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Standard Guide for Preparation of Materials Used for the Collection and Preservation of Atmospheric Wet Deposition¹

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~~ε¹ Note—Section 9 was editorially added in April 1994.~~

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1. Scope

1.1 This guide presents recommendations for the cleaning of plastic or glass materials used for collection of atmospheric wet deposition (AWD). This guide also presents recommendations for the preservation of samples collected for chemical analysis.

1.2 The materials used to collect AWD for the analysis of its inorganic constituents and trace elements should be plastic. High density polyethylene (HDPE) is most widely used and is acceptable for ~~all~~ most samples including samples for the determination of the anions of acetic, citric, and formic acids. Borosilicate glass is a collection alternative for the determination of the anions from acetic, citric, and formic acid; it is recommended for samples for the determination of other organic compounds.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water³

D 1129 Terminology Relating to Water³

D 1193 Specification for Reagent Water³

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres⁴

D 1695 Terminology of Cellulose and Cellulose Derivatives⁵

D 2914 Test Methods for Sulfur Dioxide Content of the Atmosphere (West-Gaeke Method)⁴

D 4453 Practice for Handling of Ultra-Pure Water Samples³

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this guide, refer to Terminology D 1129.

3.1.2 For definition of **plastic** refer to Terminology D 1695 and Terminology D 883.

3.1.3 For definition of AWD (**precipitation, meteorological**) refer to Terminology D 1356.

4. Significance and Use

4.1 Some chemical constituents of AWD are not stable and must be preserved before chemical analysis. Without sample preservation, it is possible that analytes can be lost through decomposition or sorption to the storage bottles.

4.2 Contamination of AWD samples can occur during both sample preservation and sample storage. Proper selection and cleaning of sampling containers are required to reduce the possibility of contamination of AWD samples.

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 11.03.

⁵ Annual Book of ASTM Standards, Vol 06.03.

4.3 The natural sponge and talc-free plastic gloves used in the following procedures should be recognized as potential sources of contamination. Individual experience should be used to select products that minimize contamination.

5. Apparatus

5.1 Instruments shall be selected in accordance with an applicable test method given in Test Methods D 1125.

5.2 The conductivity cell shall be pipet or dip type with a cell constant (K) of 0.1 .

TABLE 1 Preservation of AWD Samples Collected for Inorganic Cation and Anion Determinations

Preservation Technique	Species Determined	Remarks	Reference
No preservation	All inorganic cations and anions	Rapid analysis is required after collection because ion concentrations may change in samples. Ammonium, nitrate, and ortho-phosphate concentrations may be reduced in samples that are biologically active. Cation and trace metal concentrations may be reduced by sorption onto container surfaces.	(10-14)
Refrigerate 4°C	All inorganic cations and anions	Chilling may reduce the loss of ammonium, nitrate, and ortho-phosphate in samples that are biologically active. Samples must be allowed to come to ambient temperature (23–27°C) before performing pH and specific conductance determinations. Specific conductance and pH determinations should be performed on-site as soon as possible after sample collection.	(15-17)
HNO ₃ pH ≤ 2	Ca ²⁺ , Mg ²⁺ , Na ⁺ , K ⁺	Samples must first be filtered, or acid addition may dissolve particles in the AWD samples (see 7.4.1). Acid addition will interfere with anion determination, so a separate aliquot will be needed for other ion determinations.	(11,12)
HgCl ₂ or tetrachloro mercurate (Na ₂ HgCl ₄) ^A	Ammonium, nitrate, nitrite, phosphate	Mercury (II) is a known biocide and will preserve samples for 9 to 16 days and possibly longer periods of time. Dissolved mercury may interfere with other ion determinations, and this will require an additional aliquot. Final dissolved mercury (II) concentration in the AWD sample must be at least 30 mg/L.	(1,17)
HCl, 5 mL/L	All dissolved Hg and total Hg	Samples for dissolved Hg are filtered through 0.45 μm capsule filter. Samples collected directly into specially cleaned, pretested, fluoropolymer bottle.	(19,20)
Filtration	All inorganic cations and anions	pH and specific conductance determinations may be affected by filtration. Care must be taken to minimize the possibility of sample contamination during filtration.	(18)

^A Several of the suggested preservatives, such as tetrachloromercurate and mercuric chloride, are environmental and human health hazards and the use and disposal of these preservatives should be carefully controlled (Test Method D 2914, Section A4).

