on the sample receiving records, such as the COC, and handle the sample appropriately.

5.4.3 The laboratory must establish procedures for how, when, and who is responsible for the residual chlorine check and what happens if residual chlorine is present in samples where it is required to be removed (either at the time of collection or at another point in the sample preparation or analysis) or when the presence of residual chlorine will jeopardize the validity of the test. The laboratory's sample acceptance/rejection policy must address these situations.

5.5 Documentation Requirements – Section 252.401(f)(2) provides that the laboratory must establish procedures for a recordkeeping system to document the receipt of all sample containers. The procedures should explain what to do if any of the required information is not provided or available. The laboratory may establish procedures for obtaining this information after receipt.

5.5.1 At a minimum, the system must include procedures for recording and maintaining the client/project name; the date, time, location of sample collection; the name of sample collector (the Department recommends that the affiliation of the sample collector also be noted, but this is not a requirement); the field ID code; the date and time of laboratory receipt, and the identification of the individual receiving the sample at the laboratory.

5.5.1.1 The laboratory may allow the use of initials, collector ID, or some other form of identification to identify the individuals responsible for making the records or collecting the samples. The laboratory must maintain documentation that links the individual's name to their initials, collector ID, employee ID, or other form of identification.

5.5.1.2 The documentation must include enough information to accurately identify who is making every entry in the laboratory's records for sample drop-off and receipt. Other information that the laboratory should maintain for non-laboratory employee entries would include: What is the affiliation of that person to the sample? How would the laboratory contact this person if additional information is necessary?

5.5.1.3 One noted exception for non-SDWA subcontracted samples: If the testing laboratory will not be reporting the results directly to the customer, then the testing laboratory is not required to obtain the following information itemized in § 252.401(f)(2) provided that the originating laboratory obtains this information and includes the required information on the final test report along with any data qualifiers or other pertinent information required for the interpretation of the final test results.

- Client/Project Name
- Location of Sample Collection
- Name of Sample Collector

5.5.2 The procedures should include ways to identify how the sample was transported and received. Such as: Was the sample shipped via courier or directly received from the customer?

5.5.3 The laboratory may choose to use specific Chain of Custody forms or may document the information in various log books, LIMS system, or other recordkeeping procedures provided they meet all Chapter 252 requirements for recordkeeping and record retention. All entries must be traceable to the laboratory's sample identification number.

5.5.4 When a laboratory receives subcontracted samples, the laboratory must treat those samples as if they were collected by untrained personnel, even if the samples were collected by a satellite or sister laboratory. The receiving/testing laboratory is required to check and document the condition of the sample, including all chemical and thermal preservation checks of the samples.

## 6.0 Sample Acceptance/Rejection Policy § 252.401(g)

Laboratories are required to have a sample acceptance policy that clearly outlines the circumstances under which the samples will be accepted or rejected. The laboratory's sample acceptance policy must include the items listed in § 252.401(g). The sample acceptance and rejection policy must explain the laboratory's policies related to what is acceptable and what is cause for rejection. The policy must provide specific instructions for handling samples that do not meet the sample acceptance policy.

### 6.1 Acceptance/Rejection Decisions

6.1.1 The sample acceptance policy must directly relate to the sample receipt and handling procedures developed by the laboratory in accordance with § 252.401(f). The laboratory must compare the documented observations regarding the condition of the sample to the requirements for sample collection and handling for the particular test. Any anomalies or deviations from the requirements observed during sample receipt, analysis and reporting would result in a sample that might need to be rejected, qualified, or additional information gathered before a final decision can be made.

6.1.2 The sample acceptance policy must include the review and evaluation of the sample collection and handling information recorded during sample receipt to determine if the sample was collected in compliance with any specific sample collection, handling, preservation, and documentation requirements.

6.1.3 The laboratory's procedures for accepting and rejecting a sample for analysis can include instructions to reject any sample that does not meet the specific acceptance criteria for sample analysis, or the procedure can be to instruct the laboratory staff to seek further guidance from a project manager or supervisor.

6.1.4 All decisions for sample acceptance and rejection must be documented.

6.2 Temperature/Thermal Preservation Checks—The sample acceptance policy must specify how to handle samples that are checked for temperature/thermal preservation, as required by § 252.401(f)(1) but do not meet the expectations or are not received at the required storage temperature.

6.2.1 Samples that are received by a laboratory on the same day as collection might not have been exposed to ice long enough to be cooled to the established storage temperature. A temperature that is not within the thermal preservation requirements specified in the regulations or method for sample collected on the day of receipt would not require rejection or data qualification on the final test report, provided that other anomalies are not noted or observed.

