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Standard Methods for Beryllium Sampling and Analysis: Availabilities and Needs³

ABSTRACT: Conformity in methods for sampling and analysis of beryllium in workplace air and on surfaces is desired, but inconsistencies in sampling and analytical practices often occur among industrial hygienists and laboratory personnel. In an effort to address these issues, this paper gives an overview of standardized methods for sampling and analysis of beryllium in the workplace. A number of published methods is currently available to the industrial hygiene and analytical community, but shortfalls in the use of standardized practices require attention. Also, questions remain concerning the performance of some of the sampling and analytical methodologies that have been promulgated. We attempt to identify needs for new or improved standard sampling protocols, sample preparation techniques, analytical methods, and reference materials. Where applicable, performance data are summarized for standardized methods that are either published or are under development. These include not only ASTM and ISO international standards, but also methods published by government agencies in the USA and abroad. Significant gaps in standard methods and requirements for reference materials remain. For example, consistent practices are lacking for: (a) surface sampling of beryllium in dust; (b) extraction of beryllium from surface dust samples prior to instrumental analysis; and (c) reference materials containing beryllium oxide (especially high-fired BeO). These and other gaps will be identified and shortcomings addressed. An ultimate goal is to provide standard methods which will ensure comparability of data obtained from different sites around the globe.

KEYWORDS: aerosols, analysis, beryllium, reference materials, sample preparation, sampling, standards, surfaces, workplace

Introduction

The health of workers in a number of industries and activities is at risk through occupational exposure to beryllium. In order to estimate workers' exposures to beryllium, occupational contact with airborne and dermal sources of beryllium is typically monitored by sampling and analyzing workplace atmospheres and surfaces, respectively. Workplace air monitoring is carried out because in occupational settings inhalation is ordinarily the most likely route of entry of hazardous substances, such as beryllium, into the body. Dermal contact is another important potential route of occupational exposure and possible sensitization to beryllium. Thus, procedures for measuring surface contamination of beryllium in workplaces are also required.

Conformity in methods for sampling and analysis of beryllium in workplace air and on surfaces is desired, but inconsistencies in sampling and analytical practices often occur among

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industrial hygienists and laboratory personnel. If sampling and analytical methods are not standardized, analytical results from different investigators, locations, or points in time may not be comparable. Variations in sampling practices are of special concern, since the greatest contribution to measurement uncertainty in the overall analysis is ordinarily associated with sampling. Efforts to minimize measurement uncertainty through method standardization have been realized for many workplace toxins and, as a consequence, standard methods for numerous hazardous substances in workplace atmospheres have been promulgated [1]. However, gaps remain in a number of areas where standard techniques are as yet unavailable. For example, surface sampling techniques have not been adequately standardized or, where such standards exist, they have not been put to use in the relevant areas of application. Also, reference materials for beryllium are few and limited. In an effort to address these requirements for new standards, we have endeavored to identify and suggest correction of situations where standard techniques are needed.

Another matter of concern is where existing standards may not meet desired performance criteria or may not be applicable to a given area of application. Questions concerning method performance might only be answered following additional research, but this requires resources (human and capital) that are often unavailable. So in the absence of research results, standards are often produced by consensus with the best intentions in mind. Yet shortcomings can appear when problems in the application of sub-par standards are realized and identified. Thus, in consideration of possible shortcomings of existing standards, we have made an effort to identify problematic standards with a view to improving them or, where necessary, producing new standards that will hopefully be fit for purpose.

In this paper, standards for sampling, sample preparation, analysis, and reference materials for the determination of beryllium in workplace environments are at issue. For each instance we have made an effort to identify existing standards and to provide performance data, where available. In cases where available standards are found to be lacking, the shortcomings have been pointed out, with an ultimate intention of filling these gaps. It is intended that this article will provide potential users of standards relating to sampling and analysis of beryllium in occupational settings with a useful body of information which will enable them to solve their industrial hygiene and analytical chemistry problems.

