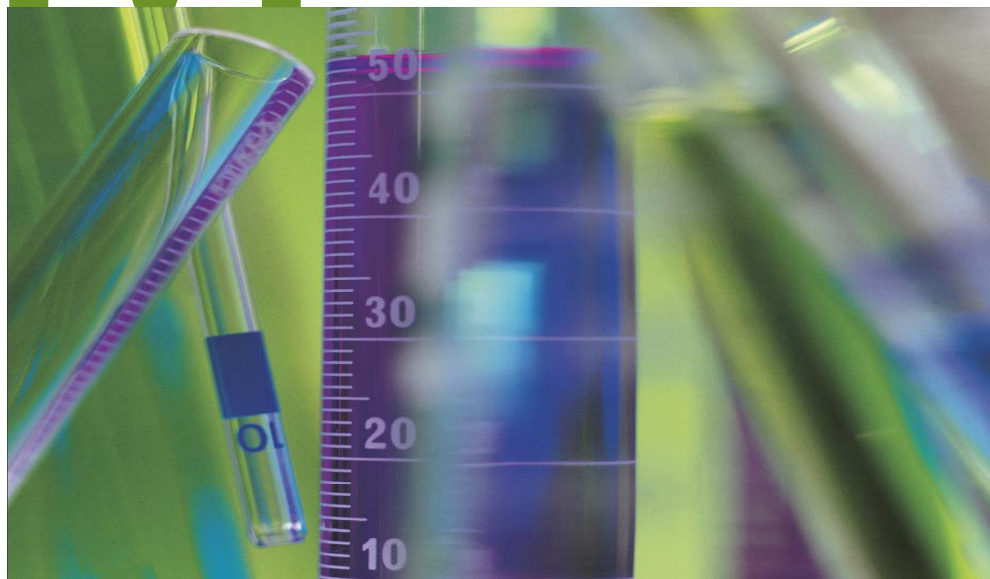


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Analytical Method

Determination of beryllium [7440-41-7] in workplace air

ANALYTICAL METHOD 359



Applicability

This method applies to the determination of beryllium and its compounds in workplace air.

Standard

TWA¹ (Time-Weighted Average): 0.00015 mg/m³

Sampling system

MCE filter, 0.8µm, 25 or 37 mm

Recommended sampling volume and flow rate

TWA¹: 180L at 1.5 L/min

Analysis

Inductively coupled plasma mass spectrometry (ICP-MS)

Minimum reported value (MRV)

0.0005 µg per sample, or 0.0000028 mg/m³ for a recommended sampling volume of 180 L

Range of application

0.001 to 0.04 µg per sample, or 0.0000056 to 0.0002 mg/m³ for a recommended sampling volume of 180 L, or 0.1 to 4 µg/L for a final volume of 10 mL

Reliability

1.2% replicability; 1.8% repeatability

Analytical Uncertainty (CV_A)

2.6%



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en santé et en sécurité du travail,
2008

Analytical Method

Determination of beryllium [7440-41-7] in workplace air

 ANALYTICAL METHOD 359

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IN CONFORMITY WITH THE IRSST'S POLICIES

The results of the research work published
in this document have been peer-reviewed.

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Preamble

The goal of the [Act respecting occupational health and safety](#) in Québec is to eliminate, at the source, dangers to the health, safety and physical well-being of workers. Permissible exposure values (PEVs) for chemical substances have been established in Schedule I of the [Regulation respecting occupational health and safety](#) (RROHS). Section 44 of this regulation entitled “*Methods*” specifies that:

“... These dusts, gases, fumes, vapours and mists found in the workplace environment shall be sampled and analyzed to obtain an accuracy equivalent to that obtained by applying the methods described in the Sampling Guide for Air Contaminants published by the Institut de recherche Robert-Sauvé en santé et en sécurité du travail du Québec, as it reads at the time that it is applied.”

To achieve these objectives, analytical methods for quantifying the workers’ degree of exposure are developed and written to establish appropriate means of control. In order to help health and safety professionals in workplaces, the IRSST publishes, periodically revises, and disseminates the [Sampling Guide for Air Contaminants in the Workplace](#), and the Laboratory Services and Expertise Department publishes contaminant analytical methods.

