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The Real Issue with Wall Deposits in Closed Filter Cassettes—What's the Sample?

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The measurement of aerosol dusts has long been utilized to assess the exposure of workers to metals. Tools used to sample and measure aerosol dusts have gone through many transitions over the past century. In particular, there have been several different techniques used to sample for beryllium, not all of which might be expected to produce the same result. Today, beryllium samples are generally collected using filters housed in holders of several different designs, some of which are expected to produce a sample that mimics the human capacity for dust inhalation. The presence of dust on the interior walls of cassettes used to hold filters during metals sampling has been discussed in the literature for a number of metals, including beryllium, with widely varying data. It appears that even in the best designs, particulates can enter the sampling cassette and deposit on the interior walls rather than on the sampling medium. The causes are not well understood but are believed to include particle bounce, electrostatic forces, particle size, particle density, and airflow turbulence. Historically, the filter catch has been considered to be the sample, but the presence of wall deposits, and the potential that the filter catch is not representative of the exposure to the worker, puts that historical position into question. This leads to a fundamental question: What is the sample? This article reviews the background behind the issue, poses the above-mentioned question, and discusses options and a possible path forward for addressing that question.

Keywords exposure assessment, workplace monitoring, air sampling

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INTRODUCTION

For more than a century, sampling of airborne dust has been a key element of workplace exposure assessment. Reviews of the history of workplace aerosol sampling procedures have been provided by Walton and Vincent(1) and Harper.(2) While the equipment and techniques utilized for dust sampling have evolved over time, the closed-face cassette (CFC), developed over a half-century ago, is still widely used for particulate sampling in the United States. Because early collection devices were basically open-face filter holders, what was collected on the filter was considered to be the sample.

Devices such as the CFC introduce the possibility that some particulate might be deposited elsewhere, such as on the interior walls of the cassette. Over the last 20 years, this possibility has been touched on in the literature.(2,3) Introduction of particle size-selective sampling conventions in the mid-1990s (4) has given attention to particulate not effectively sampled by the CFC, and to concerns that particulate not captured on the filter may be of concern in proper characterization of workplace exposure.

Recent work by Harper and Demange(3) has brought about a renewed focus on the issue of wall deposits, and whether they should be included as part of samples collected for exposure assessment. ASTM International Subcommittee D22.04 on Workplace Air Quality, in recent standards under its jurisdiction, has included a statement on wall deposits.(5) The Beryllium Health and Safety Committee (BHSC), a group composed primarily of federal and contractor personnel from a variety of U.S. government agencies, and the sponsor of the 2005 and 2008 Symposia on Beryllium Particulates and Their Detection, has also discussed the issue. This article is an outgrowth of these discussions.

The purpose of this article is to provide a brief review of the background behind the wall deposits issue as a means to frame the question, previously posed by Harper and Demange,(3) as to what the actual sample is, or should be, in the specific context of beryllium. Options for addressing the issue, and a possible path forward for resolving the open questions, are then presented. The applicability of these options may vary when considering particulate other than beryllium.

This article provides a limited discussion of fate and transport of particulate in the respiratory system. For more detailed information, the reader is referred to a literature review by the National Academy of Sciences,$^{(6)}$ documentation supporting the Threshold Limit Value (TLV®) for beryllium and compounds,$^{(7)}$ and to environmental toxicology textbooks such as the one written by Zakrzewski.$^{(8)}$

This article also focuses on beryllium sampling for compliance with occupational exposure limits (OELs). The authors recognize that this is only one aspect of characterization and assessment of occupational exposures. The reader is referred to publications on exposure assessment strategies, such as the book published by the American Industrial Hygiene Association (AIHA).$^{(9)}$

BACKGROUND

In the early part of the 20th century, particle concentrations were commonly measured in terms of number.$^{(2,10)}$ The impinger sampler was used for this purpose, as it collected aerosol dust into a liquid, from which a suspension of particles in a drop could be placed on a microscope slide for counting. All particles below a certain limit (e.g., 5 or 10 μm projected area diameter) were counted and these data were used to determine exposure.

In 1944, the U.S. Bureau of Mines recommended the use of mixed cellulose-ester (MCE) membrane filters$^{(11,12)}$ based on their applicability to various analytical methods, including gravimetric, microscopic, and chemical analytical techniques. Filter holders were developed to allow for exposure determination to be based on both number of particles and concentration of a particular analyte of interest.

These early holders, designed both for gravimetric and chemical analyses, were made of stainless steel and were generally supports that exposed the filter to the air (e.g., open face cassettes). They had the drawback of leaving the filter free to be tampered with or damaged. The CFC, which houses 37 mm diameter filters, was developed in 1956 for "clean-room" analysis.$^{(13)}$ The 4 mm entry inlet made the filter much less likely to be accidentally or deliberately damaged, which contributed to its growth in popularity. Various methods for dust and particulate collection for gravimetric and chemical analysis were validated using these cassettes.

