9105

valences +2, +4 in salts

LEAD in DUST WIPES by Chemical Spot Test (Colorimetric Screening Method)

Pb MW: 207.19 CAS: 7439-92-1 RTECS: OF7525000

METHOD: 9105, Issue1 EVALUATION: PARTIAL Issue 1: 15 March 2003

OSHA: no PEL **PROPERTIES:** soft metal; d 11.3 g/cm³; MP 327.5 °C

NIOSH: no REL ACGIH: no TLV

PRECISION (Ŝ,T):

ACCURACY:

SYNONYMS: Elemental lead and lead compounds except alkyl lead

Not applicable

Response may differ for different wipe

materials, different matrices and different rhodizonate solutions.

	SAMPLING		MEASUREMENT
SAMPLER:	WIPE, meeting the specifications of ASTM E1792 [1].	TECHNIQUE:	CHEMICAL SPOT TEST, Rhodizonate- based solution or spot test kit applied to wipe sample [2]
SAMPLE:	Human skin (e.g., hands) or non-dermal surfaces (e.g., floors, walls, furniture)	ANALYTE:	Rhodizonate complex of lead
SAMPLE STABILITY: BLANKS:	Stable At least 5% of samples, min. of 2 per batch	POSITIVE INDICATOR:	Observed color change on wipe from yellow/orange to pink/red (under acidic conditions) [2,3]
ACCURACY		RESPONSE:	The method is ordinarily positive for the presence of lead in the range of 5 - 15 µg/sample, upwards to and exceeding
RANGE STUDIED: <0.01 to >1,000 µg Pb per wipe			milligram amounts [3,4].
BIAS:	Not applicable		
OVERALL			

APPLICABILITY: This is a qualitative, colorimetric screening method, designed for field use. The method was designed as a handwipe method for human skin, but is also applicable to various non-dermal surfaces including floors, walls, furniture, etc. A characteristic color change on the wipe (i.e., from yellow/ orange to pink/ red hues) indicates the presence of lead. By estimating the performance parameters for a given wipe/rhodizonate solution combination, the performance characteristics can be evaluated [5]. If quantitative results are needed, lead in the wipe samples can be measured on-site using a modification of NIOSH method 7701, or in a fixed-site laboratory using NIOSH methods 7082, 7105, 7300, or equivalent.

INTERFERENCES: TI $^{+}$, Ag $^{+}$, Cd $^{2+}$, Ba $^{2+}$, and Sn $^{2+}$ also form colored compounds with rhodizonate ion, but with less sensitivity than that of Pb $^{2+}$, and only the lead-rhodizonate complex gives the characteristic pink or red color [3]. Interferences from the wipe medium, e.g., surfactants, are possible.

OTHER METHODS: Laboratory methods for the determination of lead in dust wipes include modifications of NIOSH methods 7082 (Lead by Flame AAS), 7105 (Lead by Graphite Furnace AAS), 7300 (Elements by ACP), and 7701 (Lead by Portable ASV).

REAGENTS:

- 1. Vinegar (e.g., 5% acetic acid as white vinegar) or 1% nitric acid (v/v).*
- 2. Sodium or potassium rhodizonate salt (rhodizonic acid).
 - NOTE: Rhodizonate solutions may degrade quickly over time. Solutions should be prepared fresh daily and kept refrigerated.
- 3. Distilled or deionized water.
 - * See SPECIAL PRECAUTIONS

EQUIPMENT:

- 1. Plastic pump spray bottles (fine spray preferred) with a capacity of at least 125 mL.
- 2. Wipes for sample collection, meeting the specifications of ASTM E1792 [1].
- 3. Safety glasses or goggles.
- Gloves, vinyl disposable or powderless latex gloves.
- 5. Scintillation vials, plastic screw top containers or plastic centrifuge tubes.

NOTE: Commercial kits are available.

SPECIAL PRECAUTIONS: If 1% nitric acid is used, use common sense safety precautions when handling the solution (e.g., wear vinyl gloves, safety glasses/goggles, and avoid contact with clothing and exposed skin).

