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# Cleaning and decontamination of workplaces containing beryllium: Techniques and cleaning solutions

Stéphanie Viau IRSST

Chantal Dion IRSST

**Guy Perrault** 

André Dufresne *McGill University* 

Valérie Turcotte McGill University, MHV Services d'hygiène industrielle Inc.

See next page for additional authors

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#### Auteurs

Stéphanie Viau, Chantal Dion, Guy Perrault, André Dufresne, Valérie Turcotte, Hooman Golshahi, Bethany Campbell, Teodor Mocanu, Annie Ouellet, and Pierre-Jean Désormeaux

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**Chemical Substances and Biological Agents** 

# Studies and Research Projects

REPORT R-614



# Cleaning and decontamination of workplaces containing beryllium

Techniques and cleaning solutions

Stéphanie Viau Chantal Dion Guy Perrault André Dufresne





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With the collaboration of : Valérie Turcotte<sup>4,5</sup>, Hooman Golshahi<sup>4</sup>, Bethany Campbell<sup>4</sup>, Teodor Mocanu<sup>4</sup>, Annie Ouellet<sup>6</sup> and Pierre-Jean Désormeaux<sup>6</sup>

> <sup>1</sup>IRSST <sup>2</sup>Research Department, IRSST <sup>3</sup>Consultant <sup>4</sup>McGill University <sup>5</sup>MHV Services d'hygiène industrielle Inc. <sup>6</sup>Laboratory Services and Expertise, IRSST



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#### SUMMARY

The relationship between beryllium (Be) exposure via the respiratory pathway and health effects has not been well defined. The contribution of skin absorption has been suggested to explain the development of sensitivity (BeS). In this context, monitoring of surface contamination levels is an important tool in controlling emissions. Good cleaning practices for premises where beryllium is used or handled are necessary in preventing the accumulation of beryllium on work surfaces and tools.

The present study evaluated: (1) the effectiveness of different cleaning solutions on surfaces of materials made of copper-beryllium (CuBe) and materials without Be; (2) three surface sampling techniques (wipe sampling with moistened wipes, "micro-vacuuming," and colorimetry using the ChemTest<sup>®</sup>), and (3) cleaning and decontamination methods used in the field.

The results indicate that it is difficult to comply with the threshold value of  $0.2 \,\mu g/100 \,\text{cm}^2$  of Be on surfaces made of copper-beryllium. However, cleaning with a neutral or alkaline product (less aggressive than an acid cleaner) keeps the surface contamination level below  $3.0 \,\mu g/100 \,\text{cm}^2$  (value to be complied with in a zone containing Be with exposure control measures and the wearing of protective equipment). For Be recovery from a surface made of materials without Be, the effectiveness of the different solutions is similar during laboratory tests as well as in field tests on the different surfaces evaluated.

In general, the surface sampling results show a better Be recovery rate with moistened wipes than with the vacuum technique during laboratory as well as field evaluation. The surface (smooth or porous) as well as its condition (condition of the paint, presence of oil, dust accumulation, etc.) have an impact on the effectiveness of these two types of sampling. The colorimetric technique with the ChemTest<sup>®</sup> was not effective on the surfaces studied in the field. In fact, a large amount of dust on the surfaces or the presence of oil overloads the surface of the wipe and inhibits the colorimetric reaction. In addition, the positive results with the ChemTest<sup>®</sup>, obtained under these conditions, are not confirmed by the two other techniques. The laboratory tests clearly show that the technique is ineffective in the presence of beryllium oxide (BeO).

In locations where Be is present, the decontamination procedure can vary, depending on the type of surface to be cleaned. A cleaning cycle generally has two steps: vacuuming of the dusts by using a vacuum equipped with a high efficiency (HEPA) filter, and wet cleaning with detergent. A second cleaning cycle is recommended when the surface contamination remains above  $0.2 \,\mu g/100 \,\mathrm{cm^2}$ . After several decontamination tests were carried out on a friable and worn concrete floor, a sealant was used. Worker exposure to beryllium particles during cleaning work varies with the worker and his work method, the work tool, the task performed, as well as the surface contamination. The wearing of protective equipment (skin and respiratory) remains necessary to protect the workers.

Based on these results, a guide will be prepared to support industries where beryllium is present in developing a cleaning or decontamination protocol so that they can achieve acceptable levels of beryllium.

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#### 1. INTRODUCTION

Beryllium is a metallic element used in alloys because of its specific properties. It is light, nonmagnetic, corrosion-resistant, a good thermal conductor, and has a high melting point. Beryllium is present in a large variety of materials used in various activity sectors. Besides its incorporation in alloys (Be-Cu, Be-Ni, etc.), it is also present in workplaces in the form of beryllium oxide (BeO), and more rarely in the elemental form (Be).

When beryllium is present in the air as dust or fumes, it can be harmful to health. Respiratory diseases linked to this metal have been recognized for many years and are widely studied. Among others, chronic beryllium disease (CBD) is a lung disease essentially characterized by the presence of granulomas in the lungs, which can develop after prolonged exposure to low beryllium concentrations. Chronic beryllium disease is preceded by a sensitization phase (BeS) that is asymptomatic (*Newman, 2005*). Not everyone with beryllium sensitization develops the disease. Skin diseases of the contact dermatitis type have also been observed in workers who have had skin exposure to beryllium.

Since 1999, 38 cases of sensitization and 38 cases of chronic and subclinical beryllium disease diagnosed in workers have been compensated by the Commission de la santé et de la sécurité du travail (CSST, Québec workers' compensation board). The CSST has developed an action plan with its partners (Health network, the IRSST, and joint sector-based associations (ASP)) in order to evaluate the situation in Québec's industries, by giving priority to certain activity sectors (primary metal products (foundries), aeronautics, environment, and the machining of beryllium-containing parts) (*CSST*, 2004).

In the foundry sector, the presence of beryllium was detected in 44 enterprises that employed 13,000 workers. Approximately 400 workers in 18 different enterprises were exposed to beryllium levels above  $2 \mu g/m^3$  (*CSST*, 2004). In the aeronautical sector, beryllium was found in 28 enterprises out of 117 (21,154 workers). Twelve workers in 4 enterprises were exposed to beryllium levels above  $0.2 \mu g/m^3$ , including 3 workers in one enterprise where the exposure was above  $2 \mu g/m^3$  (*CSST*, 2005). The monitoring activities carried out in 83 enterprises in the environmental sector (1900 workers) revealed the presence of beryllium in 26 enterprises where 43 workers were exposed to levels above  $0.2 \mu g/m^3$  (*CSST*, 2006). Québec's occupational hygienists are currently completing their interventions in the 1200 companies in the metal machining sector. It is very likely that several industries will have to clean or decontaminate their workplaces due to the presence of beryllium.

Prevention of BeS and CBD is based on controlling exposure in the worker's breathing zone. The time-weighted average exposure value (TWAEV) is  $0.15 \,\mu$ g/m<sup>3</sup> according to Schedule I of the Regulation respecting occupational health and safety (ROHS) (2007). The previous TWAEV of  $2 \,\mu$ g/m<sup>3</sup>, still in effect in different countries, does not prevent beryllium sensitization (*Kreiss, 1996; Henneberger, 2001; Kolanz, 2001; Deubner, 2001*). The contribution of skin absorption is increasingly suspect in the development of sensitization (*Tinkle, 2003; Day, 2006*).

Good cleaning practices for premises where beryllium is used or handled are necessary in preventing the accumulation of beryllium on work surfaces. Such an accumulation can result in resuspension of the beryllium particles in the air of the work area where these particles were generated, as well as in adjacent zones. Also, the presence of beryllium particles on work equipment increases the workers' potential for skin contact with beryllium (US Federal Register, 1999).

During decontamination of premises where beryllium is present, as with other contaminants such as asbestos and lead, safety measures must apply for maintenance workers and for the people working close to the decontamination zone. The techniques used during maintenance of contaminated zones or equipment must prevent the dispersion and re-suspension of the dust. Two cleaning principles are generally used: vacuum cleaning using a vacuum equipped with a high efficiency (HEPA) filter, and wet cleaning with detergent.

The present study will allow industries where beryllium is present to be supported in developing a cleaning and/or decontamination protocol so that they can achieve acceptable levels of beryllium. The project's objective is to produce a decontamination guide that will be a complement to the publication "Summary of good cleanup and decontamination practices for workplaces with beryllium containing dust" to meet specific needs (*Dion, 2005*). The research report presents the laboratory and field studies that will improve knowledge about the evaluation of cleaning solutions, three surface sampling techniques, and the cleaning and decontamination techniques used in the field. A decontamination guide will be published separately.

#### 2. STATE OF KNOWLEDGE

#### 2.1 Health impacts and environmental monitoring

The relationship between respiratory exposure to beryllium and health impacts is not well established, and the importance of another absorption pathway, through the skin, has been suggested in the development of sensitization (BeS) (*Day*, 2006). Skin exposure to soluble beryllium salts as well as to insoluble beryllium particles of small size ( $\leq 1 \mu$ m), which can cross skin barriers, could contribute to the development of BeS (*Tinkle*, 2003; *Day*, 2006). Several factors can contribute to skin exposure according to the model of Schneider (*Schneider*, 1999). Beryllium can be transferred to the skin directly by different sources (air, surfaces or clothing) during various processes, including cleaning and decontamination work.

A study carried out by Sackett *et al.* (*Sackett, 2004*) revealed the appearance of new cases of BeS and CBD after the start of cleaning operations in some U.S. Department of Energy (DOE) plants. The authors emphasize the importance of environmental and medical monitoring of the workers at the cleaning station, who are likely to be exposed to high beryllium concentrations. The Cardiff Atomic Weapons Establishment (AWE), in the United Kingdom, used beryllium from 1961 to 1997 and implemented its own beryllium control program. The latter imposes strict occupational hygiene principles: process control, control relating to workers, material control and medical monitoring (*Johnson, 2001*).

The authors of one study carried out in a ceramics plant containing beryllium oxide (BeO) concluded that a prevention program reduces the cases of sensitization in newly hired workers during their first year of employment (*Cummings, 2007*). This program must cover respiratory protection as well as the control of skin exposure, clothing contamination, the cleanliness of the working area, and the reduction in the migration of beryllium particles.

The Third International Conference on Beryllium Disease, held in Philadelphia in October 2007, summarized the state of knowledge on the subject (*Conference, 2007*). The main conclusions were that the value of  $2 \mu g/m^3$  (ACGIH, TWA) does not protect the health of workers for all types of beryllium particles (state, size and chemical composition), that the skin may represent a risk of absorption that can lead to BeS, and that uncertainties remain regarding the immunological and genetic aspects of BeS and CBD.

Current knowledge supports the need for reducing worker exposure from beryllium dust absorption through the respiratory and skin pathways by controlling the emission sources.

### 2.2 Surface contamination

Monitoring surface contamination levels is an important tool in controlling emissions (*Dion*, 2004). In fact, surface dust sampling is useful in different situations in order to: (1) detect the presence of Be in enterprises; (2) identify sources of Be contamination; (3) verify the effectiveness of housekeeping procedures or decontamination activities; (4) confirm the absence of cross-contamination between the considered zones where beryllium is present and zones without beryllium; and (5) ensure the proper cleaning of an object, piece of equipment, or establishment becoming accessible to the public.

