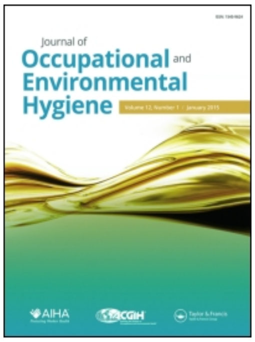

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Interlaboratory Evaluation of a Standardized Inductively Coupled Plasma Mass Spectrometry Method for the Determination of Trace Beryllium in Air Filter Samples

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A collaborative interlaboratory evaluation of a newly standardized inductively coupled plasma mass spectrometry (ICP-MS) method for determining trace beryllium in workplace air samples was carried out toward fulfillment of method validation requirements for ASTM International voluntary consensus standard test methods. The interlaboratory study (ILS) was performed in accordance with an applicable ASTM International standard practice, ASTM E691, which describes statistical procedures for investigating interlaboratory precision. Uncertainty was also estimated in accordance with ASTM D7440, which applies the International Organization for Standardization Guide to the Expression of Uncertainty in Measurement to air quality measurements. Performance evaluation materials (PEMs) used consisted of 37 mm diameter mixed cellulose ester filters that were spiked with beryllium at levels of 0.025 (low loading), 0.5 (medium loading), and 10 (high loading) μg Be/filter; these spiked filters were prepared by a contract laboratory. Participating laboratories were recruited from a pool of over 50 invitees; ultimately, 20 laboratories from Europe, North America, and Asia submitted ILS results. Triplicates of each PEM (blanks plus the three different loading levels) were conveyed to each volunteer laboratory, along with a copy of the draft standard test method that each participant was asked to follow; spiking levels were unknown to the participants. The laboratories were requested to prepare the PEMs by one of three sample preparation procedures (hotplate or microwave digestion or hotblock extraction) that were described in the draft standard. Participants were then asked to analyze aliquots of the prepared samples by ICP-MS and to report their data in units of μg Be/filter sample. Interlaboratory precision estimates from participating laboratories, computed in accordance with ASTM E691, were 0.165, 0.108, and 0.151 (relative standard deviation) for the PEMs spiked at 0.025, 0.5, and 10 μg Be/filter, respectively. Overall recoveries were 93.2%, 102%, and 80.6% for the low, medium, and high beryllium loadings, respectively. Expanded uncertainty estimates for interlaboratory analysis of low, medium, and high beryllium loadings, calculated in accordance with ASTM D7440, were 18.8%, 19.8%, and 24.4%, respectively. These figures of merit support promulgation of the analytical procedure as an ASTM International standard test method, ASTM D7439.

Keywords beryllium, consensus standards, inductively coupled plasma-mass spectrometry, interlaboratory analysis, workplace air

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INTRODUCTION

The ability to measure beryllium at trace levels in workplace air samples is a requirement in view of the low occupational exposure limits (OELs) that have been established for this element.(1–3) Prevention of occupational exposures to airborne beryllium particles by inhalation (and consequent disease) requires the ability to measure this element at extremely low levels in workplace atmospheric samples.(4) In industrial hygiene laboratories, atomic spectrometric techniques have been used historically to determine beryllium in occupational air samples, especially graphite furnace atomic absorption spectrometry (GFAAS)(5) and inductively coupled plasma atomic emission spectrometry (ICP-AES).(6,7)

In efforts to obtain even lower detection limits for toxic metals in workplace atmospheres than those offered by historically used analytical methods, inductively coupled plasma mass spectrometry (ICP-MS) is becoming more widely used in the occupational hygiene field. This trend is evidenced by the availability of a relatively new OSHA ICP-MS method(8) and a recently published ASTM International voluntary consensus standard ICP-MS test method.(9) Compared with GFAAS and ICP-AES techniques, ICP-MS offers improved detection limits for beryllium and other elements, thereby enabling ultra-trace analysis capabilities that may be required for short-term measurements and/or reduced OELs. Also, beryllium determination by ICP-MS avoids spectral interferences from several other metals to which measurement of beryllium by ICP-AES is prone.(10,11)

