Vacuum sampling techniques for industrial hygienists, with emphasis on beryllium dust sampling†‡

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Received 1st February 2006, Accepted 18th May 2006
First published as an Advance Article on the web 25th May 2006
DOI: 10.1039/b601572g

The U.S. Department of Energy (DOE) *Chronic Beryllium Disease Prevention Program Rule, 10 CFR Part 850* became effective in 2000 in response to the prevalence of Chronic Beryllium Disease (CBD) in workers. The rule requires surface and air monitoring for beryllium to determine exposure levels and the evaluation of the effectiveness of controls used to minimize or eliminate that risk. The most common methods for surface sampling use wet or dry wipes. Wipe sampling techniques may be impractical for many surfaces common to most buildings such as cinder block, textured wall surfaces, fabric and carpet. Vacuum sampling methods have been developed for the evaluation of lead or pesticides on residential surfaces such as carpets, bare floors and window sills. However, the current vacuum methods may be impractical for many workplace situations such as sampling of protective clothing, complex facility structures, or equipment surfaces. Recent work using vacuum sampling for potential bio-terrorism agents such as anthrax spores may have significant application to industrial hygiene evaluations of the workplace and may be extendable for use in sampling of metals such as beryllium. Validated vacuum sampling methods that provide meaningful data would be of great value to industrial hygienists in identifying areas having surface contamination, evaluating existing controls and work practices and determining the potential of toxic material on surfaces to become airborne and present a potential risk to workers and the public. This article discusses various vacuum sampling methodologies and recommends harmonization of sampling methods.

Introduction

Chronic Beryllium Disease (CBD) is an immune disease that affects the lungs. In affected individuals, the body reacts to inhalation of beryllium particulate matter by forming tissue in the lungs that can reduce oxygen transfer.[1] Sensitization is the precursor to the disease and is defined as a double positive result to a blood test showing a reaction to beryllium.[2] Sensitization can occur shortly after the first exposure, while the onset of clinical symptoms for the actual disease occurs, on average, seven to ten years from first exposure. While it is known that inhalation causes the disease, a protective airborne exposure level is unknown.[1,2] Nor is it known if skin exposure plays a role in beryllium sensitization, although some evidence suggests this is a potential action.[3]

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† Presented in part at the 2nd Symposium on Beryllium Particulates and Their Detection, 8th–9th November 2005, University Park Marriot, Salt Lake City, UT, USA.

Surface contamination limits and hazard assessment.

Regulatory surface contamination limits at U.S. Department of Energy (DOE) sites were established by the *Chronic*

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Beryllium Disease Prevention Program Rule.[4] Those limits are 0.2 μg/100 cm^2 for release of beryllium-contaminated equipment to a non-beryllium area and 3 μg/100 cm^2 as a housekeeping limit inside areas where beryllium work is performed. Prior to the DOE rule, surface contamination limits were independently developed and used at locations where beryllium was routinely used, primarily at sites where weapons were designed, produced and stored. The surface limits, then and now, were developed based on practicality (*i.e.*, the surface could be cleaned to achievable levels). The limits were not based on known health risk, but rather on what was achievable, the site's experience and what appeared to be a reasonable and conservative level to protect workers' health.

Since the adoption of the rule,[4] DOE-funded facilities are required to evaluate their sites and identify active beryllium areas and legacy beryllium areas that could be contaminated. In addition, there are many formerly funded DOE sites where beryllium contamination concerns exist. Activities are underway to determine the extent of the current hazard by evaluating surface contamination and to test former workers for sensitization and disease. Yet it must be emphasized the problem extends beyond DOE sites: beryllium exposures to workers have been documented in a number of industries in the United States.[5]

Various DOE sites have developed methods to sample surfaces independently, but the primary methods used are wet or dry wipe methods.[6] Given the need to determine the extent of contamination in legacy areas that span from office buildings, to machine shops and to records storage facilities, there are a variety of surfaces that need to be assessed for contamination potential. This poses technical challenges, not only for the sampling method to be used for surfaces that are not easily wiped, but also for the level of beryllium contamination to assess whether (a) there is a risk, or (b) if the surface should be cleaned, and/or (c) if the contaminated equipment should be retained or disposed of.

