

Designation: D5085 - 02 (Reapproved 2013) D5085 - 21

# Standard Test Method for Determination of Chloride, Nitrate, and Sulfate in Atmospheric Wet Deposition by Chemically Suppressed Ion Chromatography<sup>1</sup>

This standard is issued under the fixed designation D5085; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This test method is applicable to the determination of chloride, nitrate, and sulfate in atmospheric wet deposition <u>samples</u> (rain, snow, sleet, and hail) by <u>ehemically</u> suppressed ion <u>ehromatography chromatography</u>. (1). For additional <u>applications</u> referapplications, see to Test Method D4327.
- 1.2 The concentration ranges for this test method are <u>as</u> listed below. The range tested was confirmed using the interlaboratory collaborative test (see <u>Table 1</u>Table 1 for statistical summary of the collaborative test).

		Range of Local	Range
	MDL	Method	Tested
	(mg/L) (2)	(mg/L)	<del>(mg/L)</del>
	Method	Range of	Range
	Detection	Method	Tested
	<u>L (mg/L) (1)</u>	<u>(mg/L)</u>	<u>(mg/L)</u>
Chloride	0.03	0.09–2.0	0.15-1.36
Nitrate	0.03 <u>ASTM 1</u>	0.09–5.0	0.15-4.92
Sulfate	talog/standargs/sist/d367d1	99-haad-40-0.09-8.070-42 ddeft 58	0.15–6.52

- 1.3 The method detection limit (MDL) is based on single operator precision (21)<sup>2</sup> and may be higher or lower for other operators and laboratories. The precision and bias data presented are insufficient to justify use at this low level, however, many workers have found level; however, it has been reported that this test method is reliable at lower levels than those that were tested. The MDLs listed above were determined following the guidance in 40 CFR Part 136 Appendix B. Other approaches to the determination of MDLs may yield different MDLs.
- 1.4 Method Detection Limits will vary depending on the type and length of column(s) used, the composition and strength of eluent used, the bore size of the instrumentation (that is, microbore or standard bore), eluent flow rate and other variables between instruments. The method detection limits listed above are those used in determining the Precision and Bias of this method as given in Table 1.
- 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 safety, health, and health environmental practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 9.

<sup>&</sup>lt;sup>1</sup> This test method 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>&</sup>lt;sup>2</sup> The boldface numbers in parentheses refer to references at the end of this test method.

TABLE 1 Precision and Bias for Chloride, Nitrate, and Sulfate Determined from the Synthetic Atmospheric Wet Deposition Samples Used in the Interlaboratory Comparison Study

Analyte Add	A 4	Mean Recovery, mg/L	Precision mg/L						
	Amount Added, mg/L		n <sup>A</sup>	$S_t^{C}$	95 % Reproducibility Limit	$S_o^D$	95 % Repeatability Limit	Bias, mg/L	Significant Bias <sup>B</sup>
Chloride	0.15	0.157	36	0.0535	0.150	0.0116	0.0325	0.007	no
	0.30	0.293	35	0.0554	0.155	0.0291	0.0815	-0.007	no
	0.68	0.652	36	0.0549	0.154	0.0237	0.0664	-0.028	biased low
	1.36	1.368	36	0.1	0.28	0.0431	0.121	0.008	no
Nitrate	0.15	0.138	24	0.0362	0.101	0.0289	0.0809	-0.012	no
	1.08	1.077	24	0.0495	0.139	0.0421	0.118	-0.003	no
	2.44	2.486	22	0.0197	0.0552	0.0183	0.0512	0.046	biased high
	4.92	4.999	24	0.126	0.353	0.075	0.21	0.079	biased high
Sulfate	0.15	0.172	36	0.055	0.154	0.0304	0.085	0.022	no
	1.43	1.442	35	0.0683	0.191	0.0369	0.103	0.012	no
	3.23	3.358	36	0.13	0.364	0.046	0.129	0.128	biased high
	6.52	6.775	36	0.37	1.04	0.109	0.305	0.255	biased high

<sup>&</sup>lt;sup>A</sup> Number of samples included in final statistical analysis after removal of outlier data.

