



Designation: D8208 – 19

Standard Practice for Collection of Non-Fibrous Nanoparticles Using a Nanoparticle Respiratory Deposition (NRD) Sampler¹

This standard is issued under the fixed designation D8208; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This practice describes specified apparatus and procedures for collection of non-fibrous airborne metal nanoparticles generated during work activities.

1.2 Nanoparticle respiratory deposition (NRD) samplers are designed to follow a nanoparticulate matter (NPM) deposition curve based on the International Commission on Radiological Protection (ICRP) model for deposition of particles smaller than 300 nm (the minimum deposition for submicrometre particles) while removing the larger particles (1).²

1.3 This practice is applicable to personal and area sampling during work processes and situations where metal nanoparticles may be generated (for example, welding, smelting, shooting ranges).

1.4 This practice is intended for use by professionals experienced in the use of devices for occupational air sampling (such as cyclone samplers).

1.5 This practice is not applicable to the sampling of fibrous nanoparticles such as carbon nanotubes.

1.6 Detailed operating instructions are not provided owing to differences among various makes and models of suitable devices and instruments. The user is expected to follow specific instructions provided by the manufacturers of particular items of equipment. This practice does not address comparative accuracy of different devices nor the precision between instruments of the same make and model.

1.7 This practice contains notes that are explanatory and are not part of the mandatory requirements of the method.

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

1.9 *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.

1.10 *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:*³

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D4532 Test Method for Respirable Dust in Workplace Atmospheres Using Cyclone Samplers

D4840 Guide for Sample Chain-of-Custody Procedures

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

D6785 Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry

D6832 Test Method for the Determination of Hexavalent Chromium in Workplace Air by Ion Chromatography and Spectrophotometric Measurement Using 1,5-diphenylcarbazide

D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D7202 Test Method for Determination of Beryllium in the Workplace by Extraction and Optical Fluorescence Detection

D7439 Test Method for Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma–Mass Spectrometry

E1370 Guide for Air Sampling Strategies for Worker and Workplace Protection

¹ This practice 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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² The boldface numbers in parentheses refer to a list of references at the end of this standard.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at 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 ISO Standards:⁴

[ISO 7708 Air Quality—Particle Size Fraction Definitions for Health-Related Sampling](#)

[ISO 13137 Workplace Atmospheres—Pumps for Personal Sampling of Chemical and Biological Agents—Requirements and Test Methods](#)

[ISO 13138 Air Quality—Sampling Conventions for Airborne Particle Deposition in the Human Respiratory System](#)

[ISO 14644-1 Cleanrooms and Associated Controlled Environments—Part 1: Classification of Air Cleanliness by Particle Concentration](#)

[ISO 17205 General Requirements for the Competence of Testing and Calibration Laboratories](#)

3. Terminology

3.1 *Definitions*—For definitions of pertinent terms, see Terminology [D1356](#).

4. Summary of Practice

4.1 An NRD sampler is assembled into a sampling train and the flow rate is set to 2.5 L/min using a calibrated flow meter. The sampler is designed to collect non-fibrous nanoparticles of aerodynamic equivalent diameter <0.3 μm in accordance with a published performance specification (1).

4.2 The NRD sampler is used to collect non-fibrous nanoparticles (for example, airborne metal nanoparticles) in the target workplace environment. Either personal or area samples can be collected.

4.3 The collected sample in the diffusion stage of the NRD sampler can be transported to a laboratory for subsequent sample preparation and analysis (for example, trace metals dissolution in accordance with Test Method [D7035](#) and analysis by Test Method [D7439](#)).

NOTE 1—Samples obtained using this practice may be suitable for analysis by other elemental measurement methods besides inductively coupled plasma–mass spectrometry (ICP-MS) (for example: graphite furnace atomic absorption spectrometry (GFAAS; see Test Method [D6785](#)), ion chromatography for hexavalent chromium (Cr(VI)) (see test method [D6832](#)), optical fluorescence method for ultra-trace beryllium (see Test Method [D7202](#)), and so forth).

