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Standard Test Method for
Metal Removal Fluid Aerosol in Workplace Atmospheres[1]

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 fluid aerosol concentrations in the range of 0.05 to 5 mg/m^3 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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
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

2.2 *Government Standards:*

29 CFR 1910.1000 Air Contaminants[3]
29 CFR 1910.1450 Occupational Exposure to Hazardous Chemicals in Laboratories[3]

2.3 *NIOSH Document:*

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

3. Terminology

3.1 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.

3.2.2 *extractable mass, n*—the 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.

3.2.3 *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.

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

3.2.4.1 *Discussion*—Metal removal fluids addressed by this practice include straight or neat oils, not intended for further dilution with water, and water-miscible soluble oils, semisynthetics, and synthetics, which are intended to be diluted with water before use. Metal removal 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

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[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.

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[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.

[4] Available from National Institute for Occupational Safety and Health, 4676 Columbia Pkwy., Cincinnati, OH 45226.

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 have the potential to adversely affect the health and welfare of employees in contact with 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.

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 of metal removal fluid aerosol concentrations in workplace atmospheres.

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

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

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

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

6. Interferences

6.1 The total particulate matter portion of the 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 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 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 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 pump and filter cassette.

7.1.1 *Pump,* a constant-flow personal sampling pump capable of a flow rate of 2.0 L/min (±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-µm PTFE membrane filter.

7.1.4 Suitable means of attaching the pump and filter cassette to the worker for breathing zone sampling.

7.1.5 *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.

7.1.6 *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 ± 2°C and ±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 $CaSO_4$ 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

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[5] Information from Independent Lubricant Manufacturers Assoc., Health and Safety Task Force, 651 S. Washington St., Alexandria, VA 22314.

[6] See Test Method D4532, a plane-parallel press description to aid in the assembly of cassettes.

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.

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.

8.6 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 29 CFR 1910.1000 and 29 CFR 1910.1450.

10. Sampling

10.1 *Preparation of Filter Cassettes:*

10.1.1 Desiccate the filters (7.1.3) over $CaSO_4$ (7.1.11) for no more than 2 h.

10.1.2 Equilibrate or condition the filters in the weighing room (7.1.8) for a minimum of 2 h.

10.1.3 Place the filter under a ^{210}Po antistatic strip (7.1.9.1). Place a second antistatic strip in the balance (7.1.9) 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 (7.1.10).

10.1.6 Place tape around the circumference of the cassette (7.1.2) and affix a unique number on cassette. Place the ID number so it remains visible when 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) 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.)

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 the final flow rate. Record the stop time and remove the sampling equipment.

10.8 Replace the filter plugs.

10.9 For each sampling day, submit three field blank (7.1.5) filter cassettes or 10 % of the total used, which ever is greater.

10.10 If field blanks (7.1.5) 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.

10.11 Calculate the LOD and LOQ using individual day field blanks.

10.12 Prepare and analyze all field blanks (7.1.5) 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.

11. Calibration and Standardization

11.1 Calibrate the air flow rate of the sampling pump (7.1.6 and 7.1.7) before each sampling period. The final flow rate

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[7] Harper, M., "A Simplified Procedure for the Extraction of Metalworking Fluid Samples in Accordance with Provisional ASTM/NIOSH Methods," *AIHAJ*, 63, 2002, pp. 488-492.

shall be determined after sample collection is complete. Samples should be voided if flow-rate changes significantly (> + 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 (7.1.9) 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 (7.1.11) over $CaSO_4$ 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 (7.1.8) for a minimum of 2 h.

12.6 Place the filter under a ^{210}Po antistatic strip (7.1.9.1). 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 (7.1.9) as was used for the initial weighing.

12.8 Place the filter in the filter funnel (7.1.12).

12.9 Apply 10 mL of the ternary extraction solvent (8.5). 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 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 (8.5) to the filter and extract in the same manner as Steps 12.10 and 12.11, allowing at least one minute contact time.

NOTE 1—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 (8.5) 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 ^{210}Po antistatic strip (7.1.9.1). 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 (7.1.9) 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 (7.1.13) 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

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

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

where:

m_1 = tare mass of the filter before sampling,
m_2 = mass of the filter after sampling,
m_B = mean field blank mass change,[8] and
M_{TP} = total particulate mass.

13.2 Sample volume during collection:

$$V_s = Q \times T/1000 \tag{2}$$

where:

Q = mean flow rate, L/min,
T = sample time, min,
1000 = conversion of L to m^3, and
V_S = sample volume in m^3 of air.

13.3 Total particulate matter concentration:

$$C_{TP} = M_{TP}/V_S \tag{3}$$

where:

M_{TP} = total particulate mass from Eq 1,
V_S = sample volume in m^3 from Eq 2, and
C_{TP} = total particulate matter concentration in mg/m^3.

13.4 Extractable mass:

$$M_E = (m_2 - m_3) - m_E \tag{4}$$

where:

m_2 = mass of the filter after sampling,
m_3 = mass following solvent extraction,
m_E = mean lab extraction blank, and
M_E = extractable mass.

