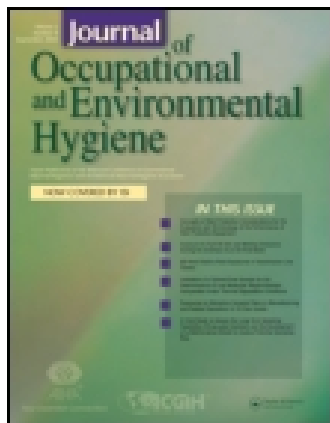


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### New NIOSH Methods for Sampling and Analysis of Airborne Inorganic Acids

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# Analytical Performance Issues

## New NIOSH Methods for Sampling and Analysis of Airborne Inorganic Acids

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### INTRODUCTION

Acid gases/vapors and aerosols are highly corrosive and can irritate the eyes and mucous membranes of the nose, pharynx, and respiratory tract, even at low airborne concentrations. Hydrofluoric acid (HF), hydrochloric acid (HCl), nitric acid (HNO<sub>3</sub>), hydrobromic acid (HBr), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), and phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) are used widely in industrial processes such as ore extraction, metal processing, pickling, electroplating, and myriad other chemical processes. It is common for several of these acids to occur simultaneously in the air of occupational settings. Applicable occupational exposure limits (OELs) for these substances in air generally range between 0.1–10 mg/m<sup>3</sup> for the various acids.<sup>(1)</sup>

The physical states of the different acids in workplace atmospheres may vary from liquid aerosols (mists) for nonvolatile acids like H<sub>2</sub>SO<sub>4</sub> to gases/vapors for volatile acids such as HCl or HNO<sub>3</sub>. Procedures for sampling and analyzing inorganic acids in workplace air must take into account the physical state(s) of the target analyte(s). Hence new National Institute for Occupational Safety and Health (NIOSH) methods for sampling and analysis of inorganic acids in workplace atmospheres, Methods 7906, 7907 and 7908,<sup>(2)</sup> have been promulgated to account for the collection of inorganic acid gases/vapors and/or aerosols. The technical contents of these new NIOSH methods are based on related recently developed International Organization for Standardization (ISO) consensus standards.<sup>(3)</sup>

### NIOSH METHOD 7906 FOR PARTICULATE FLUORIDES AND GASEOUS HYDROGEN FLUORIDE

NIOSH Method 7906 for particulate fluorides and gaseous HF updates the previous issuance of NIOSH 7906<sup>(4)</sup> by providing full method accuracy information for both analytes. Also, in the updated version, the filter media used for sampling and the sample preparation procedure for the prefilter are different from the earlier version of NIOSH 7906. The uncomplicated sampler design consists of a cellulose nitrate (0.8 μm pore size, 37-mm diameter) pre-filter placed before an alkali-treated cellulose nitrate filter, the latter of which is impregnated with sodium carbonate. (Mixed-cellulose ester (MCE) filters are unsuitable since they cause an acetate interference.) Alkali-impregnated filters are not commercially available, thus treated filters used for sampling must be prepared in the laboratory before use. Sodium carbonate-treated filters are stable for at least 2 weeks.

Sampling is carried out at normal flow rates for personal sampling pumps of 1–2 L per min. Using this sampling scheme, the pre-filter section of the sampler (i.e., filter plus internal wall deposits)<sup>(5)</sup> is meant for the collection of particulate fluorides, while the alkali-impregnated filter is used for collection of gaseous HF. Analysis of

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sample extracts is carried out by means of ion chromatography (IC) after ultrasonic extraction of fluoride from each filter using basic (high pH) aqueous buffer solutions. A significant advantage of this method is that it can allow for the collection of the inhalable fraction<sup>(6)</sup> of particulate fluorides by means of the pre-filter (housed within an optional inhalable sampler). The method<sup>(2)</sup> also specifies the use of an impregnated filter for collection of gaseous HF in lieu of a treated cellulose pad or sorbent tube<sup>(4)</sup> downstream of the untreated pre-filter. The procedure, which is consistent with ISO 21438-3,<sup>(3)</sup> replaces NIOSH 7903<sup>(4)</sup> for the determination of particulate fluorides and gaseous HF in workplace air samples by IC. The method also provides for lower limits of detection and quantitation for gaseous HF due to higher sampling flow rates than are attainable using sorbent tubes.