Although further investigations$^{(14,15)}$ concerning the performance of the CFC compared to the open-faced cassette tended to solidify its preference among practitioners, these evaluations did not compare the CFC with any performance standard for collection as one did not exist at that time for dust sampling.

Throughout that intervening period, the main focus of workplace monitoring was on particles smaller than about 10 μm aerodynamic equivalent diameters (AED), which are sampled relatively efficiently by the CFC.$^{(15)}$ When the CFC was introduced, it was described as a method for total suspended particulate$^{(16)}$; however, the data now available suggest that such a description was clearly a misnomer, since the CFC is less efficient for particles larger than 10 μm AED.

In the late 1980s the concepts behind the inhalability of larger particles arose$^{(17)}$ In 1993, a convention was agreed upon. It was based on a body of measurements of human inhalation efficiency, pushing inhalability to particles up to 100 μm AED. This is now referred to as the Inhalable Sampling Convention and was published in a standard, ISO 7708.$^{(4)}$ Recently, the American Conference of Governmental Industrial Hygienists (ACGIH®) accepted the ISO convention and proposed a number of threshold limit values, (TLVs), including one for beryllium, based on this convention. All of this has led to the interest in examining samplers for their ability to meet the inhalable convention.

INHALABLE CONVENTION AND WALL DEPOSITS

Various samplers have since been tested for their aspiration efficiency against the ISO 7708 model.$^{(18)}$ The sampler most closely matching the convention was the U.K. Institute of Occupational Medicine (IOM) sampler developed in 1986.$^{(19)}$ This sampler is popular in Europe but not as popular in the United States. The sampler is marketed as reusable, and the cost is higher than disposable samplers such as the CFC. In addition, methods to ensure cleanliness of the sampler for reuse are time-consuming and costly. Interestingly, even in the early years of use of the IOM, internal wall deposits were recognized when using this sampler, with Mark and Vincent$^{(19)}$ recommending that they be accounted for in the analysis.

Other samplers have been evaluated against the inhalable convention.$^{(20)}$ The Respicon sampler$^{(21)}$ has three stages; combining the results of all three has been shown to provide a reasonable match of the inhalable convention (if the manufacturer correction factor of 1.5 is not used). However, this sampler is relatively expensive, and should be closely monitored and the unit cleaned to prevent plugging of the first receiving tube. Another sampler being evaluated is the Button sampler.$^{(22)}$ An advantage to this sampler is that there are no internal walls and thus no wall deposits to consider. However, its performance against the inhalable convention has not been explicitly tested.$^{(23,24)}$ This sampler is also non-disposable and requires cleaning for re-use.

The recent study by Harper and Demange$^{(3)}$ postulates that wall deposits, those particulates that impact and settle on the wall of a sampler and not on the filter, could be a significant part of sample aspiration for the CFC. Since the CFC is efficient for particulate up to 10 μm,$^{(25)}$ it can be supposed that particulate on the walls is in the 10 to 100 μm range, but data to support that premise are lacking.

Wall deposits have been included in the analysis since the early 1980s by the Institut National de Recherche et de Sécurité (INRS) in France. The U.S. Occupational Health and Safety Administration (OSHA), in the 1976 version of its Atomic Absorption Spectrometry (AAS) analytical method, I-1, first specified that if loose dust was present, the cassette should be rinsed with deionized water and then, if necessary, be wiped out with a clean filter.$^{(26)}$

Later versions of OSHA sampling and analysis methods(27) included wall deposits in its gravimetric procedure and some of its chemical analyses procedures, although primarily for visible loose dust. More recently, OSHA has called for inclusion of wall deposits for hexavalent chromium(28) and for metals generally.(29) In the National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods* (NMAM), the concept of wall deposits is discussed in the preamble.(30)

Studies have been conducted to evaluate the adequacy of the CFC for the inhalable convention.(31–33) It has generally been found not to meet the standards of the inhalable convention when operated under the assumption that the "sample" is represented by the "filter catch." At issue is the amount of particulates that deposit on the walls of the CFC sampler. The causes for these depositions are not yet well understood. Some particles possess a static charge which can lead to an electrostatic deposition on the walls. Particles may also bounce off the filer and onto the walls. Particle size, particle density and airflow turbulence may also be contributing factors.(25) When analysis of particulates from the CFC includes wall deposits, the performance of the CFC more closely matches the inhalable convention.(18,34–35) For beryllium, data are limited to four points in the Harper and Demange study,(3) but the same trend is found for a large number of metals.

