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## Standard Guide for Assessment and Inclusion of Wall Deposits in the Analysis of Single-Stage Samplers for Airborne Particulate Matter<sup>1</sup>

This standard is issued under the fixed designation D8358; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 Many methods for sampling airborne particulate matter entail aerosol collection on a substrate (typically a filter) housed within a container (or holder), the whole apparatus being referred to as an aerosol sampler. In operation, the sampler allows a vacuum (pressure below ambient or room air pressure) to be applied to the rear of the substrate so that sampled air will pass through the substrate, leaving collected particles on the substrate for subsequent analysis. The sampler may also protect the substrate, while the opening (orifice) of the container may further play some role in determining what size range(s) of particles approach the collection substrate (size-selective sampling).

1.2 All particles entering the container orifice are considered part of the sample, unless stated otherwise in the method, but not all particles are necessarily found on the substrate after sampling (1).<sup>2</sup> Particles may be deposited on the inner walls of the sampler during sampling or may be deposited on the inside walls of the sampler or on the orifice plug or cap following transportation (2). These particles are often loosely referred to as wall deposits. This guide presents background on the importance of these wall deposits and offers procedures by which these deposits can be assessed and included in the sample.

1.3 Wall deposits may also occur in multi-stage samplers (for example, cascade impactors), but this guide does not cover such samplers.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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<sup>2</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>3</sup>

- D1356 Terminology Relating to Sampling and Analysis of Atmospheres
- D4185 Test Method for Measurement of Metals in Workplace Atmospheres by Flame Atomic Absorption Spectrophotometry
- D4532 Test Method for Respirable Dust in Workplace Atmospheres Using Cyclone Samplers
- D6061 Practice for Evaluating the Performance of Respirable Aerosol Samplers
- D6062 Guide for Personal Samplers of Health-Related Aerosol Fractions
- D6552 Practice for Controlling and Characterizing Errors in Weighing Collected Aerosols
- D6785 Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry
- D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D7439 Test Method for Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma–Mass Spectrometry
- D7948 Test Method for Measurement of Respirable Crystalline Silica in Workplace Air by Infrared Spectrometry

NOTE 1—Other standards under the jurisdiction of ASTM Committee D22 on Air Quality, including standards that are not the direct responsibility of Subcommittee D22.04 on Workplace Air Quality, may also be affected by this guide and should be considered on a case-by-case basis.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## 2.2 Other Standards:

**ISO 7708 Air quality — Particle size fraction definitions for health-related sampling**<sup>4</sup>

**EN 13205 Workplace exposure — Assessment of sampler performance for measurement of airborne particle concentrations (in four parts)**<sup>5</sup>

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this standard, refer to Terminology **D1356**.

3.1.1 *wall deposit, n*—particles which have entered the container (holder) of an air sampler but which are found elsewhere than on or in the principal particle collection medium (for example, a filter), including on any internal surfaces of the holder or container, such as O-rings and inlet plugs or covers.

## 4. Summary of Guide

4.1 Aerosol samplers that are used to collect “total particulate matter” (TPM), as dictated by the aspiration efficiency of the sampler, and samplers that are used to collect “inhalable particulate matter” (IPM), as dictated by the ISO 7708 inhalable particle convention, consist of filters housed in containers (holders). The design of the holder, normally referred to as a sampler, may be optimized so that aspiration follows the ISO 7708 inhalable particle convention. All particles passing through the orifice of these samplers, particularly where the orifice is designed for a specific sampling performance, typically are considered as part of the sample, unless otherwise dictated by the method.

4.2 Samplers that are used to collect the “thoracic” particulate fraction, as defined by ISO 7708, or the “respirable” particulate fraction, as defined by ISO 7708, have a pre-selector to provide a defined size selection. These samplers may also exhibit wall deposits that could require inclusion for subsequent analysis.

4.3 This guide details procedures that have been used to ensure that all collected particles are assessed in the report of mass collected, or airborne concentrations calculated from the collected mass.

4.4 This guide focuses mainly on samples taken for two types of analysis: gravimetric analysis for Particles Not Otherwise Regulated/Specified/Classified (PNOR/S/C), and acid extraction/digestion for metals analysis. However, the principles of the guide are applicable to samples collected for other analytical procedures, such as inorganic acid mists and organic aerosols.

4.5 Three main methods and some variants for assessing wall deposits are discussed in this guide. The selection of methods is dictated by the ability of the method to meet data quality objectives. Recovery must be validated as being suffi-

ciently efficient and reproducible to meet the expanded uncertainty requirements of the overall sampling and analytical method. In the absence of a published standard method it is the responsibility of those collecting samples and the analytical laboratory to review the validation data and assess the fitness-for-purpose of the procedure.