These methods must be used in combination with the following regulatory and normative references:

- ✓ *Act respecting occupational health and safety*. R.S.Q., chapter S-2.1. Éditeur officiel du Québec, (August 1, 2007).
http://www2.publicationsduquebec.gouv.qc.ca/dynamicSearch/telecharge.php?type=2&file=%2F%2FS_2_1%2FS2_1_A.htm
- ✓ *Regulation respecting occupational health and safety*. S-2.1, r.19.01, O.C. 885-2001. Éditeur officiel du Québec (July 25, 2007).
http://www2.publicationsduquebec.gouv.qc.ca/dynamicSearch/telecharge.php?type=2&file=%2F%2FS_2_1%2FS2_1R19_01_A.htm
- ✓ *Sampling Guide for Air Contaminants in the Workplace*. Operations Division, IRSST, T-15 Guide technique, Montréal, Québec, (March 2005) <http://www.irsst.qc.ca/files/documents/PubIRSST/T-15.pdf>
- ✓ NIOSH, National Institute for Occupational Safety and Health.
- ✓ ISO Guide 30, Terms and definitions used in connection with reference materials, 2nd edition, 1992.
- ✓ ISO, International vocabulary of basic and general terms in metrology (VIM), 2nd edition, 1993.
- ✓ American Industrial Hygiene Association (AIHA), organization that accredits the IRSST laboratory in the field of workplace chemical contaminant analysis and microbiological environmental analysis.

Furthermore, all the terminology used in this method is described in work instruction “I-G-014” of the document management system associated with the IRSST’s quality system.

1. WORKING RANGE

This document presents an inductively coupled plasma mass spectrometry (ICP-MS) analytical method for beryllium metal and its compounds present in airborne particles. The tests used in method development were carried out on a PerkinElmer Sciex Elan DRCII ICP-MS.

The analytical method's linearity was verified for quantities from 0.001 to 0.04 µg of beryllium, which corresponds to concentrations from 0.0000056 to 0.0002 mg/m³ in the air for a recommended sampling volume of 180 L, and from 0.1 to 4 µg/L for a final volume of 10 mL. The coefficient of determination (R²) obtained during method development was greater than 0.999 for this working range.

The document also presents the method's procedure, performance controls, and proposes a routine analytical sequence.

2. PRINCIPLE OF THE METHOD

In accordance with the parameters described in the Sampling Guide, particles containing beryllium metal and its compounds suspended in air are collected by passing a volume of air through a mixed cellulose ester (MCE) filter.

The collected filter and sample are then processed in such a manner as to solubilize the beryllium metal and its compounds. The solution obtained is then analyzed for beryllium by ICP-MS.

3. INTERFERENCES

Two types of interference are differentiated with this analytical technique, namely isobaric and non-isobaric. For beryllium determination, only non-isobaric type interferences are present. They are generally called matrix effects and show up as variations in sensitivity in relation to the composition of the solutions to be analyzed.

Matrix effects can originate in three different processes or locations: in the nebulization process, in plasma, or in the zone of the interface and lens. This type of interference also includes plugging of the nebulizer, the injector tube of the torch and the sampling cone, due to high concentrations of dissolved salt.

The use of a peristaltic pump and the addition of an internal standard to the calibration standards and samples may reduce and effectively compensate for the effects of these physical interferences.

4. MATERIAL

NOTE 1 – The stability of the samples to be analyzed and the solutions depends, to a great extent, on the material making up the container. It is recommended that polyhexafluoroethene propene (FEP), high-density polyethylene (HDPE), polypropylene (PP) or polytetrafluoroethylene (PTFE) plasticware be used.

NOTE 2 – All reusable plasticware must be carefully washed before use and decontaminated by soaking it in dilute nitric acid (for example: 10% HNO₃) for at least 12 hours, and then rinsed several times with demineralized water.

NOTE 3 – All disposable plasticware must previously be subjected to a control to detect the presence of beryllium.

