FLUORIDE (F^- & HF) IN WORKPLACE ATMOSPHERES

Method Number:	ID-110
Matrix:	Air, Wipe (Smear Tabs for particulate fluoride)
OSHA Permissible Exposure Limits	
Final Rule Limits	
Fluorides (as F):	2.5 mg/m^3 Time Weighted Average (TWA)
Hydrogen Fluoride:	3 ppm TWA 6 ppm Short-Term Exposure Limit (STEL)
Transitional Limits	
Fluoride Dust (as F):	2.5 mg/m^3 TWA
Hydrogen Fluoride:	3 ppm TWA
Collection Procedures	
Fluorides:	A known volume of air is drawn through a cassette containing a mixed-cellulose ester (MCE) filter and backup pad using a calibrated personal sampling pump.
Hydrogen Fluoride:	A known volume of air is drawn through a three-piece cassette containing a MCE filter and a sodium-carbonate treated backup pad using a personal sampling pump.
Recommended Air Volumes:	
TWA Determinations	90 L
STEL Determinations	22.5 L
Recommended Sampling Rate:	1.5 L/min
Analytical Procedure:	Filters (MCE) are fused with sodium hydroxide, and treated back-up pads are desorbed with deionized water. Analysis is performed using an ion specific electrode and the method of standard additions. All samples are analyzed for total fluoride content.
Detection Limit	
Quantitative:	25 µg (25-mL sample solution volume)
Precision and Accuracy	
Validation Range:	350 to 700 µg load (as HF)
CV_1	0.057
Bias	-0.01
Overall Analytical Error	±12.4%
Method Classification:	Validated Analytical Method
Date (Date Revised):	December 1988 (Feb. 1991)

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1. Introduction

This method describes the collection and analysis of airborne hydrogen fluoride or particulate fluoride-containing compounds in the workplace. It is applicable for both Short-Term (STEL) and Time Weighted Average (TWA) exposure evaluations.

1.1 History

In the past, samples for determination of particulate and gaseous fluoride compounds were collected using the sampling procedure mentioned in this method with one exception. For HF sampling, the MCE filter was previously placed on the chemically-treated backup pad. The filter and treated pad are now separated within a three-piece cassette. Samples have always been analyzed at the OSHA laboratory using an ion specific electrode (ISE)/standard addition technique.

1.2 Principle

An air sample is taken by drawing a known amount of air through a cassette containing a mixed-cellulose ester (MCE) membrane filter for the collection of particulate fluoride compounds. For the collection of hydrogen fluoride (HF) gas, a sodium carbonate- treated back-up pad is placed behind the MCE filter.

The MCE filter containing particulate fluoride is fused with sodium hydroxide to facilitate solubility of the particulate. The resulting alkali flux is dried, neutralized with hydrochloric acid, and then diluted to a specified volume with deionized water. The sodium carbonate-treated back-up pad is desorbed with deionized water. The pH of each sample solution is adjusted to be within a pH of 5 to 10. Immediately before analysis, each sample is combined with Tris-Tartrate (T-T) complexing buffer solution. The concentration of each sample is determined by a standard addition technique using an ISE for fluoride.

1.3 Advantages and Disadvantages

1.3.1 This method is a simple sampling and analytical procedure able to detect fluoride over a large concentration range.

1.3.2 Analytical interferences are minimized by the addition of a buffer and use of the standard additions technique. Ions commonly associated with fluoride in work atmospheres (i.e., chloride, bromide, iodide, sulfate, nitrate, phosphate, and acetate) do not interfere with the analysis.

1.3.3 The instrumentation and sample preparation are inexpensive.

1.3.4 A disadvantage of this method is the sample preparation which involves a tedious sample flux technique.

1.3.5 Another disadvantage is the tendency for ISE readings to drift when measuring low concentrations.

1.4 Physical and Chemical Properties (8.1)

Some properties and additional information regarding hydrogen fluoride are listed below. Particulate fluoride compounds are too numerous to describe; sodium fluoride (NaF) is included below as an example of a soluble fluoride-containing particulate.