5. Significance and Use

5.1 Exposures to high concentrations of aerosolized fine and ultrafine non-fibrous metal particles, including manganese (Mn), chromium (Cr), and nickel (Ni) generated during processes that involve high energy such as welding or smelting, may elicit deleterious health effects. Animal and epidemiological studies have associated welding and related work processes with a wide range of adverse health effects, including upper respiratory effects (rhinitis and laryngitis), pulmonary effects (pneumonitis, chronic bronchitis, decreased pulmonary function), potential neurological disorders (manganese-

induced Parkinsonism), and high lung cancer and pneumoconiosis death rates. Manganese has been associated with neurological diseases.

5.2 Nanoparticles produced from metals, or their oxides and chalcogenides, have found many industrial uses. Examples of nanometals include silver (Ag), gold (Au), iron (Fe), copper (Cu), cadmium (Cd), zinc (Zn), platinum (Pt), and lead (Pb); examples of nanometal oxides include aluminium oxide (Al₂O₃), magnesium oxide (MgO), zirconium dioxide (ZrO₂), cerium(IV) oxide (CeO₂), titanium dioxide (TiO₂), zinc oxide (ZnO), iron(III) oxide (Fe₂O₃), and tin(II) oxide (SnO); examples of nanometal sulfides include copper monosulfide (CuS), cadmium sulfide (CdS), zinc sulfide (ZnS), silver sulfide (Ag₂S), tin sulfide (SnS), and many sulfides of Ni and cobalt (Co); examples of nanometal selenides include zinc selenide (ZnSe), cadmium selenide (CdSe), and mercury selenide (HgSe). Both the manufacture and use of these nanoparticles can result in particle inhalation, and consequent ill-effects. A stronger association has often been found between adverse health and cellular effects and inhalation of nanoparticles compared to larger particles of the same composition.

5.3 Aerosol sampling methods generally specify the collection of workplace air samples using inhalable and related samplers. These exposure assessment methods, as well as the use of respirable and thoracic samplers (ISO 7708), are inadequate for measurements of nanoparticle exposure when paired with gravimetric analysis. Large particles (>1 μm) weigh substantially more than nanoparticles typical of fumes and, consequently, obscure the ability to detect nanoparticles through gravimetric filter sampling. Additionally, most size-selective samplers collect all particles in the fraction of aerosol that can penetrate into the respiratory tract. Particle deposition, which is governed by the principles of impaction, interception, and diffusion (ISO 13138), is typically overestimated by these samplers.

5.4 There is a need to measure nanoparticle airborne concentrations apart from larger particles. An NRD sampler selectively collects nanoparticles in a manner similar to their typical deposition in the human respiratory tract. The constant motion of nanoparticles causes them to diffuse and potentially deposit in all regions of the respiratory tract, from the head airways to the deep alveolar region, as described by the ICRP (2). NRD samplers are designed to follow a nanoparticulate matter (NPM) deposition curve based on the ICRP model for deposition of particles smaller than 300 nm (the minimum in deposition for submicrometre particles) while removing the larger particles (1). Size-selective samplers (respirable, thoracic, and inhalable) mimic particle penetration rather than particle deposition. Many studies of welding fume have noted that size distribution of welding fume particles brackets the airways deposition minimum so that a substantial proportion of the fume is not deposited in the airways following inhalation (3-7). The use of an NRD sampler, however, approaches exposure assessment from a deposition estimation perspective (8) and provides a more relevant and physiological procedure for measuring actual hazards to workers (such as welders) posed by nanoparticle exposure. This knowledge is critical to the development of toxicological studies aimed at finding links

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

between deposition of metal-containing nanoparticles and adverse health effects.

5.5 Welding fumes are dominated by incidental nanoparticles (particles with any external dimension in the nanoscale), but also include larger particles generated by splatter. Current animal and epidemiological studies investigate exposure to welding fumes without differentiating between nanoparticles and larger particles. Welding fume nanoparticles have been found to induce more toxic effects at the cellular level and to generate more reactive oxygen species (ROS) activity when compared to larger particles.