There is no regulation in Québec on surface contamination by beryllium. In its prevention program, the Department of Energy (DOE) indicates levels of surface contamination to be respected in workplaces with and without beryllium (US Federal Register, 1999). These "acceptable" levels were retained as threshold values in Québec. The beryllium concentration must not exceed  $0.2 \,\mu g/100 \,\mathrm{cm^2}$  on work, equipment and object surfaces in order to be considered "beryllium-free." The value of  $3.0 \,\mu g/100 \,\mathrm{cm^2}$  was retained as the maximum value during non-operating periods in work areas where beryllium is present. However, in this latter case, general and personal protective measures must be applied and a housekeeping program must be implemented. The DOE does not specify the surface sampling method to be used for evaluating surface contamination, except for a recommendation on the use of moistened wipes for better particle recovery.

### 2.3 Cleaning methods

Two cleaning procedures are commonly used to limit surface contamination by dust: vacuum cleaning with a vacuum with a high efficiency filter (HEPA filter), and wet cleaning using a detergent. A vacuum equipped with a HEPA filter is able to trap 99.97% of particles whose dimensions are equal to or greater than 0.3  $\mu$ m. The use of compressed air and dry methods of cleaning is prohibited (*US Federal Register*, 1999). The wet process can be carried out using disposable sponges, cloths or mops or ones that can be reused after cleaning. The sponge mop for floors is not recommended because it only spreads the particles around on the floor without collecting them (*US HUD*, 1995). The use of a high-pressure air jet can be useful for large areas and on surfaces that are difficult to decontaminate. Several documents describe the main safety aspects during the use of this equipment (*ASTM*, 1998; INRS, 2005).

When lead dust is present, three cleaning steps are suggested: (1) vacuuming with a HEPA filter in order to remove the most dust and debris, (2) wet cleaning with a cleaning agent, and rinsing to loosen the dust from the surface, and (3) a second vacuuming with a HEPA filter to remove the particles loosened but not recovered during wet cleaning (US HUD, 1995; US Navy Environmental Health Center, 2002). A one-hour waiting period is suggested before the second vacuuming treatment so that the particles that may have been loosened from the surface and become airborne during the cleaning process can redeposit. The second vacuuming would contribute to a greater reduction in lead on surfaces, particularly when they are difficult to clean (*Yiin, 2004, Grinshpun, 2002*). A second cleaning is also suggested for rough surfaces such as wood. The second vacuuming after wet cleaning may not be required if the acceptable lead concentration is reached after the first two steps (*Dixon, 1999*).

Still relating to the problem of lead, the U.S. Department of Housing and Urban Development (US HUD, 1995) identified points that may explain the difficulty in doing effective cleaning: (1) low reference value; (2) inexperienced workers who favour removing the visible dust, while the invisible dust is just as important; (3) work methods that avoid the propagation of dust, and the storage of dust in tightly sealed containers; (4) respect for work schedules and costs. These aspects can be transposed to beryllium cleaning and decontamination, where the rarity of data on the effectiveness of work methods highlights the usefulness of continuing research in this field.

### 3. OBJECTIVE OF THE STUDY

The general objective of this study was to develop a decontamination guide (French and English) that includes an exhaustive description of the occupational hygiene and prevention practices that should be applied in the context of cleaning and decontamination activities.

The objectives more specifically included:

- identifying means of controlling contaminant dispersion in the air, and identifying the protective equipment required for workers carrying out cleaning and decontamination activities;
- evaluating the cleaning or decontamination efficiency of different cleaning solutions on various types of surfaces to be decontaminated;
- evaluating the effectiveness of different cleaning and decontamination techniques;
- developing a list of cleaning or decontamination practices or steps, including environmental monitoring.

#### 4. DESCRIPTION OF THE WORK

Surface contamination and recovery rates were estimated in the laboratory on components made of copper-beryllium (CuBe) as well as in Petri dishes enriched with beryllium (addition of a known concentration) in order to evaluate the effectiveness of different cleaning solutions. In addition, three surface sampling techniques were studied in the laboratory in Petri dishes enriched with different beryllium compounds. In conjunction with these tests, the research team accompanied enterprises in cleaning or decontamination activities. A follow-up of the work was then done through the collection of air and surface samples.

### 4.1 Cleaning solutions

The effectiveness of six cleaning solutions was evaluated in the laboratory on a surface made of copper-beryllium (CuBe) alloy, in enriched Petri dishes, as well as in enterprises on different beryllium-contaminated surfaces.

#### 4.1.1 Cleaning solutions used

The cleaning solutions were chosen on the basis of literature data or because they had already been used in some enterprises. Liquinox<sup>®</sup> from Alconox was used for decontamination work carried out in Rocky Flats in the United States (*Rocky Flats, 1997*). Following a discussion with this supplier, based on the needs expressed, three detergents were chosen for the project: Citranox<sup>®</sup> (basic solution), Luminox<sup>®</sup> (basic solution) and Alconox<sup>®</sup> (acid solution). The solutions were prepared according to the instructions in the supplier's technical bulletins (*Alconox, Web site*). Another solution tested was the household product "Fantastik<sup>®</sup>," from S.C. Johnson (alkaline solution), which is used to decontaminate beryllium-contaminated surfaces, mainly by the Los Alamos National Laboratory (LANL) (*Taylor, 2002*). A 5% NaCl solution (pH of 6.57) at 21°C and at 60°C as well as tap water were studied for comparison purposes. The industrial cleaning product and degreaser "Z-99<sup>®</sup>," is an inorganic salt (basic solution) used by the company owning the moulds that were used for the CuBe surfaces in this study. This product was studied during laboratory as well as field tests. The two following solutions were the subject of field tests only: "Ledizolv<sup>®</sup>," a cleaning product, liquid degreaser, alkaline (pH of 12).

The safety data sheets as well as the technical bulletins for these products are available on the Internet<sup>1</sup>.

<sup>&</sup>lt;sup>1</sup> <u>http://www.alconox.com/static/msds/msds\_alconox.asp</u> <u>http://www.alconox.com/static/msds/msds\_citranox.asp</u> <u>http://www.alconox.com/static/msds/msds\_luminox.asp</u> <u>http://www.additionconfort.com/Fichessignaletiques/fantastik.pdf</u> <u>http://www.ledizolv.com/LearnAbout/LedizolvMSDS/lszmsds.asp</u> <u>http://maunco.com/photos/custom/MSDS/Z-99%20msdseng.pdf</u> <u>http://www.resolvesolvents.co.uk/pages/index.asp?area=4&area2=7</u>

## 4.1.2 **Procedures on copper-beryllium surfaces**

The goal of evaluating the cleaning of a beryllium surface with various cleaning solutions was to verify the possibility of reducing the quantity of this contaminant present at the surface. In fact, significant quantities of beryllium had been observed during routine sampling with moistened wipes (Ghost Wipes<sup>®</sup>), on surfaces of components made of copper-beryllium (2% Be) alloy.

The first surface used originated from two moulds (A and B) made of aluminum (Al) and CuBe alloy (2% Be), used in producing plastic bottles (Figure 1 A). The moulds were divided into twelve surfaces whose variable area (45 to 135 cm<sup>2</sup>) was estimated using a ruler. The surfaces were classified according to their location (inside and outside the mould) as well as their visual aspect (porous or smooth surface). Figure 2 shows the different surfaces for mould A; the same procedure was applied to mould B. Each cleaning solution was applied to two surfaces (generally one inside and the other outside the mould). For both moulds, three surfaces (A1-R, A5-R and A9-R) received no cleaning treatment and were used as a reference.

The second surface studied corresponded to five new CuBe plates (1.6 to 2% Be), approximately 2 mm thick and with a surface area of 100 cm<sup>2</sup>, from Brush Wellman (Figure 1 B). The top side of the plate was used for the surface sampling tests with the solutions, and the bottom was used as a reference. The safety data sheet for the CuBe surface is available on Brush Wellman's Web site<sup>2</sup>.



Figure 1: A - mould with Al and CuBe surfaces. B - new CuBe plates

<sup>&</sup>lt;sup>2</sup> http://www.brushwellman.com/EHS/MSDS/A08.pdf



Figure 2: Evaluated surfaces (A2-A4, A6-A8, A10- A12) and reference surfaces (A1-R, A5-R and A9-R)) for mould A (n=12)

A1-R = Reference for A2-A4; A5-R = Reference for A6-A8; A9-R = Reference for A10-A12; A9-R-A12 = porous surfaces

Surface sampling was done by wiping with moistened wipes (Ghost Wipes<sup>®</sup>), according to the procedures in kit #3080 available on the IRSST's Web site (*IRSST, 2008*).

For each of the cleaning solutions, a four-step protocol was followed:

- 1. Surface sampling with a Ghost Wipe<sup>®</sup> was done first on the surface. In addition to estimating the surface's starting contamination, this sampling verified the homogeneity of the beryllium content on all the surfaces.
- 2. One sample or a series of several samples were done on the same surface with a Ghost Wipe<sup>®</sup> soaked in a cleaning solution (Figure 3, A and B). Two wipes could be used for a single sample, depending on the contamination visible on the first wipe.
- 3. All the cleaned surfaces were rinsed three times with deionized water. This step removed the salts and other possible residues from the surface.
- 4. One sample with a Ghost Wipe<sup>®</sup> finally verified whether there was residual Be on the surface after cleaning.

All the steps in the protocol were performed under a hood equipped with a local ventilation system. At the end of each day of sampling, the work area was cleaned and surface samples were collected in order to ensure that no beryllium was present.

Two other decontamination tests were performed on the surface of mould A: brushing of the CuBe surfaces with a toothbrush soaked in the cleaning solution and immersion of the mould in soapy water with brushing of the surface with a scrubber (Figure 3, C and D).



Figure 3: Techniques for cleaning the surface of moulds and plates made of CuBe: A - soaking the wipe in the cleaning solution, B – cleaning the surface with the wipe soaked in the solution, C – brushing the surface, D – immersion of the mould in soapy water

### 4.1.3 Procedures in Petri dishes

Experiments in the laboratory were carried out in Petri plates in order to compare the cleaning solutions and the sampling methods, under controlled conditions, in relation to different beryllium compounds

Two solutions (Be acetate  $(Be_4O(C_2H_3O_2)_6)$  and beryllium chloride  $(BeCl_2)$ ) and a suspension (beryllium oxide (BeO)), at a mass of approximately 0.2 µg of beryllium, were deposited, using a pipette, in Petri dishes (Figure 4, A). After evaporation of the liquid phase for approximately 70 hours under the hood, the deposits of Be compounds were sampled, on the Petri surface, with the moistened wipes soaked in the solutions studied (Figure 4, B).



