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Designation: D3269 – 96 (Reapproved 2001)$^{\varepsilon 1}$

Standard Test Methods for Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Manual Procedures)[1]

This standard is issued under the fixed designation D3269; 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.

This standard has been approved for use by agencies of the Department of Defense.

ε^1 NOTE—Footnotes were deleted editorially throughout in March 2001.

1. Scope

1.1 These test methods describe manual procedures for the determination of fluoride in various types of samples. The procedures outlined, consequently, are appropriate to the analysis of ambient air samples taken by diverse sampling techniques when properly applied.

1.2 The values stated in SI units are to be regarded as the standard.

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.* Specific precautionary statements are given in 10.7.1.3 and Ref (**9**).

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D3267 Test Method for Separation and Collection of Particulate and Water-Soluble Gaseous Fluorides in the Atmosphere (Filter and Impinger Method)

D3268 Test Method for Separation and Collection of Particulate and Gaseous Fluorides in the Atmosphere (Sodium Bicarbonate-Coated Glass Tube and Particulate Filter Method)

D3270 Test Methods for Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Semiautomated Method)

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology D1356.

4. Summary of Test Methods

4.1 Fundamentally, isolation of the fluoride followed by analytis constitutes each of the methods. Because of the wide range of types of samples and the care required in the analysis to provide a representative sample, reliable isolation of the fluoride, and accurate measurement, the methods are prefaced by discussion of general precautions and sample preparation as applied to specific cases.

5. Significance and Use

5.1 These test methods may be used for the determination of the fluoride content of particulate matter and gases collected in the atmosphere by passive and active monitors, including plant material. The user is warned that the fluoride content of passive collectors (including plant materials) gives a qualitative or semiquantitative measure of atmospheric concentrations or deposition rates of fluorides.

6. Apparatus

6.1 *Crucible, nickel, inconel, or platinum.*

6.2 *Beakers, nickel or platinum.*

6.3 *Muffle Furnace.*

[1] These test methods are under the jurisdiction of ASTM Committee D22 on Air Quality and are the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

Current edition approved April 10, 1996. Published June 1996. Originally published as D3269 – 73 T. Last previous edition D3269 – 91$^{\varepsilon 1}$. DOI: 10.1520/D3269-96R01E01.

[2] 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.

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6.4 *Wiley Cutting Mill*.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3]

7.2 *Purity of Water*—Water shall be Type II reagent water conforming to Specification D1193. Additionally, the water used in the sampling and analytical procedure shall be demonstrated by testing with a specific ion electrode or by concentration and photometric analysis to contain less than 0.005 µg/mL of F.

7.3 *Calcium Oxide*, (CaO), with known low fluoride concentration.

7.4 *Hydrochloric Acid, (2 %)*—Dilute 5 mL of hydrochloric acid (HCl, sp gr 1.19) to 100 mL with water.

7.4.1 *Hydrochloric Acid, (4 %)*—Dilute 10 mL of hydrochloric acid (HCl, sp gr 1.19) to 100 mL with water.

7.5 *Hydrogen Peroxide Solution*, (30 %), (H_2O_2).

7.6 *Nitric Acid, (5 %)*—Dilute 5 mL of nitric acid (HNO_3, sp gr 1.42) to 100 mL with water.

7.7 *Phenolphthalein Indicator Solution (0.05 g/L)*—Dissolve 0.5 g of phenolphthalein in 60 mL of ethyl alcohol and dilute to 1 L with water.

7.8 *Sodium Hydroxide (10 %)*—Dissolve 10.0 g of sodium hydroxide (NaOH) in water and dilute to 100 mL with water.

7.8.1 *Sodium Hydroxide (20 %)*—Dissolve 20.0 g of sodium hydroxide (NaOH) in water and dilute to 100 mL with water.

7.9 *Sodium Hydroxide Alcoholic Solution*, (1 *N*)—Dissolve 4 g of NaOH in 5 mL of water and dilute to 100 mL with ethyl, methyl, or Formula 30 denatured alcohol.

8. Sampling

8.1 See Practice D1357 for general sampling procedures, and Test Methods D3267 and D3268D3267D3268 for procedures and guidelines applicable to sampling atmospheric fluorides.