Synonyms	(HF)	Hydrofluoric acid, hydrogen fluoride
	(Fluoride)	Variety of compounds, fluoride ion, perfluoride
CAS no.	(HF)	7664-39-3
	(Fluoride)	16984-48-8

	(NaF)	7681-49-4
Physical properties		
	(HF)	Colorless, fuming mobile liquid. Attacks glass and any silicon-containing material.
	(NaF)	Clear crystals or white powder
Boiling Point		
	(HF 38% solution)	112 ℃
	(HF)	19.5 ℃
	(NaF)	1695 ℃
Density	(HF)	0.988 (liquid @ 14 ℃)
	(NaF)	2.558 (14 ℃)
Solubility	(HF)	Soluble in water
	(NaF)	Soluble in water
Physiologic effect		
	(HF)	Strong irritant to eyes, skin, and mucous membranes. Toxic by inhalation or ingestion.
	(NaF)	Tissue irritant, toxic by inhalation or ingestion.
Uses	(HF)	Aluminum production, fluorocarbons, stainless steel pickling, glass etching, oil well acidizing, gasoline production, uranium processing, automotive chromium brightening.
	(NaF)	Water fluoridation (~ 1 ppm), degassing steel, fungicide and rodenticide, wood preservative, electroplating, toothpaste additive, glass manufacture, disinfectant (in fermentation), dental prophylaxis.

2. Range and Detection Limit
   The analytical range is from 25 to 2,000 µg fluoride. Samples larger than 2,000 µg can be diluted and analyzed. An estimated detection limit of 25 µg is used. This detection limit is based on the lowest concentration standard used in the analysis.

3. Method Performance

   Quality control data (8.2) from the OSHA Technical Center (OSHA-SLTC) indicate an average recovery of 99% with a coefficient of variation of 0.057 for 60 samples spiked with sodium fluoride. The overall analytical error for these quality control samples (analyzed from 1986 to 1990) was ±12.4%. Factors which may influence precision and accuracy include electrode temperature, drift, and noise.

   The sampling portion of the method had previously been evaluated at a flow rate of 1.5 L/min (see Reference 8.3 for more information).

4. Interferences (Analytical) (8.4)

   Interference due to OH^- ion can be controlled by maintaining the pH between 5 and 10. Loss of fluoride from complex formation of fluoride ion with polyvalent cations is controlled by the addition of a buffer during analysis. Interferences can further be minimized using a standard additions technique. Formation of HF and HF^- at low pH is avoided by using a sample matrix having a pH $\geq$ 5. Interferences from complexing agents such as hydrogen ion (H^+), aluminum, silicon, or iron [$Fe^{(3+)}$] are minimized by using a buffer and a standard additions technique.

5. Sampling

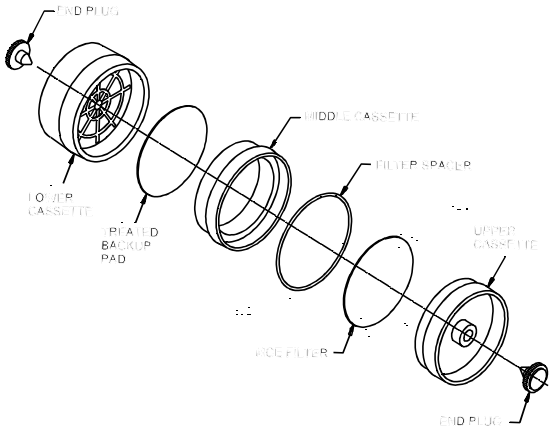
   5.1 Equipment - Air Samples (Note: Any chemicals used in sampling media preparation should be reagent grade or better)

      5.1.1 Particulate collection: Mixed cellulose ester (MCE) filters (0.8 µm pore size), cellulose backup pads for filter support, and two- or three-piece cassettes, 37-mm diameter, (part no. MAWP 037 A0, Millipore Corp., Bedford, MA).

5.1.2 Particulate and hydrogen fluoride collection: In addition to the MCE filter, a filter spacer (Cat. No. 225-23, SKC Inc., Eighty Four, PA) and a chemically-treated backup pad is also used. The spacer is a support ring for the MCE filter. The treated pad ensures capture of HF. The backup pads are treated using the following scheme:

a) Forceps
b) Pipets, 0.5 mL
c) Sodium carbonate (Na_2CO_3)
d) Glycerol ($C_3H_8O_3$)
e) Impregnation solution [Na_2CO_3 solution with glycerol] - prepare by dissolving 4.0 g Na_2CO_3 in 50 mL deionized water, add 2 mL glycerol, and dilute this solution to 100 mL with deionized water.