Skin exposures and surface sampling

Beryllium particles entering a cut or abrasion in the skin can result in lesions, ulcerations and granulomas that do not heal properly until the beryllium is removed. Therefore, skin exposure in a workplace situation where skin is not normally protected (*e.g.*, an office worker with a paper cut) creates the risk of the wound becoming contaminated. Exposure of the skin to soluble beryllium compounds can result in dermatitis and hypersensitivity to beryllium.[7] A hypothesis is that individuals can become sensitized through skin exposure to fine particles (< 1 μm) combined with natural flexing movements. Studies have shown that particles can penetrate the intact stratum corneum,[3] and work into the epidermis and dermis, where they may initiate a cutaneous immune response. If beryllium particles penetrated the skin in this manner, the immune response could result in sensitization to beryllium. Because larger particulate matter can penetrate cut or abraded skin, the size of the particles does not need to be below 1 μm to be a hazard. Given these factors, it is important to understand and assess the risk of skin exposure. This increases the need to be able to identify surface contamination in order to evaluate and monitor work areas to reduce the risk of beryllium exposure to the skin.

Surface sampling methods and vacuum sampling

Wipe sampling methods have been used for years in efforts to assess surface contamination levels[8] and approximate the potential for dermal exposures.[9] As an alternative to wipe sampling, vacuum sampling has been proposed for instances where obtaining wipe samples is deemed impractical. Wipe sampling may not be recommended for rough and/or porous surfaces, so a viable alternative for consideration is sampling by means of a vacuum. But because vacuum methods can often collect large amounts of dirt and debris, there is some question as to when one should identify the sample in terms of bulk sampling, using the units of measure of percent by weight, *versus* using units of, *e.g.*, μg/100 cm^2.

While there is no standard method that is universally applicable to sample surfaces by use of a vacuum, there are many that have been used. A comparison of wipe and vacuum sampling methods showed that the vacuum method is inferior for sampling smooth, hard surfaces.[10,11] However, because some surfaces cannot be sampled easily using wiping methods, an alternative method is needed for these surfaces. Applications for residential contamination risks have been primarily developed and used to determine the extent of lead, pesticides and carcinogen contamination.[12,13] An emerging need is to assess surface contamination to biological agents. A prime example of this need occurred when U.S. Post Offices were contaminated with the contents of an envelope that contained *Bacillus anthracis* (anthrax).[14] Vacuum sampling methods have been used extensively, but harmonization of sampling protocols is still an elusive goal.[15] Yet there remains a need for effective vacuum sampling techniques for hazardous contaminants such as beryllium. It is noted that vacuum sampling as described in this review is not meant for the assessment of potential inhalation hazards.

Standardized vacuum sampling methods

ASTM Practice D7144. A new voluntary consensus standard, American Society for Testing and Materials (ASTM) D7144, has been published, which is based on the use of "microvacuum" sampling.[16] The method allows for ease of

Fig. 1 Microvacuum sampler for use with ASTM Practice D7144.

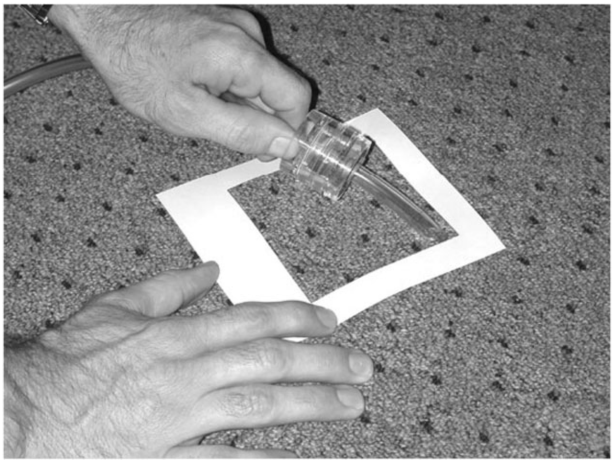

Fig. 2 Microvacuum sampling in accordance with ASTM D7144.