## 5. Significance and Use

5.1 The following is a non-exclusive list of standards to which this guide applies: Guide **D6062**; Test Methods **D4185**, **D4532**, **D6785**, **D7035**, **D7439**, **D7948**; and Practices **D6061** and **D6552**.

5.2 The applicability of this guide to other standards under the jurisdiction of ASTM Committee D22, but not the direct responsibility of Subcommittee D22.04, should be considered where analyte entry into the sampler is considered the sample and where analyte adherence to internal sampler surfaces (“walls”) is likely to scavenge analyte from the collection substrate.

5.3 Aerosol samplers typically consist of a filter or other collection substrate, for example an impaction plate or foam, supported in a container or holder. The entire device typically is considered an aerosol sampler. The sampling efficiency of the aerosol sampler, that is, the ratio of the concentration collected by the collection substrate to the undisturbed concentration in the air, has three components: (1) aspiration (or entry) efficiency; (2) transport efficiency (depending on design, both from entry “plane” to internal separator and from any internal separator to collection substrate); and (3) penetration (through the internal separator). For a sampler of a specific design, the three efficiency components are functions of particle (aerodynamic) size and flow rate. The aspiration efficiency also depends on wind speed and direction, while the sampler’s angle to the vertical influences both the aspiration efficiency and the transport efficiency. Ideally, when a sampler is designed and tested for its sampling performance, or both, it should first be established what is considered as the collected sample (that is, the deposit on the collection substrate, but also any deposits on any internal surfaces if these are to be analysed).

5.4 Part of the aerosol entering a sampler will deposit on the internal surfaces of the sampler prior to reaching the collection substrate. There are number of mechanisms by which this can occur, including bounce from the filter, inertial impaction, gravitational settling and electrostatic attraction after entry. In addition, after sample collection, if the collection substrate is transported while mounted in the sampler, it is possible that particles originally deposited on the collection substrate may dislodge during transportation. Such particles can thereby contribute to deposits on the walls, as well as on the base of any cover plate or plug. All particles found elsewhere than on or in the collection substrate are often loosely termed “wall deposits.” If the sample of interest entails the entire aspirated air particulate into the container or holder (sampler), it is necessary to account for these wall deposits, especially if it cannot be shown that they should be disregarded.

5.5 The research underpinning the information in this guide has arisen partly from studies of inert particles (**3**, **4**), but

<sup>4</sup> Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <https://www.iso.org>.

<sup>5</sup> Available from British Standards Institution (BSI), 389 Chiswick High Rd., London W4 4AL, U.K., <http://www.bsigroup.com>.

mostly from investigations of methods for the determination of airborne metalliferous particulates (2, 5-15). However, the issues at hand are also important in sampling airborne organic materials, including bacterial endotoxin (16), wood (17), and pharmaceutical dusts (18); another relevant study reported results from investigations in thermosetting plastics, wood, paper, and animal breeding (19). Except in the case of very large wood dust particles, there is no evidence to suggest that wall deposited particles are sufficiently different from those found on the collection substrate to warrant their exclusion (13, 14). Wall deposits are not limited to aerosol samplers for larger airborne particles but may also be found in samplers for finer particles (20, 21). There may be a justification for excluding wall deposits where the performance of an aerosol sampler tested to EN 13205 shows appropriate compliance with the relevant ISO 7708 size-selective convention without their inclusion.

5.6 The findings of studies that have been carried out to assess wall deposits in two commonly used samplers are summarized in Table 1 and Table 2. A commonly used sampler, the 37-mm closed-face polystyrene cassette (CFC), is specified as the sampler of choice in many U.S. National Institute for Occupational Safety and Health (NIOSH) and U.S. Occupational Safety and Health Administration (OSHA) methods (1). While the specific methods may not explicitly call for the recovery and analysis of CFC wall deposits, inclusion of wall deposits is called for by both agencies (22). Another widely used sampler, the Institute of Occupational Medicine (IOM) personal inhalable sampler, was specifically developed for the purpose of collecting the inhalable fraction of aerosol in accordance with ISO 7708 specifications (23). Wall deposits in this sampler were noted during its development and are specifically included as part of the sample (24), although no standard protocol has been published for their inclusion other than for gravimetric analysis. Side-by-side studies have shown little difference between these two samplers when used to collect aerosol in metals industries (12), provided they are analyzed by the same procedure (that is, filter only or filter plus wall deposits). Fewer studies have been carried out in non-metal industries. However, in the study of sewage composting

