

QAM-Q-111

## Aliquot Preparation and Sample Preservation

Revision 15

Approval:

  
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Laboratory Manager

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Texas Institute for Applied Environmental Research

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**1. Applicability and Purpose**

This procedure applies to all samples received in the laboratory at the Texas Institute for Applied Environmental Research (TIAER), Tarleton State University, Stephenville, Texas. This procedure is derived from several procedures in Standard Methods and EPA manuals, and it is part of the TIAER Quality Assurance Manual. The purpose of this procedure is to provide a method for splitting samples into aliquots for various analytical procedures, and to delineate the procedures for sample preservation in the laboratory.

**2. Definitions**

- 2.1. Test group code- a code assigned to a sample that designates a group of analyses through which the sample is to be processed
- 2.2. Aliquot- a portion of the sample taken from the original container to be used for specific analyses
- 2.3. Preservative/container code- a letter designation indicating the bottle type and preservation used in the bottle

**3. Equipment, Reagents and Standards**

- 3.1. Various sizes and types of laboratory containers and lids cleaned in accordance with QAM-I-116, "Preparation of Labware"
- 3.2. Sulfuric acid ( $H_2SO_4$ ), concentrated ACS grade
- 3.3. Nitric acid ( $HNO_3$ ), concentrated ACS grade
- 3.4. Sodium thiosulfate ( $Na_2S_2O_3$ ), ACS grade
- 3.5. Other types of method and program specific preservatives or filters may be required (see QAM-Q-101, "Laboratory Quality Control")
- 3.6. Eyedroppers, standard laboratory size
- 3.7. Refrigerated laboratory cooler ( $>0-≤6^{\circ}C$ ) or freezer ( $<0^{\circ}C$ ). Radioactive samples are stored separately.
- 3.8. Vacuum pump with side-arm flask and filter apparatus
- 3.9. 0.45  $\mu m$  nitrocellulose membrane filters (47mm diameter, phosphorus & contaminant-free; Millipore®, Whatman® or equivalent)
  - 3.9.1. Cover 50 filters with 2 L of DI water and soak for 1 hour.
  - 3.9.2. Pour off the water and soak for an additional 3 hours in fresh DI water.

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- 3.9.3. Pour off the water and store in DI water in a small dish covered with Parafilm™ or other clean container with lid. Label the container with the ID number, initials and date.
- 3.9.4. Shake excess water off each filter before use.
- 3.9.5. Record beginning and ending date and time of filter soaking and the number of filters prepared in the Equipment Prep Log Q-103-4, found in the E-logs.
- 3.10. pH paper, wide range (pHydrion™ or equivalent)
- 3.11. Graduated cylinders (“A”) and other miscellaneous labware
- 3.12. Thermometers, normally infrared digital, calibrated
- 3.13. Deionized water (DI)
- 3.14. Survey meter with appropriate detector (QAM-Q-101, “Laboratory Quality Control”)
- 3.15. Filters for swipe testing (SOP-RC-111, “Swipe Testing for Surface Contamination”)

**4. Procedure**

- 4.1. Refer to Attachment 1 of QAM-Q-101, “Laboratory Quality Control” for specific container type, preservative requirements, sample amount and holding time for each specific analysis.
- 4.2. Receive samples in accordance with QAM-Q-110, “Sample Receipt and Login”.
- 4.3. Segregate samples according to analysis type or test group code, which should be on the sample bottle, COC or obtained from the Laboratory Manager or designee. Also segregate samples that are radioactive by survey or swipe testing performed during Sample Receipt and Login (QAM-Q-110, “Sample Receipt and Login”). Radioactive samples are prepared or preserved in a separate location from stable chemistry samples.
- 4.4. Bacteriological samples receive priority for time and should be analyzed upon receipt. After login and labeling, bottles for these analyses are taken immediately to the analytical area. Ensure they remain on ice until analysis begins.
- 4.5. If needed, check the pH of acid or base preserved samples with pH paper by touching the paper to a drop of sample removed from the container. Never put the pH paper into the sample itself. Other preservations may need to be checked

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where appropriate. Refer to QAM-Q-101, "Laboratory Quality Control" for acceptable preservation requirements. Initiate corrective action if preservations are not proper (see QAM-Q-110, "Sample Receipt and Login"). Document field preservation and filtering for each bottle in the Sample Preservation Logbook, Attachment 1, which may be an Elog. Record the inventory control number or identification of the pH paper on all documentation where used.

4.6. Aliquot division

4.6.1. Label all aliquots with sample number, and any analytical codes, preservative/container codes or abbreviations, and other appropriate information required to perform analysis. Preservative/container codes are described in QAM-Q-110, "Sample Receipt and Login" and on the TIAER COC. All aliquots from the same sample have the same sample number, but also have different preservative/container codes for further designation. If more than one of the same sample has containers with the same code, follow the code designation with a "-1", "-2", etc.