Sampling

Collection of Workplace Air Samples

Sampling of beryllium and other metals in workplace atmospheres using filter samplers has been well standardized. In the United States, governmental methods have been published by the National Institute for Occupational Safety and Health (NIOSH) [2] and the Occupational Safety and Health Administration (OSHA) [3]. Workplace air sampling entails personal monitoring using portable, battery-powered sampling pumps that pull air at desired flow rates through the samplers, which are placed within the workers' personal breathing zones. Area sampling is also possible, but workers' exposures are best estimated by personal sampling [4]. The NIOSH and OSHA methods describe "total" air samplers consisting of closed-faced sampling cassettes housing mixed-cellulose ester (MCE) membrane filters. These methods have been widely used worldwide for both regulatory and non-regulatory (e.g., research and hazard assessment) purposes.

International voluntary consensus standards describing workplace air monitoring for metals (including beryllium) have been published recently, which are based, at least in part, on the NIOSH and OSHA methods. Procedures promulgated by the International Organization for Standardization (ISO) [5] and ASTM International [6] describe sampling of the inhalable fraction of atmospheric aerosols using filter samplers and personal sampling pumps. Performance-based options in these consensus standards also allow for sampling of the respirable aerosol fraction, the use of various sampler designs, and the use of fibrous or other membranous filters (besides MCE), depending on the intended application. Some of the technical options for sampling of workplace aerosols that are described in the ISO and ASTM International standards, which differ significantly from the NIOSH and OSHA procedures, have appeared in governmental methods published in France [7], Germany [8], and the United Kingdom [9].

Table 1 summarizes several governmental and two new consensus standard sampling methods for beryllium in workplace atmospheres. Collection efficiencies of 99.5 % or better are afforded by use of these air sampling methods. Generally, the methods listed in Table 1 recommend full 8-h time-weighted average (TWA) personal sampling, but shorter sampling periods can also be employed for certain applications, such as task-based workplace exposure monitoring [10]. The sampling procedures listed in Table 1 allow for subsequent determination of trace quantities of collected airborne beryllium.

TABLE 1—*Governmental and consensus standard procedures for sampling beryllium aerosols in workplace atmospheres.*

Method(s)	Aerosol Fraction(s) Sampled	Filter Type(s) Used
NIOSH 7102 & 7300	“Total”	Membrane
OSHA ID-125G & ID-206	“Total”	Membrane
HSE 29/2 (UK)	Inhalable	Membrane
BIA 6015 (Germany)	Inhalable	Membrane
INRS Fiche 003 (France)	Inhalable	Quartz Fiber
ISO 15202-1	Inhalable or Respirable	Membrane or Fibrous
ASTM D7035	“Total,” Inhalable or Respirable	Membrane or Fibrous

Collection of Surface Samples

Workers can become sensitized to beryllium through dermal contact with beryllium-containing dust, and this can ultimately lead to chronic beryllium disease [11]. In order to assess contamination and prevent dermal contact with beryllium-contaminated surfaces, methods are desired for collection of surface dust samples for subsequent determination of this highly toxic metal. For maximum collection efficiency, “wet” sampling techniques using wipes are generally preferred. However, there are instances where wet sampling of certain components and equipment are not desirable and dry sampling techniques are required. As an example, for technical reasons, surfaces of certain materials and components must be protected against damage from sample collection; hence, sampling methods that are less aggressive than wipe sampling are required. As shown in Table 2, several standardized procedures for wet and dry sampling have been produced by various organizations. However, standardized sampling methods have not been applied uniformly, and this has led to difficulties in comparing data obtained from different sites and locations. Also, performance data are often lacking for surface sampling techniques, and, as a consequence, collection efficiencies may not be known. Thus, efforts are underway to characterize surface sampling procedures that have been promulgated by

various sources, in order that: (a) comparisons may be made among data from wet and dry sampling procedures; and (b) recommendations can be made on the best sampling techniques for beryllium in surface dust.

