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DETERMINATION OF SUBMICROGRAM
AMOUNTS OF BERYLLIUM IN
AIR FILTER SAMPLES BY NONFLAME
ATOMIC ABSORPTION SPECTROMETRY

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SUBJECT DESCRIPTORS

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DETERMINATION OF SUBMICROGRAM AMOUNTS OF BERYLLIUM IN AIR FILTER SAMPLES BY NONFLAME ATOMIC ABSORPTION SPECTROMETRY

Jeffrey A. Hurlbut and Dale L. Bokowski

Abstract. A routine method of analysis for nanogram amounts of beryllium collected on air filters is described. A filter is ashed in oxidizing acids, diluted with sulfuric acid (H_2SO_4) and water, and analyzed with a single-beam, nonflame atomic absorption spectrometer. Aqueous standards containing 0.9M H_2SO_4 are used; the sensitivity and detection limits are 1.0×10^{-12} g and 0.4×10^{-12} g respectively. Four nanograms of beryllium per paper filter are recoverable with a relative accuracy of $\pm 5\%$ and a relative standard deviation of $\pm 8\%$. The aluminum in glass filters causes a nonatomic absorption peak that interferes with the beryllium atomic absorption peak. As a result, the detection is limited to about $0.1 \mu\text{g}$ of beryllium per glass filter. Perchloric acid (HClO_4) and hydrofluoric acid (HF) also interfere with the absorption signal and have to be removed during digestion.

INTRODUCTION

The toxicity of some forms of beryllium is well documented, but the mechanism of beryllium action in biological systems is still the subject of research.¹⁻⁵ Trace levels of beryllium compounds can cause toxic effects in the body,¹ and the most common way for beryllium to enter into the body is by inhalation of respirable size particles (less than five micrometres). Beryllium compounds can accumulate in the lungs and lead to the chronic form of beryllium disease from one to fifteen years later;¹⁻⁵ therefore, analytical procedures for monitoring beryllium in air filter samples must be as sensitive as possible. Since hundreds of air filter samples may be taken weekly or even daily, the analytical procedure should be rapid.

A thorough search of the literature¹ revealed that there are three, potentially suitable, beryllium

analysis procedures: gas chromatography,⁶⁻⁸ flame atomic absorption spectrometry,^{9, 10} and nonflame atomic absorption spectrometry. Of these three procedures, nonflame atomic absorption spectrometry appears to be the most promising in terms of ease of analysis, determination of nanogram amounts of beryllium per filter, and speed of analysis.¹¹⁻¹³

The purpose of this report is to describe the application of the heated graphite furnace, atomic absorption technique for determining submicrogram quantities of beryllium in air filter samples.

EXPERIMENTAL

Apparatus

A Varian Techtron Model 1000, single beam, atomic absorption spectrophotometer and a Model 20 Varian, 1-mV chart recorder were used in conjunction with a Varian Techtron Model 63, carbon rod atomizer. The atomizer consisted of a power supply, a gas control unit, and a workhead. Only nitrogen was used as the inert gas, and the flow rate was 4.0 litres per minute. The support rods were made of RWO spectrographic grade graphite (Ringsdorff-Werke GMBH, Bonn-Bad Godesberg, W. Germany). Sample cups and rods were pyrolytic graphite coated (Ultra Carbon Corp., Bay City, Michigan). Solutions were introduced with 5- and 10- μl Eppendorf-type pipettes.

A Perkin-Elmer Model 303, atomic absorption spectrophotometer equipped with a DCR-1 concentration readout, a single slot burner, and nitrous oxide-acetylene gas was used for the comparison study.

FIGURE 1. Plot of Absorbance versus Concentration for Beryllium Standards in 1M HNO₃ Using the Carbon Cup.

FIGURE 2. Plot of Absorbance versus Concentration for Beryllium Standards in 1M HNO₃ Using the Carbon Rod.

Reagents

Reagent-grade acids and singly distilled water were free of picogram quantities of beryllium. All glassware was washed with dilute nitric acid (HNO_3). The dilute beryllium standards were prepared from a 1000-ppm Ventron, Alfa Inorganics Standard 88053. All dilute stock solutions of beryllium were 1% (V/V) in HNO_3 , and dilute standards were stable for at least two weeks. Whatman No. 41, 4.7-cm paper filters, and Gelman Type E, 4.7-cm glass filters were used.

