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PAPER

Preparation, certification and interlaboratory analysis of workplace air filters spiked with high-fired beryllium oxide†

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Occupational sampling and analysis for multiple elements is generally approached using various approved methods from authoritative government sources such as the National Institute for Occupational Safety and Health (NIOSH), the Occupational Safety and Health Administration (OSHA) and the Environmental Protection Agency (EPA), as well as consensus standards bodies such as ASTM International. The constituents of a sample can exist as unidentified compounds requiring sample preparation to be chosen appropriately, as in the case of beryllium in the form of beryllium oxide (BeO). An interlaboratory study was performed to collect analytical data from volunteer laboratories to examine the effectiveness of methods currently in use for preparation and analysis of samples containing calcined BeO powder. NIST SRM® 1877 high-fired BeO powder (1100 to 1200 °C calcining temperature; count median primary particle diameter 0.12 μm) was used to spike air filter media as a representative form of beryllium particulate matter present in workplace sampling that is known to be resistant to dissolution. The BeO powder standard reference material was gravimetrically prepared in a suspension and deposited onto 37 mm mixed cellulose ester air filters at five different levels between 0.5 μg and 25 μg of Be (as BeO). Sample sets consisting of five BeO-spiked filters (in duplicate) and two blank filters, for a total of twelve unique air filter samples per set, were submitted as blind samples to each of 27 participating laboratories. Participants were instructed to follow their current process for sample preparation and utilize their normal analytical methods for processing samples containing substances of this nature. Laboratories using more than one sample preparation and analysis method were provided with more than one sample set. Results from 34 data sets ultimately received from the 27 volunteer laboratories were subjected to applicable statistical analyses. The observed performance data show that sample preparations using nitric acid alone, or combinations of nitric and hydrochloric acids, are not effective for complete extraction of Be from the SRM 1877 refractory BeO particulate matter spiked on air filters; but that effective recovery can be achieved by using sample preparation procedures utilizing either sulfuric or hydrofluoric acid, or by using

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Environmental impact

This research is significant because it examines the effectiveness of methods currently in use for preparation and analysis of filter samples containing high-fired beryllium oxide powder, which represents a particulate material present in workplace samples that is known to be resistant to dissolution. Regulatory reductions in occupational exposure limits for beryllium and other toxic materials present analytical challenges related to obtaining quantitative recovery and improving capabilities for ultra-trace measurement. Knowledge garnered from this interlaboratory study can assist in understanding and addressing sampling and analytical challenges for other refractory materials.

methodologies involving ammonium bifluoride with heating. Laboratories responsible for quantitative determination of Be in workplace samples that may contain high-fired BeO should use quality assurance schemes that include BeO-spiked sampling media, rather than solely media spiked with soluble Be compounds, and should ensure that methods capable of quantitative digestion of Be from the actual material present are used.

Introduction

Beryllium (Be) has numerous applications in a wide variety of materials owing to its unique properties such as low density along with high melting point, strength, and heat capacity: advantages for which there are no suitable substitutes.[1] Beryllium oxide (BeO; also known as beryllia) is noteworthy in that it is thermally conductive but electrically insulating, attributes that have led to its extensive use in electronic micro-circuitry and myriad commercial, specialized materials.[2] However, occupational exposures to airborne Be can lead to beryllium sensitization and chronic beryllium disease (CBD) in workers who are exposed.[3,4] Some people have inherited genes that make them more likely to become sensitized or develop chronic beryllium disease when exposed.[5] Sensitization is an immune response, not a disease, and does not have any symptoms. Research suggests that sensitization may result from skin contact with beryllium dusts, fumes, and beryllium-containing solutions and suspensions.[5] CBD is a progressively debilitating and potentially fatal lung disease[6] that is suspected to arise from exposures to even minute airborne concentrations of Be.[7] The US Occupational Safety and Health Administration (OSHA) has issued a Hazard Information Bulletin for beryllium exposure.[8] The OSHA permissible exposure limit (PEL) for Be has been under debate, with new information available that suggests the accepted exposure limit of 2 μg m^{-3} of air averaged over an eight hour work shift is inadequate to prevent the occurrence of CBD in exposed workers. The National Institute for Occupational Safety and Health (NIOSH) has issued an Alert on Preventing Sensitization and Disease from Beryllium Exposure.[5] The value of 0.5 μg m^{-3} of air averaged over an eight hour work shift suggested in 1977 by NIOSH as a recommended exposure limit (REL) was intended to reduce the risk of Be-related lung cancer.[9] NIOSH has stated that this REL does not protect workers from risk of developing beryllium sensitization or CBD.[5] Reduced Be occupational exposure limits (OELs) have been promulgated by some organizations in efforts to minimize the possibility of workers becoming sensitized or contracting CBD.[10,11,12] Those OELs represent tenfold to fortyfold reductions of the current OSHA PEL. Challenges in measuring such low levels of airborne Be include aspects of sample preparation and analytical detection,[13,14] entailing a requirement for quantitative recovery along with capabilities for ultra-trace measurement.[15,16]

With the realization that prevention of beryllium sensitization and CBD requires control of Be exposures to very low levels, a heightened awareness has resulted regarding the effectiveness of analytical methods used to quantify Be exposure. Due to concerns about the performance of laboratories analyzing samples of Be in air or settled dust, the Sampling and Analysis Subcommittee (SAS) of the Beryllium Health and Safety Committee (BHSC)[17] undertook a project to conduct an interlaboratory analysis of BeO spiked on air filters that are used in workplace exposure monitoring. The project was designed to gather data on the effectiveness of methods currently in use for sample preparation and analysis of BeO. NIOSH recommends the use of BeO for analytical media and blind spikes if it is suspected that BeO may be present.[18] A well-characterized, high-fired BeO was chosen as the spiking material, representing a known, difficult-to-digest Be material that may exist in typical work sites where this toxic metal can be found. This BeO material, which is refractory, may not completely dissolve when using sample preparation procedures that are commonly employed by industrial hygiene chemistry laboratories.[19] Due to reaction kinetics, Be dissolution is more effective from smaller *versus* larger BeO particles;[19] hence extraction chemistry, duration, and temperature requirements are greater for the sample dissolution step when material is encountered as large primary particles compared to when material is encountered as small particles or as agglomerates of small primary particles.[20] Consequently, this study was carried out to collect data on the efficiency, accuracy, precision and robustness of sample preparation and analytical methods used for air samples of Be-containing aerosol particles. The investigation relied upon the participation of volunteer laboratories having interest and experience in the measurement of metals in occupational hygiene samples.

