

**SAMPLING BERYLLIUM SURFACE CONTAMINATION
USING WET, DRY, AND ALCOHOL WIPE METHODS**

By
Kent Kerr

An Abstract

of a thesis presented in partial fulfillment
of the requirements for the degree of
Master of Science in Industrial Hygiene
in the Department of Safety Sciences
Central Missouri State University

December 2004

ABSTRACT

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This research compared three wipe sampling techniques currently used to test for beryllium contamination on room and equipment surfaces in Department of Energy facilities. Efficiencies of removal of beryllium contamination from typical painted surfaces were tested by wipe sampling without a wetting agent, with water-moistened wipe materials, and by methanol-moistened wipes. Analysis indicated that methanol-moistened wipe sampling removed about twice as much beryllium/oil-film surface contamination as water-moistened wipes, which removed about twice as much residue as dry wipes. As facilities change wipe sampling methods among the three compared in this study, these results may be useful for approximate correlations. Accurate decontamination decision-making depends on the selection of appropriate wetting agents for the types of residues and surfaces. Evidence for beryllium sensitization via skin exposure argues in favor of wipe sampling with wetting agents that provide enhanced removal efficiency such as methanol when surface contamination includes oil mist residue.

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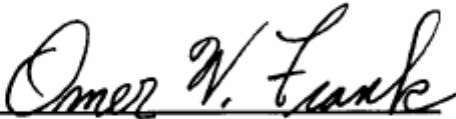
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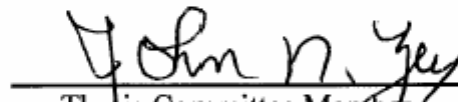
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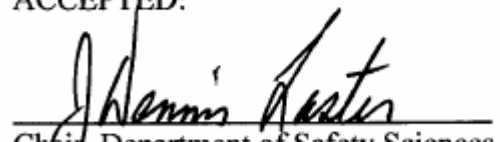
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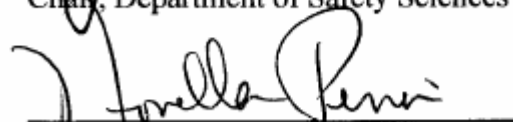

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CHAPTER 1

NATURE AND SCOPE OF THE STUDY

Beryllium was identified in its oxide form in 1798, named “Glucinium,” reduced to metallic form in 1828, renamed beryllium (Be), and began being commercially used as a hardening and toughening agent in metal alloys in 1918. Additional uses have continued to emerge. Today beryllium is a key ingredient in aerospace products, weapons, electronics, nuclear reactors, sports equipment and automobile accessories, springs, non-sparking tools, molds for resin injection, dental applications, golf clubs, fiber optic equipment, cellular network communication systems, bicycle frames, wheels, and precision instruments.

Evidence of negative health effects associated with Be began emerging from Europe during the 1930s but was ignored in the United States (U.S.). In the U.S., medical literature began documenting chemical pneumonia associated with occupational exposures to beryllium oxide in 1943, but controversy regarding health effects was perpetuated by faulty research reporting by the U.S. Public Health Service.¹ Between 1947 and 1949, Be was confirmed to be the active agent associated with lung disease in Be factory workers.

The Atomic Energy Commission (AEC), a major governmental user of Be, began proposing occupational exposure limits for respiratory exposures to Be starting in 1949. In the interim Be has been determined to be a known human carcinogen with the principle route of exposure being inhalation.

The Department of Energy (DOE, successor agency of the AEC) has remained committed to studying the health effects of Be and finding effective methods of protecting workers, their families, and the public from Be contamination. Pursuant to those obligations, the DOE promulgated 10 CFR 850 “Chronic Beryllium Disease Prevention Program,” (CBDPP) which became effective January 7, 2000.²

The DOE’s CBDPP requires that each of the employers of contracted operations at the various sites establish their own site-specific CBDPP prior to beginning Be operations but no later than April 6, 2000. Site CBDPP must contain requirements to minimize:

- the number of employees exposed or potentially exposed to Be;
- opportunities for workers to be exposed; and
- disability and lost work time due to Be health effects.

Among other provisions, the CBDPP addresses minimally acceptable cleanliness criteria for housekeeping in Be work areas, as well as facilities and equipment being released for other uses when no longer needed for Be operations. In particular, this study seeks to expand the understanding of those who:

- must implement the requirements for sampling removable surface contamination contained in housekeeping and release criteria provisions of the CBDPP (10 CFR 850.30 and 850.31 respectively);
- interpret the analytical results; and
- make decisions that impact worker and public protection from Be contamination based on the analytical results from removable surface contamination samples.

The rationale for the housekeeping and release criteria found in the preamble to the final rule clearly established removable surface contamination limits in order to minimize or eliminate worker and public exposure to Be inhalation hazards.³

The housekeeping section of the regulation stipulates that during periods when the shop is not machining or working with Be-containing materials, room and equipment surfaces should be cleaned to prevent dust buildup and assure that removable surface contamination not exceed $3 \mu\text{g Be}/100 \text{ cm}^2$. At this level, machine operators who have been educated about Be hazards and know how to protect themselves from exposure can presumably continue to work in the area without negative health effects. Release criteria apply when a shop is being permanently retired from working with Be-containing materials, or if a tool or piece of equipment is no longer needed for operations with Be-containing materials and will be released for other uses. When an organization turns previously Be-contaminated facilities or equipment over to others for non-Be work, it is important to assure that the new users will not be exposed to health-threatening residual surface contamination. To protect workers and the public from Be exposure associated with the former Be working areas or equipment, their surfaces must not have more than $0.2 \mu\text{g Be}/100 \text{ cm}^2$ of removable surface contamination remaining.

The nature of surface contamination that would pose the most obvious potential for resuspension is dry loose dust. For that type of contamination the use of water-dampened surface wipe sampling, as recommended in the preamble, would undoubtedly provide adequate collection efficiency. However, in many facilities with a history of machining parts from Be-containing alloys, surface contamination includes a small amount of aged metal working fluid (MWF) residue that may not be readily removable

by casual wiping with water-dampened wipes. The slight oiliness of the surface may complicate wipe sampling results while, at the same time, making a portion of the Be contamination less likely to become resuspended in air and inhaled. On the other hand, the slight oiliness of the surface contamination may increase the biological availability to the relatively lipophilic surface layers of human skin. Thus a tradesperson working on utility lines in the vicinity of contaminated I-beams near the ceiling may be less likely to disturb and subsequently breathe Be-containing dust stuck in oxidized mineral oil mist residue. The same tradesperson resting a sweaty wrist on the beam while working with the adjacent conduit could nonetheless become sensitized to Be through skin exposure.

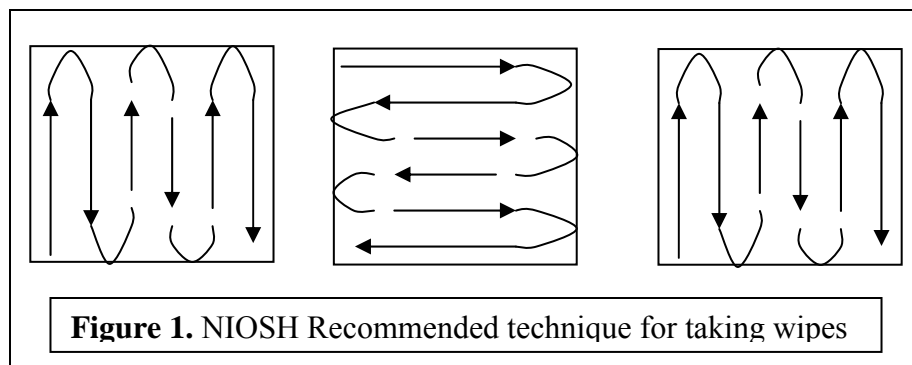
Statement of Problem

DOE's CBDPP specified not-to-exceed removable surface contamination but did not specify a standard required sampling method.*

The Preamble to the final rule at 10 CFR 850 recommended the wipe sample technique used with NIOSH method 9100.⁴ That method uses Ghost Wipes, which are individually-packaged deionized water-moistened towelettes. The towelette is folded once then wiped over the surface in three or four vertical s-curves (see Figure 1 on the next page); then folded again (exposing a fresh face), wiped across same 10 cm square in three to four horizontal s-curves, then folded and wiped vertically, placed in sample tube, and sent to the laboratory for digestion and analysis.

* A special committee was convened in September 2004 at Headquarters DOE to adopt a standard wipe sampling method (see 9/24/04 Memo at Appendix A). ASTM 6966 was designated as the standard DOE method for wipe sampling.¹⁵

Although the NIOSH 9100 wipe sampling technique was recommended, the writers of the CBDPP recognized that alternate methods were already established at various sites, and decided not to prescribe a single, standard sampling procedure. The CBDPP authors recognized that “the use of diverse sampling methods (e.g., differences in type of sample media, type of solvent (if any) on the sample media, area sampled, etc.) may easily lead to the reporting of inconsistent results.”⁵ Though stating a preference that all sites eventually transition to the damp wipes, no incentive to do so was provided. In the intervening years, sites have continued to use various sampling methods. Some use dry filter paper wipes, some use water-dampened fabric wipes, while others use alcohol-dampened wipes. A recent survey indicated that of eighteen DOE installations, twelve use wetted (mostly water) wipe sampling materials, while six facilities wipe sample with dry materials.⁶



The authors of the regulation acknowledged that the term “removable” contamination was not necessarily clear. In response, they provided the definition: “*Removable contamination* means beryllium contamination that can be removed from surfaces by nondestructive means, such as casual contact, wiping, brushing or washing.”⁷ Although this definition purported to clarify the degree of removableness of the sampled contamination, there is a perceptible difference between “casual contact wiping” and

“washing.” “Washing” would seem to imply the use of a solvent designed to remove more contamination than would be removed with “casual contact.” This issue becomes more cogent when considering the nature of surface contamination that has accumulated for decades in shops working with MWF. Oil mist aerosols generated in such metal machining shops leave somewhat tacky surface residues on walls, ceilings, shelving, and equipment that respond significantly differently to dry paper wipes than to alcohol wipes. Without a solvent to cut the aged mineral oil residue, dry paper wipes can potentially contribute (fibers) to the residue more than they remove (beryllium). Discussions with a professional having extensive experience in beryllium decontamination provided valuable insights regarding wipe materials versus the nature of surface residues (see Memo of 8-31-04 conversation with Lloyd Lazarus at Appendix A, [Memoranda of Conversations](#)). This illustrates the basis for sampling differences when one site samples using a casual contact dry wipe versus a site that samples by washing surface contamination with a solvent that removes the contamination-bearing matrix. The results are obviously not comparable or consistent. If a less-than-adequate method of wipe sampling is employed, erroneous decisions may be made not to decontaminate and facilities or equipment could be released with surface contamination significantly more than $0.2 \mu\text{g Be}/100 \text{ cm}^2$.

The surface sampling provisions of the CBDPP was explicitly intended to verify minimization of respiratory exposure to beryllium particulates.³ Since the implementation of the CBDPP, an increasing body of evidence has developed through scientific research and practical experience implicating skin contact with beryllium sensitization. Researchers have demonstrated that 0.5 and 1 μm particles penetrate the outer layers of repeatedly flexed human skin and that topical application of Be resulted in

Be sensitization in mice.⁸ Brush Wellman, the major U.S. producer of Be, implemented sophisticated engineering controls which reduced inhalable Be particulates by a hundredfold. Together with a rigorous respirator program, they virtually eliminated inhalation exposure to Be in their processing facilities. However, Be sensitization rates continued essentially unchanged at 10-12% per year. Suspecting that sensitization was occurring through skin contact, Brush Wellman implemented their comprehensive exposure pathway control program, including skin protection. Since the provision of skin protection, the rate of sensitization has dropped to zero.^{9, 10} This raises additional issues regarding the importance of removing of beryllium contamination from surfaces. Using dry paper or water-dampened wipes can prejudice sampling results by avoiding the collection of lipid-laced contamination residues in shops with historical use of MWF. Since lipophilic contamination is a greater concern for skin exposures,^{11, 12} consideration would be appropriate for broadening the rationale for surface sampling and recommended sampling methods.

Statement of Purpose

The purpose of this experimental research is to determine if there is a consistent significant relative difference in the removal efficiency of alternate wipe sampling methods currently used within the DOE complex. Specifically, this research seeks to establish the removal efficiency of wipe sampling with dry Whatman smear tab paper, versus wipe sampling with Ghost Wipes, versus wipe sampling with methanol-moistened gauze wipes on surface texture and residues typical of metal-machining shops.

Hypothesis

It is hypothesized that methanol-moistened wipe sampling removes a greater percentage of beryllium surface contamination associated with metal-working fluid residue from a typical painted surface than dry or water-moistened wipe sampling. Hypothetically, by maintaining identical conditions of plate size, plate material, surface texture, residue, surface Be mass loading, wipe pressure, and pattern, while varying only the wipe materials, relative differences in mean recovery efficiencies will be distinct enough to demonstrate a consistent correlation. The null hypothesis is that methanol wipe sampling removes the same proportion of beryllium surface contamination associated with metal-working fluid residue from a typical painted surface as dry or water-moistened wipe sampling.

Assumptions

The solvent action of methanol-dampened wipes is assumed to dissolve and remove surface contamination containing MWF residues more effectively than distilled-water dampened wipes or dry wipes.

Efficient mist elimination systems are a relatively new development in the metal machining industry. Enclosed machining centers with air cleaning systems do an excellent job of removing most oil mist aerosols generated by cutting heads doused with streams of MWF, but no capture system can be 100% efficient. Smog-Hog®, a well-known brand of air filtration equipment for enclosed metal machining, claims 95% collection efficiency.¹³ Over time, fugitive MWF aerosol released in the metal working

shop collects on walls, ceilings, and equipment. Flat painted surfaces having some minimal accumulation of oily residue are relatively typical for metal machining shops.

Baking the MWF residue at 230°F for 25 minutes changes the nature of the residue, making it slightly tacky rather than oily. Individuals experienced in decontaminating metal machining shops state that this degree of tackiness is typical.

Complete uniformity could not be guaranteed though an effort was made to maintain consistency in the application of spray on bond coat primer paint on the glass Petri dishes. Slight variations in the nature of the painted surface texture could contribute to variability in the sampling results. However, any such slight surface variability would also be typical of surfaces in facilities and equipment used for processing Be-containing materials.

Whether the wipe pattern is the s-strokes of NIOSH 9100 (see Figure 1), the concentric squares of OSHA 125G, or the patterns described in American Society of Testing and Materials (ASTM) Procedure 6966, it is clear that the objective is to maximize removal efficiency while maintaining consistency in the way wipe samples are collected. The variant method used in this experiment used 10 cm diameter round test plates (78.54 cm²) rather than the 10 cm square (100 cm²) areas used in other methods. Due to the round area of the test plates, concentric circular wiping patterns were consistently used for sampling surface contamination residues from each test plate. Since surface loading can be readily correlated on a loading per cm² basis, and an identical pattern of wiping test plate surfaces was consistently maintained, it was assumed that removal efficiency would not be significantly affected by using round test areas and wipe patterns instead of square test areas and wipe patterns.

The standard 10 µg/ml solution used to make up the solution containing 1µg Be/10 ml that was added to each test plate also contained aluminum, iron, lithium, sodium and nickel, in addition to beryllium. Since all of these elements are typically in the mix of surface contamination in metal-machining shops, it was assumed that any interference in the analytical method would be typical of real-world conditions. According to the Backup Data Report for OSHA's Method ID-125G, those interferences should be minimal with most state of the art equipment and good lab technique.¹⁴

Limitations

The results of this study will be generalizable to similar painted surfaces with traces of metal working fluid residues. These experimental results will not necessarily apply for other surfaces. Glassy, very porous, and absorbent surfaces can be expected to have differing removal efficiencies when comparing wipe sampling methods.

Contamination residues of varying consistency can be expected to have diverse removal efficiencies when comparing wipe sampling methods. Dry dusty residues can be expected to result in greater relative removals when compared to oily, sticky contamination residues depending on solubility.

Significance of Study

This study comparing the removal efficiencies of the dry, water-moistened, and methanol-moistened wipe sampling methods will be useful as DOE sites transition from pre-existing alternative methods to the ASTM D 6966 wipe sampling method.¹⁵

Historical data from different sites using diverse sampling methods will hopefully be more comparable as a result of this study.

With known surface loading of beryllium, these comparisons will provide objective evidence of the percentage of surface contamination removed by wipe sampling from the experimental standard surface using dry, water-moistened, and alcohol-moistened wipes. This information will be useful in understanding how much surface contamination actually remains when wipe sampling results indicate that “removable contamination” is below established limits.

This study is significant to the degree that understanding of human health protection is advanced. In order to be adequately protective, the selection of wipe sampling methods must be influenced by the nature of surfaces and contamination consistency. For purposes of human health protection, the relative removal efficiency of the selected method should at least be understood. The results of this study may be useful for policy development if Be skin contact health effects influence DOE and others to adjust their recommended wipe sampling method(s) to suit the nature of the contamination and affected surfaces.

Definition of Terms and Acronyms

- ACGIH – American Conference of Governmental Industrial Hygienists – is a professional organization with the mission of advancing worker health and safety through education and dissemination of scientific and technical knowledge. ACGIH authors the widely used Threshold Limit Values (TLVs) and Biological Exposure Indices (BEIs).

- AEC – Atomic Energy Commission – the predecessor agency for the Department of Energy (DOE) having responsibility for nuclear power and defense systems.
- Be – Beryllium – symbol Be, is a hard, light, gray-white alkaline earth metal with atomic number 4, atomic weight 9.01, and density of 1.85 g/cm³.
- Beryllium activity – An activity that potentially exposes workers to beryllium including fabrication, construction, maintenance, and decontamination.
- Beryllium worker – Beryllium Worker – A beryllium-associated worker who conducts beryllium activities at least once per month.
- BEI – Biological Exposure Index (Indices), are concentrations of materials in blood, urine or exhaled air below which adverse health effects are not generally expected with healthy workers.
- BeLPT – Beryllium Lymphocyte Proliferation Test - a medical test that cultures lymphocytes (preferably from bronchoalveolar lavage) and subsequently challenges the culture with BeSO₄ solution. Lymphocytes from beryllium-sensitized individuals proliferate significantly when challenged by the Be salt solution.
- CBD – Chronic Beryllium Disease – A lung disease in which inhaled beryllium particulates result in the formation of granulomas and fibrous reduction of lung volume.
- CBDPP – Chronic Beryllium Disease Prevention Program – a DOE program requiring elimination of occupational exposures that have historically caused CBD.

- CDC – Centers for Disease Control and Prevention – is an agency of the United States Department of Health and Human Services serving to develop and apply methods of disease surveillance, prevention and control to improve citizen health.
- CFR – Code of Federal Regulations – a structured system of federal rules of the United States organized into parts according to the departments responsible for the execution of those rules.
- DOE – Department of Energy – is a cabinet-level department of the United States federal government responsible for special defense systems and domestic power policies, including nuclear power.
- DOE Complex – The various offices, laboratories, industrial operating sites comprising the official activities of the Department of Energy
- EPA – Environmental Protection Agency – Is an agency of the United States federal government responsible for the stewardship of the Nation’s natural resources.
- HEPA – High Efficiency Particulate Air – is a term generally applied to air filtering equipment with an efficiency of 99.97% for retaining particles of 0.3 microns or larger.
- H-FMT – Honeywell Federal Manufacturing and Technologies – a division of Honeywell International contracted to manage and operate NNSA’s Kansas City Plant.
- ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy – is an analytical method that injects analyte into high temperature energy plasma of ionized neutral molecules induced by an electro-magnetic field. The electrons of

the analyte atoms are excited to higher energy configurations in the superheated plasma. Upon retreat to the unexcited state, diagnostically specific wavelengths are emitted, which are measured by photomultiplier tubes.

- IPM – Inhalable Particulate Mass – is one of three particle size-selective criteria used by ACGIH for establishing TLVs for concentrations of airborne particulate contamination. IPM are considered hazardous when deposited anywhere in the respiratory system and are characterized by particle sizes up to 100 μm with a significant portion of particles in the range between 25 and 100 μm .
- KCP – NNSA’s Kansas City Plant operated by Honeywell Federal Manufacturing and Technologies (H-FMT)
- MCL – Maximum Contaminant Level – is the concentration of contaminant that is not to be exceeded in drinking water, per EPA regulation.
- MWF – Metal Working Fluid – also known as metal reducing fluid, serves to flush away swarf (metal particles abraded by grinding/cutting tool) while cooling high speed cutting edges, and lubricating metal cutting operations.
- NESHAPS – National Emissions Standards for Hazardous Air Pollutants – there are 188 toxic chemicals for which airborne releases are regulated by EPA under 40 CFR 61 subpart M, per the 1990 Clean Air Act Amendments.
- NIOSH – National Institute for Occupational Safety and Health – is part of the CDC in the U.S. Department of Health and Human Services, and is responsible for research and recommending measures to prevent work-related injury and illness.

- NNSA – National Nuclear Security Administration – An agency of the U.S. Department of Energy responsible for the stewardship of nuclear defense systems and nuclear power plants for Navy vessels.
- OELs – Occupational Exposure Limits – consensus and regulatory restrictions on the concentrations and duration of exposures normally healthy workers may receive without expectation of adverse health effects. Examples of OELs are OSHA PELs, NIOSH RELs, ACGIH TLVs, etc.
- OSHA – Occupational Safety and Health Administration – An agency of the U.S. Department of Labor, OSHA is responsible for setting and enforcing health and safety standards for occupational environments, along with providing training for the promotion of safe and healthful workplaces.
- PEL – Permissible Exposure Limit – The legally enforceable 8-hour time-weighted average occupational exposure not to be exceeded, as regulated by OSHA. PELs may be found at 29 CFR 1910.1000 Table Z-1, Limits for air contaminants; Table Z-2, Time-weighted averages and ceiling values; and Table Z-3, Mineral Dusts. The beryllium 8-hr TWA PEL is $2 \mu\text{g}/\text{m}^3$, and the ceiling is $5 \mu\text{g}/\text{m}^3$.
- PPE - Personal Protective Equipment – gear designed to shield people from chemical, radiological, physical, electrical, mechanical, or other hazards. PPE includes a wide variety of eye protection, steel-toed shoes, gloves, hearing protection plugs and muffs, and hard hats.
- REL – Recommended Exposure Limits – NIOSH occupational exposure limits based on 8- or 10-hour time-weighted-average or ceiling exposure.

- RPM – Respirable Particulate Mass - is one of three particle size-selective criteria used by ACGIH for establishing TLVs for concentrations of airborne particulate contamination. RPM is considered hazardous when deposited in the gas-exchange area of the lungs, and is characterized by particle sizes up to 10 μm with more than 50% of particles smaller than 4 μm .
- RTECS – Registry of Toxic Effects of Chemical Substances - a congressionally mandated activity established by Section 20(a)(6) of the Occupational Safety and Health Act of 1970 (PL 91-596). RTECS was previously the NIOSH-maintained database of toxicological information, but was privatized in 2001.
- RQL - Reliable Quantitation Limit - ten standard deviations greater than the analytical instrument response for the medium (field) blank.
- SARA – Superfund Amendments and Reauthorization Act – A 1986 amendment to the Comprehensive Environmental Response, Compensation and Liability Act. Title III, Section 313, requires annual reporting for facilities having more than threshold amounts of hazardous substances.
- STEL – Short Term Exposure Levels – not-to-exceed levels of exposure that healthy workers may receive for a relatively short time, usually 15 minutes, without expectation of adverse health effects.
- TLV – Threshold Limit Value – TLVs are widely-used standard occupational exposure levels continuously reviewed and updated by the ACGIH.
- TSCA – Toxic Substances Control Act of 1976 – charged EPA with responsibility for tracking 75,000 industrial chemicals including polychlorinated biphenyls,

asbestos, lead, dioxins, beryllium, etc. TSCA regulation can be found at 40 CFR 700-799.

