



Designation: D 6328 – 98

# Standard Guide for Quality Assurance Protocols for Chemical Analysis of Atmospheric Wet Deposition<sup>1</sup>

This standard is issued under the fixed designation D 6328; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This guide describes quality assurance (QA) protocols for the determination of the anions and cations in Atmospheric Wet Deposition (AWD) shown in **Table 1**.

1.2 Included in this guide are minimum recommended requirements for the preparation of calibration standards and suggested procedures for validating laboratory measurement results.

1.3 This guide describes minimum requirements for the frequency of analysis of quality assurance samples and recommends procedures for the evaluation of quality assurance data.

1.4 The guide's recommendations are based upon expected anion and cation concentrations in AWD (**1**)<sup>2</sup> and **Appendix X2**.

1.5 *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:<sup>3</sup>

- D 596** Practice of Reporting Results of Analysis of Water
- D 1193** Specification for Reagent Water
- D 1356** Terminology Relating to Sampling and Analysis of Atmospheres
- D 3856** Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water
- D 5012** Guide for Preparation of Materials Used for the Collection and Preservation of Atmospheric Wet Deposition
- D 5015** Test Method for pH of Atmospheric Wet Deposition Samples by Electrometric Determination

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this guide.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

**TABLE 1 Common Techniques of Analysis for Atmospheric Wet Deposition Samples**

|                                                                                                                     |                                                                                                                                                                                                                 |
|---------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Automated Colorimetry<br>NH <sub>4</sub> <sup>+</sup>                                                               | Ion Chromatography<br>Cl <sup>-</sup> , NO <sub>3</sub> <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> , NH <sub>4</sub> <sup>+</sup> , Ca <sup>2+</sup> ,<br>Mg <sup>2+</sup> , Na <sup>+</sup> , K <sup>+</sup> |
| Flame Atomic Absorption Spectrophotometry<br>Na <sup>+</sup> , K <sup>+</sup> , Ca <sup>2+</sup> , Mg <sup>2+</sup> | Electrometric<br>pH, specific conductance                                                                                                                                                                       |

**D 5085** Test Method for Determination of Chloride, Nitrate, and Sulfate in Atmospheric Wet Deposition by Chemically Suppressed Ion Chromatography

**D 5086** Test Method for the Determination of Calcium, Magnesium, Potassium, and Sodium in Atmospheric Wet Deposition by Flame Atomic Absorption Spectrophotometry

**D 5111** Guide for Choosing Locations and Sampling Methods to Monitor Atmospheric Deposition at Non-Urban Locations

**E 177** Practice for Use of the Terms Precision and Bias in ASTM Test Methods

**E 178** Practice for Dealing with Outlying Observations

**E 200** Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

## 3. Terminology

3.1 **Definitions**—For definitions of terms used in this guide refer to Terminology **D 1356** or the ASTM Dictionary of Engineering Science and Technology.<sup>4</sup>

## 4. Summary of Guide

4.1 This guide describes QA procedures to be used in conjunction with standard test methods.

4.2 This guide does not include all components of a complete QA program for AWD measurement systems but provides minimum protocols to assist in the development of such a program. The procedures for the preparation of materials used for the collection and preservation of AWD are included in Guide **D 5012**. The procedures for choosing locations and sampling atmospheric deposition are included in Guide **D 5111**.

<sup>4</sup> ASTM Dictionary of Engineering Science and Technology, Ninth Edition, 2000.



## 5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available.<sup>5</sup> Other reagents may be used provided it can be demonstrated that they are of sufficiently high purity to permit their use without decreasing the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193, Type II.

5.3 *Standard Solutions*—Unless otherwise indicated, reference to standard solutions shall be understood to mean solutions conforming to Practice E 200. Standard Solutions are prepared from primary standards or ACS reagent grade salts or may be purchased as secondary standards from commercial laboratory suppliers.

## 6. Storage of Standard Solutions

6.1 TFE-fluorocarbon, polyethylene, and polypropylene containers are recommended for the storage of standard solutions. Glass containers are not suitable for storage of most standard solutions needed to analyze AWD.

## 7. Verification of Standard Solutions

7.1 Use two or more of the following procedures to ensure that the standard solutions are correctly formulated.

7.1.1 Confirmation of standard solution analyte concentration by an independent laboratory determination;

7.1.2 Confirmation of standard solution analyte concentration by an independent analytical procedure within the laboratory.

7.1.3 Comparison of the standard solution analyte concentrations of the same standard solution prepared by different analysts from the same laboratory or comparison of the analyte concentration of the new standard solution with the analyte concentration of a prior standard solution; or

7.1.4 Comparison of the analyte concentration from the standard solution with the concentration of a standard reference material (SRM) or certified reference material (CRM) (2).

7.2 The results of the confirmation analyses must be within the confidence limits of each measurement. New standard solution(s) must be prepared if the results are not in statistical agreement.

## 8. Reference Materials (RM)

8.1 The RM should be a SRM or a commercially available CRM.

8.2 Immediately following calibration (Test Methods D 5015, D 5085, D 5086), at least one RM is to be analyzed to ensure that the system is functioning properly and that stan-

dards were correctly prepared and that no degradation or contamination of the standards has occurred. The frequency of RM analysis is specified in the test method but must be at least one per analytical run.

8.3 *Evaluation of RM Data*—Compare the measured RM concentration to the certified value immediately after measurement. The analyst must ensure that the concentration value falls within the limits previously established from the repeated analysis of solutions at that concentration level. The measurement of samples must be suspended whenever the RM measurement system is out of control.

NOTE 1—If the confidence interval of the measurement intersects the confidence or tolerance interval of the RM, there is agreement. If not, then a discrepancy exists that needs to be investigated (2).

8.3.1 When the concentration of the RM differs from the certified value by greater than the established acceptance limits, reanalyze the RM immediately to determine if the current measurement is reproducible. If this second measurement also differs from the acceptance limits about the certified value, cease analyzing samples.

8.3.2 Whenever RM values indicate that the system is out of control, determine the reason and correct the condition. Reanalyze all samples measured after the last RM value that was in control.

## 9. Blanks

### 9.1 Preparation and Frequency of Analysis:

9.1.1 Prepare reagent blanks according to the procedures recommended in the appropriate test method. Use water conforming to Specification D 1193, Type I.

9.1.2 Measure reagent blanks each day determinations are performed or whenever new reagents are used to check for contamination in sample preparation or analysis.

9.1.3 Use field blanks for analytes whose expected concentrations are less than 1 mg/L. Field blanks are Type I water samples subjected to all aspects of sample collection, field processing, preservation, transportation, and laboratory handling as an environmental sample.

9.1.4 Other types of blanks may be necessary to determine the cleanliness of collection vessels, sample storage bottles, and membrane filters. Refer to Guide D 5012 for specific procedures.

### 9.2 Evaluation of Blank Data:

9.2.1 Reagent blank contamination can be highly variable depending on the source of contamination. When variable concentrations of analytes are found in reagent blanks, the source(s) of contamination should be determined and minimized.

9.2.2 Subtract the concentration of the analyte in the reagent blank from the concentration of the analyte in the sample only when the analyte concentration in the blank is low, for example <1 %, in relation to the samples being measured and its value is constant. When the analyte concentration in the blank is highly variable, reanalyze samples suspected of contamination.

9.2.3 Field blank contamination is often more variable than reagent blanks. The field blank concentrations should be used to determine if the AWD sample analyte concentration is rea-

<sup>5</sup> *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.