facilities (16), wall deposits of endotoxin exceeded 40 % of the total sample in 34 % of cassettes and exceeded more than half the total sample in some. In the laboratory study of wood dust (17) 85 % of the sample aspirated was found on the cassette walls. In the pharmaceutical industry study (18), averages of 51 %, 62 %, and 72 % of the sample was found on non-filter internal surfaces, depending on compound. Figure 8.2 of Aitken and Donaldson (3) provides a graph of mass fraction wall deposits of inert particles in the IOM sampler versus particle size. Although the actual data points are not provided the median is approximately 18 % and the maximum approximately 55 %, in accordance with the data in Table 2. Witschger, et al., (4) provides similar data, with a maximum wall deposit of 50 %. While both these studies were performed in a laboratory, Lidén, et al., (19) presents averages of 24–37 % wall deposits in a range of field samples from non-metal industries, depending on industry.

5.7 The Gesamtsstaubprobenahme (GSP) inhalable sampler, and similar metal or plastic versions referred to as a conical inhalable sampler (CIS), has not been the subject of similar extensive investigations of wall deposits. While the GSP met the inhalable convention in a European study without considering wall deposits for particles up to 25 µm AED (25), for particles up to 50 µm AED it under-samples by an average of 21 % with respect to the IOM sampler (when wall deposits are considered in the IOM sampler) (26). A study of wall deposits at a lead mine concentrate mill (5) showed up to 40 % (median 24 %) of total aspiration on the walls, while the laboratory wood study (17) found an average of 42 %, suggesting that wall deposits be considered with this sampler. Other samplers not specified in this practice may also have wall deposits; these should be evaluated on a case-by-case basis.

5.8 No pattern has been discerned that might allow for correction factors to be used in any single sampler without introducing too great an uncertainty into the result (1, 12)). Therefore, it is necessary to account for the wall deposits in all cases where the sample is meant to include the total aspirated aerosol into the sampler. On the other hand, enough data have now been accumulated to allow rough assumptions to be made

**TABLE 1 Summary of Findings of Internal Wall Deposits as a Percentage of Total Analyzed Mass (Filter Plus Walls) in Field Samples for Metals Using the 37-mm CFC Sampler**

Work Environment/Activity (Reference)	<i>n</i>	Agent	Median Wall Deposit	Maximum Wall Deposit
Copper smelter (1)	18	Cu	21 %	55 %
Lead ore mill (5)	9	Pb	19 %	35 %
Solder manufacture (6)	30	Pb	29 %	74 %
Battery production (7)	16	Pb	28 %	66 %
Battery recycling (8)	54	Pb	29 %	54 %
Welding (9)	10	Cr(VI)	5 %	55 %
Plating (9)	12	Cr(VI)	12 %	17 %
Paint spray (9)	29	Cr(VI)	7 %	12 %
Zn Foundry (7)	9	Zn	53 %	62 %
Zn plating (7)	18	Zn	27 %	91 %
Cast iron foundry (7)	18	Fe	22 %	46 %
Grey iron foundry (7)	18	Fe	24 %	77 %
Bronze foundry (10)	6	Cu, Pb, Zn	19 %, 13 %, 15 %	45 %, 17 %, 21 %
Cuproberyllium alloying (7)	4	Cu, Be	31 %, 12 %	40 %, 39 %
Solder manufacturer (15)	50	Pb	45 %	77 %
Solder manufacturer (15)	47	Sn	56 %	93 %

**TABLE 2 Summary of Findings of Internal Wall Deposits as a Percentage of Total Analyzed Mass (Filter Plus Walls) in Field Samples for Metals Using the IOM Sampler**

Work Environment/Activity (Reference)	<i>n</i>	Agent	Median Wall Deposit	Maximum Wall Deposit
Lead ore mill (5)	8	Pb	19 %	30 %
Copper smelter (1)	17	Cu	16 %	38 %
Copper electrorefinery (2)	48	Cu	18 %	36 %
Battery production (11)	11	Pb	8 %	33 %
Welding (11)	18	Al	3 %	13 %
Cast iron foundry (7)	18	Fe	8 %	69 %
Grey iron foundry (7)	18	Fe	5 %	16 %
Bronze foundry (9)	6	Cu, Pb, Sn, Zn	0 %, 0 %, 0 %, 3 %	10 %, 3 %, 23 %, 6 %

regarding the effect of wall deposits on a large population of samples, either historically or for predictive purposes, including estimating the proportion of likely overexposures. These estimates become more precise where there is a body of data involving filter-only and filter plus wall deposits from the specific environment of interest.