- TWA – Time-Weighted Average – is calculated by breaking the workday into segments, multiplying the concentration level measured during a workday segment by the segment duration, then adding that to the product of the next segment duration and concentration, and so on for the full workday. The sum of the products is then divided by the total length of the workday to yield the time-weighted average concentration over the full workday.
- Wipe Sampling – also known as swipe sampling or smear sampling – is a method of testing for removable surface contamination by wiping a (usually dampened) paper or gauze over a known area (usually 100 cm²), then digesting the wipe material (usually in strong mineral acid) and analyzing the digestate for the material of interest.

CHAPTER 2

REVIEW OF LITERATURE

“Microgram for microgram, beryllium is one of the most toxic elements on the periodic table.”¹⁶

The fundamental reason for wipe sampling beryllium (Be) residues from environmental surfaces is the prevention of diseases caused by Be contamination released by industrial activities. Wipe sampling shows if surfaces have dust that could be disturbed and resuspended to be subsequently inhaled. Surface contamination also poses potential health risk via skin contact that may be exacerbated by the presence of oil in surface residues. This review of the literature will recount the history of industrial uses of Be, its health effects, and the development of standard wipe sampling practices within the overall context of controlling exposures to Be.

Discovery and Physical Properties of Beryllium

Beryllium (Be) is a hard, light, gray-white alkaline earth metal with atomic number 4, atomic weight 9.01, and density of 1.85 g/cm³. Identified in 1798 by the French chemist Vauquelin and named *glucinium*, from the Greek word glykys that means “sweet,” (beryllium oxide powder tastes sweet) the element was reduced to metal form in 1828 by the German metallurgist Wohler, and renamed *beryllium*. Assigned CAS number 7440-41-7, UN 1966 and 1567, and RTECS number DS1750000, beryllium is a solid at normal room conditions, melts at 1287°C, and boils at 2471°C. Elemental

beryllium is insoluble in cold water, slightly soluble in hot water and aqueous alkalis, but dissolves vigorously in dilute acids.^{17, 18, 19} The concentration of Be in MWF (alkaline solution heated in metal grinding) is observed to increase over time (see 11/15/04 Memo at Appendix A, [Memoranda of Conversations](#)). Fugitive oil mists can contribute to Be surface contamination in metal working shops with recirculating MWF.

Metallic Be reacts with hydrogen gas and incandesces when heated in halogen gases or with phosphorus.²⁰ Aerosolized Be powder poses a moderate fire or slight explosive hazard if there is an ignition source. Mixtures of powdered Be with carbon tetrachloride or trichloroethylene will flash on impact. Metallic Be reacts with lithium,²¹ and powdered beryllium ignites on heating in a mixture of CO₂ and N₂. Be is incompatible with acids, bases, oxidizing agents, halogenated carbon compounds, halogens, and alkali metals. Toxic fumes of BeO are emitted when heated to decomposition in air.²²

Production and Commercial Uses of Beryllium

The U.S. is the world's leading processor, producer, and consumer of Be materials and products. The richest U.S. Be deposits are found in the State of Utah.²³

Commercial uses were found for the new hard light metal starting with a 1918 patent for a beryllium-aluminum alloy. Brush Laboratories of Cleveland, Ohio, and the Siemens Company in Germany researched and found other uses for the metal. The first commercial production of Be-containing materials in the U.S. was by The Beryllium Corporation of America in 1927 (later Berylco, later Kawecki Berylco Industries, later Cabot Corporation, NGK). Brush Laboratories, renamed The Brush Beryllium Company

(later Brush-Wellman), became a commercial producer of Be materials in 1931. Clifton Products incorporated in 1939 as a supplier of Be-oxide for the refractory and fluorescent lamp industries. Clifton products ceased operation in about 1949, because health concerns associated with Be phosphors used in the manufacture of fluorescent lighting prompted lighting manufacturers to use other phosphor materials. The Cabot Corporation discontinued Be production 1986, selling its beryllium-copper operations to NGK Metals Corporation of Japan.²⁴ Brush-Wellman continues as the principle U.S. company that is still processing beryllium ore and producing beryllium materials.

Early uses of Be focused on its metal-strengthening properties, its electrical conductance and its use for electrical filaments. Nuclear scientists soon discovered Be's usefulness for absorbing and generating neutrons, which are key aspects of regulating nuclear reactions. In 1947, the Atomic Energy Commission became one of the primary users of Be materials in the United States. Since those early days, uses of Be have continued to proliferate.¹⁶

Its extreme light weight and high melting point have resulted in many practical uses for beryllium. Lighter than aluminum, beryllium is over 40% more rigid and 33% more elastic than steel. Added to other metals, Be yields alloys with much greater conductivity and strength. Just 2% beryllium in copper alloys results in six-fold increases in strength. In air, a thin film of oxide forms on the surface of beryllium metal resulting in significantly increased resistance to corrosion. Be also lends corrosion resistance when alloyed with other metals.^{25, 26}

The pure beryllium metal is used in space vehicle optics, heat shields, X-ray transmission windows, disc brakes for aircraft, audio components, structural members for

aircraft and satellites, missile parts, nuclear reactor neutron reflectors, nuclear weapons, rocket propellants, and navigational systems.^{19, 25, 26} As noted above, Be has great heat absorption capacity and high melting point making it ideal for high temperature applications. It is nonmagnetic and, of all metals, it has the lowest thermal neutron absorption cross-section.²³

Copper, aluminum, and other metals alloyed with beryllium are used in aerospace, weapons, electronics, nuclear reactors, sports equipment and automotive industries for springs, non-sparking tools, molds for resin injection, dental applications, golf clubs, fiber optical equipment, cellular network communication systems, bicycle frames, wheels, precision instruments, and many other uses. Be ceramics are used in semiconductor chips and rocket covers. Most homes have multiple components containing Be, including controls for air conditioners, microwaves, freezers, water heater, and computers. It's even used in dental appliances like bridges and crowns.^{16, 19, 25, 26, 27}

Beryllium oxide finds wide commercial industrial usage in electrical insulators, gyroscopes, armor plating, nuclear reactor fuels, laser structural components, high-tech ceramics, automotive ignition systems and an additive to plastics and glass.^{19, 25} A more recent Be-aluminum alloy called Beralcast® is finding applications in U.S. military aircraft and missiles.²³

Manufacturing Process

Of about 40 beryllium-containing minerals, the two listed below are commercially important:

- Beryl ($3\text{BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$) containing about 4% beryllium (predominant mineral mined in Brazil and Eurasia).
- Bertrandite ($4\text{BeO} \cdot 2\text{SiO}_2 \cdot \text{H}_2\text{O}$) containing < 1% beryllium (predominant mineral mined in the United States). Bertrandite is efficiently processed into beryllium hydroxide, which is then reacted to produce beryllium oxide, the most important high-purity commercial form of beryllium.^{19, 25}

Bertrandite is strip-mined and processed in Utah. The ore is wet-milled, leached with sulfuric acid near the boiling point, dissolved in water, solvent extracted with di-2-ethylhexyl-phosphoric acid in kerosene, precipitated by reheating to yield beryllium basic carbonate ($2 \text{BeCO}_3 \cdot \text{Be}(\text{OH})_2$), which is filtered, repulped in deionized water, and further heated to form beryllium hydroxide.

Beryllium hydroxide is dissolved in ammonium bifluoride solution, ammonium fluoroberyllate crystals are then thermally decomposed to beryllium fluoride at about 900-1000°C. Beryllium fluoride is reacted with magnesium in large graphite crucibles at about 900°C producing molten beryllium.^{19, 28} The resulting Be metal can be pulverized by mechanical milling and consolidated by vacuum hot-pressing at 1100°C and 1200 psi. Most users of Be buy rough-machined hot-pressed Be blanks a little larger than the shape of the desired parts and then machine them to the ultimately desired shape.¹⁹

Beryllium hydroxide powder is mixed with carbon powder; copper chips and melted together at 3500°F in an arc furnace to produce beryllium-copper alloy. The molten copper, containing about 4% beryllium, is poured into ingot molds. The ingots are then hot rolled, extruded, or used for other alloying operations.

Beryllium ceramics are produced by redissolving Be oxide powder and refiring in a car hearth furnace. The purified oxide powder is formed into ceramic shapes by various processes such as dry pressing, hot pressing, extruding, or tape casting.¹⁹

Non-occupational Exposures to Beryllium

Practically everyone on the planet is exposed to small amounts of Be. It is a naturally occurring element in coal, soils, dusts, and bodies of water. The natural background levels of beryllium in rocks, soils and minerals normally ranges from 0.038 to 11.4 mg/kg. The United States Environmental Protection Agency (EPA) has estimated that the general U.S. population has a daily beryllium intake of 420 ng from background environmental exposure.²⁵ The natural background levels of beryllium in the environment are greater or lesser depending on varying natural soil composition. In addition to the naturally occurring beryllium in the environment, some areas are contaminated by releases from coal burning, mining and industrial processing operations. The combustion of coal and oil contributes about 1250 or more tons of Be to the environment each year, which is about five times the annual production from other industrial sources. Groundwater in the State of New Mexico has an average Be concentration of 3 µg/l.²³ Researchers have found that the average person has about 0.26 µg/l of Be in their urine.²⁹ The EPA has estimated the manufacturing industries release about 50,000 pounds of beryllium to the environment each year, with most (> 90%) released to land and as much as 5,500 lb. going to the atmosphere from point sources.²⁵ The Toxics Release Inventory estimated that between 1987 and 1993, over 340,000

pounds of beryllium were released to land and water in the United States. These releases were primarily from copper beryllium operations in Ohio and Pennsylvania.³⁰

Industrial occupational exposures to even low-Be content alloy particulates and fumes have resulted in disease, as have extra-occupational exposures to those handling the clothes of Be workers and people living in the neighborhood of Be processing facilities. Ambient air sampling and voluntary radiographic surveys of residents in communities having Be manufacturing facilities provided evidence that the lowest observable adverse effects level for beryllosis, or chronic beryllium disease (CBD), was 0.01 to 0.1 $\mu\text{g Be}/\text{m}^3$. That data was the basis for Eisenbud team's recommendation that ambient air in the neighborhood of beryllium plants should not exceed a monthly average concentration of 0.01 $\mu\text{g Be}$ per cubic meter.^{24, 31} People who have breathed Be dust for even a few days have an increased potential for sensitization and disease that will stay with them for the rest of their lives¹. Recent information indicates that some people may develop CBD after short exposures below the current occupational exposure limits.²⁷

Health Effects of Beryllium

The most vulnerable populations are those having occupational exposures to Be and Be-containing materials. Beryllium miners, jewelers (emeralds and aquamarine are beryllium containing crystals), and workers in processing, alloying, fabrication, reclaiming, and nuclear technology facilities have greater occupational exposures. The Occupational Safety and Health Administration (OSHA) has estimated that about 25,000 U.S. workers have been exposed to beryllium. Of those having occupational exposures to

beryllium, physiological factors such as age, sex, overall health status, and genetic factors make some individuals more susceptible to the diseases associated with beryllium.²⁵

Early Confusion about Toxicity

Reports of disease conditions related to Be began to emerge from European sources in the 1930's but went unnoticed in the United States. Dr. Howard Van Ordstrand's paper "Chemical Pneumonia in Workers Extracting Beryllium Oxide," published in 1943, was the first U.S. medical science literature regarding the health effects of Be.³² Unfortunately, the authorities of the U.S. Public Health Service published a report the same year claiming that Be was inert with regard to human health effects. According to the Public Health Service report (the Fairhall report), any toxicity previously attributed to Be was actually caused by fluoride compounds present as byproducts in Be materials.^{1,33} The infamous Fairhall report confused the issue, but evidence of Be's toxicity kept accumulating in the wake of its ever widening industrial uses. Dr. Van Ordstrand published an update in 1945 documenting 170 cases of dermal and respiratory illness (including five acute lung disease fatalities) among Be workers.³² In 1946, cases of similar diseases were reported among employees of a Massachusetts fluorescent lighting industry where Be phosphors were handled.²⁴

Toxicity Confirmed

In 1947, the Atomic Energy Commission (AEC) began to be a major user of Be materials. The AEC's Health and Safety Director, Merrill Eisenbud, took an active

interest in resolving the controversies surrounding the health effects of Be. Working together with Brush & Clifton Products and the Ohio Health Department, Eisenbud confirmed Be to be the active agent causing lung disease in Be factory workers, community residents affected by industrial emissions, and those laundering the clothes of factory workers. Brush employees received a memorandum from their management in October 1949, informing them about beryllium disease and advising medical consultation if they were having symptoms. Brush also notified their customers and started applying warning labels on their products in 1949, in response to the findings of the AEC investigation.³³

In 1949, the Eisenbud team recommended a time-weighted average occupational exposure limit of $2 \mu\text{g}/\text{m}^3$ for airborne Be in factory air; a maximum limit of $25 \mu\text{g}/\text{m}^3$, and a monthly average of $0.01 \mu\text{g}/\text{m}^3$ in neighborhood air around a beryllium processing plant.^{31, 33, 34} That limit was later adopted by the EPA as the National Emission Standard for Beryllium, which is still the clean air standard for ambient air in the vicinity of Be processing facilities.³⁵ The recommended $2 \mu\text{g}/\text{m}^3$ limit for air inside Be processing facilities was specified in federal government contracts with Brush and was later adopted as a Threshold Limit Value by the American Conference of Government Industrial Hygienists (ACGIH) in 1957.^{33, 36}

Toxic AND Carcinogenic

Be and Be-containing materials are toxic, and Be has been determined by the International Agency for Research on Cancer (IARC), the U.S. National Toxicology Program (NTP), OSHA, the National Institute for Occupational Safety and Health (NIOSH), and the ACGIH to be a known human carcinogen.^{25, 37, 38, 39, 40} The principle

route of exposure is inhalation and the primary target organ is the lung. Skin and eyes are routes of exposure and subject to harmful effects. It has historically been thought that the most common way for beryllium to get into the human body is via inhalation of particles or fumes. Most exposures of concern are associated with occupational exposures, but coal burning and even some brands of cigarettes can be significant sources of beryllium aerosols. Some Be can get into the body through pores and breaks in the skin. Very little Be gets into the body through ingestion. Results of lab experiments indicate that only about 1% of ingested Be gets absorbed into the bloodstream. However, once through exterior membranes and into contact with the phagocytes and lymphocytes in the blood, lungs, skin or lymph, sensitization is possible. Once in the bloodstream (usually in the form of a salt), Be can deposit in bone or be excreted by the kidneys.^{25, 29}

Exposures at Ore Processing Facilities

Those who work in facilities where beryllium ore is refined and processed are potentially exposed to acidic and caustic forms of Be the fluoride (BeF_2), the ammonium fluoride and sulfate (BeSO_4), and also to beryllium oxide (BeO), and hydroxide [$\text{Be}(\text{OH})_2$]. These acidic and caustic forms of Be have been shown to cause dermatitis, chronic skin ulcers, inflammation of naso-pharyngeal passages, rales (rattling breath heard in chest with stethoscope), bronchitis, pneumonia with fever, coughing and chest pain. In plants where Be is alloyed with other metals or formed into ceramics, workers are potentially exposed to the oxide. The oxide form is of great concern regarding potential health effects due to its powdery and insoluble nature. The temperature at which BeO is fired plays a significant role in its toxicity. Low-fired BeO has a threshold

dose concentration of 1-to-3 mg Be/m³. Ten times that concentration of high-fired BeO is required for the same threshold response.⁴¹ Many other potential industrial exposures occur in the milling of Be and Be-containing alloys.^{42, 43} Those who machine parts from Be and Be-alloy metal stock have been shown to have a greater potential for developing Be sensitization and CBD than the custodial employees; however, a significant number of custodial employees were found to have Be health effects.⁴⁴

Acute Beryllium Disease

Acute pulmonary beryllium disease is a condition primarily associated with the early days of Be industries when workers were exposed to high airborne levels of Be ore dust and fine particulates and fumes from Be processing. More soluble (BeCl₂, BeF₂, BeSO₄) forms and larger particle sizes would cause severe irritation and inflammation of the nasal and pharyngeal mucosa. Less soluble, smaller particles and fumes penetrated deeper into the alveolar region of the lung and caused acute chemical pneumonitis²¹. Virtually 100% of workers exposed to more than 1000 µg/m³ of airborne Be developed acute pulmonary disease. Few workers exposed to less than 100 µg/m³ developed acute Be pulmonary disease. The acute disease was generally observed to develop rapidly within 72 hours of massive exposure; a more insidious form of the disease developed several days or weeks following a major exposure. About 10% of rapid-onset Be pulmonary disease resulted in fatalities. Recovery was reportedly complete within three weeks if no further major exposure events occurred. The insidious form of the disease was slower to diagnose and had a longer recovery period (probably because the worker continued to work in a Be dust environment). Of the workers diagnosed with acute

pulmonary Be disease, researchers estimated that about 15-30% developed chronic beryllium disease.^{26, 27, 41, 45} Acute Be pulmonary disease was virtually eliminated by the installation of engineering controls and personal protective equipment, which improvements are attributed to the cooperative efforts of the AEC and Brush-Wellman Industries in the late 1940s.^{33, 45} The few cases since the 1940s have been the result of a few industrial accidents that caused major releases of beryllium-laden dust or fumes.

Dermal Health Effects

Cutaneous effects of Beryllium include contact dermatitis, hypersensitivity dermatosis, chemical ulcers, ulcerating granulomas, dermal granulomas, and systemic sensitization.^{27, 38} Most of these cases were associated with the processing of Be ore and were a function of the solubility and magnitude of exposure. The more soluble salts cause relatively quick responses, whereas Be metal or oxide-induced skin pathologies develop after some latency period. Be particles that get into breaks in the skin or otherwise become imbedded in the skin can cause lesions that do not heal properly, ulcers, granulomas, wart-like bumps, and/or rashes.²⁷ Studies in the late 1960s raised questions about the relationship between skin hypersensitivity and potential for pulmonary disease. Those researchers demonstrated that Be can penetrate the skin, become complexed with plasma proteins, and subsequently trigger cell-mediated immune reactions.^{46, 47}

A recent definitive study demonstrated conclusively that fine particulates (0.5 and 1 μm) can work their way through flex points of uninjured human skin. This same research project found that Be activated Langerhans cells and cluster of differentiation

protein molecules on the surface of lymphocytes located in the area of the skin where Be particles had penetrated the epidermis of mice. Neighboring lymph nodes were activated. T-lymphocytes were released into the peripheral blood, and beryllium-specific changes are initiated in the molecular sorption properties of the lymphocyte cell membranes. These findings confirmed that systemic sensitization to Be can be caused by skin exposure.⁸ The results of this cutaneous sensitization study also help explain why Be sensitization had not been essentially eliminated like pulmonary Be disease with the advent of improved engineering controls and respiratory protection. Without appropriate skin protection, like gloves, workers were still getting Be through their skin when handling the metal or getting dust on hands, wrists, and arms. There is increasing evidence that sensitization developed by skin exposure may lead to systemic sensitization.^{9, 10, 48}

*Beryllium Sensitivity and Chronic
Beryllium Disease (Berylliosis)*

Be has a small ionic size (0.35Å) and high reactivity (charge-to-radius ratio of 5.7), and the highest Pauling electronegativity (1.5 kcal/ g atom) of all the alkaline earth elements. As a result, Be is capable of bonding tightly with key sites of complex molecules such as nucleic acid and proteinaceous structures that are relative inaccessible to other metallic ions. It is able to displace other divalent positively charged ions in biomolecular environments. These characteristics probably contribute to Be's toxicity to living cells, impacting chromosomal gene replication, cell division, enzyme function and protein phosphorylation.²³

Available data indicates that a relatively small percentage of the general population is predisposed to Be sensitivity. A significant number of patients with CBD have been found to share similar genetic vulnerabilities.⁴⁹ Ninety-seven percent of individuals with CBD have the HLA-DPB1 Glutamate 69 genetic marker.^{45,50} Once sensitized, conditions may progress to CBD even without continued exposure.⁵¹

Chronic beryllium disease develops in stages after lung exposure to Be particulates and/or fumes. The mucociliary escalator does not clear the fine particulates (<5 μm) that deposit in the alveolar regions of the lung. If the particulate is of the more soluble forms of Be (halide or sulfate salts), it may dissolve into systemic circulation, be staged in the liver, stored in the bone, or be eliminated through the kidneys. However, BeO and Be-metal particles are essentially insoluble and remain as foreign bodies in the gas exchange region of the lungs. Bound with protein, the Be-hapten triggers a chain of events that can lead to sensitization and the formation of granulomas in the lungs. Macrophages and lymphocytes accumulate around the Be hapten. Clusters of differentiation molecules CD4+ are activated on T-lymphocytes and lymphokines are released, recruiting other defensive cells (neutrophils, lymphocytes, monocytes and fibroblasts). Granulomas, which under a microscope appear as tight-packed macrophages, monocytes, and lymphocytes, form around the Be particles in the alveolar sacs. Fibroblasts are recruited; collagen is produced resulting in reduced lung volume and loss of elasticity.^{38, 42, 43} Symptoms include labored breathing, coughing, and shortness of breath, loss of appetite, chest pain, and fatigue. Forced vital capacity (a measure of lung volume) and carbon monoxide diffusion capacity (a measure of lung function) both significantly decline as CBD begins to manifest. Red blood cell count, pulse and

respiratory rates are often increased to compensate for the decreased gas exchange ability of the lungs. Chest x-rays show the progressive damage as nodules, fibrous tissues, scarring and alveolar destruction increase.^{27, 41, 42, 43, 52, 53} The disease may develop slowly over several years or can occur over a period of months. If the person with CBD is removed from Be exposure and provided with medical support (certain steroids have been effective), the disease may go into remission or its progression can be substantially slowed. About 30% of people who have developed CBD have eventually succumbed.²¹ Death usually results from respiratory and cardiac failure.