The aim of the present study was to conduct an interlaboratory evaluation of the ASTM International ICP-MS standard test method,(9) with the goal of establishing estimates of analytical method performance for beryllium based on a round-robin trial. This interlaboratory study (ILS) was performed in accordance with an applicable ASTM International standard practice, ASTM E691,(12) which describes statistical procedures for investigating interlaboratory precision of candidate ASTM standard test methods. Uncertainty of the ICP-MS method for beryllium was also estimated in accordance with ASTM D7440,(13) which applies the International Organization for Standardization (ISO) *Guide to the Expression of Uncertainty in Measuremen*t (*ISO GUM*)(14) to air quality measurements. It is intended that the method performance parameters estimated from this ILS will be used to provide supporting data for the ASTM D7439 (ICP-MS) standard test method. To date, there is a paucity of validation data for ICP-MS analysis of beryllium in occupational air samples, and it was a goal of this study to endeavor to fill this data void.

EXPERIMENTAL

The performance evaluation materials (PEMs) used for the ILS consisted of 37 mm diameter mixed cellulose ester (MCE) membrane filters that were spiked with beryllium at levels of 0.0250 ± 0.0004 (low loading), 0.500 ± 0.006 (medium loading), and 10.00 ± 0.12 (high loading) μg Be/filter. Standard solutions prepared from beryllium sulfate were used for spiking of the filters.

Soluble forms of 20 other metals (Al, Ag, As, Ba, Cd, Cr, Co, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Sb, Se, Sn, U, V, Zn) were also spiked onto the filters at similar loading levels. Media blanks consisted of MCE filters that were spiked with the solution reagent containing no metal and were certified to have a beryllium content of less than 0.01 μg/filter. The as-received filters were mailed in sealed plastic containers in sets of five and were isolated from one another by nonabsorbent separator pads.

Volunteer laboratories possessing ICP-MS equipment and having experience in industrial hygiene elemental measurements were solicited from Europe, North America. and Asia. From over 50 laboratories that were originally contacted, two dozen agreed to participate in the ILS, and ultimately, 20 laboratories submitted results. PEM filter samples were mailed to each volunteering laboratory. Each laboratory received one triplicate set of samples for each beryllium spiking level, plus blanks, yielding a total of 12 PEM filter samples per laboratory. Beryllium spiking levels (as well as spiking levels of other metals present) in all samples were unknown to the participants in the ILS.

The volunteer laboratories were requested to carry out sample preparation (hotplate or microwave acid digestion or hotblock acid extraction) and ICP-MS analysis in accordance with the procedure described in ASTM D7439.(9) Participants were requested to report their results to the coordinator of the ILS in units of micrograms of beryllium per filter sample. Volunteer laboratories were identified by number to ensure confidentiality.

Interlaboratory precision of the beryllium ILS results reported by the participating laboratories was investigated using the statistical analysis described in ASTM E691,(12) which requires a minimum of six participants. In accordance with this consensus standard practice, repeatability and reproducibility of the beryllium results reported were calculated for each of the four PEMs that were analyzed by the volunteer laboratories. Repeatability (r) is an estimate of within-laboratory variability, and this was computed by averaging the squares of the standard deviations of within-laboratory results for each sample having a given beryllium level and taking the square root of this average. Thus, the average within-laboratory standard deviation for each filter sample of a particular beryllium loading is expressed by the repeatability standard deviation, s_r. Reproducibility (R) is an estimate of the variability of both within-laboratory and between-laboratory results. The reproducibility standard deviation s_R is given by the greater of s_r and

$$\{(s_x)^2 + [(s_r)^2(n-1)n^{-1}]\}^{1/2}$$

where s_x is the standard deviation of the mean value as estimated by the average of all ILS results for a given PEM, and n is the number of test results at a particular spiking level. Relative standard deviation (RSD) was calculated by dividing the overall standard deviation s_x by the mean interlaboratory study result for a given spiking level. The RSD for each spiking level was then compared against the minimum precision that is desired (e.g., RSD < 0.20(15)).