TABLE 2—*Standardized sampling procedures for collecting beryllium dust samples from surfaces.*

Method(s)	Sampling Media/Device	Substrate(s) Sampled	Comments
OSHA ID-125G & ID-206	“Wet” or “dry” filter or wipe	Smooth surfaces, dermal samples	Alcohol wipes widely used
NIOSH 9100 & 9102	“Wet” wipe	Smooth surfaces, dermal samples	Individually packaged wipes
ASTM D 6966	“Wet” wipe	Smooth surfaces	Individually packaged wipes; ASTM E 1792 wipes acceptable for Be sampling
ASTM E 1216	Adhesive tape	Smooth surfaces	May damage fragile surfaces
OSHA Technical Manual	Patch samples, hand rinsates	Dermal samples	Various protocols; may sample clothing, gloves, etc.
NIOSH 2600, 3601, 9202 & 9205	Patch samples, hand rinsates	Dermal samples	Developed for sampling pesticides, metalworking fluids, etc.
ASTM D 5438	Modified upright vacuum cleaner	Floors	Sampling from carpets
ASTM E 1973	Sampling cassette with collection nozzle	Rough, porous, uneven surfaces	“microvacuum” Pb dust sampling (withdrawn standard)
ASTM WK4996 (work item)	Sampling cassette with collection nozzle	Rough, porous, uneven surfaces; fragile surfaces	“microvacuum” dust sampling for metals (under development; to replace ASTM E 1973)

On its web page, OSHA has published procedures for obtaining surface samples for assessment of surface contamination [12]. These methods have been recommended for surface sampling for contaminants such as beryllium [3]. The OSHA procedures describe wipe sampling techniques for worksite and dermal surfaces, generally using filters (membrane or fiber, depending on the application) as sampling media. These procedures are currently the only ones published that describe the use of dry sampling media. While filters are easy to handle and their sample preparation and analysis are uncomplicated, the collection efficiency of sampling surface dust when using filters is highly questionable. OSHA has also promulgated evaluation guidelines for surface sampling methods [13], but the procedures described therein prescribe sample collection from ultra-smooth surfaces that are not representative of real-world situations. Due in part to the shortcomings of available surface sampling methods described by OSHA, others have sought to employ and evaluate alternative surface sampling methods for standardization purposes.

Apart from sampling media, there are other confounders in evaluating surface sampling methods. Notably, non-uniformity of surface contamination, e.g., in terms of beryllium loading levels, beryllium compounds, and particle size distribution, can lead to variable results. This is another factor to consider in efforts to harmonize surface sampling methods.

In a recent investigation, a comparison of wipe sampling methods for beryllium was carried out wherein dry, wet, and alcohol wipe methods were evaluated for their application in removing beryllium-containing dust from painted surfaces [14]. This study found alcohol to be most effective for removing beryllium dust from oily surfaces, while (not surprisingly) dry wipes were least effective for this purpose. Wipe sampling of surfaces using robust wipe media, wetted with either distilled water or an organic solvent (such as methanol), has been described in a standard practice developed by ASTM International [15]. This consensus standard, ASTM D 6966, describes the collection of surface wipe samples from delineated areas of smooth, flat surfaces from components such as furniture, room components, and equipment encountered in worksites. The ASTM D 6966 wipe sampling procedure is based on a NIOSH method for collection of surface dust samples for subsequent determination of lead [16]. NIOSH recently extended the wipe sampling procedure for lead to multi-element determination [17]. Candidate wipe materials for sampling may include wipes meeting the specifications of ASTM E 1792 [18], which was developed to address wipe materials for lead dust collection. But besides the results from sampling of oily surfaces presented in the aforementioned study [14], general performance data for beryllium dust sampling from representative surfaces when using ASTM D 6966 are as yet unavailable.

Another consensus standard, ASTM E 1216, describes the collection of surface contaminants in particulate matter using pressure-sensitive tape [19]. This standard practice is targeted for use on surfaces that are not damaged by the use of adhesive tape for sampling purposes. It is meant for the collection of particles 5 μm in diameter and larger, but does not efficiently collect ultrafine ($<1 \mu\text{m}$) beryllium particulate matter, which is of concern [20]. ASTM E 1216 is generally applicable to surface sampling from metal platings, oxide coatings, and robust materials. But the use of this method on painted, vapor-deposited, and optical coatings is recommended with caution and should be carried out only after preliminary evaluation.