6.2.2 The laboratory should evaluate all circumstances available to evaluate whether the temperature of a sample received the same day it was collected is acceptable. For example: If a sample is collected from a stream in winter, and the laboratory receives the sample later in the day at a temperature of 25°C, then the sample was probably not handled properly and further investigation or action might be appropriate.

6.3 pH Checks—The sample acceptance policy must specify how to handle samples that are checked for pH, as required by § 252.401(f)(1)(i) and (ii) but do not meet expectations.

6.3.1 The laboratory is responsible for defining what the expectations for sample acceptance would be based on sample type, client history, or other information specifically related to the sample.

6.3.2 For example, SDWA samples collected for nitrite analysis require an unpreserved bottle in order to obtain a valid result. If the laboratory checks the sample bottle and the pH is <2, then the sample is invalid for compliance and the sample acceptance policy must explain how this situation is handled.

6.3.3 Another example would be SDWA cyanide samples received at the laboratory that are not pH >12. The samples must be preserved with sodium hydroxide at the time of collection to a pH >12. The sample is invalid for compliance and the sample acceptance policy must explain how this situation is handled.

6.4 Residual Chlorine Checks—The sample acceptance policy must specify how to handle samples that are checked for residual chlorine.

6.4.1 What is the laboratory's policy if a sample is supposed to be dechlorinated, and laboratory staff checks the sample for residual chlorine, as required by 252.401(f)(iii), and determines residual chlorine is present?

6.4.2 Some SDWA samples are obtained from chlorinated sources and the presence of chlorine in these samples, when the sample collection requirements do not specify dechlorination, might not invalidate the sample. The laboratory's sample acceptance policy would need to address these types of samples too.

6.5 SDWA Compliance Samples—For most chemistry and radiochemistry SDWA compliance testing, the testing laboratory has the authority and responsibility to evaluate the validity of the final test results and determine if the results meet all method, program, and regulatory requirements before reporting the result to DWELR. So, for these samples, analysis of an improperly collected, preserved, or stored sample will only cost the laboratory the expense of testing a sample that cannot be used for compliance purposes. **NOTE**: The laboratory must have a technically justified reason for invalidation of sample results after analysis. However, 25 Pa. Code Chapter 109 establishes the Department's authority and responsibility for invalidation of microbiology and lead and copper samples. Analysis of improperly collected, preserved, or stored microbiology or lead and copper samples will require Department approval before the samples may be invalidated. **The Department strongly recommends that any SDWA compliance sample should be automatically rejected if ALL sample acceptance criteria are not met.**

6.5.1 *SDWA Microbiology Samples*—§ 109.304(3)(iii) establishes that only the Department may invalidate total coliform samples. Laboratories should NOT begin sample analysis of microbiology samples that do not meet all collection, preservation, and storage requirements.

6.5.1.1 The analysis of non-compliant microbiology samples that yield a positive result still requires check samples, and the laboratory is still required to perform the 1-hr notification to the PWS and 24-hr written notification to the Department regardless of the condition of the sample.

6.5.1.2 The analysis of non-compliant microbiology samples that yield a negative result would require specific Departmental approval to be reported, and those samples will often be rejected by the DEP for compliance purposes. A replacement sample would likely be required.

6.5.2 *SDWA Chemistry Samples*—As stated above, most chemistry samples can be validated/invalidated by the accredited laboratory performing the testing. However, §

109.1105(j)(1) establishes the Department's authority and responsibility to invalidate lead or copper tap water samples.

6.5.2.1 The laboratory should NOT begin analysis of any improperly collected, preserved, or stored chemistry samples, but if the laboratory does begin analysis of non-compliant lead and copper samples, the laboratory must seek approval from the Department to invalidate those results.

6.5.2.2 The analysis of non-compliant chemistry samples would require specific approval to be reported, and the sample will often be rejected for compliance purposes. A replacement sample would likely be required.

6.5.3 *Request to Report Qualified DW Results for Chemistry*—The Department has established that some SDWA compliance results that do not meet all method, program or regulatory requirements might, in some specific cases, still be valid for compliance purposes. In all cases where the analytical test results are associated with any non-compliance with the program, method, or regulatory requirements the laboratory cannot report the result without specific written approval by the Department. In almost all cases of improperly collected, preserved, stored, or transported chemistry samples, the Department rejects the request to report the qualified DW result to DWELR.

6.5.4 Analysis of non-compliant SDWA samples will unnecessarily cost time and money, for the laboratory, the PWS, and the DEP.