Using a forceps, remove the MCE filters from the three-piece cassettes and use the opened cassettes as supports for backup pad impregnation. Each backup pad should be resting on the ridge of the middle insert of the cassette and not in contact with the cassette base when impregnating.



Slowly pipet 0.5 mL of the impregnation solution over the entire backup pad and let dry overnight. Assemble the cassettes such that the backup pad resides in the lower section (the cassette outlet), and the MCE filter and filter spacer is in the upper section (the inlet) as shown.

Use the treated backup pads within 2 months of preparation

5.1.3 Gel bands (Omega Specialty Instrument Co., Chelmsford, MA) for sealing cassettes. Immediatly seal cassettes using the assembly shown in the above diagram. The cassettes must be sealed with gel bands prior to use.

5.1.4 Sampling pumps capable of sampling at 1.5 liters/min (L/min) with less than ±5% pump error.

5.1.5 Assorted flexible tubing.

5.1.6 Stopwatch and bubble tube or meter for pump calibration.

5.2 Equipment - Wipe Samples

5.2.1 Smear tabs (part no. 225-24, SKC Inc., Eighty Four, PA).

5.2.2 Deionized water.

5.2.3 Scintillation vials, 20-mL (part no. 74515 or 58515, Kimble, Div. of Owens-Illinois Inc., Toledo, OH) with polypropylene or Teflon cap liners.

5.3 Sampling Procedure - Air Samples (Also see note in Section 7.3)

5.3.1 Connect sampling media for calibration purposes to each pump and calibrate to approximately 1.5 L/min.

5.3.2 Remove the calibration media and connect the appropriate sampling media to each pump. For particulate fluoride sampling only, use the media described in Section 5.1.1. For HF and particulate fluoride, use the media listed in Section 5.1.2. Make sure the filter cassette is connected to the pump with flexible tubing such that sampled air enters the MCE filter first.

5.3.3 Place the sampling assembly on the employee or workplace area so it does not interfere with the work being performed.

5.3.4 Collect air samples at a flow rate of 1.5 L/min. Whenever possible for TWA measurements, take consecutive samples for 1 h each. Take enough samples to cover the entire workshift. Observe each cassette during sampling to make sure the filter does not become overloaded.

5.3.5 Take samples for at least 15 minutes for STEL measurements. The minimum suggested total air volume for STEL determinations is 22.5 L.

5.3.6 Replace the end plugs into the filter cassettes immediately after sampling.

5.3.7 Securely wrap each sample cassette end-to-end with an OSHA Form 21 sample seal.

5.4 Sampling Procedure - Wipe Samples

5.4.1 Wear clean, impervious, disposable gloves when taking each wipe sample.

5.4.2 Moisten the wipe filters with deionized water prior to use.

5.4.3 If possible, wipe a surface area covering 100 cm^2.

5.4.4 Fold the wipe sample with the exposed side in.

5.4.5 Transfer the wipe sample into a 20-mL scintillation vial and seal with vinyl or electrical tape. Securely wrap an OSHA-21 seal length-wise from vial top to bottom.

5.5 Shipment

5.5.1 Document the operation sampled and record other chemical substances in use.

5.5.2 Request fluoride analysis. Also request hydrogen fluoride analysis if the impregnated backup pad was used. For smear tabs, only total particulate fluoride will be analyzed and reported.

5.5.3 Submit at least one blank sample with each set of air or wipe samples. The blank sample should be handled in the same manner as the other samples except that an actual sample is not taken.

5.5.4 Ship the samples to the laboratory for analysis as soon as possible in a suitable container designed to prevent damage in transit.

6. Analysis

6.1 Safety Precautions

6.1.1 All work with concentrated acids or bases is potentially hazardous. Always wear safety glasses and protective clothing. HF and fluoride salts pose a particular hazard. All safety materials must be reviewed before handling either chemical.

6.1.2 Prepare all fusions in an exhaust hood.

6.1.3 Care should be exercised when handling any acidic or basic solutions. If any acid or base contacts the eyes, skin, or clothes, flush the area immediately with copious amounts of water. Medical treatment may be necessary. Acid or base contact with work surfaces should be avoided. Fluoride safety kits should be located near the fluoride work station. Instructions must be carefully followed.

6.1.4. Use a pipet bulb, never pipet by mouth.

6.1.5 Before using any instrument, the operator should consult the Standard Operating Procedure (SOP) (8.5) and any instrument manuals.