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade acids and other chemicals shall be used to reduce the risk of contaminating the AWD samples. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁶

6.2 Nitric acid, ultra pure.

6.3 Nitric acid (1+9)—Dilute 1 volume concentrated nitric acid with 9 volumes of water.

6.4 Chloroform, high performance liquid chromatography reagent.

~~6.5 Sodium tetrachloromercurate (II), 40 g/L Hg—Dissolve 54.4 g of mercuric chloride (HgCl₂)~~

~~6.5 Hydrochloric acid, pretested for total and 23.4 g sodium chloride (NaCl) in distilled water and adjust volume to 1000 mL.~~

~~6.6 Mercuric chloride.~~

~~6.7 Sodium chloride.~~

~~6.8 methyl mercury.~~

6.6 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D 1193. AWD samples collected for organic analysis may require Type II (distilled) reagent water.

7. Sample Preservation

7.1 Interaction between the sample and the atmosphere must be minimized. The sample container should be sealed as soon as possible after collection or sub-sampling. AWD samples can be easily contaminated because of the low concentration levels of their analytes. Trace metals, and possibly other ions in AWD samples, can be lost through sorption with the bottle or container in which they are stored. AWD samples may contain biologically active microorganisms which could affect the concentration of many analytes. Immediate analysis of AWD samples is best and chemical preservation should be used only when the preservation is shown not to interfere with the analysis being performed. Samples collected for total and methyl mercury determinations are to be collected directly into specially cleaned, pretested, fluoropolymer bottle(s) (19 and 20).⁷

7.2 *Samples Collected for Inorganic Cation and Anion Determinations:*

⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

⁷ The boldface numbers in parentheses refer to the list of references at the end of this standard.

7.2.1 Samples collected for pH, specific conductance, calcium, magnesium, potassium, sodium, chloride, fluoride, and sulfate analysis are often only placed in pre-cleaned plastic containers (see Section 8) before analysis. If there is a delay between time of collection and time of analysis, a preservation technique may eliminate or moderate chemical and biological changes in the AWD samples. Table 1 summarizes the recommended sample preservation techniques for AWD samples.

7.3 Samples Collected for Organic Acids:

7.3.1 Samples collected for the analysis of acetate, citrate, formate and other low molecular weight organic acid anions (C_1 - C_{12}) should be preserved within minutes after collection. Organic acids have been determined in AWD samples collected from locations around the world (1-7). These compounds (in particular formic and acetic acids) can constitute from a small fraction to mostly all of the free acidity in AWD samples. Because these acids are unstable in AWD samples, samples must be analyzed within hours after collection or else a preservation technique is required. Table 2 summarizes the recommended sample preservation techniques for AWD samples.

7.4 Samples Collected for Trace Dissolved Metals:

7.4.1 Samples collected for trace metals should be filtered to remove insoluble particulate matter often found in AWD samples unless an AWD particulate trace element analysis is desired. Filter pore size should be $\leq 0.45 \mu\text{m}$. Filter materials may contain trace elements, and the filters should be preconditioned before use by filtering 300 mL of water in order to leach soluble impurities on the filter and from the filtration apparatus itself (8). Any filters used for AWD samples should be tested to determine if the filter causes losses or gains of trace elements to the AWD sample. Test the preconditioned filters by filtering 50 mL of water and determine the analyte concentrations of interest in the filtrate.

7.4.2 Acidify the AWD sample with nitric acid to $\text{pH} \leq 2$ to minimize container adsorption of trace metals. The highest purity acid available should be used. Most AWD samples are poorly buffered and only small quantities of acid (about 1 mL/L) are required to reduce the AWD sample to a $\text{pH} \leq 2$. Samples acidified with an acid may not be used for the determination of pH and certain other analytes.

7.4.3 Acidify the AWD sample with 5 mL/L of pretested HCl for dissolved mercury and total mercury.

8. Sample Containers

8.1 Many AWD sampling networks use samplers that utilize either a bucket or bottle that is an integral component of the AWD samplers. These collection techniques allow “wet-only” samples to be obtained, meaning the bucket or bottle is only exposed to the atmosphere during the precipitation event. The preparation or cleaning procedures described in Section 8 apply to the automated AWD samplers that use buckets, and the sample storage bottles to which the AWD sample is transferred from the collection bucket. The procedure described in 8.3 could also be used as a cleaning procedure for AWD sampling that uses a bottle as the principal collection container.