Experimental

Equipment and instrumentation

Two Sartorius model AC211P and AC210P analytical balances (Edgewood, NY) weighing to 0.1 mg were used to prepare suspension standards of National Institute of Standards and Technology (NIST) standard reference material SRM 1877® high-fired BeO (NIST, Gaithersburg, MD)[21] and monitor the weights during dispensing of the suspension standard on filters. A model P4002 Denver Instruments top-loading balance (Arvada, CO) was used to measure final weight of suspension solutions. An Aquasonic model 50HT ultrasonic bath (VWR, West Chester, PA) was used to mix and homogenize the suspension solutions. A model 776 Dosimat automatic pipettor from Brinkman Metrohm (Westbury, NY) was calibrated and used to deposit a suspension of BeO onto filters. Optima models 7300 DV and 5300 DV Inductively Coupled Plasma - Optical Emission Spectrometers (ICP-OES), each equipped with a Ryton spray chamber, a *gem*-tipped cross-flow nebulizer, and a demountable torch with an alumina injector (Perkin-Elmer, Norwalk, CT), were used to confirm concentrations of BeO suspension standards and BeO filters (detection limit 0.0001 mg L^{-1}). Whatman 40 (Maidstone, England), 47-mm filter papers were used to wipe the inner surfaces of petri dishes that housed BeO-spiked filters to test for potential Be residue.

Preparation of BeO-spiked filters

The NIST SRM 1877 high-fired BeO powder used to prepare the standard suspensions is a well-characterized reference material comprising BeO that was high-fired between 1100 and1200 °C; this material was characterized as agglomerates of individual BeO particles having a count median primary particle diameter of 0.12 μm with a geometric standard deviation of 1.5 and a mass median primary particle diameter of 0.17 μm with a geometric standard deviation of 1.4.[21] The uniformity of the submicrometer size of the BeO primary particles reduces concerns for particle size variations in the preparation and dispensing of highly homogeneous suspension standards. To prepare the BeO suspension standards, the SRM 1877 material was dried at 100 °C in a model 6522 Thermo Economy Oven (Waltham, MA) for one hour to remove incidental moisture and was then placed in a standard laboratory desiccator and allowed to achieve ambient temperature. Using the calibrated analytical balance isolated within a chemical fume hood, a pre-calculated mass of SRM 1877 was placed and weighed on tared transfer paper for suspension preparation. The weighed material was then transferred to a tared, pre-cleaned 250 mL Pyrex® (Corning, Lowell, MA) glass beaker. The transfer paper and sides of the receiving container were rinsed with a small amount of deionized water to ensure complete transfer and capture of the reference material powder. A 10% (volume fraction) proprietary solution matrix (High-Purity Standards, Charleston, SC) was then added to the beaker to form an aqueous BeO suspension. Final dilutions of the suspension to desired concentrations were conducted on the calibrated top-loading balance.

To disperse the reference material within the suspension, the prepared suspension was covered with Parafilm® (VWR, Batavia, IL) and placed in the ultrasonic bath filled with deionized water and subjected to ultrasonic agitation for 30 min. Homogeneity during subsequent pipetting for preparation of spiked filters was maintained by means of mechanical stirring with a polytetrafluoroethylene (PTFE)-coated stir bar. Based on evaluations of the temporal stability of the suspension concentration, the suspension was re-sonicated if the period of use extended to times beyond 30 min. A 776 Dosimat automatic pipette (Thermo Fisher Scientific, Waltham, MA) was used to spike the suspension standard onto 37 mm, 0.8 μm pore size, mixed cellulose ester (MCE) filters (Millipore Corporation, Billerica, MA). Using standard suspensions with Be concentrations of 30.0 $\mu g\ g^{-1}$ and 397.2 $\mu g\ g^{-1}$, the mass dispensed from the suspension standards ranged between 20 and 45 mg per filter, depending on the mass concentration of the standard suspension and the target mass concentration (0.6, 0.9, 1.2, 12, and 18 μg Be per filter) for the five filter loadings produced.

To ensure calibration of the automatic pipettor, the equipment was programmed at the desired setting, and four replicate weights were taken by dispensing aliquots of the suspension standard into 1.5 mL pre-weighed microcentrifuge tubes that were manually assigned unique identification numbers. The sample tubes were re-weighed on the analytical balance and the weights were recorded. All balance calibrations are verified weekly by an in-house method using National Bureau of Standards (NBS; now NIST) weights Inventory No. 20231A (Christian Becker, New York, NY). The balances are calibrated yearly and the calibration weights are checked biennially by a qualified independent metrology company with weights traceable to the primary standards developed by NIST. The volume delivery of the pipette syringe was adjusted when necessary to obtain the target weight. Calibration of the autopipettor was repeatedly checked after every ten filters were spiked and at the end of production using the same process.

A weight summary is provided in Table 1, indicating that the dispensed weights remained consistent with the target weights. Blank filters were prepared similarly by depositing an aliquot of a blank composite solution (BeO-free) of the spiking suspension matrix. The filters were air dried in a clean environment and then treated with a thin polymer protective coating to prevent displacement of particles during handling and transport. Filters were placed between inert spacers and packaged in 47 mm (i.d.) plastic petri dishes. The above procedure was used for each of the five spiked filter mass concentrations. Suspension standards were prepared freshly on each day of use.