Figure 4: Procedures in Petri dishes: A – samples enriched with 0.2 µg (BeO), B – sampling of fortified Petri dishes

The procedure followed to prepare the solutions and suspension is described in Appendix A. The preparations were analyzed using the inductively coupled plasma mass spectrometry (ICP-MS) technique to obtain the actual beryllium concentration that will be used as the target value. Surface samples were collected in 108 Petri dishes (Table 1), and the recovery rate was calculated using the following formula:

$\mathbf{R}$ ecovery rate (%) –	Quantity of beryllium sampled with the cleaning solution ( $\mu g$ )			
(70) =	Target value of the preparation ( $\mu g$ )			

	Number of samples							
	Alconox®	Citranox <sup>®</sup>	Luminox®	Water	NaCl 5% (21°C)	NaCl 5% (60°C)	Control	Total
Be acetate	6	6	6	6	6	6	3	39
BeCl <sub>2</sub>	6	6	6	6	NA	NA	3	27
BeO	6	6	6	6	6	6	6	42
Total	18	18	18	18	12	12	12	108

#### Table 1: Number of Petri dishes prepared to evaluate the cleaning solutions

NA: Not applicable

### 4.1.4 Field procedures

The laboratory protocol used to evaluate the cleaning solutions was applied in the companies on different surfaces (Figure 5). The visited companies were: a plastics industry using moulds made of CuBe (enterprise 1), a plant manufacturing parts made of beryllium (enterprise 2), and a foundry (enterprise 5).



Figure 5: Tests carried out in parallel in the evaluation of solutions: A – on a painted concrete surface, B – on a concrete wall

#### 4.2 Surface sampling techniques

The second laboratory study consisted of evaluating three sampling techniques: sampling with moistened wipes (Ghost Wipes<sup>®</sup>), micro-vacuuming, and colorimetry (ChemTest<sup>®</sup>). Five parameters were considered in this study: the sampling technique (n = 3), the beryllium compound (n = 4), the quantity of beryllium (n = 3), the type of surface (n = 3), and interindividual variability (for wipes only).

#### 4.2.1 Surface sampling techniques

The sampling technique using individually sealed moistened wipes (Ghost Wipes<sup>®</sup>), applied according to method ASTM D 6966 (2003), is the one most commonly used (Figure 6 A).

Among the sampling techniques using vacuuming, the "micro-vacuum" system is the most appropriate for sampling a 100 cm<sup>2</sup> surface (Figure 6 B). The system combines the standard method ASTM 7144 (2005) and a non-standard method used for the national monitoring of lead by the U.S. Department of Housing and Urban Development (HUD) (*Farfel, 1994*). It uses a pump that can supply a flow of 16 L/min coupled with a 37-mm polystyrene sampling cassette containing a mixed cellulose ester (MCE) membrane with a pore size of 0.8  $\mu$ m, to which was added a collecting tube approximately 5.5 cm in length, bevelled to 45°.

The third sampling technique, the ChemTest<sup>®</sup> (*Microteq*<sup>®</sup>, *Web site*), uses a system with wipes to detect the presence of beryllium in a few hours on the sampling site using a colorimetric method (Figure 6 C). The presence of Be on the surface causes the wipe to change colour (from pink to blue) due to a chain of chemical reactions. The wipes are sold in a box containing all the material necessary for the sampling and analytical method: 20 wipes, a wetting solution (deionized water), 3 developing solutions, 20 developing plates, and 20 sample bags for the wipes after sampling. The method's limit of detection is  $0.2 \mu g$  of beryllium per wipe, namely the threshold value for confirming the presence of beryllium on a 100 cm<sup>2</sup> surface.



Figure 6: Sampling techniques . A – moistened wipes (Ghost Wipes<sup>®</sup>), B – micro-vacuuming, C – colorimetry (ChemTest<sup>®</sup>)

### 4.2.2 Procedures

Plastic, glass and aluminum Petri dishes were enriched with a known quantity (0.1, 0.2 and 0.4  $\mu$ g) of a beryllium compound (Be acetate, BeCl<sub>2</sub>, AlBeMet and BeO). The surface samples (wipe samples with Ghost Wipes<sup>®</sup>, vacuuming, and ChemTest<sup>®</sup>) were carried out in the Petri dishes following an evaporation period of approximately 70 hours in a laboratory hood. The procedure followed in preparing the solutions and suspensions is presented in Appendix B. The recovery rate was calculated using the following formula:

$\mathbf{P}_{acovery}$ rate $(\%)$ –	antity of beryllium sampled by the cleaning technique (µ		*100
Recovery rate $(70) =$	Target val ue of the preparatio n (µg)		100

A total of 192 Petri dishes were sampled (Table 2). The same person carried out the procedures. However, three additional moistened wipes were used by a second operator on the three surfaces, with the quantity of 0.4  $\mu$ g of Be. This allowed the inter-individual difference in the moistened wipe sampling to be evaluated.

Groufe ee	µg of Be	Number of samples					
Surface		Moistened wipe	Vacuuming	ChemTest <sup>®</sup>	1 otal		
	0,1	3	3	3	9		
	0,2	3	3	3	9		
Plastic	0,4	6	3	3	12		
	Control	2	2	2	6		
Glass	0,4	6	NA	NA	6		
Aluminum	0,4	6	NA	NA	6		
Total		26	11	11	48		

Table 2: Number of Petri dishes prepared for each beryllium compound

NA: Not applicable

All the steps in the protocol were carried out in a hood equipped with a local ventilation system. After each day of sampling, the work area was cleaned, and surface samples were collected in order to ensure that there was no beryllium present.

The surface sampling techniques were also evaluated in the field in three enterprises: dental technique teaching college (enterprise 3), mould manufacturing company (enterprise 6), and plastic bottle manufacturing company using CuBe moulds (enterprise 7).

#### 4.3 Workplace interventions

In order to evaluate the effectiveness of the cleaning techniques in various work environments, the research team accompanied four enterprises in the context of their occasional cleaning or decontamination activities. Three of them did a complete decontamination of the work areas (enterprises 1, 2 and 3). Another enterprise had to set up a "beryllium contaminated" area with worker protection measures and establish a cleaning protocol for the premises (enterprise 4).

One intervention in an aluminum plant involved the testing of cleaning solutions but no decontamination or cleaning activity (enterprise 5). Finally, two enterprises were visited to evaluate the three surface sampling techniques (enterprises 6 and 7).

#### 4.3.1 Types of enterprises visited

#### 4.3.1.1 Enterprise 1

This company manufactures plastic containers for the food industry. The extrusion process uses moulds, some of which are partially made of a copper and beryllium (CuBe) alloy. The shop where the moulds are machined was completely decontaminated by means of dust and debris vacuuming followed by wet cleaning with a liquid cleaner and degreaser (Resolve<sup>®</sup>). Surface samples were collected in this company in order to evaluate different cleaning solutions.

#### 4.3.1.2 Enterprise 2

This company was specialized in the manufacture of metal components for the military, aerospace and telecommunications industries. At the time of decontamination, the company had not occupied the premises for more than two years. Due to the size of the area to be decontaminated and the presence of many contaminated machines, the decontamination activities extended over a period of close to two years. In the context of this study, seven interventions were carried out over a period of approximately one year. It should be noted that contamination was present on different surfaces, vertical as well as horizontal (cement, steel, painted concrete, porous concrete, metal, etc.). The interventions were organized so that air and surface sampling was carried out at each step in the decontamination. These steps involved vacuuming, mainly of the horizontal surfaces, followed by wet cleaning with an industrial solvent (Ledisolv<sup>®</sup>) using a high-pressure jet. Surface sampling was done in this company in order to evaluate the effectiveness of the different cleaning solutions.

#### 4.3.1.3 Enterprise 3

Enterprise 3 is a college that provides training in dental techniques where the use of certain beryllium-containing materials had sometimes been required. These materials had not been used for about ten years. Beryllium contamination was present inside the teaching facilities where the practical work was done (window frames, tops of lights, above ventilation ducts) as well as in the ventilation ducts. Complete decontamination of the premises was done in two steps: vacuuming of debris and dust, followed by wet cleaning with an industrial solvent (Ledisolv<sup>®</sup>). The ventilation system was dismantled so that it could be replaced by a new one. For this, the ducts were detached from the structure (ceiling) in sections, cut along their lengths, cleaned by vacuuming and wet cleaning, and folded and packaged for shipping to a metal recycling company. The results provided by the company on the follow-up of the decontamination will be briefly discussed. However, the three surface sampling techniques were evaluated during a visit before the work was started.

#### 4.3.1.4 Enterprise 4

This company, similar to enterprise 1, manufactures plastic bottles from moulds made of CuBe alloy. The problem in this company mainly involved the presence of beryllium on the moulds at concentrations above the reference value, even after wet cleaning with a solvent. Two of these moulds were transported to the IRSST's laboratories to evaluate the effectiveness of the different cleaning solutions.

#### 4.3.1.5 Enterprise 5

The effectiveness of the cleaning solutions was also evaluated in an aluminum plant. The experiments were conducted in the anode sealing department where spent anodes (waste anodes) are cleaned, which consists of removing the electrolytic bath (containing beryllium) that adheres to the surface of the anodes during electrolysis. The intervention was carried out during the department's normal activities.

#### 4.3.1.6 Enterprise 6

Enterprise 6 mainly produces injected plastic components for industries such as medical and computer industries. It manufactures its moulds, some of which contain inserts made of CuBe. The visit followed a cleaning activity and evaluated the three surface sampling techniques.

#### 4.3.1.7 Enterprise 7

This enterprise is specialized in the manufacture of moulds (for automobile components, plastic bottles, etc.) by extrusion, intrusion, self-moulding and electroerosion processes. It manufactures and repairs moulds, some of which contain inserts made of CuBe. The visit followed a cleaning operation and evaluated the three surface sampling techniques.

## 4.3.2 Sampling techniques

During the visits to the enterprises, air samples and surface samples were collected during the decontamination work. Surface samples were also collected before the work began in order to determine the initial contamination, as well as after the work to determine the effectiveness of the decontamination.

#### 4.3.2.1 Air samples

Air samples were collected in the workers' breathing zones during the cleaning work. Other stationary samples, close to the work area, were used for evaluating the extent of the air contamination as a function of the work methods. The samples were collected according to the IRSST's "Sampling Guide for Air Contaminants in the Workplace" (2005).

The sampling devices used during the interventions is presented in Figure 7: (A) 37-mm cassette with a 4-mm opening and a mixed cellulose ester (MCE) membrane with a pore size of 0.8  $\mu$ m ("total" dusts); (B) IOM<sup>®</sup> (Institute of Occupational Medicine) sampling head with MCE membrane, 25 mm (inhalable dusts); (C) 3-part 37-mm open cassette with an MCE membrane, 0.8  $\mu$ m connected to an aluminum cyclone (respirable dusts); (D) 8-stage Sierra<sup>®</sup> cascade impactor (Andersen<sup>®</sup> model 298) with 34-mm MCE substrates for collecting aerosols in a particle range varying from 0.52  $\mu$ m to 21.3  $\mu$ m (inhalable and respirable dusts).



# Figure 7: Sampling devices : A – sampling cassette, B - IOM<sup>®</sup>, C – aluminum cyclone, D - Sierra<sup>®</sup> impactor

#### 4.3.2.2 Surface samples

The effectiveness of the cleaning and decontamination techniques was verified with surface samples collected with moistened wipes (Ghost Wipes<sup>®</sup>) (*IRSST*, 2008).

#### 4.4 Analysis of samples

The MCE filters were subjected to acid digestion in order to solubilize the dusts and determine their beryllium concentration. Beryllium analysis was done using inductively coupled plasma mass spectrometry (ICP-MS) according to IRSST method 359 (*IRSST, 2008b*). The minimum reported value (MRV) is 0.0005  $\mu$ g.

