LEAD BY FIELD PORTABLE XRF 7702

Pb MW: 207.19 (Pb) 223.19 (PbO) CAS: 7439-92-1 (Pb) 1317-3608 (PbO) RTECS: OF7525000 (Pb) OG1750000 (PbO)

METHOD: 7702, Issue 1 **EVALUATION: FULL** **Issue 1: 15 January 1998**

OSHA: 0.05 mg/m^3
NIOSH: <0.1 mg/m^3; blood lead ≤ 60 µg/100 g
ACGIH: 0.05 mg/m^3; BEI blood 30 µg/ 100 mL

PROPERTIES: soft metal; d 11.3 g/mL @ 20 °C; MP 327.5°C; BP 1740°C; valences +2, +4 in salts

SYNONYMS: elemental lead and lead compounds except alkyl lead

SAMPLING

SAMPLER: FILTER (0.8-µm, 37-mm, mixed cellulose ester membrane)

FLOW RATE: 1 to 4 L/min

VOL-MIN: 570 L @ 30.0 µg/m^3 [1]
-MAX: 1900 L @ 9.0 µg/m^3

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.1 to 1514.6 µg/m^3 (as Pb) (based upon lead mass loadings)

BIAS: 0.069 [1]

PRECISION ($\bar{S}_{rT}$): 0.054 @ 10.3 to 612 µg Pb per sample

ACCURACY: ±16.4%

MEASUREMENT

TECHNIQUE: X-RAY FLUORESCENCE (XRF), PORTABLE, L-SHELL EXCITATION (e.g., ^{109}Cd source)

NOTE: Performance parameters are based upon research conducted with the NITON® 700 XRF [1].

ANALYTE: lead

CALIBRATION: lead thin-film standards (Micromatter Co., or equivalent); internal instrument calibration

RANGE: 17 to 1500 µg of Pb per sample [1]

ESTIMATED LOD: 6 µg of Pb per sample [1]

APPLICABILITY: This method was evaluated for air samples on filters only. The working range of this method is 0.017 mg/m^3 to 1.5 mg/m^3. This is a field portable analytical method, particularly useful for the analysis of initial exposure assessment samples, or for applications where laboratory analysis is impractical. Additionally, the method is non-destructive; samples analyzed in the field can later be analyzed in a laboratory. The method is applicable to all elemental lead forms, including lead fume, and all other aerosols containing lead.

INTERFERENCES: The presence of bromine will cause XRF readings for lead to be elevated, resulting in a positive bias error. Other interferences may exist in other XRF instruments.

OTHER METHODS: Laboratory-based methods include atomic spectrophotometric methods following hot plate acid digestion: NIOSH methods 7082 (flame atomic absorption spectrophotometry) [2], 7105 (graphite furnace atomic absorption spectrophotometry) [3], and 7300 (inductively coupled plasma atomic emission spectrophotometry) [4]. A field-portable analytical method for lead air filter samples using ultrasound/ASV has been developed, NIOSH Method 7701[5]. A field-portable screening method by spot test kit has been developed, NIOSH Method 7700 [6].

REAGENTS:

1. None

EQUIPMENT:

1. Sampler: Mixed cellulose ester filter, 0.8-µm pore size, 37-mm diameter, with cellulose back-up pad, in a closed-faced cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Field portable, L-shell X-Ray Fluorescence (XRF) instrument with a Cadmium-109 source.
4. Filter sleeve: thin cardboard with 37-mm dia. cut out, and covered with a light adhesive between two pieces of acetate (Mylar™) (NITON, Bedford, MA, or equivalent).
   NOTE: Material must be transparent to X-ray.
5. Filter test platform to hold the filter (specific to instrument).
6. Forceps
7. Thin film standard reference materials from 15 µg/cm^2 to 150 µg/cm^2 (Micromatter Co., Deer Harbor, WA), or equivalent [7,8].

SPECIAL PRECAUTIONS: None

SAMPLING:

1. Calibrate each sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate (1 to 4 L/min) for a total sample size of approximately 1000 L. Do not exceed a filter loading of 2 mg total dust.

SAMPLE PREPARATION:

3. With forceps, transfer the MCE filter without the backup pad to a filter sleeve. The sleeve material must be transparent to X-rays (see EQUIPMENT, Item 4).
   NOTE: Take special care when removing the filter from the backup pad to avoid loss of lead-containing dust.
4. Place the filter into 37-mm opening and seal with Mylar™ film to prevent losses and allow undisturbed analysis of the filter.
5. Place the sealed filter onto the filter test platform of the instrument for analysis.
   NOTE: The NITON® 700 Series XRF has a filter test platform that allows for three readings with no substrate effect.

