## LEAD in Air by Chemical Spot Test
7700

<table>
<thead>
<tr>
<th>Pb</th>
<th>MW: 207.19</th>
<th>CAS: 7439-92-1</th>
<th>RTECS: OF7525000</th>
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<td>METHOD: 7700, Issue 1</td>
<td>EVALUATION: PARTIAL</td>
<td>Issue 1: 15 May 1996</td>
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**OSHA:** 0.05 mg/ m³  
**NIOSH:** <0.1 mg/ m³  
**ACGIH:** 0.15 mg/ m³

**SYNONYMS:** None

**PROPERTIES:** soft metal; d 11.3 g/cm³; MP 327.5 °C; valences +2, +4 in salts

**SAMPLING**

| SAMPLER: FILTER (0.8-µm cellulose ester membrane) | TECHNIQUE: CHEMICAL SPOT TEST KIT (Rhodizonate-based) |
| FLOW RATE: 2 L/min [1] | ANLYTE: rhodizionate complex of lead |
| VOL-MIN: 10 L@0.05 mg/m³[1] | POSITIVE INDICATOR: yellow/orange to pink/red color change (under acidic conditions) [2] |
| -MAX: 240 L | PERFORMANCE PARAMETERS: |
| SAMPLE STABILITY: stable | Positive result: 0.57 µg Pb @ 95% confidence level [1] |
| FIELD BLANKS: 2 to 10 per set (for laboratory analysis) | Negative result: 10.2µg Pb @95% confidence level [1] |

**ACCURACY**

| RANGE STUDIED: < 0.01 to >100.0 µg Pb per filter | BIAS: not applicable |
| OVERALL PRECISION \( \bar{S}_{R-T} \): not applicable | ACCURACY: not applicable |

**APPLICABILITY:** This is a qualitative method only, designed for use in the field. A characteristic color change indicates the presence of lead above a certain mass, as determined by performance parameters for a given test kit. If quantitative results are needed, the filter samples, test kit components, and backup pads may be shipped to the laboratory for analysis by NIOSH methods 7300, 7082, 7105, or equivalent.

**INTERFERENCES:** Tl⁺, Ag⁺, Cd⁺², Ba⁺², and Sn⁺² also form colored compounds with sodium rhodizonate, but with less sensitivity than that of Pb⁺² and only the lead-rhodizionate complex gives the characteristic red color [2].

**OTHER METHODS:** Laboratory methods for the determination of lead include NIOSH methods 7300 (Elements by ICP), 7082 (Lead by Flame AAS), and 7105 (Lead by Graphite Furnace AAS). ASTM E 1553 is an alternate sample collection procedure [3].
REAGENTS:

1. Rhodizonate-based spot test kit
   (Merck EM Quant Lead Test™ or equivalent).
   
   NOTE 1: Rhodizonate may degrade quickly over time. Follow manufacturer’s recommendations for maintaining viability of reagents.

   NOTE 2: Performance parameters on page 7700-1 apply only to Merck EM Quant Test kit (See APPENDIX).

EQUIPMENT:

1. Sampler: Cellulose ester membrane filter, 0.8-μm pore size, 37-mm, in 2- or 3-piece cassette with cellulose backup pad.

2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.

3. Sealable plastic bags.

4. Gloves, powderless, plastic

SPECIAL PRECAUTIONS: None

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 (2 L/min recommended) for a total sample size of 10 to 240 L.

SPOT TESTING (Qualitative measurement):

3. Don a clean pair of gloves.

4. Using an appropriate tool, remove the top portion of the filter cassette (after sample has been collected for desired time period).

5. Apply the spot test to the center of the filter, following manufacturer’s instructions.

6. Record results as positive for lead if a characteristic color change is observed, or negative if no characteristic color change is observed.

   NOTE: For rhodizonate-based lead spot tests (under acidic conditions), the characteristic color change is from yellow or orange to pink or red (2).

LABORATORY ANALYSIS (Optional):

7. Re-cap filter cassettes.

8. Place filter samples and test kit components in resealable plastic bag for shipment to laboratory.

9. Analyze by NIOSH method 7300, 7082, 7105, or equivalent method for lead.

   NOTE: The backup pad must be analyzed for lead that may have wicked through the filter during qualitative measurement (step 5 above).

EVALUATION OF METHOD:

A commercial rhodizonate-based spot test kit (Merck EM Quant Lead Test™) was evaluated for its potential use in the detection of lead in airborne particulate [1]. Personal sampling pumps were used to collect 371 air samples on cellulose ester membrane filters at various worksites where lead was a suspected air contaminant. Each filter sample was tested with an individual chemical spot test, and the samples (test kits included) were then analyzed using NIOSH method 7105. Experimental data were statistically modeled in order to estimate the performance parameters of the spot test kit (see APPENDIX). A positive reading was found at 95% confidence for lead mass values above 10.2 μg Pb per filter, while 95% confidence of a negative reading was found for lead masses below 0.57 μg Pb per filter.
REFERENCES:


METHOD WRITTEN BY:

Kevin E. Ashley, Ph.D., DPSE/MRB

APPENDIX: CALCULATION OF PERFORMANCE PARAMETERS

Example calculations are illustrated here for the performance parameters of the Merck EM Quant Lead Test™; performance parameters for other spot test kits should be estimated by statistical modeling before being used for field screening applications. Note that these calculations assume that the short-term exposures are representative of a full 8-h workday.

Consider first the case of a positive test result after 5 min of sampling at 2.0 L/min (minimum lead mass of 0.57 µg for a total sampling volume of 10 L):

\[
0.057 \text{ µg Pb/L} \times 1000 \text{ L/m}^3 = 57 \text{ µg Pb/m}^3,
\]

which is in excess of the OSHA permissible exposure limit (PEL) of 50 µg/m³ for an 8-h workday.

Consider secondly the case of a negative test result after 2 h of sampling at 2.0 L/min (maximum lead mass of 10.2 µg for a total sampling volume of 240 L):

\[
0.0425 \text{ µg Pb/L} \times 1000 \text{ L/m}^3 = 42.5 \text{ µg Pb/m}^3,
\]

which is below the OSHA PEL for an 8-h workday.

Similar computations may be carried out for other sampling volumes and spot test kits, provided that the performance parameters for the test kits are known.