6.2 Equipment

6.2.1 Ion Specific Electrode (ISE) and filling solution, fluoride (Model 96-09, Orion Research Inc., Cambridge, MA).

6.2.2 Electrode, pH and filling solution (Model 81-02 Ross Combination pH electrode, Orion Research Inc.).

6.2.3 Reference electrode and filling solution (Model 90-01, Orion Research Inc.).

6.2.4 Millivolt/pH meter, capable of relative mV, pH, standard addition or concentration measurements (Model EA 940 Expandable Ionanalyzer, Orion Research Inc.).

6.2.5 Stirrer, electronic, or magnetic with Teflon stirring bars.

6.2.6 Drying oven, vacuum-assisted (Model 5851, National Appliance Co., Portland, OR).

6.2.7 Nickel or monel crucibles, 75-mL.

6.2.8 Laboratory glassware including 25-, 250-, 500-, and 1,000-mL volumetric flasks, various sizes of Class A volumetric pipets, and 20-mL glass scintillation vials.

6.2.9 Polyethylene centrifuge tubes, 100-mL.

6.2.10 Forceps.

6.2.11 Desiccator.

6.2.12 Eyedroppers or disposable Pasteur pipets.

6.2.13 Automatic pipets, adjustable, 0.1- to 5.0-mL range (models P-1000 and P-5000, Rainin Instruments Co., Woburn, MA).

6.2.14 Analytical balance (0.01 mg).

6.3 Reagents (All chemicals should be reagent grade or better)

6.3.1 Deionized water (DI H_2O).

6.3.2 Sodium hydroxide (NaOH) pellets.

6.3.3 Sodium hydroxide, 5 N: Dissolve 200 g NaOH pellets in approximately 600 mL of DI H_2O and dilute to 1-L. Store in a polyethylene bottle.

6.3.4 Sodium hydroxide, dilute: 0.5 and 0.05 N for pH adjustments.

6.3.5 Sodium fluoride (NaF).

6.3.6 Sodium fluoride stock solution, 1,000 µg/mL as F^-: Dissolve 1.10525 g NaF in DI H_2O and dilute to 500 mL. Store in a polyethylene bottle.

6.3.7 Tris(hydroxymethyl)aminomethane [$(CH_2OH)_3CNH_2$].

6.3.8 Hydrochloric acid (HCl), concentrated (36.5 to 38% w/w).

6.3.9 Hydrochloric acid, dilute (2%) for pH adjustments.

6.3.10 Sodium tartrate ($Na_2C_4H_4O_6 \bullet 2H_2O$).

6.3.11 Tris-Tartrate (T-T) buffer (concentrated): To approximately 500 mL DI H_2O add 84 mL of concentrated HCl, 242 g tris(hydroxymethyl)aminomethane and 230 g sodium tartrate. Stir to dissolve and let cool to room temperature. Dilute to 1-L with DI H_2O. Use this concentrated solution to prepare dilute T-T buffer. Do not use concentrated T-T buffer in any samples or standards.

6.3.12 Tris-Tartrate (T-T) buffer (analytical): Dilute 360 mL of concentrated T-T buffer to 2-L with DI H_2O. Use this buffer with an equal volume of sample or standard solution for analysis.

6.3.13 1:1 T-T buffer/DI H_2O (for dilutions only): Dilute equal volumes of analytical T-T buffer with DI H_2O. Use this buffer **only** for sample dilutions (i.e. when the sample displays an mV reading above the largest standard).

6.3.14 Buffer solutions, in the range of pH 4 to 10.

6.4 Standard Preparation

Prepare dilutions of the 1,000 µg/mL F^- stock standard. Use DI H_2O as the diluent and store all standards in polyethylene bottles. An example of preparation of three standards in the analytical working range is shown:

Standard	Dilution of 1,000 µg/mL Stock Standard
80. µg/mL	20 mL to 250 mL
40. µg/mL	20 mL to 500 mL
5.0 µg/mL	5 mL to 1,000 mL

Prepare 20 mL of each standard with 1:1 tris(hydroxymethyl)aminomethane:sodium tartrate added before analysis.

6.5 Sample Preparation

6.5.1 Rinse all glassware and crucibles with 10% HNO_3 and DI H_2O. Rinse all plasticware with DI H_2O. Allow labware to air dry before using.

