

various organic-soluble compounds MW: various CAS: 65996-93-2 RTECS: GF8655000

**METHOD:** 5023, Issue 2

**EVALUATION:** PARTIAL

**Issue 1:** 15 May 1985  
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**OSHA :** 0.2 mg/m<sup>3</sup> (benzene-solubles)  
**NIOSH:** 0.1 mg/m<sup>3</sup>; carcinogen;  
(cyclohexane-solubles)  
**ACGIH:** 0.2 mg/m<sup>3</sup> (benzene solubles)

**PROPERTIES:** liquid; d ~1.06 g/mL @ 38 °C;  
60 to 85% distills @ ≤ 355 °C;  
creosote distills @ 270 to 395 °C

**SYNONYMS:** benzene-solubles, cyclohexane-solubles, coal tar pitch volatiles, creosote from coal tar.

SAMPLING	MEASUREMENT
<p><b>SAMPLER:</b> FILTER (2-µm, 37-mm PTFE membrane)</p> <p><b>FLOW RATE:</b> 1 to 4 L/min</p> <p><b>VOL-MIN:</b> 500 L @ 0.2 mg/m<sup>3</sup> <b>-MAX:</b> 2400 L</p> <p><b>SHIPMENT:</b> routine</p> <p><b>SAMPLE STABILITY:</b> unknown</p> <p><b>BLANKS:</b> 2 to 10 field blanks per set</p>	<p><b>TECHNIQUE:</b> GRAVIMETRIC</p> <p><b>ANALYTE:</b> organic-solubles (includes anthracene, benzanthracene, benzo(a)pyrene, carbazole, chrysene, phenanthrene, pyrene and others [1,2,3,4])</p> <p><b>EXTRACTION:</b> benzene, cyclohexane or other appropriate solvent; ultrasonic 20 min</p> <p><b>CALIBRATION:</b> NIST Class M weights</p> <p><b>RANGE:</b> 0.1 to 2 mg per sample</p> <p><b>ESTIMATED LOD:</b> 0.05 mg per sample [5]</p> <p><b>PRECISION (<math>\bar{S}_r</math>):</b> 0.02 at 1.35 mg [5]; 0.23 for blanks [5]</p>
ACCURACY	
<p><b>RANGE STUDIED:</b> not studied</p> <p><b>BIAS:</b> unknown</p> <p><b>OVERALL PRECISION (<math>\bar{S}_{rT}</math>):</b> not determined</p> <p><b>ACCURACY:</b> unknown</p>	

**APPLICABILITY:** The working range is 0.1 to 2 mg/m<sup>3</sup> for a 1000-L air sample. The method is useful for air monitoring of coke oven emissions, petroleum combustion products such as diesel emissions, and petroleum asphalt fumes. The method may be applied to bulk samples. The method is non-specific and measures all substances in the sample which are soluble in the solvent selected and which can be desorbed from particulate matter present on the filter.

**INTERFERENCES:** Changes in temperature or humidity during pre- and post-collection weighing affect accuracy. Losses may occur due to volatilization of collected aerosol during or after sampling.

**OTHER METHODS:** This method modifies and combines P&CAM 217 [6] and the criteria document method [2].

**REAGENTS:**

1. Solvent, Benzene,\* cyclohexane or other solvent, reagent grade.
2. Dichromic acid cleaning solution.
3. Acetone, reagent grade.
4. Hexane.

\* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler: PTFE laminated membrane filter, 2- $\mu$ m pore size, 37-mm diameter (Zefluor, Membrana Inc., Pleasanton, CA; Gelman Sciences, or equivalent) backed by a gasket 37-mm OD, 32-mm ID, cut from a cellulose support pad (SKC, Inc. or equivalent) in plastic filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Ultrasonic bath.
4. Microbalance, readable to 1  $\mu$ g, with NIST Class M weights.
5. Environmental chamber for balance, e.g., 20 °C  $\pm$  0.3 °C and 50%  $\pm$  5% relative humidity.
6. Weighing cups, PTFE, 2-mL, approximate tare weight 60 mg, in metal rack.
7. Vacuum oven.  
NOTE: Keep the interior of the vacuum oven dust-free for maximum sensitivity, reproducibility, and accuracy.
8. Forceps.
9. Test tubes, PTFE-lined screw caps, 13-mm x 100-mm.\*
10. Filter, 0.5- $\mu$ m (Millex-SR, Millipore Corp., Bedford, MA or equivalent).
11. Pipets, 1- and 5-mL\* with bulb.

