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## Closed-Face Filter Cassette (CFC) Sampling—Guidance on Procedures for Inclusion of Material Adhering to Internal Sampler Surfaces

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The sampling and analytical methods published in the *NIOSH Manual of Analytical Methods* (NMAM)<sup>(1)</sup> represent state-of-the-art methods for assessing worker exposures to toxic chemicals. Aerosol sampling methods generally specify the collection of workplace air samples using samplers containing filters such as 37-mm closed-face cassettes (CFCs). NIOSH considers that all particles entering CFCs, as well as some other samplers, such as the Institute of Occupational Medicine (IOM) sampler, should be included as part of the sample whether they deposit on the filter or on the inside surfaces of the sampler. This matter has been discussed in detail in the section on “Sampler Wall Losses” of NMAM Chapter O<sup>(2)</sup> and has also been specifically addressed in a gravimetric method promulgated by the Occupational Safety and Health Administration (OSHA).<sup>(3)</sup> The most accurate measure of occupational exposures therefore includes all aerosol particles entering workplace air samplers, both for gravimetric analysis and for analytes such as metals and metalloids. Hence, following sampling, i.e., during sample preparation and analysis, procedures should be used to account for material adhering to the internal surfaces of air sampling cassettes.

Research studies have shown that material deposited on the inside surfaces of air sampling cassettes often constitutes a significant fraction of the aerosol that enters the sampler.<sup>(4–7)</sup> Example data for CFCs are presented in Table I,<sup>(8)</sup> which demonstrate that, in some instances, the wall deposit can even exceed the amount of material collected on the filter. There are several mechanisms that can lead to particles being deposited on internal surfaces rather than on the filter, including direct impaction, gravitational settling, interception during transport by eddies within the sampler, bounce from the filter, and electrostatic attraction. Losses of collected particulate matter from the filter can also occur during sample shipment and handling. It is likely that multiple mechanisms are in play simultaneously, with varying degrees of importance depending on factors such as particle density, inertial velocity, wind

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speed and orientation, and so on. However, it is unlikely that these mechanisms would interfere with particles entering human airways during breathing.

Recent laboratory and field investigations on airborne particles of up to 20  $\mu\text{m}$  aerodynamic equivalent diameter containing lead (Pb) have shown that there is no qualitative difference in the size distribution of aerosols deposited on the filter or on the internal surfaces of CFCs.<sup>(9,10)</sup> Both components therefore would contribute to the inhaled dose that is delivered to the airways of exposed workers. This is a concern not only for sampling and analysis of lead and other elements but can apply to aerosol sampling in general, and to certain targeted aerosol fractions (i.e., inhalable, respirable, or thoracic).<sup>(11,12)</sup> Sampling cassettes composed of conductive materials do minimize the internal deposits that occur through static attraction but will not eliminate internal deposits entirely.<sup>(2)</sup> Losses of collected particulate matter from the filter during sample transport and handling are also possible, regardless of the composition of the sampler.

Consideration of deposits adhering to internal sampler walls is included in related international voluntary consensus standards published by ASTM International (formerly, American Society for Testing and Materials) and the International Organization for Standardization (ISO), which describe the sampling and analysis of airborne metals and metalloids in occupational atmospheres.<sup>(13-17)</sup> The recommendation to account for sampler wall deposits is consistent with the position of OSHA, whose sampling and analytical methods for metals and metalloids specify transferring loose dust, washing internal surfaces, and wiping internal cassette walls.<sup>(18,19)</sup>

Depending on the analyte(s), suitable techniques for accounting for internal sampler wall deposits include: (1) wiping (with inclusion of the wipe along with the filter during subsequent sample preparation);<sup>(18,19)</sup> (2) carrying out sample extraction directly within the cassette;<sup>(20-22)</sup> (3) thorough washing of internal cassette surfaces;<sup>(21)</sup> and (4) the use of internal sampling capsules or cartridges (sampler inserts).<sup>(3,23)</sup> These techniques are consistent with NIOSH methodologies that, as noted, should include not just the filter catch but all material that enters the sampler. In some cases an exception might be made where sampling cassettes composed of conductive materials are employed and internal wall deposits are thereby minimized.<sup>(2)</sup>

NIOSH methods that are presently affected by the recommendation to account for cassette wall deposits are listed in Table II. Specific guidance on techniques to include internal wall deposits is provided below.