5.6 An NRD sampler was initially designed with nylon screens as the diffusion stage for the collection of nanoparticles (1), including welding fume (8, 9), although it was noted at the time that laboratory tests of this embodiment had not also included agglomerated particles, such as those which characterize welding fume. An additional collection mechanism, interception, was later found to play an important role as the sample collection of agglomerated nanoparticles progressed to higher loadings. Performance of the nylon screens for agglomerated particles was found to be affected by accumulated nanoparticle fraction loadings greater than 1 mg. The change in performance was accompanied by an increase in pressure drop across the screens to 14.3 kPa (57 in. of water) (5), which would cause many sampling pumps to fault. At the American Conference of Governmental Hygienists (ACGIH) Threshold Limit Value (TLV)⁵ for welding fume of 5 mg/m³, a one-hour sample at 2.5 L/min will collect 0.75 mg. Since the nanoparticle fraction of welding fume is typically less than half the total mass in air (3), the nylon screens are effective in sampling welding fume for one-hour or less as was borne out in field studies (9).

5.7 A new diffusion stage substrate, polyurethane foam, has characteristics more closely resembling human airways (example, Ref (10)) and may be preferable for collecting agglomerated materials in higher loading scenarios (11). In addition, polyurethane foam does not contain titanium dioxide allowing this sampler to be used to assess nanoparticle titanium dioxide.

5.8 The sampler with polyurethane foam has been shown to mimic the ICRP deposition curve closely when sampling spherical nanoparticles up to 100 nm diameter. Agglomerated particles collected in foam begin to show significant deviations from the simple curve as their size and shape factor increase (11). In Figure 3 of Ref (11), the curve modeling behavior of particles through foam is adjusted according to the dynamic shape factor of the aerosol and the sampler collection is shown to continue to match the modified curve at larger particle sizes. Since foam has proven to be a useful surrogate for lung deposition at larger particle sizes, it can be hypothesized that the adjusted foam model also will mimic the behavior of nanoparticle agglomerates in the lung. Enhanced deposition of larger agglomerates has been observed for agglomerated silica particles in human lung-casts (12) demonstrating that it may be

necessary for an adjustment to the ICRP curve for agglomerates in this size range. However, until future research has identified a more precise adjustment to the ICRP deposition curve for agglomerated particles in the human airways the relationship of foam collection to human airways deposition remains a hypothesis.

5.9 An accurate measurement of flow rate through an NRD sampler is required for experiments where sampling devices and filter materials are to be compared as to the size distribution aerosol they capture. Air flow rate affects the efficiency with which a sampler will capture a particular aerodynamic size of particles. Furthermore, air flow rate through a sampler may affect the distribution of aerosol particles captured on the filters and deposited on the sampler collection substrates and walls. To determine aerosol concentration from a mass of captured particles it is necessary to set and measure flow rates accurately.

NOTE 2—Refer to Guide E1370 for guidance on the development of appropriate exposure assessment and measurement strategies.

6. Interferences

6.1 Trace elemental contamination is a common problem which requires extreme care during sampling and analysis (9). Background levels of certain elemental analytes in sampling media (for instance, Cr) can sometimes be problematic, hence the need for particular care when conducting ultra-trace elemental analysis. Corrective action is required should significant backgrounds levels of target elements be found (for example in media blanks, field blanks, reagents, laboratory equipment.)

6.2 Titanium dioxide is often used to give materials a white appearance, and this is the case with nylon. Hence the nylon screens are not suitable for assessing exposure to nano-titanium dioxide. Low levels of Cd have been detected in blank polyurethane foam (5) but this may be due to mass spectroscopic interference from high levels of tin. Future research may confirm that this interference can be resolved (13).

6.3 The practice may not be applicable to the measurement of carbon nanotubes as fibrous or high aspect ratio materials may not be sampled in accordance with the preferred collection criterion for these materials. Further research is required to establish the appropriate size-selection performance for these materials, and if that is matched by this sampler.

7. Apparatus

7.1 Sampling Equipment:

7.1.1 Typical *handling equipment*, including plastic gloves (powder free) and sample transportation containers; and typical ancillary equipment, including labels and label marker; field notebook or electronic record keeping device; calculator, as necessary.