Collection of dermal samples from workers' hands has been described briefly in a new NIOSH method for colorimetric lead screening in dust wipe samples [21]. The procedure entails the use of wetted wipes to remove contaminated dust from hands for subsequent analysis. The worker to be sampled is asked to use a fresh wipe to remove dust from first the palms, then the fingers, and lastly, the backs of the hands, with wiping to be done for a period of 30 s per pair of hands. The method is similar to the procedure described by OSHA [12], except that wetted wipes, rather than filters, are specified in the NIOSH 9105 method for dermal (hand) sampling. Other dermal sampling methods that have been used for various skin contaminants, such as patch sampling and collection of hand rinsates, have also been described by NIOSH [22] and OSHA [12]. Unfortunately, dermal sampling procedures have not been well standardized, and this has led to difficulties in evaluating and comparing data from a variety of different studies [23]. Dermal sampling methods for beryllium need to be harmonized, and this remains an important area for further research and development efforts.

Vacuum collection techniques are sometimes used in "dry" methods to sample surface dust, and some of these procedures have been standardized. Sample collection methods using a vacuum may be preferred in lieu of "wet" methods requiring wipes for situations where surfaces are rough or highly porous, or both. For collection of floor dust for subsequent analysis, a

standardized vacuum sampling method has been developed by ASTM International [24]. The procedure is applicable to indoor environments including workplaces, but it has not been evaluated for purposes of beryllium sampling and analysis. The method is especially useful for sampling larger areas of uneven substrates, such as carpets. Vacuum sampling might also be required in cases where it is necessary to prevent damage to the substrate being sampled through direct contact with a collection medium, such as a wipe. To this end, a "microvacuum" dust sampling method is under development in ASTM Subcommittee D22.04 on Sampling and Analysis of Workplace Atmospheres, with collected samples intended for subsequent determination of metals, including beryllium [25]. The method is an extension of an earlier (and now withdrawn) consensus standard describing microvacuum sampling for subsequent lead determination [26]. This procedure entails vacuum collection of surface dust using sampling cassettes to which collection nozzles are attached [27]. With vacuum generated by a low-flow sampling pump, the collection nozzle is used to sample settled dust on delineated areas of surfaces of interest. While it is known that only smaller particles will be collected, no damage will befall the substrate when using this technique since the collection nozzle does not touch the substrate being sampled.

Standardized surface sampling methods that can be applied to the collection of beryllium in surface dust are summarized in Table 2. Unfortunately, performance data are mostly lacking for these procedures, and additional research is needed for the assessment of surface dust sampling techniques for the subsequent determination of beryllium. Nevertheless, in the absence of performance figures of merit, the use of harmonized surface sampling methods will enable better comparability of data from different samplers, sampling sites, locations, and times.

Methods for obtaining bulk samples are outside the scope of this article, but they are briefly mentioned here. An excellent source of information on bulk sampling of soils, solid waste, water, field equipment, etc. is the U.S. Environmental Protection Agency (EPA), which has published an exhaustive tome [28] that covers issues such as: (a) sampling strategies and design; (b) sampling techniques, media, and equipment; (c) standardized sampling procedures developed through voluntary consensus (notably ASTM standards); (d) data quality considerations pertaining to sample collection, sample handling, and transport; and (e) numerous related subjects. A great many relevant ASTM standards on collecting bulk samples have also appeared in ASTM publications on environmental sampling [29,30]. Further study is also needed regarding when it is more appropriate to use bulk sampling as opposed to surface sampling for beryllium. Guidelines and performance data are limited in this area.

Sample Preparation

Laboratory methods are required to prepare beryllium samples for subsequent analysis. To this end, several standardized procedures describing beryllium dissolution techniques have been published. Owing to the refractory nature of beryllium particulate matter, including beryllium oxide (especially the high-fired variety) and beryllium silicates, rigorous strong acid high temperature digestion techniques are generally needed in order to dissolve beryllium samples. Sulfuric or hydrofluoric acids (in concert with other acids such as nitric or hydrochloric) are ordinarily required to put refractory beryllium compounds into solution.