Conformity of BeO suspension standard and filters

Concentration confirmation. The concentration of the suspension standard was verified by dispensing aliquots of the standard into small pre-cleaned 50-mL PTFE beakers (Thermo Fisher) prior to use and after every 60 filters were prepared. Each aliquot was treated with 10 mL 1 : 1 nitric acid and two drops of hydrofluoric acid, and then heated with a lid on a hot plate for one hour between 150 and170 °C to achieve dissolution of BeO (for subsequent analysis by ICP-OES). The lid was then removed and the solution was evaporated to approximately 3 mL. The beaker was removed from heat and cooled. The sides of the beaker were rinsed with approximately 5 mL of deionized water. Contents of the beaker were quantitatively transferred to a calibrated Class A 25 mL volumetric flask and diluted to volume.

Homogeneity testing. Aliquots were taken at the beginning, middle, and end of filter production to verify both the concentration and homogeneity of Be in the spiking standard. In addition to periodic sampling of dispensed suspension weights and suspension aliquots, three spiked filters were randomly selected from each batch for digestion and analysis. The digestion method described above for dissolution of suspension standard aliquots was also used for analysis of the spiked filters. Sample concentrations were verified *via* ICP-OES against NIST SRM 3105a (lot #892707) Be standard solution. Table 2 summarizes Be recoveries obtained from the aliquots and filters.

Certification of BeO-spiked filters

Certification was performed for the mass of Be on the spiked filters. The assignment of established masses and their uncertainties for the BeO filters was based on the results from gravimetric preparation, homogeneity testing, and stability studies. Homogeneity testing incorporated the measurements of mass and Be concentration of the aliquots of the suspension standard randomly sampled, as well as measurements of Be concentration of filters during filter preparation. The traceability of these filters was maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties.

Table 1 Gravimetric preparation summary for dispensed BeO standard suspensions

Target Be mass (μg filter^{-1})	Suspension standard [Be] (μg g^{-1})	Target dispensed mass (mg)	Dispensed mean mass (mg)	Std. Dev. (mg)	RSD[a] (%)	No. of samples (*n*)	Blinded Sample ID
0.6	30.0	20	19.9	0.3	1.7	13	Sample 3
0.9	30.0	30	29.8	0.4	1.4	14	Sample 2
1.2	30.0	40	39.8	0.4	1.1	12	Sample 5
12	397.2	30	30.3	0.2	0.7	14	Sample 1
18	397.2	45	45.1	0.4	0.8	13	Sample 4

[a] Relative standard deviation.

Gravimetric measurement – Calculation of results. The amount of Be as BeO (μg Be per filter) was calculated based on gravimetric preparation associated with masses taken and the Be mass fraction in SRM 1877 as indicated in eqn (1):

$$C_{Be} = 10^{6} \cdot \frac{m \cdot f}{W} \cdot M \quad (1)$$

Where

C_{Be} is the Be concentration (μg filter^{-1})

10^{6} is the conversion factor for grams (g) to micrograms (μg)

m is the mass of BeO material (SRM 1877) used to prepare the suspension standard (g)

f is the certified mass fraction of Be in the BeO material (SRM 1877)

W is the total prepared mass of the suspension standard (g)

M is the mass of the BeO suspension standard dispensed on the filter (g)

Uncertainty estimation. Uncertainty components arising from gravimetric preparation, homogeneity measurement, and the stability study were identified during BeO filter preparation and measurements. Combined effects of uncertainty components on the results were evaluated and estimated by Type A and/or Type B evaluation of standard uncertainty, and the combined uncertainty was calculated using the law of propagation of uncertainty in accordance with NIST protocols.[22]

The combined standard uncertainty of the gravimetric preparation involved four major uncertainty components: (a) the

Table 2 Homogeneity testing of randomly dispensed aliquots of BeO suspension standards spiked on filters

	Sample recovery from homogeneity testing[a]								
Suspension standard Be conc. (μg g^{-1})	Target Be mass (μg filter^{-1})	Sampling method	Be mass (μg filter^{-1})	Recovery (%)	Average recovery (%)	Recovered mean mass (μg filter^{-1})	Std. Dev. (μg)	RSD (%)	No. measured (n)
30.0	0.598	Pipetting 1	0.668	112	103	0.615	0.038	6.2	6
		Pipetting 2	0.570	95.4					
		Pipetting 3	0.618	103					
		Filter 1	0.630	105					
		Filter 2	0.633	106					
		Filter 3	0.570	95.4					
	0.895	Pipetting 1	0.843	94.1	95.3	0.853	0.063	7.4	6
		Pipetting 2	0.840	93.9					
		Pipetting 3	0.800	89.4					
		Filter 1	0.923	103					
		Filter 2	0.935	105					
		Filter 3	0.780	87.2					
	1.20	Pipetting 1	1.15	95.4	104	1.25	0.070	5.6	5
		Pipetting 2[b]	0.88	73.5					
		Pipetting 3	1.31	109					
		Filter 1	1.21	101					
		Filter 2	1.24	103					
		Filter 3	1.32	110					
397.2	12.0	Pipetting 1	11.7	97.3	98.7	11.8	0.70	5.9	6
		Pipetting 2	11.6	97.0					
		Pipetting 3	12.6	105					
		Filter 1	12.4	103					
		Filter 2	12.2	102					
		Filter 3	10.6	88.1					
	18.0	Pipetting 1	17.2	96.0	100	18.0	1.0	5.6	6
		Pipetting 2	18.6	104					
		Pipetting 3	17.0	95.2					
		Filter 1	17.0	94.8					
		Filter 2	19.2	107					
		Filter 3	18.8	105					

[a] All analyses by ICP-OES. [b] Results omitted from calculation of recovery statistics owing to the absence of HF during digestion.

balance, (b) the Be mass fraction of the powder, (c) the mass of the suspension standard prepared, and (d) the mass of the standard suspension dispensed as shown in eqn (2):

$$\boldsymbol{u}_{gravimetric} = C_{Be}\sqrt{\boldsymbol{u}_m^2 + \boldsymbol{u}_f^2 + \boldsymbol{u}_W^2 + \boldsymbol{u}_M^2} \tag{2}$$