The uncertainty in the beryllium ILS data was also examined in accordance with ASTM D7440,(13) which applies the ISO GUM(14) to air quality measurements. This procedure calls for the identification of individual uncertainty components of a given measurement method, thereby enabling an overall uncertainty to be computed via the propagation of uncertainty approximation. To wit, the overall uncertainty is given by $u_c = (\sum u_i)^{1/2}$, where u_i represents the i^{th} individual uncertainty component.(16) In this manner, the overall uncertainty of the test method under investigation is estimated based on the ILS result reported for a particular spiking level and on uncertainty components that are common to each participating laboratory. An expanded uncertainty is then given as $U = k \times u_c$, with k normally taken to be equal to 2 for air quality measurements.(13) Using a coverage factor of $k = 2$ yields approximately a 95% confidence limit about the mean.

For each beryllium spiking level, an estimate of analytical bias was calculated by dividing the difference between the mean of the laboratory-reported triplicate results and the reference value by the reference value. That is, bias $B_i = (\mu_i - R_i)R_i^{-1}$, where B_i, μ_i and R_i are the bias, mean measured concentration and reference concentration, respectively, for the i$^{\text{th}}$ laboratory-reported value for each spiking level.

RESULTS AND DISCUSSION

A list of the 20 laboratories that participated in the collaborative round-robin study and returned results for beryllium is presented in Table I. The raw beryllium ICP-MS data that were received from the collaborating laboratories are presented in Table II. Each volunteer laboratory reported results after sample preparation and analysis of each of the filter PEMs, with sample preparation and analysis carried out on the samples in triplicate. As can be seen from Table II, a number of the laboratories did not submit numerical results for lower beryllium levels and/or for blank filters; i.e., less-than (<) signs were reported in many instances, and many laboratories reported values of zero. Seven laboratories did not report a numerical value for low beryllium loadings (Table II).

Even though the anticipated ICP-MS method quantification limit for environmental samples(17) is in the range of a few nanograms of beryllium per filter, many laboratories' reporting limits were significantly higher. ILS statistical computations were performed using only reported numerical values. Statistical outliers were omitted for the purposes of the statistical treatments performed here (Dixon's Q-test, $P = 0.05$(18)). High apparent blank values reported by four laboratories in particular (Laboratories 7, 11, 13, and 20, Table II) produced statistical outliers for low-spike data, so these laboratories' spiked filter results were blank corrected in subsequent statistical calculations.

Overall ILS precision statistics, based on results reported by the 20 participating laboratories and treated in accordance with ASTM E691,(12) are summarized in Table III. Overall mean values were computed based on the pooled means for the average of the three results reported by each laboratory for each sample. Excluding results for media blanks, computed interlaboratory RSDs ranged from 0.11 to 0.16 for the spiked filter samples (Table III). These precision results are acceptable in view of an overall goal of obtaining interlaboratory RSDs of less than 0.20.(15)

Moreover, these precision estimates are comparable to RSDs obtained during beryllium proficiency analytical testing rounds.(19,20) The data in Table III show that at the low and medium beryllium spiking levels (i.e., Levels A and B), recoveries were quantitative (within 100% ± 10%).

TABLE I. Volunteer Laboratories That Participated in the ICP-MS ILS

Name of Laboratory	Location
Japan National Institute of Occupational Safety and Health (JNIOSH)	Kawasaki, Japan
Hungarian Institute of Occupational Health (HIOH)	Budapest, Hungary
Institut National de Recherche et de Sécurité (INRS)	Vandœuvre-les-Nancy, France
Laboratoire Central de la Préfecture de Police (LCPP)	Paris, France
Eurofins Environnement	Saverne, France
Institut Technique des Gaz de l'Air (ITGA)	Saint-Etienne, France
Health and Safety Laboratory (HSL)	Buxton, Derbyshire, England
Institute of Naval Medicine, Occupational and Environmental Safety Laboratory (OESL)	Gosport, Hampshire, England
Institut de Recherche Robert Sauvé et de Sécurité en Travail (IRSST)	Montréal, Québec, Canada
Y-12 National Security Laboratory	Oak Ridge, Tennessee
Occupational Safety and Health Administration (OSHA)	Sandy, Utah
DataChem Laboratories	Salt Lake City, Utah
University of Cincinnati, Department of Chemistry (UC)	Cincinnati, Ohio
Bureau Veritas North America (BVNA)	Novi, Michigan
Environmental Resource Associates (ERA)	Arvada, Colorado
United States Geological Survey (USGS)	Denver, Colorado
RTI International	Research Triangle Park, North Carolina
Savannah River National Laboratory (SRNL)	Aiken, South Carolina
Navy Central Industrial Hygiene Laboratory (Navy CIHL)	San Diego, California
U.S. Army Center for Health Promotion and Preventive Medicine (USACHPPM)	Aberdeen Proving Ground, Maryland