- ✓ Single-use nitrile gloves (powder-free), to prevent any possibility of contamination by hands and contact with corrosive substances;
- ✓ Container, with a capacity of at least 30 mL, for hot plate extraction;
- ✓ Tweezers with flat plastic tips, to remove the filters from the cassettes;
- ✓ Stirring hot plate, thermostatically-controlled, that can maintain a surface temperature of at least 200°C.

NOTE 4 – The surface temperature can vary considerably, depending on the position on the hot plate. It may therefore be useful to characterize the hot plate's performances before use.

- ✓ Piston stroke volumetric pipettes with tips, for the preparation of calibration solutions, calibration control solutions, and dilution of the sample solutions;
- ✓ Calibrated centrifuge tubes, 10 mL, with leakproof caps, and uncalibrated test tubes compatible with the tube holder of the instrument's autosampler;
- ✓ Centrifuge, with a capacity of 3500 revolutions per minute;

NOTE 5 – Since the dissolution method does not always result in complete solubilization of the sample, undissolved material can be eliminated from the sample solution by centrifugation or filtration (single-use 10 mL polypropylene syringes, with a filter incorporating a membrane with a porosity less than or equal to 0.8µm).

- ✓ Volumetric flasks and bottles with leakproof caps of appropriate volume;
- ✓ Inductively coupled plasma mass spectrometer (ICP-MS), autosampler with rinse station, peristaltic pump with several channels and T connection, all interfaced with a computer;
- ✓ Supply of high purity argon (>99.99%).

5. REAGENTS

NOTE 6 – The concentration of the element to be determined and found in the reagents, water and nitric acid must ideally be negligible compared to the lowest concentration to be determined.

NOTE 7 – Use commercial solutions with a certified concentration and guaranteeing traceability to national standards.

NOTE 8 – Commercial solutions used for preparing the calibration control solutions and the control samples on filter must ideally come from a different manufacturer than the one used for the calibration standards for beryllium, or from a different lot or, as a last resort, from a different intermediate solution.

- ✓ Water, with resistivity greater than 18 MΩ-cm at 25°C (CAS 7732-18-5);
- ✓ Concentrated nitric acid (HNO₃), for example: Fisher Trace Metal Grade (CAS No. 7697-37-2);

WARNING – Concentrated nitric acid is corrosive and an oxidant. Avoid all exposure from skin or eye contact, or vapour inhalation. Nitric acid vapours are irritating. Use personal protective equipment (appropriate gloves, face shield or safety glasses, etc.) for any work with concentrated or dilute nitric acid.

- ✓ Triton X-100, for example SigmaUltra (CAS No. 09002-93-1);
- ✓ Commercial solution of beryllium (for example: SPEX Claritas at 1000 mg/L) for calibration solutions;
- ✓ Commercial solution of beryllium (for example: SCP Science PlasmaCal at 10 mg/L) for calibration control solutions and control samples on filter;
- ✓ Commercial solution of lithium (⁶Li) at approximately 1000 mg/L (for example: SPEX Claritas), as internal standard;
- ✓ Commercial solution of scandium (⁴⁵Sc) at approximately 1000 mg/L (for example: SPEX Claritas), as internal standard.

NOTE 9 – Scandium can be used as an internal standard to countercheck the results obtained with lithium.

6. SAMPLING

Beryllium metal and its compounds suspended in the air are sampled using a cassette containing a mixed cellulose ester (MCE) membrane with a porosity of 0.8µm, and a sampling pump whose flow rate has been previously adjusted. For each series of samples, a control cassette containing a membrane from the same lot must be planned for. This control must be processed in the same manner as the cassettes used for sampling for all aspects relating to storage and transport to the sampling point, but air must not be passed through the filters. The samples can be stored at ambient temperature. The sampling parameters recommended for beryllium are described in Table 1.

Table 1. Recommended sampling parameters

| | |
|------------------|-----------|
| Flow rate | 1.5 L/min |
| Volume | 180 L |

These parameters take into account the exposure standard, the sensitivity of the analytical method, and the capacity of the sampling system; however, the sampling volume can be changed to adapt to the environmental constraints. When high concentrations of airborne particulates are expected, the sampling period should be shorter to avoid overloading the filter.

