

Analytical Method

Determination of beryllium concentrations by ICP-MS – Sampling using Solu-Sert™ filter capsules

Method Technical Manager

Mickaël Calosso, M. Sc., Chemist

Contributor to this version of the method

Marie-France Bisson, Laboratory Technician

ANALYTICAL
METHOD

MA-396



Disclaimer

The IRSST makes no guarantee regarding the accuracy, reliability or completeness of the information contained in this document. In no case shall the IRSST be held responsible for any physical or psychological injury or material damage resulting from the use of this information. Note that the content of the documents is protected by Canadian intellectual property legislation. Analytical or calibration methods are the ones developed or chosen by the IRSST to carry out its different mandates. They may require the use of hazardous materials, operations or equipment. These methods do not mention all the safety problems related to their use. It is the user's responsibility to establish the appropriate health and safety practices. Use of the data included in these methods is at the user's own risk: the IRSST is not responsible for any errors or damage that may result from such use and application. The hyperlinks that appear in this document were validated at the time of publication.

A PDF version of this publication is available on the IRSST Web site.

Legal Deposit

Bibliothèque et Archives nationales du Québec, 2018

ISBN : 978-2-89797-026-0

ISSN : 0820-8395

Lorsque imprimé, ce document est non contrôlé.
SVP vous référer au document disponible sur support informatique

No. Révision : 1 - Date de diffusion: 2019-01-29

Lorsque imprimé, ce document est non contrôlé.
SVP vous référer au document disponible sur support informatique

No. Révision : 1 - Date de diffusion: 2019-01-29

SUBSTANCE	CAS No.	STANDARD ¹ (TWAEV ²)
Beryllium, metals and compounds	7440-41-7	0.00015 mg.m ⁻³ ↔ 0.15 µg.m ⁻³

¹ Quebec's regulation respecting occupational health and safety ("Règlement sur la santé et la sécurité du Travail du Québec")

² Time-weighted average exposure value

APPLICABILITY

This method applies to all beryllium and beryllium oxides that can be put into a solution for acid digestion.

Range: 0.0014 µg.m⁻³ to 0.22 µg.m⁻³ of beryllium

N.B.: These values are based on a recommended sampling volume of 180 litres, dissolution in 10 mL, and 1/10 dilution of the sample.

Coefficient of determination (r^2) > 0.998 or **Correlation coefficient** r > 0.999

LIMITATIONS AND INTERFERENCES

The concentrations obtained depend on the method's capacity to solubilize the beryllium present in the samples.

Non-spectral interferences, or matrix effects, originate in the matrix composition and result in the physical suppression of the analyte signal. This occurs when the matrix has too much dissolved salt or acid. The matrix effect can be reduced or eliminated by using an internal standard and/or diluting the sample if possible.

SAMPLING

1) Sampling system

Media	Solu-Sert™ 25 mm and 37 mm (0.8 µm MCE filter membrane in a cellulose shell)
Device	2-piece polypropylene cassette with cellulose support pad

Notes: A control sample containing a filter from the same lot number must be set aside. See reference [1] for the frequency and number of control samples.

Due to the hygroscopic properties of cellulose, Solu-Sert™ filters are not recommended for gravimetric analysis.

2) Recommended sampling conditions

Flow : 1.5 L.min⁻¹ (ajustable from 1.5 to 4 L.min⁻¹)

Sampling time : 120 min

Sampling volume : 180 litres (L)

3) Tested and validated conservation time

Time not determined, stable at ambient temperatures

4) Storage

At ambient temperature

Maximum storage time : Not determined

5) Details

Remove the caps from the cassette and connect the upper part (red cap) to the pump through a flexible tube.

Perform the sampling according to the recommended flow and duration.

After sampling, put the caps back on the cassette and package it for shipping.

For more information on sampling, see the Sampling Guide for Air Contaminants in the Workplace [1].

REAGENTS AND STANDARDS

- Nitric acid, TraceMetal™ grade (CAS 7697-37-2)
- Sulfuric acid, TraceMetal™ grade (CAS 7664-93-9)
- Hydrogen peroxide, Suprapur® (CAS 7722-84-1)
- Beryllium solution at 1000 mg.L⁻¹ for quality control
- Beryllium solution at 10 mg.L⁻¹ for quality control
- Lithium internal standard solution at 100 mg.L⁻¹
- Argon (purity > 99.998%), helium (purity > 99.9999%)

EQUIPMENT

- 30 mL disposable containers for the digestion of filters
- Leakproof graduated flasks of the appropriate volume for preparing standard or control solutions
- Electronic pipettes with disposable tips
- Eppendorf pipette with Combitip adapted as needed
- 15 mL test tube with a conical end for centrifugation
- Filter forceps with corrosion-resistant tips
- Hotplate stirrer
- Centrifuge with accessories for 15-mL test tubes
- ESI SC-4 DX sampler with prepFAST S400V autodilution system
- Inductively coupled plasma mass spectrometer (ICP MS), Perkin Elmer Nexion 350D

Comments:

Due to the carcinogenicity of beryllium (RSST's notation: C1), personnel handling these samples are recommended to wear all safety equipment (gloves, safety goggles, lab coat and at least a half-mask with P-100 particulate filter).