obtaining surface dust samples and the necessary equipment is readily available. The prior standard that this one replaced was ASTM E1973,[17] which was applicable to lead-in-dust sampling and was based on a published methodology.[18] The ASTM D7144 procedure uses a standard 37 mm three-piece air cassette with filter and backup pad.[19] The cassette is modified with an inlet that has flexible hose extension, with the hose cut at a 45° angle (Fig. 1). A portable, battery-powered, personal air sampling pump calibrated to 2.5 L min^{-1} is used as the suction source. The sampling area, *e.g.*, 100 cm^2, is vacuumed three times in succession (Fig. 2). To prevent cross-contamination, a new inlet hose and sampler are used for each sample. The entire contents of the cassette, *i.e.*, the filter plus the collected particulate matter, are targeted for subsequent analysis. Cassette inserts can be used for sampling if gravimetric analysis is desired. This technique is also described as an EPA method for dust sampling.[20]

Benefits of the ASTM D7144 sampling technique include: (a) low cost; (b) ready availability of typical industrial hygiene sampling equipment; (c) pre-assembled microvacuum samplers are commercially available; and (d) the method is very easy to put into practice. However, some concerns with the ASTM D7144 procedure include: (a) sample losses occur within the collection nozzle owing to static charge buildup during sample collection; (b) no agitation is used during sampling, which may result in a relatively lower collection efficiency as compared to methods where agitation is used; (c) a new inlet hose must be used for each sample, which results in additional waste; (d) the collection efficiency is unknown, but is significantly lower than 100%;[18] and (e) smaller dust particles will be sampled preferentially.[21] However, it must be pointed out that collection efficiency has not yet been fully evaluated for any beryllium vacuum sampling method.

ASTM Test Methods D5755 and D5756. There are other ASTM methods that are similar to ASTM D7144 which have been developed for sampling surface contamination and subsequent transmission electron microscopy (TEM) analysis for asbestos. They are ASTM Test Methods D5755[22] (asbestos structure number concentrations) and D5756[23] (asbestos mass concentration) for asbestos-microvacuum sampling and in-

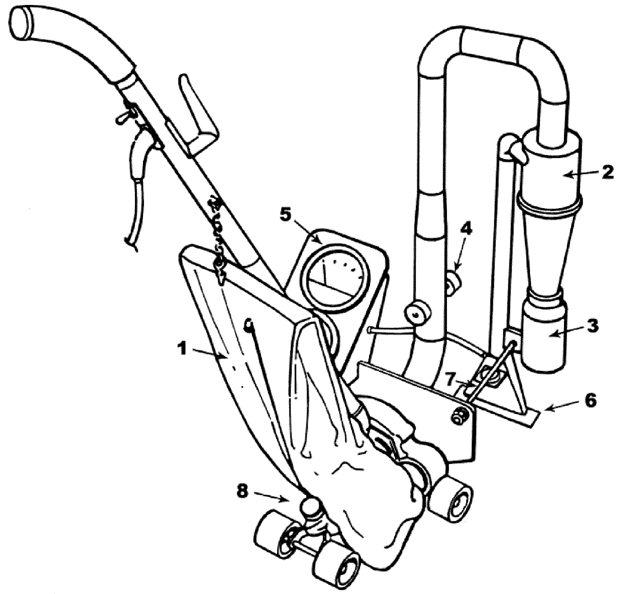

Fig. 3 High-volume vacuum sampling device for use with ASTM D5438, based on the HVS3 cyclone vacuum sampler: (1) commercial vacuum cleaner; (2) cyclone with 5 μm cut-point; (3) sample catch bottle; (4) flow control valve; (5) vacuum gauges; (6) 10-cm suction nozzle; (7) nozzle level adjustment screw; (8) platform level adjustment knob (Courtesy of Dr R. G. Lewis).

direct analysis of dust by TEM. The only major difference between ASTM D7144 and the two ASTM microvacuum methods for asbestos sampling and analysis is that a 25 mm, three-piece cassette commonly used for air sampling for asbestos analysis is used (in lieu of the 37 mm cassette).

ASTM Practice D5438. A voluntary consensus standard method, ASTM D5438,[24] uses a special vacuum cleaner known as the High Volume Sampler, or HVS3 (CS^3 Inc., Sandpoint, ID, USA), which is equipped with a cyclone attachment (Fig. 3) designed to collect particle sizes with a mean aerodynamic size of 5 μm or larger. The sampled particulate matter is deposited in a bottle that can be used for sample digestion. A filter can be added downstream of the cyclone to collect smaller sized particles down to 0.3 μm. The device is meant for sampling dust from carpets and bare floors. An evaluation of the HVS3 for sampling polycyclic aromatic hydrocarbons and polychlorinated biphenyls in household dust indicated that greater than 95% of these materials were collected by the cyclone.[25]

When using the D5438 sample collection procedure, the pressure gauges must be calibrated and the flow rate adjusted for different types of carpets. Parallel tapes are placed on the floor to demarcate the sampling areas. The HVS3 nozzle is wiped to remove prior contamination. The nozzle height is adjusted and the carpet vacuumed twice down the tapeline, then the second parallel tape is used until a total of 6–8 grams of material is collected. The sample bottle is removed and the surface area sampled is recorded.