1.6 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

# 2. Referenced Documents

2.1 ASTM Standards:<sup>3</sup>

D883 Terminology Relating to Plastics

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water Ocument Preview

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D3670 Guide for Determination of Precision and Bias of Methods of Committee D22

D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn  $2002)^4$ 

D4327 Test Method for Anions in Water by Suppressed Ion Chromatography

D5012 Practice for Preparation of Materials Used for the Collection and Preservation of Atmospheric Wet Deposition

E694 Specification for Laboratory Glass Volumetric Apparatus

E1154 Specification for Piston or Plunger Operated Volumetric Apparatus

IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System

2.2 Other Documents:

40 CFR 136 Appendix B Definition and Procedure for the Determination of the Method Detection Limit<sup>5</sup>

#### 3. Terminology

3.1 Definitions—For definitions of terms used in this test method, refer to Terminologies D883, D1129, and D1356 and Test Method D4327 and Practice IEEE/ASTM SI-10.

# 4. Summary of Test Method

4.1 Ion chromatography combines conductometric conductimetric detection with the separation capabilities of ion exchange resins. (1)A filtered aliquot of the sample, ranging in size from 505 to 250 μL, depending on instrumental system, is pumped

 $<sup>^{</sup>B}$  95 % confidence level.

 $<sup>^{\</sup>it C}$  Between laboratory precision, reproducibility.

<sup>&</sup>lt;sup>D</sup> Within laboratory precision (pooled single operator precision), repeatability.

<sup>&</sup>lt;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.

<sup>&</sup>lt;sup>4</sup> The last approved version of this historical standard is referenced on www.astm.org.

<sup>&</sup>lt;sup>5</sup> Dionex P/N 030986 (AG3) available from Dionex Corp., 1228 Titan Way, PO Box 3603, Sunnyvale, CA, 94088-3603, or equivalent has been found to be satisfactory: Available from U.S. Government Publishing Office (GPO), 732 N. Capitol St., NW, Washington, DC 20401, http://www.gpo.gov.



through an ion exchange column where the anions of interest are separated. Each ion's affinity for the exchange sites, known as its selectivity quotient, is largely determined by its radius and valence. Because different ions have different selectivity quotients, the sample ions clute from the column as discrete bands. Each ion is identified by its retention time within the exchange column. The sample ions are selectively eluted off the separator column and onto into a suppressor column, suppressor, where the conductivity of the eluent ions is reduced and the sample ions are converted to their corresponding strong acids. The separated anions are detected by a conductance cell. The chromatograms produced are displayed on a strip chart recorder or other data acquisition device, conductivity detector. Data is collected using acquisition software specific to the system in use. Measurement of peak height or area is used for quantitation. The ion chromatograph is calibrated with standard solutions containing known concentrations of the anion(s) of interest. Calibration curves are constructed from which the concentration of each analyte in the unknown sample is determined. For additional information on ion chromatography refer to Test Method D4327.

### 5. Significance and Use

- 5.1 This test method is <del>useful</del> for the determination of the anions: chloride, nitrate, and sulfate in atmospheric wet deposition.
  - 5.2 Fig. X1.1 in the appendix represents cumulative frequency percentile concentration plots of chloride, nitrate, and sulfate obtained from analyses of over 5000 wet deposition samples. These data may be used as an aid in the selection of appropriate calibration solutions (32).

#### 6. Interferences

- 6.1 Unresolved peaks will result when the concentration of one of the sample components is 10 to 20 times higher than another component that appears in the chromatogram as an adjacent peak. Decreasing the eluent concentration or flow rate, increasing eolumn length, diluting the sample with reagent water, or decreasing sample size-injection volume may correct this problem.
- 6.2 Interferences may be caused by ions with retention times that are similar to the anion of interest. The retention time of sulfite may be similar to nitrate or sulfate. Other possible interfering ions are bromide and phosphate. Before analyzing precipitation samples, measuredetermine the retention times of these possible interfering ions. Interference is common in some types of wet deposition samples. If this interference is anticipated, decreasing the eluent concentration or flow rate, increasing column length, or decreasing sample size will result in improved peak resolution.