Diagnosing the Disease

The first case of CBD was documented in 1946 and the Massachusetts Institute of Technology originated the Beryllium Case Registry in 1952.⁵⁴ To qualify as a case of Be disease, the following criteria are generally checked:

- Evidence of significant beryllium exposure
- Clinical syndrome compatible with chronic lower respiratory tract disease
- Abnormal chest radiograph
- Abnormal pulmonary function tests with either obstructive or restrictive changes
- Histology consistent with chronic beryllium disease
- Evidence of elevated tissue levels of beryllium.^{43, 52, 53}

Although not always conclusive at first attempts, the beryllium-lymphocyte proliferation test (BeLPT) is a relatively conclusive test for Be-sensitization and for differentiating CBD from other granulomatous lung disorders such as sarcoidosis. Ideally

the BeLPT is done with lymphocytes from a bronchoalveolar lavage but peripheral blood lymphocytes can be used. The cultured lymphocytes are challenged with a BeSO_4 solution. Lymphocytes from individuals with Be disease are Be-sensitized and proliferate significantly when challenged by the Be salt solution. Lymphocytes from individuals whose granulomatous lung disease has been caused by something other than Be do not evidence Be sensitivity by proliferating upon challenge with the Be-sulfate solution.^{43, 53, 55, 56} A study involving BeLPT of 136 beryllium workers exposed to an average of about $0.5 \mu\text{g Be/m}^3$, but with occasional excursions about $5 \mu\text{g Be/m}^3$, determined that the lowest observable level at which Be sensitization occurred was $0.55 \mu\text{g Be/ m}^3$.⁴⁹

There are now several techniques for detecting Be in human tissues and fluids. Combining atomic absorption spectrophotometry and laser ion mass spectrometry, gross amounts and microscopic distributions of Be can be determined. By confirming the absence or presence of Be in biopsied granulomas, the accuracy of CBD diagnosis is significantly enhanced.⁵⁷ However, the bronchoscopy and biopsy is an invasive procedure. Researchers are looking for better tests to improve early detection of Be-induced health effects.⁵⁶

The Cancer Connection

Cohort mortality studies of Be workers have indicted Be as a lung carcinogen.^{38, 58, 59} Supporting evidence from animal studies and in vitro assays are sufficiently convincing.^{41, 57} As a result, Be is listed as: “B1, Probable Human Carcinogen” by EPA; “Group 1, Known Human Carcinogen” by IARC; “A1, Confirmed Human Carcinogen by ACGIH; “Carcinogen” by NIOSH and OSHA. EPA based its more limited assessment of the carcinogenicity of Be on incomplete smoking data and probable exposure to other

potential lung carcinogens.⁵⁷ A recent review of the earlier cohort mortality study, with adjustments for smoking, found that "there is no statistical association between beryllium exposure in these workers and lung cancer when using the most appropriate population cancer rates."⁶⁰

An excellent resource that thoroughly reviews Be health effects including human epidemiological and animal studies is found in the Public Health Service "Toxicological Profile for Beryllium."⁶¹

Evaluating and Regulating Be in the Occupational Environment

Workers who need to know how to protect themselves from becoming Be casualties can be more effectively targeted for hazard communication training if their numbers and general segment of the workforce are more accurately determined.

In 1970, the U.S. Public Health Service (PHS) and the Bureau of Occupational Safety and Health estimated that 30,000 U.S. workers were potentially occupationally exposed to Be. NIOSH estimated 21,233 workers potentially exposed to Be in the National Occupational Hazard Survey (NOHS) conducted between 1972 and 1974. Others using NOHS data postulated that over 800,000 U.S. workers could be exposed to Be. A recent effort to provide a more accurate estimate of Be-exposed worker was conducted because the 1970 PHS survey and the NOHS excluded employees of government agencies and mining operations, and the 800,000 number was not developed with any approved method of estimation. That recent effort calculated estimates with upper and lower bounds based on two possible assumptions. One assumption was that, in

a facility where OSHA found at least one worker with Be exposure greater than 0.1 $\mu\text{g}/\text{m}^3$, all workers were potentially exposed. The alternate assumption was that only the numbers of workers with similar job taskings, as recorded by OSHA, were potentially exposed. Due to the alternative assumptions, the authors estimated the total number of U.S. workers with current potential exposure to Be as either 54,400 or 134,000.⁶²

Occupational Airborne Beryllium Exposure Limits in Transition

To verify the adequacy of engineering controls, maintenance activities, and to minimize exposures to unsafe levels, the air of the work environment must be sampled and tested for Be. Determination of a safe limit remains elusive in the case of Be. Current airborne Occupational Exposure Limits (OELs) were based on best available data 50 years ago. ACGIH and the regulators agree that current data demand more protective OELs. But until better approaches are devised, the outdated ones remain on the books. The threshold limit value (TLV) recommended by the ACGIH is 0.002 mg/m^3 as a time-weighted average over a workday, allowing for a short-term exposure level (STEL) of 0.01 mg/m^3 . ACGIH has published a Notice of Intended Change to reduce its recommended beryllium TLV from 0.002 mg/m^3 to 0.0002 mg/m^3 . OSHA's permissible exposure limit (PEL) is an 8-hr. TWA of 0.002 mg/m^3 , allowing for a ceiling level of 0.005 mg/m^3 , and a maximum peak of 0.025 mg/m^3 not to be exceeded for more than 30 minutes during the workday.^{36, 40} NIOSH's Recommended Exposure Limit (REL) is not to exceed 0.0005 mg/m^3 , with 4 mg/m^3 considered immediately dangerous for life and health.¹⁰¹ The Department of Energy has established an action level of 0.0002 mg/m^3 in the chronic beryllium disease prevention program.⁶³

Of 26 other nations listed on the Registry of Toxic Effects of Chemical Substances (RTECS) Beryllium data sheet, most have adopted the equivalent of the TLV and PEL standards. A few, like Russia, Denmark and Hungary, have established occupational exposure limits at 0.001 mg/m^3 and some have more conservative STELs.³⁹

None of the standards, as they currently stand, take into account important factors such as particle size, solubility, and manner of breathing (through the nose or through the mouth). Medical scientists have long understood that the size of the particle and its solubility are determining factors in the fate of inspired Be particles. A recent study evaluated differences in lung deposition resulting from breathing through the mouth rather than through the nose. Mouth breathing can result in significantly more small particles being deposited in the alveolar gas-exchange region of the lung.²⁸ Another recent study concluded that the concentration of respirable particles less than $3.5 \mu\text{m}$ is probably a better measure than total mass concentrations for minimizing potential for CBD.⁶⁴ Due to significant differences in potential for causing CBD observed for various forms (salts, oxide, powders, solutions, solid, alloys, etc.), and particle sizes, there may need to be a whole series of TLVs. Having a TLV for copper beryllium, another TLV for metallic beryllium, and another for beryllium oxide or ore dust would probably make sense.⁶⁵

Data continues to accumulate regarding particle size distribution for varying machining operations involving Be and Be-containing metals. One study found that, of the particles generated by milling Be metal, 4-9% were smaller than $5 \mu\text{m}$.⁶⁶ Another study found that 21% of particles generated during the thermal processing of ammonium fluoroberyllate crystals 21% were smaller than $6 \mu\text{m}$.⁶⁴ Machining Be generated particles

with a mass median aerodynamic diameter of 7.9 μm plus or minus a standard deviation of 3.3.²⁸ The current researchers are agreed that more thorough studies of particle size distribution and solubility for varying processes are needed to inform the standards developing process.^{21, 28, 64}

ACGIH has published their intention to reduce their TLV for Be to 0.0002 mg/m^3 ⁽¹⁾, but factors other than total mass of airborne dust are still causing debate. In establishing an adequately protective recommended standard, the respirable fraction will need to be addressed, perhaps similar to the way the TLV was set for crystalline silica but with consideration for solubility.³⁶ On the other hand, since access to equipment to measure respirable particulates will not be as available as simple personal monitors employing pumps, cyclones and filter cassettes, perhaps total mass TLVs would be more practicable. While the ACGIH, NIOSH and OSHA are redrafting exposure limits, mouth breathing is another variable worth considering. Beryllium and other cancer-causing particulates are changing the way exposure limits have historically been established.²⁶

Whatever the exposure limits ultimately decided upon, those individuals who sample and analyze occupational atmospheres and interpret results will need to take particle size into consideration. A respirable particulate mass-TLV (RPM-TLV) has been suggested at 0.002 mg/m^3 for less soluble forms of Be and also for inhalable particulate mass-TLV (IPM-TLV) for soluble forms. The ACGIH (2003) book of TLVs and BEIs Appendix D: "Particle Size-Selective Sampling Criteria for Airborne Particulate Matter" provides recommended calculations for RPM and IPM (as well as thoracic particulate mass TLVs).³⁶

Sampling/Monitoring the Air in the Work Environment

Multicyclone samplers collecting air from the breathing zone are recommended for quantifying various particle sizes of airborne Be. Typically, air is sampled from the general area of worker activities as well as breathing zone sampling within a foot of the operator's head during peak exposure periods. Personal samplers for collecting IPM and RPM fractions are also available. Since the amounts to be captured are so minute, rigorous quality control must be applied to sampling, digesting and analytical techniques.^{67, 68} OSHA recommends analysis of air by the inductively coupled argon plasma (ICP) method (OSHA ID-125G & ID-206) using mixed cellulose ester filters (0.8 microns), sampling between 480 and 960 liters of air at a maximum flow rate of 2.0 L/min., preferably collected over an 8-hour workshift.⁶⁹ The NIOSH recommended method is by atomic absorption spectrophotometry in a graphite furnace, Method 7102, Issue 2, with an estimated limit of detection of 0.005 µg per sample.⁷⁰

Hazard Communication Requirements Pertain⁷¹

Employers involved in processes entailing Be-containing materials must assure employees are provided access to the pertinent material safety data sheets. A formal training program must be established and maintained by which Be-workers and Be-associated workers are educated regarding the hazards of Be exposure and how to protect themselves. They should be encouraged to avoid skin contact in addition to complying with regulated area and PPE policies. Workers should be trained on the proper use of necessary PPE, including respiratory protection and informed about the availability of

medical testing. Warning signs must be posted at approaches to regulated Be areas and labels must be affixed to Be materials, containers of Be-materials, Be-contaminated materials, waste, and scrap. An accurate inventory of all Be-containing materials must be kept current. Copies of results of industrial hygiene sampling for beryllium should be provided to, and discussed with the workers.^{27, 63} Additionally, copies of OSHA's Hazard Information Bulletin entitled *Preventing Adverse Health Effects From Exposure to Beryllium on the Job* should be provided to the workers.²⁷

Controlling Occupational Exposures to Beryllium

Beryllium production facilities have the greatest potential for worker exposure to powdered forms of Be, and therefore must have the most elaborate engineering controls. Pellets should be used in place of powders to the extent possible. Batch digesting processes, processing furnaces and blending of Be-containing powders are operations performed in totally enclosed vessels. Of course, the vessels must be opened as part of process or maintenance. Air sampling must accurately characterize the potential exposure to workers at those times of increased vulnerability.

Engineering Controls

Protective clothing and equipment must be provided and correctly utilized. Local exhaust ventilation equipment and air cleaning equipment must be properly designed and diligently maintained.^{27, 43, 112}

In selecting alloys for commercial products, less toxic metals should be substituted whenever possible. The number of workers having potential exposures to airborne Be particulates should be minimized.²⁷ Cutting operations in which beryllium and beryllium-containing alloys are shaped by processes such as lathes and milling machines should be carried out in enclosed environments using metal removing fluids or vacuum systems to contain metals dusts and fragments. Particulate Be suspended in metal working fluid can be removed by centrifugation. Air exhausted from the enclosed machining operations is cleaned using high efficiency particulate air HEPA filters before being returned to the machine shop room environment. Local exhaust ventilation is provided to control background levels of air contamination. Maintenance of filtration systems is essential in the overall effort to minimize potential for airborne exposures.^{27, 43, 112} If handling beryllium and beryllium compounds, nitrile rubber gloves covering hands and wrists are recommended,²⁰ along with disposable protective clothing adequate to prevent dusts or fumes from contacting skin.¹¹²

Action Level Prompts Interim Protection Strategies

The Department of Energy (DOE) has promulgated an action level of $0.2\mu\text{g}/\text{m}^3$ for their contractors whose operations involve Be-containing materials.⁶³ When performing maintenance operations or handling materials that may present a potential for exposure to beryllium dust or fumes above the action level, workers should be provided full protective clothing and full-faced air-purifying respirators with filtering capacity appropriate for the maximum level of airborne Be particulates potentially present in the work environment. In order to avoid exposure to concentrations greater than the PEL,

only the “most protective” respirators are permitted for use in concentrations exceeding 4 mg Be/cubic meter.¹⁰¹ Actually, for cancer prevention purposes, NIOSH recommends the “most protective” respirators for concentrations above 0.0005 mg Be/m³. Following the intervention or maintenance operation, workers should be decontaminated with HEPA vacuum, protective clothing doffed inside out; they should bathe and change into uncontaminated clothing, including uncontaminated street shoes. Any potentially contaminated clothing should never be worn outside of the facility or taken home, but should be laundered at the industrial facility.^{27, 43, 63}

Administrative Controls

Workers must not eat, drink, smoke, or apply cosmetics at their work stations. If skin becomes contaminated with Be-containing particles, the affected worker(s) should promptly wash with mild non-abrasive soap and lots of water. Following operations that may have generated Be particulates, the equipment and surfaces of the work area should be cleaned with HEPA vacuums. Contact lenses should not be worn when working with this chemical.² Shoes should be cleaned with dampened wipes or HEPA vacuums. Care should be taken to avoid releasing collected dust from the vacuum system when it is turned off and hoses are moved. Compressed air should not be used for blowing off parts or work surfaces in machining operations involving Be or Be-containing metals or ceramics.²⁷

Areas within a facility that have been shown, by air sampling, to be subject to airborne Be concentrations greater than the action level must be demarcated as regulated areas. To maintain Be-exposure as low as reasonably possible, access to regulated areas

must be restricted to those employees whose work requires them to go into the area and who are properly trained and equipped.^{27, 63} Employee exposure needs to be monitored on a regular recurring basis to assure that exposures remain below the appropriate occupational exposure levels. In operational and maintenance phases where personal protective equipment (PPE) is needed to avoid exposure, personnel should be monitored to assure necessary PPE is properly utilized.^{27, 43}

Medical Surveillance

It is required that all employers processing Be-containing materials and having contracts with DOE (and their contractors) must establish and maintain a medical monitoring program for current and former Be-workers and Be-associated workers (those whose work requires them to go into areas potentially exceeding action levels, like custodial workers). OSHA recommends a similar medical monitoring program for any employer of Be-workers.²⁷ Such medical surveillance programs should include baseline and periodic evaluations, as well as evaluations after any inadvertent exposure incident. If symptoms develop, or upon employee request, the employer should arrange for a BeLPT to determine possible Be-sensitization. Upon detecting Be-sensitization or CBD, the Occupational Medical Director must advise the employer and the affected worker should be offered a work transfer to an area of the operation not subject to Be exposure.⁶³

Additional Be literature about animal studies, first aid recommendations, environmental and transportation regulations can be found at Appendix B ([Summary of Animal Studies, First Aid, and Environmental Regulations](#)) of this paper.

Sampling Surfaces for Removable Beryllium Contamination

In addition to air monitoring, employers can add surface sampling to their arsenal of tools to minimize and eliminate worker exposure to Be. By keeping surfaces clean of accumulated dust, a potential source of inhalable contamination is removed and potential for skin contact is taken away. In the personal protective equipment (PPE) provisions of the OSHA regulations found at 29 CFR 1910.132, employers are required to assess workplaces for hazards in order to identify the need for PPE. Wipe sampling for surface contamination is a useful tool not only for determining where PPE may be required but also for identifying surfaces that require decontamination, verification testing for decontaminated surfaces, routine checks of housekeeping maintenance in work areas where hazardous materials may settle out on surfaces, and for checking the insides of air-purifying respirators or other PPE for possible breakthrough.⁷²

Wipe Sampling Methods Compared

Standardized wipe sampling for environmental surface contamination developed in large part due to reports of lead poisoning among children in government subsidized housing⁷³ that led to the Residential Lead-Based Paint Hazard Reduction Act, the provisions of which are enforced by U.S. Department of Housing and Urban Development (HUD).⁷⁴ Methods of sampling surface dust were evaluated for various surface conditions in a 1995 review conducted by EPA for HUD. That study noted that there was no correlation between the various methods of sampling for surface contamination. Wipe sampling methods reviewed included the Vostal Method (ca. 1974)

that used paper towels moistened with denatured alcohol or commercial moistened towelettes to wipe a square foot area inside a template or an entire windowsill. Lead was then eluted from the paper towel in dilute hydrochloric acid solution. The Farfel Method (ca. 1993) modified the Vostal Method by including paint chips that Vostal excluded and specified back-and-forth wipes twice in orthogonal directions. Farfel noted that removal efficiency was inversely related to surface roughness. The 1990 HUD Method was similar to the Vostal and Farfel Methods but specified total digestion of the wipe sample material and an additional wipe over the sampled area to improve removal efficiency. The Rabinowitz Method (ca. 1985) included the use of preweighed wipe sampling materials, making possible the determination of concentration of lead in the dust (as lead mass / total dust mass) in addition to surface loading in mass per area. The OSHA method, which allowed for either dry or moistened wipe materials, and a wiping pattern of concentric squares within a 100 cm² area was criticized for vagueness and failing to determine concentration. The Lioy-Weisel-Wainman (LWW) Wipe Method (ca. 1993) used either a rectangular 100 or 50 cm² template, and three preweighed 37 mm polyethylene draindisc filters as wipe materials. The entire surface area tested was wiped with three successive filters that were subsequently reweighed before being digested. The LWW was tested with two types of dust particulates (road dust and potting soil dust) on painted shelving, Formica, and wood paneling.⁷⁵

Wipe Sampling Identified Take-Away Beryllium

Wipe sampling has gained broader application as asbestos and additional metallic particulates such as nickel, cadmium, and beryllium have been identified as carcinogenic

contaminants anticipatable in surface residues associated with industrial processes. Although the health hazards associated with Be have been widely publicized since the 1940's, and Be-workers are routinely cautioned about hygienic practices to avoid carrying contamination away from the workplace, a relatively recent article showed that there is continual need for vigilance. In that study, Be-workers from an industrial facility were regularly provided with work clothing and shoes for use in the Be-processing areas. Showers were also available but to reach them required passing through the Be-work area. Many of the workers did not change shoes or clothes before leaving work in the afternoon. Wipe samples taken from worker hands and inside their automobiles revealed that Be particulate contamination was being carried away, especially on worker shoes as shown by samples from floor mats.⁷⁶

*Surface Contamination Limits
Tested by Wipe Sampling*

In the Chronic Beryllium Disease Prevention Program (CBDPP), DOE adopted 3 µg/100 cm² as the not-to-exceed housekeeping level of removable surface contamination based on a range of pre-existing best management practices in their various manufacturing and research facilities (see 9/01/04 memo of conversation with Paul Wambach in Appendix A, [Memoranda of Conversations](#)). This limit is intended to protect against respiratory exposures through disturbance and resuspension of beryllium-containing dust accumulated on surfaces in beryllium work areas.³ When facilities or equipment will no longer be used for working with beryllium-containing materials, the DOE established more stringent protective levels for decontamination. Prior to being turned over for other uses, room(s) and equipment surfaces must be cleaned of all

removable beryllium contamination to a not-to-exceed limit of 0.2 $\mu\text{g}/100\text{ cm}^2$ or the concentration of beryllium in the soils in the area of the facility where the equipment will be used.⁷⁷ Of course, as is hypothesized in this paper, differences in surface conditions, wipe materials, and solvents can make significant differences in the amount of surface contamination found by surface wipe sampling. As noted by an experienced professional in the area of beryllium decontamination, “sampling methods can be strategically selected to get the desired results. If you do not want to see removable surface contamination, a method can be selected that will minimize the removal efficiency.”⁷⁸

Though for the first five years of the CBDPP, DOE had no established standard method of collecting wipe samples, in September 2004, a decision was made to adopt ASTM D 6966-03 as the standard wipe sampling method for DOE facilities (see 9/24/04 Memo for Record in Appendix A, [Memoranda of Conversations](#)).⁷⁹ This ASTM method requires the use of moistened wipes meeting the specifications of ASTM 17920-03, but does not specify the wetting agent.⁸⁰ Wipes can vary in size from 200 cm^2 to 625 cm^2 and can be from 0.05 to 0.5 mm thick. The wiping pattern is similar to that described by NIOSH 9100 (see Figure 1, page 5) except, on the third pass, the edges of the area are wiped rather than repeating the “S” curve pattern across the whole surface.¹⁵

The CBDPP rule is open to potential dispute in situations where area soils have relatively elevated, widely varying Be content. There is no definitive guidance provided regarding which concentration would apply to the alternate release criteria provision.⁷⁷ A standard correlation between concentration (mass of Be *vs.* mass of total dust) and content per area of minimal depth would be needed in order to determine alternate release criteria based on concentration level of Be in soil at the specific release site. That would

require further research perhaps involving preweighed, moistened wipes, dehydrated after wiping, reweighed, then digested and analyzed for total Be. Knowing average concentration in the geographic area of release and concentrations found on interior surfaces would make an objective alternative release criterion tenable.

Methods of Analyzing Wipe Samples

OSHA method number ID-125G, “Metal and Metalloid Particulates in Workplace Atmospheres,” includes extensive information about wipe sampling.⁸¹ The method uses moistened Whatman paper, moistened Smear Tabs, or Ghost Wipes. The wipe is folded once, pressed onto the surface being sampled, and wiped in progressive concentric square patterns from outside edge of the 10 cm square template to the inside. The wipe is folded with contaminant-side in, and the concentric wipe process repeated at least three times. The wipe is folded a final time, contaminant in, and placed in a scintillation vial, sealed and sent to the lab for analysis along with a blank sample for ICP analysis. The OSHA Salt Lake City Technical Center used 100 cm² glass test plates to which aqueous solution containing 4 µg of beryllium was added, evaporated, and wiped with water-moistened wipes when testing the removal efficiency of their method. The researchers compared three sampling events involving different individuals using Ghost Wipes in the prescribed concentric squares wipe method. Their recovery rates ranged from 90 to 92.8% for one sampler to 92.2 to 100.6% for the second sampler, and 94.2 to 100.6% for a third sampling event. In other words, almost all of the beryllium surface contamination was removed from the glass using the distilled water-dampened Ghost Wipes.⁸² Of course,

the nature of the surface and the contamination residue would be expected to affect the recovery rates.

The OSHA Salt Lake City Technical Center has published *Evaluation Guidelines for Surface Sampling Methods* that specifies glass surface, numbers of wipe tests, adequate removal efficiency, numbers of blanks, and spikes, among other considerations. That same publication also recommended that appropriate solvents should be selected as the wetting agent for wipe sampling.⁸³

The EPA methods 200.7 (for water-borne sediments)⁸⁴ and 6010 (for extraction from solid wastes),⁸⁵ like OSHA's 125G, use ICP-AES and have a detection limit of 0.3 µg/l. EPA method 7090 for waters uses atomic absorption (AA) spectrophotometry and has a detection limit of 0.005 mg/l.⁸⁶ EPA method 7091 is used for extracting Be from soils, wastes, and groundwater and utilizes graphite furnace AA with a detection limit of 0.2 µg/l.⁸⁷

Taylor et al., list 22 methods for analyzing aqueous solutions for Be using colorimetric detection and another 8 fluorimetric methods.²³ Laser-induced breakdown spectroscopy instruments have been developed that are portable enough to be used in the field for analyzing metals (including Be) in soils and air. They operate like ICP-AES by kicking the elemental sample constituents into excited electron states. As the electrons fall back to their normal orbits, wavelengths characteristic of the elements are emitted. Los Alamos National Laboratory has developed a portable microwave-induced plasma atomic emission spectrophotometer for metal analyses of liquids and air with detection limits of 0.009 ng/ml and 0.21 µg/m³ respectively.²³

*Previous Dry and Water-moistened
Wipe Sampling Comparison*

Results from dry wipes and water-moistened wipes of surface beryllium contamination were compared in a study conducted by Lawrence Livermore National Labs. Analyses showed differences in removal using dry wipes and wet wipes in side by side comparisons. There were also differences when wiping with dry wipes followed by wet wipes over the same area. The researcher concluded that surface wipe sampling does not quantitatively remove all surface contamination; however there was reportedly no consistent correlation between wet and dry methods.⁸⁸ Two individuals with extensive experience at collecting surface wipe samples in former metal machining shops observed that surface loading of contaminant residues frequently differ significantly in side-by-side comparisons on industrial equipment and other surfaces.⁸⁹ Among other variables, proximity to operations and air currents over surfaces differ resulting in dissimilar deposition of surface contaminants. By holding surface loading constant in surface residues, these confounding variables were avoided in this present study.

