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Method no.:	ID-216SG
Matrix:	Air
OSHA Standard:	1 ppm
Collection Procedure:	A known volume of air is drawn through a midget glass bubbler containing 10 mL of 0.1 M NH <sub>4</sub> F.
Recommended sampling rate:	1 liter per minute.
Analytical Procedure:	The volume of the bubbler solution received is measured and diluted to volume. The sample is then analyzed by Ion Specific Electrode.
Detection Limit:	10 µg or 0.4 µg/mL of solution.
Method Classification:	Partially Validated
Date:	September 1989

## 1. Introduction

This method describes the collection and analysis of airborne Boron Trifluoride. The analysis is based on measurement of fluoroborate using an ion specific electrode.

## 2. Sampling Procedure

2.1 Ten milliliters (10 mL) of 0.1 M NH<sub>4</sub>F solution is placed in an all glass midget impinger. Air is sampled at a flow rate of 1.0 liter per minute. A maximum air volume of 480 liters is recommended. The sampling pump should have been previously calibrated with the midget impinger attached.

2.2 Following sampling, the sample solution is poured into a 20-mL glass vial. The impinger is rinsed with 2 or 3 mL of the collecting solution and the rinsings are poured into the sample vial. A Teflon lined cap is placed tightly on the vial. The cap is secured by wrapping tape around the cap in the same direction the cap is screwed on.

2.3 The sample vials are sealed with OSHA Form 21, packed and shipped to the laboratory for analysis.

## 3. Working Range and Detection Limit

3.1 A lower analytical limit of 0.5 µg/mL of BF<sub>4</sub><sup>-</sup> was selected for this method.

3.2 The working range for fluoroborate using this method is 12.5 µg to 2500 µg of BF<sub>4</sub><sup>-</sup>. This represents a range of approximately 10 µg to 1950 µg BF<sub>3</sub>.

3.3 A detection limit of 10 µg was chosen for this method. Assuming a 25 mL solution volume this allows an air volume of as low as 30 liters to give a detection limit at one tenth of the OSHA standard.

## 4. Analytical Procedure

### 4.1 Apparatus

#### 4.1.1 Sample Collection

Midget fritted glass impingers.  
Personal sampling pumps.  
20-mL glass vials with Teflon lined caps.

#### 4.1.2 Sample Analysis

Orion Model 93-05 Fluoroborate electrode or equivalent.  
Millivolt meter - Orion Model 940 IonAnalyzer or equivalent.  
Laboratory plasticware including beakers, volumetric flasks, graduated cylinders, and disposable beakers.

### 4.2 Reagents

All reagents used should be ACS analytical reagent grade or better.

4.2.1 0.1 M  $\text{NH}_4\text{F}$  for collection solution: Dissolve 3.7037 grams (g)  $\text{NH}_4\text{F}$  in deionized water. Dilute to 1 liter with deionized water. Use this solution for all dilutions of samples and standards.

4.2.2 1000  $\mu\text{g}/\text{mL}$   $\text{BF}_4^-$  standard solution: Dissolve 0.1265 g  $\text{NaBF}_4$  in 0.1M  $\text{NH}_4\text{F}$ , and dilute to 100 mL. Store the solution in a polyethylene bottle. Discard after 1 week to avoid errors introduced by hydrolysis of fluoroborate.

### 4.3 Standard Preparation

Make serial dilutions of the 1000  $\mu\text{g}/\text{mL}$  standard to make 100, 10, 5, 2, and 1  $\mu\text{g}/\text{mL}$   $\text{BF}_4^-$ . Make 100 mL of each concentration.

### 4.4 Sample Preparation

Measure the volume of each sample and dilute each sample to 25 mL with 0.1 M  $\text{NH}_4\text{F}$ .

### 4.5 Analysis of Sample

Use the fluoroborate specific-ion electrode and a double junction reference electrode. Use the 0.1 M  $\text{NH}_4\text{F}$  as the Inner filling solution for the reference electrode. Follow manufacturer's instructions for instrument operation. Analyze the samples and standards using either direct mV readings or concentration readings. Make any necessary dilutions of the samples with 0.1 M  $\text{NH}_4\text{F}$ .

## 5. Calculations

Use the OSHA Colorimetric program for calculating the results. A gravimetric factor of 0.781 is used in the calculations to convert the  $\text{BF}_4^-$  readings obtained to  $\text{BF}_3$ . A conversion factor of 0.36 is used to convert from  $\text{mg}/\text{m}^3$  to ppm air concentration.