Where

$u_{gravimetric}$ is the combined standard uncertainty of the gravimetric preparation

u_m is the uncertainty associated with the mass of BeO material taken, related to the uncertainty of the balance

u_f is the uncertainty associated with the mass fraction of Be in the SRM 1877 BeO material

u_W is the uncertainty associated with the total mass of the suspension standard prepared, related to the uncertainty of the balance

u_M is the uncertainty associated with the mass of BeO suspension standard dispensed on the filter, related to uncertainty of the balance and repeatability of mass measurements

The combined standard uncertainty for the gravimetric preparation was then combined with the uncertainty components determined from the homogeneity measurements and from the suspension stability study to obtain the combined standard uncertainty for preparation of the BeO-spiked filters as shown in eqn (3):

$$\boldsymbol{u}_c = C_a \cdot \sqrt{\boldsymbol{u}_{gravimeric}^2 + \boldsymbol{u}_{homo}^2 + \boldsymbol{u}_{stab}^2} \tag{3}$$

Where

u_c is the combined standard uncertainty for preparation of the spiked BeO filters

C_a is the assigned value of Be (μg filter^{-1}) from repeatability of measurements by analytical techniques and methods (Note: the assigned C_a values were taken from the C_{Be} calculations)

$u_{gravimetric}$ is the uncertainty from gravimetric preparation (eqn (2))

u_{homo} is the uncertainty from homogeneity measurement related to dispensed masses and filter homogeneity testing

u_{stab} is the uncertainty from the stability study

The combined standard uncertainty (u_c) is intended to represent one standard deviation, and the expanded uncertainty was calculated as $U = ku_c$, with a coverage factor $k \sim 2$ commonly used for an estimated 95% confidence interval.

Mass value and estimated uncertainty. BeO levels, as Be filter certified values = C_{Be} (μg filter^{-1}) ± U (μg filter^{-1}) for a 95% confidence interval, are given in Table 3. The assignments of Be (μg filter^{-1}) mass values were calculated using eqn (1) by gravimetric measurement, associated with the certified value[16] of Be mass fraction. The uncertainties in Table 3 were estimated using eqn (2) and eqn (3), respectively. During filter production and validation, the uncertainty from gravimetric preparation was estimated to be about 1% to 2% of the mass values. The uncertainty arising from all uncertainty components was estimated to be about 5% to 10% of the mass values. These uncertainty components were due to bias or errors from the filter and testing sample preparations, digestion and measurement methods, homogeneity and stability effects.

Suspension stability study. Long-term stability for BeO suspensions in the range of 10 to 300 μg g^{-1} Be was evaluated over a one-year span. Re-suspension and sampling of the standards demonstrated consistent, reproducible results from the time of initial preparation to the date of evaluation over one year later. In addition, stabilities of the suspension standards used in preparation of the BeO-spiked filters were confirmed during the preparation time of the filters by periodic weighing and analysis of aliquots of the suspension standards throughout production of the spiked filters and at the conclusion of filter production.

Shipping stability study for BeO-spiked filters. Additional stability testing was performed on the filters to guarantee that Be material was not lost due to possible dropping or rough handling during shipment to the participating laboratories. Two sets of representative samples of the BeO-spiked filter media at four different filter concentrations within the defined range of this study were packaged for shipment and drop tested *via* International Air Transport Association (IATA) regulations.[23] This test involves a series of drops from a height of four feet above the ground and requires multiple surfaces of the box, including one corner, to impact the ground. As an additional precautionary measure, requirements of the regulations were then exceeded by subjecting half of the drop-tested packages to mechanical vibration on a sieve shaker to simulate additional rough handling that may occur to the package during transit. To verify that no significant Be loss had occurred, each filter was digested and analyzed separately from the corresponding spacers surrounding it; also, a wipe (Whatman 40) of the cassette that housed the filter was digested and analyzed. Each component was digested according to the nitric acid and hydrofluoric acid treatment and heating method described above in the section on confirmation of the suspension standard concentrations, and then diluted to 25 mL for ICP-OES analysis.

Table 4 shows the observed values of Be recovered from the digested filters, spacers, and wipes as analyzed by ICP-OES (with extract aliquots diluted by a factor of 25). No significant traces of Be were found to have transferred to the surrounding packaging during either drop testing or drop testing with additional rough handling, and the data indicate that the BeO-spiked material remained adhered to the filter. All data for the spacers and wipes analyzed following drop testing alone indicated Be levels at or below the detection limit of ICP-OES (0.1 μg Be L^{-1}).

Data collection and analysis from interlaboratory study

The participating laboratories were requested to analyze sets of samples that consisted of five BeO-spiked filters (in duplicate) and two blank filters, for a total of 12 unique air filter samples per laboratory. The Be levels of the spiked filters were chosen to be in the range of levels used in the American Industrial Hygiene Association Beryllium Proficiency Analytical Testing (AIHA BePAT) program,[24] and were between 0.5 μg and 25 μg of Be as BeO. Certified values for the BeO-spiked filters (High-Purity Standards) that were distributed to the participating laboratories are presented in Table 3. The certified spike levels of these samples were unknown to all participants during the study and were revealed only to the program coordinators.

