



Designation: ~~D7049–04 (Reapproved 2010)~~ **D7049 – 17**

Standard Test Method for ~~Metal Removal~~ **Metalworking Fluid Aerosol in Workplace Atmospheres¹**

This standard is issued under the fixed designation D7049; 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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for the determination of both total collected particulate matter and extractable mass ~~metal removal~~ metalworking fluid aerosol concentrations in the range of ~~0.050~~ 0.07 to 5 mg/m³ in workplace atmospheres.

1.2 This test method describes a standardized means of collecting worker exposure information that can be compared to existing exposure databases, using a test method that is also more specific to metal removal fluids.

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

1.4 *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~~ safety, health, and health ~~environmental~~ practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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:*²

[D1356 Terminology Relating to Sampling and Analysis of Atmospheres](#)

[D3195 Practice for Rotameter Calibration](#)

~~[D3670 Guide for Determination of Precision and Bias of Methods of Committee D22](#)~~

[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](#)

~~[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)~~ [/astm-d7049-17](#)

2.2 *U.S. Government Standards/Regulations:*³

[29 CFR 1910.1000 Air Contaminants](#)

[29 CFR 1910.1450 Occupational Exposure to Hazardous Chemicals in Laboratories](#)

2.3 *NIOSH Document:*⁴

~~[Method 0500 NIOSH Manual of Analytical Methods \(NMAM\), 4th Ed](#)~~

3. Terminology

3.1 *Definitions*—For definitions of terms relating to this test method, refer to Terminology [D1356](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *breathing zone, n*—the worker's breathing zone consists of a hemisphere 300-mm radius extending in front of the face, centered on the midpoint of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head and the larynx.

¹ This test method is under the jurisdiction of ASTM Committee [D22](#) on Air Quality and is the direct responsibility of Subcommittee [D22.04](#) on Workplace Air Quality. Current edition approved April 1, 2010/Oct. 1, 2017. Published June 2010/October 2017. Originally approved in 2004. Last previous edition approved in 2004/2010 as D7049 – 04–04 (2010). DOI: 10.1520/D7049-04R10:10.1520/D7049-17.

² 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.

³ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401/Occupational Safety and Health Administration (OSHA), 200 Constitution Ave., NW, Washington, DC 20210, [http://www.osha.gov](#).

3.2.1 *extractable mass, n*—the amount of material removed by liquid extraction of the filter using a mixed-polarity solvent mixture. ~~This mass is an approximation of the metal removal fluid portion of the workplace aerosol.~~

3.2.1.1 *Discussion*—

This mass is an approximation of the metal removal fluid portion of the workplace aerosol.

3.2.2 *filter set, n*—a group of filters from the same production lot that are weighed and assembled into the filter cassettes at one time. The filter set may be used for sampling on multiple days with the appropriate field blanks being submitted for each sampling day.

3.2.2.1 *Discussion*—

The filter set may be used for sampling on multiple days with the appropriate field blanks being submitted for each sampling day.

3.2.3 *metal removal fluids, n*—the subset of ~~metal working~~ metalworking fluids that are used for wet machining or grinding to produce the finished ~~part.~~ Metal removal part; such fluids are often characterized as straight, soluble, semisynthetic, and synthetic.

3.2.3.1 *Discussion*—

~~Metal removal~~ Metalworking fluids addressed by this ~~practice test method~~ include straight or neat oils, not intended for further dilution with water, and water-miscible soluble oils, ~~semisynthetics; semi-synthetics,~~ and synthetics, which are intended to be diluted with water before use. ~~Metal removal~~ Metalworking fluids become contaminated during use in the workplace with a variety of workplace substances including, but not limited to, abrasive particles, tramp oils, cleaners, dirt, metal fines and shavings, dissolved metal and hard water salts, bacteria, fungi, microbiological decay products, and waste. These contaminants can cause changes in the lubricity and cooling ability of the metal removal fluid ~~as well as and may~~ have the potential to adversely affect the health and welfare of employees ~~in contact with~~ exposed to the contaminated metal removal fluid.