9. General Precautions and Sample Preparation

9.1 *General Precautions*:

9.1.1 Fluorine is one of the more common elements and occurs in at least trace amounts in virtually all natural and manufactured materials. Contamination by extraneous fluoride may, therefore, come from such sources as sampling and laboratory apparatus, reagents, and from exposure to laboratory dust and fumes. Care must be exercised in the selection, purification, and testing of reagents and apparatus, and only minimal exposures of samples should be permitted.

9.1.2 Vessels used for evaporation, ashing, or caustic fusion of samples are first rinsed with warm, dilute HCl or HNO_3 (see 7.4 or 7.6) solution, then with water and air dried under clean toweling. Inconel crucibles used for fusion of ash may require additional cleaning by boiling in 10 % NaOH (see 7.8) solution for 1 h. Glassware is washed with hot detergent solution followed by a rinse in warm, dilute HCl or HNO_3 (see 7.4 or 7.6); it is finally rinsed with water and dried (see **Warning**). All sampling devices, containers, volumetric glassware, reagent solutions, and so forth, are stored under suitable conditions of protection from airborne dusts and fumes and are reserved for exclusive use in low-fluoride analysis. (**Warning—**The distilling flasks should be cleaned using only a brush and water. Repeated use of alkaline cleaning solution produces an etched surface that is difficult to clean and that tends to retain fluoride.)

9.1.3 Before proceeding with analysis of samples, blank determinations are repeated until satisfactorily low values (5 µg, or less, total fluoride per determination) are consistently obtained. Calibration standards are analyzed whenever new batches of reagent solutions are prepared. In addition, one blank and one standard determination are carried through the entire analytic procedure with each set of ten or fewer samples. If samples are handled in larger sets, the ratio of one blank and one standard per ten samples should be maintained.

9.2 *Sample Preparation*:

9.2.1 The techniques of sample recovery and preparation will vary, as described below, with the sampling method and equipment, and will also depend upon the procedures selected for isolation and measurement of fluoride. Until proof to the contrary is established, samples are assumed to contain fluoride in refractory forms in addition to the commonly encountered interfering materials. Many details involved in the determination of gaseous and particulate fluorides in the atmosphere and in vegetation are discussed by Pack, et al, (**1**)[4] and automatic apparatus for the determination of ambient atmospheric HF concentrations down to 0.1 ppb (v) has also been described (**2**).

9.2.2 *Particulate Fluorides*:

9.2.2.1 Particulate matter collected in air sampling generally requires fusion with NaOH for conversion into soluble form before separation of fluoride by Willard-Winter distillation (**3**). This treatment is also necessary for materials containing fluoride associated with aluminum, for materials high in silica, and for many minerals.

9.2.2.2 Transfer the sample-bearing paper filter to a resistant crucible (see 6.1), for example, nickel, platinum, or Inconel, moisten with water, and make alkaline to phenolphthalein (see 7.7) using CaO (see 7.3). After evaporation to dryness, ignite the paper in a muffle furnace at a temperature of 550 to 600°C until all carbonaceous material has been oxidized. Control combustion of filters of the membrane (cellulose ester) type by drenching with alcoholic NaOH (see 7.9) solution and igniting with a small gas flame.

[3] *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.

[4] The boldface numbers in parentheses refer to the references at the end of these methods.

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9.2.2.3 Remove particulate matter, collected by electrostatic precipitation, from the surfaces of both electrodes with the aid of a rubber policeman and water. Make the resulting suspension alkaline to phenophthalein (see 7.7) with NaOH (see 7.9) solution and evaporate to dryness.

9.2.2.4 Integrated samples, that is, those containing both gaseous and particulate fluorides, that have been collected on glass fiber filters, are not amenable to fusion; filters are transferred directly to the distillation flask. Integrated samples collected in impingers are transferred to beakers made of nickel or other resistant materials, evaporated to dryness in the alkaline condition, and the residue ashed if organic matter is present.

9.2.2.5 Fuse the impinger sample, residue from ashing of a filter, or electrostatic precipitator catch, with 2 g of NaOH. Dissolve the cold melt in a few millilitres (mL) of water, add four or five drops of 30 % H_2O_2 (see 7.5) to oxidize sulfites to sulfates, and boil the solution to destroy excess peroxide. The sample solution is then ready for isolation of fluoride.

9.2.3 *Ambient Gaseous Fluorides, Dry Collectors*:

9.2.3.1 Treat filter papers impregnated with calcium-based fixative agents as described above for particulate fluorides, except that caustic fusion of the ashed residue is not required.