The sampling period must also be long enough to allow the beryllium to be determined at significant levels for industrial hygiene. To calculate the minimum sampling time necessary for the sampled quantity of beryllium to exceed the analytical method's minimum reported value for an estimated time-weighted exposure value, the following equation is used:

$$T_{\min} = MRV / X * TWA * Q \quad [1]$$

where

- T_{\min} = Minimum sampling time, in minutes (min)
- MRV = Minimum reported value for the analytical method, in μg
- X = Estimated time-weighted average exposure value, as a decimal fraction
- TWA = Time-weighted average exposure value, in mg/m^3 of beryllium
- Q = Flow rate recommended for sampling, in L/min

7. ANALYTICAL PROTOCOL

7.1 Solutions

- HNO_3 solution, 1% v/v;
- HNO_3 solution, 3% v/v;
- Intermediate solutions of beryllium in 1% v/v HNO_3 for the calibration solutions;
- Intermediate solutions of beryllium in 1% v/v HNO_3 for the calibration control solutions and the control samples on filter;
- Calibration solutions of beryllium in 1% v/v HNO_3 ;
Prepare the calibration solutions that correspond to the required working range by diluting the corresponding intermediate solutions of beryllium (for example: the calibration blank solution and four calibration solutions)
- Control solutions of beryllium in 1% v/v HNO_3 ;
Prepare the calibration control solutions and the solutions for the controls on filter at the required concentrations by diluting the corresponding intermediate solutions of beryllium (see NOTE 8)
- Intermediate solution of 1% v/v Triton X-100 in 1% v/v HNO_3 ;
- Internal standard solution: 50 $\mu\text{g}/\text{L}$ of ^6Li , 50 $\mu\text{g}/\text{L}$ of ^{45}Sc , 0.01% v/v Triton X-100 in 1% v/v HNO_3 ;

- Rinse solution: 0.01% v/v Triton X-100 in 3% v/v HNO₃.

7.2 Preparation of samples

NOTE 10 – The dissolution method may not completely solubilize the sample. Undissolved material is then eliminated from the sample solution by centrifugation. An appropriate written note is added to the laboratory records to include this observation, and the client is informed in the analytical report.

NOTE 11 – The dissolution method using nitric acid has proven effective for beryllium sulphate, beryllium metal, and alloys with copper and aluminum, and almost completely for beryllium oxide. It is ineffective for solubilizing beryllium in minerals such as Beryl.

NOTE 12 – It is recommended that single-use nitrile gloves be worn during the preparation of the samples, in order to prevent any possibility of contamination by hands and to protect against contact with toxic and corrosive substances.

NOTE 13 – Note in the laboratory records any pertinent remark relating to the state of the sample.

- 7.2.1 Open the cassettes and transfer, using a pair of clean tweezers, each filter into a separate dissolution container bearing the number of the corresponding sample. Follow the same procedure for the field blanks and quality controls on filter;

NOTE 14 – If there is a loss of dust during transfer into the dissolution container, an appropriate written note is added to the laboratory records to include this observation, and the client is informed in the analytical report.

- 7.2.2 Rinse the internal surfaces of the cassette (inlet section) with 7 mL concentrated nitric acid in order to transfer any particles adhering to these surfaces to the corresponding container;
- 7.2.3 Place the containers on a hot plate at approximately 115°C, and then evaporate to dryness without stirring (if the solution becomes darker during evaporation, slowly add some concentrated nitric acid, dropwise, until the solution becomes colourless or slightly yellowish);
- NOTE 15 – Dissolution can take up to 6 hours.
- 7.2.4 Remove each container from the hot plate and allow to cool;
- 7.2.5 Rinse the walls of each container carefully with a few mL of 1% HNO₃;
- 7.2.6 Return the containers to the hot plate and heat with agitation for a few minutes;
- 7.2.7 Transfer the contents to a centrifuge test tube. Rinse the container quantitatively with 1% HNO₃ by transferring after each rinse;
- 7.2.8 Place the caps on the test tubes and allow to cool at ambient temperature;
- 7.2.9 When the samples have cooled, fill the test tubes to the 10 mL mark with 1% HNO₃, cap and then store for later analysis;

- 7.2.10 Centrifuge the test tubes for 10 minutes at 3500 rpm, transfer an aliquot of each sample to test tubes compatible with the tube holders in the autosampler, and then analyze the samples.