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- 6.3 Water fromin the sample injection-will cause a negative peak (water dip)("water dip") in the chromatogram when it elutes because its conductance is less than that of the suppressed eluent. Chloride Depending on the column used, chloride may elute near the water dip and must be sufficiently resolved from the dip to be accurately quantified. This can be achieved by changing the eluent concentration or decreasing the flow rate. The potential interference of the negative peak can be eliminated by adding an equivalent of 100 ptluL of a prepared eluent concentrate (solution that is 100 times more concentrated than the eluent used for analysis) per 10.0 mL of sample. Identical eluent additions must also be included in calibration and quality control solutions.
- 6.4 Decreases in retention times and resolution are symptoms of column deterioration which may be caused by the buildup of eontaminants on the exchange resin. Refer to the manufacturer's guidelines for instructions on cleaning the column resin and column filter beds. Excising the contaminated portion of the column and changing the filters may also improve performance. If the procedure in this section do not restore the retention times, replace the column deterioration.
- 6.5 Contaminated valves and sample lines may also reduce system performance causing decreased retention times and resolutions. Refer to the manufacturer's guidelines for instructions on cleaning the valves and replacing the lines.
  - Note 1—Review operational details and refer to the trouble shooting guide in the Operator's Manual to determine the cause of decreased retention times and resolution prior to extensive cleaning or changing of all valves, columns, filters, sample lines, or all of the above.
  - 6.6 The presence of air bubbles in the columns, tubing, or conductivity detector cell may cause baseline fluctuations and peak variability. Prevent introducing air into the system when injecting samples and standards. The use of degassed water for eluents and regenerants may help to minimize minimizes the introduction of air (See 8.2).
  - 6.7 For more information on interferences refer to Test Method D4327.

# 7. Apparatus

- 7.1 *Ion Chromatograph*—Chromatograph (IC)—Select an instrument equipped with an injection valve, a sample loop, separator column(s), suppressor column(s), pump(s), and detector meeting requirements specified. Peripheral equipment includes empressed gas, a guard column, separator column, suppressor, pump(s), conductivity detector, and suitable data acquisition device such as a strip chart recorder, an integrator, or computer, and may include an automatic sampler.software. An autosampler is recommended. Compressed gas, typically high purity helium or nitrogen, may be required for some IC systems.
- 7.1.1 *Tubing*—Tubing that comes in contact with samples and standards must be manufactured from inert material such as polyethylene plastics or TFE-fluorocarbon.polyethylethylketone (PEEK) or tetrafluoroethylene (TFE).
- 7.1.2 Anion Guard Column—Also called a precolumn, it is placed before Located upstream from the separator column. The guard column contains the same resin as the separator column and is used to protect it the separator column from being fouled by particulates or organic constituents. Using an anion guard column will prolong the life of the separator column.
- 7.1.3 Anion Separator Column—This column is a column generally packed with a pellicular low-capacity anion exchange resin constructed of polystyrene-divinylbenzene beads coated with quartenary ammonium active sites resin.
- 7.1.4 Anion Suppressor Column—Place following between the separator column. This may be in the form of an anion micro-membrane suppressor or an anion self-regenerating suppressor. The first type of suppressor utilizes a semipermeable membrane containing anion exchange sites to suppress cluent conductance. Column and the detector. The second type of suppressor uses the neutralized cell effluent as the source of water for the regenerant chamber water.
- 7.1.5 Compressed Gas (Nitrogen or Air)—Helium—Use ultra-high purity 99.999 % (High purity grade.v/v) compressed gas that is oil, particulate, and water free to actuate the valves and to pressurize the regenerant flow system as needed.
- 7.1.6 *Detector*—Select a A flow-through, temperature-compensated, electrical conductivity eell with a volume of approximately 6 µL coupled with a meter capable of reading from 0 to 1000 µs/cm on an analog or digital scale.cell.
- 7.1.7 Pump—Use a pump capable both Capable of delivering a constant flow rate of approximately 1 to 5 mL/min and of tolerating a pressure 1379 to 13 790 kPa. A constant pressure, constant flow pump is recommended for enhanced baseline stability. rate. Flow rates and back pressures are dependent on the specific manufacturer's IC system. All interior pump surfaces that will be in contact with samples and standards must be manufactured from inert, non-metallic materials.
- https://standards.iteh.ai/catalog/standards/sist/d367df99-baad-40e2-a570-42ddef158bc1/astm-d5085-2
- 7.1.8 Data Acquisition System: System—
- 7.1.8.1 Recorder—This must be compatible with the maximum conductance detector output with a full-scale response time of 0.5 s or less. A two pen recorder with variable voltage input settings is recommended. A computer operating system-specific acquisition software to collect and process data.
- 7.1.8.2 *Integrator*—If an integrating system is employed, the data acquisition unit must be compatible with the maximum detector output to quantitate the peak height or area. If an integrator is used, the maximum peak height or area measurement must be within the linear range of the integrator.
- 7.1.9 Sample Loop—Select a sample loop with a capacity of 505 to 250 µL.
- 7.1.10 Sample Introduction System—Autosampler—Select one of the following: An autosampling system capable of precise delivery.
- 7.1.10.1 Syringe—A syringe equipped with a male fitting with a minimum capacity of 2 mL.
- 7.1.10.2 Autosampler—An autosampling system capable of precise delivery, equipped with a dust cover to reduce airborne contamination.
- 7.2 Eluent and Regenerant Reservoirs—Select containers with a 4 to 20 L capacity that are designed to minimize introduction of air into the flow system for storing eluents and regenerants. Reservoirs may be blanketed with helium or nitrogen per manufacturer's guidelines.