The Ghost Wipes<sup>®</sup> were subjected to the same treatment and were analyzed in a similar way as the MCE filters for their beryllium content, according to IRSST method 359. The MRV is  $0.05 \mu g$ .

For the vacuuming system (micro-vacuum), the plastic sampling tube was cleaned with concentrated nitric acid. The resulting solution was added to the digestion liquid for the corresponding cassette's filter in order to ensure that all of the collected dust was analyzed. The analyses were done in the same way as for the MCE filters.

### 5. **RESULTS**

The results are presented in three sections: cleaning solutions (laboratory part and field part), surface sampling techniques (laboratory part and field part), and cleaning and decontamination techniques (field).

## 5.1 Cleaning solutions

The cleaning solutions were first evaluated in the laboratory on CuBe surfaces (moulds and plates) as well as in beryllium-enriched Petri dishes. The methodology was then applied in the workplace on different types of surfaces.

## 5.1.1 Evaluation on surfaces made of copper-beryllium alloy

#### 5.1.1.1 Moulds

The tests were carried out on two moulds (A and B). Surface wipes (F) were performed using moistened wipes as well as moistened wipes soaked in the studied solution to do the cleaning (N).

Figure 8 presents the results for mould A:

- N1-N7 = wipe samples with wipes soaked in cleaning solutions;
- F1-F6 = wipe samples with moistened wipes;
- F7 = wipe samples after cleaning with a toothbrush;
- F8 = wipe samples after rinsing with water;
- F9 = wipe samples after total immersion of the mould in soapy water.

Figure 9 presents the results for mould B:

- N1-N10 = wipe samples with wipes soaked in cleaning solutions;
- F1-F4 = wipe samples with moistened wipes.

The wipe samples were collected over a period of a few weeks, with week 1 corresponding to the start of sampling.

The results presented in these two figures show that Citranox<sup>®</sup> (acid solution) is much more effective than the other tested products (neutral or basic solutions) in recovering beryllium from the surface. For the two moulds, the beryllium concentrations were generally above the reference value, even for wipe sampling with moistened wipes (F) following cleaning (residual contamination) for the majority of the solvents tested.



Figure 8: Beryllium concentrations on the different surfaces of mould A

- F wipe samples
- N wipes soaked in cleaning solution


Figure 9: Beryllium concentrations on the different surfaces of mould B

F wipe samples

N wipes soaked in cleaning solution

Note that the inside and outside surfaces of the mould that were cleaned with the Citranox<sup>®</sup> acid solution returned to their copper colour, suggesting the absence of oxidation (Figure 10). This observation is specific to Citranox<sup>®</sup>; the other studied solutions did not show this capability.



Figure 10: Cleaning of surfaces (inside and outside the mould) with the acid solution

#### 5.1.1.2 Plates

The results of the tests performed on the CuBe plates with the solutions also demonstrated that the Citranox<sup>®</sup> solution is the most effective for loosening beryllium from the surface (Figure 11). For each solution, the different samples (F and N) were collected successively. The treatments were performed twice with Citranox<sup>®</sup> only, on the same surface, at a 2-week interval. Note that, compared to the results for the sampling on the moulds, the concentrations are below the reference value for all the other solvents.



**Figure 11: Beryllium concentrations on the CuBe plates** 

#### 5.1.2 Evaluation of the solutions in the enriched Petri dishes

The target values obtained for the two solutions (Be acetate and BeCl<sub>2</sub>) and the suspension (BeO) following analysis by ICP-MS are presented in Table 3. Considering the difficulties involved in reaching a target value of 0.2  $\mu$ g for the BeO suspension (insoluble compound), a concentration close to 0.4  $\mu$ g was retained.

Beryllium compound	Preparation	Target value (µg)
Beryllium acetate	Solution diluted in water	0,203
BeCl <sub>2</sub>	Solution diluted in nitric acid	0,204
BeO	Suspension in nitric acid	0,387

Table 3: Be target value for the solutions / suspension of Be compound

The beryllium recovery rate varied with the solvent used and with the compound studied (Figure 12). With Luminox<sup>®</sup>, recovery in the beryllium acetate-enriched Petri dishes was above 100%, which would be acceptable, considering the uncertainty related to the analytical method for beryllium (in the order of 3% with MCE filters). With beryllium oxide, the recovery rate varied from 50 to 81%. This was expected since the average recovery rate for acid digestion of BeO alone, as measured in the validation of IRSST analytical method 359, is in the order of 80%.



Figure 12: Beryllium recovery rate in the Petri dishes

#### 5.1.3 Field evaluation of the solutions

The results of the tests performed in the three enterprises with the cleaning solutions are presented in Appendix C. Six types of surfaces were sampled: metal, painted metal, wood, Plexiglas<sup>®</sup>, concrete, and painted concrete. The beryllium concentration decreased after the cleaning steps on all the surfaces, except for cement floor 2, where the beryllium concentrations were lower before cleaning (F1 = 0.4; 0.65; 0.79; 0.31 µg/100 cm<sup>2</sup>) than after cleaning with the solutions (F2 = 1.1; 2.3; 1.8; 0.41 µg/100 cm<sup>2</sup>).

#### 5.2 Surface sampling techniques

This section presents the laboratory and field results for the three surface sampling techniques studied.

#### 5.2.1 Target value for the beryllium compound preparations

The target values obtained for the two solutions (Be acetate and BeCl<sub>2</sub>) and the two suspensions (AlBeMet and BeO) following analysis by ICP-MS are presented in Table 4. The target value in the preparation of the solutions and suspensions was  $0.1 \mu g$ .

Beryllium compounds	Preparation	Target value (µg)
Beryllium acetate	Solution diluted in water	0,1
BeCl <sub>2</sub>	Solution diluted in water	0,1
AlBeMet	Suspension in isopropanol	0,033
BeO	Suspension in isopropanol	0,09

Table 4:	Target	<b>Be value</b>	in the	prepared	l solutions	or sus	pensions	of the	Be compounds
				F . F					

Due to a low result obtained with the beryllium and aluminum compound (AlBeMet), a new suspension was prepared under the same conditions, and a quantity of 0.041  $\mu$ g was obtained. The first suspension (result presented in Table 4) was analyzed again after several weeks, and a quantity of 0.037  $\mu$ g was then found. The results, while reproducible, show the difficulty obtaining a suspension of specific concentration for this compound.

#### 5.2.2 Wipe sampling with moistened wipes

Table 5 presents the average of the recovery rates calculated for the three samples collected in Petri dishes with the moistened wipe (performed by the same person) based on the surface, compound, and beryllium concentration. The beryllium recovery rate varies with the type of surface, being higher for the aluminum Petri dishes. The coefficients of variation (CV) vary from 1.5 to 45.7%. In general, beryllium acetate was the compound most effectively recovered during the sampling, while the AlBeMet mixture had the lowest recovery rates and highest coefficients of variation (CV).

S. C	µg of	Beryllium acetate		BeCl <sub>2</sub>		AlBeMet		BeO	
Surface	Be	% Recovery	CV (%)	% Recovery	CV (%)	% Recovery	CV (%)	% Recovery	CV (%)
	0,1	73	6,2	73	7,9	52	45,7	81	7,9
Plastic	0,2	86	7,6	57	5,1	43	24	63	18,4
	0,4	73	12,4	48 68	7,9 6,4	49	16,3	67	4,2
Glass	0,4	74	5,1	60	26	74	29,1	71	2,2
Aluminum	0,4	96	1,5	97	1,5	45	5,6	79	7,3

 Table 5: Average of the recovery rates for the moistened wipe samples (same operator)

The inter-individual evaluation was done using the results of the principal operator (operator 1) with the results of a variable operator (operator 2). Table 6 presents the average of the recovery rates for the three moistened wipe samples for the different surfaces and beryllium compounds, in relation to the operator doing the sampling. The operator involved redid the two results with a statistically significant difference in order to obtain a second result, thus eliminating the statistical difference. Except for these two results, the ratio of the recovery rate of operator 1 (Op1) and that of operator 2 (Op2) fluctuates around 1.

			Beryllium acetate		BeCl <sub>2</sub>		AlBeMet		)
Surface	Operator	Recovery (%)	CV (%)	Recovery (%)	CV (%)	Recovery (%)	CV (%)	Recovery (%)	CV (%)
Plastic	1	73	12,4	48 68	7,9 6,4	49	16,3	67	4,2
	2	82	8,8	78	7,4	52	32,2	62	6,8
Ratio (O	Ratio (Op1/Op2)		)	0,6 /	0,9	1,0		1,1	
	1	74	5,1	60	26	74	29,1	71	2,2
Glass	2	75	14,5	14 68	96,8 6,9	59	28,1	77	4,2
Ratio (O	p1/Op2)	1,0	)	4,3 /	0,9	1,3	3	0,9	)
A 1,	1	96	1,5	97	1,5	45	5,6	80	7,3
Aluininum	2	96	1,5	97	1,5	46	19,2	56	13,2
Ratio (Op1/Op2)		1,0	)	1,0	)	1,0	)	1,4	ļ

Table 6: Inter-individual difference presented with the average of the recovery rates for
moistened wipe samples for the two operators

# 5.2.3 Vacuuming (micro-vacuum)

The average of the beryllium recovery rates with the vacuum technique for each compound and each concentration is presented in Table 7. A single person collected the vacuum samples only in plastic Petri dishes. The recovery rate is low for the four beryllium compounds (between 0.13 and 12.4%), with CVs varying from 18.7 to 125.8%.

	Beryllium acetate		BeCl <sub>2</sub>		AlBe	eMet	BeO	
µg of Be	Recovery (%)	CV (%)	Recovery (%)	CV (%)	Recovery (%)	CV (%)	Recovery (%)	CV (%)
0,1	1,9	125,8	2,1	53,7	4,8	75,9	2,5	31,2
0,2	4,5	78,6	3,0	83,0	3,9	72,2	1,5	37,1
0,4	0,13	48,1	3,0	65,4	12,4	125,4	1,8	18,7

 Table 7: Average of the recovery rates by vacuuming in the Petri dishes

# 5.2.4 ChemTest® (colorimetry)

The results with the ChemTest<sup>®</sup> wipes, after an analysis carried out according to the manufacturer's instructions, are presented in Figure 13. Note that a slight blue colour starts to appear at 0.1  $\mu$ g of beryllium (the manufacturer mentioned a lower limit of 0.2  $\mu$ g), and the colour increases when the quantity of beryllium increases for three of the beryllium compounds studied. Note that the test does not detect the presence of beryllium oxide, under the utilization conditions.

The waiting time between the use of the three developing solutions to dry the wipes was longer than the time indicated by the manufacturer (more than 3 hours, rather than 2 hours), even though the manipulations were done in a location promoting rapid drying (laboratory hood).



Figure 13: ChemTest<sup>®</sup> wipe samples after analysis

# 5.2.5 Field evaluation of the sampling techniques

The results for the three surface sampling techniques in the three enterprises are presented in Appendix D. The samples collected with the moistened wipes gave a better Be recovery rate than the vacuum sampling, as did the laboratory results. However, the relationship between the beryllium concentrations for these two sampling techniques was close to 1 on the surfaces with a large amount of deposited dust.