TABLE II. ICP-MS ILS Beryllium Results Reported by Participating Laboratories

Laboratory No.	Media Blank	Level A (0.0250 μg Be)	Level B (0.500 μg Be)	Level C (10.00 μg Be)
1	<0.008 (×3)	0.0184, 0.0177, 0.0185	0.440, 0.415, 0.495	8.75, 8.35, 8.80
2	(0) (×3)	0.0264, 0.0278, 0.0260	0.494, 0.496, 0.516	9.38, 9.56, 8.61
3	<0.05 (×3)	<0.05 (×3)	0.481, 0.481, 0.470	8.99, 7.81, 7.16
4	<1 (×3)	<1 (×3)	<1 (×3)	8.6, 7.6, 6.0
5	(0) (×3)	0.03, 0.02, 0.02	0.44, 0.46, 0.46	8.84, 8.80, 6.95
6	(0) (×3)	0.023, 0.022, 0.023	0.43, 0.45, 0.44	4.92, 5.24, 5.87
7[A]	0.0156, 0.0243, 0.0214	0.0414, 0.0427, 0.0409	0.536, 0.535, 0.544	10.1, 8.86, 8.04
8	<0.04 (×3)	<0.04 (×3)	0.464, 0.473, 0.450	9.01, 8.36, 8.19
9	(0) (×3)	0.023, 0.022, 0.022	0.480, 0.496, 0.484	9.81, 9.44, 8.16
10	0.00036, 0.00032, 0.00016	0.0251, 0.0253, 0.0250	0.496, 0.501, 0.503	6.42, 4.92, 4.40
11[A]	0.027, 0.028, 0.031	0.068, 0.056, 0.058	0.60, 0.65, 0.59	10, 9.6, 9.0
12	<0.006 (×3)	0.025, 0.030, 0.025	0.47, 0.48, 0.48	9.0, 7.6, 6.5
13[A]	0.074, 0.070, 0.071	0.095, 0.095, 0.094	0.558, 0.538, 0.563	9.28, 7.91
14	<0.010 (×3)	0.021, 0.022, 0.021	0.476, 0.483, 0.490	9.40, 8.82, 8.95
15	<0.50 (×3)	<0.50 (×3)	0.57, 0.58, 0.56	6.93, 7.29
16	<0.25 (×3)	<0.25 (×3)	0.46, 0.44, 0.46	8.9, 7.1, 4.8
17	(0) (×3)	(0) (×3)	0.478, 0.494, 0.493	7.90, 8.33, 7.48
18	0.000, −0.001, 0.002	0.030, 0.024, 0.020	0.544, 0.502, 0.505	9.32, 9.22, 10.1
19	(0) (×3)	<0.10 (×3)	0.61, 0.57, 0.59	7.89, 8.64, 6.11
20[A]	[0.48][B], 0.05, 0.04	0.06, 0.06, 0.07	0.62, 0.62, 0.59	9.93, 8.55, 7.72

Note: Results in units of μg Be/filter sample.

[A] Significant high media blank levels reported by these laboratories resulted in statistical outliers for low-level spike results; thus, values for spiked samples were blank corrected in subsequent statistical calculations.

[B] Statistical outlier.