4. Summary of Test Method

4.1 Workplace air is drawn into a 37-mm filter cassette containing a tared polytetrafluoroethylene (PTFE) filter for a measured period of time. ~~The total particulate matter concentration is calculated from the mass gain of the filter and the volume of air sampled.~~

4.2 The filter is extracted with a ternary mixture of both nonpolar and polar solvents, a second mixture of methanol and water, dried, and reweighed. The extractable mass concentration is calculated from the loss of mass following extraction and the volume of air sampled. <https://standards.iteh.ai/catalog/standards/sist/0d0f20e2-71dd-4355-b27a-a546a4968bd1/astm-d7049-17>

4.3 As a cost-control procedure, the nonspecific total particulate matter concentration may be used in place of the extractable mass if the total particulate concentration is acceptable to the user of this test method.

5. Significance and Use

5.1 This test method covers the gravimetric ~~determination~~ measurement⁴ of metal removal fluid aerosol concentrations in workplace atmospheres.

5.2 ~~The~~ This test method provides total particulate matter concentrations for comparison with historical exposure databases collected with the same technology.

5.3 ~~The~~ This test method provides an extension to current ~~nonstandardized~~ non-standardized methods by adding an extractable mass concentration which reduces interferences from nonmetal removal fluid aerosols.

5.4 ~~The~~ This test method does not address differences between metal removal fluid types, but it does include extraction with a broad spectrum of solvent polarity to adequately remove any many of the current fluid formulations from insoluble background aerosol ~~adequately; aerosol.~~⁵

5.5 ~~The~~ This test method does not identify or quantify any specific putative toxins in the workplace that can be related to metal removal fluid aerosols or vapors.

5.6 ~~The~~ This test method does not address the loss of semivolatile compounds from the filter during or after collection.

⁴ Available from National Institute for Occupational Safety and Health, 4676 Columbia Pkwy., Cincinnati, OH 45226; "NIOSH Method 0500," in *NIOSH Manual of Analytical Methods*, 4th ed. National Institute for Occupational Safety and Health, Cincinnati, OH. Available from National Institute for Occupational Safety and Health, 4676 Columbia Pkwy., Cincinnati, OH 45226.

⁵ Information from Independent Lubricant Manufacturers Assoc., Health and Safety Task Force, 651 S. Washington St., Alexandria, VA 22314; "NIOSH Method 5524," in *NIOSH Manual of Analytical Methods*, 5th ed., <http://www.cdc.gov/niosh/nmam>.

6. Interferences

6.1 The total particulate matter portion of ~~the~~this test method is not specific to metal removal fluid in the workplace and is subject to positive bias by other aerosol sources.

6.2 The extractable mass concentration measurement improves the specificity of ~~the~~this test method by eliminating insoluble background aerosol from the determination of the metal removal fluid aerosol concentration. This is an important consideration at ~~low-exposure~~low exposure limits.

6.3 Any metal removal fluid components that are insoluble in either extraction solvent mixture will not be measured in the extractable mass fraction.

6.4 The total particulate and extractable mass concentrations measured with this test method are subject to a negative bias, to the extent that semivolatile compounds are lost from the filter during sampling.

6.4.1 Samples of workplace atmospheres in which metal removal fluids containing lower viscosity petroleum fractions or volatile alkanolamines are used may be particularly subject to this negative bias both during sampling and during storage ~~time before~~preceding analysis.

6.5 Any insoluble materials that are lost from the filter during the extraction process will be reported as extractable mass, resulting in a positive bias.

7. Apparatus

7.1 The sampling unit consists of a sampling pump and filter ~~cassette~~cassette sampler, connected by tubing.

7.1.1 Sampling Pump, a constant-flow personal sampling pump capable of a flow rate of 2.0 L/min ($\pm 5\%$) through the filter cassette for a full work shift (8 h).

7.1.2 Filter Cassette, a closed-face (4-mm opening) two- or three-piece 37-mm filter cassette with filter-support pad and inlet and outlet plugs.

7.1.3 Filter, the filter shall be a 2- μ m PTFE membrane filter.

7.1.4 Clips or harnesses, Suitable to provide a suitable means of attaching the pump and filter cassette to the worker for breathing zone sampling.

7.1.5 Field Blank, Tubing, a filter cassette prepared for sampling that has been taken to the workplace and handled in the same manner as the analytical filters, but which has not had any air drawn through it. flexible, for connecting the sampling pump to the sampler.

7.1.6 Precision Flow Meter, for calibration of sampler flow rates (for example, bubble flow meter, dry seal flow meter, or burette and stopwatch).