The LLNL comparison study was presented in a Beryllium Health and Safety Committee (BHSC) meeting of DOE and their contractors in October 2001. At that same meeting, members debated the merits of having a standard method of wipe sampling for beryllium surface contamination.⁹⁰ Some may be reluctant to adopt a single approved method, such as OSHA ID-125G or ASTM 6966, because they have established precedence for using dry wipes at their facilities. Use of moist wipes could make it more difficult to achieve the housekeeping or release cleanliness criteria. Some sites having mixed beryllium and radioactive contamination object to using a dampened wipe sample

method because of water's interference with alpha particle detection.⁹¹ The controversy regarding surface sampling methods continued at the October 2004 BHSC meeting, where prevention of skin exposures was also a repeated topic of discussion. The outline of a proposed study to compare methods of surface sampling methods was presented at that recent BHSC meeting. The proposed study would study removal of BeSO₄ solution residues from smooth, slightly rough, and porous surfaces using dry Whatman paper, Ghost Wipes, dry linen paper, methanol wipes, vacuum method, and a pressure controlled method.⁶

Wipe sampling may not always be the best method of determining levels of surface contamination depending on the character of the surface being sampled. The EPA-HUD Review of Surface Sampling Methods provided a comparison of various non-wipe sampling methods such as tape lifts and vacuum techniques.⁷⁵ In the CBDPP Preamble, DOE authors agreed that more research is needed and encouraged development of new technologies like direct reading instruments.⁹²

CHAPTER 3

METHODOLOGY

The list of materials and equipment used for this research and the detailed step-by-step test plate preparation and sampling protocol can be seen immediately following the “Summary of Method.” While the salient points are covered in the summary, there are significant details noted in the ensuing step-by-step sections.

Summary of Method

According to the Evaluation *Guidelines for Surface Sampling Methods*, wipe sampling removal of $\geq 50\%$ of contaminant from an ideal surface (glass) is considered adequate. That same publication also recommended that appropriate solvents should be selected as the wetting agent for wipe sampling.⁸³ For this project, the surface and the residue were deliberately not “ideal.” The intent was to compare three wipe sampling methods in current use within the DOE complex on surfaces and residues typically found in metal working shops. Dry wipe sampling with Whatman smear tab filter paper was in use at some DOE facilities, water-moistened wipe sampling with Ghost Wipes was practiced elsewhere within DOE, and methanol-moistened gauze wipes were used at still other DOE sites. The “typical” surface selected by consensus (see 9/24/04 memo for record in Appendix A, [Memoranda of Conversations](#)) for this comparison study was a flat painted surface. Test plates were prepared using 100 mm X 15 mm Pyrex Petri dishes with interior surfaces spray painted with a bond coat primer. To achieve uniform

deposition over the test plate surface, precisely-measured aliquots of solution with known Be mass were transferred by pipette to the test plates and subsequently evaporated (see Photo 6 in Appendix C, [Photographs](#)). Similar to deposits on kitchen cabinets over cooking ranges, a slight oiliness is common on surfaces of metal working shops where fugitive oil mists accumulate over time. A small amount of a semi-synthetic metal working fluid (solubilized mineral oil) was added to the solution used to “contaminate” the test plates. Sixteen test plates for each wipe method (dry, water, and methanol) were chosen based on a recommendation from a professional statistician (see 9/01/04 memo of conversation with Jim Wyckoff in Appendix A, [Memoranda of Conversations](#)).

The residue applied to the painted surface of the test plates was formulated to contain $1 \mu\text{g} \pm 0.002 \mu\text{g}$ Be and 0.1 ml of a metal working fluid concentrate per 78.54 cm^2 (the equivalent of $1.25 \mu\text{g}$ Be and 0.13 ml of a metal working fluid per 100 cm^2). The oily film typically oxidizes after extended periods producing a slight tackiness. Test plates were baked at 230°F for 25 minutes to simulate the aging effect (see Photo 7 in Appendix C, [Photographs](#)). Control and field blanks were collected in an attempt to control for possible sources of contamination inherent in the preparation, and sampling processes (see 9/30/04 memo for professors, et al. at Appendix A, [Memoranda of Conversations](#)). Control blank plates were prepared exactly like test plates except the “contamination” aliquots contained only 0.1 ml of metal working fluid in 10 ml of deionized water and no Be. Field blanks were collected in triplicate by folding the respective unused wipe materials and sealing them in test vials, dated and numbered like other test vials.

All test and control plates were wiped with uniformly consistent patterns (see Figure 2) and pressures using the three wipe materials typically used at DOE facilities. The Whatman paper was too stiff to bend into the edges of the test plates. It was pre-conditioned by wadding into a tight ball and then unwadding (see Photos 8, 9, & 10 in Appendix C, [Photographs](#)). In this more supple form it could be more effectively pressed into the inside angle of test plate bottoms and side walls (see Photo 11 in Appendix C, [Photographs](#)).

Test and control plates had slightly reddish-brown deposits visible on top of the white painted surfaces (see Photo 6 in Appendix C, [Photographs](#)). The color was from the metal working fluid (see Photo 2 in Appendix C, [Photographs](#)). The first set of test plates to be sampled was the series wiped with dry Whatman paper. The paper firmly pressed against the surface did not stick to the residue or meet with any remarkable resistance when wiped across the painted surface. Those observations led the researcher to conclude that the oily deposit had not been baked long enough to achieve the intended tackiness (see “Late August 2004” entry at beginning of Appendix D, [Beryllium Wipe Correlation Lab Journal](#)). The red-brown surface contamination came off on the paper without noticeable resistance. Use of the same pressure with the Ghost Wipes for the next series of plates squeezed water out of the folded wipes. Water was sopped up to avoid loss of removal efficiency and wiping pressure was subsequently reduced with final folded moist wipes (including methanol gauze wipes). The water-moistened wipes dissolved the surface residue to a surprising extent. There was no significant visible difference between the removal accomplished by water-moistened and alcohol-moistened wipes. All used wipe materials were placed in lab vials, sealed, marked with date and

time and transferred to the analytical lab (see Appendix E, [Chain of Custody](#)). A second lab with more sensitive instruments reanalyzed the digested samples returned from the first lab. Analytical results were used to calculate comparative removal efficiencies, the variances of which were analyzed for statistically significant difference using one-way analyses of variance (ANOVA) and Multiple-Fs post hoc testing.

Materials and Equipment

The materials, equipment, analysis, and facilities needed for the study were provided by the NNSA's Kansas City Plant operated by Honeywell FM&T. This included the following:

- 1 clean dehydrator
- 1 uncontaminated drying oven
- 58 Pyrex Petri dishes 100mm diameter, 15mm deep
- Zinsser Cover Stain® Primer Sealer Stain-killer, Bond Coat spray paint
- 23 Ghost Wipes
- 23 Whatman Grade 42 smear tabs
- 24 digestible gauze methanol wipes
- 72 pairs of latex gloves
- 72 sample tubes
- 10 ml of 10 µg/ml standard Be solution
- distilled deionized water
- a small amount of MWF concentrate
- glassware (volumetric flask, pipette)

- micropipette to deliver 0.1ml spikes of 10 µg/ml standard Be solution
- analysis of 72 samples

Step-by-Step Preparation of Test plates

1. Coated each test plate (100 mm X 15 mm Pyrex Petri dishes) with Zinsser Cover Stain® Primer Sealer Stain-killer, Bond Coat spray paint (see MSDS at Appendix F, [Material Safety Data and Specification Sheets](#)). This provided a typical flat painted surface – representative of the surfaces decontaminated in actual metal machining shops.
2. Prepared 2L of a dilute metal working fluid solution containing 0.10 ml of concentrated CIMSTAR 3700T (see MSDS at Appendix F, [Material Safety Data and Specification Sheets](#)) in 10 ml, i.e., 1:100MWF. Pipetted 10 ml CIMSTAR 3700T concentrate into volumetric flask and added deionized water to the mark.
3. Prepared 1 liter of 100 µg Be/L MWF solution by pipetting 10 ml of 10 µg Be/ml standard solution into a 1L volumetric flask and filling to mark with 1:100 MWF. The stock standard solution was Inorganic Ventures certified custom-grade standard GN 30-1, with 100 µg/ml each: Rb, Tl; and 10 µg/ml each: Al, Be, Fe, Li, Ni, and Na; Lot No: W-MEB161016; Density 1.015 g/ml; components: water (CAS# 7732-18-5), and nitric acid (CAS # 7697-37-2); expiration date 1 Nov 2004 (See Specification Sheet and MSDS at Appendix F [Material Safety Data and Specification Sheets](#)). We added a little 1:100 MWF solution to the volumetric flask before transferring the standard Be solution to the flask. When the Be solution was added, there was an immediate precipitation reaction observed by

cloudiness. We brought the solution in the volumetric flask to the line with 1:100 MWF. After an hour and a half, there was no apparent flocculation or settling out – just uniformly milky suspension. The KCP ICP analyst ran the solution through ICP-AES to determine if there had been a loss of Be in solution. The results, 0.0978 mg/l, showed that the Be concentration was still essentially at the target concentration of 0.1 mg/l (see [Post Precipitation Analysis Results](#), at Appendix G). Pipette accuracy was $\pm 0.02\text{ml}$.

4. Donned powderless latex gloves.
5. Cleaned the lab bench work area with new methanol gauze wipes to remove any loose material.
6. Wiped off and arranged the following on the lab bench:
 - Box of powderless latex gloves,
 - two 10 ml pipettes,
 - pipette suction syringe bulb,
 - micropipette for 0.1ml,
 - volumetric flask with 1:100 MWF,
 - volumetric flask with 100 μg Be/L MWF solution,
 - squeeze bottle filled with deionized water,
 - three racks of sample vials containing 57 sample vials from laboratory,
 - 20 packets of Ghost Wipes, and
 - box containing new grade 42 Whatman filter paper 125mm circles.
7. Removed painted test plates from cartons in opposite bench shelving, wiped all dish surfaces with methanol wipes to remove any stray paper dust, etc., and laid

- the dishes out in three groups of 19 each on the clean surface (see photograph 1 in Appendix C, [Photographs](#)).
8. Wiped a permanent marker for writing on glass with a methanol wiper. And placed it on the clean work surface.
 9. Removed and disposed of gloves
 10. Donned powderless latex gloves.
 11. Labeled painted test plates: D1, D2, D3,...D19; W1, W2, W3,...W19; and A1, A2, A3,...A20.
 12. Labeled sample vials provided by analytical lab D1, D2, D3,...D23; W1, W2, W3,...W23; A1, A2, A3,...A24; and Q1, and Q2.
 13. Removed and disposed of gloves.
 14. Donned powderless latex gloves.
 15. Prepared spikeless control blank plates. From the 1:100 MWF solution, added 10 ml aliquots to each of 9 test plates labeled D17-19, W17-19, and A17-19.
 16. Prepared test plates. From the 100 µg Be/L-1:100MWF solution, added 10 ml aliquots to each of 48 painted test plates labeled D1, D2, D3,...D16; W1, W2, W3,...W16; and A1, A2, A3,...A16 (see photos 3 and 4 in Appendix C, [Photographs](#)). Based on pipette accuracy (± 0.02 ml printed on pipette) this aliquot would contain 1 ± 0.002 µg Be.
 17. Evaporated the 58 test plates in a 145°F evaporation oven 16 –to- 24 hours (see Photo 5 in Appendix C, [Photographs](#)).
 18. Collected 1 pre-use wipe of the drying oven with alcohol wipe placed in sample vial number Q1, sealed and marked with date and time.

19. Baked test plates at 230°F for 25 minutes to produce test plates having 1.0 µg of Be surface contamination associated with a slightly tacky oil-containing residue. The baking at 230°F for 25 minutes simulates the oxidation of metal working fluid (fugitive oil mist) residues that normally occurs over a period of several years. The oxidation process changes the texture from slick to slightly sticky. (See oven calibration record in Appendix H, [Equipment Performance and Calibration](#)).
20. Removed test plates from oven and allowed to cool for 2 hours prior to wipe sampling.
21. Collected 1 post-use wipe of the drying oven with alcohol wipe place in sample vial number Q2, sealed, and marked with date and time.

Sampling Protocol

1. Collected spikeless dry wipe samples from test plates marked D17-D19 using 125 mm round Whatman grade 42 filter papers per modified OSHA 125G method, placed wipe papers in sample vials provided by analytical lab labeled D17-D19, sealed them, and marked date and time. The modified wipe technique consisted of the following steps (also see Figure 2 below):

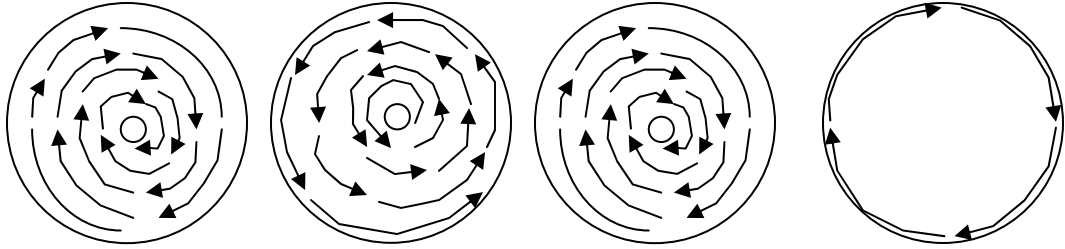


Figure 2. Modified OSHA Wipe Sampling Technique

- a. Don powderless latex gloves.
- b. Remove wipe material from its container and if folded, unfold it, if very thin and of a large enough area fold in half once prior to wiping surface.
In the case of the Whatman filter papers, due to stiffness, they were preconditioned by wadding into a tight ball and then unwadding to provide a more flexible wipe for accessing the recessed edge of the Petri dish test plates (see photos 9 and 10 in Appendix C, [Photographs](#)).
- c. helical wipe the test plate surface in a clockwise direction from outer rim to center, covering entire bottom surface in about four cycles,
- d. fold wipe material in half with used surface inside,
- e. helical wiping in counterclockwise direction from center to outer rim,
- f. fold wipe material a second time,
- g. helical wiping in clockwise direction from outer rim to center
- h. fold wipe material a third time,
- i. press corner of folded wipe into the outer corner of outer rim and wipe the circumference,
- j. roll contaminated edge into the folded wipe material,

- k. place wipe in new sample tube for transport to lab.
 - l. Remove and dispose of gloves
2. Collected spikeless water-moistened wipe sample from test plates marked W17-19 using Ghost Wipes per modified OSHA 125G method, placed used Ghost Wipes in sample vials provided by analytical lab labeled W17-W19, sealed them, marked date and time.
 3. Collected spikeless alcohol-moistened wipe samples from test plates marked A17-A19 using alcohol-moistened wipes per modified OSHA 125G method, placed used alcohol-moistened wipes in sample vials provided by analytical lab labeled A17-A19, sealed them, and marked date and time.
 4. Prepared dry field blanks by folding and placing new unused Whatman grade 42 125 mm circles in the laboratory vials labeled D20-D22, sealed them, and marked with date and time. Fresh latex gloves were used for the preparation of each field blank.
 5. Prepared Ghost wipe field blanks by folding and placing a new unused Ghost Wipes in the laboratory vials labeled W20-W22, sealing them, marked date and time.
 6. Prepared alcohol-moistened field blanks by folding and placing new unused alcohol-moistened wipes in laboratory vials labeled A21-A23, sealed them, marked date and time.
 7. Collected a paint control blank wipe sample by wiping the Petri dish marked A20 with an alcohol wipe, then folding and placing it in the laboratory vial labeled A20, sealed it, and marked date and time.

8. Collected 15 dry wipe samples from test plates numbered D1-D15, using 125 mm Whatman grade 42 filter paper circles, placed in sample tubes numbered D1-D15, sealed, marked with date and time.
9. Collected 1 dry wipe sample with an additional $1 \mu\text{g} \pm 0.008 \mu\text{g}$ Be spike by wiping Petri dish number D16 with a dry filter paper, then using a micropipette to add 0.1 ml of $10 \mu\text{g Be/ml}$ standard solution to sample in the lab vial, sealing it, and marking with date and time (see micropipette product specification at Appendix H, [Equipment Performance and Calibration](#)).
10. Collected 15 Ghost wipe samples from test plates numbered W1-W15, using Ghost Wipes, placed in sample tubes numbered W1-W15, sealed, marked with date and time. The water was surprisingly effective at removing the visible residue from the painted test plates. The light reddish-brown Be/MWF deposit seemed to dissolve readily and appeared to be mostly removed by the wiping operations. Another surprise: trying to use the same pressure as with the dry filter papers proved impossible. After the Ghost Wipes were folded a couple of times that much pressure squeezed the water out of the material. To leave water behind would have resulted in a loss of removal efficiency. I sponged up the water so as not to leave it behind.
11. Collected 1 Ghost wipe sample with additional $1 \mu\text{g}$ Be spike by wiping Petri dish number W16 with a Ghost wipe, then adding 0.1ml (using micropipette) of $10 \mu\text{g Be/ml}$ standard solution to sample in the lab vial number W16, sealing it, and marking with date and time.

12. Collected 15 dry alcohol-moistened wipe samples from test plates numbered A1-A15, using alcohol-moistened wipes, placed in sample tubes numbered A1-A15, sealed, marked with date and time. The same squeezing out happened with the methanol wipes as with the Ghost Wipes. I reduced the pressure somewhat (relative to that used with the dry wipes) as not to leave Be-bearing wetting agent behind and thus lose removal efficiency.
13. Collected 1 alcohol-moistened wipe sample with additional 1 µg Be spike by wiping Petri dish number A16 with an alcohol-moistened wipe, then adding 0.1 ml (using micropipette) of 10 µg Be/ml standard solution to sample in the lab vial number A16, sealing it, and marking with date and time.

Analytical Method

The wipe samples were analyzed using EPA method 6010B “Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES).”⁸⁵

The lab was informed that the samples originated from matrices common to surface contamination samples from the Kansas City Plant (KCP) with a potential for Al, Fe, Li, Ni, Na, Rb, and Tl, in addition to Be. The analytical lab provided their spike recovery protocol documentation and Be calibration curve for 10/15/04 (see Appendix I, [Lab Analytical Quality Control Documents](#)).

Wipe sample media were initially sent to a contracted analytical laboratory with a 0.1 µg limit of detection. When the results returned with almost the entire dry wipe series “non-detect,” the Honeywell FM&T Analytical Laboratory agreed to reanalyze the digestates returned from the other lab using ICP-AES-Mass Spec equipment with

sensitivity down to 0.00001 µg. The vials were set up in an automated carousel arrangement with standard concentration control vials set to run after approximately every 15 sample vials to assure the instrument remained in calibration throughout the run. The instrument was calibrated to read ppb (µg/1000 ml), but the wipe media were digested and brought to a volume of 100 ml, so the ppb reading was multiplied by 0.1 to yield the total µg present on the wipe medium. According to the *Evaluation Guidelines for Surface Sampling Methods*,⁸³ the reliable quantitation limit (RQL) is about ten standard deviations greater than the response of the medium blank. The mean reading for the blanks (n=16) for this analysis was 0.005706 and the standard deviation was 0.001383, therefore the RQL was calculated as $0.005706 + 10(0.001383) = 0.0195$ µg, which was approximately four to five times less than the lowest sample results.

CHAPTER 4

ANALYSIS OF DATA

The results of the analyses are shown in Appendix J, Table 1. [Sampling Results](#). The removal efficiency rates were calculated for the different wipe materials as $[(\text{Be}_{\text{Mass}}$ on Wipe Medium) / (Be_{Mass} present on test plate)] X 100. The mean removal efficiency rates (n=15 for each method) were as follow:

Dry wipes: 9.33 % (S.D.= 2.80)

Water-moistened wipes: 22.97 % (S.D.= 6.05)

Alcohol-moistened wipes: 50.62 % (S.D.= 9.54),

as shown on the histogram in Figure 3:

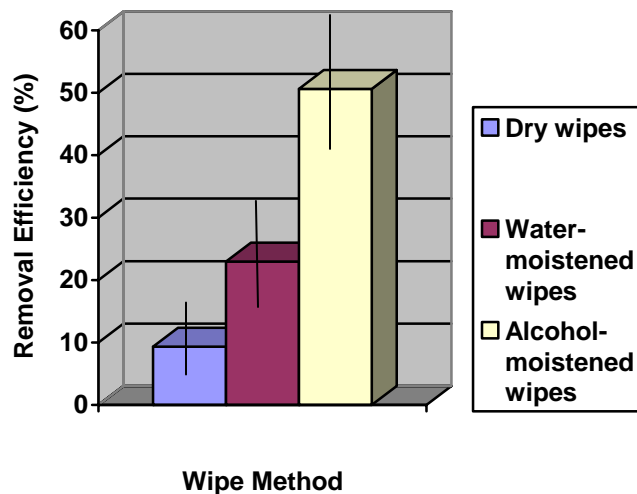


Figure 3. Mean Removal Efficiencies (%)

Removal efficiencies for dry, water-moistened, alcohol-moistened wipe sampling were also plotted as a frequency distribution bar chart. Values along the x-axis are removal efficiency range intervals and the heights of the bars reflect the numbers of sample per range, see Figure 4. This visual aid illustrates the relatively tighter distribution (less variance) of removal efficiencies experienced with dry wipe sampling. This may be partially attributable to the fact that while moistened sampling techniques removed more of the surface contamination, they also left behind varying amounts of liquid.[†] It would appear reasonable that the greater the solvent effect of the wetting agent, the more contaminant would be contained in the moisture left behind. As noted in steps 12 and 14 of the Sampling Protocol section of Chapter 3 (pages 61 & 62), the pressure used with the dry wipe sampling squeezed liquid from the wet wipes. For that reason, manual pressure was reduced for the water and alcohol-moistened wipe samples. Imperfect control of wiping pressures and differing moisture content in wipe materials could account for variable quantities of liquid left behind and hence more diverse removal efficiencies.

[†] F-max test revealed a lack of homogeneity of variance, which was interpreted as an indication of divergent population characteristics when comparing dry with moistened wipe efficiencies. Dry wipes lack a key variable inherent with wetted wipes, i.e., left behind moisture.

