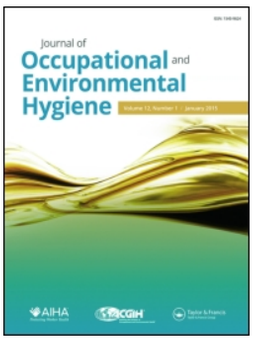

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Analytical Performance Criteria

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Analytical Performance Criteria Concerning Sampler Wall Deposits in the Chemical Analysis of Airborne Metals

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The findings and conclusions in this report are those of the author and do not necessarily represent the views of the National Institute for Occupational Safety and Health.

A question has arisen as to what constitutes the actual sample collected by an aerosol sampler. In the past, the sampler was considered to be simply a filter holder, with the sample being the material that is collected on the filter. More recent considerations of particle size-selective sampling suggest that the sample should include the entire aspiration of particles into the sampler no matter where they come to rest inside the sampler. The following is a discussion of the issues and the current state of the art.

DEVELOPMENT OF THE CLOSED-FACE CASSETTE SAMPLER

Various tools have been used for the measurement of aerosols, and it is sometimes possible to track down a specific instance where a transition can be observed from one instrument to another. For example, one can find the 1922 Bureau of Mines report[(1)] describing the initial experiments on the Greenburg-Smith impinger as an improvement over the sugar tube[(2)] used to that point. It has been more difficult to pinpoint the moment when the 37-mm, closed-face plastic cassette (CFC) with membrane filter replaced the impinger. In 1944, the Bureau of Mines published a report[(3)] comparing glass fiber filter concentrations with impinger results.

Outside the mines, the U.S. Public Health Service in Cincinnati, Ohio, the precursor to the National Institute for Occupational Safety and Health (NIOSH), published[(4)] in 1957 a recommendation for the use of mixed cellulose-ester (MCE) membrane filters based on their potential for various analytical finishes, including gravimetric, microscopic, and chemical analytical techniques. The paper suggests that their laboratory had been using these filters (in metal holders) since 1953.

The Millipore Company was founded in 1954 to manufacture membrane filters, and around 1956 a plastic cassette was developed to replace the stainless steel holder used for air sampling. This field monitor was designed to make the sampling easier and less expensive. One of its uses was sampling of liquids, such as water for coliform bacteria; for this application the monitor needed to be sterile. The 1960 (1st) edition of the American Conference of Governmental Industrial Hygienists (ACGIH®) handbook, *Air Sampling Instruments*,[(5)] includes a photograph of this "Millipore Monitor" and describes it as a new development (and also as disposable and sterile).

Another major application for the Millipore Monitor was in determining cleanroom requirements for the aerospace industry. It was specified under the more generic title of "air monitor" in U.S. Federal Standard 209,[(6)] which was first published in 1963 as *Cleanroom and Work Station Requirements, Controlled Environments*. This standard was revised several times over succeeding years before being superseded by ISO Standard 14644.[(7)] The original plastic material (Tenite) used for the manufacture of the monitor released toxic plasticizers that killed many organisms, making the method for coliform bacteria unreliable, and so it was replaced by polystyrene for water-based microbiological applications.

In 1969–1970, it was also discovered that the extracted plasticizers changed the weight of the membrane filter and chemically interfered with some airborne

contaminants of interest; thus, the polystyrene version was also recommended for air sampling. At some point in the early 1970s, Millipore began to use the designation "cassette" for the styrene version and "monitor" for the Tenite version (E. Smyrloglou, Smyrloglou Consulting, personal communication, January 2007).

Throughout the 1950s and 1960s, the most common methodology for determining aerosol concentrations remained the impinger. A paper published by Linch and Corn[8] of the University of Pittsburgh in 1965 evaluated the performance characteristics of a miniaturized impinger. As late as 1971, a subsequent paper from the same source,[9] responding to the criticism by Davies[10] that air samplers may not have efficient aspiration characteristics, evaluated several samplers including the micro-impinger, but the CFC was not among them (except possibly as the holder for the filter used in conjunction with the 10-mm cyclone). The CFC was certainly in some use for occupational measurements in 1970, as an assembly consisting of a 37-mm (closed-face) Millipore Monitor attached to the inlet of midget impinger was described for sampling inorganic lead particulate matter separate from tetraethyl lead vapor collected by reaction in the impinger fluid.[11] A Bureau of Mines paper from 1975[12] used the CFC as a basis for designing inlets of various dimensions for testing the theories of Davies. NIOSH Method S349 for total dust was validated and published in 1977[13,14] and, when the performance of the CFC was intensively investigated in 1980,[15] the text of that paper makes it fairly clear that the sampler was in common use by this time. A further evaluation covering the performance of the CFC in moving air was published in 1986.[16] This evaluation did not compare the CFC with any performance standard, as one did not exist at that time for coarse dust sampling.