7.3 Glassware—Labware—Glassware, Glassware or plasticware, including volumetric pipettes and flasks, must be dedicated only for use onwith atmospheric wet deposition samples only. Volumetric pipettes should be used to measure the stock solutions. The pipettes may be either fixed or variable volume and either glass or plastic. Volumetric glassware samples. Volumetric glassware or plasticware must meet the requirement for Class A items given in Specification E694. Pipettes with disposable tips are preferred in order to reduce contamination. The pipettes must have a precision and a bias of 1 % or better. Precision and bias are determined by weighing a minimum of ten separately pipetted aliquots.

Note 2—More sensitive instruments may have issues with contamination from borosilicate glassware. High density polyethylene (HDPE), low density polyethylene (LDPE), or polystyrene is a suitable alternative to glass volumetrics.

7.4 Laboratory Facilities—Pipettes—Laboratories used—Fully adjustable, air-displacement pipets, for the analysis of wet deposition samples must be free from sources of contamination. The use of laminar flow clean air work stations is recommended for sample processing and preparation to avoid the introduction of airborne contaminants. Samples must always be capped orsmall-volume dispensing of aqueous fluids of moderate viscosity and density. Pipets must comply with Specification E1154 covered prior to analysis. A positive pressure environment within the laboratory is also recommended to minimize the introduction of external sources of contaminant gases and particulates. Room temperature fluctuations must be controlled to within ±°C to prevent baseline drift and changes in detector response. Windows within the laboratory must be kept closed at all times and sealed if air leaks are apparent. The use of disposable tacky floor mats at the entrance to the laboratory is helpful in reducing the particulate loading within the room for piston operated volumetric devices.

## 8. Reagents and Materials

- 8.1 *Purity of Reagents*—Use reagent grade or higher grade chemicals for all solutions. 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>6</sup>
- 8.2 *Purity of Water*—Use water conforming to Specification D1193, Type II. Point of use 0.2 µm filters are recommended for all faucets supplying water to prevent the introduction of bacteria, ion exchange resins, or both, into reagents, standard solutions, and internally formulated quality control check solutions. If degassing is necessaryperformed (see 6.6), de-gas the water prior to use by placing in a polyolefin or glass container, stirring vigorously, and aspirating off the liberated gasses.
- 8.3 <u>Eluent Solution—Eluent—(The eluent solution given here is for use with the AS3 or AS4 separator column. Other columns are available.) Sodium bicarbonate 0.0028—Eluent solutions are specific M, sodium carbonate 0.0022 M (eluent strength recommended for wet deposition analysis). Dissolve 0.941 g sodium bicarbonate (NaHCO<sub>3</sub>) and 0.933 g of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) in water and dilute to 4 L with water. Mix the solution well and de-gas before use when necessary to the column being used. Refer to column manufacturer's instructions for eluent preparation. Automated eluent generators may be used.</u>
- 8.4 Regeneration Solution: Regenerant—
- 8.4.1 Sulfuric Acid (0.009 M)—(Regenerate for the Anion Micro-Membrane Suppressor.) Add 2.02 mL of concentrated H<sub>2</sub>SO<sub>4</sub> to 2 L of water, mix well, and dilute to 4 L:Regenerant solutions are specific to the suppressor being used. Refer to manufacturer's instructions for regenerant preparation and use. Self-regenerating suppressors do not require a separate regenerant solution.
- 8.4.2 Water—Reagent water, ASTM Type I, for use with some supressors.
- 8.4.3 The self-regenerating supressors need no regenerant solution.
- 8.5 Stock Standard Solutions—Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent

<sup>&</sup>lt;sup>6</sup> Dionex P/N 030985 (AS3) available from Dionex Corp., 1228 Titan Way, PO Box 3603, Sunnyvale, CA, 94088-3603, or equivalent has been found to be satisfactory.

<sup>7</sup> Dionex P/N 35350 (AFS) or Dionex P/N 38019 (AMMS) available from Dionex Corp., 1228 Titan Way, PO Box 3603, Sunnyvale, CA, 94088-3603, or equivalent has been found to be satisfactory.

<sup>&</sup>lt;sup>6</sup> Reagent Chemicals, American Chemical Society Specifications, ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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 Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



grade materials as listed in NIST-traceable commercially-prepared 1000 mg/L solutions of chloride, nitrate 8.5.1 – 8.5.3 and dried to constant weight at 105°C. Store the solutions at room temperature in high density polyethylene or polypropylene containers.sulfate.

- 8.5.1 Chloride Solutions, Stock (1.000 mL = 1.000 mg Cl)—Dissolve 1.648 g of sodium chloride (NaCl), in water and dilute to + L.
- 8.5.2 Nitrate Solution, Stock (1.000 mL = 1.000 mg NO<sub>3</sub>)—Dissolve 1.371 g sodium nitrate (NaNO<sub>3</sub>) in water and dilute to 1 L.
- 8.5.3 Sulfate Solution, Stock (1.000 mL = 1.000 mg  $SO_4$ )—Dissolve 1.479 g anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) in water and dilute to 1 L.
- 8.6 Sample Containers—Use polyolefin or glass sample cups that have been rinsed thoroughly with water before use: analyte-free glass, high density polyethylene (HDPE) or low density polyethylene (LDPE) containers. Containers include both autosampler vials and any bottles that may be used in the transportation of the sample.

#### 9. Hazards

- 9.1 The calibration standards, sample types, and most reagents used in this test method pose limited hazard to the analyst providing routine when following typical laboratory safety precautions are practiced practices (see 9.39.2). Use a fume hood, protective clothing, and safety glasses when handling concentrated sulfuric acid.
- 9.2 Keep the doors of the instrument column compartment closed at all times when pumps and columns are in use to prevent injury to the operator from column explosion if the pump pressure or column backpressure increases.
- 9.2 Follow American Chemical Society guidelines regarding the safe handling of chemicals used in this test method.method (43).
- 10. Sampling, Test Samples Sample Preservation and Test Units
- 10.1 Some chemical constituents found in atmospheric wet deposition are not stable and must be preserved before analysis. Proper selection and cleaning of sampling containers are required to reduce the possibility of eontamination.contamination (32)).
- 10.2 For additional information on sample collection and preservation of atmospheric wet deposition refer to Guide D5012.
- 10.3 Data are reported in mg/L as CL<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, or SO<sub>4</sub><sup>2-</sup>.

# 11. Calibration and Standardization

- 11.1 Determination of Retention Times:
- 11.1.1 The retention time for each anion is determined by injecting a standard solution containing only the anionanions of interest and noting the time required for the center of a peak to appear on the chromatogram. Retention times vary with operating conditions and are influenced by the concentration of ion(s) present, the columns used, the flow rate of eluent, and the eluent composition. Prepare separate standard solutions of each anion for at least two concentrations by pipetting theas described in 11.2.2 appropriate amount of stock standard solutions into 1 L volumetric flasks and diluting with water. Analyze each standard of interest as defined in Section 11.12.8. Note the time in hundredths of minutes for each peak to appear on the chromatogram.
  - 11.1.2 A locator mix multi-anion mixed standard must be used to determine the retention time of each standard ion in solution with the others. It is prepared by Prepare as described in 11.2.2 pipetting an appropriate amount of each of the stock standard solutions into a 1 L volumetric flask and diluting with water. The concentrations of each standard chosen must be proportional to the expected concentrations of the samples. The retention times are determined by injecting the locator mix mixed standard and noting the time required for the center of each peak to appear on the chromatogram.
  - 11.2 Calibration Solutions:
- 11.2.1 A minimum of five uniformly distributed calibration solutions and one zero standard (blank) are needed to generate a