NOTE 16 – If the volume of the residues at the bottom of the test tube is significant following centrifugation, this must be taken into account in the final volume of the sample.

NOTE 17 – Note in the laboratory records any relevant remark observed when the sample was solubilize.

7.3 Verification of the ICP-MS performance criteria

Before turning on the instrument, some parts of the instrument should be visually checked: excessive deposits on the cones, cleanliness of the torch, mainly the introduction tube, and flatness of the piping of the peristaltic pump. Cones should be changed and/or cleaned when the deposits are considered excessive, when the sensitivity is insufficient, or when the level of polyatomic ions and/or doubly charged ions is too high.

The manufacturer's recommendations for turning on the instrument must be followed, for example, regarding compliance with the required laboratory environmental conditions, requirements relating to electrical power, cooling water, exhaust ventilation, preheating time, etc.

Once the visual checks have been performed, activate the plasma and allow the instrument to warm up for a minimum of 30 min before using it. The calibration blank solution should be aspirated into the plasma during the heating period in order to avoid changing the conditions of the plasma during the analysis.

Parameterize the instrument according to the manufacturer's specifications for performance verification, and begin the application. The performance results must be equal to or better than the manufacturer's; otherwise different parameters of the ICP-MS must be adjusted, and the verification must be started again. The instrument must be adjusted to reduce interferences (formation of oxides, formation of ions with a charge doublet, for example) to a minimum and to achieve sufficient sensitivity.

The instrument's performances are checked daily, before the samples are analyzed, generally by aspiration of a prescribed solution of elements. Mass calibration and detector optimization must be performed as often as required by the manufacturer or according to the instrument's performance history.

7.4 Calibration

The instrument, a PerkinElmerSciex Elan DRCII (Woodbridge, Ontario, Canada), is equipped with a quadrupole mass spectrometer with platinum cones, a standard torch with a quartz injector (2.0 mm orifice), a cyclonic spray chamber, and a Meinhard quartz nebulizer for introducing the samples.

The ICP-MS is also equipped with an autosampler (PerkinElmer, Ontario, Canada) to automate the analyses. Data acquisition is performed by the company's ELAN software. All calibration solutions, control solutions, internal standard solution and samples are directed to the nebulizer by a peristaltic pump with several channels.

The internal standard elements are added and mixed into all of the solutions before analysis by means of a T connection and the peristaltic pump. The mixture is then transferred to the nebulizer. The sample/internal standard mixture ratio is 5 to 1.

Before and after each analysis, the system's tubing is rinsed with a solution containing 0.01% Triton X-100 in 3% (v/v) nitric acid. The instrument parameters are indicated in Table 2 and have been optimized for beryllium analysis.

Table 2. Analytical parameters

| PARAMETERS | VALUE |
|---|--------------------------------------|
| Isotope | ⁹ Be |
| Plasma power (Watts) | 1100 |
| Argon flow rate (L/min) | 15 |
| Flow rate of auxiliary gas (L/min) | 1.2 |
| Flow rate of gas at the nebulizer (L/min) | ~ 0.9 |
| Measurement protocol | Peak hopping |
| Sweeps (per reading) | 3 |
| Dwell time (ms) | 500 |
| Readings (per replicate) | 10 |
| Mode of analysis | Standard |
| Internal standards | ⁶ Li and ⁴⁵ Sc |
| Calibration | External standardization |

The instrument is calibrated by measuring, in increasing order of concentration, the intensity of beryllium in a number of known calibration standards beginning with a blank solution. When all the calibration standards have been run, the software generates a calibration curve using linear regression via the instrument's computer (measured intensity versus beryllium concentration in the standard solutions). Typically, a calibration blank solution and up to three standards are run. It is seldom necessary to run a calibration with more than five standards.