7.1.7 Rotameter, calibrated in accordance with Practice ~~D3195~~ for field check of sampler flow rate.

7.1.8 Weighing Room, with temperature and humidity control to allow weighing under reproducible environmental conditions of $22 \pm 2^\circ\text{C}$ and $\pm 5\%$ relative humidity in a range of 30 to 55 %.

7.1.9 Analytical Balance, capable of weighing to ± 0.001 mg.

7.1.9.1 Antistatic Strips, of ^{210}Po < 200 days old since packaging.

7.1.10 *Plane-Parallel Press*, for assembling of filter cassettes.⁶

7.1.11 *Chemical Desiccator*, with indicating CaSO₄ desiccant for drying of filters.

7.1.12 *Filter Funnel*, for solvent extraction of 37-mm filters using a dichloromethane, methanol, and toluene mixed solvent. Two choices are available, a 37-mm aluminum funnel and 37-mm disposable polypropylene cassettes, modified for use with this method, with polypropylene support pads.⁷ Ensure the filter funnel does not allow mechanical loss of nonsoluble particulate and that it does allow for quantitative collection of the washings for further chemical analysis of specific constituents, if desired. The funnel should be able to be cleaned between uses to prevent cross-contamination of samples. The use of disposable cassettes eliminates the possibility of cross-contamination, but cassettes should be checked by running blanks to ensure that material is not extracted and added to the filter.

7.1.13 *Laboratory Extraction Blank*, a filter that has been subjected to the extraction procedure in the same manner as the analytical filters, but which has not had any air drawn through it.

7.2 *Field Blank*, a filter cassette prepared for sampling that has been taken to the workplace and handled in the same manner as the analytical filters, but which has not had any air drawn through it.

7.3 *Precision Flow Meter*, for calibration of sampler flow rates (for example, bubble flow meter, or dry seal flow meter).

7.4 *Rotameter*, calibrated in accordance with Practice D3195 for field check of sampler flow rate.

7.5 *Weighing Room*, with temperature and humidity control to allow weighing under reproducible environmental conditions of 22 ± 2°C and ± 5 % relative humidity in a range of 30 to 55 %.

7.6 *Analytical Balance*, capable of weighing to ±0.001 mg.

7.7 *Antistatic Strips*, of ²¹⁰Po, < 200 days old since packaging.

7.8 *Plane-Parallel Press*, for assembling of filter cassettes.⁶

7.9 *Chemical Desiccator*, with indicating CaSO₄ desiccant, for drying of filters.

7.10 *Filter Funnel*, for solvent extraction of 37-mm filters using a dichloromethane, methanol, and toluene mixed solvent.

NOTE 1—At least two choices of filtration funnels are available: a 37-mm aluminum funnel and 37-mm disposable polypropylene cassettes, modified for use with this test method, with polypropylene support pads.⁷

7.11 *Laboratory Extraction Blank*, a filter that has been subjected to the extraction procedure in the same manner as the analytical filters, but which has not had any air drawn through it.

8. Reagents

8.1 *Dichloromethane*, for solvent extraction of nonpolar compounds, analytical reagent grade with a maximum residue on evaporation of 5 ppm(v).

8.2 *Methanol*, for solvent extraction of polar compounds, analytical reagent grade with a maximum residue on evaporation of 5 ppm(v).

8.3 *Toluene*, for solvent extraction of nonpolar compounds, analytical reagent grade with a maximum residue on evaporation of 5 ppm(v).

8.4 *Water*, analytical reagent grade, distilled, deionized, and filtered.

8.5 Prepare an extraction solvent from equal volumes of dichloromethane, methanol, and toluene.

8.5 Prepare a second extraction solvent from equal volumes of methanol (8.2) and water (8.4). *Extraction Solvent Mixtures:*

8.5.1 *Ternary Extraction Solvent*—Prepare an extraction solvent from equal volumes of dichloromethane, methanol, and toluene.

8.5.2 *Methanol/Water Solvent*—Prepare a second extraction solvent from equal volumes of methanol (8.2) and water (8.4).

9. Hazards

9.1 The dichloromethane used as a nonpolar extraction solvent could pose a carcinogenic hazard in the laboratory.

9.2 Perform all operations involving extraction in a chemical fume hood.

9.3 Follow good laboratory procedures for worker protection and waste disposal including (see for example 29 CFR 1910.1000 and 29 CFR 1910.1450:1910.1450).

⁶ See Test Method D4532, where a plane-parallel press description is described (to aid in the assembly of cassettes/sampling cassettes).