The ChemTest<sup>®</sup> colorimetric technique did not demonstrate good effectiveness on the different surfaces sampled in the field. A large quantity of dust or the presence of oil on the sampled surfaces obstructs the surface of the wipe and prevent its coloration. The positive results (blue coloration) observed with the ChemTest<sup>®</sup> were not always corroborated by the two other techniques (for example, Appendix D, Table D2, enterprise 6, numbers 4 and 6).

# 5.3 Cleaning and decontamination techniques

The Be concentrations obtained from the air samples (if applicable) and surface samples are presented for the enterprises visited within the context of the cleaning work.

# 5.3.1 Enterprise 1

#### 5.3.1.1 Surface contamination

In the machining shop where the CuBe moulds were made, repaired and stored, beryllium was determined to be present during a preliminary visit by means of surface sampling of the walls, tools, moulds and machines. The maximum concentration found was 463  $\mu$ g/100 cm<sup>2</sup> on the protective panel of a lathe used for machining moulds (Table 8).

During decontamination in the shop, the results in the different work steps show the effectiveness of the cleaning methods used. In fact, after the final decontamination step, the results were below the MRV on the different surfaces.

Period	Number of samples	Average (µg Be/100 cm <sup>2</sup> )	Median (µg Be/100 cm²)	(min max.) (µg Be/100 cm²)
Preliminary visit	5	100,3	3,3	(0,57 - 463)
Before vacuuming	6	5,2	1	(0,34 - 18)
After vacuuming	7	1,8	0,24	(0,11 - 9,9)
After wet cleaning	4	< MRV	< MRV	All < MRV

 Table 8: Be concentrations in surface samples during the work (enterprise 1)

MRV: minimum reported value (0.05 µg)

#### 5.3.1.2 Air samples

The air samples were collected by means of different sampling devices in the workers' breathing zones during vacuuming of debris and dust (Table 9). The beryllium concentrations were all below  $0.15 \,\mu$ g/m<sup>3</sup> (TWAEV applicable for total dusts).

 Table 9: Air samples in the workers' breathing zones during the work (enterprise 1)

<b>D</b>	Concentration				
Fraction	dusts (mg/m <sup>3</sup> )	beryllium (µg/m³)			
	<mrv< td=""><td><mrv< td=""></mrv<></td></mrv<>	<mrv< td=""></mrv<>			
Respirable	0,27	0,0051			
	<mrv< td=""><td>0,0073</td></mrv<>	0,0073			
lotar	<mrv< td=""><td>0,0020</td></mrv<>	0,0020			
T 1 1 1	1,3	0,13			
Inhalable	0,42	0,0065			
Inhalable and respirable	<mrv< td=""><td><mrv< td=""></mrv<></td></mrv<>	<mrv< td=""></mrv<>			
(Sierra <sup>®</sup> )	1,2	0,014			

MRV: Minimum reported value (0.0005 µg/sample)

# 5.3.2 Enterprise 2

#### 5.3.2.1 Surface contamination

For this enterprise, some surfaces had beryllium concentrations above the threshold value of  $0.2 \,\mu g/100 \,\text{cm}^2$ , even after vacuuming followed by wet cleaning with solvent (Table 10). The application of a sealant on the concrete floor surfaces finally reduced the accessible beryllium contamination below the reference value.

Period	Number of samples	Average (µg Be/100 cm <sup>2</sup> )	Median (µg Be/100 cm²)	(min max.) (µg Be/100 cm²)
Before vacuuming	8	8,1	7,3	(1,4 - 16)
After vacuuming	8	2,8	2,1	(< MRV - 7.4)
After wet cleaning	8	0,22	0,2	(< MRV - 0.71)
Several days later	21	0,17	0,08	(< MRV - 1.5)
After sealant	2	< MRV		All < MRV

 Table 10: Be concentrations in surface samples during the work (enterprise 2)

MRV: Minimum reported value (0.05 µg)

#### 5.3.2.2 Air samples

Ambient air samples were collected close to the worker (sampling devices generally attached to the lift, Figure 14) during the vacuuming of dust and debris as well as during the wet cleaning. The beryllium concentrations in these samples were all below the limit value of  $0.15 \,\mu$ g/m<sup>3</sup>.



Figure 14: Ambient air sampling with sampling devices attached to the lift

Breathing zone samples were collected only during the vacuuming of dust and debris. Table 11 presents the results, with some concentrations exceeding the TWAEV. Note that the inhalable dust samples (IOM<sup>®</sup>) and "total" dust samples (37-mm cassette) were not collected on the same day.

Collector	Number of samples	Average (µg Be/m <sup>3</sup> )	Median (µg Be/m³)	(min max.) (µg Be/m <sup>3</sup> )
IOM®	4	0,23	0,22	(0,11 - 0,39)
Cassette, 37-mm	6	0,45	0,13	(0,024 - 1,7)

 Table 11: Air samples in the breathing zone during vacuum cleaning work (enterprise 2)

#### 5.3.3 Enterprise 3

This establishment did the wipe sampling and vacuum sampling (micro-vacuum) by itself at the end of the decontamination work on different types of surfaces (painted cement, wood, metal, fabric). The results, generally below  $0.2 \ \mu g/100 \ cm^2$ , demonstrated that the cleaning techniques used were effective. Surface sample results above the reference value were explained by a forgotten cleaning or by surfaces that were more difficult to clean, such as fabric on chairs.

### 5.3.4 Enterprise 4

Surface samples were collected from two moulds (1 and 2), similar to the one in Figure 2, before and after their cleaning, in order to verify the variation in surface contamination (Table 12). The results show that the value of  $0.2 \ \mu g/100 \ cm^2$  is exceeded on the CuBe surfaces, even after wet cleaning with solvent. The presence of beryllium on the aluminum surface, in higher concentrations after the cleaning than before it, suggests the migration of beryllium particles.

# Table 12: Beryllium concentration in the moistened wipe samples before and after cleaning of two moulds in industry

		Beryllium concentration (µg/100 cm <sup>2</sup> )					
Mould	Surface	Sampling in February	Sampling in June (just before cleaning)	Sampling in June (just after cleaning)			
1	CuBe	3,6	3,2	2,0			
1	Al	NA	< MRV	0,10			
2	CuBe	0,70	3,2	1,4			
2	Al	NA	0,20	0,24			

NA: Not applicable MRV: Minimum reported value (0.05 µg)

#### 6. **DISCUSSION**

The discussion is presented separately for each of the studies carried out in the context of this project: evaluation of the cleaning solutions (industrial and commercial), evaluation of three surface sampling techniques, and evaluation of the cleaning and decontamination techniques.

# 6.1 Cleaning solutions

# 6.1.1 CuBe surface

The objective of the cleaning solution study was mainly to answer the two following questions:

- Can the concentration of free beryllium at the surface of a metal part made of a copperberyllium alloy (2% Be) be reduced to a level below the criterion of  $0.2 \,\mu g/100 \,\text{cm}^2$ ?
- Are there more effective cleaning solutions for loosening the beryllium from the surface of these metal parts?

Citranox<sup>®</sup>, with its acid character, was shown to be the most effective for recovering beryllium from the surface of moulds and plates. However, the residual contamination following cleaning with this product was greater than with the other products, and generally well above the reference value of 0.2  $\mu$ g/100 cm<sup>2</sup> (for moulds and plates). Therefore, while the acid product cleans the beryllium from the surface better, there is always beryllium present at the surface.

Nakao *et al.* (1979) demonstrated that beryllium oxide (BeO) at 10% (w/w) or more formed at the surface of a metal made of CuBe alloy (2% Be), by producing rather homogeneous thin layers. The Be concentration was 0.2% (w/w) at the surface and 1.9% at a depth of 300 nm. In another study (*Belkiat, 1998*), a thin layer of BeO was seen to form at the surface when a metal part made of CuBe alloy (4% Be) was heated. This suggests a migration of Be towards the surface as well as its oxidation.

Therefore, when a surface is cleaned with Citranox<sup>®</sup>, the latter's acid character eliminates layers made of BeO, as shown in Figure 12. After the cleaning, thin layers of beryllium oxide would form again at the surface. The use of an acid product such as Citranox<sup>®</sup> to clean parts made of CuBe is therefore not recommended, either on a small or large scale, considering the quantity of beryllium generated during the process, which could increase the risk of skin exposure.

Also, the observations reported in the two mentioned studies could explain the difference in the amount of beryllium collected by wipe sampling for the new plates (no apparent oxidation) and the moulds (apparent oxidation). Hence, an oxidized surface would be more likely to release beryllium (with wiping). Therefore, keeping the contamination at a level below 0.2  $\mu$ g/100 cm<sup>2</sup> on the surface of a material made of CuBe alloy seems difficult.

In an area where beryllium will continue to be used, the company must ensure regular monitoring of the contamination level and establish a housekeeping program, associated with exposure control measures and the wearing of protective equipment for workers, such as:

- storage of beryllium-containing parts in a closed location, with a sign at the entrance indicating the presence of beryllium on the premises and the protective equipment required;
- wrapping of the part when it is moved;
- enclosure around the part when it is used in a process;
- label on the part (or its packaging) mentioning the exposure risks and the required protective equipment;
- skin protection when these parts are handled (gloves, long sleeves, etc.);
- respiratory protection recommended in the *Guide de protection respiratoire (CSST, 2002)*;
- training and informing workers.

To limit the accumulation of dusts and their propagation towards other areas, surface contamination must be below  $3.0 \ \mu g/100 \ cm^2$ . To clean contaminated parts or areas, the use of a neutral product (Luminox<sup>®</sup>) or alkaline product (Alconox<sup>®</sup>, Fantastik<sup>®</sup> and Z-99<sup>®</sup>) would then be suggested, due to their less aggressive character. These products have demonstrated their capacity to recover beryllium from the surface while leaving a residual contamination that is generally below  $3.0 \ \mu g/100 \ cm^2$ .

### 6.1.2 Recovery rate

The tests carried out in Petri dishes evaluated the capacity of moistened wipes, soaked in different cleaning solutions, to recover beryllium (in the form of three different compounds) from a smooth surface (polystyrene).

In the evaluation of IRSST analytical method 359, the incomplete solubilization of beryllium oxide (85%) was revealed (IRSST, 2008b). Therefore, the recovery rate obtained with BeO for this experiment compares well with the results obtained with beryllium acetate and beryllium chloride.

For the recovery of beryllium from the surface of a material that did not contain beryllium, the different solutions were similar in effectiveness, compared to the tests on CuBe surfaces where the acid solution recovered much more beryllium than the other solutions. This was demonstrated during the tests in the Petri dishes as well as on the different surfaces evaluated in enterprises.

# 6.1.3 Field evaluation

In the workplace, cement floor 2 (enterprise 2) was the only surface where the Be concentrations were higher after three successive cleanings than before the cleaning. The material's porosity as well as the surface's friability and wear could be important factors in the migration of beryllium towards the surface; wood did not demonstrate the same behaviour. These characteristics make the surface difficult to sample with the wipe method, which may have affected the reliability of the results. Cleaning would therefore have increased the beryllium concentration at the surface.

This effect has already been observed for lead-contaminated surfaces. Consequently, a second vacuuming would be more effective on surfaces that are difficult to decontaminate and would

allow the particles of lead loosened during wet cleaning to be recovered (*Grinshpun*, 2002; Yiin, 2004). The US HUD (1995) proposed a three-step cleaning cycle: (1) vacuuming of debris and dust, (2) wet washing with detergent, and (3) vacuuming of dust.