Three NIOSH CFC methods entailing gravimetric analysis are listed in Table IIa. When CFC sampling is carried out, gravimetric analysis procedures for aerosol measurement should employ internal capsules<sup>(24)</sup> such as the polyvinyl chloride (PVC) Accu-Cap (SKC Inc., Eighty Four, Pa.), the Woodcheck (MSA, Pittsburgh, Pa.), or equivalent. Weighing only the filter is generally insufficient, since this practice will not account for material collected on internal sampler walls and on the interior of the cap. (While, in principle, each CFC sampler could be weighed in its entirety, contamination of outside cassette surfaces would contribute greatly to sampling bias, especially in dusty work environments. There is

also a loss in precision caused by the much greater absolute mass.) For respirable sampling and gravimetric analysis where cyclone/cassette assemblies are used, internal non-filter deposits are still noted,<sup>(7)</sup> but it is more difficult to account for them; thus, they should be minimized where possible through the use of cassettes composed of conductive materials, with collection of the aerosol deposits onto pre-weighed filters.<sup>(2)</sup>

NIOSH CFC sampling and analytical methods for which inclusion of internal non-filter deposits is already described in each method, at least to some extent, are listed in Table IIb. These methods, which entail rinsing, prescribe rinsing if there is visible deposit on internal cassette surfaces (and it is expected that the target analyte is soluble in the solution used for rinsing). The rinsate is then included along with the filter in subsequent sample extraction steps prior to analytical measurement. In fact, rinsing should take place even if there is no visible deposit on the internal walls or inside the cap. Although often invisible to the naked eye, internal non-filter deposits of aerosol particles can contribute significantly to the total amount of aerosol entering the sampler. Another technique that merits consideration is within-cassette extraction, which could prove to be effective for some or all of the methods listed in Table IIb.

Table IIc lists elemental analysis methods, in which there are options for inclusion of CFC internal non-filter deposits. The use of a digestible capsule that is fused to the filter (e.g., an internal cartridge composed of cellulose acetate attached to a mixed-cellulose ester (MCE) filter) is especially appealing as this obviates any need for a separate wiping or rinsing step. After sampling, the capsule can be simply removed from the cassette and placed into a sample preparation vessel for dissolution in its entirety. (Since many digestion vessels do not easily accommodate a 37-mm diameter internal capsule, the fabrication of a 25-mm diameter version is a worthwhile goal.) Cassette wiping, where particles are dislodged using manual pressure through a wetted fabric or similar material, is a practice that OSHA uses for elemental analysis, and the effectiveness of this technique has been demonstrated.<sup>(18)</sup> The wipe is added to the filter used for sampling, and both are digested together prior to subsequent instrumental analysis. Alternative techniques such as cassette rinsing with dilute acid solution could be considered, whereby the rinsate from the cassette walls is added to the sample preparation vessel containing the filter used for sampling. These practices may be acceptable, but their effectiveness must be validated through demonstrated quantitative recoveries. In cases where extraction and not digestion is called for, it is possible to carry out the extraction directly within the cassette, which automatically incorporates non-filter deposits into the dissolution medium.<sup>(21,22,25)</sup>

Other methods for which internal non-filter deposits should be included but do not fit into the above three categories (i.e., for methods listed in Table IIa–c) are listed in Table II d. For hexavalent chromium (Cr[VI]) methods, cassette wiping with a non-reactive medium such as PVC can be used. (With Cr[VI]-containing samples, cellulosic wiping materials should not be used since these media can cause reduction of Cr[VI].) This wipe, which may be a filter wetted with extraction solution, is then added to the filter used for sampling prior to further analytical processing. As an alternative, Cr[VI] can be extracted directly within the cassette using the extraction solution (basic buffer),<sup>(21)</sup> and aliquots can then be analyzed. Still another option could entail rinsing of Cr[VI] wall deposits with basic buffer solution.