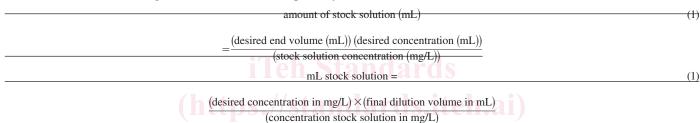


suitable calibration curve. The lowest calibration solution <u>concentration</u> must contain the analyte(s) of interest at a concentration approaching or equal to the <u>MDL</u>. <u>detection limit</u>. The highest <u>solution must approximate the 95 percentile of the expected range of the solutions being analyzed</u>. <u>calibration solution concentration is per the column manufacturer's guidelines</u>. Samples above the highest calibration standard must be diluted <u>for analysis</u>. If more than one detector sensitivity scale setting is used to increase the instrument's concentration range, calibrate at each sensitivity level using five calibration standards and one zero standard. <u>Suggested calibration standard concentrations for each analyte are listed in with reagent water for analysis</u>. <u>Table 2</u>.

11.2.2 Calibration solutions are prepared by diluting the stock standard solutions. Dedicated volumetric glassware meeting the requirement for Class A items given in Specification E694 must be used to obtain the required accuracy. Calibrated volumetric pipettes with disposable tips may also be used.

Note 2—The precision and bias of pipettes with disposable tips should be validated (5).

- 11.2.2 Standards may be prepared using two different methods. Serial dilutions are necessary when using glass volumetric pipettes. Disposable Calibration solutions are prepared by diluting the stock standard solutions. Use dedicated volumetric labware (7.3 tipped pipettes may be used for ) and calibrated volumetric pipettes with disposable tips (7.4direct dilution of stock solutions.).
- 11.2.2.1 When using pipettes with disposable tips, select either fixed or variable volume pipettes. Rinse each new tip before use with water at least three times. Aspirate and discard a minimum of three aliquots of the stock standard. Add the amount of stock solution, calculated from Eq 1, to a volumetric flask partially filled with water. Dilute to volume and mix well.



- 11.2.3.2 When preparing standards by serial dilution, it is important to not use more than three dilutions in a series. Glass pipettes must be dedicated for use with one analyte and one concentration of that analyte. Pre-rinse all glass pipettes with the analyte solution prior to preparing standards.

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- 11.2.3 <u>Standards Calibration standards</u> are stable for one week when stored at room temperature in high density polyethylene or polypropylene containers. If <u>there is evidence</u> of a change in the <del>concentration of the standards, concentration, prepare the standards more frequently. <u>Refrigeration at or below 4 °C can prolong the shelf life of the standards.</u></del>
- 11.2.4 Chloride, nitrate, and sulfate can be combined into a single solution at each of the five standard concentration levels.
- 11.2.5 For additional information on calibration refer to Test Method D4327.
- 11.3 Whenever a new cluent or regenerant solution is made, re-establish the calibration curve. Retention times may change in the middle of a run. If this occurs, regeneration of the calibration curve is necessary.

### 12. Procedure

- 12.1 Laboratory temperature must be maintained within  $\pm 3^{\circ}C \pm 3^{\circ}C$  while conducting analyses or a temperature controlled conductivity cell should be used.
  - 12.2 Use the eluent strength inconcentration recommended 8.3 for wet deposition analyses. by the column manufacturer. If peak resolution is not adequate, it may be necessary to decrease the eluent strength or increase the ionic strength of the eluent.
  - 12.3 Adjust the instrument flow rate for optimal peak <u>resolution.separation</u>. Decreasing the flow rate may provide improved peak <u>resolutionseparation</u> but lengthens retention times. Increasing the flow rate decreases peak <u>resolutionseparation</u> and shortens retention times. Refer to the manufacturer's recommendations for guidelines on optimizing flow rate.