Procedure

Sample Preparation

The digestion procedure for paper filters consists of heating the filters in a 30- or 50-ml beaker with 6 ml of 5:1 HNO_3 : HClO_4 in a wash-down fume hood until dryness is reached. About 2 ml of HNO_3 is added then, and the mixture is again heated to dryness. The remaining salts are taken up in either hot dilute HNO_3 or hot 6M H_2SO_4 , placed in a volumetric flask, mixed with the appropriate amount of H_2SO_4 (5 ml/100 ml), and brought to volume with water.

The digestion procedure for glass filters consists of wetting the filter with HNO_3 in a platinum crucible, adding about 5 ml of HF, and heating to dryness. This adding and drying sequence is then repeated with 3 ml of HF, with 3 ml of 1:1 HNO_3 : HClO_4 , and with 3 ml of HNO_3 . The salts are taken up in hot 6M H_2SO_4 . Additional H_2SO_4 is added so that the final solution is 5% (V/V) in H_2SO_4 , and the mixture is filtered through Whatman No. 40 filter paper into a volumetric flask. This filtration is needed to remove the insoluble sulfate salts. Beryllium apparently is not coprecipitated.

Analysis

The graphite rods and cups were adjusted for maximum transmittance. The nonflame atomic absorption spectrometer was operated according to manufacturer's recommendations (wavelength equals 235 nm, slit equals 0.5 mm, current equals 6.0 mA). Five- μl samples were used with the

carbon rod, and 10- μl samples generally were used with the carbon cup. The furnace was programmed as indicated below. The times and settings for ashing and drying varied somewhat depending upon the type and amount of acid used.

Step	Carbon Cup	Carbon Rod
Drying	50 sec at 3.7	25 sec at 4.2
Ashing	15 sec at 4.5	15 sec at 4.0
Atomization	3 sec at 8.0	3 sec at 8.0

The rod had to be retightened and readjusted after every 10 to 20 runs. This resulted in larger standard deviations.

RESULTS AND DISCUSSION

Relationship Between Absorbance and Concentration for Beryllium

Figures 1 and 2 are calibration plots of absorbance versus concentration, in ng/ml, for solutions that are 1M in HNO_3 . The carbon cup and carbon rod were used for Figures 1 and 2 respectively.

The sensitivity (amount of beryllium that yields 1% absorption) and detection limits (the amount of beryllium that will give a signal twice that of the baseline signal)¹⁴ are 4×10^{-12} g and 2×10^{-12} g, respectively, for the carbon cup and 0.8×10^{-12} g and 0.3×10^{-12} g, respectively, for the carbon rod. Similar detection limits were obtained by an alternate procedure.¹⁵ Five- μl samples were used for both the rod and cup with the 1M HNO_3 .

Figures 3 and 4 are similar plots for solutions that are 0.9M (5% V/V) in H_2SO_4 . The sensitivity and detection limits are 6×10^{-12} g and 2×10^{-12} g, respectively, for the carbon cup with 10- μl samples, and are 1.0×10^{-12} g and 0.4×10^{-12} g, respectively, for the carbon rod with 5- μl samples.

Effect of Acids on Beryllium Recovery

Since Sighinolfi previously indicated that both the type and concentration of acid were variables,¹²

FIGURE 3. Plot of Absorbance versus Concentration for Beryllium Standards in 0.9M H_2SO_4 Using the Carbon Cup.

FIGURE 4. Plot of Absorbance versus Concentration for Beryllium Standards in 0.9M H_2SO_4 Using the Carbon Rod.

we decided to further investigate the effects of acids on the absorbance. Table 1 summarizes the absorbance effects for 1M acids on 5.0 ng/ml beryllium standards. Small differences in absorbance were mainly the result of different splattering characteristics of the acid solutions during the ashing step; however, large depressant effects were seen when HClO_4 and HF were present. A hydrogen continuum lamp indicated that either or both large scattering and molecular absorption peaks (nonatomic absorbance) occurred for both phosphoric acid (H_3PO_4) and a HF- HClO_4 mixture. The nonatomic peak for H_3PO_4 was eliminated by increasing the ash setting to 7.5, but the nonatomic absorbance from HF- HClO_4 could not be separated effectively from the beryllium atomic absorbance peak.

Sulfuric acid acts as a masking agent for the calcium interference;¹² so, the potential masking effect of various acids was investigated. Tables 2 and 3 summarize the effects of 1M acids in the presence of other 1M acids and 0.1M acids. Hydrofluoric acid (HF), H_3PO_4 , and HClO_4 drastically lower the recovery of beryllium from 1M HNO_3 and from 1M HCl solutions. The apparent loss is between 10 and 40% in the presence of 1M H_2SO_4 . Hydrofluoric acid, H_3PO_4 , and HClO_4 also cause bothersome nonatomic absorbance. These interfering acids should be removed from any sample during the sample workup. All air filter samples were therefore ashed to dryness.