5.9 Samplers for the ISO 7708 respirable fraction of dust have filters contained in holders downstream of (after) the size-separation device, typically a cyclone. These sample holders, where not electrically conductive, have also been shown to exhibit significant proportions of wall deposits. In a study of field samples (19), up to 32 % of total collected quartz was found on the walls of 2-piece non-conductive styrene cassettes and up to 55 % on the walls of 3-piece styrene non-conductive cassettes, which is similar to what was found in laboratory studies (20).

## 6. Reagents and Equipment

6.1 For information on equipment and reagents to be used, see the specific applicable test method(s) (see 5.1 or other).

## 7. Procedural Guidance

7.1 Samples are typically collected in accordance with the applicable test method (see 5.1 or other).

7.2 After sample collection, the exterior of the sampler is carefully cleaned by wiping with an appropriate material to ensure the sample is not contaminated by material not considered part of the sample. Where the sample is considered to be all particles entering the aerosol sampler, the measurement procedure is intended to analyze all of the collected particles within the sampler.

7.2.1 For gravimetric analysis, when the filter is bonded to a tared internal capsule (usually made from polyvinyl chloride (PVC)), which includes all particles of interest, the capsule is weighed in its entirety by means of a high-sensitivity analytical balance (typically accurate to the nearest 0.001 mg) in an environmentally-controlled area. The mass of collected aerosol is given by the difference in mass (in units of grams or milligrams) between the pre-weighed (prior to sampling) and post-weighed (subsequent to sampling) internal capsule.

7.2.2 It is also possible to recover collected metalliferous aerosols through acid extraction/digestion within an aerosol sampler, but the sampler must necessarily be watertight and resistant to the extraction solution employed. A procedure for in-situ (that is, within-sampler) extraction in France (27) uses a 3-piece polystyrene cassette as the container for both

sampling and extraction. Originally the filter used consisted of quartz fiber media, but high background of some metals in these filters, together with the need for large quantities of hydrofluoric acid for complete dissolution of the filter, has led to replacement with a cellulose support pad and mixed cellulose ester (MCE) filter (28). After sampling, the cassette is inverted and opened from the rear and the support pad removed, leaving the MCE filter in place. Acids (perchloric first, followed by nitric and hydrochloric, with hydrofluoric where necessary) are pipetted into the cassette, which is then sealed and placed in an ultrasonic bath for 10 minutes (being upended after 5 minutes).

NOTE 2—While this procedure purports to include wall deposits, and very likely does so, the efficiency of recovery of wall deposits has not been investigated systematically, for example by wiping the interiors of field sample cassettes after extraction.

7.2.3 A procedure that has often been used is to rinse the interior of the sampler after removing the filter and adding the washings to the solution used to extract or digest the filter. The rinsate typically is acidic for metals analysis, aqueous for acid mists, basic for alkaline dusts or hexavalent chromium, or organic for organic aerosols. However, rinsing procedures have been tested extensively for the 37-mm polystyrene cassette and have been shown to result in recovery that is less than satisfactory (8, 29). Available evidence (see Table 2) suggests that rinse recovery from other samplers by rinsing also is less than fully quantitative. It is likely that the precision of recovery is better (lower) where there is fully quantitative removal of material on internal sampler walls.

7.2.4 Another procedure that is often used entails wiping the interior surfaces of the sampler (container or holder), digesting and analyzing the wipe separately or adding the wipe material to the filter in the digestion step. This has been found to be much more efficient than the rinsing procedure, even with a single wipe (8, 29), although it is a manual technique potentially subject to inter-personal or intra-personal variation. Wiping with a moistened wipe allows a combination of mechanical removal with wetting or solubilization. The choice of wipe is important: it must be free of significant background contamination and it must be compatible with the digestion and analytical procedure. Typically, the same material as would be used for a surface wipe sample is to be preferred. These wipe materials are typically fully digestible in any acid mixture that would be used to digest the filter. However, where the

procedure has not been validated using laboratory samples, the analysis of a second wipe of field samples can be a reasonable guide to recovery.

7.3 Another procedure that has been shown to be effective is to use a weighable or digestible capsule housed within the sampler (30-38). After sample collection, the internal capsule is removed from the sampler housing and then analyzed.

7.3.1 A weight-stable capsule has been used by OSHA in a 37-mm closed-face cassette for gravimetric analysis since the inception of the agency (30). However, the specific gravimetric capsule used by OSHA is expensive and may be replaced by a PVC filter and capsule in other uses (31).