13.5 The extractable mass concentration:

$$C_E = M_E/V_S \tag{5}$$

where:

M_E = extractable mass from Eq 4,

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[8] A pooled mean field blank mass change can be used for a set of filters (12.17).

V_S = sample volume from Eq 2, and
C_E = extractable mass concentration.

13.6 Limit of detection for total particulate matter:

$$LOD = 3 \times STD_{FB} \tag{6}$$

where:

STD_{FB} = standard deviation of field blanks, and
LOD = limit of detection.

13.7 Limit of quantitation for total particulate matter:

$$LOQ = 10 \times STD_{FB} \tag{7}$$

where:

STD_{FB} = standard deviation of field blanks, and
LOQ = limit of quantitation.

13.8 Limit of detection for extractable mass:

$$LOD = 3 \times STD_{EB} \tag{8}$$

where:

STD_{EB} = standard deviation of laboratory extraction blanks, and
LOD = limit of detection.

13.9 Limit of quantitation for extractable mass:

$$LOQ = 10 \times STD_{EB} \tag{9}$$

where:

STD_{EB} = standard deviation of laboratory extraction blanks, and
LOQ = limit of quantitation.

14. Report

14.1 Report total particulate concentration (13.3), extractable mass concentration (13.5), limit of detection for total particulate matter (13.6), limit of quantitation for total particulate matter (13.7), limit of detection for extractable mass (13.8) and limit of quantitation for extractable mass (13.9).

15. Precision and Bias[9]

15.1 *Analytical Range,* 0.05 to 5 mg per sample (approximately 0.2 to 5 mg/m^3 for full-shift sample).

15.2 *Estimated Limit of Detection:*[10]

15.2.1 *Total Particulate Matter*—See Eq 6. While the full validation of this test method is pending, NIOSH Method 0500 reports a LOD of 30 µg or a corresponding full-shift sample LOD of 0.03 mg/m^3. The test method described here improves on NIOSH Method 0500 by reducing the blank variability through the use of PTFE filter medium.[11]

15.2.2 *Extractable Mass*—See Eq 8. The LOD range for this test method will be established during the validation process.

15.3 *Estimated Limit of Quantitation:*[12]

15.3.1 *Total Particulate Matter*—See Eq 7. While the full validation of this test method is pending, NIOSH Method 0500 reports a LOQ of 100 µg or a corresponding full-shift sample LOQ of 0.10 mg/m^3. The test method described here improves on NIOSH Method 0500 by reducing the blank variability through the use of PTFE filter medium.[11]

15.3.2 *Extractable Mass*—See Eq 9. The LOQ range for this test method will be established during the validation process. Glaser, et al, report that limits of quantitation, estimated from blanks carried through the entire analytical procedure, were 30 mg for the weighing technique and 60 µg for the extraction technique.[13]

15.4 *Analytical Precision*—Analytical precision has not been determined for the entire test method at this time.

15.5 *Range Studied*—NIOSH Method 0500 was 8 to 28 mg/m^3, but this test method has been used extensively in field work at concentrations less than 0.5 mg/m^3.

15.6 *Bias*—NIOSH Method 0500 reports 0.01 %. Full validation of this test method is pending.

15.7 *Precision*—NIOSH Method 0500 reports a CV of 0.026 for their method. Glaser, et al, report that the percent relative standard deviation for those weights of all fluids spiked at levels ≥ 200 µg and the precision (% relative standard deviation or % RSD) was estimated to be 4 % for the total weight procedure and 5 % for the extraction procedure.[14] The precision of this test method is being determined and will be available on or before January 2007.

16. Keywords

16.1 aerosol sampling; air monitoring; grinding fluid; machining fluid; metal removal fluid; metal working fluid; sampling and analysis; workplace atmospheres

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[9] This test method has not been fully validated.

[10] The limit of detection for total particulate matter is dependent on the variability in the field blank values and not on the sensitivity of the balance or other equipment. The limit of detection for extractable mass is dependent on the variability of the laboratory extraction blanks.

[11] A full validation of this test method is being conducted in accordance with Guide D3670 and Practice E691.

[12] The limit of quantitation for total particulate matter is dependent on the variability in the field blank values. The limit of quantitation for extractable mass is dependent on the variability of the laboratory extraction blanks.

[13] Glaser, R. A., Shulman, S., and Klinger, P., "Data Supporting a Provisional American Society for Testing and Materials (ASTM) Method for Metalworking Fluids, Part 2: Preliminary Report of Evaluation of a Ternary Solvent Blank in a Provisional ASTM Method for Metalworking Fluids," *Journal of Testing and Evaluation*, Vol 27, No 2, March 1999, pp. 131-136.

[14] Glaser, R., Shulman, S., Kurimo, R., and Piacitelli, G., "Data Supporting a Provisional ASTM Method for Metalworking Fluids, Part 3: Evaluation of an ASTM Method for Metalworking Fluids in a Survey of Metalworking Facilities," *Journal of Testing and Evaluation*, Vol 30, No 5, Sept. 2002, pp. 439-451.

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