\* Rinse with distilled water, acetone, and hexane; dry.

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**SPECIAL PRECAUTIONS:** Benzene and coal tar pitch volatiles are suspect carcinogens [1,2,3,4].

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**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample volume of 500 to 2400 L. Do not exceed a filter loading of ca. 2 mg total particulate.
3. Replace caps in cassette and ship to laboratory.

**SAMPLE PREPARATION:**

4. Transfer filter carefully using forceps to test tube. Add 5.0 mL solvent via pipet. Cap the tube.  
NOTE 1: Cyclohexane is recommended as solvent because of the carcinogenic potential of benzene [2].  
NOTE 2: This extraction is also applicable to bulk samples (ground and sieved to ca. 250  $\mu$ m). Extract 250 mg bulk sample with 5.0 mL solvent.
5. Place tube upright in beaker containing water to the same level as the liquid in the tube. Place beaker and tube in ultrasonic bath. Ultrasonicate for 20 min.
6. Filter solution through a 0.5- $\mu$ m filter into a clean, preweighed weighing cup. Discard the filter.

NOTE: An aliquot of the solution may be taken at this step if other analyses (e.g., polynuclear aromatic hydrocarbons) are to be performed on the sample. Apply the appropriate aliquot factor in calculations.

#### CALIBRATION AND QUALITY CONTROL:

7. Zero the microbalance on the 1.0 mg range and calibrate per balance manufacturer's directions. NOTE: Perform weighings at constant temperature and relative humidity.
8. Process three blank filters through the extraction and measurement procedures.

#### MEASUREMENT:

9. Transfer via pipet a 1.0-mL aliquot of sample extract to a preweighed weighing cup.
10. Place weighing cup in vacuum oven preheated to 40 °C. Apply vacuum until pressure in the oven is 7 to 27 kPa (50 to 200 mm Hg). Allow solvent to evaporate for 2 hrs. Release vacuum by slowly opening release valve which has an in-line filter to remove room dust.
11. Equilibrate the weighing cup to the temperature and relative humidity of the balance room for at least 30 min. Weigh the weighing cup to the nearest microgram.

#### CALCULATIONS:

12. Determine the mass of organic-soluble residue found in the sample aliquot,  $W$  ( $\mu\text{g}$ ), and in the average media blank,  $B$  ( $\mu\text{g}$ ), and multiply by 5 to give total sample mass.
13. Calculate concentration,  $C$ , of organic-solubles in the air volume sampled,  $V$  (L):

$$C = \frac{(W - B) \cdot 5}{V}, \text{ mg/m}^3.$$

#### EVALUATION OF METHOD:

Several benzene extracts of samples of aluminum reduction plant emissions were combined to give a solution containing 1.35 mg of benzene-soluble material per sample; nine aliquots of this solution gave residue weights with a relative standard deviation of 0.02. Benzene extracts of six blank filters gave residue weights with a relative standard deviation of 0.23 [6].

#### REFERENCES:

- [1] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, Coal Tar Pitch Volatiles, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.
- [2] Criteria for a Recommended Standard ... Occupational Exposure to Coal Tar Products, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-107 (1978).
- [3] Criteria for a Recommended Standard ... Occupational Exposure to Coke Oven Emissions, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 73-11016 (1973).
- [4] 1992-1993 Threshold Limit Values and Biological Exposure Indices. American Conference of Governmental Industrial Hygienists, Cincinnati, OH (1992).
- [5] UBTL Report, NIOSH Sequences #4229-T,U,V (unpublished, April 27, 1984).
- [6] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, P&CAM 217, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).

**METHOD REVISED BY:** B. R. Belinky, NIOSH/DPSE.