Based on the results given in Tables 2 and 3, and based on the work by Sighinolfi,¹² H_2SO_4 was added to all air filter samples. Finally, the carbon cup and 10- μl samples were used for these acid studies, but 5- μl sized samples would have given better precision since splattering occurred with the larger sample size.

Recovery of Beryllium from Spiked Paper Filter Samples

Table 4 summarizes recovery data for paper air filter samples spiked with beryllium. The filters were spiked with various amounts of beryllium, and were dried, digested, diluted with H_2SO_4 and

water, and run using either the carbon cup or carbon rod. The relative standard deviation varied from 5 to 11%, and the relative accuracy was better than $\pm 10\%$ for all runs. Flame-type atomic absorption spectrometry^{9, 10} was used as a comparison technique for the 50- and 20- μg spikes, and the recoveries were complete as indicated by the results of both methods. Four nanograms of beryllium can be quantitatively recovered from a paper filter, and the lowest amount of beryllium per paper filter that can be accurately detected is about two nanograms.

Figure 5 is a plot of absorbance versus μg of beryllium added to the paper filters; all of the digested filters were diluted to 100 ml. The plot is identical, essentially, to a plot of absorbance versus concentration for the aqueous beryllium standards. This indicates again that recovery is complete and that aqueous standards can be used directly. The correlation coefficients for Figures 1 through 5 are all better than 0.997.

Figure 6 is a histogram plot of ng of recovered beryllium versus the number of each determination for filter papers spiked with 0.75 μg of beryllium. Thirty-nine determinations were performed with a

TABLE 1. Effect of 1M Acids on the Absorbance of 5 ng/ml Beryllium (Be) Standards Using the Carbon Cup and 10- μl Samples.

1M Acid	Be Concentration (ng/ml)	Absorbance ^a
HNO_3	5.0	0.031
HCl	5.0	0.029
H_2SO_4	5.0	0.027
H_3PO_4	5.0	0.025 ^{b, c}
HClO_4	5.0	0.014 ^{b, c}
HF	5.0	0.009 ^b
HF, HClO_4	5.0	0.08 ^{b, c}

^aDifferences in absorbances were caused partially by different splattering characteristics of the solutions during the ashing step.

^bAtomic absorbance and nonatomic absorbance peaks occurred during the atomization step.

^cOverlap of the atomic and nonatomic peaks caused the recovery to vary considerably from run to run.

TABLE 2. Effect of 1M Acids on the Recovery of ng/ml Amounts of Beryllium (Be) from Other 1M Acids Using the Carbon Cup and 10- μ l Samples.

Parent 1M Acid	1M Acid	Be Concentration (ng/ml)	Be Recovery Based on 1M Parent (ng/ml)
HNO ₃	—	5.0	5.0 ^a
HNO ₃	H ₂ SO ₄	5.0	4.1 ^a
HNO ₃	HCl	5.0	3.4 ^a
HNO ₃	H ₃ PO ₄	5.0	2.5 ^b
HNO ₃	HClO ₄	5.0	1.9
HNO ₃	HF	5.0	1.6
H ₂ SO ₄	—	10.0	10.0
H ₂ SO ₄	HNO ₃	10.0	10.4 ^a
H ₂ SO ₄	HCl	10.0	10.3
H ₂ SO ₄	H ₃ PO ₄	10.0	8.9 ^b
H ₂ SO ₄	HClO ₄	10.0	6.1 ^b
H ₂ SO ₄	HF	10.0	5.0 ^b

^a Differences in recovery were caused partially by different splattering characteristics during the ashing step.

^b Atomic absorption and nonatomic absorption occurred during the atomization step. The overlap of the atomic and nonatomic peaks caused the recovery to vary considerably from run to run.

TABLE 3. Effect of 0.1M Acids on the Recovery of 7.5 ng/ml Beryllium (Be) Standards from 1M Acid Solutions Using the Carbon Cup and 10- μ l Samples.