6.5.2 MCE filters

Prepare filters (also wipe smear tabs) suspected of containing particulate fluoride as follows:

a) Carefully remove each filter from its cassette using a forceps. Place each filter in a separate, labeled 75-mL nickel or monel crucible. Record the crucible number in a log book next to the appropriate sample number. Using an automatic or glass volumetric pipet, carefully add 5 mL of 5 N NaOH to each crucible.
b) Turn on the vacuum-assisted drying oven. Place the heat setting at 90 °C and ramp the temperature slowly to 140 °C to avoid splatter and loss of sample. Maintain a drying temperature of 140 °C (Note: For the equipment mentioned in Section 6.2.6, 140 °C is approximately 85% of the maximum setting). Place the samples on a metal tray and secure with tape to prevent the crucibles from "walking" off the surface due to vibration. Place the tray containing the samples on the bottom of the drying oven. Close the oven door tightly and dry for 1 h at 140 °C.
c) Before proceeding, make sure the drying oven air vents are open to the surrounding atmosphere. Turn on the vacuum to the drying oven and then close the vents. Continue drying with the vacuum on for an additional hour. When drying is complete, open the oven air vents. When the vacuum reaches 10 mmHg or less, close off the vacuum. Open the oven door when the internal oven pressure is equal to ambient atmospheric pressure. Carefully remove the tray of samples and turn off the oven.
d) Place dried samples in a desiccator until the next step (5) is performed, since the samples will reabsorb moisture from the air if there is a time gap between removal from oven and fusing process.
e) In an exhaust hood, fuse the samples with the added NaOH by carefully heating the crucible over a Bunsen burner and slowly swirling the molten NaOH around the inside of the crucible until bubbles subside. Samples will appear translucent.

Note: If splattering occurs, you have probably lost some of the sample and results will be low. Place the crucibles back into the oven for more complete drying.

f) Allow the samples to cool, then add 10 mL DI H_2O to each crucible.
g) Carefully add 1.5 to 2 mL of concentrated HCl to neutralize the basic solution.
h) Quantitatively transfer the contents of each crucible into separate 50-mL centrifuge tubes.

6.5.3 Chemically-treated backup pads

Note: The MCE filter should always be prepared and analyzed even if the industrial hygienist specifies only an HF analysis. See Section 7.3 for more details.

Place each impregnated backup pad into a clean scintillation vial and add 20 mL of DI H_2O. Allow the pads to desorb for at least 1 h. Agitate each solution occasionally while desorbing.

6.6. Instrument Set-up

Follow the manufacturers' instructions or the SOP (8.5) for operation of the analytical instrument and electrodes. Use a battery-powered or electronic stirrer with stirring bar to stir any sample or calibration solution (Note: If a magnetic stir bar is used, make sure the bar does not contact the electrodes).

6.6.1 Connect the pH electrode (and reference electrode, if necessary) to the millivolt/pH meter. Calibrate the instrument using two buffers in the pH 4 to 10 range.

6.6.2 Individually adjust the pH of each sample to within 5 to 10 using an eyedropper or Pasteur pipet with dilute HCl or NaOH as needed. Dilute particulate fluoride samples to 25 mL.

Note: Do not use a large amount of solution to adjust the pH. A few drops should be sufficient. The desorbed solutions from the backup pads normally should not need any adjustment to achieve a pH within 4.5 to 10.5.

Rinse the electrode after each standard or sample measurement.

6.6.3 While adjusting the pH of the samples, periodically check the instrument for drift by measuring the pH buffers.

6.6.4 Connect the fluoride ISE and reference electrode leads to the appropriate sites of the instrument, place the electrodes in a standard solution, and allow to stabilize.

6.7 Analytical Procedure

6.7.1 Prepare working standards immediately before analysis as follows: Add 20 mL of dilute T-T buffer to each polyethylene beaker. Depending on the size of the sample set, prepare enough standards to analyze at the beginning, during, and at the end of the analysis. A fresh standard should be analyzed periodically throughout the analysis according to laboratory QC rules.

Note: The total microgram content of the three standards is 2,000, 1,000, and 125 µg, respectively. Other standards in this range may be used if desired.