9.2.3.2 Filters impregnated with soluble alkalies are leached with water, as are fixative-coated beads or tubes. Evaporate the washings in a suitable vessel and maintain in an alkaline condition during reduction to a volume convenient for the subsequent fluoride separation procedure. Add four or five drops of 30 % H_2O_2 (see 7.5) and boil the solution to destroy excess peroxide.

9.2.3.3 Gaseous fluorides collected on glass fiber filters cannot be quantitatively removed by leaching with water. Transfer such filters directly to the flasks in which Willard-Winter distillations are to be conducted.

9.2.4 *Ambient Gaseous Fluorides, Wet Collectors*:

9.2.4.1 Transfer a sample collected in water or alkaline solution to a suitably sized vessel, make alkaline to phenolphthalein (see 7.7) with NaOH (see 7.9), and evaporate to the desired volume. Treat the solution with 30 % H_2O_2 (see 7.5) and destroy the excess peroxide by boiling before proceeding with the isolation and determination of fluoride.

9.2.5 *Vegetation*:

9.2.5.1 Reduce the gross specimen to manageable size for mixing by use of hand shears or, in the case of dried materials, a Wiley cutting mill. Take a small portion (10 to 25 g) of the mixed specimen for determination of moisture by oven drying at 80°C for 24 to 48 h.

9.2.5.2 Adjust the mass of material taken for fluoride determination according to condition of the specimen: 100 to 150 g of fresh or frozen vegetation is satisfactory, while for dried materials, such as cured hay, dried leaves, straw, and so forth, a 50-g portion is adequate.

9.2.5.3 Weigh the sample into a resistant vessel, (see 6.2). Make alkaline to phenolphthalein (see 7.7) by addition of low-fluoride CaO slurry (see 7.3), and maintain alkalinity during evaporation to dryness on a hot plate. Raise the temperature of the hot plate until charring and partial ashing have occurred. Complete the ashing at 550 to 600°C in an electric furnace reserved for the ignition of low-fluoride materials. The ash should be white or gray, indicating removal of organic matter.

9.2.5.4 When the ash has cooled, pulverize it and scrape all material from the dish; mix and determine the net mass. Store in a tightly stoppered bottle.

9.2.5.5 To effect quantitative release of fluoride combined with silica in many varieties of vegetation, fusion of the limed-ash with NaOH is required and is routinely performed on all vegetation specimens (**4-6**). Transfer approximately 1 g of ash into a tared nickel, platinum, or Iconel crucible (see 6.1) and weigh accurately. Add about 5 g of NaOH pellets, cover the vessel, and fuse the contents for a few minutes over a gas burner. After cooling the melt, note its color; a blue-green color indicates the presence of manganese and treatment with H_2O_2 (see 7.5) is required as described in 11.7.2. Disintegrate the melt with hot water, washing down the lid and walls of the crucible. Reserve the resulting material for isolation of fluoride.

10. Isolation of Fluoride (Willard-Winter Distillation)

10.1 *Principle of Method*—The prepared sample is distilled from a strong acid such as H_2SO_4 or $HClO_4$ in the presence of a source of silica. Fluoride is steam distilled as the fluosilicic acid under conditions permitting a minimum of volatilization and entrainment of the liberating acid (**7**).

10.2 *Range and Sensitivity*—The Willard-Winter distillation method, on the macroscale, can accommodate quantities of fluoride ranging from 100 mg down to a few milligrams (mg).

10.3 *Interferences*—Samples relatively free of interfering materials, and containing fluoride in forms from which it is easily liberated, may be subjected to a single distillation from $HClO_4$ at 135°C. Samples containing appreciable amounts of aluminum, boron, or silica require a higher temperature and larger volume of distillate for quantitative recovery. In this case, a preliminary distillation from H_2SO_4 at 165°C is commonly used (Note 1). Large amounts of chlorides are separated by precipitation with $AgClO_4$ following the first distillation. Small amounts are held back in the second distillation from $HClO_4$ by addition of $AgClO_4$ to the distillation flask. See Note 1.

NOTE 1—Generally, samples from the atmosphere do not present a problem with aluminum and silicon inhibiting the distillation of fluoride. The presence of boron in amounts sufficient to interfere with subsequent determinations of fluoride, is also unlikely. Where there is concern over these interferences, however, the distillation and analytical methods should be selected to avoid the interferences or to establish the fact that they are not significant.