Rinsing with dilute acid solution should be used for removal of alkaline dusts from internal cassette surfaces; alternatively, a wiping procedure may be employed.

In the future, additional NIOSH methods may require incorporation of internal sampler wall deposits; a number of these methods are listed in Table III. Effective procedures and performance data are needed for all of the listed methods; thus, for the time being, the general recommendation to account for internal sampler wall deposits does not apply to these methods.

For most of the aerosol sampling methods discussed here, alternatives to the CFC samplers are available, and some of these have been optimized or found by experiment to collect the ISO-defined “inhalable fraction.” Several such samplers are available commercially.<sup>(2,26)</sup> Deposits also occur on internal surfaces of these samplers (e.g., within the cones of the IOM and Gestamtstaubprobenahme (GSP) samplers, and on the Oring of the Button sampler), and the user should be aware of and, if necessary, account for them.

In summary, owing to the significance of internal non-filter sampler deposits, it is generally recommended to account for this contribution to worker exposures to airborne chemical agents. This recommendation may apply to methods that collect and analyze both aerosols and vapor where, for example, the sampler consists of a filter cassette and sorbent tube in series; in such cases, inclusion of the particulate material adhering to the filter cassette internal surfaces should be considered. Inclusion of internal non-filter sampler deposits may not pertain to methods that entail the use of impregnated filters (e.g., isocyanates). Other NIOSH aerosol sampling methods not addressed here (Table III) may deserve consideration of internal wall deposits in the future. It is intended to revise the texts of affected NIOSH methods to specifically account for internal wall deposits. However, until revised methods are approved and published, the recommendations given here shall apply.

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**TABLE I**

Closed-Face Filter Cassette Internal Wall Deposits from Samples Obtained in Various Workplace Environments

Work Environment/Activity	n	Chemical Agent(s)	Maximum Wall Deposit(s) (%)	Median Wall Deposit(s) (%)
Copper smelter	17	Pb	55	21
Lead ore mill	28	Pb	35	19
Solder manufacture	30	Pb	74	29
Battery production	16	Pb	66	28
Welding	10	Cr[VI]	55	5
Electroplating	12	Cr[VI]	17	12
Paint spray	29	Cr[VI]	12	7
Foundry	9	Zn	62	53
Zinc plating	18	Zn	91	27
Cast iron foundry	18	Fe	46	22
Grey iron foundry	18	Fe	77	24
Bronze foundry	6	Cu, Pb, Zn	45, 17, 21	19, 13, 15
Cuproberyllium foundry	4	Cu, Be	40, 39	31, 12

TABLE II

NIOSH Methods Affected by the Recommendation to Account for Cassette Internal Non-Filter Deposits, and Suitable Techniques for Inclusion of All Aerosol Entering the Sampler