Parent 1M Acid	0.1M Acid	Be Concentration (ng/ml)	Be Recovery Based on 1M Parent ^a (ng/ml)
HNO ₃		7.5	7.5
HNO ₃	HCl	7.5	7.1
HNO ₃	H ₂ SO ₄	7.5	6.6
HNO ₃	H ₃ PO ₄	7.5	6.2 ^b
HNO ₃	HClO ₄	7.5	1.5
HNO ₃	HF	7.5	0.7
HNO ₃	HF + HClO ₄	7.5	0.5 ^b
H ₂ SO ₄		7.5	7.5
H ₂ SO ₄	HCl	7.5	7.8
H ₂ SO ₄	HNO ₃	7.5	7.2
H ₂ SO ₄	H ₃ PO ₄	7.5	6.7 ^b
H ₂ SO ₄	HClO ₄	7.5	6.2 ^b
H ₂ SO ₄	HF	7.5	4.5 ^b
H ₂ SO ₄	HNO ₃ + HClO ₄	7.5	5.8 ^b
HCl		7.5	7.5
HCl	HNO ₃	7.5	7.5
HCl	H ₂ SO ₄	7.5	7.8
HCl	H ₃ PO ₄	7.5	5.4 ^b
HCl	HClO ₄	7.5	2.8
HCl	HF	7.5	1.4 ^b

^a Differences in recovery were caused partially by different splattering characteristics during the ashing step.

^b Atomic absorption and nonatomic absorption occurred during the atomization step. The overlap of the atomic and nonatomic peaks caused the recovery to vary considerably from run to run.

TABLE 4. Recovery of Beryllium (Be) from Spiked Paper Air Filter Samples.

Added Be (μ g)	Be Recovered ^a by Nonflame A.A. ^b (μ g)	Number of Determinations	Furnace Type	Be Recovered ^c by Flame A.A. ^b (μ g)
50.0	48 $\pm$ 3	15	Cup	52 $\pm$ 1
20.0	19 $\pm$ 2	10	Cup	20.1 $\pm$ 0.4
0.75	0.72 $\pm$ 0.05	40	Cup	—
0.075	0.069 $\pm$ 0.005	10	Cup	—
0.020	0.018 $\pm$ 0.002	10	Rod	—
0.0040	0.0038 $\pm$ 0.0003	10	Rod	—

^a All samples were made 5% (V/V) in H₂SO₄ before running.

^b A.A. — Atomic Absorption.

^c The samples were taken up in 8N.HNO₃ for flame analysis, then diluted for nonflame analysis. The solutions were made 5% (V/V) in H₂SO₄ for nonflame analysis.

recovery average of $0.72 \mu\text{g}$ and with a standard deviation of $\pm 0.05 \mu\text{g}$. The recovered values ranged from a low of $0.62 \mu\text{g}$ to a high of $0.86 \mu\text{g}$.

Recovery of Beryllium From Spiked Glass Filter Samples

Table 5 summarizes recovery data for glass air filter samples spiked with beryllium. The relative standard deviation varied from 4 to 13%, and the relative accuracy varied from 4 to 20%.

The practical lower limit of detection was approximately $0.1 \mu\text{g}$ per glass filter because of a large nonatomic absorbance peak that interfered with the beryllium atomic absorption peak. Blank glass filters gave a large peak when digested and brought to 10 ml. Attempts were made to eliminate

the nonatomic peak by varying the instrumental ashing conditions, by switching to a ramp atomization, and by using acids other than $0.9\text{M H}_2\text{SO}_4$, but satisfactory conditions were not found. Preliminary results from another Rocky Flats laboratory indicate that the nonatomic peak from glass filters is not a serious problem when using a double beam instrument equipped with a deuterium background corrector.

For glass filter samples containing more than $0.2 \mu\text{g}$ of beryllium and less than $1 \mu\text{g}$ of beryllium, the carbon rod proved superior to the carbon cup. The rod is superior because the atomic and nonatomic peaks occur simultaneously, and the nonatomic peak is smaller with the carbon rod. If the conditions are set so that the nonatomic and atomic absorbance peaks occur simultaneously, then the blank, nonatomic peak can be subtracted from the combined peak.

FIGURE 5. Plot of beryllium added to paper filters versus absorbance. All digested filters were diluted to 100 ml using $0.9\text{M H}_2\text{SO}_4$. The carbon cup was used.

Emission spectroscopy analysis of the glass filters indicate that 1 g of glass filter contains more than 0.1 mg each of aluminum, boron, barium, potassium, sodium, silicon, and zinc, in addition to smaller quantities of silver, calcium, copper, iron, lithium, magnesium, rubidium, antimony, and strontium. The residue from ashed glass filters was also analyzed, and the major constituents found were aluminum and sodium. Barium, potassium, and zinc were present at lesser concentrations, and calcium, iron, magnesium, and strontium were present at much lower levels.