# 6.2 Surface sampling techniques

## 6.2.1 Surface wipe sampling

The method of surface wipe sampling using moistened wipes has already been studied to evaluate lead contamination (*Lanphear*, 1995; *Reynolds*, 1997) and also beryllium contamination (*Dufay*, 2006; Ashley, 2005).

The study by Dufay was carried out in order to compare wet and dry sampling methods in relation to different collecting media including Ghost Wipes<sup>®</sup>, for concentrations of soluble beryllium (0.003 to 3  $\mu$ g), on a surface of non-porous glass. The results indicate a large difference between the different types of collecting media. Ghost Wipes<sup>®</sup> showed an average recovery rate of 85.9% (CV of 7.1%).

The results of the present study with beryllium acetate (soluble compound) showed recovery rates with Ghost Wipes<sup>®</sup> in the order of 73% (CV of 12.4%) to 96% (CV of 1.5%) for the three types of surfaces (plastic, glass and aluminum). These results are therefore comparable to those of Dufay. However, with beryllium chloride, BeCl<sub>2</sub>, (another soluble compound), the results are more variable, with a recovery rate varying from 48 to 97% (CV: 1.5 to 26%).

The recovery rates observed with the aluminum and beryllium compound (AlBeMet) are lower (between 43 and 74%) with higher CVs than for the other compounds, namely between 5.6% (aluminum surface) and 45.7% (plastic surface). To calculate the concentration of Be in suspension in isopropanol, the concentration ratios submitted by the product's supplier (62% Be and 38% aluminum) were used without any other certification. It is therefore difficult to state that the amount of product taken from the bottle for weighing is representative of the entire mixture. As a result, the heterogeneity of the product used or the heterogeneity of the suspension prepared for the laboratory tests could explain why such results were obtained.

The sampling done in the BeO-enriched Petri dishes showed recovery rates varying from 63 to 81% (CV of 7.9 to 18.4%), comparable to those observed with soluble compounds (Be acetate and BeCl<sub>2</sub>). Note, however, the possible underestimation of beryllium in this compound, due to the difficulty dissolving it completely under these analytical conditions.

The tests on the smooth aluminum surface gave better recovery rates, except for the AlBeMet compound, with CVs in the order of 1.5 to 13.2%, compared to the other surfaces.

Studies published on the evaluation of the surface sampling technique using moistened wipes also showed recovery rates below 100% (*Dufay, 2006; Lichtenwalner, 1992; Reynolds, 1997*). However, these recovery rates can be indicators of the effectiveness of the sampling, more particularly with Ghost Wipes<sup>®</sup>.

For the evaluation of inter-individual variation, two average recovery rates of the two operators showed a statistically significant difference (95% confidence interval and p-value below 0.05). These results were redone by the operator involved, thus eliminating this difference. This shows that there can be an intra-individual difference and that the surface wipe remains a sampling technique in which the human factor has an impact on the result. The ratios of the average recovery rate of operator 1 and operator 2 are generally close to 1, showing a good reproducibility for the method. However, the fact that the two operators come from the same laboratory and have similar training must be taken into account. The results could be very different if they came from two different laboratories (inter-laboratory variation).

# 6.2.2 Vacuuming (micro-vacuum)

Some surfaces are more difficult to evaluate by surface wipe sampling, and thus the interest in using the vacuum sampling technique. This technique was mainly studied during its use on carpets in lead-contaminated homes (*Bai, 2003; Reynolds, 1997; Farfel, 1994; Lanphear, 1995*). Creek discusses the possibility of using the "micro-vacuum" technique for surface sampling of Be particles and concludes that the method, as described in ASTM 7144, should be improved in order to obtain a better recovery efficiency (*Creek, 2006*). For the present study, the pump flow was adjusted to 16 L/min (instead of 2.5 L/min), as in the studies on lead carried out by the U.S. Department of Housing and Urban Development (*US HUD, 1995*) and that of Farfel *et al. (1994*).

The laboratory results obtained have very low recovery rates (between 0.13 and 12%) and high CVs (between 18.7 and 125.8%). This may be due to the methodology used. In fact, the beryllium particles are first dissolved or suspended in a liquid and then returned to the solid form following evaporation of the liquid. The morphology of the particles and their adherence to the surface would then be different from that of deposited particles, which are generally found in companies.

### 6.2.3 ChemTest®

The reliability of the semi-quantitative surface sampling technique using colorimetry (ChemTest<sup>®</sup>) was verified in the laboratory with four beryllium compounds. The tests on the two soluble compounds (Be acetate and BeCl<sub>2</sub>) responded positively. Also, blue coloration was observed starting at a concentration of 0.1  $\mu$ g of Be, instead of 0.2  $\mu$ g, as indicated by the supplier. Therefore, as soon as a slight blue colour appeared, which would indicate a concentration close to the reference value, surface wipe sampling would be recommended with a moistened wipe in order to confirm the presence of Be.

In the case of the aluminum and beryllium compound (AlBeMet), the ChemTest<sup>®</sup> reacted in a similar way as for the soluble compounds, except that the blue colour appeared as distinct spots. This could be due to the insolubility of AlBeMet and the heterogeneity of the deposit in the Petri dish.

According to our results, the ChemTest<sup>®</sup> sampling and analytical technique, carried out according to the supplier's instructions, does not react to the presence of beryllium oxide.

A study by Taylor (2002) discusses the validation of beryllium detection by colorimetry by using a method similar to the ChemTest<sup>®</sup>. The authors explain the difficulties encountered in dissolving BeO. They conclude that the use of a 10% sulfuric acid solution ( $H_2SO_4$ ) would be appropriate for validating the method with BeO, when the wipe and the surface are in contact for a period of 15 minutes. The solubilization solution for the ChemTest<sup>®</sup> is in fact 10%  $H_2SO_4$ , but the user manual does not mention favouring 15-minute contact with the surface. Other tests could be carried out to verify whether longer contact would solve the problem.

Tekleab *et al.* (2006) from the Microteq company (supplier of the ChemTest<sup>®</sup>) published an article mentioning that there was no interference with their sampling and analytical method by colorimetry for beryllium quantification. The validation report on the company's Web site does not mention difficulty detecting BeO.

The unavailability of a reference material, for evaluation of the digestion and extraction protocol with BeO, is discussed by the scientific community (*Brisson, 2006*). Such a material would validate the different analytical methods used in beryllium determination.

The ChemTest<sup>®</sup> kit for beryllium determination costs \$350 for 20 wipes (\$17.50 per wipe) and requires a waiting time of approximately 3 hours to obtain the results. Laboratory analysis of a moistened wipe (Ghost Wipe<sup>®</sup>) costs approximately \$50, with a waiting time of as long as a few days.

The reliability of the ChemTest<sup>®</sup> remains to be determined, particularly for BeO. The absence of coloration therefore does not indicate that the Be concentration is below 0.2  $\mu$ g. The presence of BeO must be confirmed by laboratory analysis unless the compound's precise composition is known. Also, elemental beryllium, which can oxidize to BeO on contact with air, may be present in several work environments.

# 6.2.4 Field evaluation

The laboratory evaluation involved some limitations, as compared to the reality in enterprises: the dust was not deposited dust, the surface was smooth and homogeneous, the surface area was limited, beryllium was the only contaminant present, etc. Field tests were therefore carried out with the three surface sampling methods. In general, the results for the samples collected in the enterprises (3, 6 and 7) confirm what was observed in the laboratory, namely a better Be recovery with the moistened wipes than with the vacuum technique. However, the ratio of the concentration obtained for wipes and for vacuuming is close to 1 when a lot of dust is present. The surface (smooth or porous) as well as its condition (condition of the paint, presence of oil, dust accumulation, etc.) would explain the difference in beryllium concentrations obtained with the two types of sampling.

Our results are consistent with what was observed in other studies (*Farfel, 1994; Reynolds, 1997; Ashley, 2007*). Note that the "micro-vacuum" technique was mainly used to estimate carpet contamination (*Reynolds, 1997; Bai, 2003*). Vacuuming techniques should be standardized in order to improve their reliability and precision, and can be an alternative of choice when the use of wipes is impractical (*Ashley, 2005; Ashley, 2007; Farfel, 1994; Byrne, 2000*).

The colorimetric technique with the ChemTest<sup>®</sup> did not give as good results as the laboratory tests. In fact, a large amount of dust on the sampled surfaces or the presence of oil on the surface of the wipe would alter the different chemical reactions that result in wipe coloration. Finally, the reliability of the results with the ChemTest<sup>®</sup> technique is affected by the condition of the surface. The ChemTest<sup>®</sup> should therefore be used mainly in areas assumed to be "clean" in order to confirm the absence of Be.

### 6.3 Cleaning and decontamination techniques

### 6.3.1 Effectiveness of the cleaning techniques

The cleaning procedure generally used during decontamination consists of vacuuming debris and dust, followed by wet cleaning with the addition of detergent. For enterprise 1, this two-step cleaning cycle resulted in effective decontamination of the premises (Be concentrations below  $0.2 \,\mu g/100 \,\mathrm{cm^2}$ ) (Table 8). Note that the four samples collected after wet cleaning were not necessarily collected from the surfaces that showed significant contamination in the previous step (after vacuuming).

Enterprise 2, a much larger location, had larger scale beryllium contamination. The decontamination work began with cleaning and moving of equipment and machines to another location. Vacuuming of debris and dust was then done on the horizontal surfaces, followed by wet cleaning with detergent using high-pressure jets. The samples collected after wet cleaning gave concentrations varying from < MRV to 0.71 µg/100 cm<sup>2</sup> and up to 1.5 µg/100 cm<sup>2</sup> a few days after the cleaning (Table 10). Some of the results above 0.2 µg/100 cm<sup>2</sup> come from samples collected on vertical surfaces such as walls. We can assume that in this specific case, wet cleaning with high-pressure jets on a surface with a significant amount of dust, if it hadn't been preceded by vacuuming, was not effective. For cement floor 2, which was friable and worn, it was difficult to achieve a beryllium concentration below 0.2 µg/100 cm<sup>2</sup>. In fact, even after repeating the cleaning cycle, the concentrations were still above 0.2 µg/100 cm<sup>2</sup>. Application of a sealant was the remedy chosen by the company to confine the contamination. The results of wipe sampling performed after application of this barrier clearly show that the contamination was no longer present at the surface. However, sealant degradation must be monitored in order to ensure the ongoing effectiveness of this barrier. As well, if structural modification work is begun, workers must be informed about the presence of this contamination in the material in order to apply the appropriate prevention measures.

In enterprise 3, the cleaning methods used were effective on all the types of surfaces, except for porous surfaces, which required a second cleaning cycle, according to the contractor responsible for decontamination.

According to the report published on *Opération béryllium*. Activités dans les établissements du secteur de l'environnement (Operation beryllium in establishments in the environmental sector), enterprises generally use vacuuming or wet cleaning (*CSST*, 2006). Also, it seems that the air jet is still used for periodic maintenance, even though this practice is known to only redistribute the contamination.