The revised NIOSH Method 7906 is applicable to the determination of masses of 0.005 mg to > 1 mg of particulate fluorides per sample and 0.0125 mg to > 1 mg of HF per sample. The concentration range of particulate fluorides and gaseous HF in air for which the measurement procedure is applicable is associated with the selected sampling protocol. For a 120-L air sample, the working range is approximately 0.04 mg/m<sup>3</sup> to at least 10 mg/m<sup>3</sup> for particulate fluorides and approximately 0.13 mg/m<sup>3</sup> to at least 10 mg/m<sup>3</sup> for gaseous HF. (If there is a risk of sampler overloading, consecutive samples are required for 8-hr time-weighted average (TWA) sampling.) NIOSH 7902<sup>(4)</sup> and ASTM D4765<sup>(7)</sup> are alternative methods that are recommended for the measurement of insoluble fluorides; these methods rely on the detection of F<sup>-</sup> by means of fluoride ion-selective electrodes.

In the course of method evaluation and validation, quantitative recoveries of 100% ± 2% were obtained after sample collection for both particulate fluorides and gaseous HF.<sup>(8)</sup> Also, no breakthrough of gaseous HF was observed from impregnated filters for up to 5 mg fluoride.<sup>(9,10)</sup> The component of the coefficient of variation of the method that arises from analytical variability, determined from the analysis of spiked filters, was 2.4% to 5.6% for HF and 1.7% to 3.3% for fluorides.<sup>(8,9)</sup> No effect of relative humidity (RH 20–80%) was observed on recovery of particulate fluoride over the concentration range of 0.3–5 mg/m<sup>3</sup>.

Laboratory investigations with test atmospheres of gaseous HF in the range of 0.5–5 mg/m<sup>3</sup> demonstrated quantitative sampling efficiencies (>95%) at RH of 20–60%, but recovery was reduced to ≈60% at RH values near 80%.<sup>(8,9)</sup> Yet because the influence of high humidity on HF analytical recovery was found to be reproducible and correctible (see Figure 1), the recovery  $\eta$  of HF as a function of RH > 60% at room temperature can be reliably calculated as:

$$\eta = [226.5 - (2.0914 \times \text{RH})]/100.$$

Results for precision and uncertainty of Method 7906 for test atmospheres of gaseous HF and fluoride aerosol (RH ≈50%) are summarized in Table I. The technical merits of the equivalent method (ISO 21438-3<sup>(3)</sup>) have been verified

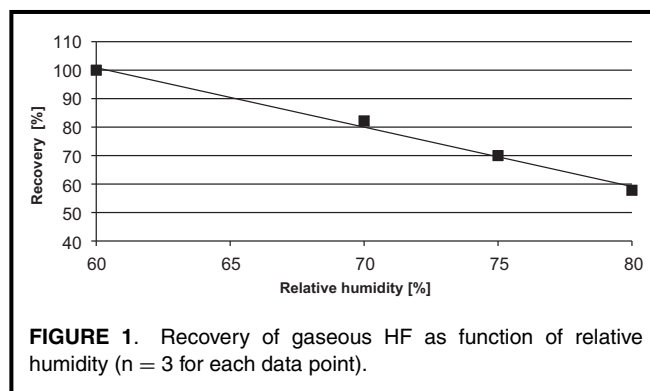


FIGURE 1. Recovery of gaseous HF as function of relative humidity (n = 3 for each data point).

through successful applications in independent laboratory and field investigations.<sup>(9)</sup>

Co-sampled formate and acetate compounds in the work environment can cause a positive interference to fluoride measurement; thus fluoride detection by ion-selective electrodes using NIOSH 7902<sup>(4)</sup> or ASTM D4765<sup>(7)</sup> is a suitable alternative if airborne formate and/or acetate are expected in samples from certain workplace environments. Cations that form insoluble fluorides, such as Ca<sup>2+</sup>, Mg<sup>2+</sup>, Fe<sup>3+</sup>, and Al<sup>3+</sup>, can cause negative interferences; in such cases the use of more rigorous sample dissolution schemes as described in NIOSH 7902<sup>(4)</sup> and/or ASTM D4765<sup>(7)</sup> would be required.