A total of 27 laboratories from the U.S., U.K., France and Canada volunteered to participate in this study (Table 5). Several participants requested more than one sample set so as to check

Table 3 BeO-spiked filter mass values and estimated uncertainties

	Gravimetric Preparation			All Uncertainty Components	
Target Be mass (μg filter^{-1})	Be mass from preparation (μg filter^{-1})	Estimated uncertainty U (%)	Certified Be value (μg filter^{-1})	Estimated uncertainty U (%)	Be value (μg filter^{-1})
0.6	0.5978	2.0	0.598 ± 0.012	10	0.60 ± 0.06
0.9	0.8951	2.0	0.895 ± 0.018	7.7	0.90 ± 0.07
1.2	1.196	1.7	1.20 ± 0.020	6.7	1.20 ± 0.08
12	12.04	0.83	12.0 ± 0.10	5.0	12.0 ± 0.6
18	17.91	1.1	17.9 ± 0.20	5.0	17.9 ± 0.9

the performance of more than one sample preparation and analysis method for this sample matrix. Accordingly, a total of 36 sets of samples were distributed to the 27 participants. All volunteer laboratories were requested to follow their normal sample preparation and analysis methods currently in use for samples of this type (*i.e.*, workplace air filter samples). A data report form was submitted to each participating laboratory in an effort to collect information on each participant's preparation and analysis method details and to allow for data comparison and categorization. Each sample set contained documentation on safe handling and data reporting requirements, along with a Material Safety Data Sheet.[25] Ultimately, data sets were submitted by the 27 participants for 34 of the 36 sample sets that were conveyed. To preserve the identity of individual laboratories, only the unique identifier code for each sample set is listed in the table.

Table 4 Shipping stability test results for BeO-spiked filters[a, c]

	After drop testing alone				After drop testing and rough handling			
Be mass (μg filter^{-1})	Analyzed item	ICP-OES value (μg l^{-1})	Recovery (%)	Average recovery (%)	Analyzed item	ICP-OES value (μg l^{-1})	Recovery (%)	Average recovery (%)
0.5[b]	Filter 1	17.9	89.5	90.8	Spacer 1 top	0.7	3.5	89.3
					Filter 1	17.8	89.0	
					Spacer 1 bottom	0.4	2.0	
					Wipe 1	0.4	2.0	
	Filter 2	18.4	92.0		Spacer 2 top	N.D.	0.0	
					Filter 2	17.9	89.5	
					Spacer 2 bottom	0.2	1.0	
					Wipe 2	0.1	0.5	
1.2	Filter1	48.1	100	101	Spacer 1 top	0.2	0.4	97.1
					Filter 1	46.2	96.3	
					Spacer 1 bottom	0.2	0.4	
					Wipe 1	0.3	0.6	
	Filter 2	49.0	102		Spacer 2 top	0.2	0.4	
					Filter 2	47.0	97.9	
					Spacer 2 bottom	0.2	0.4	
					Wipe 2	0.3	0.6	
2	Filter 1	77.4	96.8	98.4	Spacer 1 top	0.2	0.3	98.5
					Filter 1	78.4	98.0	
					Spacer 1 bottom	N.D.	0.0	
					Wipe 1	0.3	0.4	
	Filter 2	80.1	100		Spacer 2 top	0.3	0.4	
					Filter 2	79.2	99.0	
					Spacer 2 bottom	N.D.	0.0	
					Wipe 2	0.1	0.1	
18	Filter 1	720.4	100	102	Spacer 1 top	0.8	0.1	105
					Filter 1	744.8	103	
					Spacer 1 bottom	0.3	0.0	
					Wipe 1	2.3	0.3	
	Filter 2	750.8	104		Spacer 2 top	0.4	0.1	
					Filter 2	767.7	107	
					Spacer 2 bottom	0.1	0.0	
					Wipe 2	1.2	0.2	

[a] Note: After drop testing alone, filters, spacers, and wipes were digested separately and then analyzed, but beryllium values for the spacers and wipes were below the ICP-OES detection limit (0.1 μg L^{-1}) and only the filter data are shown here. All values are shown for analysis results after drop testing and rough handling. [b] This lot of filters was produced 4 years prior to the full study and was expected to be more fragile due to long-term instability of the filter material. They were in the concentration range of interest and were tested for long-term stability, but filters from this lot were not distributed for analysis in the interlaboratory study. [c] N.D. = None detected.

Data submitted by the volunteer laboratory participants were subjected to statistical assessments in accordance with established procedures for interlaboratory analysis.[26] Results were pooled into groups depending upon the reagents used for sample preparation. All submitted data were compared directly to the certified Be spike levels, and recovery was determined for each sample in the study.

Results and discussion

Reported results for individual sample sets from each participating laboratory are presented in Table 6. In almost all instances the paired results were close to one another in magnitude; although there were a few exceptions (notably two identified outliers from one laboratory), internal laboratory repeatability was generally good. For the majority of cases the reported Be values were near the certified value; however, nine laboratories out of 34 reported results that appear to be significantly low (mean recovery <85%, Table 6; more on this observation below). Results reported for media blanks were largely negligible. Generally, the spiking levels on the filters did not affect the reported Be recoveries. That is, recoveries obtained for low-level spikes (*i.e.*, ≈0.6 μg per filter) did not differ significantly from recoveries obtained from intermediate and high level spikes (*i.e.*, up to ≈18 μg per filter).

Table 7 summarizes the various analytical methods, detection methods, and dissolution reagents used for each of the 34 sample sets. Mean recoveries and standard deviations for all sample results are also included. Data groupings by dissolution reagent combination in Table 7 helped to most concisely explain differences in the results obtained from the various analytical methods employed by the participant laboratories.

Scrutiny of the data shown in Table 6 had suggested four dissolution reagent categories, which were attributed to the principal reagents employed in the sample preparation portions of the various methods used.[27] In Table 7, results from the use of sulfuric acid, or combinations of reagents that included sulfuric acid, are grouped in the Sulfuric Acid group. Results from use of ammonium bifluoride (ABF: NH_4HF_2), or combinations using NH_4HF_2, are grouped in the Ammonium Bifluoride (ABF) group. Results from the use of hydrofluoric acid, or combinations using HF, are grouped in the Hydrofluoric Acid group. The Nitric Acid group combined results from all the laboratories that had used either nitric acid alone, or had used some combination of nitric acid and other reagents not already grouped above. The Nitric Acid group comprises nitric acid alone or a combination of nitric acid and hydrochloric acid, which are acids common to dissolution procedures described by the NIOSH, Environmental Protection Agency (EPA), and similar environmental sample preparation methods. Note that although NIOSH Method 7300[28] is formally described to use a combination of nitric acid and perchloric acid at a ratio of 4 : 1, Table 7 shows that none of the methods cited in the current study as "NIOSH 7300" or "NIOSH 7300 modified" used that formulation. This emphasizes the importance of verifying and documenting the details and performance of any modifications to standard methods. At any rate, mixtures of nitric and perchloric acids are reportedly ineffective for extracting Be from calcined BeO.[33]