10.4 *Precision and Bias*—Recovery data for the Willard-Winter distillation, as given in the literature, are difficult to dissociate from inaccuracies inherent in various methods of sample preparation and final evaluation of fluoride. Recovery data from field samples are further complicated by variability of interfering substances and ranges of fluoride contained. In general, recoveries should be within ±10 % of the amount of fluoride present. Under favorable circumstances of sample composition and fluoride range, mean recoveries of approximately 99 % with standard deviation of about 2.5 % have been reported (**7**).

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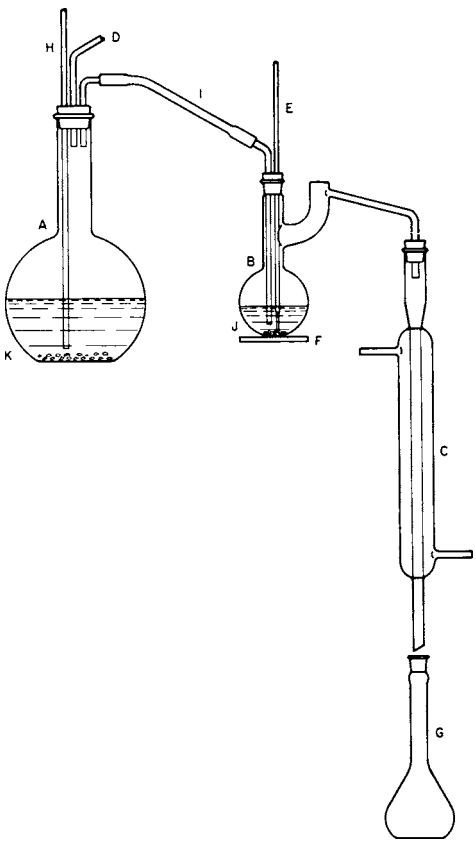
10.5 *Apparatus*:

10.5.1 *Steam Generator (see* Fig. 1*A)*—2-L Florence flask made of borosilicate glass. The flask is fitted with a stopper having at least three holes for inserting 6-mm outside diameter borosilicate glass tubing. Through one of the glass tubes, bent at right angles, steam is introduced into the distilling flask. The second tube is a steam release tube (see Fig. 1D) that controls the steam pressure. The small piece of rubber tubing that is slipped over the end of the steam release tube is clamped shut during sample distillation. The third tube is a safety tube (see Fig. 1H). If desired, other tubes may be added to permit the steam generator to supply a maximum of three distilling flasks. Any suitable heating device may be used.

10.5.2 *Distilling Flask (see* Fig. 1*B)*—A 250-mL modified Claisen flask made of borosilicate glass. The auxiliary neck of this flask is sealed and the outer end of the side tube is bent downward so that it may be attached to an upright condenser. The side tube is fitted with a one-hole rubber stopper to fit the condenser and the main neck with a two-hole stopper through which passes a thermometer and a 6-mm outside diameter heat-resistant glass inlet tube for admitting the steam. Any suitable heating device may be used.



A–STEAM GENERATOR
B–DISTILLING FLASK
C–CONDENSER
D–STEAM RELEASE TUBE
E–THERMOMETER
F–PLATE
G–RECEIVER
H–SAFETY TUBE
I–RUBBER TUBING
J–FLINTGLASS BEAD
K–BOILING CHIPS

FIG. 1 Apparatus for Distillation of Fluoride

10.5.3 *Liebig Condenser* (see Fig. 1*C*)—borosilicate glass, 300-mm jacket.

10.5.4 *Steam Release Tube* (see Fig. 1*D*)—See 11.5.1.

10.5.5 *Thermometer* (see Fig. 1*E*)—ASTM Thermometer 88C conforming to Specification E1 will be satisfactory for this purpose.

10.5.6 *Support Plate* (see Fig. 1*F*)—Metal or ceramic.The plate shall have a perfectly round 50-mm hole in which the distilling flask is placed as shown in Fig. 1. The Claisen flask must fit well in the 50-mm hole so that the flask wall, above the liquid level, is not subjected to direct heat. Excessive heat on the wall of the flask causes the liberating acid to be distilled.