Standardized high temperature acid digestion techniques are available for the purpose of dissolution of beryllium in samples collected from workplaces. A number of options for dissolution procedures has been described in recently published international standards [6,31], but performance data for beryllium recoveries from refractory materials have not yet been

obtained through interlaboratory testing. Hot plate or microwave digestion techniques are widely recommended, but ultrasonic extraction is an option that can be used with hydrofluoric acid digestion. Various governmental and consensus standard methods describing the preparation of beryllium samples from occupational environments are summarized in Table 3. Procedures for the dissolution of wipe samples require larger volumes of acids than the quantities needed for air filter samples. Also, if microwave digestion of wipe samples is carried out, this may require a preliminary hot plate dissolution step. Unfortunately, methods for preparing wipe samples for beryllium determination have not been well standardized to date. An ASTM standard describing sample preparation methods for wipes is currently being considered for development within ASTM Subcommittee D22.04 on Sampling and Analysis of Workplace Atmospheres.

Acid digestion methods for the preparation of bulk samples for subsequent beryllium determination are generally similar to those for air filters. Besides the OSHA procedures listed in Table 3, there is a number of EPA [28] and ASTM [30] hot plate and microwave acid digestion methods that may be applicable to the dissolution of beryllium in bulk samples collected from workplaces.

TABLE 3—*Governmental and consensus standard procedures for preparation of beryllium samples obtained in workplaces.*

Method	Sample Type(s)	Acid Mixture	Digestion Method(s)
NIOSH 7102	Air filter	Nitric & Sulfuric	Hot plate
NIOSH 7300	Air filter	Nitric & perchloric	Hot plate or microwave
NIOSH 7302	Wipe	Nitric & perchloric	Hot plate or microwave
OSHA ID-125G	Air filter, wipe, or bulk	Nitric, sulfuric, & hydrochloric	Hot plate
OSHA ID-206	Air filter, wipe (smear tab), or bulk	Hydrochloric, nitric	Hot plate
HSE 29/2 (UK)	Air filter	Nitric & Sulfuric	Hot plate
INRS Fiche 003 (France)	Air filter	Nitric & hydrofluoric	Hot plate or microwave
ASTM D7035	Air filter	Various options	Hot plate or microwave
ISO 15202-2	Air filter	Various options	Hot plate, microwave, or ultrasound

Laboratory Analysis

Following sample dissolution, laboratory analytical methods are then used to measure the concentrations of dissolved beryllium in sample extracts. Standardized instrumental analytical methods for beryllium are generally based on atomic spectrometric techniques. Graphite furnace atomic absorbance spectrometry (GFAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) are the two most widely used instrumental methods for determining beryllium in extracts from workplace samples. Atomic spectrometric methods for beryllium, as evaluated by NIOSH [2] and OSHA [3], are very precise and unbiased.

New ICP-AES voluntary consensus standards, which were in development for nearly a decade, have recently been published [6,32]; these international standard methods are applicable to workplace beryllium measurements. If ultra-trace beryllium determinations are required, inductively coupled plasma mass spectrometry (ICP-MS) can be employed. EPA methods for

determining beryllium in environmental samples based on GFAAS [28,33], ICP-AES [28,34], or ICP-MS [28,35] have been published. Methods using flame atomic absorption spectrometry (FAAS) are also available [28,36], but method detection limits for FAAS are generally inadequate for the trace analyses that are ordinarily required in workplace beryllium monitoring.

Several standardized analytical methods that can be used to determine beryllium in workplace samples are summarized in Table 4. All of these methods entail aspiration of aliquots of sample extracts into the instrument. GFAAS and ICP-AES yield comparable method detection limits for beryllium. ICP-AES is especially useful for analysis since other elements besides beryllium can also be monitored simultaneously. For cost considerations, ICP-MS is generally not recommended for routine sample analysis. But this methodology may be necessary for determining beryllium in, for example, short-term task-based workplace air samples, or in other instances where very low detection limits may be required. All method detection limits for the methods listed in Table 4 are well below the action level of 0.2 μg Be per sample established for air [37] (1000-L sampling volume) and surface [38] (100 cm^2) samples.