Benefits of the ASTM D5438 procedure are: (a) the method uses agitation, thereby increasing dust pick-up efficiency; (b) the procedure has been reported as having a relatively high level of precision when compared to other methods using vacuum cleaners for dust sampling;[26] and (c) it is considered a recommended vacuum sampling method by NIOSH.[27] Disadvantages include: (a) a special vacuum device that is very costly is required; (b) there is a potential for cross contamination of samples; (c) the vacuum cleaner nozzle must be cleaned prior to each use; (d) a large amount of material must be collected, which may not be possible on some surfaces; (e) the method is cumbersome to conduct since the pressure gauges must be calibrated, the nozzle height adjusted, flow rate adjusted depending on surface type, tape must be placed on the sampling surface and then removed, *etc.*; and (f) the standard HVS3 does not have high-efficiency filtration, thereby allowing for the potential of ultra-fine particulate suspension due to the sampling activity itself unless a supplemental filter is added.

Fig. 4 Indoor Biotechnologies Mitest Dust Collector©.

Non-standard vacuum sampling methods

In many studies, a great number of non-standard vacuum methods have been used to obtain samples of dust from a variety of surfaces,[15] and some of the more widely used techniques are highlighted here.

The minivacuum method (MVM) or dust vacuum method (DVM) is similar to ASTM D7144 but with minor changes: the surface area sampled is 0.25 m^2 rather than the more commonly used surface area of 100 cm^2.[26] Also, the sampler is passed over the surface twice, as compared to three times for the ASTM D7144 protocol. Essentially the MVM/DVM method can be construed as a modification of the procedure described in ASTM D7144.

The ''GS80 method'' for vacuum sampling uses a commercially available GS80 (or the similar GS90) high-efficiency industrial vacuum (NILFISK of America, Inc., Malvern, PA, USA) that has three stages of filtration.[26,28] However, only the contents of the first-stage vacuum cleaner bag are analyzed. This vacuum sampling device can be used to collect dust from large surface areas.

A method used for a national survey of lead dust in the U.S. housing, hereto referred as the HUD vacuum method,[10] employs an in-line, 37 mm, three-piece filter cassette with a polytetrafluoroethylene (PTFE) nozzle that slips over the exterior of the cassette. A high-volume (16 L m^{-1}) rotary vane vacuum pump is used for suction. The method is similar to that described in ASTM D7144 except that a high-volume sampler, rather than a personal sampling pump, is used.

The High Volume Furniture Sampler (HVFS) is a cyclone with a flexible wand and a notched nozzle.[13] This method yielded surface sampling efficiencies above 80% on bare floors and collection efficiencies on foam cushions were reportedly as high as 90.5%.

The High Volume Tripod Sampler (HVTS) method is a modification to the HVS3 in which the sampling head is suspended from a tripod and has a portable vacuum cleaner that is slung over the shoulder. This method reportedly had an average efficiency on carpets comparable to the HVS3.[13] The HVTS shows lower collection efficiencies than the HVS3, but was consistent. This sampler is lower in cost than the HVS3.

Cyclone samplers were reported to be reliable for sampling lead in dust from floors or hard surfaces.[29] A modified method, the R&M Cyclone Sampler (CS^3 Inc., Sandpoint ID, OR, USA), used the same cyclone as the HVS3 device, with a flexible tubing sampling wand and nozzle. The nozzle/wand was attached to a hand-carryable vacuum device, which was used as the suction source. The sample collection vessel consisted of a conventional paper vacuum cleaner bag. This sampler is significantly lower in cost than the HVS3.

