

Source Test Procedure **ST-25**

FLUORIDE, INTEGRATED SAMPLE

(Adopted January 20, 1982)

REF: Regulation 10-1-301

1. APPLICABILITY

1.1 This method is used to quantify emissions of fluoride. It determines compliance with Section 10-1-301 of Regulation 10

2. PRINCIPLE

2.1 Sample gas is drawn through a solution of 0.1 normal (0.1N) sodium hydroxide which absorbs the fluoride. The fluoride is then analyzed according to Analytical Procedure Lab-24.

3. RANGE AND SENSITIVITY

3.1 The minimum and maximum measurable concentrations of fluoride is 0.05 ppm at the sample volume specified in this procedure.

4. INTERFERENCES

Interferences are accounted for in Lab Procedure 24.

5. APPARATUS

5.1 Probe. The probe is constructed of borosilicate glass tubing.

5.2 Absorbers. Use three Greenberg-Smith impingers as absorber/condensers. The final impinger has a thermometer attached to the inlet stem.

5.3 Cooling System. Use an ice bath to contain the impingers.

5.4 Sample Pump. Use a leak-free vacuum pump capable of maintaining a 14.3 liter/min (0.5 CFM) flow rate at 15 inches of mercury. The pump must have a control valve and vacuum gauge attached to the inlet.

5.5 Silica Gel Tube. Use approximately 500 cc of silica gel (with Drierite indicator) to insure that the gas entering the dry gas meter is dry.

5.6 Dry Test Meter. Use a dry gas test meter accurate with $\pm 2\%$ of the true volume and equipped with a thermometer to measure the outlet temperature.

5.7 Connections. Use Teflon tubing in making all connections that come in contact with the sample. Vinyl tubing is acceptable for all other connections.

5.8 Barometer. Use a barometer that is accurate to within ± 0.2 inches of mercury.

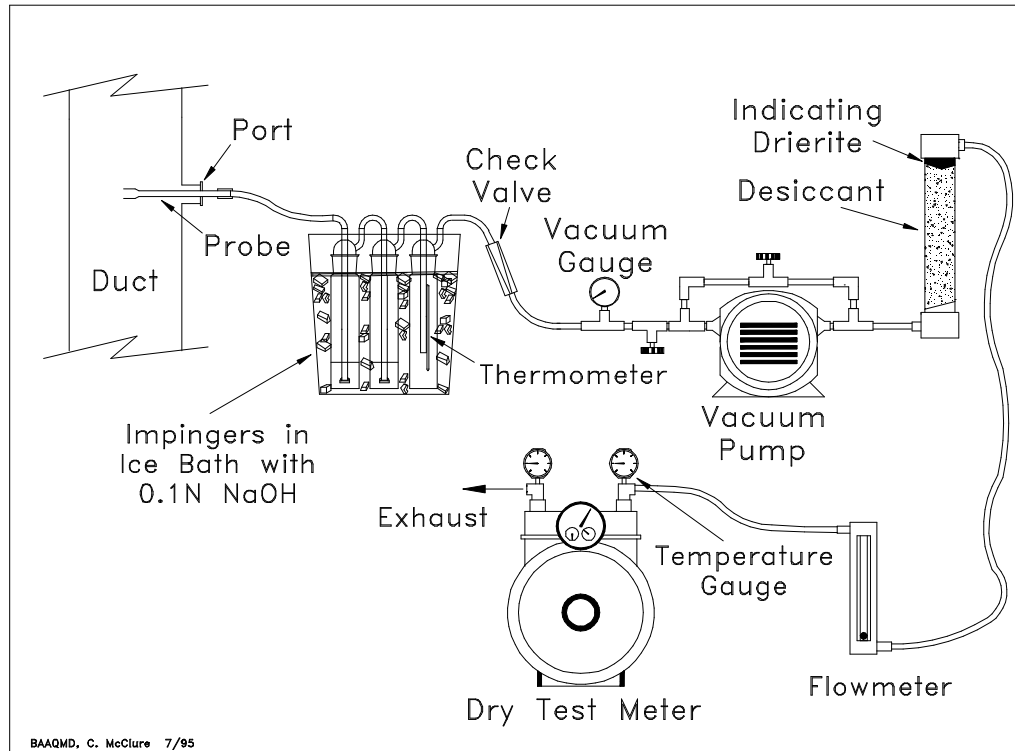
5.9 Rotameter. Use a calibrated Rotameter to measure the sampling rate.

6. REAGENTS

- 6.1 Sodium hydroxide, 0.1N. Dissolve 4.0 grams of sodium hydroxide ml concentrated HCl pellets in sufficiently distilled water to make a 1.0 liter solution.

Figure 25-1

Fluoride Sampling Train



7. PRE-TEST PROCEDURES

- 7.1 Add 100 ml of the NaOH solution to each of two impingers.
- 7.2 Stopper the impingers.
- 7.3 Retain 100 ml of the NaOH solution to analyze as a blank.
- 7.4 Assemble the sampling train as shown in Figure 25-1.
- 7.5 Leak-test the sampling train by starting the pump, plugging the probe, and adjusting the pump inlet vacuum to 10 inches Hg. The leak rate must not exceed 0.6 liter/min (0.02 CFM) through the dry test meter. Before stopping the pump, carefully release the plug in the sample probe to avoid backflow of the impinger solution.
- 7.6 Record the initial dry test meter reading and barometric pressure on the sampling data sheet, Form 25-1.
- 7.7 If there is evidence of concentration stratification, select the sampling traverse points according to ST-18. Otherwise, sample at single point.