THE CONCEPT OF THE INHALABLE SAMPLING CONVENTION

The International Organization for Standardization (ISO) Technical Committee 146, Sub-committee 2 (Workplace Air Quality), published a standard (ISO 7708[17]) on particle size-based collection efficiencies of samplers designed to match the probability of penetration to the lung. This was initially published as a Technical Report, where the largest particle convention, based on experimental data up to ~35 μm aerodynamic diameter (AED) and extrapolated to larger sizes, was termed "inspirable." This convention was redefined when further experimental data in the range 35 to 100 μm AED was obtained, and it is now termed the "inhalable" sampling convention. (The "inhalable" fraction defined for workplaces in the ISO standard is not the same as the inhalable coarse particle fraction defined by the U.S. Environmental Protection Agency.)

This convention was proposed initially for both still and moving air and for both oral and nasal breathing modes. However, experimental data obtained since that time has provoked recent discussion[18] concerning the nature of the convention with respect to calm air and also with respect to very large particles.

The ACGIH has accepted the ISO conventions and has proposed a large number of "inhalable" threshold limit values (TLVs). The ACGIH requires sampling using a sampler whose performance conforms to the ISO inhalable convention when assessing exposures to these substances.[19] The CFC has been tested against both the original inhalable convention in moving air[20] and one proposed modification for calm air.[21,22] In both cases, it matched the convention at the finer end of the range (< about 15 μm AED) well, but the sampling efficiency dropped significantly for larger particles. This performance was then confirmed in field trials for a range of different environments covering many different aerosols.[23]

Thus, the CFC has obtained a reputation as not being an inhalable sampler. In some ways this is lamentable; the CFC has advantages of cost, disposability, and ease of calibration over other, more expensive samplers. However, all experiments involving the CFC have operated under the assumption that the filter catch is representative of the aspiration into the cassette, i.e., deposition of particles on the internal surfaces of the cassette was ignored.

THE INSTITUTE OF OCCUPATIONAL MEDICINE (IOM) SAMPLER

A sampler designed at the U.K. Institute of Occupational Medicine (IOM) best met the ISO inhalable convention in European laboratory trials,[20] although this is hardly surprising as it was the only sampler tested that was actually designed with the convention in mind. It is of interest to note that internal wall deposits were recognized as potentially significant in this sampler, and it was recommended that their presence should be accounted for in the analysis.[24]

In an early study,[25] the amount of material found on the walls of the IOM sampler was 25–44% of the total sample for particles between 6 and 34 μm AED and a wind speed of 1 m/sec. In the European study,[20] however, smaller losses were found, and losses were also found to be particle size dependent, increasing to 25% at 100 μm. A more recent study[26] gave a loss range of 17 to 28% between 10 and 100 μm AED, also noting some particle dependence. Finally, Witschger et al.[27] also showed a particle size dependence of wall deposits, from around 20% at 6.9 μm AED to 55% at 76 μm AED.

In field studies, Lidén et al.[28] found wall losses 24–37% over a range of industries where the aerosols were organic, while Demange et al.[29] found less in industries where metalliferous dusts were encountered. Samples from a lead battery producer in Norway[30] also showed less of the total deposit on the walls of the IOM (1–33%, median 8%). Nevertheless, the amount of the total sample that could be anticipated to be found on the walls of IOM field samples is likely to be significant in all industries. The main mechanisms by which particles aspirated into the filter housing may be lost between the entry orifice and the filter is (*a*) electrostatic attraction, which affects

mainly small particles; and (*b*) gravitational settling or inertial impaction, which mainly affect large particles.[31]

Where aerosols are coarse and the metric of interest is mass, the cube relationship between diameter and volume ensures that the large particle losses dominate. Assuming these particles are of biological significance and are sampled in accordance with the inhalable convention, they should be included as part of the exposure measurement strategy.

WALL DEPOSITS IN THE CLOSED-FACE CASSETTE SAMPLER

The two U.S. government agencies concerned with occupational aerosol exposure assessment are NIOSH and the Occupational Safety and Health Administration (OSHA). Even though NIOSH has accepted the ISO size-selective conventions, it has only one method for a specific substance (formaldehyde on wood dust) specifying sampling in accordance with the inhalable convention. It is the CFC that is specified in all other NIOSH and OSHA methods, and thus it could be argued that these methods do not meet the inhalable convention.