A wet vacuum sampling method was used in studies of street dust deposition.[30] Two different domestic canister-type vacuum cleaners were used: one with a water jet, the other without. In-line between the vacuum and the inlet was a plastic drum designed to be a hydrocyclone. Initially it was filled with one litre of water. An 80 cm × 80 cm area was sampled. For the vacuum equipped with a water jet, four litres of water was sprayed onto the surface as the inlet was moved at a set pace along four directions (*i.e.*, back and forth and side to side). The method using the vacuum without the water jet consisted of an initial 250 ml water deposit in the middle of the sampling location. This is followed by hand brushing and then vacuuming of the resulting slurry. Each procedure took approximately 20–30 minutes and showed large variability for collection efficiency of metals.[30]

A method used for sampling of *Bacillus anthracis*, *i.e.*, anthrax, employed an inner cone-shaped filtering sock (X-cell 100, Midwest Filtration, Fairfield, OH, USA).[31] A similar type of filter was fitted into a nozzle of a HEPA vacuum cleaner (Atrix International Inc., Burnsville, MN, USA) that has a potential flow rate of approximately 800 L min^{-1}. The socks were pre- and post-weighed in order to determine the sample mass collected. The vacuum cleaner nozzle was cleaned in between samples to reduce the potential for cross contamination.

The Mitest Dust Collector© (Indoor Biotechnologies, Charlottesville, VA, USA), developed to sample for dust mites, uses a molded plastic inlet wand that holds a filter sock (Fig. 4).[32] The wand is attached to a vacuum cleaner; the type of vacuuming device is unspecified. This technique suggests collecting four subsamples for 30 seconds, for a total sampling

Fig. 5 Microvacuum wet sampling method.

time of 2 minutes with a sampling area of 0.25 m^2. The device is easy to use and has been employed in a study wherein non-technicians obtained vacuum dust samples.[33]

The M-vac™ sampler (Microbial Vacuum Systems, Inc., Bozeman, MT, USA) is a wet vacuuming system wherein an aqueous buffer is gently sprayed on the sampling surface (Fig. 5). The liquid and dust is vacuumed and collected in a reservoir. The sampling head has a rotating area that agitates the surface. This method has been developed for biological sampling of public settings and sampling of food products.

Comparisons of sampling methods

To compare the uses of the above methods, it is clear that the majority of studies utilizing vacuum sampling techniques have been for sampling residential settings for lead contamination. There are a few methods that have been evaluated for biological hazards, some for allergic-type hazards to mold, dust mites and endotoxins and others, *e.g.*, for biological agents.[34,35] Relatively few efforts have been attempted to conduct vacuum sampling methods for beryllium. The most widespread methods used to date include the ASTM D5438 and ASTM D7144 procedures or slight modifications of these versions. Extensions of applications of vacuum sampling methods beyond locales investigated to date are required for proper evaluation and assessment of the various sampling techniques.

Several studies have been carried out in efforts to compare efficiencies of surface dust sampling methods. In one investigation, a study was conducted to evaluate four sampling methods, three of which were vacuum sampling methods.[26] The HVS3 sampler was found to be the vacuum method that proved most precise and highest in collection efficiency. As discussed earlier, the wipe sampling methods collect more particulate matter than the vacuum sampling methods,[10,11] and should be used when a vacuum sampling method is not needed. In other work, both wet and dry vacuum sampling methods were evaluated and high variabilities were cited for both sampling techniques.[30] Elsewhere, a comparison between swab, wipe and HEPA vacuum sock collection methods was made.[31] In this work, good agreement was found between the vacuum and wipe collection method, but agreement was poor with the swab sampling method. It was noted that contamination was still present after sampling for each technique. Another study compared two wipe sampling methods to a microvacuum sampling method on surfaces spiked with lead containing dust.[36] The authors reported thet the wipe sampling methods yielded higher recoveries on smooth hard surfaces, but that on carpet, the vacuum method had a significantly higher recovery.

Unfortunately quantitative information is often lacking for many studies where vacuum sampling methods were used and that is an issue that we wish to highlight through publication of this review. Hopefully, this will initiate discussions for a harmonized approach such that collected data can be compared and contrasted.

Despite a few successes, there is a general paucity of reliable performance data for vacuum sampling methods.[15] This is true of standardized protocols as well as of non-standard vacuum sampling methods. Further studies are necessary in order to address this knowledge gap.