#### NIOSH METHOD 7907 FOR VOLATILE ACIDS: HYDROCHLORIC, NITRIC, AND HYDROBROMIC ACIDS

In NIOSH Method 7907,<sup>(2)</sup> similarly to Method 7906, the sampler for the volatile acids HCl, HNO<sub>3</sub>, and HBr also contains a 37-mm diameter pre-filter and impregnated filter in series; however, instead of cellulose nitrate, the sampling filter and prefilter are comprised of quartz fiber.<sup>(2)</sup> Sample collection

TABLE I. Performance Evaluation for HF and F<sup>-</sup> Measurements from Test Atmospheres<sup>(8)</sup>

Analyte	Conc. (mg/m <sup>3</sup> )	s <sub>r</sub> (n = 6) <sup>A</sup>	RSD (%) <sup>B</sup>	U (%) <sup>C</sup>
HF	0.25	0.007	2.8	22
HF	1.2	0.044	3.7	21
HF	2.3	0.026	1.1	20
HF	5.0	0.099	2.0	11
Fluoride	0.04	0.002	5.0	19
Fluoride	0.52	0.006	1.2	12
Fluoride	2.95	0.009	0.3	11
Fluoride	5.9	0.065	1.1	11

<sup>A</sup> Standard deviation for 6 test samples.

<sup>B</sup> Percent relative standard deviation.

<sup>C</sup> Expanded measurement uncertainty;  $k = 2$  (calculated in accordance with EN 482<sup>(11)</sup>).

is performed using typical personal sampling pump flow rates for filtration sampling of 2 L per min. The NIOSH 7907 procedure, which is consistent technically with ISO 21438–2,<sup>(3)</sup> is intended to replace NIOSH 7903<sup>(4)</sup> for the determination of HCl, HNO<sub>3</sub>, and HBr in workplace air samples by means of silica sorbent tubes. With application of NIOSH 7907, the impregnated quartz fiber filter collects HCl, HNO<sub>3</sub>, and HBr vapor, while the pre-filter functions to capture potentially interfering airborne particles and mists. Ultrasonic extraction of chloride, nitrate, and/or bromide from the impregnated quartz filter is then carried out using a basic buffer solution, and dissolved Cl<sup>-</sup>, NO<sub>3</sub><sup>-</sup> and/or Br<sup>-</sup> anions are subsequently measured by IC. The main advantage of this method is that it provides for lower limits of detection and quantitation for these volatile acids due to higher sampling flow rates (up to 5 L per min) that cannot be attained by using sorbent tube samplers. Optional analysis of the pre-filter for other analytes of interest (along with internal wall deposits)<sup>(5)</sup> can be performed if measures of co-sampled airborne particulate matter are desired.

In the course of method validation using sodium carbonate-impregnated quartz fiber filters,<sup>(10,12,13)</sup> the estimated method detection limit (MDL) was 0.003 mg/filter for all three acids; this corresponds to an airborne concentration of 0.0012 mg/m<sup>3</sup> for a 240-L air volume. The working range was found to be (at least) 0.01 to 2.0 mg/sample for a 240-L air sample. (If there is a risk of sampler overloading, consecutive samples would be required for 8-hr TWA sampling.) Sample stability was demonstrated by recoveries of >95% for hydrochloric and nitric acid 4 weeks after sample collection, and no breakthrough was observed at sample loadings of up to 2.5 mg HCl or 5 mg HNO<sub>3</sub>.<sup>(12,13)</sup>

In these investigations, mean analytical recoveries determined from the analysis of spiked filters were found to be in the range of 97–100% for the three acids HBr, HCl, and HNO<sub>3</sub>. The component of the coefficient of variation of the method that arises from analytical variability, as determined from the analysis of filters sampled at a dynamic test gas apparatus, was 0.8% to 1.3% for HCl and 1.1% to 1.8% for HNO<sub>3</sub>; and as determined from the analyses of spiked samples of HBr, this value was 0.8% to 1.4%.<sup>(12)</sup> The method has been independently verified for all three acids<sup>(13)</sup> in accordance with applicable performance criteria.<sup>(10)</sup> An inter-laboratory study with five participants found negligible biases and good agreement for HCl and HNO<sub>3</sub> at concentrations between 0.6 and 8 mg/m<sup>3</sup> for HCl and 0.8 and 10 mg/m<sup>3</sup> for HNO<sub>3</sub>.<sup>(14)</sup> Expanded method uncertainty estimates found were  $U < 12\%$  for HCl and HBr and  $U < 14\%$  for HNO<sub>3</sub>.