However, again, wall deposits in the CFC were not evaluated in any laboratory or field comparison of the CFC against the inhalable convention. In the *NIOSH Manual of Analytical Methods (NMAM)*,[32] neither the Particles Not Otherwise Regulated (PNOR) method (0500) nor any substance-specific method using the CFC calls for an appraisal of these internal wall deposits, although the concept is discussed in the so-called "blue pages" (Chapter O, part 7) in the preamble to the manual. In the *OSHA Sampling and Analysis Methods* manual,[33] the gravimetric procedure (Method PV2121) calls for a filter as part of an internal capsule, weighed in its entirety, so that the wall deposits are included. Some OSHA methods that involve chemical analyses have a procedure for including the wall deposits, while others do not.

It is unclear exactly when the OSHA Salt Lake Technical Center (SLTC) began to require analysis of wall deposits along with the filter. The OSHA general methods for metals (ID121, ID125G) have been revised relatively recently and include a statement that the internal surfaces of cassettes must always be rinsed and wiped, and the rinse solution and filter swab must be added to the filter for digestion. It has been shown that rinsing alone is unable to remove all material collected on plastic cassette walls,[34,35] hence the recommendation of OSHA for mechanical wiping with a wetted fabric. In an OSHA method for hexavalent chromium (ID103), last updated in 1990, there is no reference to this procedure.

In a method for inorganic arsenic (ID105), last updated in 1991, there is a recommendation for following this procedure *if necessary*. It appears that the driving force behind the recommendation was a concern by the laboratory over filters so heavily overloaded that loose dust was found inside cassettes, rather than a specific concern for materials deposited on the walls during sampling. A method for metals from solder operations (ID206) of the same vintage does not contain the recommendation, possibly because overloaded filters were never observed in this situation.

As a result of their work to characterize the extent of wall deposits in general metals samples,[35] and based on their view that the appropriate air sample should include all particles aspirated into the sampler, the OSHA SLTC laboratory now routinely rinses and wipes the interiors of all cassette samples for chemical analysis of metals, as noted in the new method recently completed for hexavalent chromium, Method ID215 version 2, paragraph 1.1.1.[33] In this method, it is recommended to wipe with the rough side of a polyvinyl chloride (PVC) filter, wetted with a buffer solution specific to the method (results were similar for deionized water, but using the buffer solution avoids interferences). A study reported in the method (paragraph 4.8.6) of the removal efficiency of this procedure gave a mean of 97.5% recovery for spiked soluble potassium dichromate and 92.8% for spiked insoluble lead chromate. A study of actual compliance samples received by the SLTC laboratory showed wall deposits of up to 13% for samples from spray painting, up to 20% for samples from chromium plating, and up to 123% for samples of welding fume (wall deposits as a percentage of filter deposit).

In France, filter cassettes were already in use in 1985,[36] and the analysis of wall deposits, by solubilization of the samples inside the cassettes, dates from 1986. The main reason for doing this was to avoid contamination during handling of the samples and to avoid losses due to transportation of the samples before analysis. However, Annex II of the European Lead Directive, published in 1982[37] (withdrawn in 1998), which required both a specific speed of the air at the entrance of the sampler (1.25 m/s $\pm$ 10%) and a specific orifice (intake orifice diameter of at least 4 mm diameter to avoid wall effects), was interpreted by the Institut National de Recherche et Sécurité (INRS) to mean that all particles entering the cassette were to be regarded as part of the sample.

A French Standard for Lead published in 1988[38] (but since withdrawn) contains an informative annex recommending this method of *in situ* digestion. In 1990,[39] the results of separate analyses of filters and wall deposits were published, showing the importance of wall deposits. In 1991, additional experiments (reported in 2002)[29] showed that these deposits were not only due to losses in transportation. The official method of the INRS (MetroPol), first published in 2000, includes sample digestion within the cassette,[40] and this has also been included in the 2002 revision of the French standard for metals and metalloids analysis.[41]

EVALUATING WALL DEPOSITS IN THE IOM SAMPLER

Reference can be made to methods published by the U.K. Health & Safety Executive[42] (Methods for the Determination of Hazardous Substances, or MDHS) for guidance with regard to the assessment of wall deposits in the IOM sampler. For gravimetric analysis (MDHS14/3) it is simple enough to weigh the entire IOM capsule, and that is the recommendation.