6.7.2 Quantitatively transfer fused particulate centrifuge tube samples into 100-mL Griff beakers. Pipet 25 mL of the dilute T-T buffer into each empty centrifuge tube and then transfer these rinses into each corresponding beaker. Pipet 15 mL of each HF sample into 50 mL beakers and add 15 mL T-T buffer solution.

6.7.3 Using the mV scale on the millivolt/pH meter, scan each sample and compare the mV reading to the standards. Always rinse the ISE with DI H_2O. If any mV reading is lower (less negative) than that of the highest standard, the sample is above the calibration curve and therefore must be diluted.

6.7.4 If a sample appears to be greater than the PEL, the sample may be split into two aliquots, which can be diluted and analyzed separately. To decide whether a sample should be split for duplicate analyses, estimate the concentration of the sample using data presented in the Appendix. For example, if the sample mV reading is near the 1,000 µg standard and the air volume of the sample is near 200 L, then the estimated air concentration is about 5 mg/m^3.

Note: This calculation is only an estimate - it does not include blank corrections, Time Weighted Average calculations, etc. and is offered only as a convenience to allow for duplicate sample analyses. Final assessment of an overexposure is performed by the industrial hygienist.

6.7.5 Sample dilutions

To estimate the approximate concentration of any samples above the highest standard, apply the following rule: Doubling the concentration of the analyte will change the initial mV reading (E_o) by about 18 mV. Therefore, if the sample has an initial mV reading 18 mV less than the initial mV reading of the prepared standard, it is twice as concentrated as the standard. Similarly, a sample reading 36 mV lower than the standard is four times as concentrated. For samples that are 18 mV or less below the highest standard, pipet a 25-mL aliquot of the sample/T-T buffer mixture into a clean beaker. Add 25 mL of 1:1 T-T buffer/DI H_2O (Section 6.3.13). This results in a two-fold dilution that now is within the analytical concentration range. If further dilutions are necessary, repeat the two-fold dilutions until the sample is in the range of the standards. Save the unanalyzed portion for a duplicate analysis.

Note: The 1:1 T-T buffer/DI H_2O used for two-fold dilutions is necessary to maintain a constant ionic strength.

6.7.6 Place the fluoride and reference electrodes into a standard solution. Allow the reading to stabilize and record the reading. Remove the electrodes from the standard solution, rinse with DI H_2O. Analyze a different concentration standard (usually a ten- to twenty-fold concentration difference) and determine the slope from the two readings. Slope values using the instruments specified in Section 6.2 of this method have been approximately -56 to -59 mV.

Note: The fluoride ISE can be affected by changes in temperature. Standards and samples should be at the same temperature before analyzing. Fluctuations in the ambient temperature during analysis can sometimes be compensated for by slope adjustment (see Reference 8.4 for further details).

6.7.7 If available, use a standard additions program intrinsic within the instrument to calibrate and convert readings directly to concentration values. If an automated program is not available, record the mV reading prior to standard addition (E_o) and after addition (E_s). The "standard addition" is a 500-µL aliquot of the 1,000 µg/mL fluoride stock standard (Section 6.3.6).

6.7.8 Analyze a sample or standard (E_o). Using a glass or automatic pipet, add a 1,000 µg (as F^-) spike, and then take a final reading (E_s or concentration for automated programs). Follow the SOP for the particular instrument (8.5.) or manufacturers' guidelines. Analyze a standard in the concentration range of the samples after every tenth sample and at the end of the analysis.

7. Calculations

7.1 Determine the total µg fluoride content of each sample and blank using a concentration-response (concentration units versus µg) linear regression curve if readings were measured in concentration units.

If mV readings were taken, plot the mV readings using an appropriate standard additions program. An example of equations used for standard additions can be found in reference 8.6 or in ISE manuals.

Note: Recall that 25 mL of dilute T-T buffer and 1 mL NaF spike is added to each standard and sample. Since this volume is constant for all samples and standards, the total µg content of each sample and standard can be calculated after standard addition computation as:

µg fluoride = µg/mL fluoride x Solution Volume x Dilution Factor

Where:
Solution Volume = Standard or sample volume (mL) without the addition of T-T buffer and NaF spike.
Dilution Factor = Factor from Section 6.7.5

7.2 Each air sample is blank corrected and the concentration is then calculated to determine particulate fluoride or hydrogen fluoride exposure using the following equations:

Particulate fluoride

$$\text{mg/m}^3 \text{ fluoride} = \frac{\text{µg Sample} - \text{µg Blank}}{\text{(Air Volume, L)}}$$