7.3.2 MCE filters bonded to cellulose acetate capsules have been evaluated for the analysis of metals (32-35), including interlaboratory studies. Following sample collection, internal capsules are subjected to sample preparation by means of acid digestion, with sample aliquots then analyzed by means of appropriate atomic spectrometric or other applicable analysis. These capsules can be digested using any acid procedure employed for filters alone, although the time for digestion may need to be extended. Background contaminant levels in cellulose internal capsules are sufficiently low to enable their use in compliance with current occupational exposure limit values.

NOTE 3—Similar PVC and cellulose capsules are commercially available for a current version of the IOM sampler, known as a disposable inhalable sampler, for gravimetric and elemental analysis, respectively.

7.4 Recently, it has been noted that a small percentage of sampled aerosol may dislodge from the filter or walls during transportation and be deposited on the interior of the inlet cap of the IOM sampler (2). The percentage losses may be considered tolerable, but further investigation of this issue is warranted.

7.5 Filters that are used for respirable size-selected particulate (as in Test Method D4532, Practice D6061, and Test Method D7948) should be housed only in conductive (static-dissipative) cassettes unless the filter deposits will be wiped and added to the analysis. Wall deposits still occur in conductive cassettes, but a study showed that 90 % of samples collected in conductive cassettes contained wall deposits that were less than 10 % of the total collected by the air sampler (21). This small percentage loss may be considered tolerable (35).

NOTE 4—While not explicitly evaluated, the above recommendation probably also applies to thoracic samplers.

## 8. Performance Data

8.1 Current NIOSH policy is that all particles entering the sampler should be included in the analysis, except in the case of respirable particles sampled using conductive cassettes (35), and methods have been published for this purpose (36, 37).

8.2 NIOSH Method 0501 (36) was tested in the NIOSH laboratory using blank internal PVC capsules and with capsules spiked with 0.1–4 mg of National Institute of Standards and Technology (NIST) Standard Reference Material (SRM)

1648: Urban Particulate Matter, and with Arizona Road Dust (Air Cleaner Test Dust). The method reports a bias of 0.058 and an overall precision of 0.059, for an accuracy of  $\pm 15.5$  %. Weight stability over 28 days was verified for both blanks and spiked capsules. Independent laboratory testing on blanks and field samples was used to verify long-term weight stability as well as sampling and analysis uncertainty estimates. The working range is given as 0.25 to 5 mg per sample, with an estimated limit of detection of 0.075 mg per sample and a precision at approximately 2 mg per sample of 0.031.

8.3 In using MCE filters bonded to cellulose acetate internal capsules, NIOSH Method 7306 (37) reported the data shown in Table 3 and Table 4 by element as measured by inductively-coupled plasma (ICP) optical emission spectroscopy after acid digestion.

8.4 The PVC capsule for the disposable inhalable sampler has been evaluated for gravimetric analysis by the Institut de recherche Robert-Sauvé en santé et en sécurité du travail (IRSST) in Québec, Canada (36). The IRSST evaluation yielded a 60  $\mu\text{g}$  reporting limit after 24 hours equilibration at constant temperature and humidity pre- and post-sampling, and measurement uncertainty of 2.3 %. In performance tests collecting flour dust in bakeries, the disposable inhalable sampler gave results equivalent to the IOM sampler.

8.5 The MCE capsule for the disposable inhalable sampler has undergone preliminary performance testing.<sup>6</sup> The median blank values from analysis of ten unused capsules are provided in Table 5. The blank-corrected limits of quantitation from 10 $\times$  the standard deviation of these analyses are given in Table 6. Digestion was carried out in a microwave oven using nitric-hydrochloric-hydrofluoric acids with hydrogen peroxide and analyses were carried out using magnetic-sector ICP-Mass Spectrometry (38).

## 9. Report

9.1 Mass or concentration data reported without assessment of wall deposits shall be reported as such in any laboratory report and flagged as a possible underestimation of exposure.

9.2 When data are reported from gravimetric analysis or acid digestion of an internal capsule with attached filter, or where a rinse or wipe of the sampler interior is combined with the filter for analysis, it is not possible to provide separate values for filter and wall deposits, but the expanded uncertainty of the procedure also is a single value. If the wall deposits are analyzed separately from the filter, then each has its own uncertainty which must be combined for the calculation of expanded uncertainty.

## 10. Keywords

10.1 aerosols; air samplers; workplace atmospheres

<sup>6</sup> Preliminary performance testing took place at the Wisconsin State Laboratory of Hygiene Trace Element Research Group in Madison, WI. For inquiries about this, please contact the D22 Staff Manager at ASTM International Headquarters.