However, the IOM sampler is also recommended in the general method for metals (MDHS 99), but there is no discussion in this method about removing wall deposits for analysis by the chemical procedure. (Note: ISO Standard 15202-2 [2001] Workplace air—Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry—Part 2: Sample preparation that might be assumed to supersede the MDHS methods, suggests rinsing the interiors of cassettes with dilute nitric acid if necessary but not wiping.)[43] Indeed, MDHS 91, which uses X-ray fluorescence analysis of filters (and also recommends using an inhalable sampler such as the IOM), cannot account for these deposits at all. Thus, there is evidently a need for more consistent recommendations regarding the analysis of wall deposits in the IOM sampler as well as in the CFC.

COMPARING IOM AND CFC SAMPLES IN METALS INDUSTRIES

In a French study in various metals industries,[29] it was found that by adding wall deposits of the CFC to the filter analysis, results much closer to those of the IOM (filter + wall deposits) could be obtained in samples collected side-by-side. Figure 1 shows results from workplace evaluations in France where the CFC was compared vs. the IOM while accounting for wall deposits; in four different industries at 43 locations, duplicate pairs of CFC and IOM samples were collected side-by-side. For the traditional evaluation of CFC (filter only) to IOM (filter + wall deposits), the slope is 0.59 ($r^2 = 0.73$). However, the data shown are for CFC (+ wall deposits) vs. IOM (filter + wall deposits) where the slope is 0.79 ($r^2 = 0.88$), much closer to 1.

In a NIOSH study of a lead ore processing mill,[44] we found that wall deposits of lead were an average of 19% (maximum 35%, n = 28) of the total sample in CFC samplers, and 17% (max. 30%, n = 22) in the total sample of IOM samplers. We are also able to report here for the first time that in a similar study of samplers in a copper smelter, we found an average of 25% (maximum 55%, n = 17) of the total copper sample on the walls of CFC samplers, and 19% (max. 38%, n = 18) of the total copper sample on the walls of IOM samplers. Figure 2, first published in 2006,[45] contains data from a bronze foundry, a solder manufacturer, a lead-acid battery recycler, a nickel-chromium-cadmium recycler, and lead ore processing mill; this graph has been updated with the side-by-side results for lead in this copper smelter (lead is a contaminant of the ore).

In Figure 2, 51 pairs of side-by-side CFC and IOM personal and area samples, with a concentration range extending from approximately 10 to 5000 $\mu g/m^3$ are shown by analysis of their filters only. There is a good correlation ($r^2 = 0.93$) with almost 1:1 correspondence. In both the INRS and NIOSH studies of metalworking industries, the collection efficiencies of the IOM and CFC samplers are very similar when both the collected material on the walls and the filters is taken into account. The most likely explanation for these observations is that very little of the aerosol in these environments is in the extra-thoracic fraction, i.e., larger than about 25 μm AED. An increase in the CFC collection of about 25% in the range 15–25 μm AED would provide a result similar to the inhalable convention and, thus, to the IOM sampler. The particle size distributions in these workplaces are not known, but the processes included many likely to produce a coarse aerosol, including ore crushing, battery breaking, chipping

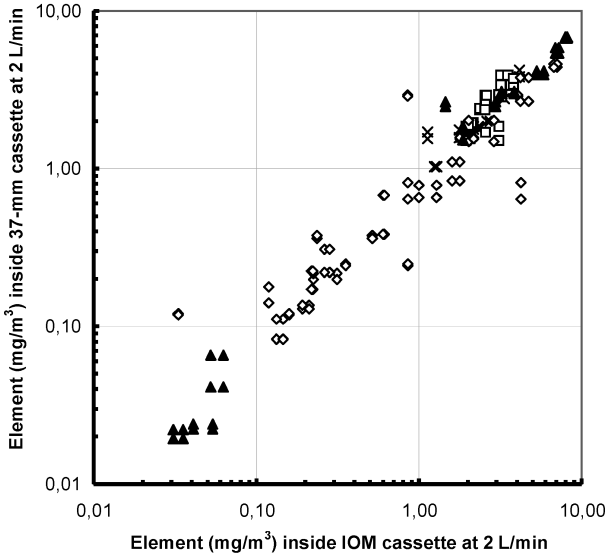

FIGURE 1. Logarithmic comparison of CFC vs. IOM results (filter + wall deposits) for metals analysis from field samples collected side-by-side. ×: Fe—cast iron machining and welding, □: Fe—grey cast iron machining, ◇: Pb—lead and zinc refinery, ▲: Ni—nickel refinery (n = 172, y = 0.79x, $r^2 = 0.88$).