TABLE III. Precision Statistics for Beryllium Measurements from ILS PEM Filter Samples, Computed in Accordance with ASTM E691

Level/Ref. μg Be per Filter	μ,[A] μg Be per Filter	s_x[B]	s_r[C]	s_R[D]	r[E]	R[F]	*RSD*[G]	*% Rec.*[H]
Level A (low) 0.0250 μg Be	0.0233[I] (n = 13)	0.0038	0.0030	0.016	0.010	0.044	0.16	93.2
Level B (medium) 0.500 μg Be	0.509[I] (n = 19)	0.055	0.019	0.061	0.052	0.170	0.11	102
Level C (high) 10.00 μg Be	8.06[I] (n = 20)	1.22	0.946	1.44	2.65	4.04	0.15	80.6

[A] Overall mean.

[B] Overall standard deviation.

[C] Repeatability standard deviation.

[D] Reproducibility standard deviation.

[E] Repeatability limit (2.8 s_r).

[F] Reproducibility limit (2.8 s_R).

[G] Relative standard deviation (s_x/μ).

[H] Percent recovery ([μ/ref.] ×100).

[I] Values are blank corrected.

TABLE IV. Uncertainty Budget at Various Be Spiking Levels, Using Application of ASTM D7440 to the ILS Data

Uncertainty Source	Uncertainty Component at Be Spiking Level 0.025 μg, 0.50 μg, 10 μg	Degrees of Freedom at Be Spiking Level 0.025 μg, 0.50 μg, 10 μg
Interlaboratory variance	0.083, 0.091, 0.065	6, 18, 16
Intralaboratory variance	0.029, 0.033, 0.101	14, 38, 32
Bias estimation	0.032, 0.021, 0.021	—
Pipetting/dilution	0.008, 0.008, 0.008	—

TABLE V. Uncertainty Estimates Obtained for Different Be Spiking Levels, After Application of ASTM D7440 to the ILS Data

	Low Spike Level (0.025 μg Be)	Medium Spike Level (0.50 μg Be)	High Spike Level (10 μg Be)
95% Bias Confidence Limits	–0.14, 0.00	–0.04, 0.04	–0.12, –0.19
Correction Factor	1.08	1.00	1.18
Combined Uncertainty	9.4%	9.9%	12.2%
Expanded Uncertainty at $k^A = 2$	18.8%	19.8%	24.4%

^{A}Coverage factor.

However, low recoveries (∼80%) were obtained from the highly loaded (Level C) samples. Several participating laboratories reported that, for this spiking level, material had been visibly transferred from the MCE filter to the separator pads between the filters on arrival at their laboratories. Hence, it is thought that loss of material from the highly loaded filters during transport resulted in lower than expected results for this spiking level.

To augment the above analysis, uncertainty estimations through application of ASTM D7440(13) were carried out on the ILS data. Outliers were identified graphically(21) and were not included in subsequent computations. For the low loading level (0.0250 μg Be per filter sample), the estimated overall bias Δ for all laboratories L was:

$$\begin{aligned}\Delta &= \frac{1}{L}\sum_{l=1}^{L}\Delta_l \\ &= -7.1\%\end{aligned}$$

(where l refers to individual laboratories), giving a correction factor $corr$:

$$corr = 1/(1+\Delta) = 1.08$$

Furthermore, the laboratory-bias variance estimate s^2 is:

$$s^2 = \frac{1}{L-1}\sum_{l=1}^{L}(\Delta_l - \Delta)^2 = 0.0790^2 = 0.00624,$$

with $L – 1 = 6$ degrees of freedom, which yields the estimated variance in the bias estimate:

$$Variance[\Delta] = s^2/L = 0.030^2 = 0.00090.$$

The within-lab variance estimate s^2_{intra} is

$$\begin{aligned}s^2_{intra} &= \frac{1}{L}\frac{1}{R-1}\sum_{l=1}^{L}\sum_{r=1}^{R}(err_{lr} - \Delta_l)^2 = 0.0267^2 \\ &= 0.00071,\end{aligned}$$

with $L \times (R – 1) = 14$ degrees of freedom; here r and R refer to individual laboratory results and all laboratory results, respectively,(13) and are not to be confused with the symbols for repeatability and reproducibility as used above.(12) The estimated interlaboratory variance is then found from:

$$s^2_{inter} = s^2 - \frac{1}{R}s^2_{intra} = 0.0774^2 = 0.00599.$$

The magnitude of the pipetting/dilution systematic error in sample preparation is limited to < 0.014 by ISO 8655-2(22) for positive displacement piston pipettes with disposable plunger and capillary and with fixed volume equal to 50 μL. Therefore, this uncertainty component, assuming uniform distribution, is:

$$S_{\text{pipette bias}} = 0.014/\sqrt{3} = 0.008.$$

With corrected data, the above "raw" uncertainties, aside from pipette/dilution bias, are multiplied by the correction factor resulting in the uncertainty budget shown in Table IV. Using a coverage factor $k = 2$, an expanded uncertainty estimate of 18.8% is arrived at for the low-spike ILS data (Table V). Similar treatment of results at medium and high loading yielded the remaining results shown in Table IV. Expanded uncertainty estimates for medium- and high-loading ILS sample results are 19.8% and 24.4%, respectively (Table V). These uncertainty estimates for the ICP-MS method are acceptable in consideration of the NIOSH ±25% accuracy criterion(23) and an allowable uncertainty of ±30% for air analyses conducted in comparison with a relevant European standard.(24) These figures of merit support the ASTM D7439 analytical procedure.

CONCLUSION

Overall, the results of the ILS study indicate that the ICP-MS method is effective for the determination of trace beryllium in air filter samples. These results are to be used as backup data for ASTM D7439 and support promulgation of this ASTM International standard. However, several limitations of this work must be pointed out. For one, the soluble compound of beryllium used here is not difficult to dissolve; hence, this work should not be viewed as a serious challenge of the sample preparation techniques used. Follow-up sample dissolution/ICP-MS studies on refractory beryllium compounds, such as high-fired BeO[25,26] and siliceous material,[15,27] are recommended.

Also, the reported loss of material from the high-spiked filters during transport resulted in low beryllium results for these samples. Considering the difficulty encountered in the preparation and transport of highly loaded filters, further efforts are needed to improve the fabrication and distribution of such materials. High reporting limits for a number of laboratories reflected that, in many cases, significant dilution of acid-digested samples may have resulted in higher method detection limits than were otherwise anticipated. Presently the use of ICP-MS for the determination of beryllium at ultra-trace levels appears to be beyond the capabilities of many laboratories. Better interlaboratory performance for low-level samples could be forthcoming in the future as laboratories attain more experience in ICP-MS analysis of industrial hygiene samples.