A graphical presentation of individual sample set recoveries in each of the four reagent groupings is shown in Fig. 1. The high, low, and mean recoveries are plotted against 100% recovery,

Table 5 List of the 27 laboratories that participated in the interlaboratory study

Name of Laboratory	Location
ALS DataChem	Salt Lake City, UT, USA
B&W Pantex	Amarillo, TX, USA
Beryllliant, Inc.	Tucson, AZ, USA
Bureau Veritas North America (BVNA)	Novi, MI, USA
Comprehensive Industrial Hygiene Laboratory (CIHL), Navy and Marine Corps Public Health Center	San Diego, CA, USA
Fluor Hanford	Richland, WA, USA
Forensic Analytical Laboratories	Hayward, CA, USA
Galson Laboratories	East Syracuse, NY, USA
Health and Safety Laboratory (HSL)	Buxton, England, U.K.
Health Physics Analytical Laboratory	Abingdon, England, U.K.
Institut National de Recherche et de Sécurité (INRS)	Vandœuvre-les-Nancy, France
Institut de Recherche Robert Sauvé et en Sécurité du Travail (IRSST)	Montréal, Québec, Canada
Los Alamos National Laboratory (LANL)	Los Alamos, NM, USA
Lawrence Livermore National Laboratory (LLNL)	Livermore, CA, USA
LLNL, Environmental Monitoring Radiochemistry Laboratory (EMRL)	Livermore, CA, USA
Navy & Marine Corps Public Health Center Laboratory	Norfolk, VA, USA
National Security Technologies	Mercury, NV, USA
National Institute for Occupational Safety and Health (NIOSH)	Cincinnati, OH, USA
Oak Ridge National Laboratory (ORNL)	Oak Ridge, TN, USA
Occupational Safety and Health Administration (OSHA)	Sandy, UT, USA
Savannah River Nuclear Solutions (non-rad IH lab)	Aiken, SC, USA
Savannah River Nuclear Solutions (rad IH lab)	Aiken, SC, USA
Savannah River National Laboratory (SRNL)	Aiken, SC, USA
US Air Force School of Aerospace Medicine (USAFSAM)	San Antonio, TX, USA
United States Enrichment Corporation (USEC)	Piketon, OH, USA
Wisconsin Occupational Health Laboratory (WOHL)	Madison, WI, USA
Y-12 National Security Laboratory	Oak Ridge, TN, USA

Table 6 Laboratory-reported results (μg Be filter^{-1}) for analyses of duplicate BeO-spiked filters

	Be mass per filter					
Sample Set ID No.	0.598 μg	0.895 μg	1.20 μg	12.0 μg	17.9 μg	Blank filter media
1001	0.598; 0.607	0.872; 0.868	1.09; 1.18	12.0; 11.5	17.6; 17.1	0; 0
1002	0.405; 0.410	0.579; 0.525	0.707; 0.768	7.15; 7.60	12.5; 11.4	< 0.15; < 0.15
1003	0.588; 1.65[a]	2.21[a]; 0.954	1.16; 1.19	12.9; 11.6	18.0; 17.9	< 0.005; < 0.005
1004	0.613; 0.607	0.921; 0.908	1.18; 1.18	11.9; 12.0	17.1; 17.8	NR[b]
1005	0.552; 0.566	0.834; 0.833	1.11; 1.07	11.1; 10.8	16.8; 17.2	NR
1006	0.652; 0.632	0.988; 0.937	1.28; 1.27	12.0; 11.9	18.3; 18.6	NR
1007	0.659; 0.576	0.839; 0.817	1.15; 1.14	11.5; 11.3	16.9; 17.1	< 0.02; < 0.02
1008	0.573; 0.581	0.920; 0.973	1.18; 1.24	12.9; 12.0	18.6; 19.4	< 0.01; < 0.01
1009	0.597; 0.617	0.903; 0.910	1.23; 1.20	12.0; 12.1	17.8; 17.7	< 0.01; < 0.01
1010	0.641; 0.628	0.933; 0.930	1.25; 1.28	12.9; 12.4	18.6; 19.4	< 0.1; < 0.1
1011	0.603; 0.565	0.880; 0.897	1.25; 1.17	11.9; 12.0	17.7; 17.8	0.0051; 0.0027
1012	0.590; 0.588	0.873; 0.871	1.18; 1.18	11.8; 11.7	17.0; 17.2	< 0.03; < 0.03
1013	0.620; 0.590	0.870; 0.880	1.20; 1.10	12.3; 11.3	17.4; 17.8	< 0.03; < 0.03
1014	0.469; 0.470	0.694; 0.645	0.947; 0.932	7.49; 5.67	11.1; 12.1	< 0.025; < 0.025
1015	0.531; 0.528	0.726; 0.823	1.13; 1.11	10.5; 10.5	14.5; 13.8	< 0.05; < 0.05
1016	0.627; 0.651	0.924; 0.930	1.21; 1.25	13.3; 12.9	17.9; 18.6	< 0.05; < 0.05
1017	0.598; 0.601	0.951; 0.883	1.28; 1.27	12.4; 12.2	18.5; 15.7	< 0.05; < 0.05
1018	0.215; 0.289	0.274; 0.274	0.378; 0.493	5.99; 4.93	8.81; 5.10	< 0.020; < 0.020
1019	0.491; 0.530	0.543; 0.589	0.821; 0.993	10.6; 9.87	14.5; 15.4	NR
1020	0.583; 0.494	0.739; 0.785	1.11; 1.10	10.4; 11.6	15.0; 14.8	< 0.0005; < 0.0005
1021	0.380; 0.388	0.633; 0.646	0.878; 0.655	7.89; 8.05	12.4; 11.8	NR
1022	0.364; 0.417	0.447; 0.400	0.726; 0.799	7.42; 8.42	12.3; 11.0	NR
1023	0.555; 0.559	0.836; 0.835	1.14; 1.20	12.4; 11.9	16.0; 17.2	NR
1024	0.541; 0.540	0.813; 0.800	1.19; 1.19	11.7; 11.6	17.4; 16.9	0.0002; 0.0001
1025	0.560; 0.620	0.930; 0.870	1.19; 1.26	12.1; 11.9	17.0; 16.9	0; 0
1026	0.638; 0.615	0.943; 0.898	1.19; 1.26	12.1; 12.9	17.8; 17.2	< 0.01; < 0.01
1027	0.579; 0.565	0.877; 0.819	1.16; 1.15	11.5; 12.1	16.1; 16.4	< 0.01; < 0.01
1028	0.608; 0.583	0.896; 0.880	1.20; 1.17	11.7; 12.3	17.0; 17.3	0; 0
1029	0.600; 0.610	0.870; 0.870	1.15; 1.18	11.9; 11.9	17.1; 17.0	< 0.02; < 0.02
1030	0.537; 0.499	0.772; 0.765	0.864; 0.889	10.4; 9.99	14.4; 14.0	< 0.005; < 0.005
1032	0.493; 0.471	0.611; 0.609	0.968; 0.927	8.59; 9.45	14.7; 12.0	0.0051; 0.0070
1033	0.523; 0.579	0.796; 0.793	1.04; 1.05	10.9; 10.7	15.3; 15.6	0.0088; 0.0072
1035	0.372; 0.374	0.597; 0.596	0.828; 0.758	6.87; 7.50	11.3; 10.8	0.0041; 0.0033
1036	0.532; 0.526	0.789; 0.828	1.03; 1.03	10.8; 11.1	15.9; 15.7	< 0.05; < 0.05