⁷ Harper, M., "A Simplified Procedure for the Extraction of Metalworking Fluid Samples in Accordance with Provisional ASTM/NIOSH Methods," *American Industrial Hygiene Association Journal*, Vol 63, 2002, pp. 488-492:492.

10. Sampling

10.1 Preparation of Filter Cassettes:

- 10.1.1 Desiccate the filters (7.1.3) over CaSO₄ (7.1.17.9) for no more than 2 h.
- 10.1.2 Equilibrate or condition the filters in the weighing room (7.1.87.5) for a minimum of 2 h.
- 10.1.3 Place ~~the~~ each filter under a ²¹⁰Po antistatic strip (7.1.9.17.7). Place a second antistatic strip in the balance (7.1.97.6) weighing chamber, if possible.
- 10.1.4 Weigh the PTFE filters (7.1.3) and record the mass to the nearest 0.001 mg (*m*₁).
- 10.1.5 Place the tared filter and filter support (7.1.2) in the filter cassette and close fully using the plane-parallel press (7.1.107.8).
- 10.1.6 Place tape around the circumference of the cassette (7.1.2) and affix a unique number on cassette. Place the ~~H~~Identification number so it remains visible when the cassette is opened to access the filter.

10.2 Assemble the sampling apparatus as shown in Fig. 2 of Practice D5337.

- 10.2.1 Remove filter plugs, attach prepared filter cassette (7.1.2, 10.1), and turn on the sampling pump (7.1.1).

10.3 Check the sampling unit for proper operation, check for leaks, and adjust the flow rate to 2.0 L/min according to Practice D5337.

- 10.4 Install the sampling unit on the worker with the filter cassette (7.1.2) placed in the breathing zone (7.1.4). Place the filter cassette in a manner that prevents the entry of falling or splashing material, but which does not restrict the inlet.

- 10.5 Record the start time and make appropriate field notes to document the process and work practices being monitored.

10.6 Sample at 2.0 L/min for a full shift. ~~Sampling times shorter than a full shift are permitted under any of the following conditions:~~

- 10.6.1 ~~The filter becomes overloaded. (This may be identified by discoloration of the support pad where the fluid has broken through the filter.)~~ Sampling times shorter than a full shift are permitted under any of the following conditions:

- 10.6.1.1 The filter becomes overloaded.

NOTE 2—This may be identified by discoloration of the support pad where the fluid has broken through the filter.

- 10.6.1.2 Specific working operations of shorter duration are being evaluated.

NOTE 3—This raises the limit of detection (LOD) of this test method.

- 10.6.1.3 The sample pump stops at a known time before the end of the shift.

NOTE 4—This may also raise the LOD of this test method.

- 10.6.2 ~~Specific working operations of shorter duration are being evaluated (raises the limit of detection, LOD):~~

- 10.6.3 ~~The sample pump stops at a known time before the end of the shift (raises LOD):~~

- 10.7 ~~Determine~~Measure the final flow rate. ~~Record~~rate, record the stop time, and remove the sampling equipment.

- 10.8 Replace the filter cassette plugs.

- 10.9 For each sampling day, submit at least three field blank (7.1.57.2) filter cassettes or 10 % of the total used, which ever is greater.

- 10.10 If field blanks (7.1.57.2) within a set of filters remain consistent between days, then the field blanks may be pooled for the set of filters to reduce the LOD and limit of ~~quantitation, LOQ:~~quantitation (LOQ) of this test method.

- 10.11 Calculate the LOD and LOQ of this test method using individual day field blanks.

- 10.12 Prepare and analyze all field blanks (7.1.57.2) in the same manner as the analytical filters (7.1.3) used for workplace sampling.

- 10.13 Return the filter cassettes (7.1.2) to the laboratory via overnight delivery service in a container that minimizes sample damage in transit.

- 10.14 Refrigerate received samples at 4 ± 2°C immediately after receipt to preclude bacterial decomposition. ~~Analyze as soon as possible after receipt.~~

- 10.14.1 Store samples no longer than two weeks prior to analysis.

- 10.15 Analyze samples (12) as soon as possible after receipt.

11. Calibration and Standardization

- 11.1 Calibrate the air flow rate of the sampling pump (7.1.67.3 and 7.1.77.4) before each sampling period. The final flow rate shall be determined after sample collection is complete. Samples should be voided if flow-rate changes significantly ($> +5\%$) ($> \pm 5\%$) during the sample period.