Volatile inorganic acids can react with co-sampled particulate matter on the pre-filter, potentially leading to low measurement results. One such example is in the galvanizing industry, where the presence of zinc oxide can be a major confounding factor in the measurement of HCl in occupational atmospheres. Also, potentially interfering particulate chlorides and nitrates removed by the pre-filter can react with sampled

strong non-volatile airborne acids (such as H<sub>2</sub>SO<sub>4</sub>) and liberate HCl and HNO<sub>3</sub> that is subsequently collected on the sampling filter, leading to results that are biased high.<sup>(14)</sup>

## NIOSH METHOD 7908 FOR NON-VOLATILE ACIDS: SULFURIC AND PHOSPHORIC ACIDS

NIOSH Method 7908 specifies the use of a 37-mm diameter quartz fiber filter or 0.45- $\mu$ m pore size polytetrafluoroethylene (PTFE) filter for the collection of H<sub>2</sub>SO<sub>4</sub> and/or H<sub>3</sub>PO<sub>4</sub> in workplace air.<sup>(2)</sup> Since these acids exist as liquid aerosols (mists) in occupational atmospheres, no pre-filter is required or desired in the sampler. Similarly to NIOSH Methods 7906 and 7907, sample extraction is carried out using sonication of the sample filters (plus material rinsed from internal cassette walls)<sup>(5)</sup> in basic buffer solution. This procedure, which is technically consistent with ISO 21438–1,<sup>(3)</sup> replaces NIOSH 7903<sup>(4)</sup> for the determination of H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> in workplace air samples by IC. (The use of sorbent tubes for collecting acid mists is not appropriate or fit for purpose.) A significant advantage of this method is that it can allow for the collection of the inhalable fraction of H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> aerosols in situations where an inhalable sampler is employed.

In method validation on quartz fiber filters, 97–100% recovery of H<sub>2</sub>SO<sub>4</sub> was obtained 4 weeks after sample collection, and no breakthrough was observed at sample loadings of up to 1 mg H<sub>2</sub>SO<sub>4</sub> per sample.<sup>(15)</sup> Laboratory testing with generated atmospheres of H<sub>2</sub>SO<sub>4</sub> mist yielded a collection efficiency of >95% over the range 0.5 to 10 mg/m<sup>3</sup> of H<sub>2</sub>SO<sub>4</sub> on 0.45- $\mu$ m pore size PTFE filters;<sup>(16)</sup> also, >95% recovery of H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> was found 4 weeks after sample collection. Mean analytical recoveries determined from the analysis of spiked quartz fiber filters has been found to be quantitative for both acids.<sup>(16,17)</sup> The component of the coefficient of variation of the method that arises from analytical variability, determined from the analysis of spiked quartz fiber filters, was 0.7% to 3.2% for H<sub>3</sub>PO<sub>4</sub> and 0.5% to 2.6% for H<sub>2</sub>SO<sub>4</sub>. An inter-laboratory study with 26 participants found negligible biases and inter-laboratory relative standard deviations of 12–15% for H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> concentrations between 0.05 and 1 mg/m<sup>3</sup>.<sup>(18)</sup> The method has also been successfully field tested for workplace H<sub>2</sub>SO<sub>4</sub> measurements at sample volumes of up to nearly 2000 L.<sup>(19)</sup> While some limit values for H<sub>2</sub>SO<sub>4</sub> pertain to the thoracic fraction, the use of inhalable samplers has been found to be appropriate.<sup>(18)</sup>

The working range of NIOSH Method 7908 was evaluated to be (at least) H<sub>2</sub>SO<sub>4</sub> = 0.002 to 1.0 mg/sample and H<sub>3</sub>PO<sub>4</sub> = 0.004 to 1.0 mg/sample for a 420-L air sample.<sup>(18,19)</sup> Estimated MDLs for H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> were 0.002 mg/m<sup>3</sup> and 0.003 mg/m<sup>3</sup> for a sampled air volume of 1 m<sup>3</sup>. Expanded measurement uncertainty obtained for both acids was <23%.<sup>(17)</sup> It should be underscored that particulate salts of sulfate and/or phosphate will give a positive interference.

## SUMMARY

Newly published NIOSH methods for inorganic acids, that are technically harmonized with relevant parallel ISO standard, have been promulgated. These methods have been extensively validated and represent significant improvements in sampling and analytical methodologies for inorganic mists and vapors in workplace atmospheres. The equivalent constructs of method accuracy, as defined by the NIOSH Accuracy Criterion,<sup>(20)</sup> and expanded uncertainty, as elucidated in EN 482<sup>(11)</sup> and ASTM D7440<sup>(21)</sup> (which apply the ISO/IEC *Guide to the expression of uncertainty in measurement* (GUM)<sup>(22)</sup> to air quality measurements), have been previously explained by Bartley.<sup>(23)</sup> Under these considerations is intended that, in the near future, additional occupational exposure monitoring methods based on international voluntary consensus standards will be incorporated into the *NIOSH Manual of Analytical Methods*.

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