SAMPLE PREPARATION

Number of preparatory steps : 1

Step 1	Acid digestion of samples (Solu-Sert™ + collected particles). Samples retrieved and filled to 10 mL with 1% nitric acid.
---------------	--

Comments

All quality control (QC) samples are subjected to the same procedure as the other samples. Prepare an appropriate number of blanks (recommended: 3) for estimating the contamination level of the reagents and of the digestion step.

The presence of beryllium could not be found in the different lots of Solu-Sert™ encapsulated filters used in the laboratory. Adding a filter blank for each digestion has enabled us to verify this information.

ANALYTICAL CONDITIONS

Analytical technique	: Inductively coupled plasma mass spectrometer (ICP-MS)
Injector	: 1.5 mL injection loop
Pump	: Peristaltic pump set to 6 rpm
Auto-sampler	: ESI SC-4 DX
Auto-dilution system	: PrepFast S400V. Automated calibration curves and QC. Auto-dilution of out-of-range samples
Eluant	: Mixture of 1% nitric acid and 0.1% sulfuric acid
Isotope analyzed	: ⁹ Be
Internal standard	: ⁶ Li
Analysis mode	: Standard mode
Detector	: SimulScan™ dual-stage (pulse counting ou analog)
Plasma torch power rating	: 1600 Watts
Argon gas flow	: 16 L.min ⁻¹
Flow at nebulizer	: ≈ 1 L.min ⁻¹

CALIBRATION

The concentrations in the sample extract are determined by a $Y = AX$ type equation.

Comments:

The beryllium concentration determined in the sample must be within the calibration range of the analysis method. If the beryllium concentration in the sample exceeds the highest concentration in the range, the sample is appropriately diluted with matrix-matching, then the analysis is repeated and the dilution factor is taken into account in the calculations. Consequently, the reported limit (RL) will be adjusted as a function of the additional dilution.

CALCULATION AND EXPRESSION OF RESULTS

Calculation of the concentration of beryllium element in the air sample:

$$C = \frac{(Q_{éch} \times F \times V_{éch}) - (Q_{3b} \times V_b)}{V_{air}}$$

Where:

C	=	Concentration of Be in the air sample, in mg.m⁻³
Q _{éch}	=	Concentration of Be in the sample solution, in µg.L⁻¹
V _{éch}	=	Volume to which the sample was filled, in L
F	=	Total dilution factor (if no dilution, F=1)
Q _{3b}	=	Average concentration of the element in the digestion blanks (3), in µg.L⁻¹
V _b	=	Volume to which the solution blanks were filled, in L
V _{air}	=	Volume of air sampled, in L

Comments:

- The beryllium concentrations in the solution blanks are obtained by averaging the results from analysis of the three digestion blanks. The average contamination of these three blanks is subtracted if that value exceeds the method quantification limit (MQL) and is greater than $0.5 \times RL$ for the element in question.
- An evaluation of beryllium contamination in the Solu-Serts™ showed that the contamination was negligible and therefore did not have to be taken into account. A Solu-Sert™ blank is digested with each analysis sequence for monitoring purposes.
- The results obtained for the samples are not corrected with the results of the field blanks.

METHOD EVALUATION

Note: The validation data show the method's performance at the time of publication. For updated values, see the IRSST website.

Detection limit and Quantification limit

COMPOUND OR ELEMENT	DETECTION LIMIT ($\mu\text{g}\cdot\text{L}^{-1}$)	QUANTIFICATION LIMIT ($\mu\text{g}\cdot\text{L}^{-1}$)
Beryllium	0.0009	0.0031

Precision

COMPOUND OR ELEMENT	REPLICABILITY (%)	REPEATABILITY (%)
Beryllium	1.3	1.5

Accuracy

COMPOUND OR ELEMENT	ACCURACY (%)
Beryllium	98.1

Recovery

COMPOUND OR ELEMENT	RECOVERY (%)
Beryllium	101.4

Measurement uncertainty

Note: The validation data show the method's performance at the time of publication. For updated values, see the IRSST website.

Analytical measurement uncertainty (CV_a) was calculated using the individual results of samples subjected to the entire analytical procedure. It does not take into account a probability threshold (confidence level of 95%, for example) or the contribution of sampling uncertainty.

COMPOUND OR ELEMENT	CV_a (%)
Beryllium	1.2

For more information on calculation of uncertainties, see *Explanatory Document for Analytical Methods Validation Elements, I-G-041*, produced by the IRSST Laboratory Division.

REFERENCES

1. Institut de recherche en santé et sécurité au travail (IRSST). "Sampling guide for air contaminants in the workplace", *Studies and Research Projects*, Technical Guide T-15, 8th edition (update 8.1), 2012, 191.
<http://www.irsst.qc.ca/media/documents/PubIRSST/T-15.pdf>
2. *Regulation respecting occupational health and safety*, CQLR c S-2.1, r 13, updated to 1 April 2018, Quebec, Éditeur officiel du Québec.
<http://legisquebec.gouv.qc.ca/fr/pdf/cr/S-2.1,%20R.%2013.pdf>