8. SAMPLING

- 8.1 Each test run shall be of thirty minute duration when testing emissions from continuous operations. Each test run at batch process operation shall be for 90% of the batch time or thirty minutes, whichever is less.
- 8.2 Position the probe at the sampling point and start the pump.
- 8.3 Sample at a constant rate of 14.3 liter/Min (0.5 CFM) during the test as determined by the Rotameter. Use the rotameter only to establish the initial sampling rate. Then remove it from the system.
- 8.4 Record the following information at five-minute intervals:
- Dry Test Meter Temperature
Impinger Outlet Temperature
Dry Test Meter Volume
- 8.5 Add ice as necessary to maintain impinger temperatures at 7 °C (45°F) or less.
- 8.6 At the conclusion of each run, stop the pump, remove the probe from the stack, and record the final meter volume. Point the probe upward and purge the sample train with ambient air. Rinse the probe and connecting tubing with 50 ml of the NaOH solution and drain into the first impinger. Immediately transfer the impinger solutions into polyethylene bottles for subsequent analyses.
- 8.7 Take three consecutive samples.

9. POST-TEST PROCEDURES

- 9.1 Individually analyze the sodium hydroxide solutions and blank for total fluoride content according to Analytical Procedure Lab 24.

10. AUXILIARY TESTS

- 10.1 Stack gas velocity and volumetric flowrate, ST-17.

11. CALCULATIONS

- 11.1 Standard sample volume:

$$V_o = \frac{17.71V_m P_b}{T_m}$$

Where:

- V_o = Corrected sample volume, SDCF at 70°F and 29.92 inches Hg.
- V_m = Uncorrected meter volume, ft³
- T_m = Average Run Meter Temperature, °R
- P_b = Barometric pressure, inches Hg
- 17.71 = A constant correcting to 70F and 29.92 inches Hg

11.2 Fluoride concentration:

$$C = \frac{2.73 \times 10^3 (V)}{V_0}$$

Where:

- C = Fluoride concentration, ppm by volume on a dry basis
 V = Total volume of fluoride in the impinger catch, for each run, microliters.
 2.73×10^3 = A constant derived from molecular weight and correcting to 70°F and 29.92 inches Hg.

11.3 Pounds of fluoride per ton of product:

$$M = \frac{2.94 \times 10^{-6} (\text{ppm})(Q_0)}{P}$$

Where:

- M = Pounds of fluoride per ton of product
 Q_0 = Stack gas volumetric flow rate ST-17
 P = Production rate, tons per hour (daily average)
 2.94×10^6 = A constant derived from corrections to standard conditions, time and molecular weight.

12. REPORTING

Report the data indicated on Form 25-2.

Bay Area Air Quality Management District

939 Ellis Street, San Francisco, CA 94109

Form 25-1

Source Test Data Sheet

Plant # _____
 Source I.D. _____
 Sample Type _____
 Process Cycle _____
 Duct Size _____
 Duct Shape _____
 Duct Pressure _____
 Assumed %H₂O _____

Run # _____
 Temp Meter # _____
 Mag. Gauge # _____

Date: _____
 Box ΔH@ _____
 Meter (Y) _____

Nozzle Diameter _____
 Pitot Tube I.D., Cp _____
 Gas System _____
 Pbar, Barometer _____
 Leak Test Rate _____
 Time @ Point _____
 # of Points _____
 Time/Run (Min.) _____

Sampling Train: Probe # _____ Filter # _____ Imp. # _____ Imp. # _____ Pump/Box # _____

Initial Traverse Data					Sampling Data									
Trav. Point I.D.	Dist. from Wall	Duct Temp. °F	ΔP "H ₂ O	Angle of Flow	Traverse Point I.D.	ΔP "H ₂ O	Duct Temp. °F	Vs FPS	Time (minutes)	Meter Rate CFH	Meter Temp. °F	Meter Volume Ft ³	Train Vacuum "Hg	Sat'd Gas Temp. °F

Post Run Impinger Catch (ml) = _____
 Assumed O₂ = _____
 Assumed CO₂ = _____
 Post Run Calculated %H₂O = _____

Source Test Team

Comments: _____

Form 25-2

Distribution: Firm Permit Services Enforcement Services Technical Services Planning Requester DAPCO	BAY AREA AIR QUALITY MANAGEMENT DISTRICT <i>939 Ellis Street San Francisco, California 94109 (415) 771-6000</i> Summary of Source Test Results	Report No.: _____ Test Date: _____ Test Times: Run A: _____ Run B: _____ Run C: _____
Source Information		BAAQMD Representatives
Firm Name and Address	Firm Representative and Title Phone No. ()	Source Test Engineers
Permit Conditions:	Source:	Permit Services Division/Enforcement Division
	Plant No. Permit No. Operates	Test Requested By:
Operating Parameters:		
Applicable Regulations:		VN Recommended:

Source Test Results and Comments:

<u>METHOD</u>	<u>TEST</u>	<u>RUN A</u>	<u>RUN B</u>	<u>RUN C</u>	<u>AVERAGE</u>	<u>LIMIT</u>
ST-17	Stack Volume Flowrate, SDCFM Stack Gas Temperature, °F					
ST-25	Fluoride, ppmv Fluoride, pounds per ton of product					

Air Quality Engineer II	Date	Supervising Air Quality Engineer Date	Approved by Air Quality Engineering Manager
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