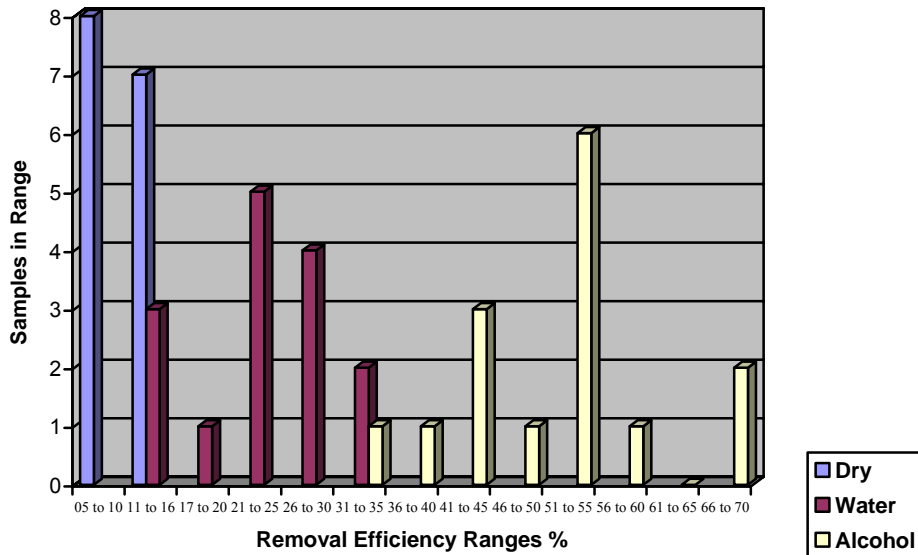


Figure 4. Frequency Distribution of Removal Efficiencies

The sampling results were analyzed using a single factor Analysis of Variance (ANOVA) showing that there were significant differences in the removal efficiencies of the three wipe sampling methods being compared [F=146.87 (2,42), P = 1.1E-19, SIG]. ANOVA tabulation is at Appendix K, Table 2. [Analysis of Variance](#). Post Hoc Multiple-F analysis of the test results (Appendix K, Table 3. Mean Removal Efficiency Differences) showed that, as predicted, water-moistened wipe sampling had significantly greater removal efficiency than dry wipe and alcohol-moistened wipe sampling had significantly greater removal efficiency than water-moistened wipe sampling ($p < 0.01$).

CHAPTER 5

CONCLUSIONS AND DISCUSSION

Removal efficiency was tested by wipe sampling without a wetting agent, as compared with water-moistened wipe material, as compared with methanol-moistened wipes. Analysis indicated that methanol-moistened wipe sampling removed significantly more Be/oil-film surface contamination than water-moistened wipes, which removed significantly more residue than dry wipes. Whereas moistened wipe methods removed significantly more surface contamination, they apparently also left behind varying amounts of Be in moisture remaining on sampled surfaces. The issue of leaving behind Be in moisture remaining on surfaces sampled with moistened wipe materials could be resolved by adding a step to dry the surfaces with ashless filter paper to be included in the sample vials for digestion along with the moistened wipe materials. This modified sampling procedure would provide more consistent and accurate information regarding levels of surface contamination when making decisions regarding the need for decontamination.

Comparing Wipe Methods Has Little Significance Apart from Contact Sensitization

Removal efficiencies varying with the nature of surfaces, residues, and wetting agents only have significance if the portions remaining after the wipe sampling genuinely present a health hazard. It could be argued that even the method with the least removal

efficiency removes most of the surface contamination that poses an inhalation hazard. After all, the CBDPP was developed with the express intent of eliminating the risk of breathing Be particulates in occupational environments. The limits on removable surface contamination focused on dusty accumulations that could be resuspended if disturbed and pose an inhalation hazard. If dry wipe sampling removes the majority of disturbable surface contamination, some would question the need for moistened wipe methods. Of course, dry wipe sampling can expose the one doing the sampling to disturbed dust, and reduce removal efficiency by resuspension of particles. For those reasons, it would be advisable to use a dampened wipe material. Alcohol moistened wipe sampling may well remove more Be from surfaces having a tacky oil film, but the additional Be removed would have little, if any, potential to become airborne and present an inhalation hazard. Some might argue that a dirty surface oil film might be unsightly and just smear when wiped, but it would not present a health hazard because there is no potential for inhalation. Oil mist residues could essentially encapsulate the Be surface contamination making it less “removable” by “casual contact” (per CBDPP definition).⁷ If, in fact, Be-sensitization via skin contact is not a valid hazard, there would be little reason to be concerned about surface contamination that is not removable by either dry or water-dampened wipe sampling.

Conclusions Hinge on Skin-Mediated Sensitization

Though research and practical findings since the promulgation of the CBDPP have been extremely convincing, some maintain that the role of skin contact in Be

sensitization remains theoretical. This present study is founded on the proposition that corporate management can ill afford to ignore such excellent evidence as that provided by the research of Tinkle et al.,⁸ and the dramatic drop in sensitization experienced by Brush Wellman following implementation of skin protection policies.^{9,10} Those responsible for decontamination of facilities and equipment in the interest of human health must give serious consideration to the possibility of skin-mediated Be-sensitization. Be-sensitized individuals are at greater risk of developing CBD even if not occupationally exposed to Be (see [Non-occupational Exposures to Beryllium](#)). The choice of wipe sampling materials, including wetting agents, becomes important if skin contact plays a valid role in Be disease conditions. The rest of the conclusions and discussion presented in this paper will address the issues of wipe sampling methods from the perspective that skin contact with a somewhat oily surface residue containing Be can contribute to sensitization.

The results of this study lead to the conclusion that dry wipe sampling may identify less than a tenth of the actual Be on surfaces having a residual oil film. Alcohol-moistened wipe sampling may be expected to remove approximately half of the surface Be contamination associated with such oil-mist residues. That is more than twice the amount that would be found with water-moistened wipes from the same type of surface and residue. Therefore, it is clear that wipe-sampling without or with inappropriate wetting agents can lead to misinformed decision-making regarding the need for decontamination and potential for dermal-mediated health effects.

Though skin-mediated Be sensitization is almost certainly a real possibility, the levels of surface contamination required to consistently trigger immune response in

genetically predisposed individuals has not been established. Even the use of optimal wetting agents cannot be expected to result in 100%-efficient wipe sampling in actual practice. There will always be some level of surface contamination that is not removed by even the best non-destructive sampling methods. In this present study, methanol-moistened wipe sampling provided the greatest removal efficiency but still left about half of the Be contamination behind. It was possibly adsorbed onto the surface coating. Whereas there is no certainty that the remaining not-so-removable surface contamination poses any real threat to unprotected users, there would probably be no ethically-supportable rationale for using less effective sampling methods.

When a decontaminated facility or piece of equipment previously used for Be work still has any remaining oil film residual, dry wipe sampling would be an inappropriate method for verification of the release criteria specified in 10 CFR 850.31.⁷⁷ Whereas removable dry dusty surface contamination may be less than $0.2 \mu\text{g Be}/100 \text{ cm}^2$, Be contamination could still be present in an oil film at levels 10 times the release criteria. Likewise, wipe sampling with water-moistened materials could yield deceptively low results when surface contamination includes some degree of oil mist residue. A decontaminated surface validated with a water-moistened wipe sample at the $0.19 \mu\text{g}/100 \text{ cm}^2$ level could still contain over four times the Be specified by the CBDPP release criterion if an oily film is present. Unprotected users of equipment or facilities with Be/oil-film surface contamination would be at greater risk of Be-sensitization.

As facilities change methods among the three compared in this study, these results could be used for approximate correlations. Where facilities transition from methanol-moistened to water-moistened wipe sampling, old data and new data could be correlated

based on the fact that surface contamination removable by the previous (methanol) method equals approximately two times the surface contamination removed by water-moistened wipe sampling (where oil film is present). More conservative criteria for housekeeping and release may be appropriate when surface contamination includes an oil film.

Other practical uses of the results of this study could include selection of water-moistened or alcohol-moistened wipe materials based on the historical use of the facility/equipment and the observed nature of the surface contamination. If deposits are dry and dusty water-moistened wipes would probably be appropriate, whereas methanol wipes would be more appropriate if an oil film is present. A more conservative approach, (most protective of human health) may be to adopt alcohol-moistened materials as the standard wipe sampling method.

Evidence for Be sensitization via skin exposure argues in favor of wipe sampling with wetting agents that provide greater residue removal efficiency. In the case of surfaces contaminated with Be, together with fugitive oil mist residues typical for metal working shops, this study indicates that methanol is a better wetting agent than water. Selection of inappropriate wetting agents can jeopardize the health of current Be workers as well as future users of facilities or equipment released according to the provisions of CBDPP regulations.

APPENDIX A

Memoranda of Conversations

8/31/04

Memorandum of conversation with: Lloyd Lazarus
 Subject: Beryllium Wipe Research Project

I briefly described the proposed research: Flood test plates with known concentration and volume of metal-working fluid beryllium solution, dry in oven, then compare dry wipes, distilled water wipes, and methanol wipes. Then I asked him what form of beryllium he would recommend.

He told me about Bob Bitter's Black Belt project to prove in their ultra-sonic cleaning process. In that project, they used a petroleum jelly with a know concentration of beryllium. Tape around a 100 cm² stainless steel plate then apply beryllium petroleum jelly to tape thickness with a squeegee – ultrasonically clean the plate – then wipe with 2 or 3 wipes to determine cleanness.

Lloyd described problems with various wipe materials versus nature of residue. The metal-working fluid residues in the plant are dried in place. Distilled water moistened Ghost Wipes do not cut the residue unless laid on the area to be sampled allowing time for residue to dissolve/loosen – then the Ghost wipe can scrub a little and remove the dried on residue. Paper wipes tend to shred or roll up when scrubbing the tacky surface contamination residue. He believes that using dry paper wipes in Crib 14 would not have shown any removable contamination (would be more likely to leave more paper on the surface than remove residue). Alcohol wipes typically cut through the residue quickly.

Decontamination of machining parts exposed to metal working fluid (water-oil-detergent) is an entirely different proposition than dried on residues on room surfaces. When placed in the metal working fluid environment, residues are removed by the washing action of metal working fluid.

He proposed that the study should also consider the material of the wipe: dry filter paper vs. the gauzy materials used with ghost and alcohol wipes. The filter paper tends to break when bent. Use of locking forceps to handle the wipes would cause filter paper to crack and tear apart versus the fabric type wipes.

He has experimented a little with BeO powder suspended in oil, baked in 200° oven – never could get dry. He recommended using straight 9704-2454 (pink semi-synthetic) metal working fluid concentrate to prepare a stirred suspension of BeO. He will put some in a Petri dish, put it in an oven and let me know how well it dries.

.....
 9/1/04

Memorandum of phone conversation with: Paul Wambach DOE/EH-6, 301-903-7373,
paul.wambach@eh.doe.gov

Subject: Background of the 3 and 0.2 µg/100 cm² removable surface contamination limits

I briefly introduced myself and my proposed research to compare methods of wipe sampling.

Paul explained that the 3 ug limit goes back to an earlier AIHA guide dating from the 1980s and 1950s era that stipulated 25 $\mu\text{g}/\text{ft}^2$. These were empirical levels usually achievable at Be machining shops.

The 0.2 μg limit came from poling the complex, asking “what’s possible?” The 0.2 μg was basically established by consensus assuming starting with a space or article that was contaminated and subsequently thoroughly cleaned.

The Health Physicists don’t like wet wipes in cases where surface contamination contains both beryllium and radioactive particles. The water masks particle detection.

The preamble to the Final Rule is available on the TIS website @ <http://www.eh.doe.gov/be/>

9/1/04

Memorandum of conversation with: Jim Wyckoff
Subject: Statistically derived sample size

Null hypothesis proposes that the two methods of swiping (wet vs. dry) provide the same results

The Alternate hypothesis proposes that the two methods provide different results.

He explained that the α value (the probability of finding the null hypothesis false when it is actually true) should be greater than the β value (the probability of finding the alternate hypothesis false when it is actually true). He explained them using the analogy of finding a guilty person innocent vs. finding an innocent person guilty.

Using a sample size of 18 would result in $\alpha = 0.1$ and $\beta = 0.1$ with a difference of 1.73 μg between the wet and dry wipes assuming 3 μg loading. Standard deviation at 1.73.

When all was said and done he recommended a sample size of 16 wet and 16 dry. If using 3 wipe materials, he would recommend doing 6 each, then if there is no conclusive consistent correlation could increase the sample size.

9/10/2004

Garrett, Bill, & Dan -

Thanks for taking time to look over my rough draft and estimate of cost. I've noticed some inconsistencies in that document. For example, in the estimate BeO is listed but in the text Be₂(PO₄)₂ is proposed. The objective is to prepare test plates with a known amount of beryllium. I had considered using a suspension of beryllium oxide, but subsequently decided a soluble form would provide a more precisely transferable aliquot. When talking about 0.5 micrograms of beryllium per test plate - use of BeO might require splitting a grain of powder. If

there is a stock of soluble beryllium salt in the plant that I can use, it won't matter if it is phosphate, sulfate, or whatnot. Surely, there is a beryllium reagent of some sort in plant for use in spiking samples, etc.

I've been trying various surface treatment for the test plates. Using 100mm Petri dishes still seems like a reasonable approach except for the fact that many of the surfaces being decontaminated are not glassy. So I found that spray painting them with something like Kilz provides a flat painted surface more typical of the surfaces being decontaminated.

Then to get a little metal working fluid onto the surface together with the beryllium contamination has been another issue. I've tried:

- pouring in a small amount swirling it around to coat the bottom and draining off excess, dehydrating at 145 degree F 24 hours. That was too much metal working fluid and it remained fluid.
- olive oil and powdered sugar at 145 degree F but that also remained too liquid. Might have been able to achieve the tackiness I was looking for, but some could argue that we don't use olive oil in our metal working operations. (Not representative of a typical industrial surface residue). Decided I needed to try again with metal working fluid.
- brushed on a very thin coat of metal working fluid over painted surface, baked at 400 for 30 minutes. Too hot too long.
- brushed on thin coat of metal working fluid over paint, baked at 250 for 15 minutes. Not bad. Still probably a little too much metal working fluid but the texture was close to what I believe to be more typical of aged residues we encounter in our efforts to decontaminate.

For the test plates to get just a touch of metal working fluid, I think it will be better to add the MWF to the Be salt solution (about 0.5 - 1 ml of concentrate per Petri dish) pipet into the Petri dish enough of a volume of Be salt solution to provide 0.5 micrograms per test plate, dehydrate at about 200 F, and then bake at 250 for 15 minutes.

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9/24/04

MEMORANDUM FOR: Record

SUBJECT: Beryllium Wipe Sampling Research

Of 30 samples of painted surfaces provided by Mark Smith of the Paint & Plasma Lab, Dan Ham, Mike Davis, Garrett Wilkie, and Bill Frede independently picked the same sample when asked to pick one surface texture that would be a typical & representative surface encountered in decontamination efforts in the Plant. It was conceded that no one surface can represent all the surfaces being decontaminated. Unpainted steel, painted metal and painted walls vary in surface texture. The surfaces targeted by the CBDPP would predominantly be painted and a semi-gloss rolled-on paint texture would probably approximate the average surface. That would generally describe the consensus choice of the four individuals, which Mark identified as Dupont Teflon S (Teflon-filled Epoxy Powder Coating - Black).

Bill would prefer that the study compare actual wipe sampling materials, i.e., compare dry Whatman filter paper, Ghost Wipes, and the alcohol wipes they have been using. I agreed.

Bill suggested using a weight to maintain equal pressure for all wiping. I like the idea – but need to figure out how to do it. Bill also suggested that I contact Belinda Thompson or Eric Grother of the Materials Engineering Lab regarding simulation of aging. They do aging studies.

Bill was in a select group in Washington D.C. last week. They were locked in until they came up with a consensus method for taking wipe samples. They agreed on ASTM D 6966-03, *Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals*, which uses a wipe material specified in ASTM E 1792 – 03, *Standard Specification for Wipe Sampling Materials for Lead in Surface Dust*.

Took Petri Dishes to Mark Smith. He asked for a charge number. Called Garrett, he said it would be O.K. for Mark to charge against the number 06953794.

I brought personal food dehydrator into the Analytical Lab Control Area for use in evaporating solution on test plates. Each tray can hold 12 Petri Dishes. Should be able to fit all 58 at once.

I checked depth in Petri dish. Ten ml will cover bottom if kept level. Leveled oven.

.....
9/30/04

Memorandum for: Professors, and health professionals at Honeywell, FM&T.

Some of my big challenges come from environmental protection and health safety policies:

- One can't just spray paint anywhere in the plant - must be in a special booth or hood; the Paint and Plasma lab could coat the Petri dishes - but it was Teflon, epoxy filled powder coating - not a representative / typical surface;
- Can't bring own paint into the plant - they don't have the paint I need in the plant - so got a property pass to take government-owned Petri dishes out of the plant - painted them on the driveway and took them into the garage to dry - is airborne dust a potential contaminant? Probably to some degree;
- Target loading = 1 microgram per test plate. Removal efficiencies will be measured at the fractional microgram level;
- Does the paint contain fractional micrograms of beryllium that could affect the results - wouldn't be surprised;
- Not very many people are willing to let Petri dishes with beryllium solution be put into their evaporation oven - got a property pass to take personal food dehydrator into the plant to evaporate beryllium solution in Petri dishes;
- Dust in dehydrator?
- Food dehydrator fan pulls air from the control area lab in an around the Petri dish test plates - is there airborne beryllium in fractional microgram levels that could contaminate?
- New Petri dishes come in cartons with some minimal amount of paper dust - must clean them before preparing test plates - will cleaning process introduce fractional micrograms of beryllium contamination?
- How does one adequately correct for all the potential contamination? Field blanks, spiked samples - how many and what kind?

- There is a small amount of liquid left in the pipette - they're designed to deliver accurate volume with remainder considered - but it makes me nervous when working at the sub-microgram level.

So many questions - and I've just started.

I spent last night re-reading ASTM 6966 "Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals." It requires a minimum of 5% field blanks (at least one for every 20 wipe samples collected) and a minimum of three for each batch of wipe samples. Their field blanks are unused wipe towelettes. I want to do some blanks that have wiped control plates that have had the solution dehydrated on them but without the microgram of beryllium. How many of those? When you get the time - you can see that I will genuinely appreciate some expert advice. Thanks in advance.

.....

11/15/04

MEMORANDUM FROM: Lloyd Lazarus

SUBJECT: Review Comments

According to the Handbook of Chemistry and Physics, Beryllium is insoluble in cold water and slightly soluble in hot water. It is also slightly soluble in alkaline solutions. Metalworking fluids are slightly soluble. We maintain the FMS fluid at a pH of 9. Since the tool-work piece interface is hot when machining and should affect the water temperature we can have the circumstances that allow slight solubility. I also noticed during the monitoring of the metalworking fluid in the FMS that the minimum beryllium counts went up during the two years no matter how much filtering or centrifuging I did. I believe I had some dissolved beryllium. At the end my counts were 10 parts/ billion in the fluid (PACE results) (Page 10). For your short term test you are correct.

In actual usage we deliver 5% solutions of 97022454. Because of evaporation we find the average solution on the floor is 6 to 7%. I realize for your mixtures 1:100 was a good way to deal with the numbers. Page 38 last paragraph using straight 9702-2454 concentrate and it should be 97022454.

The tooling from crib 14 and the films on the high voltage buss ducts the film is no longer sticky it is hard (free dried the old fashioned way). Some crib 14 items have been stored over 25 years. This film will not give up the beryllium unless the coating has been softened. My greatest fear is that contaminated tooling will go on a clean machine and exposed to water and the film softens and then dissolves. We now have a contaminated machine.

APPENDIX B

Summary of Animal Studies, First Aid, and Environmental Regulations

The following information about beryllium is of general interest but has only marginal application to the issues of surface contamination and its control by wipe sampling. Of course, findings from animal studies have provided some of the most valuable understandings about health effects caused by beryllium. And there is an interrelationship between surface contamination and the environment, so the information is provided for the reader interested in the more complete story.

Beryllium Toxicity Data from Animals Experimentation

Animal studies of the health effects of Be began in the early 1930s and continue to the present. Large doses of Be have caused bone lesions in rats.⁹³ Lung disorders have resulted in several species.^{94, 53, 95} Particle size and the temperature of firing in the production of BeO affects toxicity. The “low-fired” (400°C) BeO was significantly more toxic than that produced at 1150-1350°C. Later experiments tested mice and rats inhaling 13 µg/m³ Be for one hour. At three weeks after exposure, researchers observed proliferation of alveolar epithelial, interstitial, and capillary endothelial cells, with macrophage response and polymorphonuclear leukocytes infiltration of lungs.⁹⁶ The Be dose level resulting in death for 50% of test animals (LD₅₀) have been determined for several species and routes of exposure.^{97, 98, 99, 100, 101} Lung cancers have been caused by Be in test animals in various research projects.^{102, 103, 94, 104} Be exposure via ingestion has been studied with mixed results.^{105, 106} In vitro experiments with bacteria, mouse cells and human cell cultures demonstrated that Be forms DNA adducts at dose concentrations above 30 µmol/L.¹⁰⁷ Gene mutations have not been demonstrated with Ames assays,¹⁰⁸ but have been induced in mammalian cells cultured with beryllium chloride.¹⁰⁹ More recent studies have concentrated on mechanisms by which Be exerts influence on genetic materials or gene expression. One such study examined Be’s effect on specific mouse genes in alveolar macrophages. Be had a significant effect on genes that encode cytokine production as well as affecting transcriptional activators and repressors.¹¹⁰

First Aid

In case of contact with the eyes, immediately thoroughly flush the eyes with plenty of water, occasionally lifting the lower and upper lids. Get medical attention as soon as possible.^{17, 111, 112} The affected person should be retested in their company’s medical monitoring program.⁶³ If the exposed person has difficulty breathing because of having inhaled Be particulates, remove the individual to fresh air.¹¹¹ If breathing stops, perform artificial resuscitation, and call 911.¹¹² Following any inhalation incident, the person should be retested in their company’s medical monitoring program.⁶³ Ingestion is unlikely. If someone is suspected of having ingested small amounts of Be, induce

vomiting and seek medical attention.¹¹² Skin that has been potentially exposed to Be particulates should be thoroughly washed with nonabrasive soap and plenty of water. Splinters or chips or Be-containing metal should be promptly removed. Wounds that have been caused by Be-chips or pre-existing cuts and scratches that may have gotten contaminated with Be particles should be flushed with plenty of water, disinfected, covered with sterile dressing, and then examined by a doctor.^{111, 112}

Other U.S. Regulations Pertaining to Beryllium

Since surface Be contamination is interrelated with Be contained in various environmental media that following brief summary is provided:

- The National Primary Drinking Water Regulations, promulgated in accordance with the Safe Drinking Water Act of 1974, the EPA enforces a Maximum Contaminant Level (MCL) of 4 ppb for Be. This level is considered reasonably achievable by water treatment facilities and is thought to potentially result in 10^{-4} to 10^{-6} excess cancer risk. Water found to have greater than the MCL of Be can be treated by the EPA approved method of alumina coagulation, filtration, ion exchange, lime-softening, and reverse osmosis.^{30, 113}
- The manufacture, import and export of Be and Be-containing materials are tracked as a part of that Toxic Substances Control Act (TSCA) inventory.
- Superfund Amendments and Reauthorization Act (SARA) Title III, Section 313, Emergency Planning and Community Right-to-Know Act (CERCLA) requires businesses having more than certain threshold amounts of hazardous substances to make annual reports. Be and Be-containing materials are reportable in accordance with SARA title III, having a reportable quantity of 1-to-10 pounds.¹¹⁴
- Clean Air Act Regulation, National Emission Standards for Hazardous Air Pollutants (NESHAPS) are established for certain chemicals and materials including a 30-day average of $0.01 \mu\text{g Be}/\text{m}^3$, as documented at 40 CFR 61.32.³⁵
- Resource Conservation and Recovery Act (RCRA) regulations at 40 CFR 261.33(3) designate discarded Be powder waste material as a hazardous waste with waste code number P015. This category of hazardous waste only applies to commercially pure products or manufacturing intermediates in which Be is the only active ingredient.¹¹⁵ Be-containing waste materials may also qualify as RCRA hazardous waste if a toxicity characteristic leaching procedure demonstrates the likely evolution of toxic breakdown products in the reducing environment of a landfill.^{116, 117} For disposal of Be-containing materials, the waste should be sealed in double plastic bags with the outer bag labeled with the appropriate EPA hazardous waste label and DOT hazard warning labels and shipped with a hazardous waste manifest to an appropriately licensed hazardous waste management facility.¹¹² Under land disposal restrictions, beryllium powder is prohibited from being diluted in a combustion process.¹¹⁸
- Clean Water Act regulations at 40 CFR 131, Subpart D, "Federally Promulgated Water Quality Standards," lists Be but notes that EPA has not published an aquatic life criterion value.¹¹⁹