It is suggested that the signal from the calibration blank solution be subtracted from the signal of the other calibration solutions and that the calibration curve be forced to pass through the origin. The statistical least square regression fit of the data, the coefficient of determination (R^2), should be computed at 0.999 or better. If R^2 is less than 0.999, the calibration standards should be rerun or prepared again to achieve the 0.999 level. It might be possible to remove an erroneous calibration point and then reprocess the data to obtain an acceptable calibration curve, however a minimum of 4 calibration solutions (blank and three standards) should be maintained.

7.5 Quality control

Several types of controls are analyzed during the analytical sequence in order to check the calibration and the variation in sensitivity. Each control must comply with the criteria established for this control and any exceedance or deviation must be documented, and appropriate actions planned in the quality system must be undertaken.

Initial Calibration Verification (ICV). ICV controls are calibration control solutions that are analyzed just after instrument calibration. Two solutions are recommended and their concentrations are approximately 25% (ICV-L) and 75% (ICV-H) of the maximum of the calibration curve.

The commercial solution used for preparing these calibration control solutions must ideally come from a different manufacturer than the one used for preparing the calibration solutions, or from a different lot from the same manufacturer or, as a last resort, from a different intermediate solution from the same manufacturer.

Initial Blank Verification (IBV). The IBV control is the same solution as the calibration blank solution. It is analyzed after the 2 ICV controls, and the results must be comparable to the results obtained during the analysis of the calibration blank solution.

Minimum reported value (MRV). The MRV control is a control solution analyzed after the IBV control and CCB. This control verifies the instrument's response at the analytical method's minimum reported value.

The commercial solution used to prepare these calibration control solutions must ideally come from a different manufacturer than the one used for preparing the calibration solutions, or from a different lot from the same manufacturer or, as a last resort, from a different intermediate solution from the same manufacturer.

Continuing Calibration Verification (CCV). The CCV control is the same solution as the calibration solution approximately representing the concentration in the middle of the calibration curve. The CCV is analyzed sequentially before the CCB and every 10 samples.

Continuing Calibration Blank (CCB). The CCB control is the same solution as the calibration blank solution and is analyzed after every CCV. The results of the CCB must be comparable to the results of the calibration blank solution.

Quality Control Sample (QCS). These control samples are filters spiked with soluble beryllium. They are subjected to all of the preparation and analysis steps for a sample. Two control samples are solubilized and analyzed as samples in the analytical sequence. The results are used to estimate the precision of the analytical method.

The commercial solution used to prepare these calibration control solutions must ideally come from a different manufacturer than the one used for preparing the calibration solutions, or from a different lot from the same manufacturer or, as a last resort, from a different intermediate solution from the same manufacturer.

Laboratory Reagent Blank (LRB). The *LRB* control is a sample that contains only the reagents. It is subjected to all the preparation and analysis steps as a sample. It is used to check whether the preparation steps contaminate the samples. Three (3) reagent blanks are solubilized for each analytical sequence.

7.6 Analysis

Once the calibration curve has been established, the solutions, control samples, reagent blanks and samples are successively analyzed and the beryllium intensity is read against the calibration curve.

The beryllium concentration determined in the sample must fall within the analytical method's working range. If the beryllium concentration in the sample is greater than the highest concentration in the working range, the sample is appropriately diluted to match the matrix of the calibration solutions and analyzed again by taking the dilution factor into account in the calculations.

Furthermore, if the beryllium concentration greatly exceeds the last calibration point in the working range, elimination of the beryllium in the tubing (memory effect) has to be verified by analyzing the blank solution. The result must be similar to the result obtained during the calibration. A longer rinse time corrects this situation.

When the internal standard's signal varies by more than $\pm 20\%$ for a sample, it is important to verify whether dilution of this sample significantly modifies the previously obtained result.

The analyses are done according to the following proposed sequence:

- ✓ Calibration solutions
- ✓ ICV
- ✓ ICB
- ✓ MRV
- ✓ CCV
- ✓ CCB
- ✓ 10 samples (3 laboratory reagent blanks, 2 QCs and 5 samples)
- ✓ CCV
- ✓ CCB
- ✓ MRV
- ✓ 10 samples
- ✓ Calibration solutions
- ✓ CCV
- ✓ CCB
- ✓ MRV...