4.6.2. Preserve appropriate amounts of sample for total analyte measurement in accordance with QAM-Q-101, "Laboratory Quality Control" by adding acids or other preservatives to the aliquot container. If preservatives are added to containers that will not be used right away, the containers should be tightly closed. Ensure any off-gassing is complete prior to closing the container. Check to ensure the aliquot is preserved correctly according to the analytical SOP. Record Inventory Control Number of preservatives also.

4.6.3. Liquid samples may be shaken, stirred, blended or otherwise mixed to obtain a representative composite of the sample submitted to the laboratory. Sticks, leaves, organisms or other materials may be physically removed from the sample if the client does not wish them to be considered part of the sample. Once mixed, quickly pour, pipette or otherwise transfer part of the liquid sample to the correctly labeled aliquot container, filter flask or other transfer container.

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- 4.6.4. Solid and semi-solid samples, dried or wet, may be stirred, shaken, milled, ground or otherwise mixed. Spatulas, spoons, and other utensils should be used to obtain representative portions from different sections of the original container prior to transfer.
- 4.6.5. Filter aliquots for the dissolved analyte measurements in liquid through 0.45  $\mu\text{m}$  membrane filters by vacuum filtration prior to acidification, if required. Refer to QAM-Q-101, "Laboratory Quality Control" for proper preservations. Examples are ammonia nitrogen, nitrate-nitrite nitrogen, orthophosphate phosphorus for laboratory filtration, chloride, sulfate, dissolved metals and other ions or species in a dissolved state or otherwise considered soluble. Replace filters as necessary to obtain sufficient test volumes. Other pore size filters may be used to clean up the sample, provided that the 0.45  $\mu\text{m}$  membrane filter is the final.
- 4.6.6. Add acid or other preservatives to various dissolved aliquots as required after filtration for dissolved constituents.
- 4.6.7. The filter apparatus is rinsed at least three times with DI  $\text{H}_2\text{O}$  between each sample; at least three seconds for flow-through parts. Further rinsing may be required if contamination problems are discovered. The apparatus should be removed from station and replaced with a clean apparatus at the end of each day.
- 4.6.8. Do not filter aliquots for the following analyses: total Kjeldahl nitrogen, chemical and biochemical oxygen demands, total phosphorus, solids (residue) determination, total recoverable metals or other analytes that require analysis in a total state. Preserve total aliquots as required.
- 4.6.9. Record all filtering and preservation in the Sample Preservation Logbook, Attachment 1.
- 4.6.10. Deionized water method blanks are filtered, preserved and analyzed at the station where samples are analyzed, where appropriate. They are not necessarily filtered or preserved at the sample preparation area, which is an extension of field activity.

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- 4.6.11. Laboratory Control Samples (LCS) and duplicates (LCSD) may be filtered, treated and preserved in the same manner as samples, if required by the method, at the station where analysis occurs.
- 4.6.12. Aliquots that require the same container type and preservative may be used for more than one analysis (ex. ammonia and nitrate-nitrite may be determined from the same preserved aliquot "E").
- 4.6.13. Chlorophyll-a and Pheophytin-a samples are filtered and preserved frozen in accordance with SOP-C-112, "Determination of Chlorophyll-a and Pheophytin-a". Other analytes may require special handling specific to the individual method.
- 4.6.14. Some sample portions may need to be separated into aliquots for specialized organic or metals extraction. Refer to the appropriate "C" series SOP for further details.
- 4.7. Place aliquots in refrigerated storage ( $>0\text{-}\leq 6^{\circ}\text{C}$ ), as required, in numerical order by sample number. Rotate samples through storage by age, normally corresponding to sample number. Refer to ESDMS for holding times remaining on various aliquots and the backlog of pending analyses to be run. Store radioactive samples according to analytes to be tested, separate from stable chemistry samples.
- 4.8. The Laboratory Manager directs removal and disposal of sample aliquots when holding time is past, or work is complete and acceptable, and denotes disposal on the COC.

**5. Quality Control and Safety Aspects**

- 5.1. All aspects of this procedure comply with QAM-Q-101, "Laboratory Quality Control" and QAM-S-101, "Laboratory Safety".
- 5.2. Dispose of unused aliquots and portions in accordance with QAM-W-101, "Disposal of Laboratory Waste" and QAM-Q-110, "Sample Receipt and Login" custody requirements. Refer to QAM-W-101, "Disposal of Laboratory Waste" for details on further pollution prevention measures.
- 5.3. Record all filtering and preservation in the Sample Preservation Logbook, Attachment 1.

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**6. References**

- 6.1. Standard Methods for the Examination of Water and Wastewater, latest online edition (EPA approved), edited by A. E. Greenberg, et al., APHA, AWWA, Washington, D.C.
- 6.2. Method of Chemical Analysis of Water and Wastes, John R. Kopp, et al., Environmental Monitoring and Support Lab, Cincinnati, OH, March 1983.
- 6.3. TNI standard, 2016, The NELAC Institute.

**7. Attachments**

- 7.1. Sample Preservation Logbook (Elog; may be part of Sample Logbook)

Working Copy