- The EPA's Nonferrous Metals Manufacturing Point Source Standards, Subpart O sets effluent limitations for the discharges from various operations in the processing of beryllium ore.¹²⁰

Department of Transportation does not regulate most solid Be or Be-containing materials, but Be powder is regulated as hazard class 6.1, Identification Number UN1567, packing group II, reportable quantity is 10 pounds. "Toxic – Flammable Solid" labels must be applied. Placards are required for shipments over 454kg aggregate gross weight.^{112, 121} shipment

APPENDIX C

Photographs

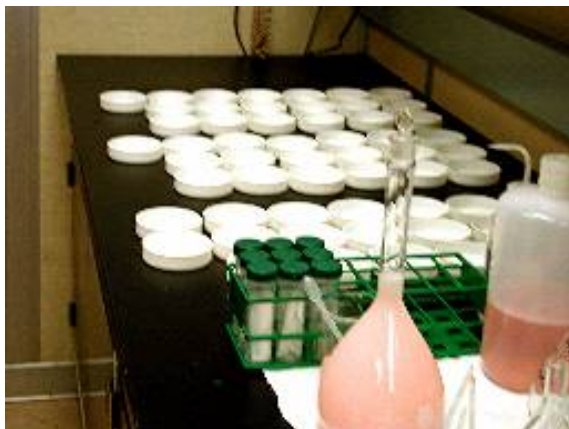


Photo 1 – Lab Bench Layout



Photo 2 – Pipetting MWF to Flask



Photo 3 – Pipetting Be Solution



Photo 4 – Delivering Aliquot to Test Plate



Photo 5 – Plates in Dryer



Photo 6 – Dried Plates with Covers



Photo 7 – Plates in Oven for Bake Out



Photo 8 – Unconditioned Paper Too Stiff



Photo 9 – Preconditioning Whatman Paper

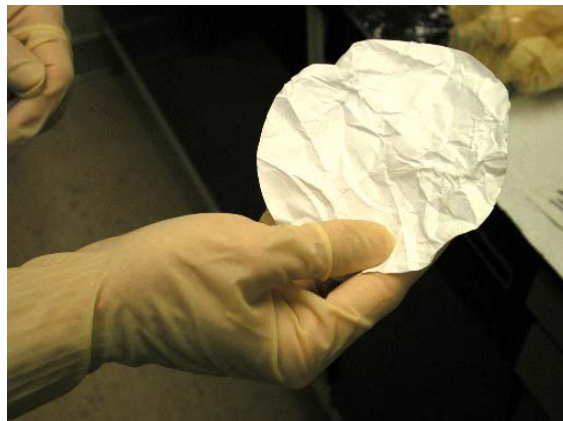


Photo 10 – More Flexible for Edge Access



Photo 11 – Press, Hold, Turn Plate



Photo 12 – Opening Ghost Wipe Packet

APPENDIX D**Beryllium Wipe Correlation Lab Journal**

Late August 2004

- I began experimenting with dehydrating and baking various oils, materials, and metal work fluid residues on Petri dishes to achieve a slightly tacky surface deposit. The tackiness was mentioned in the 8/31/04 conversation with Lloyd Lazarus (see Appendix A, [Memoranda of Conversations](#)). Some of those experiments are referenced in the memo of a 9/10/04 conversation with Garret, Bill and Dan (see Appendix A, [Memoranda of Conversations](#)).

9/28/04

- Spray painted the Petri dishes on step stool covered with plastic drop cloth out on the driveway, then moved them into the relatively quiescent air of the garage to dry on an ersatz table made from a piece of house siding placed on top of two saw horses.
 - I damp wiped the drying “table” to remove any surface contamination prior to placing the painted Petri dishes on it.
 - When removing the Petri dishes from their cartons, found small amount of paper dust on the inside surfaces. Wiped them out with isopropyl alcohol wipes (Zeiss Lens Cloths) before painting.
 - Paint can said the paint dried in 30 minutes. After two hours, I repacked the test plates in their cartons and returned them to the KCP – stored in cabinet of Designated Carcinogen Control Lab in the Analytical Lab Department.

9/30/04

Tammy showed me the micro-pipette – also the supply of Whatman 42 (ashless) filter papers. Bill Frede and Garrett agreed that Whatman 42 was appropriate for the dry wipe material in lieu of Whatman 50 (low ash hardened).

10/1/04

Got more Ghost Wipes from Lloyd Lazarus.

10/06/04

Received 57 Pace Lab methanol wipe in vials from Mike Davis, Dan Ham, and Bill Frede.

10:00 10/07/04

- Washed glassware with concentrated nitric acid, rinsed with tap water then rinsed with deionized water.
- Prepared 2 L of dilute (1:100) metal working fluid – 10 ml concentrate CIMSTAR 3700T in 1 Liter deionized water.
- Prepared 1 liter of 100 µg Be/L MWF solution by pipetting 10 ml of 10 µg Be/ml standard solution into a 1L volumetric flask and filling to mark with 1:100 MWF.

- Note: I was having trouble with the pipette bulb that Tammy Pond uses – it was a simple suction bulb – had to pull liquid up into pipette then transfer finger to end of pipette and bleed off contents down to the line. With nitrile gloves on, I could not adequately control the release of the liquid (kept shooting past the line). Tammy volunteered to pipette the 10ml standard 10 μg Be/ml solution. (She didn't wear gloves.) At her recommendation, I added a little 1:100 MWF solution to the volumetric flask before she transferred the standard Be solution. When she added the Be solution, there was an immediate precipitation reaction observed by cloudiness. I brought the solution in the volumetric flask close to the line with 1:100 MWF. Tammy offered to run the solution through her ICP spectrophotometer to determine if there had been a loss of uniformity in the concentration of Be in the solution. The MWF contains detergent, which causes some bubbles when agitated by pouring operation. I took a lunch break, allowing time for suds to subside.
- While walking through the aisle on lunch break, chanced to meet Bill Frede. We discussed field blanks and spikeless control plates and agreed that it would be best to run three each, as well as a spike directly to unused wipe material in vial.
- After lunch, there was no apparent flocculation or settling out – just uniformly milky suspension. I brought the solution up to the line in the volumetric flask and mixed it. Tammy ran the solution through ICP spec. The results, 0.0978 mg/l, showed that the Be concentration was still essentially at the target concentration of 0.1 mg/l.
- Wiped off the lab bench surfaces with dampened paper towels, then with methanol wipe material.
- Laid out the painted Petri dish test plates. Wiped both inside and outside surfaces of each with methanol moistened wipe material.
- Went to the photo lab and borrowed a digital camera. Asked Ray Gann to help take some pictures of the operations. With some help from another fellow in the lab we learned how to operate the camera. After a couple of pictures ran out of batteries. Standard AAs. Asked around – told to ask Karl. He got me four AA batteries (bless his heart!). Got pictures of the arrays of test plates, pipette and volumetric flask transfers, etc.
- In the distraction of photographing the processes, I digressed from my previously prepared step-by-step work instruction. Instead of transferring unspiked solution aliquots to my spikeless control plates, I began transferring Be-spiked solution aliquots to test plates D1, D2, and D3, for the camera. This necessitated washing another 10 ml pipette with nitric acid, repeated water and deionized water rinses in order to assure Be-free glassware for transferring 1:100 MWF to spikeless control plates.
- I had been using blue nitrile gloves interchangeably with the powderless latex gloves, until I noticed the nitrile gloves leaving cloudiness behind in droplets when rinsing the additional 10 pipette. Probably need to run a control wipe of the nitrile gloves to make sure they are not a source of Be contamination.
- Added 10 ml aliquots 1:100 to spikeless control plates D17, D18, D19, W17, W18, W19, A17, A18, and A19. I did this pipette work bare handed but still had

trouble with solution shooting past the line. Placed them on the top shelf of the evaporation chamber (food dehydrator).

- 10 ml just barely covers the bottom of the 10 cm Petri dishes
- Prepared test plates. From the 100 µg Be/L 1:100MWF solution, added 10 ml aliquots to each of 48 painted test plates labeled D1, D2, D3,...D16; W1, W2, W3,...W16; and A1, A2, A3,...A16.
 - After struggling with pipette transfers for several plates of the D-series, I went to Carl and asked if he could provide me with a 3-way jointed pipette bulb. He had two (ordered about 10 years ago but hardly ever used) and loaned me one (what a relief!).
- Placed the test plates in dehydrator - 12 on each of four shelves and the remaining 10 on the floor of the unit. Set the unit for 145°F and turned it on at 17:15.
 - Discovered that the dehydrator did not appear to be level by observing the 10 ml of liquid in a test plate sitting on top of the dehydrator. Borrowed a level and some shims from Wenski. Leveled the unit. Then discovered that the shelves were actually more level without the shimming. Levelness was desirable in the attempt to provide a more-or-less uniform residue consistency over the face of the painted test plate surface.

10/08/04

- 10:00 checked the Petri dish test plates in the dehydrator – they were dried.
- Wiped Petri dish tops with alcohol-dampened wipes.
- 10:20 took pre-use wipe test in the oven – placed in vial O2 marked with date and time.
- 10:45 began removing test plates from dehydrator and covering them.
- 10:30 started pre-heating the oven – Carl said it would take about an hour to get it up to 230°F
- Went to see Dan Ham, Mike Davis to get 15 additional Pace alcohol wipe vials; talked about chain of custody when transferring samples to Pace. Dan and Mike will enter the sample numbers into their master data base and electronically provide the information to Pace. I will provide them a sanitized listing of the vial numbers.
- Oven was reading 229 before opened to load test plates. Placed the test plates on a new-foil-covered roller cart in stacks of three to move them from the Designated Area Lab across the hall to the Instron oven room. The cart bumped into a black-foam insulated liquid nitrogen line knocked over a stack of test plates. The cover for plate W8 came off and a small fleck of black foam insulation fell onto the test plate surface. I tipped it out, recovered it and proceeded with the operation. Covered the bottom of the oven with foil. Stacked test plates on the foil-covered floor of the oven in stacks of 4-5 deep; temperature was at 130 when door was closed at 12:25; 226 @ 12:30; 228 @ 12:35; and 229 @ 12:50. Removed them from the oven at 12:50. Wished I had one I could finger to check out the texture – had to proceed based on earlier experimentation. The color change from red to more brownish red indicated some oxidation and the baked dishes looked very dry. Oven (temperature chamber was manufactured by Associated Environmental Systems Division of Craig Systems Corporation, Lawrence, MA 01842. Its

calibration sheet is included at Appendix H, [Equipment Performance and Calibration](#).

- 13:50 Started swiping test plates. Began with the dry wipes using Whatman 42 12.5 cm circle filter papers preconditioned by wadding into a tight little ball & then un-wadding to make flexible enough to bend into the corner edges of the test plates. Started by holding the test plates with my left hand while swiping with the fingers of my right hand. Soon began to suspect that I could introduce contamination from the backs of the test plates – since my left glove touched the backs and then was used to fold the paper for subsequent wipe runs. Discontinued holding plates with my left hand while swiping – instead used left hand on outside and top edges to hold the test plates and turning them while pressing filter paper onto the surface. Counted about 12 quarter turns while right fingers were tracing clockwise helical pattern from outside edge toward the center. Spent about six quarter turns mostly on outside edge and next six turns moving toward the center. For the counterclockwise pattern, held the plate still while moving right fingers from inside to outside in helical pattern. Dry wipes took longer due to preconditioning.
- At some point began to consider other possible cross-contamination. Although I had wiped the lab bench surface and the test plates, there was a remote possibility that there could be Be contamination on the bench and/or Petri dish exterior surfaces. I would put on a fresh pair of powderless Latex gloves after each Petri dish wipe, but then I would pick up the next Petri dish, usually touching the bench surface and bottom outside edge of the Petri dish; remove the cover; remove the next filter paper from the box; precondition it; wipe sample (some potential for getting Be contamination on the right glove fingers); then pick up lab vial; unscrew lids (remote potential for contamination on the exterior surfaces of the vials); if there were Be contamination on the vials now the gloves would be contaminated when picking up the used wipe and placing it in the vial; then with potentially contaminated glove, I picked up the marker to write date and time on the vial; now the marker had to be considered contaminated, but it was only handled after the sample was in the vial and immediately after writing time and date on the vial the gloves were discarded and new ones donned. There would have been wisdom in observing Mike and Dan taking wipe samples before attempting this exercise. They have probably learned techniques for avoiding cross-contamination. Now there is some unnecessary angst while awaiting the results of my control, and field blanks. Too soon old – too late wise!
- I wiped the Ghost wipe packets with alcohol wipe media in an effort to minimize potential contamination. Even so, one wonders when handling the packets, opening them, and then removing the wet wipe media about the potential for cross contamination from the outside of the packets.
- It would be best to have four hands for this operation. Two for handling vials, towelette packets, markers, etc., and additional two for removing the towelettes from their containers with uncontaminated plastic locking forceps, handling the sampling media, and performing the wipe sampling.
- Eventually, I also made another change: before removing contaminated gloves, I would slide the next Petri dish test plate over and remove its top – in this way the

exterior sides of the Petri dish were not touched with potentially contaminated gloves and the fresh pair did not touch the lab bench when picking up the next dish.

- Most of the Ghost Wipes were surprisingly soppy (only one had dried out) and seemed to dissolve the contamination residues (made me think they didn't get baked enough) with surprising efficiency. I guessed that the detergent content in the metal working fluid enhanced the ability of the water-moistened wipe materials to dissolve and remove the residue (there may not be as much contrast between the Ghost wipe and alcohol wipe removal efficiencies and originally anticipated). Found that I couldn't use as much pressure with the Ghost Wipes after the first couple of folds – if I pushed as hard as with the dry filter paper, it squeezed the water from the material – would lose collected Be if wash water were to be left behind. This was also true for the alcohol wipes. Whereas I had originally intended to provide consistent pressure while swiping the various test plates – in practice it was not possible to use equal pressure without running the risk of leaving behind Be-laden solvent when using the dampened wipe materials.
- 18:00 Finished wipe sampling.
- 18:20 Left KCP after cleaning up the Designated Area lab; packed up used test plates in their original cartons and stowed them in shelving under lab bench N side near door; locked the sample vials in their racks in my office file cabinet. Locked the camera in my desk.

10/11/04

- Dan Ham recommended that I bring the samples on 10/12/04 at about 09:00 with the Chain of Custody paperwork for him to input them into the automated PACE system, prior to taking them to Mike McNelly for transmittal to the PACE courier.
- There were two trial test plates left from preliminary experimentation with amounts of MWF and baking times to achieve the tacky surface texture that was intended to simulate aged MWF mist residue on surfaces in metal working shops. Wiping with a water-dampened paper towel quickly removed even the tackiest baked-on surface contamination from the painted test plates. This was surprising. It was expected that the oxidized mineral oil residue would be resistant to water-moistened wiping. The fact that the desiccated detergent residue would still be effective had not occurred to me.

10/12/04

- Mike McNelly was ill but had thought to call and tell me to contact Don Stedem instead. Dan and Mike discussed it with PACE and they decided these samples didn't have to be entered into the automated system in advance of transfer to the lab. I took the sample vials to Don Stedem, and he signed the Chain of Custody.
- Ray Gann helped take some additional pictures. I took the camera to Nelson Beard who reviewed the 22 photographs for security issues. None were found so I returned the camera to Jim Briggs who signed it back in on the log book and downloaded the photographs to a CD.

10/14/04

- Copied the photographs from the CD to my home computer.

10/15/04

- Called Mike McNelly to get Chris Scharff's phone number at Pace Labs.
- Called Chris at 913-599-5665 (ext. 225) to remind her that (as stated on the bottom of my Chain of Custody) I was requesting the calibration and spike recovery data for the day they ran my samples. Chris informed me that they had run my samples this morning and that the results should be available later this morning or this afternoon on Docunet. She said she's let me know if she had any trouble getting the information I'd requested.
- Mike McNelly provided me with the instruction for getting into <https://www.docunet.pacelabs.com> with the User ID and Password for Honeywell.
- Downloaded the results. Non-detect for all the dry wipe samples! Should be *something* there. Spike recovery less than 75%. EPA Method requires recovery be $\pm 25\%$.
- I called Mike and he got Chris on the line with us – requested that the samples be returned for second opinion. Hope to have Tammy Pond run them.
- Chris informed me that they did not run spike recovery since they were single samples.

10/18/04

- Mike McNelly prepared Laboratory Test Request number 34732 for Tammy Pond to do 17 second-opinion analyses of the digestate sample solutions returned from Pace having their sample numbers affixed to top of vials on paper labels. Re-analysis was requested for my sample numbers D1, D4, D8, D11, D14, D16, W3, W7, W10, W13, W16, W23, A2, A7, A10, A13, and A16.
- Tammy said she should have them done "later in the week."

10/25/04

- Got the results from Tammy.

10/27/04

- Ran ANOVA analysis of the 3 sets of removal efficiencies. Significant. Reject H_0 .

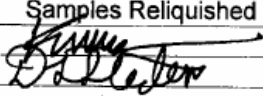
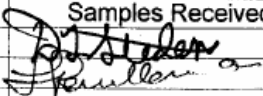
APPENDIX E

Chain of Custody

Be Swipe Correlation Sample List and Chain of Custody

Vial Number	Vial Fill Date	Analysis Requested	Turnaround requested
D1	10/8/2004	Total Be by ICP	Normal (not rush)
D2	10/8/2004	Total Be by ICP	Normal (not rush)
D3	10/8/2004	Total Be by ICP	Normal (not rush)
D4	10/8/2004	Total Be by ICP	Normal (not rush)
D5	10/8/2004	Total Be by ICP	Normal (not rush)
D6	10/8/2004	Total Be by ICP	Normal (not rush)
D7	10/8/2004	Total Be by ICP	Normal (not rush)
D8	10/8/2004	Total Be by ICP	Normal (not rush)
D9	10/8/2004	Total Be by ICP	Normal (not rush)
D10	10/8/2004	Total Be by ICP	Normal (not rush)
D11	10/8/2004	Total Be by ICP	Normal (not rush)
D12	10/8/2004	Total Be by ICP	Normal (not rush)
D13	10/8/2004	Total Be by ICP	Normal (not rush)
D14	10/8/2004	Total Be by ICP	Normal (not rush)
D15	10/8/2004	Total Be by ICP	Normal (not rush)
D16	10/8/2004	Total Be by ICP	Normal (not rush)
D17	10/8/2004	Total Be by ICP	Normal (not rush)
D18	10/8/2004	Total Be by ICP	Normal (not rush)
D19	10/8/2004	Total Be by ICP	Normal (not rush)
D20	10/8/2004	Total Be by ICP	Normal (not rush)
D21	10/8/2004	Total Be by ICP	Normal (not rush)
D22	10/8/2004	Total Be by ICP	Normal (not rush)
D23	10/8/2004	Total Be by ICP	Normal (not rush)
W1	10/8/2004	Total Be by ICP	Normal (not rush)
W2	10/8/2004	Total Be by ICP	Normal (not rush)
W3	10/8/2004	Total Be by ICP	Normal (not rush)
W4	10/8/2004	Total Be by ICP	Normal (not rush)
W5	10/8/2004	Total Be by ICP	Normal (not rush)
W6	10/8/2004	Total Be by ICP	Normal (not rush)
W7	10/8/2004	Total Be by ICP	Normal (not rush)
W8	10/8/2004	Total Be by ICP	Normal (not rush)
W9	10/8/2004	Total Be by ICP	Normal (not rush)
W10	10/8/2004	Total Be by ICP	Normal (not rush)
W11	10/8/2004	Total Be by ICP	Normal (not rush)
W12	10/8/2004	Total Be by ICP	Normal (not rush)
W13	10/8/2004	Total Be by ICP	Normal (not rush)
W14	10/8/2004	Total Be by ICP	Normal (not rush)
W15	10/8/2004	Total Be by ICP	Normal (not rush)
W16	10/8/2004	Total Be by ICP	Normal (not rush)
W17	10/8/2004	Total Be by ICP	Normal (not rush)
W18	10/8/2004	Total Be by ICP	Normal (not rush)
W19	10/8/2004	Total Be by ICP	Normal (not rush)
W20	10/8/2004	Total Be by ICP	Normal (not rush)
W21	10/8/2004	Total Be by ICP	Normal (not rush)
W22	10/8/2004	Total Be by ICP	Normal (not rush)
W23	10/8/2004	Total Be by ICP	Normal (not rush)
A1	10/8/2004	Total Be by ICP	Normal (not rush)
A2	10/8/2004	Total Be by ICP	Normal (not rush)
A3	10/8/2004	Total Be by ICP	Normal (not rush)
A4	10/8/2004	Total Be by ICP	Normal (not rush)
A5	10/8/2004	Total Be by ICP	Normal (not rush)

Be Swipe Correlation Sample List and Chain of Custody

A6	10/8/2004	Total Be by ICP	Normal (not rush)
A7	10/8/2004	Total Be by ICP	Normal (not rush)
A8	10/8/2004	Total Be by ICP	Normal (not rush)
A9	10/8/2004	Total Be by ICP	Normal (not rush)
A10	10/8/2004	Total Be by ICP	Normal (not rush)
A11	10/8/2004	Total Be by ICP	Normal (not rush)
A12	10/8/2004	Total Be by ICP	Normal (not rush)
A13	10/8/2004	Total Be by ICP	Normal (not rush)
A14	10/8/2004	Total Be by ICP	Normal (not rush)
A15	10/8/2004	Total Be by ICP	Normal (not rush)
A16	10/8/2004	Total Be by ICP	Normal (not rush)
A17	10/8/2004	Total Be by ICP	Normal (not rush)
A18	10/8/2004	Total Be by ICP	Normal (not rush)
A19	10/8/2004	Total Be by ICP	Normal (not rush)
A20	10/8/2004	Total Be by ICP	Normal (not rush)
A21	10/8/2004	Total Be by ICP	Normal (not rush)
A22	10/8/2004	Total Be by ICP	Normal (not rush)
A23	10/8/2004	Total Be by ICP	Normal (not rush)
A24	10/8/2004	Total Be by ICP	Normal (not rush)
O1	10/8/2004	Total Be by ICP	Normal (not rush)
O2	10/8/2004	Total Be by ICP	Normal (not rush)
72 vials			
Date	Time	Samples Relinquished By	Samples Received By
10/12/04 10/12/04	09:45 1324		
Samples have potential for Al, Fe, Li, Ni, Na, Rb, and Tl, in addition to Be.			
Please provide spike recovery and calibration data for day of analyses with results.			