Recommendations

To ensure comparability of data obtained from different sites and/or times, it is generally recommended that voluntary consensus standards be used.[37,38] However, some existing standard methods may suffer shortcomings as far as beryllium sampling is concerned. For instance, while ASTM D5438 (HVS3, Fig. 3) may have been proven to be the superior method for collection efficiency and precision, there are several concerns with using this technique for evaluating a potential DOE beryllium legacy area. The method is suitable only for sampling floors. The necessary equipment is costly and subject to cross-contamination of samples. Also, when put into practice, the ASTM D5438 method is time-consuming and not very user-friendly. However, the most significant concern is that the use of ASTM D5438 poses the risk of suspending ultra-fine particulate matter in the breathing zone, since this system does not rely on a HEPA vacuum. For this reason, the ASTM D5438 method is not generally recommended for sampling of dust that potentially contains beryllium.

ASTM D7144 (Microvacuum, Fig. 2) is another voluntary consensus standard method currently available for use by industrial hygienists for vacuum sampling of beryllium particles. This standard was recently developed in coordination with experts from DOE sites and it is hoped that ASTM D7144 will fill a need for a reliable vacuum sampling method which was previously lacking. Yet there remains a concern of low collection efficiency when using this practice, thus improved techniques are desired.

Improved vacuum sampling methods for use in beryllium areas are still needed. New or modified methods should have the following features: (a) they should be easy to use; (b) they should be low in cost; (c) they should eliminate cross contamination; (d) their use should not cause settled dust to become airborne; (e) samples should be easy to transport; and (f) subsequent sample preparation and analysis should be straightforward and uncomplicated. Methods recently developed using HEPA vacuum cleaners that are equipped with an inlet that has a filter sock show great promise. Vacuum sampling methods that have been developed for biological sampling are potentially applicable to sampling of dust that also may contain metal contaminants such as beryllium.

There are somewhat opposing positions on the need for agitating a surface while collecting a vacuum sample. While agitation may significantly improve the efficiency of the sample collection,[28] there is concern that agitation would resuspend dust, possibly causing an exposure to the sampler. However, the very act of moving something over a surface will resuspend some portion of dust. For instance, a wipe sample method can suspend dust by the disturbance of air caused by the moving hand. Therefore, when developing a vacuum sampling method, it is advised to look at the flow rate and sampler inlet design to ensure that the particulate that is being suspended is captured by the sampling method. Vacuum sampling techniques should minimize the possibility of dust resuspension, whether agitation is used or not.

Concluding remarks

As shown in this document, there are numerous vacuum sampling methods for conducting surface sampling. In addition, there are many methods for sampling surfaces, not discussed here. The most common method for sampling surfaces for beryllium contamination is the wet or dry wipe using a filter media. This method is effective in non-porous, smooth surfaces. However, there are many surfaces that limit this method. Surfaces that are porous or rough tend to cause pillage of the filter media reducing the collection efficiency. High surface dust loading can reduce the effectiveness of the wet wipe due to the dust absorbing the moisture. Therefore, there are some surfaces that call for sampling with an alternative vacuum sampling method as the wet or dry wipe is impractical.

A number of vacuum sampling methods using a variety of sampling media and vacuum sources have been developed and tested to varying levels. It is probably more important to select, refine and standardize a few of these methods, rather than endeavor to develop entirely new techniques. For instance, a high volume HEPA-filtered vacuum cleaner type system should be selected and standardized for larger scale sampling of, for example, facilities and structures. Likewise, a standardized low volume sampling pump/filter system should be used for smaller scale sampling of items such as personal protective equipment and small areas.

Standardizing methods is important for attaining consistency of results and ensuring the ability to determine compliance with surface contamination regulations and guidelines for beryllium and other toxic materials. Standardization is also needed to allow for comparisons between vacuum and other surface sampling methods.

Acknowledgements

We thank the members of the Beryllium Health and Safety Committee for their invaluable input and guidance. This work was supported in part by the U.S. Department of Energy. Chandran Achutan, Eugene Kennedy and Ken Martinez of CDC/NIOSH kindly reviewed the draft manuscript.

Disclaimer: Mention of company names or products does not constitute endorsement by the U.S. Department of Energy or the Centers for Disease Control and Prevention. The findings and conclusions in this paper are those of the authors and do not necessarily represent the views of the National Institute for Occupational Safety and Health.

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