7.1.2 *High-vacuum grease and spare o-rings* for sampler assembly, and cleaning reagents as necessary when not using commercially available clean samplers (re-use is not recommended because of the risk of contamination; if samplers are re-used, users must select reagents to ensure compliance with 8.1.1).

⁵ Threshold Limit Values (TLV) is a registered trademark of the American Conference of Governmental Industrial Hygienists (ACGIH).



FIG. 1 Housing of NRD Sampler



FIG. 4 NRD Sampler/Aluminum Cyclone Assembly

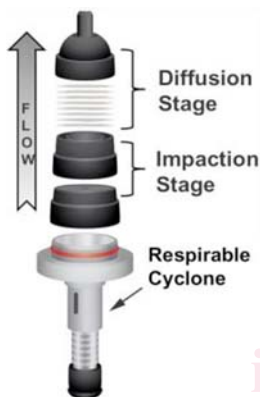


FIG. 2 NRD Sampler Components



FIG. 3 Aluminum Respirable Cyclone

7.1.3 *NRD samplers* (Fig. 1), designed to collect nanoparticles in accordance with the nano-particulate matter criterion (1). Disposable NRD samplers are commercially available; alternatively, non-disposable NRDs may be cleaned and re-loaded by the user, but this is not recommended (see 8.1).

7.1.4 *Nylon screens*: eight 25-mm hydrophilic nylon mesh screens with 11- μm pore size and 6 % porosity, or *polyurethane foam*: 25-mm diameter \times 40-mm length, 90–100 pores per square inch (ppi), to fit the NRD sampler. The screens or foam are placed in the diffusion stage of an NRD device (Fig. 2). The size of the diffusion stage holder is different to accommodate the different size of the substrate (screens or foam). Prior to use foam must be protected from exposure to ultraviolet light, including sunlight, and stored in a cool, dry place.

7.1.5 *Sampling pumps*, with an adjustable flow rate, portable, for use in personal sampling. Pumps shall comply

with ISO 13137, and be capable of maintaining the selected flow rate of 2.5 ± 0.1 L/min and to within ± 5 % of the nominal value throughout the sampling period. Sampling pumps meeting the requirement of ISO 13137 of being able to maintain a flow of 3 L/min against a pressure drop of 4 kPa should be able to maintain flow against the 3.54 kPa of an unloaded NRD sampler at 2.5 L/min.

7.1.6 *Flexible tubing*, 5-mm inner diameter, 0.8-mm outer diameter, of length suitable for making a connection from the sampling pumps to the samplers.

7.1.7 *Respirable aluminum cyclone*, 25-mm, meeting the requirements of Test Method D4532 (Fig. 3).

7.1.8 *Adapter* for connection between the cyclone inlet and flow meter, suited for use with a 25-mm aluminum cyclone.

7.1.9 *Filter cassette holder*, conductive, 25-mm, suited to hold the NRD sampler and cyclone (Fig. 4).

7.1.10 *Flow meter*, portable, with an accuracy that is sufficient to enable the volumetric flow rate to be measured to within ± 2 %. The flow meter shall be calibrated against a standard that is traceable to national standards (see Practice D5337) by a calibration provider accredited to ISO 17205 to provide such calibration services. Many modern flow meters provide electronic data output, including the calculation of averages.

7.1.11 *Flat-tipped forceps*, metal-free, for loading and unloading filters into NRD samplers.

7.1.12 *Timer*, for use in recording of starting and ending times of sampling periods.

8. Sampling Procedures

8.1 Sampler Assembly:

8.1.1 *Cleaning of Samplers*—Disposable NRD samplers are preferred because of the risk of contamination, but otherwise to re-use samplers, a cleaning procedure should be devised by the user to ensure cleaning materials (for example, detergents) thoroughly clean, and do not contaminate the sampler.

8.1.2 *Assembling the NRD Sampler*—Don clean gloves before handling the samplers to minimize the risk of damage or contamination.

8.1.3 *Handling Polyurethane Foam or Nylon Screens*—Handle foam and screens only with clean, flat-tipped, metal-free forceps, and in a clean, uncontaminated area free from high concentrations of air particles (such as a Class 5 environment; see ISO 14644-1).