[a] Indicates outlier. [b] NR = not reported.

along with upper and lower acceptance limits (±25%) Fig. 1 illustrates the significance of the reagent effectiveness for recovery as a function of the data grouping. None of the laboratories that used either sulfuric or hydrofluoric acid in their sample preparation methods yielded results outside the acceptance limits. Only one of the eight laboratories that used ammonium bifluoride in their preparation methods failed to recover within the 75%–125% limits; this failure was found to be due to an inadvertent lack of sample heating during extraction. However, only one of the nine laboratories employing nitric acid alone (without sulfuric acid, hydrofluoric acid or ammonium bifluoride) was able to successfully recover Be within the acceptance limits. These results demonstrate conclusively that heat-assisted sample preparation with sulfuric acid, hydrofluoric acid or ammonium bifluoride is required for the attainment of acceptable recoveries of Be from refractory BeO.

Conclusions

Preparation and certification of BeO-spiked filters demonstrated uniformity of the samples that were distributed to the volunteer laboratories. Confirmation of the stability of on-filter Be composition at the time of receipt and following handling by the laboratories offers further validation of the uniformity of the distributed samples. Observed differences in Be recoveries are attributed to differences in sample preparation methods used by the laboratories. Based on this data analysis, the interlaboratory data also show conclusively that effective recovery of Be from this refractory BeO particulate matter spiked on air filters can be achieved using sample preparation procedures utilizing either sulfuric or hydrofluoric acid. Methodologies utilizing ammonium bifluoride with heating are also highly effective in recovery of Be from this highly insoluble material. Several standard methods have been promulgated that utilize these reagents.[11,14,27,34–39] Based on the data analysis of this work, the interlaboratory data also show conclusively that sample preparation using nitric acid alone, or combinations of nitric and hydrochloric acids, is not effective for complete extraction of Be from high-fired BeO. It is noted that the use of HNO_3 alone or in combination with HCl or $HClO_4$ may be suitable for dissolving Be from some matrices, but the results herein indicate that HNO_3 should not be the sole or primary acid employed when the sample is expected to contain refractory BeO. If it is suspected that BeO may be present, then it is recommended to use BeO for media and blind spikes. In addition, it should be

Table 7 Beryllium recoveries from BeO-spiked filters with results grouped according to analytical method used by the participating laboratories

Lab ID no.	Analytical method cited	Detection[c]	Dissolution reagents used[b] H_2SO_4	 NH_4HF_2	 HF	 HNO_3	 HCl	 $HClO_4$	Mean recovery (%) ± std dev (n = 10)
1007	Microwave digestion	ICP-OES	X						96.0 ± 5.12
1011	NIOSH 7300[28]	ICP-OES	X						98.6 ± 2.64
1005	H_2SO_4 + HNO_3	ICP-OES	X			X			92.5 ± 1.86
1009	Microwave digestion	ICP-OES	X			X			100.4 ± 1.39
1013	Acid digestion	GFAAS	X			X			98.0 ± 3.58
1020	Not specified	ICP-MS	X			X			88.2 ± 5.93
1004	OSHA ID-125G[29]	ICP-OES	X			X	X		99.9 ± 1.65
1036	Not specified	ICP-OES	X			X	X		88.6 ± 2.30
1012	NIOSH 7300 modified	ICP-OES	X			X		X	97.1 ± 1.34
1023	NIOSH 7102[28]	ICP-OES	X				X		95.2 ± 4.24
1029	NIOSH 7300/OSHA ID-125G	ICP-OES	X				X		97.7 ± 2.34
1001	NIOSH 7704[28]/ASTM D7202[30]	Fluorescence		X					97.1 ± 3.02
1003	NIOSH 7704[28]	Fluorescence		X					100.1 ± 3.76[a]
1010	ASTM D7202 modified	Fluorescence		X					105.1 ± 1.68
1017	NIOSH 7704	Fluorescence		X					101.1 ± 5.62
1022	NIOSH 7704	Fluorescence		X[d]					61.3 ± 8.48
1025	ASTM D7202	Fluorescence		X					98.9 ± 4.23
1028	NIOSH 9110[28]	Fluorescence		X					98.5 ± 2.41
1016	NIOSH 7300 modified	ICP-MS		X		X			104.6 ± 3.49
1008	NIOSH 7300 modified	ICP-MS			X				101.9 ± 4.13
1033	EPA 3052[31]	ICP-MS			X	X			88.6 ± 3.28
1006	HCl + HNO_3 + HF	ICP-OES			X	X	X		104.4 ± 3.43
1024	Not specified	ICP-MS			X	X	X	X	94.3 ± 4.13
1026	NIOSH 7300 modified	ICP-OES			X	X	X	X	102.0 ± 3.90
1027	NIOSH 7300 modified	ICP-OES			X	X	X	X	95.0 ± 3.49
1015	NIOSH 7300 modified	ICP-MS				X			86.7 ± 5.70
1018	NIOSH 7300 modified	ICP-MS				X			38.6 ± 8.40
1035	NIOSH 7300 modified	ICP-OES				X			63.1 ± 3.36
1002	NIOSH 7300 modified/OSHA 125G modified	ICP-OES				X	X		63.7 ± 3.89
1014	EPA 3051[31]	ICP-OES				X	X		70.1 ± 10.4
1019	OSHA 206[29]	ICP-OES				X	X		78.4 ± 9.96
1021	NIOSH 7303[28]	ICP-OES				X	X		66.5 ± 5.27
1030	NIOSH 7301[28] modified	ICP-MS				X	X		81.7 ± 5.62
1032	NIOSH 7303	ICP-OES				X	X		75.3 ± 6.26