APPENDIX F

Material Safety Data and Specification Sheets

inorganic ventures / iv labs

MATERIAL SAFETY DATA SHEET

EMERGENCY TELEPHONE #'S

Inorganic Ventures: (800) 669-6799 Chemtrec (24 hr): (800) 424-9300

195 lehigh avenue, suite 4
lakewood, new jersey 08701
usa

Issue Date: 10-29-03

Effective Date: 10-29-03

tel: 732.901.1900
fax: 732.901.1903
sales: 1.800.669.6799
tech support:
1.800.569.6799
website:
www.ivstandards.com
e-mail:
IVsales@ivstandards.comSECTION II - PRODUCT IDENTIFICATION/HAZARDOUS INGREDIENTSProduct Name: Custom Multielement Solution Standard Formula: 100 µg/mL ea: Rb, Tl
10 µg/mL ea: Al, Be, Fe, Li, Ni, NaCommon Names: GN30-1Component : CAS NO:All elements are below reportable levels under 29 CFR Subpart 1910.1200d 5(ii) 7-1-89 edition.
Nitric Acid 2% (abs) (A)
Water (W)7697-37-2
7732-18-5STEL: 10 mg/m³ (A)PEL/TLV/TWA: 5 mg/m³ (A)Toxicity:

LC50 (ihl-rat) 1 hr (A)

N/A - Not Applicable or Not Available

N/E - Not Established

SECTION III - PHYSICAL/CHEMICAL CHARACTERISTICSB.P.: 100°C (212°F)Sp. G (H₂O = 1): 1SOL./H₂O: CompleteAPPEARANCE/ODOR: Clear, colorless solution with no odor.SECTION IV - FIRE AND EXPLOSION HAZARD DATAFlash Point: N/ANFPA - Rating: 3-0-0Extinguishing Media: Use appropriate for surrounding fire.Special Fire-Fighting Procedures: Firefighters should wear proper protective equipment and self-contained breathing apparatus with full facepiece operated in positive pressure mode.Unusual Fire Explosion Hazards: N/AToxic Gases Produced: Nitrogen oxidesSECTION V - REACTIVITY DATAUnstable(): Conditions to Avoid: N/A
Stable(X):Incompatibles: Organic materials, strong reducing agents.Hazardous Decomposition: Oxides of nitrogen

It is the policy of Inorganic Ventures to provide its customers with a Material Safety Data Sheet for each lot of material supplied, since lots may vary in composition. For your convenience, we are labeling this MSDS with the expiration date of the lot accompanying this document.

Expires:

EXPIRES
10/29/04IN
OUT 11/12/2003

SECTION VI - HEALTH HAZARD DATA

Routes of Entry: Inhalation, eye contact, skin contact

Signs and Symptoms of Exposure: Liquid may cause burns to skin and eyes. Inhalation of vapors may cause coughing and difficulty breathing. Ingestion may cause nausea, vomiting and loss of consciousness.

Medical Conditions Generally Aggravated by Exposure: None identified.

Carcinogenicity: None identified.

Emergency First Aid Procedures: CALL A PHYSICIAN!!! If swallowed, do NOT induce vomiting; if conscious give water, milk or milk of magnesia. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before re-use.

SECTION VII - PRECAUTIONS FOR SAFE HANDLING AND USE

Special Precautions: Keep container tightly closed. Store in corrosion-proof area.

In Case of Spill or Discharge: Wear self-contained breathing apparatus and full protective clothing. Stop leak if you can do so without risk. Use water spray to reduce vapors. Take up with sand or other non-combustible absorbent material and place into container for later disposal. Flush spill area with water.

Disposal Procedures: Dispose in accordance with all applicable federal, state, and local environmental regulations.

EPA Hazardous Waste #: D002 (Corrosive Waste)

SECTION VIII - PROTECTIVE EQUIPMENT

This is a laboratory use product for which no industrial protective equipment has been designated. However, we do suggest the use of local exhaust, vent hood, lab coat, safety glasses and proper gloves.

NOTICE

The above information is believed to be accurate and represents the best information currently available to us. It has been compiled from the data presented in various technical publications and our experience. It is the user's responsibility to determine the suitability of this information for their particular purposes. We assume that only qualified individuals, trained and familiar with procedures suitable to this product will handle this material.

MSDS # 4261, Valid through 10/29/2004.

CURRENT ISSUE CALL 7090

MSDS Id #: 4261

MATERIAL SAFETY DATA SHEET

Product Name: CIMSTAR 3700T

Part #:97022454

SECTION I SUPPLIER INFORMATION

Common Name: IDENTITY (AS USED ON LABEL AND LIST): CIMSTAR 3700T
Chemical Name/Formula: NO DATA SUPPLIED
Product CAS#: NO DATA SUPPLIED

Supplier: PRODS. DIV., CINCINNATI Supplier #:3963
P.O. Box: MILACRON INC.
Address: 4701 MARBURG AVENUE
City, St, Zip: CINCINNATI OH 45209 Phone: 513-841-8964

EMERGENCY PHONE #: 513-841-8181

Date Issued: 09/01/90 Date Entered: 03/25/93 Downloaded: 21 MAR 1997

Date Revised:

SECTION II INGREDIENT INFORMATION

Table with 6 columns: INGREDIENT, %MIN, %MAX, CAS, PEL-OSHA, TLV-ACGIH. Rows include MONOETHANOLAMINE, TRIETHANOLAMINE, MINERAL OIL (SEVERELY HYDROTREATED) (MIST).

INGREDIENT HAZARD STATEMENT

HAZARDOUS COMPONENTS

- MONOETHANOLAMINE
TRIETHANOLAMINE
MINERAL OIL (SEVERELY HYDROTREATED) (MIST)

THE INGREDIENTS LISTED ABOVE MAY CONTRIBUTE TO THE PRODUCT HAZARD AS LISTED IN SECTION VI.

SECTION III PHYSICAL/CHEMICAL CHARACTERISTICS

Boiling Point: 212 DEGREES F
Specific Gravity (H2O = 1): 1.033
Melting Point: NOT APPLICABLE (NA)
Vapor Pressure (mm Hg): NOT APPLICABLE (NA)
Vapor Density (Air=1): NOT APPLICABLE (NA)
Evaporation Rate (Butyl Acetate=1): LIKE WATER
Solubility/Water: 100%
pH Level:
Percent Volatile:

***** APPEARANCE AND ODOR *****

CLEAR; CHEMICAL

NOTE: ON HARD COPY MSDS: CLEAR; CHEMICAL WAS CROSSED OFF AND PINK DYE AS REQUESTED BY KCD DESCRIPTION WAS HANDWRITTEN

***** ADDITIONAL INFORMATION *****

CIMSTAR IS A REGISTERED TRADEMARK OF CINCINNATI MILACRON

MANUFACTURER:

PRODUCTS DIVISION / CINCINNATI MILACRON MARKETING COMPANY

TELEPHONE NUMBER FOR INFORMATION 3-841-8964

SECTION IV FIRE AND EXPLOSION HAZARD DATA

Flash Point (Method): (METHOD USED): (COC) NONE; SELF-EXTINGUISHING
LEL: FLAMMABLE LIMITS: NOT APPLICABLE (NA)
UEL: FLAMMABLE LIMITS: NOT APPLICABLE (NA)
Auto-Ignition: NO DATA SUPPLIED

***** NFPA HAZARD CLASSIFICATION *****

Flammable: NO DATA SUPPLIED Health: NO DATA SUPPLIED
Reactivity: NO DATA SUPPLIED Special: NO DATA SUPPLIED

***** HMIS HAZARD CLASSIFICATION *****

Flammable: Health:
Reactivity: Special:

***** EXTINGUISHING MEDIA *****

NO FIRE HAZARD.

***** SPECIAL FIRE FIGHTING PROCEDURES *****

NO FIRE HAZARD.

***** UNUSUAL FIRE AND EXPLOSION HAZARDS *****

NONE.

SECTION V REACTIVITY DATA

Stability? PRODUCT IS STABLE.

Avoid: NOT APPLICABLE (NA)

***** INCOMPATIBILITY (Materials to Avoid) *****

AVOID CONTACT OF CONCENTRATE WITH STRONG ACIDS.

***** HAZARDOUS DECOMPOSITION OR BY-PRODUCTS *****

NONE.

Hazardous Polymerization? HAZARDOUS POLYMERIZATION: WILL NOT OCCUR

Avoid: NOT APPLICABLE (NA)

SECTION VI HEALTH HAZARD DATA

***** ROUTES OF ENTRY *****

Inhalation? YES
 Skin? YES
 Ingestion? NOT APPLICABLE (NA)
 Eyes? NO DATA SUPPLIED

***** ADDITIONAL INFORMATION *****

ROUTE(S) OF ENTRY: INHALATION? YES SKIN? YES
 INGESTION? NOT APPLICABLE (NA)

***** CARCINOGENICITY *****

NTP? NO IARC? NO OSHA? NO

***** ADDITIONAL INFORMATION (CARCINOGENICITY) *****

CARCINOGENICITY: NTP? NO IARC MONOGRAPHS? NO OSHA REGULATED? NO

***** ACUTE AND CHRONIC HEALTH HAZARDS *****

CONCENTRATE IS ALKALINE. HARMFUL IF TAKEN INTERNALLY. CONCENTRATE IS AN EYE IRRITANT. CONCENTRATE MAY BE A PRIMARY SKIN IRRITANT. NO ADVERSE CHRONIC EFFECTS ARE EXPECTED WHEN USED AS RECOMMENDED.

***** SIGNS AND SYMPTOMS OF EXPOSURE *****

EYE DAMAGE MAY OCCUR FROM CONTACT WITH CONCENTRATE. A REVERSIBLE SKIN IRRITATION (REDNESS AND SWELLING) MAY OCCUR FROM CONTACT WITH CONCENTRATE.

***** MEDICAL CONDITIONS GENERALLY AGGRAVATED BY EXPOSURE *****

MAY AGGRAVATE EXISTING SKIN IRRITATION WHERE FURTHER DEFATTING OR SKIN PENETRATION COULD OCCUR.

***** EMERGENCY AND FIRST AID PROCEDURES *****

CASE OF EYE CONTACT, FLUSH IMMEDIATELY WITH RUNNING WATER FOR 15 MINUTES, THEN GET PROMPT MEDICAL ATTENTION TO CHECK FOR POSSIBLE IRRITATION. IN CASE OF SKIN CONTACT WITH CONCENTRATE, WASH IMMEDIATELY WITH WATER. IF CONCENTRATE OR MIX IS SWALLOWED, DO NOT INDUCE VOMITING. DILUTE WITH WATER OR MILK. IMMEDIATELY CONTACT PHYSICIAN AND OBTAIN TREATMENT.

SECTION VII PRECAUTIONS FOR SAFE HANDLING AND USE

DOT Classification: NO DATA SUPPLIED

***** STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED *****

THOROUGHLY FLUSH WITH WATER TO SEWER.

***** WASTE DISPOSAL METHOD *****

FOR USED MIX: 1) ULTRAFILTRATION FOR SEWER DISPOSAL, OR 2) SEWER DISPOSAL IF APPLICABLE ACCORDING TO LOCAL REGULATIONS, OR 3) RECYCLE EQUIPMENT FOR REUSE, OR 4) TREAT WITH POLYMER OR INORGANIC DEEMULSIFIERS, THEN DISPOSE OF TOP LAYER BY INCINERATION OR LANDFILL, AND DISPOSE OF WATER LAYER IN SANITARY SEWER.

FOR UNUSED CONCENTRATE: INCINERATE, OR CONTACT VENDOR.

***** PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE/REGULATORY DATA *****

USE ONLY AS RECOMMENDED BY CINCINNATI MILACRON. AVOID ALL CONTACT OF CONCENTRATE WITH EYES OR PROLONGED CONTACT WITH SKIN. DO NOT SWALLOW. IF FROZEN, PRODUCT SEPARATES. THAW COMPLETELY AT ROOM TEMPERATURE AND STIR THOROUGHLY.

***** OTHER PRECAUTIONS *****

CONTAINS AMINES. DO NOT ADD SODIUM NITRITE OR OTHER NITROSATING AGENTS TO THIS PRODUCT. SUSPECTED CANCER-CAUSING NITROSAMINES COULD BE FORMED.

SECTION VIII CONTROL MEASURES

***** RESPIRATORY PROTECTION *****

PRODUCT NOT VOLATILE.

***** VENTILATION *****

- Local Exhaust: NO DATA SUPPLIED
- Special Exhaust: NO DATA SUPPLIED
- Mechanical Exhaust: GENERAL
- Other: NO DATA SUPPLIED

***** OTHER PROTECTION *****

Gloves: PROTECTIVE GLOVES: WATERPROOF GLOVES REQUIRED WHEN HANDLING CONCENTRATE.

Eye Protection: SAFETY SHIELD OR GOGGLES REQUIRED WHEN HANDLING CONCENTRATE.

***** CLOTHING *****

OTHER PROTECTIVE CLOTHING OR EQUIPMENT: EFFECTIVE METALWORKING PLANT PROTECTIVE
THING AS APPROPRIATE.

***** WORK/HYGIENE PRACTICES *****

GOOD PERSONAL HYGIENE SHOULD ALWAYS BE FOLLOWED.
THIS IS THE END OF THE MSDS Id #4261

Material Safety Data Sheet

Section 1 General Information

Manufacturer:
Zinsser Company, Inc.
173 Belmont Drive
Somerset, NJ 08875
(732) 469-8100

HMIS Rating
HEALTH: 1
FLAMMABILITY: 3
REACTIVITY: 0

Emergency Telephone: Chemtrec (800) 424-9300

Date: September 25, 2002

Product Name: Cover Stain Spray

Product Codes: 3608

Section 2 Hazardous Ingredients

<u>Hazardous Component</u>	<u>CAS#</u>	<u>OSHA PEL</u>	<u>ACGIH TLV</u>
Acetone	67-64-1	1000 ppm	500 ppm
Aliphatic Petroleum Distillates	64742-89-8	500 ppm	N/E
Aromatic 100	64742-95-6	500 ppm	N/E
n-Butyl Alcohol	71-36-3	50 ppm (skin)	50 ppm (skin)
Dipropylene Glycol Monomethyl Ether	34590-94-8	100 ppm	100 ppm
Ethanol	64-17-5	1000 ppm	1000 ppm
Isopropanol	67-63-0	400 ppm	500 ppm
Kaolin	1332-58-7	15 mg/m ³ * (5 mg/m ³ **)	2 Mg/m ³ **
Limestone	1317-65-3	15 mg/m ³ * (5 mg/m ³ **)	10 mg/m ³ † (3 mg/m ³ **)
Propane	74-98-6	1000 ppm	2500 ppm
Titanium dioxide	13463-67-7	15 Mg/m ³ *	10 Mg/m ³
VM&P Naphtha	8032-32-4	500 ppm	300 ppm

* Total Dust

** Respirable Dust Fraction

† Inhalable fraction

Section 3 Hazard Identification

Emergency Overview: This material is an aerosol spray coating used as a primer/sealer. The aerosol container is pressurized and extremely flammable. As packaged, this material has a flashpoint of less than 32° F.

Primary Routes of Exposure:

Inhalation
Skin Contact
Eye Contact

Potential Acute Health Effects:

N/A: Not Applicable N/D: Not Determined N/E: Not Established N/R: Not Required Est.: Estimated

Eye: May cause eye irritation.

Skin: Prolonged contact may cause skin irritation.

Ingestion: Although ingestion is not anticipated route of exposure, this material can be harmful if swallowed. It may cause depression of the central nervous system, nausea, and vomiting.

Inhalation: May cause headache, drowsiness, dizziness, nausea, and respiratory tract irritation.

Potential Chronic Health Effects: None known.

(See also Sections 4, 8, and 11 for related information)

Section 4 First Aid Measures

Eye contact: Flush eyes with water for 15 minutes.

Skin contact: Wash affected area with soap and water.

Ingestion: Call a physician or poison control center.

Inhalation: Remove person to fresh air. Contact physician or poison control center if symptoms persist.

Section 5 Fire Fighting Measures

Flash Point (method): < 32° F (< 0° C)

Extinguishing Media: Foam, CO₂, dry chemical, water fog.

Protection of Firefighters: As in any fire, wear NIOSH approved self-contained breathing apparatus in pressure-demand mode and full protective gear.

Fire and Explosion Hazards: Contents under pressure. Containers can build up pressure if exposed to heat (fire).

Section 6 Accidental Release Measures

Personal Precautions: Use with adequate ventilation. Avoid breathing vapors. Avoid contact with eyes and skin. Do not eat, drink or smoke in areas of use or storage.

Clean Up Methods: Dispose of in accordance with federal, state, local, regulations.

N/A: Not Applicable N/D: Not Determined N/E: Not Established N/R: Not Required Est.: Estimated

(See also Section 8 for information on Exposure Controls and Personal Protective Equipment)

Section 7 Handling and Storage

Handling: Keep away from heat, spark, and flame. Keep operating temperatures below ignition temperatures at all times. Use only in a well-ventilated area.

Storage: Store at ambient or lower temperature. Keep containers tightly closed and upright when not in use. Protect against physical damage. Contents under pressure. Do not expose to heat.

Section 8 Exposure Controls / Personal Protection

Engineering Controls: Local exhaust ventilation may be necessary to control vapors within applicable exposure limits.

Personal Protective Equipment (PPE):

Eye Protection: Wear safety glasses, splash goggles, or full face shield to prevent eye contact.

Skin Protection: Wear rubber gloves to prevent skin contact.

Respiratory Protection: Use NIOSH approved respirator with organic vapor cartridges if vapor levels exceed allowable workplace exposure limits.

General Hygiene Practices: Wash thoroughly after handling. Prevent Eye contact. Avoid prolonged skin or inhalation contact.

Section 9 Physical Data

Appearance:	White aerosol mist.	Odor:	Hydrocarbon type odor.
Physical State:	Spray mist	pH:	N/A
Boiling Point:	N/D	Melting Point:	N/D
Vapor Pressure:	N/D	Vapor Density:	Greater than air
Odor Threshold:	N/D	Viscosity:	N/D
Solubility in Water:	N/D	Specific Gravity (water = 1):	0.9

Section 10 Stability and Reactivity

N/A: Not Applicable N/D: Not Determined N/E: Not Established N/R: Not Required Est.: Estimated

Stability: Stable, non-reactive.

Hazardous Polymerization: Will not occur.

Hazardous Decomposition Products: None known.

Conditions to Avoid: Heat, sparks, and open flames.

Section 11 Toxicological Information

Carcinogenicity: This material is not considered a carcinogen by IARC or NTP and is not regulated as a carcinogen by OSHA.

(See also Section 15 for related information)

Section 12 Ecological Information

Chemical Fate and Effects: No data available.

Section 13 Disposal Considerations

RCRA Hazardous Waste: This material, when discarded or disposed of, could be a hazardous waste according to federal regulations (40 CFR 261) due to the characteristic of ignitability (D001). The transportation, storage, treatment, and disposal of this waste must be conducted in compliance with 40 CFR 262, 263, 264, 268, and 270. Disposal can only occur in properly permitted facilities. Check state and local regulations for any additional requirements as these may be more restrictive than federal laws and regulations. Chemical additions, processing or otherwise altering this material may make the waste management information presented in this MSDS incomplete, inaccurate, or otherwise inappropriate.

Section 14 Transportation Information

Regulated by the DOT: Yes

DOT Proper Shipping Name: Paint

UN / NA Number: UN 1950

Section 15 Regulatory Information

CERCLA:

N/A: Not Applicable N/D: Not Determined N/E: Not Established N/R: Not Required Est.: Estimated

The Comprehensive Environmental Response Compensation and Liability Act of 1980 (CERCLA) requires notification to the National Response Center for releases of quantities of Hazardous Substances equal to or greater than the reportable quantities (RQs) in 40 CFR 302.4 (for CERCLA 102).

Components present in this product at a level which could require reporting under the statute are:

<u>Chemical Name</u>	<u>CAS#</u>	<u>Maximum Concentration (Wt. %)</u>
None	N/A	N/A

SARA Title III, section 311/312:

The Superfund Amendments and Reauthorization Act of 1986 (SARA) Title III requires emergency planning based on Threshold Planning Quantities (TPQs) and release reporting based on Reportable Quantities (RQs) in 40 CFR 355 (used for SARA 302, 304, 311 and 312).

Components present in this product at a level which could require reporting under the statute are:

<u>Chemical Name</u>	<u>CAS#</u>	<u>Maximum Concentration (Wt. %)</u>
None	N/A	N/A

SARA Title III, section 313:

The Superfund Amendments and Reauthorization Act of 1986 (SARA) Title III requires submission of annual reports of release of toxic chemicals that appear in 40 CFR 372 (for SARA 313).

Components present in this product at a level which could require reporting under the statute are:

<u>Chemical Name</u>	<u>CAS#</u>	<u>Maximum Concentration (Wt. %)</u>
n-Butyl Alcohol	71-36-3	2 %

TSCA:

The components of this mixture are listed in the Toxic Substance Control Act Inventory of Chemical Substances.

This product contains the following chemicals which require export notification under section 12(b) of the TSCA regulation:

<u>Chemical Name</u>	<u>CAS#</u>	<u>TSCA Section</u>
Isopropanol	67-63-0	Sec. 4
Methy isobutyl ketone	108-05-4	Sec. 4
Acetone	67-64-1	Sec. 4
Methyl ethyl ketoxime	96-29-7	Sec. 4
n-Butyl Alcohol	71-36-3	Sec. 4

Section 16 Other Information

Legend: N/A: Not Applicable
N/E: Not Established

N/D: Not Determined
N/R: Not Required

N/A: Not Applicable N/D: Not Determined N/E: Not Established N/R: Not Required Est.: Estimated

STEL: Short Term Exposure Limit
cps: Centipoise
mppcf: million particles per cubic foot of air.
PPB: Parts Per Billion
TLV: Threshold Limit Value
ACGIH: American Conference of Governmental Industrial Hygienists
CPSC: Consumer Product Safety Commission
DOT: US Department of Transportation
FHSA: Federal Hazardous Substance Act
OSHA: Occupational Safety and Health Administration (US Dept. of Labor)
RCRA: Resource Conservation and Recovery Act
SARA: Superfund Amendment and Reauthorization Act
Skin: This substance has the potential to be absorbed systemically through the skin.
TSCA: Toxic Substance Control Act

C: OSHA Ceiling Value
mg/m³: milligrams per cubic meter
PPM: Parts Per Million
PEL: Permissible Exposure Limit
TWA: Time Weighted Average

HMIS Key

4 = Severe Hazard
 3 = Serious Hazard
 2 = Moderate Hazard
 1 = Slight Hazard
 0 = Minimal Hazard

Prepared By: Zinsser Health and Safety Manager, Regulatory Compliance Dept.
 173 Belmont Drive Somerset, NJ 08875 (732) 469-8100

Disclaimer: Zinsser Company, Inc. believes, to the best of its knowledge, information and belief, the information contained herein to be accurate and reliable as of the date of this material safety data sheet. However, because the conditions of handling, use, and storage of these materials are beyond our control, we assume no responsibility or liability for personal injury or property damage incurred by the use of these materials and make no warranty, expressed or implied, regarding the accuracy or reliability of the data or results obtained from their use. All materials may present unknown hazards and should be used with caution. The information and recommendations in this material safety data sheet are offered for the users' consideration and examination. It is the responsibility of the user to determine the final suitability of this information and data and to comply with all applicable international, federal, state, and local laws and regulations.