10.5.7 *Receiver (*Fig. 1*G)*—250- or 500-mL volumetric flask or 400-mL beaker.

10.5.8 *Safety Tube* (Fig. 1H), 6-mm outside diameter borosilicate glass, 600 mm long, one end of which is 10 mm from the bottom of the steam generator flask.

10.5.9 *Rubber Tubing* (Fig. 1I), for flask connections, made from natural rubber. Lengths of rubber tubing shall be kept as short as possible.

10.5.10 *Flint Glass Beads* (Fig. 1J), 3 mm in diameter, for use in the distilling flask to prevent superheating and to supply silica for the formation of fluosilicic acid during distillation.

10.5.11 *Porous Pumice Stones or Boiling Chips* (Fig. 1K).

10.5.12 *Pinchcock* (Fig. 1L to control steam supply from the generator.

10.6 *Reagents*:

10.6.1 *Hydrogen Peroxide Solution*, (H_2O_2) (3 %).

10.6.2 *Perchloric Acid (70 to 72 %)*—Concentrated perchloric acid ($HClO_4$, sp gr 1.66).

10.6.2.1 *Perchloric Acid* (0.05 *N*)—Dilute 4.3 mL of 70 % $HClO_4$ to 1000 mL with water.

10.6.3 *Silver Perchlorate Solution (50 %)*—Dissolve 100 g of silver perchlorate ($AgClO_4$) in 100 mL of water.

10.6.4 *Sodium Hydroxide Pellets*, (NaOH).

10.6.5 *Sulfuric Acid (96 %)*—Concentrated sulfuric acid (H_2SO_4, sp gr 1.84) (see Note 2).

NOTE 2—Acid giving excessively high fluoride blanks requires preboiling at 135°C, with admission to steam, before addition of samples.

10.7 *Procedure*:

10.7.1 *Procedure for Single Distillation, Miscellaneous Materials*:

10.7.1.1 Fill a steam generator about two thirds full of water. Add to it one NaOH pellet and a few drops of phenolphthalein indicator solution (see 7.7) to ensure that the water remains alkaline at all times. Add a piece of pumice to permit free boiling, and heat the water to boiling. Keep the steam release tube open at this time and place a pinchcock on the steam supply tubing.

10.7.1.2 Introduce the sample into a Claisen distilling flask containing five or six glass beads. Wash down the sides of the flask with water and bring the volume to 50 to 75 mL, the lesser volume being more desirable. Insert in the main neck of the flask the rubber stopper that contains the thermometer and steam inlet tube. Set the flask in the 50-mm-diameter hole in the plate and connect the outlet to a condenser.

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10.7.1.3 Rinse the sides of the beaker or crucible that contained the sample with 50 mL of $HClO_4$ (70 to 72 %) (see 10.6.2) and add 1 mL of $AgClO_4$ solution (see 10.6.3). Transfer the rinsings to the distilling flask by means of a small funnel attached to the steam inlet tube. Rinse the beaker or crucible with water and add the rinsings to the flask. Mix the contents of the flask by gentle shaking and attach the flask to the steam generator. Place a 250-mL volumetric flask under the condenser to receive the distillate and begin heating the solution in the flask. Keep the pinchcock in place on the steam inlet tube until the contents of the distilling flask reach 135°C. (**Warning—**When using $HClO_4$, the usual precautions should be taken. Hot concentrated $HClO_4$ may react explosively with reducing substances, such as organic matter. Therefore, it is wise to see that any organic matter in the sample is destroyed in the ashing process before distillation. Precautions for the use of $HClO_4$ are available in Material Safety Data Sheet Collection (**9**).)

10.7.1.4 Remove the pinchcock on the steam inlet tube and place it on the steam release tube of the steam generator. Maintain the distillation temperature at 135 ± 2°C. Swirl the contents of the distilling flask frequently to minimize deposition on the flask wall of any siliceous residues that might retain fluoride. After collecting 250 mL of distillate during a period of about 1 h, remove the pinchcock from the steam release tube and place it on the steam inlet tube. Disconnect the rubber tubing from the steam inlet tube, and discontinue heating. (See 9.1.2.)

10.7.2 *Procedure for Single Distillation, Vegetation Ash*:

10.7.2.1 Transfer the disintegrated melt to a Claisen distilling flask (see 9.1.2), as described in 10.7.1.

10.7.2.2 Rinse the sides of the crucible in which the fusion was made with 50 mL of $HClO_4$ (70 to 72 %) (see 10.6.2) and add 1 mL of $AgClO_4$ solution (see 10.6.3).