Thus, the data reported in the literature have established that particulates can adhere on the sides of the polystyrene cassettes during sampling in sometimes large but inconsistent amounts of the total sample.(34–37) While hardly new, this information has gained added attention for beryllium exposure monitoring for several reasons. First, the CFC is the primary sampler used for beryllium exposure assessment. Although it can be argued that the CFC has been used since the late 1950s, without including wall deposits, there is a legitimate concern that people can inhale these particles that are collecting on the wall of the CFC and we are not measuring them.

It can also be argued that for beryllium, particles above 10 μm are not likely to be retained in the lung, meaning that particles below this size, which are efficiently sampled by the CFC, are more important. Second, ACGIH has adopted a new TLV for beryllium based on sampling the inhalable fraction. (7) As noted earlier, without inclusion of wall deposits, available data suggest that the CFC filter catch does not meet the inhalable convention.

The U.S. Department of Energy (DOE) is among the world leaders in workplace beryllium sampling(38) for a variety of purposes, including facility characterization, process control evaluations, and regulatory compliance. In the area of compliance, the DOE Worker Safety and Health Program(39) invokes the 2005 ACGIH TLV, but not necessarily changes made to TLVs after 2005. However, DOE's Chronic Beryllium Disease Prevention Program,(40) which currently prescribes a limit of 0.2 μg per cubic meter, is under revision and may propose a limit closer to the new TLV.

Additionally, some local DOE sites, as well as some branches of the U.S. Armed Forces, have policies to follow the "latest" TLVs regardless of the year of promulgation. Irrespective of the purpose(s) for particular beryllium exposure assessment campaigns, the much lower levels of concern for beryllium, as opposed to most other metals or metalloids, magnifies the importance of wall deposits and the need to account for, or minimize, such deposits, or justify reasons for not doing so. More than one approach may apply depending on the purpose(s) for sampling, data quality objectives, and other factors. While additional studies are urgently needed to determine the best approach(es), some sites or agencies may see the need to take one or more actions before such studies can be completed.

WHAT IS THE SAMPLE?

As suggested by Harper and Demange,(3) the ultimate question that needs to be answered is, what is the sample that needs to be collected and analyzed? However, answers to a number of other questions feed into the answer to this ultimate one. We will address these other questions one at a time, although they are considerably intertwined. As a reminder, this is being addressed for beryllium specifically, but the general questions can be applied to other metal and metalloid particulate matter. The answers affect not only what sampling equipment to use, but also can affect the analytical method to be applied, and how well the data compare with historical data and results from other locations.

First, what health end point are we protecting against? The ACGIH Statement of Position, as posted on its web site, indicates that TLVs are intended to represent conditions under which nearly all workers can be exposed without experiencing adverse health effects.(41) In the case of beryllium, a certain percentage of exposed individuals develop a reaction, known as beryllium sensitization or BeS, and that a percentage of sensitized individuals later contract chronic beryllium disease or CBD.(42) The historical perspective, to which many still subscribe, has been to protect against the disease; however, in adopting the new TLV® for beryllium, the ACGIH TLV Committee has taken the position that protection against BeS is necessary, and in fact clearly so states in its Documentation of the TLV®(7) Although not stated, the implication is that any sensitized individual could potentially contract CBD, which has a long latency period. The validity of that premise, or other positions taken in the TLV, is not a subject of discussion for this article.

This leads to the second question: What air sampling fraction should be collected? The documentation of the new TLV calls for inhalable sampling, which is common in Europe but less common in the United States. Measurements made in the development of the TLV were taken using high-volume samplers assumed, but not proven, to collect at least some of the larger particles within the inhalable fraction.(2)

Inhalable sampling for beryllium is based on the premise that any particulate inhaled into the respiratory system may interact with immune-competent cells,(7) potentially leading to BeS. While particles below 10 μm will be deposited in the tracheobronchial or alveolar regions, particles above 10 μm are cleared (e.g., by the mucociliary escalator) and enter the gastrointestinal tract.(43) Less than 1% of beryllium is absorbed in the gastrointestinal tract,(6) with the balance being excreted. This would suggest a low (but non-zero) risk of BeS from larger particles, as suggested by Kent et al.(43) and Kolanz.(10)

Looking at the previous two questions together, the inhalable convention would seem to be appropriate if one wishes to protect against BeS by any and all possible pathways involving airborne particulate. However, it could be reasonably argued that the risk of BeS from gastrointestinal absorption is not great enough to justify the cost of switching from the CFC to an inhalable sampler. Additionally, an argument could be made that sampling according to the respirable convention, as defined by ISO 7708,(4) would provide a better indicator for CBD itself. Both conventions (inhalable and respirable) are somewhat different from what the CFC collects.