[a] Two outliers omitted (Grubbs test).[32] [b] Heating normally employed during sample dissolution. [c] ICP-OES: Inductively coupled plasma – optical emission spectrometry; GFAAS: Graphite furnace atomic absorption spectrometry; Fluorescence: Optical molecular fluorescence; ICP-MS: Inductively coupled plasma – mass spectrometry. [d] Samples unheated during preparation.

borne in mind that published studies have demonstrated that dissolution of Be from the smallest BeO particles occurs significantly more quickly than from the largest particles.[19] Therefore, it is also recommended that laboratories analyzing samples containing BeO particles larger in diameter than the primary particle diameter of 0.12 μm found in SRM 1877 should ensure they are achieving quantitative digestion of the actual material present. Several studies have been published related to determining the necessary conditions for quantitative recovery of Be from BeO.[14,15,27]

Results from this study demonstrate that standardized methods are available that are effective for quantitatively dissolving refractory BeO from representative air sampling media spiked with BeO SRM 1877. Communication of this information on the efficiency of the methods employed by the volunteer laboratories is one of the primary goals of the interlaboratory study. Additionally, it is recommended that Be proficiency testing schemes frequently utilize BeO-spiked sampling media, rather than solely media spiked with soluble Be compounds. Data obtained from such analyses should provide assurance that suitable methods are used by laboratories for quantitatively determining Be in workplace samples that are suspected to contain high-fired BeO.

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Note added after first publication

This article replaces the version published on 25th October 2011, which contained errors in the reference numbering.

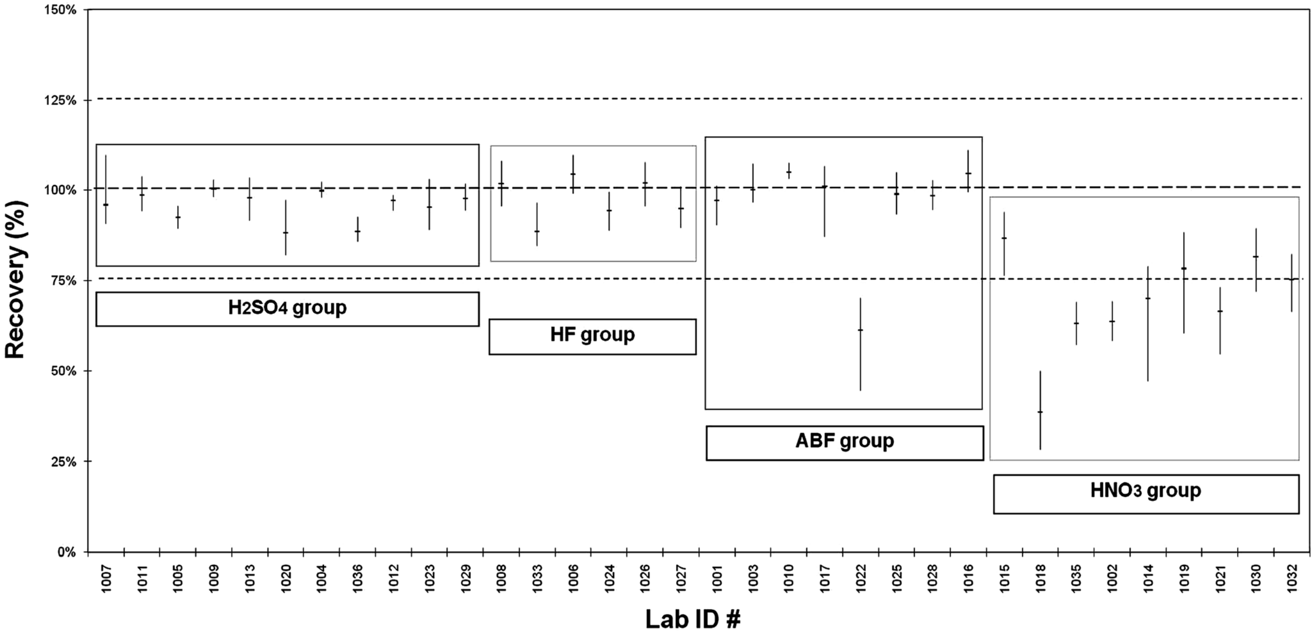

Fig. 1 Beryllium recoveries from BeO-spiked filters, with laboratories grouped according to principal dissolution reagents used. Vertical lines represent ranges of high and low results reported, with the mean values indicated by short horizontal lines. (The low outlier in the ABF [NH_4HF_2] group was attributed to an inadvertent failure to heat the samples during extraction.)

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