NIOSH Methods	Technique Recommended	Comments
<b>(a) Gravimetric Analysis Methods</b>		
0500 - Particles (“total”)	Use internal capsule for sample collection, which is analytically weighed pre- and post-sampling	Suitable examples are capsules composed of PVC attached to PVC filters
5000 - Carbon black		
0600 - Particles (respirable)	Use filter housed in static-dissipative (“conductive”) cassette, or use internal capsule	Conductive or static-dissipative sampler material can minimize internal wall deposits
<b>(b) Internal Deposits Currently Considered in Method Text</b>		
5005 – Thiram	Rinse internal wall deposits with extraction solution (e.g., water, eluent, organic solvent)	Rinsate is added to sample preparation vessel (e.g., beaker) along with the filter used for sampling
5011 - Ethylene thiourea		
5030 - Cyanuric acid		
5032 - Pentamidine isethionate		
5700 - Formaldehyde (textile or wood dust)	Use IOM inhalable sampler	Alternative procedure could entail CFC sampling, followed by rinsing as above
<b>(c) Elemental Analysis Methods</b>		
7013 - Aluminum	<b>1</b> Remove internal wall deposits with wetted wipe (e.g., filter or wipe material); wipe is then added to sample preparation vessel (e.g., beaker) along with the filter used for sampling	Alternative procedure could entail rinsing of internal sampling cassette surfaces with dilute acid solution (demonstrate effectiveness)
7024 - Chromium		
7027 - Cobalt	<b>2</b> Use digestible internal capsule (e.g., cellulose acetate attached to MCE filter) for sample collection; capsule is digested and analyzed in its entirety	
7029 - Copper		
7030 - Zinc		
7048 - Cadmium		
7046 - Barium		
7074 - Tungsten		
7082 and 7105 - Lead		
7102 - Beryllium		
7300, 7301, and 7303 - Elements		
7900 - Arsenic		
7020 - Calcium	Remove internal wall deposits with wetted filter or wipe, which is then added to sample preparation vessel (e.g., centrifuge tube) along with the filter used for sampling	Alternative procedures involve within-cassette extraction
7701 - Lead		
7704 - Beryllium		
<b>(d) Hexavalent Chromium and Alkaline Dusts</b>		
7600, 7605, and 7703 - Hexavalent chromium	Remove internal wall deposits with wetted filter, which is then added to sample preparation vessel (e.g., beaker) along with the filter used for sampling	Alternative procedures could involve rinsing or within-cassette extraction
7401 - Alkaline dusts		

TABLE III

## Other NIOSH Methods Potentially Affected by the Recommendation to Account for Internal Sampler Wall Deposits

NIOSH Methods and Analytes
5001 - 2,4-D and 2,4,5-T ((2,4-dichlorophenoxy)acetic acid and 2,4,5-trichlorophenoxy)acetic acid)
5002 - Warfarin
5003 - Paraquat
5004 - Hydroquinone
5006 - Carbaryl
5007 - Rotenone
5008 - Pyrethrum
5009 - Benzoyl peroxide
5010 - Bromoxynil and Bromoxynil octanoate
5012 - EPN (Ethyl p-nitrophenyl thionobenzenephosphonate)
5013 - Benzidine, o-Tolidine, and o-Dianisidine dyes
5014 - Chlorinated terphenyl
5016 - Strychnine
5017 - Dibutyl phosphate
5018 - 2,4,7-Trinitrofluoren-9-one
5019 - Azaleic acid
5020 - Dibutyl phthalate and Di(2-ethylhexyl) phthalate
5021 - o-Terphenyl
5022 - Organo-arsenic
5023 - Coal tar pitch volatiles
5025 - Chlorinated diphenyl oxide
5026 - Oil mist
5027 - Ribavirin
5031 - Aspartame
5033 - p-Nitroaniline
5034 - Tributyl phosphate
5035 - Super-absorbent polymers
5036 - Trimetallic anhydride
5037 - Triorthocresyl phosphate
5038 - Triphenyl phosphate
5039 - Chlorinated camphene
5041 - Capsaicin and Dihydrocapsaicin
5042 - Asphalt fume (benzene soluble and total particulate)
5043 - p-Toluenesulfonic acid
5044 - Estrogenic compounds
5516 - 2,4 and 2,6 Toluenediamine
5521, 5522, 5525 - Isocyanates
5524 - Metalworking fluids

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**NIOSH Methods and Analytes**

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5527 - Triphenyltin chloride

7500, 7501, 7601, 7602, and 7603 - Silica

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