CALIBRATION AND QUALITY CONTROL:

6. Start XRF and allow a 30-minute warm-up period. The instrument will conduct an internal self-calibration.
7. Using thin film standards [8], verify the internal calibration to within ± 5% of the calibration standard. Use a minimum of three standards at concentrations of 15 µg/cm^2, 150 µg/cm^2, and one standard concentration between these two values.
8. Restart the instrument as needed to assure instrument accuracy prior to sample analysis.
   NOTE: When the thin film standard measurements are not within the specified parameters, the instrument may need to be recalibrated at the factory.
9. Analyze one thin film standard every 2 hours to check for instrument drift.
10. Repeat step 7 when all analyses are completed as a post-calibration check.

MEASUREMENT:

11. Set instrument parameters and analyze filter samples as specified by the manufacturer. The following measurement technique is based upon the NITON® 700 XRF.
    a. Analyze the middle of the sample filter first (see Figure 1, M).
    b. Allow the instrument to take a one source-minute reading (This may take longer than one real-time minute, depending upon the source strength). A one source-minute reading will assure the accurate L-shell reading necessary for the analysis of lead air filter samples.
    c. Analyze the filter sample at the top of the filter for one source minute (see Figure 1, T).
    d. Analyze the filter sample at the bottom of the filter for one source minute (see Figure 1, B).
    e. The instrument software uses an algorithm that converts the three readings in $\mu g/cm^2$ to an analytical result in µg of lead per sample. This result will be displayed following the third filter reading [1].
    f. Analyze one standard every 2 hours (step 8).
    g. Repeat three-reading calibration check following completion of analyses (step 8).

CALCULATIONS:

12. Using the measured lead concentration, W (µg), calculate the concentration, C (mg/m^3), of lead in the air volume sampled, V (L):

$$C = \frac{W}{V}, \; mg/m^3$$

NOTE: $\mu g/L \equiv mg/m^3$

EVALUATION OF METHOD:

This method was validated on field samples [1] by collecting lead particulate samples from bridge lead abatement projects. Airborne concentrations of lead within the containment of a sand blasting bridge lead abatement project ranged from 1 to 10 mg/m^3. Area samples were collected for periods of time ranging from 15 seconds to 2 hours. This sampling protocol yielded 61 filter samples with lead loadings ranging between 0.1 to 1514.6 µg of lead per sample. Four personal samples were collected from a hand-scraping bridge lead abatement project for a total sample size of 65. The samples were first analyzed using a non-destructive, field portable XRF method. Samples subsequently were subjected to confirmatory analysis by the laboratory based NIOSH method 7105, Lead by GFAAS [3]. The method was statistically evaluated according to the NIOSH Guidelines for Air Sampling and Analytical Method Development and Evaluation [9]. The overall precision ($\hat{S}_{rT}$) of the XRF method was calculated at 0.054 with a 95% confidence interval (CI) of 0.035 to 0.073, and the bias was 0.069 with a 95% CI of 0.006 to 1.515. The XRF method accuracy was determined to be ± 16%; however, at the upper 90% CI, the accuracy is ± 27%. Since the confidence interval includes the ± 25%, meeting the NIOSH accuracy criteria of ± 25% is inconclusive. However, the samples used to evaluate this method were field samples. Laboratory prepared aerosol samples would be expected to give better precision. Additionally, the XRF method is non-destructive; samples analyzed in the field can subsequently be analyzed in a laboratory using a method with greater accuracy, as needed. The filter sleeve used with the NITON® 700 Series XRF used a Mylar film to cover and seal the 37-mm filter. The lead particulate on the surface of the filter came into contact with the Mylar™ film. Both the Mylar™ film and the filter were digested with nitric acid and hydrogen peroxide as is specified in NIOSH Method 7105 [3].

REFERENCES:

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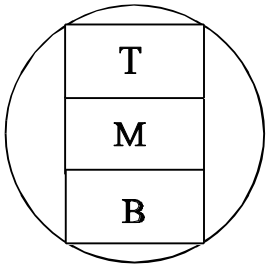

Figure 1: Analysis of a 37-mm filter using XRF
(XRF windows identified as M, T, and B are 2 cm x 1 cm)