NOTE 1—Any materials used for the collection and preservation of AWD should be dedicated to the use intended.

8.2 Inorganic Ions—Buckets and Lids:

8.2.1 Sample containers shall be made of materials that will not contaminate the sample and shall be cleaned thoroughly before use. Plastic containers of polyethylene and polypropylene are recommended for AWD samples collected for inorganic ions. HDPE is most commonly used.

8.2.2 Wipe the exterior of the bucket with damp cloth or brush.

TABLE 2 Preservation of AWD Samples Collected for Organic Acid Determinations

Preservation Technique	Species Determined	Remarks	Reference
HgCl ₂ or tetrachloromercurate (Na ₂ HgCl ₄) +4°C storage ^A	Low molecular weight acids	AWD samples preserved with Hg (II) can be analyzed by capillary GC-MS or for low molecular weight organic anions by ion exclusion chromatography. Sample disposal may be a problem because mercury and its compounds are a toxic waste. The preservative is usually coupled with sample storage at 4°C. See Table 1 for dissolved mercury (II) concentration in AWD samples.	(1,2)
Chloroform	Acetate, citrate formate, C ₁ -C ₅	Analysis is usually by ion exclusion chromatography because chloroform may interfere with capillary GC-MS analysis. This preservative is often coupled with sample storage at 4°C. When chloroform treatment is combined with storage in the dark at 4°C, samples are reportedly stable for 60 days. Final chloroform concentration in the AWD sample must be 0.2 % by weight.	(3,4)
Chloroform	Acetate, citrate, formate, C ₁ -C ₅	Analysis is usually by ion exclusion chromatography because chloroform may interfere with capillary GC-MS analysis. This preservative is often coupled with sample storage at 4°C. When chloroform treatment is combined with storage in the dark at 4°C, samples are reportedly stable for 60 days. Final chloroform concentration in the AWD sample must be 0.2 % by weight.	(3,4)
Freeze at - 20°C	Short-chain dicarboxylic acids C ₂ -C ₁₂	Analysis is by capillary gas chromatography or GC-MS. Sample must be completely in the liquid state before sample is prepared for analysis. Maximum storage time has not been reported.	(6)
Chill at 4°C	Low molecular weight acids	This is a minimum procedure for organic acid preservation. This preservation technique will not preserve acetate and formate longer than 3 days. Degradation may occur in less than 3 days.	(7)

^A Several of the suggested preservatives, such as tetrachloromercurate and mercuric chloride, are environmental and human health hazards and the use and disposal of these preservatives should be carefully controlled (Test Method D 2914, Section A4).

8.2.3 Clean collector buckets and lids only with water. The inside of the bucket and inner side of the lid should not come into contact with any object other than a natural sponge that has been preconditioned with water by soaking the sponge for at least 24 h.

NOTE 2—The initial preparation procedure for newly obtained natural sponges requires that the sponge is washed with copious amounts of water at least four times. Soaking a new natural sponge in 4 L of water for two or three days, after washing, is suggested.

8.2.4 Rinse the interior of the bucket two or more times with water using 100 mL for each rinse.

8.2.5 Place at least 1000 mL of water in the bucket.

8.2.6 Scrub all the inner surfaces of the bucket with the sponge and then swirl the water in the bucket to rinse the inner surfaces. Discard all remaining water.

8.2.7 Rinse the interior of the bucket two or more times with water using 100 mL for each rinse.

8.2.8 Fill the bucket to about 7.5 cm depth with water, cover with a clean lid (see 8.2.11 and 8.2.12) and store overnight.

8.2.9 Measure the specific conductance of the water in the bucket. Transfer a portion of the rinse water to a clean measuring vessel and determine the specific conductance. If the specific conductance is greater than 2 $\mu\text{S}/\text{cm}$ (Test Method D 1125), repeat 8.2.4-8.2.8.

8.2.10 Shake the inverted bucket to remove any excess water and place the bucket in a clean plastic bag, making sure that only the interior of the bag contacts the interior of the bucket. Seal the bag with rubber bands or twist ties.

8.2.11 Scrub the inner surface of the lid with the sponge. Rinse the lid with water.

8.2.12 Soak the lids for at least 24 h in water.

8.2.13 Rinse lids with water, shake free the excess water, and place lid in a plastic bag. Seal the bag with a rubber band or twist tie.