8. CALCULATIONS

The mass concentration of beryllium for the air sample at ambient conditions is calculated using the following equation:

$$[\text{beryllium}] = (([\text{beryllium}]_1 \times V_1 \times F) - ([\text{beryllium}]_0 \times V_0)) / V \quad [3]$$

where

| | | |
|--------------------------|---|---|
| [beryllium] | = | Mass concentration of beryllium in the air sample, in mg/m ³ |
| [beryllium] ₀ | = | Average concentration of beryllium in the blank samples, in µg/L (see NOTE 18) |
| [beryllium] ₁ | = | Concentration of beryllium in the sample, in µg/L; |
| V | = | Sampling volume, in L, of the sample |
| V ₀ | = | Final volume, in mL, of the solution blanks |
| V ₁ | = | Final volume, in mL, of the sample solution |
| F | = | Dilution factor (F=1 in the absence of dilution) |

NOTE 18 – Blank samples are laboratory reagent blank or filter blank samples solubilized and analyzed using the same method as for the sample. The results obtained for the samples are not corrected for the results of the field blanks. The results of the field blanks are reported as total mass (µg).

9. PERFORMANCE PARAMETERS

9.1 Limit of detection, limit of quantification and minimum reported value (MRV)

The analytical method's limits of detection and quantification were initially evaluated as 0.00005 µg and 0.00015 µg, respectively, for a final volume of 10 mL. The minimum reported value (MRV) is the minimum amount of contaminant that is quantified in the IRSST's laboratory. It takes into account one or more of the following aspects: linearity of the method under the experimental conditions used, the recovery efficiency, and the relevance of the determination at low levels of concentration. The MRV for beryllium is 0.0005 µg.

9.2 Recovery

The solubilization of beryllium using our dissolution procedure was evaluated on different products. Table 3 below presents the recovery percentages for each of these products. The samples consisted of enriched membranes for the soluble form of beryllium, and quantities of beryllium weighed on a membrane for the insoluble forms, all subjected to the entire analytical procedure.

Table 3. Recovery of different beryllium products

| BERYLLIUM PRODUCT | RECOVERY (%) | N | CV (%) |
|---------------------------------------|--------------|----|--------|
| Beryllium (sulfate) BeSO ₄ | 100.3 | 35 | 1.7 |

| | | | |
|---------------------------|-----|----|-----|
| Beryllium (metal), Be | 101 | 11 | 3.7 |
| Beryllium (oxide), BeO | 85 | 10 | 9.5 |
| Beryllium-Aluminum, Be-Al | 99 | 10 | 4.9 |
| Beryllium-Copper, Be-Cu | 93 | 21 | 9.2 |

9.3 Reliability

The analytical method's replicability is 1.2%, and its repeatability, 1.8%. These values were determined in the laboratory from 44 membranes spiked with soluble beryllium subjected to the entire analytical procedure (4 concentration levels, 11 membranes per level).

9.4 Accuracy

The analytical method's accuracy is 99.6% (CV = 5.1%). This value was initially determined with the data (n = 48) originating from participation in an American interlaboratory proficiency testing comparison program (BePAT, AIHA, USA) associated with this accreditation organization. The laboratory continues to participate in this interlaboratory comparison program in order to maintain its accreditation.

The analytical method's accuracy is also verified at each series of analyses using a membrane enriched with soluble beryllium prepared in the laboratory. The results obtained are compiled in the context of the interlaboratory quality control.

9.5 Uncertainty of measurement

The uncertainty of the method's analytical measurement was initially evaluated at 2.6%. It was calculated with the results obtained on 35 membranes enriched with soluble beryllium (5 concentration levels, 7 membranes per level) subjected to the entire analytical procedure. This value corresponds to the analytical method's coefficient of variation (CV_A).

In addition, the expanded measurement uncertainty (CV_E) for the entire determination and sampling is evaluated at 11%. This value was calculated by taking into account an estimated CV of 5% for the sampling, and a chosen one-sided probability threshold of 95%.

10. REFERENCES

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