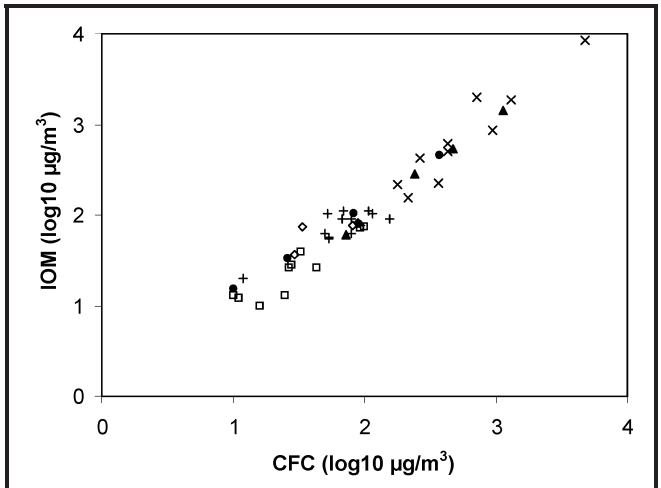

FIGURE 2. Comparison of filter-only analyses for lead (Pb) from side-by-side IOM and CFC samplers (n = 51; y = 1.02x, r^2 = 0.93). x: lead-acid battery recycler, ◇: bronze foundry, ▲: lead ore-processing mill, +: copper ore smelter, □: nickel/chromium recycler, ●: solder manufacturer.

molds away from casts, and powder handling. Examination of the aerosols in the French studies[46] indicated a mass median AED $<20\ \mu$m but with very wide geometric standard deviations (up to 7). Therefore, further studies comparing in more detail the deposits on the walls and filters of these samplers are needed to confirm this hypothesis.

There is a point of difference between the two studies, and that is in the proportion of collected materials on the walls of the IOM samplers vs. filters in the NIOSH studies compared with the INRS study. The NIOSH study showed a higher proportion of collected material on the walls of the IOM cassettes than did the INRS study. This resulted in a ratio of CFC (filter only) to IOM (filter only) of 0.64 for the INRS results compared with 1.02 for the NIOSH results. This might be due to differences in the capsules; the INRS used the older, plastic style, whereas the more recent NIOSH studies have mainly used stainless steel capsules with the longer "throats" for "multidust" (use of foam inserts for other size-selective fractions) sampling. It is not known, however, whether these differences actually influenced the performance of the samplers.

DISCUSSION AND CONCLUSIONS

The issue of sampler wall deposits has evolved from ignorance, through realization of their importance, to a need for inclusion. However, many published methods have not kept up with this trend, and so guidance in the methods manuals is inconsistent. It has now been shown in a number of studies in metals industries that filter and wall deposit masses are comparable for the CFC and IOM samplers. Thus, if the total aspiration (filter + wall deposits) of the IOM sampler is considered a sample meeting the ISO inhalable size-selection criterion, then so too could the total aspiration (filter + wall deposits) of the CFC.

Including the wall deposits with the filter is the current procedure employed by OSHA, and thus the samples may be assumed to conform to the ISO standard. (Note that this conclusion is specific to the industries studied and may not be applicable to other industries; for example, where organic dusts such as wood and flour are encountered.)

The pharmaceutical industry moved to standardize on internal capsules to include wall deposits for their in-house methods through in-situ desorption 15 years ago;[47,48] however, solubilization of metals samples within cassettes requires well-sealed cassettes for safety reasons, and digestion by means other than heat. The French method uses ultrasonic digestion directly within the cassettes.[40]

As a caveat to the conclusion above: while it has generally been assumed that the IOM sampler meets the inhalable criterion when the wall deposits are included, for the major European study, this conclusion was based on averaging data from tests over several wind speeds;[16] the report also concludes that analysis of the filter deposit only may better meet the current inhalability criterion for low wind-speeds (0.5 m/s). In an investigation of the IOM sampler for large particles in very low wind speeds (<0.3 m/s), it was also found that the filter-only catch better matched one of the proposed conventions for inhalability under near calm air conditions than did the filter plus wall deposits.[27] If these conclusions are correct, then it is possible that wall deposits should not be included in the analysis, which would allow the use of on-filter analytical techniques such as XRF. Hopefully, this issue will be explored in more detail in future research studies.

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