N/A: Not Applicable N/D: Not Determined N/E: Not Established N/R: Not Required Est.: Estimated

APPENDIX G

Post Precipitation Analysis Results

Method: GENERAL Sample Name: Be sample Operator:
Comment:
Run Time: 10/07/04 12:47 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Be3130
Line	313.042
Units	ppm
S	.0978
Stdev	.0012
%RSD	1.229
#1	.0986
#2	.0969

APPENDIX H

Equipment Performance and Calibration



Motorized Microliter Pipette

PERFORMANCE SPECIFICATIONS

13

ACCURACY

In this manual ACCURACY means the closeness with which the dispensed volume approximates the volume set on the pipette. Accuracy is specified as **Mean Error**, the maximum amount by which the mean value of a large number of replicate measurements of the same volume will deviate from the set volume. Although the pipette is designed to operate from 4° to 40°C, since the density of water changes with temperature, this specification applies to water in the temperature range of 21.5°C ± 1.0°C.

PRECISION

In this manual PRECISION means the "scatter" of individual measurements around the mean of a large number of replicate measurements of the same volume. Also expressed as **Standard Deviation**.

Specifications for precision are generally tighter than for accuracy. In most experiments, where sample measurements are compared to standards, the precision specification will determine the accuracy of results as long as both samples and standards are measured with the same instrument.

Table 13.1 – Specifications

EDP-Plus Model	VOLUME SET μL	ACCURACY		PRECISION	
		%	μL	%	μL
10 μL	0.5	5	± 0.025	2.8	≤ 0.014
	1	2.5	0.025	1.2	0.012
	5	1.5	0.075	0.6	0.03
	10	1.0	0.1	0.4	0.04
25 μL	2.5	6.0	0.15	2.0	0.05
	12.5	1.2	0.15	0.4	0.05
	25	1.0	0.25	0.3	0.075
100 μL	10	3.0	0.3	1.0	0.1
	50	0.8	0.4	0.2	0.1
	100	0.8	0.8	0.2	0.2
250 μL	25	2.0	0.5	0.6	0.15
	125	0.8	1.0	0.15	0.19
	250	0.8	2.0	0.15	0.38
1000 μL	100	3.0	3.0	0.6	0.6
	500	0.8	4.0	0.13	0.65
	1000	0.8	8.0	0.13	1.3
2500 μL	.25 mL	3.2	8.0	0.8	2.0
	1.25 mL	0.8	10.0	0.16	2.0
	2.5 mL	0.8	20.0	0.12	3.0
10ML	1 mL	5.0	50.0	0.6	6.0
	5 mL	1.0	50.0	0.2	10.0
	10 mL	0.8	80.0	0.16	16.0

Specifications are subject to change without notice, and apply to PIPETTE mode operation. Except for EP-10: if the relative and absolute values differ, use the greater value.

To determine the accuracy and precision for your model at a specific volume setting, multiply the volume setting (μL or mL) by the % specification for that model. Compare the calculated μL to the μL specification above for your EDP-Plus model. The larger μL value is the specification for your volume setting. (For EP-10ML models the specification is the value calculated as indicated.)

Example:

Model EP-1000 (1000 μL liquid end installed on EDP-Plus.)
You wish to determine accuracy at a volume setting of 475 μL .

From Table 13.1, accuracy for the EP-1000 is 3 μL or 0.8%.

$$(0.8 \times 475 \mu\text{L}) \div 100 = 3.80 \mu\text{L}$$

As 3.80 μL is more than 3 μL , accuracy at 475 μL is $\pm 3.8 \mu\text{L}$.
(You can make the same calculations for precision.)

EDP-Plus Model 100 μl was used set for 100 μl . Accuracy calculation: $(0.8 \times 100 \mu\text{l}) / 100 = 0.8 \mu\text{l}$

$0.8 / 1000 = 0.0008 \text{ ml}$; $(0.0008 \text{ ml})(10 \mu\text{g/ml}) = \pm 0.008 \mu\text{g}$

Calibration Worksheet

Job Type:
Scheduled Calibration

T004510092001.00

Status:10
WS2255-08012000

Calibration Description	Due Date	Interval	Units	Day of Week	Cal Area	Min Int	Max Int	Est Hrs	Last Cal Date
GRADIENT	23-Sep-2001	104	Week	Sunday	E08M	52	104	7	23-Sep-2000

Equipment Information:		Manufacturer		Model	
54913		ASSOCIATED CHAMBER			
Description	Type	Size		# Cal Seals	
Temperature Chamber				0	
Equipment Marking		System ID			
ASSOCIATED CHAMBER		CE54913			
Own Dept	Chamber Code	Classification	User Type	CMS-Tagkey	
835		UNCL	INSP	E2046-08012000	

Equipment Location:								
Dept	Post Loc.	Building	Room	Cabinet	Shelf	Drawer	Cart	Other
835	SF1	MSB	141					

Documents:		Document Name		Suffix	Issue	Classification	CMS-Tagkey
Document Type		CP5702			C	UNCL	D0013-01262000
CP - Calibration Procedure							

Recommended Equipment:		Equipment	Range	Accuracy
MULTI SENSOR			-100 TO 200 DEG F	+/- 1 DEG F
TEMP SYSTEM			200 TO 600 DEG F	+/- 2 DEG F

Ancillary Equipment: None

Special Instructions:
 PREVENTATIVE MAINTENANCE PERFORMED. NOTE: PLACE CHECK MARK IN BLANKS WHEN CONDITIONS ARE SATISFACTORY. CHECK DOORS AND OPENINGS FOR PROPER FIT, TIGHTNESS, BINDING, DRAGGING OR WARPAGE CONDITIONS.
 CHECKS LATCHES AND LOCKS FOR TIGHTNESS AND EFFECTIVENESS.
 CHECK FOR WORN OR DEFECTIVE GASKETS WHICH ALLOWS HEAT OR VAPOR LEAKAGE.
 CHECK ALL ALARMS SYSTEMS AND ASSOCIATED LIGHTS FOR PROPER OPERATION.
 VERIFY THAT THE SAFETY HIGH LIMIT CONTROL WILL SHUT DOWN THE OVEN IN THE EVENT OF AN OVER TEMPERATURE CONDITION.
 INITIATE A 659 FOR ANY REPAIRS MADE.
 IF PLANT MAINT SERVICES ARE NEEDED, INITIATE REQUEST TO PLANT MAINT.
 REQUEST RESPONSIBLE TEAM MANAGER TO SIGN OFF 659.
 INDICATE OVERALL CONDITION OF OVEN OR CHAMBER UNDER REMARKS.
 INSERT COPY OF THE GRADIENT 704 IN THE C OF C PACKET.

CP or Para No	Low	Nominal	High	OOT Reading
LOW TEMP CAL				
CP1	TO (AVG)	99	DEG F	
CP2	TR (AVG)	99.8	DEG F	
CP3	TL	99	DEG F	
CP4	TH	99	DEG F	
POS DEV				
CP5=(CP4-CP2)		-0.8	DEG F	TOL +/- 5 DEG F
NEG DEV				
CP6=(CP2-CP3)		0.8	DEG F	TOL +/- 5 DEG F
HIGH TEMP CAL				

Calibration Worksheet

Job Type:
Scheduled Calibration

T004510092001.00

Status:10
WS2255-0801200

Calibration Description	Due Date	Interval	Units	Day of Week	Cal Area	Min Int	Max Int	Est Hrs	Last Cal Date
GRADIENT	23-Sep-2001	104	Week	Sunday	E08M	52	104	7	23-Sep-2000
Equipment Id	Description		Dept	Post					
54913	Temperature Chamber		835	SF1					
CP or Para No	Low	Nominal		High		OOT Reading			
CP7	TO (AVG)	394	DEG F						
CP8	TR (AVG)	397.6	DEG F						
CP9	TL	397.4	DEG F						
CP10	TH	397.8	DEG F						
POS DEV									
CP11=(CP10-CP8)		0.2	DEG F		TOL +/- 10 DEG F				
NEG DEV									
CP12=(CP8-CP9)		0.2	DEG F		TOL +/- 10 DEG F				
Temperature	Barometer	Humidity	OOT - Int	Date Calibrated	Next Due Date				
70° ±	N/A	N/A	Y - 78 N - 104	9-18-01	9-21-03				
Stds Used last Calibration					Stds Used this Calibration				
Eq ID	Manuf	Model		Eq ID					
CN18088	OMEGA ENGINEERING	2176A		ME04057					
Tech	AMH	Date	Comments						
4822	9	9-18-01							

APPENDIX I

Lab Analytical Quality Control Documents

QUALITY CONTROL DATA

Lab Project Number: 6087770
 Client Project ID: Be Sampling WPWM 5256

QC Batch: 179853	Analysis Method: EPA 6010				
QC Batch Method:	Analysis Description: Metals Scan Wipe ICP				
Associated Lab Samples:	607561461	607561479	607561487	607561495	607561503
	607561511	607561529	607561537	607561545	607561578
	607561586	607561594	607561602	607561610	607561628
	607561636	607561644	607561651	607561669	607561792

METHOD BLANK: 607563566							
Associated Lab Samples:	607561461	607561479	607561487	607561495	607561503	607561511	607561529
	607561537	607561545	607561578	607561586	607561594	607561602	607561610
	607561628	607561636	607561644	607561651	607561669	607561792	

Parameter	Units	Blank Result	Reporting Limit	Footnotes
Beryllium	ug/100cm2	ND	0.100	

LABORATORY CONTROL SAMPLE: 607563574

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Footnotes
Beryllium	ug/100cm2	10.00	8.960	90	80-120	

QC Batch: 179854	Analysis Method: EPA 6010				
QC Batch Method:	Analysis Description: Metals Scan Wipe ICP				
Associated Lab Samples:	607561677	607561685	607561693	607561701	607561719
	607561727	607561735	607561743	607561750	607561768
	607561776	607561784	607561800	607561818	607561826
	607561834	607561842	607561859	607561867	607561875

METHOD BLANK: 607563582							
Associated Lab Samples:	607561677	607561685	607561693	607561701	607561719	607561727	607561735
	607561743	607561750	607561768	607561776	607561784	607561800	607561818
	607561826	607561834	607561842	607561859	607561867	607561875	

Parameter	Units	Blank Result	Reporting Limit	Footnotes
Beryllium	ug/100cm2	ND	0.100	

LABORATORY CONTROL SAMPLE: 607563590

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Footnotes
Beryllium	ug/100cm2	10.00	9.160	92	80-120	

QC Batch: 179856	Analysis Method: EPA 6010				
QC Batch Method:	Analysis Description: Metals Scan Wipe ICP				
Associated Lab Samples:	607562162	607562170	607562188	607562196	607562204
	607562212	607562220	607562238	607562246	607562253
	607562287	607562295			

METHOD BLANK: 607563624							
Associated Lab Samples:	607562162	607562170	607562188	607562196	607562204	607562212	607562220
	607562238	607562246	607562253	607562287	607562295		

Parameter	Units	Blank Result	Reporting Limit	Footnotes
Beryllium	ug/100cm2	ND	0.100	

LABORATORY CONTROL SAMPLE: 607563632

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Footnotes
Beryllium	ug/100cm2	10.00	9.350	94	80-120	

QUALITY CONTROL DATA PARAMETER FOOTNOTES

Consistent with EPA guidelines, unrounded concentrations are displayed and have been used to calculate % Rec and RPD values.

- LCS(D) Laboratory Control Sample (Duplicate)
- MS(D) Matrix Spike (Duplicate)
- DUP Sample Duplicate
- ND Not detected at or above adjusted reporting limit
- NC Not Calculable
- J Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit
- MDL Adjusted Method Detection Limit
- RPD Relative Percent Difference

Dilution Corrected Concentration Per Analyte

Run	Label	TimeStamp	9Be ppb
1	Blk	10/21/04 12:16:08 PM	(P)0.021
2	Blk	10/21/04 12:16:19 PM	(P)0.020
3	Blk	10/21/04 12:16:31 PM	(P)0.018
	Mean of Blk	10/21/04 12:16:08 PM	(P)0.019
	SD of Blk		(P)0.002
	%RSD of Blk		(P)8.384
1	10 PPB	10/21/04 12:19:24 PM	(P)9.366
2	10 PPB	10/21/04 12:19:36 PM	(P)8.787
3	10 PPB	10/21/04 12:19:48 PM	(P)8.889
	Mean of 10 PPB	10/21/04 12:19:24 PM	(P)9.014

	SD of 10 PPB		(P)0.309
	%RSD of 10 PPB		(P)3.432
1	30 PPB	10/21/04 12:22:47 PM	(P)29.400
2	30 PPB	10/21/04 12:22:59 PM	(P)26.770
3	30 PPB	10/21/04 12:23:10 PM	(P)29.260
	Mean of 30 PPB	10/21/04 12:22:47 PM	(P)28.480
	SD of 30 PPB		(P)1.480
	%RSD of 30 PPB		(P)5.199
1	50 PPB	10/21/04 12:26:07 PM	(P)49.330
2	50 PPB	10/21/04 12:26:18 PM	(PM)53.080
3	50 PPB	10/21/04 12:26:30 PM	(P)47.540
	Mean of 50 PPB	10/21/04 12:26:07 PM	(PM)49.980
	SD of 50 PPB		(PM)2.824
	%RSD of 50 PPB		(PM)5.650

Nominal Concentration (ppb)	Mean Instrument Reading (ppb)
0	0.019
10	9.014
30	28.40
50	49.98

APPENDIX J

Sampling Results

Table 1. Sample Description and Analytical Results

Plate*	Vial	Sample Identification	Be Present (μg)	1st Results (μg)	Removal Efficiency (%)	KCP Results (μg)	Removal Efficiency (%)
D1	D1	Spike 1 Dry Wipe	0.978	ND	NA	0.1003	10.256
D2	D2	Spike 1 Dry Wipe	0.978	ND	NA	0.1257	12.853
D3	D3	Spike 1 Dry Wipe	0.978	ND	NA	0.1257	12.853
D4	D4	Spike 1 Dry Wipe	0.978	ND	NA	0.0702	7.178
D5	D5	Spike 1 Dry Wipe	0.978	ND	NA	0.0535	5.470
D6	D6	Spike 1 Dry Wipe	0.978	ND	NA	0.0791	8.088
D7	D7	Spike 1 Dry Wipe	0.978	ND	NA	0.0554	5.665
D8	D8	Spike 1 Dry Wipe	0.978	ND	NA	0.0492	5.031
D9	D9	Spike 1 Dry Wipe	0.978	ND	NA	0.0874	8.937
D10	D10	Spike 1 Dry Wipe	0.978	ND	NA	0.103	10.532
D11	D11	Spike 1 Dry Wipe	0.978	ND	NA	0.0875	8.947
D12	D12	Spike 1 Dry Wipe	0.978	ND	NA	0.1195	12.219
D13	D13	Spike 1 Dry Wipe	0.978	ND	NA	0.1109	11.339
D14	D14	Spike 1 Dry Wipe	0.978	0.127	13%	0.1276	13.047
D15	D15	Spike 1 Dry Wipe	0.978	ND	NA	0.0744	7.607
D16	D16	Spike 1+1 Dry Wipe	1.978	0.862	74%	1.057	53.438
D17	D17	Spikeless Dry Wipe	0	ND	NA	0.0083	
D18	D18	Spikeless Dry Wipe	0	ND	NA	0.0067	
D19	D19	Spikeless Dry Wipe	0	ND	NA	0.0058	
	D20	Dry Field Blank	0	ND	NA	0.0067	
	D21	Dry Field Blank	0	ND	NA	0.006	

Table 1. Sample Descriptions and Analytical Results (continued)

	D22	Dry Field Blank	0	ND	NA	0.0075	
	D23	Spike 1Dry No Wipe	1	0.664	66%	0.7891	78.91
W1	W1	Spike 1 Ghost Wipe	0.978	0.196	20%	0.3274	33.476
W2	W2	Spike 1 Ghost Wipe	0.978	0.183	19%	0.2636	26.953
W3	W3	Spike 1 Ghost Wipe	0.978	ND	NA	0.1268	12.965
W4	W4	Spike 1 Ghost Wipe	0.978	0.142	15%	0.2267	23.180
W5	W5	Spike 1 Ghost Wipe	0.978	0.137	14%	0.1755	17.945
W6	W6	Spike 1 Ghost Wipe	0.978	0.149	15%	0.2497	25.532
W7	W7	Spike 1 Ghost Wipe	0.978	0.104	11%	0.1205	12.321
W8	W8	Spike 1 Ghost Wipe	0.978	0.212	22%	0.3029	30.971
W9	W9	Spike 1 Ghost Wipe	0.978	0.162	17%	0.2585	26.431
W10	W10	Spike 1 Ghost Wipe	0.978	0.147	15%	0.2203	22.526
W11	W11	Spike 1 Ghost Wipe	0.978	0.179	18%	0.2554	26.115
W12	W12	Spike 1 Ghost Wipe	0.978	0.17	17%	0.2409	24.632
W13	W13	Spike 1 Ghost Wipe	0.978	ND	NA	0.1562	15.971
W14	W14	Spike 1 Ghost Wipe	0.978	0.14	14%	0.2098	21.452
W15	W15	Spike 1 Ghost Wipe	0.978	0.171	17%	0.2354	24.070
W16	W16	Spike 1+1 Ghost Wipe	1.978	1.01	85%	1.433	72.447
W17	W17	Spikeless Ghost Wipe	0	ND	NA	0.0027	
W18	W18	Spikeless Ghost Wipe	0	ND	NA	0.0055	
W19	W19	Spikeless Ghost Wipe	0	ND	NA	0.0069	
	W20	Ghost Wipe Field Blank	0	ND	NA	0.0049	
	W21	Ghost Wipe Field Blank	0	ND	NA	0.005	
	W22	Ghost Wipe Field Blank	0	ND	NA	0.0057	
	W23	Spike 1 Ghost No Wipe	1	0.89	89%	1.277	127.7
A1	A1	Spike 1 Alcohol Wipe	0.978	0.322	33%	0.4835	49.438
A2	A2	Spike 1 Alcohol Wipe	0.978	0.291	30%	0.3971	40.603
A3	A3	Spike 1 Alcohol Wipe	0.978	0.385	39%	0.5362	54.826
A4	A4	Spike 1 Alcohol Wipe	0.978	0.402	41%	0.5367	54.877
A5	A5	Spike 1 Alcohol Wipe	0.978	0.377	39%	0.5049	51.626
A6	A6	Spike 1 Alcohol Wipe	0.978	0.393	40%	0.5736	58.650
A7	A7	Spike 1 Alcohol Wipe	0.978	0.259	26%	0.3363	34.389
A8	A8	Spike 1 Alcohol Wipe	0.978	0.456	47%	0.6656	68.057
A9	A9	Spike 1 Alcohol Wipe	0.978	0.459	47%	0.6576	67.239

Table 1. Sample Descriptions and Analytical Results (continued)

A10	A10	Spike 1 Alcohol Wipe	0.978	0.386	39%	0.433	44.274
A11	A11	Spike 1 Alcohol Wipe	0.978	0.39	40%	0.503	51.431
A12	A12	Spike 1 Alcohol Wipe	0.978	0.375	38%	0.5017	51.299
A13	A13	Spike 1 Alcohol Wipe	0.978	0.292	30%	0.3957	40.460
A14	A14	Spike 1 Alcohol Wipe	0.978	0.292	30%	0.4057	41.483
A15	A15	Spike 1 Alcohol Wipe	0.978	0.375	38%	0.4947	50.583
A16	A16	Spike 1+1 Alcohol Wipe	1.987	1.22	83%	1.482	
A17	A17	Spikeless Alcohol Wipe	0	ND	NA	0.0057	
A18	A18	Spikeless Alcohol Wipe	0	ND	NA	0.0066	
A19	A19	Spikeless Alcohol Wipe	0	ND	NA	0.0061	
A20	A20	Alcohol Wipe Paint Only	0	ND	NA	0.0045	
	A21	Alcohol Wipe Field Blank	0	ND	NA	0.0041	
	A22	Alcohol Wipe Field Blank	0	ND	NA	0.004	
	A23	Alcohol Wipe Field Blank	0	ND	NA	0.0047	
	A24	Alcohol Blue Glove Wipe	NA	ND	NA	0.0046	
	O1	Pre-Oven Wipe	NA	ND	NA	0.0045	
	O2	Post-Oven Wipe	NA	ND	NA	0.0103	
58 plates	72 vials						
Mean Dry Recovery	9.34	SDdry	2.80				
Mean Wet Recovery	22.97	SDwet	6.05				
Mean Aol Recovery	50.62	SDaol	9.54				

Appendix K

Analysis of Variance

Table 2. Statistical Evaluation of Results

% Removal Dry	% Removal Water	% Removal Alcohol
10.26	33.48	49.44
12.85	26.95	40.6
12.85	12.97	54.83
7.18	23.18	54.88
5.47	17.94	51.63
8.09	25.53	58.65
5.66	12.32	34.39
5.03	30.97	68.06
8.94	26.43	67.24
10.53	22.53	44.27
8.95	26.11	51.43
12.22	24.63	51.3
11.34	15.97	40.46
13.05	21.45	41.48
7.61	24.07	50.58

SUMMARY		α 0.05			
Groups	Count	Sum	Average	Variance	
Dry	15	140.03	9.34	7.83	
Water	15	344.53	22.97	36.64	
Alcohol	15	759.24	50.62	91.08	

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	13271.68	2	6635.84	146.87	1.1E-19	3.22
Within Groups	1897.68	42	45.18			
Total	15169.36	44				

Post Hoc Multiple F Test

Since the ANOVA showed significant difference in the three arrays of removal efficiencies, a *post hoc* test is required to determine the significance of difference between each of the three possible pairs of comparisons (dry vs. water; dry vs. alcohol; and water vs. alcohol). This was a planned comparison in that the experimental hypothesis predicted that methanol-moistened wipe sampling would remove more oil-film beryllium surface contamination than water-moistened wipe sampling, which would remove more of the deposit than dry wipe sampling. Therefore, the multiple F test is the

appropriate *post hoc* test. In the multiple F test, the critical difference (CD) was calculated from the critical F (F_{crit}), within-group variance (VAR_{wg}), and number of samples per array (n). The critical F (for $\alpha = 0.01$) was found in an “F Distribution” table based on degrees of freedom between the two groups being compared ($df = 1$), and within the samples of the arrays ($df = 42$).¹²²

$$n = 15; df = (1, 42); F_{crit0.01} = 7.27$$

$$CD_{0.01} = (2F_{crit})^{1/2}(VAR_{wg}/n)^{1/2} = (2 \times 7.27)^{1/2}(45.21/15)^{1/2} = (3.813)(1.736) = 6.62$$

Differences between the means of the removal efficiencies were compared to the critical difference. Those differences greater than the CD were significant at the 0.01 probability level. As seen in Table 3, all three paired comparisons demonstrated significant differences.

Table 3. Mean Removal Efficiency Differences

	Dry 9.33	Water 22.97	Alcohol 50.62
Dry 9.33		13.64	41.29
Water 22.97			27.65
Alcohol 50.62			

As predicted, methanol-moistened wipe sampling demonstrated significantly greater removal efficiency than water-moistened wipe sampling, which provided significantly greater removal efficiency than dry wipe sampling when removing beryllium surface contamination associated with metal working fluid residue ($p < 0.01$).

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