Finally, the decontamination procedure to be used can vary with the type of surface to be cleaned. In fact, a second cleaning cycle is recommended when the surface contamination remains above 0.2  $\mu$ g/100 cm<sup>2</sup> after the first cycle. With lead contamination, specialists suggest a second vacuuming when the surface is difficult to decontaminate (*Yiin, 2004; US HUD, 1995*) and a second wet cleaning for porous surfaces such as wood (*Grinshpun, 2002*).

### 6.3.2 Worker exposure to beryllium

Beryllium exposure was checked by air samples collected in the workers' breathing zones only during vacuuming of debris and dust in two enterprises. In enterprise 1, all the results were below  $0.15 \ \mu g/m^3$ , while in enterprise 2, a larger site where the contamination was greater, the average concentrations were  $0.45 \ \mu g/m^3$  in total dust, and  $0.23 \ \mu g/m^3$  in inhalable dust. However, the stationary samples collected were all below the TWAEV. Despite the small number of samples, the results show that exposure can vary with the decontamination steps, the work techniques, the surface to be decontaminated, and the initial surface contamination.

Several studies mention that maintenance or decontamination workers on premises where Be is present have beryllium disease or are sensitized (*Kreiss, 1997; Henneberger, 2001; Stange, 2001; Deubner, 2001; Kelleher, 2001; Madl, 2007*). These workers must therefore wear personal protective equipment (skin and respiratory). Other preventive measures are also required to prevent the propagation of dust towards the outside or the contamination of adjacent areas. These measures could include two change rooms for the workers, and closing off the areas for decontamination and having them under negative pressure.

### 7. CONCLUSION

The laboratory recovery tests, as well as the laboratory and field decontamination tests, show that the surface on which the beryllium dusts are deposited, the beryllium compound, the cleaning solution, the sampling technique, and the operator are important determining factors in obtaining a surface contamination level below the reference values.

Tools and equipment made of Be alloy must be considered as potential sources of skin exposure and their use should be guided by appropriate preventive measures. A component containing beryllium, whose surface had been decontaminated, would always retain its potential to release Be. Therefore, following cleaning with an acid detergent such as Citranox<sup>®</sup>, the surface undergoes a chemical process (oxidation) and fine layers of BeO would form there again. The use of an acid detergent is therefore not recommended, either on a small or large scale, due to the amount of beryllium generated during the process, which could increase the risk of skin exposure.

Despite the inter-individual variability of the wipe sampling method with moistened wipes, this method remains the most appropriate technique for estimating surface contamination. The use of vacuuming (micro-vacuum) can be considered for porous surfaces where the wipe technique is unsuitable. However, the colorimetric method (ChemTest<sup>®</sup>) must be used with caution. In our opinion, other laboratory and field studies must be carried out to validate the results of this technique.

The cleaning cycle that combines the two methods (vacuuming followed by wet cleaning with detergent) is an effective procedure for relatively smooth and homogeneous surfaces. Following the cleaning cycle, surface samples must be collected on all types of surfaces in order to verify the effectiveness of this procedure. Another cleaning cycle may be necessary for some more porous or damaged surfaces. The application of a sealant must be considered only when the cleaning procedure is ineffective, and the sealant's effectiveness must be monitored over time.

#### 8. **RECOMMENDATIONS**

Based on the results of this study, the recommendations are to:

- Combine the two surface cleaning methods: vacuuming (HEPA filters) followed by wet cleaning;
- Carry out a second cleaning cycle (vacuuming and wet cleaning) when the contamination is  $> 0.2 \mu g/100 \text{ cm}^2$  after the first cycle;
- Use a sealant only if surface decontamination proves ineffective, and monitor its effectiveness;
- Avoid using an acid solution for cleaning surfaces made of copper-beryllium in order to limit oxidation and to avoid the increased generation of beryllium at the surface;
- Implement a complete prevention program in order to reduce the workers' exposure to beryllium, since the decontamination of parts made of copper-beryllium seems impossible;
- Collect surface samples from all types of surfaces in order to verify the effectiveness of the decontamination;
- Use the surface-sampling vacuum technique when moistened wipe sampling is inadequate;
- Know the limits of the ChemTest<sup>®</sup> colorimetric technique before using it:
  - It does not react to the presence of beryllium oxide (BeO);
  - It is ineffective when a lot of dust or oil is present;
  - It can be used to verify the effectiveness of the cleaning when BeO is not present.

The general recommendations are to:

- Wear respiratory protective equipment as well as skin protection during cleaning and decontamination work;
- Provide workers with two change rooms, and close off the area where the work will be done and have it under negative pressure in order to avoid the contamination of areas adjacent to the work area.

For future studies:

- Do not use the beryllium and aluminum dust (AlBeMet) mixture as a reference material for validating a sampling or analytical method;
- Verify whether a longer contact time between the ChemTest<sup>®</sup> wipe and the surface will detect beryllium oxide;
- For the ChemTest<sup>®</sup>, verify whether the use of a solution more acidic than 10% H<sub>2</sub>SO<sub>4</sub> would help solubilize BeO without hindering the chain of chemical reactions leading to wipe coloration.

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# APPENDIX A: PREPARATION OF BERYLLIUM COMPOUNDS (TESTS ON CLEANING SOLUTIONS)

#### **Beryllium** acetate

A beryllium solution at 200  $\mu$ g/L was prepared by diluting a commercial solution of beryllium acetate containing 10,000 mg/L of Be (SPEX CertiPrep<sup>®</sup>Group, Lot# W10-115Be) with 1% nitric acid. A summary of the procedure is presented in Table A1. In order to verify the mass of beryllium in the final solution, three tests were carried out directly in digestion beakers and were analyzed by ICP-MS. The average amount of Be was 0.203  $\mu$ g with a coefficient of variation (CV) of 2.8%.

	Initial solution	Step 1	Step 2	Final solution
Concentration in the solution		10 000 mg/L	20 mg/L	200 µg/L
Volume sampled		200 µL	1 000 µL	1 000 µL
Dilution in		100 mL HNO <sub>3</sub> 1%	100 mL HNO <sub>3</sub> 1%	
Resulting concentration	10 000 mg/L	20 mg/L	200 µg/L	
Quantity of Be		2 mg	20 µg	0,2 µg

#### Table A1: Preparation of the beryllium acetate solution

#### Beryllium chloride (BeCl<sub>2</sub>)

The conversion factor used for BeCl<sub>2</sub>, based on the ratio of the atomic weight of Be (9 g/mol) and the molar mass of beryllium chloride (79.9 g/mol), was 0.1126. The solution was prepared by using BeCl<sub>2</sub> powder (supplied by Fluka), which was dissolved in deionized water, to obtain a final mass of beryllium of 0.2  $\mu$ g. A summary of the steps is presented in Table A2. The average mass of Be obtained, following verification of the mass of Be in the final solution by ICP-MS, was 0.204  $\mu$ g (CV of 0.6%).

Table A2: Preparation of the BeCl <sub>2</sub> solution
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	Initial solution	Step 1	Step 2	Final solution
Mass of BeCl <sub>2</sub>	182.2 mg powder	182.2 mg powder		
Concentration in the solution			20.52 mg/L Be	205.2 µg/L Be
Volume sampled			1,000 µL	1,000 µL
Dilution in	1,000 mL water	1,000 mL water	1,000 mL water	
Resulting concentration		182.2 mg/L BeCl <sub>2</sub> 20.52 mg/L Be	205.2 µg/L Be	
Quantity of Be		20.52 mg	20.52 µg	0.2 μg

#### Beryllium oxide (BeO)

The conversion factor for BeO, based on the ratio of the atomic weight of Be (9 g/mol) and the molar mass of beryllium oxide (25 g/mol), was 0.36. The suspension was prepared by using BeO powder (from Fluka) and isopropanol in order to obtain a mass of Be of 0.38  $\mu$ g. A summary of the steps is presented in Table A3. The average mass of Be obtained following verification of the mass of Be in the final solution by ICP-MS was 0.387  $\mu$ g, with a CV of 0.3%.

	-		•	
	Initial solution	Step 1	Step 2	Final solution
Mass of BeO	141.8 mg	141.8 mg		
Concentration in the solution			51.05 mg/L Be	255.25 μg/L Be
Volume sampled			5,000 μL	1,500 µL
Suspension in	1,000 mL isopropanol	1,000 mL isopropanol	1,000 mL isopropanol	10 mL HNO <sub>3</sub> (1%)
Resulting concentration		141.8 mg/L BeO 51.05 mg/L Be	255.25 μg/L Be	
Quantity of Be		51.05 mg	255.25 μg	0.387 µg

#### **Table A3: Preparation of the BeO suspension**

## APPENDIX B: PREPARATION OF BERYLLIUM COMPOUNDS (TESTS ON SAMPLING TECHNIQUES)

#### **Beryllium acetate**

A beryllium solution at 100  $\mu$ g/L was prepared by diluting a commercial solution of beryllium acetate containing 10,000 mg/L of Be (SPEX CertiPrep®Group, Lot# W10-115Be) with 1% nitric acid. A summary of the procedure is presented in Table B1. In order to verify the mass of beryllium in the final solution, three tests were carried out directly in digestion beakers and then analyzed by ICP-MS. The average mass of Be obtained was 0.10  $\mu$ g with a coefficient of variation (CV) of 0.99%.

	Initial solution	Step 1	Step 2	<b>Final solution</b>
Concentration in the solution		10,000 mg/L Be	100 mg/L Be	100 µg/L Be
Volume sampled		1 mL	0,1 mL	1 mL
Dilution in		100 mL HNO <sub>3</sub> 1%	100 mL HNO <sub>3</sub> 1%	
Resulting concentration	10,000 mg/L	100 mg/L Be	100 µg/L	
Quantity of Be		10 mg	0.01 mg	0.1 µg

 Table B1: Preparation of the beryllium acetate solution

#### **Beryllium chloride (BeCl<sub>2</sub>)**

The conversion factor used for BeCl<sub>2</sub>, based on the ratio of the atomic weight of Be (9 g/mol) and the molar mass of beryllium chloride (79.9 g/mol), was 0.1126. The solution was prepared by using BeCl<sub>2</sub> powder (supplied by Fluka), which was diluted in deionized water to obtain a beryllium concentration of 100  $\mu$ g/L. A summary of the steps is presented in Table B2. The average mass of Be, obtained following verification of the final solution by ICP-MS, was 0.10  $\mu$ g (CV of 0.96%).

	Initial solution	Step 1	Step 2	Final solution
Mass of BeCl <sub>2</sub>	92.5 mg powder	92.5 mg powder		
Concentration in the solution			20.83 mg/L Be	104 µg/L Be
Volume sampled			1 mL	1 mL
<b>Dilution in</b>	500 mL water	500 mL water	200 mL water	
Resulting concentration		185 mg/L BeCl <sub>2</sub> 20.83 mg/L Be	104 µg/L Be	
Quantity of Be		10.42 mg	104 µg	0.104 µg

Table B2: Preparation of the BeCl<sub>2</sub> solution

#### Beryllium oxide (BeO)

The conversion factor for BeO was 0.36, based on the ratio of the atomic weight of Be (9 g/mol) and the molar mass of beryllium oxide (25 g/mol). The suspension was prepared by using BeO powder (supplied by Fluka) and isopropanol in order to obtain a Be concentration of 100  $\mu$ g/L. A summary of the steps is presented in Table B3. The average mass of Be, obtained following verification of the mass of Be in the final solution by ICP-MS, was 0.09  $\mu$ g (CV of 0%).