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REFERENCES

1. **Brisson, M.J., K. Ashley, A.B. Stefaniak, A.A. Ekechukwu, and K.L. Creek:** Trace-level beryllium analysis in the laboratory and in the field—State of the art, challenges and opportunities. *J. Environ. Monit. 6*:605–611 (2006).
2. **Brisson, M.J., and K. Ashley:** Sampling and analysis issues relating to the ACGIHnotice of intended change for the beryllium threshold limit value. *J. Occup. Environ. Hyg. 2*:D97–D99 (2005).
3. **ACGIH:** *2009 Threshold Limit Values Biological Exposure Indices*. Cincinnati, Ohio: ACGIH, 2009.
4. **Ashley, K. (ed.):** *Beryllium—Sampling and Analysis* (ASTM STP 1473). West Conshohocken, Pa.: ASTM International, 2006.
5. **National Institute for Occupational Safety and Health (NIOSH):** Method No. 7102. In *NIOSH Manual of Analytical Methods*, 4th ed. Cincinnati, Ohio: NIOSH, 1994.
6. **National Institute for Occupational Safety and Health (NIOSH):** Method No. 7300. In *NIOSH Manual of Analytical Methods*, 4th ed. Cincinnati, Ohio: NIOSH, 1994.
7. **Occupational Safety and Health Administration (OSHA):** Method No. 125G. In *OSHA Sampling and Analytical Methods*. Salt Lake City: OSHA, 2002.
8. **Occupational Safety and Health Administration (OSHA):** Method No. 1006. In *OSHA Sampling and Analytical Methods*. Salt Lake City: OSHA, 2005.
9. **ASTM International:** *Standard Test Method for the Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma Mass Spectrometry* (ASTM D7439). [Standard]. West Conshohocken, Pa.: ASTM International, 2008.
10. **Robbins, J.M.:** "Advantages of Multivariate Spectral Deconvolution Techniques for Interference Correction in Beryllium Analysis by ICP-OES." Presented at the 3rd International Symposium on Beryllium Particulates and their Detection, Albuquerque, N.M., 2008.
11. **Thomas, R.:** Chap. 19. Comparing ICP-MS with other atomic spectroscopic techniques. In *Practical Guide to ICP-MS*. New York: Marcel Dekker, 2004.
12. **ASTM International:** ASTM E691, *Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method* [Standard]. West Conshohocken, Pa.: ASTM International, 2005.
13. **ASTM International:** ASTM D7440, *Standard Practice for Characterizing Uncertainty in Air Quality Measurements* [Standard]. West Conshohocken, Pa.: ASTM International, 2008.
14. **International Organization for Standardization (ISO):** *Guide to the Expression of Uncertainty in Measurement (GUM)*. Geneva: ISO, 1993.
15. **Cronin, J.P., A. Agrawal, L. Adams, et al.:** Interlaboratory evaluation of an extraction and fluorescence method for the determination of trace beryllium in soils. *J. Environ. Monit. 8*:955–960 (2008).
16. **Bartley, D.L.:** Analytical performance criteria—Reconciling traditional accuracy assessment with the ISO *Guide to the Expression of Uncertainty in Measurement* (ISO/GUM). *J. Occup. Environ. Hyg. 1*:D37–D41 (2004).
17. **U.S. Environmental Protection Agency (EPA):** *Trace Elements in Water & Wastes—ICP-MS* (Method No. 200.8). Washington, D.C.: EPA, 1994.
18. **Wernimont, G.T.:** *Use of Statistics to Develop and Evaluate Analytical Methods*, W. Spendley (ed.). Arlington, Va.: Association of Official Analytical Chemists, 1985.
19. **Welch, L.:** Beryllium Proficiency Analytical Testing Program. Presentation at the Beryllium Health and Safety Committee Meeting, Savannah River Site, S.C., 2004.
20. **Ashley, K., T.M. McCleskey, M.J. Brisson, G. Goodyear, J. Cronin, and A. Agrawal:** Interlaboratory evaluation of a portable fluorescence method for the measurement of trace beryllium in the workplace. *J. ASTM Int.* 2(9)(2005).
21. **Hund, E., D. Luc Massart, and J. Smeyers-Verbeke:** Inter-laboratory studies in analytical chemistry. *Anal. Chim. Acta 423*:145–163 (2000), and references therein.
22. **International Organization for Standardization (ISO):** *Piston-Operated Volumetric Apparatus—Part 2: Piston Pipettes* (ISO 8655-2:2002). [Standard] Geneva: ISO, 2002.
23. **Kennedy, E.R., T.J. Fischbach, R. Song, P.M. Eller, and S. Shulman:** *Guidelines for Air Sampling and Analytical Method Development and Evaluation* (DHHS [NIOSH] Pub. No. 95-117). Cincinnati, Ohio: NIOSH, 1995.
24. **Comité Européen de Normalisation (CEN):** *Workplace Atmospheres—General Requirements for the Performance of Procedures for the Measurement of Chemical Agents* (EN 482). [Standard] Brussels: CEN, 2006.
25. **Agrawal, A., J. Cronin, J. Tonazzi, et al.:** Validation of a standardized portable fluorescence method for determining beryllium in workplace air and wipe samples. *J. Environ. Monit. 8*:619–624 (2006).
26. **Ashley, K., A. Agrawal, J. Cronin, et al.:** Ultra-trace determination of beryllium in occupational hygiene samples by ammonium bifluoride extraction and fluorescence detection using hydroxybenzoquinoline sulfonate. *Anal. Chim. Acta* 584:281–286 (2007).
27. **Agrawal, A., J.P. Cronin, A. Agrawal, et al.:** Extraction and optical fluorescence method for the measurement of trace beryllium in soils. *Environ. Sci. Technol. 42*:2066–2071 (2008).