10.7.2.3 If the sample contains manganese, add sufficient (two to ten drops) 3 % H_2O_2 solution (see 10.6.1) to the contents of the distilling flask to reduce manganese dioxide and permanganates (**10**).

10.7.2.4 Carry out the distillation as previously described, except that a 500-mL volumetric flask is used as receiver and filled with distillate during a period of about 2 h.

10.7.3 *Procedure for Double Distillation*:

10.7.3.1 Fill a steam generator, as directed in 10.7.1.1. Transfer the sample solution to a Claisen flask and rinse the sides of the beaker or crucible which contained the sample with 50 mL of concentrated H_2SO_4 (see 10.6.5). Transfer the rinsings to the distilling flask through a small funnel attached to the steam inlet tube. Mix the contents of the flask by swirling, rinse and remove the funnel, and connect the distilling flask to the steam generator. Place a 400-mL beaker under the condenser and begin heating the distilling flask and steam generator. Keep the pinchcock in place on the steam inlet tube until the contents of the distilling flask reach 165 ± 5°C. Swirl contents of the flask as required to prevent accumulation of insoluble material on the walls of the flask above the liquid level. Collect about 375 mL of distillate during a period of about 1½ to 2 h.

10.7.3.2 Add NaOH (see 7.8) to the distillate until alkaline to phenolphthalein indicator (see 7.7).

10.7.3.3 Redistill the concentrated distillate from $HClO_4$ as directed in 10.7.2. Fix small quantities of chloride in the distilling flask by the addition of 1 mL of $AgClO_4$ solution (see 10.6.3). Collect a 250-mL quantity of distillate in a volumetric flask.

11. Isolation of Fluoride (Ion Exchange)

11.1 *Principle of the Method*—The sample is freed of interferences by preferential sorption on an ion exchange resin, followed by desorption of fluoride in a small volume of eluting solution. Thus, concentration of fluoride from impinger or bubbler-collection media may be achieved without the attendant danger of contamination on prolonged exposure of solutions during evaporation (**11**).

11.2 *Range and Sensitivity*—The ion exchange procedure can be adapted to quantities of fluoride in the low-mg to µg range.

11.3 *Interferences*—Interfering cations may be eliminated by sorption of fluoride on an anion exchange resin. Fluoride is then eluted with NaOH. See Note 3.

NOTE 3—When interferences are present in cationic as well as anionic forms, both may be removed by use of a strongly basic anion exchange resin. This is accomplished by conversion of cations into strongly held complex anions, while the weakly held fluoride ions are quantitatively eluted from the column (**12**).

11.4 *Precision and Bias*—Net recoveries for quantities of fluoride of 20 µg or more should be within ±5 % of the quantity present. Low recovery indicates incomplete preconditioning of a new column, while high recovery may be due to contamination or failure to elute completely the previous sample.

11.5 *Apparatus*:

11.5.1 *Chromatographic Column*—Dimensions of the column are not critical and many types, available from suppliers' stocks, are usable. A column made of borosilicate glass tubing 10-mm inside diameter and 160 mm long, having a fritted glass disk fused into the constricted base, and a reservoir of about 100-mL capacity at the top, is satisfactory. A short piece of poly(vinyl chloride) tubing attached to the bottom and closable with a screw hose clamp permits adjustment of flow rates and prevents complete drainage of liquid from the column.

11.5.2 *Quartz Sand, White*, −60 + 120 mesh, purified by hot extraction with 20 % NaOH (see 7.8.1) solution, followed by hot 10 % HCl (see 7.8.1) solution, is used as a protective layer at the top of the resin bed.

11.6 *Reagents*:

11.6.1 *Anion Exchange Resin Intermediate-Base* of the granular aliphatic polyamine type. Mesh size is not critical but, along with column diameter and height, is a factor in controlling flow rate of solutions. Mesh sizes of −60 + 100 or −100 + 200 are usable. (See Note 4.)

NOTE 4—Separation of fluoride may be achieved with other resins and appropriate eluting solutions; for example, Dowex 1-X8 in the acetate form may be used with elution by sodium acetate solution (**13**).

11.6.2 *Hydrochloric Acid (HCl) Solution*:

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