Now we move to the third question: Does the sample include only the filter catch, or does it also include wall deposits? Prior to the introduction of the CFC, when there were no cassette walls, the filter catch alone was typically considered to be "the sample." This continued even with the CFC initially, since the importance of larger particles was not generally recognized prior to the 1980s. As noted previously, the CFC loses efficiency above 10 μm, so if particulate up to 100 μm is important (as indicated by the ACGIH TLV), that would suggest including wall deposits. Remember that wall deposits can be an issue for the "inhalable" IOM sampler as well. If, however, one takes the position (in contrast to the TLV) that collecting particulate of 10 μm or less is sufficient for one's exposure assessment purposes, inclusion of wall deposits becomes less important.

OPTIONS AVAILABLE

As noted above, one option would be to continue to use the CFC and analyze only the filter catch for beryllium. This could be viable if beryllium wall deposits are found to be low enough to be essentially insignificant, either owing to a sufficiently low quantity or a determination that their particle size distribution poses an acceptably low risk (again, in contrast to the TLV). Currently, the available data are limited to four points, with a median of 12% and a maximum of 39% of total beryllium deposited.(3,35) Continuing use of the CFC without wall deposits would also be viable it were possible to establish a correlation such that the filter catch can be deemed representative of the total beryllium particulate.

However, current data with other metals do not suggest that a consistent correlation factor can be applied.(5) Additional data are needed to provide a better characterization of both the amount and particle size distribution of beryllium wall deposits, and to determine whether a correlation factor can be established.

A second option would be to use a different sampler so that the filter catch more closely matches the inhalable convention. We have previously mentioned the IOM, which was designed to match the inhalable convention, but was intended primarily for gravimetric measurements rather than chemical analyses, and also has a certain amount of wall deposits. The gravimetric method accounts for the wall deposits, but method validation to account for wall deposits has not been performed for the chemical analysis using these samplers. A sampler that does not have walls, such as the Button sampler, could be considered, but as noted previously, would need additional validation against the inhalable convention.

Yet another possibility would be a multi-stage sampler, such as the Respicon, which could account for all three of the ISO 7708 conventions (respirable, thoracic, and inhalable). There are additional samplers, which we have not mentioned, that could potentially be used.(25) None of these samplers is disposable or inexpensive; all would require re-use, which in turn would require cleaning after each use, adding to their cost. Also, for any of these samplers, laboratories accredited by the American Industrial Hygiene Association (AIHA) would need to validate their methods utilizing these samplers.

A third option would be to continue to use the CFC, but include the wall deposits in the analysis of the sample. There are several methodologies that could be employed. One method is to wipe the interior walls of the cassette and include these particulates with the sample. This is what OSHA is doing today(27,28) as well as DOE's Lawrence Livermore National Laboratory.(2) The OSHA method has the disadvantage of adding media to the sample, which requires the laboratory to use a greater amount of digestion solution, which can adversely impact laboratory reporting limits. Another method is to rinse the interior walls. OSHA believes that this is less effective than wiping and suggests that wiping should be performed in addition to rinsing.(44) Another method, used in France, involves in-cassette digestion.(45)

Regardless of the option selected, comparability of newer data to older data sets, based on filter catch only, will need to be considered. It can reasonably be expected that including wall deposits will result in higher beryllium values, and in the case of measurements for regulatory compliance, the prospect for an increased incidence of results above the OEL. Appropriate communication with affected stakeholders will help to successfully address these considerations.

CONCLUSION

The wall deposits issue can no longer be ignored. The data have established that particulates do adhere to the walls of the samplers, particularly the CFC. Furthermore, since ACGIH has in fact adopted a TLV that calls for sampling and analysis of the inhalable fraction, for beryllium, those agencies that use the CFC for the particulate analysis for beryllium will need to evaluate whether to follow the TLV and, if so, how they will do it. Although the issue is not limited to beryllium and will largely be addressed by others outside of the beryllium community, individuals in the beryllium community can have a say by understanding fully the particulars of the issue and by participating in standard bodies such as ASTM International and the International Organization for Standardization (ISO), that are addressing the issue.

Additional studies are urgently needed to fill in the information gaps which currently exist and make it difficult to determine the best path forward. We would suggest that NIOSH undertake these studies, addressing the following issues:

(1) Collection of data to better characterize beryllium wall deposits on the CFC and/or IOM samplers. This should include both quantities deposited on walls versus the filter catch, as well as particle size characteristics of the wall deposits. The latter would confirm the current belief that such deposits are primarily of larger (above 10 μm) particles.
(2) Comparison of samplers to determine the optimum sampler to use, considering both technical and cost factors, and potentially getting away from the wall deposits issue altogether.
(3) Comparison of analytical methods to include the entire sample with optimum reporting limits.

Recommendations from these studies should then be implemented by revisions to existing voluntary consensus standards (e.g., ASTM International, ISO).

Given that such studies will take appreciable time and funding, field and laboratory personnel will, in the meantime, need to evaluate the issues discussed in this article and select the option(s) that best fit their requirements. While costs and available resources are factors that must be considered, the ultimate goal should be protection of the worker.

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