Based on this analysis, aluminum was thought to be the cause of the nonatomic absorbance peak, and experimental results verified this. A 100- $\mu\text{g}/\text{ml}$ Al^{+3} standard in 0.9M H_2SO_4 gives a nonatomic peak equivalent to about 1 ng/ml of beryllium; a 1,000- $\mu\text{g}/\text{ml}$ Al^{+3} standard in 0.9M H_2SO_4 gives

a similar peak equivalent to about 5 ng/ml. Aluminum concentrations greater than 100 $\mu\text{g}/\text{ml}$ in the final assay solution cannot be tolerated if the beryllium level is less than about 5 ng/ml. Other investigators have also experienced difficulties with trace metal analysis when using glass filters.¹⁶

Stability of Standards and Sample Solutions

Results from several standards and sample solutions indicate that solutions containing 7 ng of beryllium per ml and that are 1M in HNO_3 are stable for at least a week. Stock solutions containing 1 μg of beryllium or more per ml and that are 1% in HNO_3 are stable for periods longer than a week. Standards and digested filter samples that are 0.9M in H_2SO_4 show no apparent loss of beryllium after standing for two weeks.

FIGURE 6. Histogram plot of recovered beryllium versus the number of each determination for paper filters spiked with 750 ng of beryllium. The recovered mean was 720 ng and the standard deviation was 50 ng. The carbon cup was used, and all solutions were 0.9M in H_2SO_4 .

Solutions that are 0.9M in H_2SO_4 and that contain 2.0 ng of beryllium per ml, for example, give identical absorbance readings after standing for two weeks. Treatment of the glass containers with hexamethyldisilazane^{8, 11} is not necessary.

SUMMARY

Sulfuric acid tends to mask interferences caused by calcium and acids; consequently, all solutions were made 0.9M (5% V/V) in H_2SO_4 . Larger amounts of H_2SO_4 cause splattering in the cup. Plots of absorbance versus concentration are linear from 0 to at least 4 ng/ml for the carbon rod and 5- μl samples, and from 0 to 15 ng/ml for the carbon cup and 10- μl samples. The sensitivity and detection limits for the carbon rod are 1.0×10^{-12} g and 0.4×10^{-12} g, respectively. The equivalent values for the carbon cup are about five times greater.

Nonatomic peaks occur with H_3PO_4 , HF, and HClO_4 solutions, and solutions containing 0.1M HF or 0.1M HClO_4 or both cause more than a 10% depression in the absorbance signal. These acids must be removed during digestion.

As little as 2.0 ng of beryllium can be recovered quantitatively from spiked paper filters by using aqueous standards. The relative accuracy was better than $\pm 10\%$. The standard deviation varied between 6 and 11% for paper filters spiked with 50 μg to 4 ng of beryllium. Comparison of the results obtained by standard flame atomic absorption analysis and nonflame atomic absorption analysis was good, but the nonflame technique has the advantage that as little as 2.0 ng of beryllium per paper filter can be detected, whereas the direct flame technique is limited to about 2 μg of beryllium per filter.

The beryllium also can be recovered quantitatively from spiked glass filters, but the detection is limited to about 0.1 μg of beryllium per filter on a single beam spectrometer because of an interfering nonatomic peak. The nonatomic peak is probably caused by aluminum. Aluminum concentrations greater than 100 $\mu\text{g}/\text{ml}$ in the final solution cannot be tolerated if the beryllium concentration is less than about 5 ng/ml. The aluminum interference apparently is not a major factor when a double beam instrument equipped with a deuterium background corrector is used.

For paper filters, the method is fast; 50 samples can be completed easily in a working day. The method also is accurate and sensitive. Since urban air in the United States usually contains from 0.1 to 3 ng of beryllium per cubic metre,^{1, 8} the minimum sampling volume will vary from 20 to less than 1 cubic metre.

TABLE 5. Recovery of Beryllium (Be) from Spiked Glass Air Filter Samples.

Added Be (μg)	Be ^a Recovered (μg)	Number of Determinations	Furnace Type ^b
100	108 $\pm$ 4	10	Rod
1.00	1.02 $\pm$ 0.07	10	Rod
0.75	0.71 $\pm$ 0.09	15	Cup ^c
0.25	0.30 $\pm$ 0.03	10	Rod
0.020 ^d	—	10	Rod

^a All samples were made 5% (V/V) in H_2SO_4 before running.

^b Both a beryllium atomic and a nonatomic peak occur. The conditions are such that the atomic and nonatomic peaks occur simultaneously; thus, a blank correction is made easily.

^c The nonatomic peak is larger with the carbon cup and generally occurs before the atomic peak.

^d The nonatomic peak is too large to correct for.

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