	Initial solution	Step 1	Step 2	<b>Final solution</b>
Mass of BeO	132.7 mg	132.7 mg		
Concentration in the solution			95.60 mg/L Be	95.60 µg/L Be
Volume sampled			5 mL	1 mL
Suspension in	500 mL isopropanol	500 mL isopropanol	1 000 mL isopropanol	10 mL HNO <sub>3</sub> (1%)
<b>Resulting</b> concentration		265.4 mg/L BeO 95.60 mg/L Be	95.60 μg/L Be	
Quantity of Be	Quantity of Be 47.77 mg		95.60 μg	0.09 µg

#### Table B3: Preparation of the BeO suspension

#### Beryllium - aluminum (AlBeMet)

The suspension was prepared by using a mixture of beryllium dust (62%) and aluminum dust (38%), provided by the Brush Wellman company, as well as isopropanol to obtain a Be concentration of 100  $\mu$ g/L. A summary of the steps is presented in Table B4. The average mass of Be, obtained following verification of the mass of Be in the final solution by ICP-MS, was 0.033  $\mu$ g with a CV of 42%.

	Initial solution	Step 1	Step 2	Final solution
Mass of AlBeMet 87 mg		87 mg		
Concentration in the solution			107.88 mg/L Be	107.88 µg/L Be
Volume sampled			1 mL	1 mL
Suspension in	500 mL isopropanol	500 mL isopropanol	1,000 mL isopropanol	10 mL HNO <sub>3</sub> (1%)
Resulting concentration		174 mg/L AlBeMet 107.88 mg/L Be	107.88 µg/L Be	
Quantity of Be		53.94 mg 107.88 μg		0.108 µg

#### Table B4: Preparation of the AlBeMet suspension

# APPENDIX C: FIELD EVALUATION OF CLEANING SOLUTIONS

			Citranox	Alconox	Resolve	Tap water	Ledisolf	Luminox	NaCl
	Plexiglas table	F1	0.44	0.45	0.67				
	5	N1	0.61	0.85	0.79				
		N2	0.30	0.21	0.27				
		N3	0.31	0.09	0.09				
Ţ		F2	0.13	<mrv< th=""><th><mrv< th=""><th></th><th></th><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th><th></th><th></th></mrv<>				
e S	Metal table	F1	1.7	3.2	0.62				
ris		N1	1.0	1.0	0.45				
Id.		N2	0.26	0.36	0.14				
E		N3	0.15	0.18	0.07				
ż		F2	0.06	0.06	0.07				
ш	Floor	F1	3.2	2.3	2.6				
		N1	6.7	11.0	21				
		N2	6.6	9.4	11				
		N3	9.9	4.8	5.1				
		F2	2.3	0.54	1.2				
	Painted metal	F1	3.7	5		2.8	4.6		
		N1 N2	0.42	0.87		0.65	0.41		
			0.16	0.67		0.32	0.29		
		E2	0.09	0.35		0.10	0.2 TD		
	Painted metal	F1		0.07		0.12			
	i anteo metai	N1		< <u>MR</u> \/		< <u>MR</u> V			
		N2	<mrv< th=""><th><mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<>		<mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th></mrv<>		
		N3	<mrv< th=""><th><mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<>		<mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th></mrv<>		
		F2	<mrv< th=""><th><mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<>		<mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th></mrv<>		
	Textured cement wall	F1	<mrv< th=""><th><mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<>		<mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th></mrv<>		
		N1	<mrv< th=""><th><mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<>		<mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th></mrv<>		
2		N2	<mrv< th=""><th><mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<>		<mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th></mrv<>		
se		N3	<mrv< th=""><th><mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<>		<mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th></mrv<>		
ori		F2	<mrv< th=""><th><mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th><mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<></th></mrv<>		<mrv< th=""><th><mrv< th=""><th></th><th></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th></mrv<>		
lie	Cement floor 1	F1	17	18		22	31		
Jt		N1	20	11		18	26		
ш		N2	12	7		8.3	11		
		N3	6.2	4.2		7.8	16		
	Deinted concrete well	F2 F4	5.2	3.7		4.3	5.4		
	Painted concrete wait		3.1	3.3		3.5	4.5		
		N2	0.17	0.33		0.42	0.31		
		N3	0.27	0.10		0.07	0.12		
		F2	0.31	0.08		0.06	0.17		
	Cement floor 2	F1	0.4	0.65		0.79	0.31		
		N1	0.45	1.10		0.92	0.26		
		N2	0.57	1.80		1.10	0.37		
		N3	0.85	2.70		1.30	0.40		
		F2	1.1	2.3		1.8	0.41		
	rPainted concrete	F1	0.31	0.3				0.33	0.34
		N1	<mrv< th=""><th>0.05</th><th></th><th></th><th></th><th><mrv< th=""><th>0.07</th></mrv<></th></mrv<>	0.05				<mrv< th=""><th>0.07</th></mrv<>	0.07
		N2	<mrv< th=""><th><mrv< th=""><th></th><th></th><th></th><th><mrv< th=""><th><mrv< th=""></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th><th></th><th><mrv< th=""><th><mrv< th=""></mrv<></th></mrv<></th></mrv<>				<mrv< th=""><th><mrv< th=""></mrv<></th></mrv<>	<mrv< th=""></mrv<>
		N3 E2							
ŝ	Painted motal	r 2 E1							<ivit v<br="">1</ivit>
Se		N1	0.00	0.04				0.50	0.20
, i		N2	<mr\ <="" th=""><th>0.00</th><th></th><th></th><th></th><th>0.05</th><th>0.23</th></mr\>	0.00				0.05	0.23
ep		N3	<mrv< th=""><th><mrv< th=""><th></th><th></th><th></th><th>0.06</th><th>0.1</th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th><th></th><th>0.06</th><th>0.1</th></mrv<>				0.06	0.1
nt		F2	<mrv< th=""><th><mrv< th=""><th></th><th></th><th></th><th><mrv< th=""><th><mrv< th=""></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th><th></th><th><mrv< th=""><th><mrv< th=""></mrv<></th></mrv<></th></mrv<>				<mrv< th=""><th><mrv< th=""></mrv<></th></mrv<>	<mrv< th=""></mrv<>
ш	Wood	F1	0.9	1.1				0.8	0.47
		N1	0.21	0.37				0.4	0.15
		N2	0.09	0.11				0.1	0.06
		N3	0.06	<mrv< th=""><th></th><th></th><th></th><th>0.11</th><th>0.05</th></mrv<>				0.11	0.05
		F2	<mrv< th=""><th><mrv< th=""><th></th><th></th><th></th><th><mrv< th=""><th><mrv< th=""></mrv<></th></mrv<></th></mrv<></th></mrv<>	<mrv< th=""><th></th><th></th><th></th><th><mrv< th=""><th><mrv< th=""></mrv<></th></mrv<></th></mrv<>				<mrv< th=""><th><mrv< th=""></mrv<></th></mrv<>	<mrv< th=""></mrv<>

< MRV: below the minimum reported value, 0.05 µg ; F: wipe; N: wipe soaked in cleaning solution TD: technical difficulty: no result for this sample
## APPENDIX D: FIELD EVALUATION OF SURFACE SAMPLING TECHNIQUES

			(µg Be/100 cm <sup>2</sup> )		Ratio
	Location	Surface type and conditions	Wipe	Vacuum	vacuum
1	Above light (fluorescent)	Painted metal, smooth a lot of dust	0,32	0,22	1,5
2	Above light (fluorescent)	Painted metal, low porosity, small accumulation of dust	26	6,7	3,9
3	Above ventilation ducts	Painted metal, low porosity, residual dust because of incomplete sampling	44	38	1,2
3A	Ceiling (near sample 3)	Painted gypsum board, uneven surface	0,06	0,0035	17
4	Above student desk	Painted melamine, smooth	< MRV	0,0008	31
5	Window frame	Stained wood, worn surface	0,16	0,0025	64
6	Above student desk (behind sample 4)	Melamine, smooth	-	-	-
7	Above a vacuum system (above a student desk in sample 4)	Painted metal, smooth	0,1	-	-
8	Inside a hood	Metal, smooth, greasy surface	0,23	-	-
9	Above the hood	Metal, smooth	0,19	-	-

## Table D1: Results of the surface samples for enterprise 3



Figure 1D: Result of ChemTest<sup>™</sup> wipes in enterprise 3

	Lasstian		(µg Be/100 cm <sup>2</sup> )		Relationship
Location			Wipe	Vacuuming	wipe / vacuum
1	Horizontal saw	Metal, presence of fine metallic residues, oily	0,11	0,16	0,68
2	Horizontal saw	Metal, less oily than sample 1	0,16	0,062	2,6
3	Work table	Wood fibreboard, dirty, not oily	<mrv< th=""><th><mrv< th=""><th>1,0</th></mrv<></th></mrv<>	<mrv< th=""><th>1,0</th></mrv<>	1,0
4	Machine (lathe)	Metal, not oily	<mrv< th=""><th><mrv< th=""><th>1,0</th></mrv<></th></mrv<>	<mrv< th=""><th>1,0</th></mrv<>	1,0
5	Machine (lathe)	Metal, dusty, not oily	<mrv< th=""><th>0,0051</th><th>4,9</th></mrv<>	0,0051	4,9
6	Machine (lathe)	Metal, slightly oily	<mrv< th=""><th>0,016</th><th>1,6</th></mrv<>	0,016	1,6
7	Machine		<mrv< th=""><th>0,0048</th><th>5,2</th></mrv<>	0,0048	5,2
8	Machine	Painted metal, small accumulation of dust, not oily	<mrv< th=""><th>0,0056</th><th>4,5</th></mrv<>	0,0056	4,5
9		Pressed wood, slightly dirty, not dusty, not oily	<mrv< th=""><th>0,003</th><th>8,3</th></mrv<>	0,003	8,3
10	Wall	Painted gypsum board, clean	<mrv< th=""><th>-</th><th>-</th></mrv<>	-	-
11	Wall	Painted gypsum board, clean	<mrv< th=""><th>-</th><th>-</th></mrv<>	-	-
12	Door frame	Painted wood, very dirty, dusty, oily	1,2	-	-
13	Door frame	Painted wood, very dirty, dusty, oily	1,4	-	-



Figure 2D: Results of ChemTest<sup>™</sup> wipes in enterprise 6

	Location	Surface type and conditions	Wipe	
		Surface type and conditions	(µg Be/100 cm <sup>2</sup> )	
1	Grinding machine	Wood, dirty, not oily	0.08	
2	Welding table	Metal, dirty, slightly oily	<mrv< th=""></mrv<>	
3	Polishing table	Pressed wood, dirty and damaged, slightly oily	<mrv< th=""></mrv<>	
4	Machine	Metal, dirty and oily	0.54	
5	Machine	Metal, rather clean, oily	0.08	
6	Electrical panel	Metal, clean	<mrv< th=""></mrv<>	
7	Column	Painted metal, rough	0.11	

Table D3: Results of surface samples for enterprise 7



Figure 3D: Results of ChemTest<sup>™</sup> wipes in enterprise 7