Where:
µg Sample or Blank = From above calculation (Section 7.1)

Hydrogen fluoride

$$\text{ppm F} = \frac{\text{MV x (µg Sample} - \text{µg Blank)}}{\text{Molecular Weight x (Air Volume, L)}}$$

Where:
MV (Molar Volume) = 24.45 (25 ˚C and 760 mmHg)
µg Sample or Blank = From Section 7.1.
Molecular Weight (F) = 19.00

7.3 Reporting Results

Note: Problems have occurred concerning the discrimination between particulate and hydrogen fluoride (See references 8.3 and 8.7 for further details). Past studies (8.3, 8.8) have indicated that HF did not significantly react with the MCE filter, styrene cassette, or an untreated backup pad before collection on the chemically-treated backup pad; however, one study (8.3) did indicate the possibility of HF reacting with or being absorbed by particulate on the MCE filter (especially if the particulate is an adsorbent such as alumina which is common in aluminum reduction operations). A recent study (8.7) appears to indicate some reactivity of HF with the sampling media components. Due to the possibility of HF reacting with particulate on the MCE filter, the potential for underestimating HF exposure exists. The total fluoride exposure should be considered for industrial operations having alumina or other adsorbents in the air during sampling. If possible for TWA determinations, consecutive samples should be taken over the workshift, not to exceed 1-h each. This should minimize the amount of particulate on the MCE filter.

Results are reported to the industrial hygienist as follows:

For particulate fluoride (MCE filters), sample results are reported as mg/m^3 fluoride.

For chemically-treated backup pad or MFGB samples, results are reported as ppm fluoride.

8. References

8.1 Hawley, G.G.: The Condensed Chemical Dictionary. 11th ed. New York: Van Nostrand Reinhold Co., 1987.

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8.3 Einfeld, W., and S.W. Horstman: Investigation of a dual filter sampling method for gaseous and particulate fluoride. Amer. Ind. Hyg. Assoc. J. 40: 626-632 (1979).

8.4 Orion Research Incorporated: Instruction Manual, Fluoride Electrodes, Model 94-09, Model 96-09. Cambridge, MA: Orion Research Incorporated, 1977.

8.5 Occupational Safety and Health Administration Technical Center: Ion Specific Electrode Standard Operating Procedure. Salt Lake City, UT. In progress (unpublished).

8.6 Occupational Safety and Health Administration Analytical Laboratory: OSHA Manual of Analytical Methods edited by R.G. Adler (Fluoride as F^- and HF. Method No. VI-3). Salt Lake City, UT. 1977.

8.7 Lorberau, C., and K.J. Mulligan: Problem identified with NIOSH method 7902. Appl. Ind. Hyg. 3: 302 (1988).

8.8 Laboratory Services, Worker's Compensation Board of British Columbia: Hydrogen Fluoride in Air (Analytical Method No. 0751). Vancouver, B.C., Canada: Worker's Compensation Board of British Columbia, 1989.

Appendix

Calculated mg/m^3 values for F^- or HF

Concn (µg)	50	100	250	500	1,000	2,000
Air Vol (L)	(mg/m^3)					
50	1.0	2.0	5.0	10.0	20.0	40.0
100	0.5	1.0	2.0	5.0	10.0	20.0
150		0.67	1.67	3.33	6.67	13.3
200		0.50	1.25	2.50	5.0	10.0
250			1.0	2.0	4.0	8.0
300			0.83	1.7	3.3	6.7
350			0.71	1.4	2.9	5.7
400			0.62	1.2	2.5	5.0
450			0.56	1.1	2.2	4.4
500			0.50	1.0	2.0	4.0
550				0.90	1.8	3.6
600				0.83	1.7	3.3
700				0.77	1.4	2.9
800				0.71	1.2	2.5
900				0.67	1.1	2.2
1000				0.62	1.0	2.0

mg/m^3 values are based on the equation:

$$mg/m^3 \text{ analyte} = \frac{GF \times \mu g}{L \text{ air}}$$

µg = estimated reading obtained (as F^-)
GF = gravimetric factor (1 for F^-, 1.05 for HF)
PEL = 2.5 mg/m^3 F^- and 3 ppm HF
Conversion of mg/m^3 HF to ppm values can be accomplished by multiplying the mg/m^3 HF value by 1.222.