NOTE 3—All cleaning and packaging steps should be performed while wearing talc-free plastic gloves. Both hands should be gloved. Gloves should be worn for all procedures that are used to prepare materials for the collection and preservation of AWD.

8.3 *Inorganic Ions—Plastic Bottles:*

8.3.1 Sample containers shall be made of materials that will not contaminate the sample and shall be cleaned thoroughly before use. HDPE is most commonly used.

8.3.2 Soak the bottle closures (caps) in water while the bottles are being prepared.

8.3.3 Rinse each bottle a minimum of three times with water. The amount of water used for each rinse is dependent on the bottle volume. For 250-mL or smaller bottles, use about one-half the bottle volume. For bottles larger than 250 mL, use at least 200 mL of water.

8.3.4 Fill each bottle as full as possible with water and seal with the cap. Store the full bottles at least overnight (see Note 3).

8.3.5 Randomly select bottles and determine the conductivity of the bottle contents. If there is more than one bottle with a conductivity greater than 2 $\mu\text{S}/\text{cm}$, the bottles should be rewashed.

8.3.6 Slowly drain the water from the bottles, shake free any excess water, and recap the bottles immediately.

8.3.7 The bottle soak solution should be analyzed for the ions of interest on a regular basis and each time a new supply of plastic bottles is obtained.

8.4 *Trace Elements—Plastic Bottles:*

8.4.1 Sample containers must be made of a material that will not contaminate the sample and must be acid cleaned before use. HDPE and ~~fluorohydrocarbon~~ fluoropolymer bottles are most frequently used.

8.4.2 Rinse each bottle two times with water. Volume of water used for each rinse should be at least 50 mL.

8.4.3 Prepare a 1.6 N HNO_3 (1+9) (higher normality is acceptable) solution of sufficient volume to soak the bottles and caps.

NOTE 4—The appropriate concentration needed for leaching the bottles and caps has been discussed extensively in the literature, and 1.6 N HNO_3 (1 + 9) is the minimum concentration used.

8.4.4 Soak the caps in 1.6 N HNO_3 while rinsing the bottles.

8.4.5 Fill each bottle as full as possible with 1.6 N HNO_3 and seal with the cap. Store the full bottles for at least 24 h.

8.4.6 Slowly drain the acid from the bottles, shake free any excess acid, and rinse each bottle three times with water. See 8.3.3 for rinse volumes.

8.4.7 Soak the bottle caps in water while preparing the bottles in 8.4.5.

8.4.8 Refill the bottles as full as possible with water and seal with the cap. Store the full bottles at least 24 h.

NOTE 5—A literature reference recommends soaking the bottles with water until the bottles are used for sampling (9) .

8.4.9 Rinse the bottles with water before using.

8.4.10 The bottle soak solution should be analyzed for the ions of interest on a regular basis and each time a new supply of plastic bottles is obtained.

8.5 *Organic Acids:*

8.5.1 Organic materials can be leached from HDPE materials. However, the leached compound concentrations can be reduced by the cleaning and soaking procedures described for inorganic ions. Many investigators (2-7) are using HDPE containers to collect and store organic acid AWD samples; however, borosilicate glass is recommended for dicarboxylic acids.

8.5.2 Prepare the collection vessels as described in 8.2, and prepare the storage bottles as described in 8.3.

8.6 Mercury:

8.6.1 Only fluoropolymer or borosilicate glass (if mercury is the only target analyte) containers should be used.

8.6.1.1 New bottles are cleaned with warm 4 N HCl for 48 h; rinsed with water, and stored filled and capped with 1 % HCl for 12 h.

8.6.1.2 New bottles are rinsed three additional times with water, rinsed once with 0.4 % (v/v) HCl, and dried in a mercury-free Class 100 clean bench until dry.

8.6.1.3 Store all cleaned bottles tightly capped in double-bagged new polyethylene zip-type bags.

8.6.2 Used bottles are to be cleaned as in 8.6.1, except use a hot 4 N HCl wash for 6–8 h.

8.6.3 Store cleaned bottles in wooden or plastic boxes until used.

8.6.4 Soak bottle fluoropolymer caps in 4 N HCl for 48 h.

8.6.4.1 Rinse caps three times with reagent water followed by a rinse with 1 % (v/v) HCl.

9. Keywords

9.1 atmospheric wet deposition samples; collection materials; sample containers; sample contamination; sample preservation

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