TABLE 4—*Governmental and consensus standard methods for atomic spectrometric analysis of workplace beryllium samples.*

Method	Instrumental Technique	Estimated Method Detection Limit (μg Be/sample)
NIOSH 7102	GFAAS	0.005
NIOSH 7300	ICP-AES	0.005
OSHA ID-125G	ICP-AES	0.013
OSHA ID-206	ICP-AES	0.0072
EPA 7091	GFAAS	0.005
EPA 200.7	ICP-AES	0.008
EPA 200.8	ICP-MS	0.001
EPA 6010B	ICP-AES	0.005*
EPA 6020	ICP-MS	0.0005*
ASTM D7035	ICP-AES	0.009
ISO 15202-3	ICP-AES	Not evaluated

*(Based on a 25-mL sample).

Reference Materials

To evaluate sampling and analytical methods, representative certified reference materials containing beryllium are desired. Several National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs) having certified reference concentrations of beryllium are available [39]; these are summarized in Table 5. Copper-beryllium alloy SRMs are well represented; these are in the form of chips (\approx 0.5 to \approx 1.2 mm diameter for SRMs 458, 459, and 460) or blocks (19 mm \times 31 mm for SRM C1122). Highly divided powders containing certified concentrations of beryllium are represented by bituminous coal (SRM 1632c) and waterway sediment (SRM 1944). Certified beryllium in solution is available as a single element standard solution (SRM 3105a) and as an isotope standard for applications in mass spectrometry (SRM 4325). Some older Be-Cu alloy SRMs have been discontinued, as has an old SRM consisting of beryllium and arsenic spiked onto filter media. There are several other SRMs containing beryllium at non-certified concentrations [39].

TABLE 5—*Available NIST SRMs containing beryllium at certified concentrations.*

SRM #	Description	Certified Beryllium Concentration
458	Be-Cu alloy (chips)	0.360 % (by weight)
459	Be-Cu alloy (chips)	1.82 % (by weight)
460	Be-Cu alloy (chips)	1.86 % (by weight)
C1122	Copper base alloy (block)	1.75 % (by weight)
1632c	Coal (bituminous)	1.0 $\mu\text{g/g}$
1944	NY/NJ waterway sediment	1.6 $\mu\text{g/g}$
3105a	Single element standard solution	10 mg/L
4325	Be 10/9 accelerator mass spectrometry standard (solution form)	5 mg/L; Be 10/9 ratio = 3×10^{-11}

It can be seen from the list of Table 5 that beryllium-containing SRMs are unavailable for media such as air filters and wipes. Also, environmental SRMs containing beryllium oxide are not available either. Efforts are presently underway by the National Institute of Standards and Technology (NIST) to develop representative SRMs containing beryllium oxide, including the high-fired variety, which is particularly refractory in nature. Additionally, there remains a need to certify beryllium concentrations in representative environmental matrices.

Summary

Various standardized sampling, sample preparation, and analytical methods for beryllium in workplace samples have been published by governmental agencies and consensus standards organizations. Within the beryllium analysis arena, our goal is to encourage the development of voluntary consensus standards in areas of interest for which such standards are presently unavailable. Methods for measuring beryllium in workplace air samples are well standardized, as evidenced by the availability of recently published ASTM and ISO international standards. However, taken as a whole, surface sampling methods for beryllium require better harmonization and evaluation. A new ASTM standard procedure for obtaining surface dust wipe samples using wet wiping is available, and, for collecting “dry” samples, a “microvacuum” ASTM standard sampling method is under development. A vacuum sampling method for collecting floor dust has also been standardized in the form of an ASTM procedure. There remains a need for a voluntary consensus standard to describe sample preparation procedures for beryllium in wipe samples. Also, for many of the existing standard procedures, performance data are lacking for refractory beryllium compounds. Reference materials containing beryllium oxide, as well as beryllium in several environmental matrices of interest, are unavailable to date. Candidate areas for further standardization in the beryllium sampling and analysis arena will be undertaken when needs for new standards are identified.

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