11.2 Maintenance and repairs of the sampling and analytical equipment should be performed according to the recommendations of the manufacturer and should be documented in maintenance records.

11.3 The air flow rate calibration shall be performed according to Practice [D5337](#).

11.4 Check the calibration of the analytical balance ([7.1.97.6](#)) daily using National Institute of Standards and Technology Class S-1.1 or ASTM Class 1 masses.

12. Analytical Procedure

12.1 Allow the filters to warm to ambient laboratory temperature. Clean the exterior of the filter cassette ([7.1.2](#)) with a moist lintless towel to avoid sample contamination.

12.2 Remove the tape from the cassette.

12.3 Open the filter cassette ([7.1.2](#)) and carefully remove the filter ([7.1.3](#)) from the holder by inserting a rod through the outlet and against the filter support.

12.4 Desiccate the filter ([7.1.3](#)) in a chemical desiccator ([7.1.17.9](#)) over CaSO₄ for no more than 2 h. Do not desiccate in a vacuum desiccator.

12.5 Equilibrate or condition the filters ([7.1.3](#)) in the weighing room ([7.1.87.5](#)) for a minimum of 2 h.

12.6 Place the filter under a ²¹⁰Po antistatic strip ([7.1.9.17.7](#)). A second antistatic strip should be placed in the balance weighing chamber, if possible.

12.7 Weigh the filter ([7.1.3](#)) and record the mass to the nearest 0.001 mg (*m*²) using the same analytical balance ([7.1.97.6](#)) as was used for the initial weighing.

12.8 Place the filter in the filter funnel ([7.1.127.10](#)). Ensure that the filter funnel does not allow mechanical loss of insoluble particulate and that it does allow for quantitative collection of the washings for further chemical analysis of specific constituents, if desired. The funnel should be able to be cleaned between uses to prevent cross-contamination of samples.

NOTE 5—The use of disposable cassettes eliminates the possibility of cross-contamination, but cassettes should be checked by running blanks to ensure that material is not extracted and added to the filter.

12.9 Apply 10 mL of the ternary extraction solvent ([8.58.5.1](#)). Allow at least five minutes contact time.

12.10 Apply vacuum to the filter funnel. Assure that solvent vaporized does not contaminate the vacuum system.

12.11 Apply 10 mL of the 1:1 v/v blend of methanol:water ([8.5.2](#)) to the filter and extract in the same manner as in [12.10](#), allowing at least one minute contact time.

12.12 Apply an additional 10 mL of the ternary solvent mixture ([8.58.5.1](#)) to the filter and extract in the same manner as Steps [12.10](#) and [12.11](#), allowing at least one minute contact time.

NOTE 6—If desired, the solvent washings may be quantitatively collected for chemical analysis of specific contaminants.

12.13 Rinse the inside of the extraction filter with a stream of the ternary solvent mixture ([8.58.5.1](#)) to return any insoluble particulate to the filter and to aid in drying.

12.13.1 If more rapid removal of solvent is desired, filters can be placed on a slightly warm, clean hot plate covered with new aluminum foil or in a clean, dust-free vacuum oven not to exceed 50°C for a few minutes.

12.14 Equilibrate the filter ([7.1.3](#)) in the weighing room for 2 h minimum.

12.15 Place the filter under a ²¹⁰Po antistatic strip ([7.1.9.17.7](#)). A second antistatic strip should be placed in the balance weighing chamber, if possible.

12.16 Weigh the filter ([7.1.3](#)) and record the mass to the nearest 0.001 mg (*m*³) using the same analytical balance ([7.1.97.6](#)) as was used for the previous weighings.

12.17 Follow the same procedure ([12.1 – 12.16](#)) for the field blanks.

12.18 Perform laboratory blank ([7.1.137.11](#)) analyses for total particulate matter and extractable mass at least on each new lot of filters or solvents using the procedure in [12.1 – 12.16](#).

12.19 Maintain appropriate QA/QC charts on all laboratory and field blanks and instrument calibrations.

13. ~~Calculation~~Calculations

13.1 Mass of total particulate aerosol collected on the sample filter (all masses expressed in mg):

$$M_{TP} = (m_2 - m_1) - m_B \quad (1)$$

where:

*m*₁ ≡ tare mass of the filter before sampling,