SAMPLING:

Dermal surfaces (e.g., hands):

- 1. For workplace dermal sampling, tear open a wipe packet and without touching the wipe, offer the wipe to the employee to be evaluated.
- 2. Instruct the employee to remove the wipe from the packet, and unfold it.
- 3. Instruct the person to wipe the palms first, then the top surfaces of both sides of his or her hands (using normal hand washing pressure). Tell the employee to wipe using the same side of the wipe, and to continue wiping his/her hands for a period of not less than 30 seconds.

Other (non-dermal) surfaces:

- 4. Obtain wipe samples in accordance with ASTM E1728 [6], or use the following modification.
 - a. With gloved hands, open a wipe packet and unfold the wipe.
 - b. Wipe a delineated (known) area of the surface, being sure to cover the entire area thoroughly. Wipe across the surface using repeated horizontal motions.
 - c. Then wipe the same surface again using the same side of the wipe for sampling, and wipe at a right angle to the first wiping motion.
 - d. Don a new pair of clean gloves for successive wipe samples.

SAMPLE PREPARATION:

- NOTE: The following procedure is written for sodium rhodizonate, and can be amended using potassium rhodizonate. Also, commercial spot test kits may be used to test for lead on wipe samples in lieu of the procedure described below, but skin contact with the reagents should be avoided.
- 5. Prepare a solution of rhodizonic acid using the sodium rhodizonate powder. Dissolve a pre-weighed portion of 0.135 grams (135 mg) in 105 cc of very cold water (~2 °C). This is the indicator solution. The indicator solution should turn an orange color when mixed.
- 6. Place a quantity of the indicator solution into one of the spray bottles, label the bottle: #2 or lead indicator solution. Refrigerate the remaining solution.
- 7. Fill another spray bottle with household vinegar or make up a 1% (v/v) nitric acid solution, label that bottle #1 or lead extraction solution.
 - NOTE 1: The indicator solution will remain active for 2-3 days (if kept cold). After a time the indicator solution will begin to turn color (pale yellow) and lose viability, at which time a new solution

must be mixed. A new indicator solution should be prepared at least daily, especially if not refrigerated at a very low temperature (approaching 0 $^{\circ}$ C). The lead disclosing solution should be kept cold where possible (e.g., recommend to use a small cooler for field storage of indicator solution).

NOTE 2: Alternative solutions and test indicators may be used, and this can alter the sensitivity and selectivity of the test for lead.

- 8. Place sample (wipe) on clean surface.
- 9. Spray with 3 pumps of bottle #1 (vinegar) extraction solution.
- 10. Spray wipe with 2 pumps of bottle #2 (indicator solution).
- 11. If sample turns red, lead is present. If no color change, sample is considered negative. NOTE 3: For quantitative analysis, see Method 7300 for ICP.

EVALUATION OF METHOD:

The method has been evaluated preliminarily using ASTM E1792 [1] wipes spiked with certified reference materials (CRMs), and has been found to give a positive response for at least ten micrograms of lead per wipe. The method has also been subjected to limited field testing, and shows a positive response for at least a few tens of micrograms of lead per wipe. Extremely heavy soiling on the wipe could interfere with visualization of the red color change due to darkening of the wipe, but the pink or red hues should still be visible around the area of the heaviest soiling, provided lead is present. Difficult matrices (e.g., dust wipes containing paint chips) may require leaching in dilute nitric acid before spot testing.

Persons with color blindness may not be able to detect the pink or red hues characteristic of this spot test method.

REFERENCES:

- [1] ASTM [2002]. ASTM E1792-02, Standard specification for wipe sampling materials for lead in surface dust. In: Annual Book of ASTM Standards, Vol. 04.11. West Conshohocken, PA: American Society for Testing and Materials.
- [2] Esswein EJ, Boeniger M, Ashley K [2001]. Handwipe disclosing method for the presence of lead. U.S. Patent No. 6,248,593.
- [3] Feigel F, Anger V [1972]. Spot Tests in Inorganic Analysis. Amsterdam: Elsevier, pp. 282-287, 564-566, 569
- [4] Ashley K, Fischbach TJ, Song R [1996]. Evaluation of a chemical spot test kit for the detection of airborne particulate lead in the workplace. Am. Ind. Hyg. Assoc. J. 57:161-165.
- [5] Song R, Schlecht PC, Ashley K [2001]. Field screening test methods Performance criteria and performance characteristics. J. Hazard. Mater. 83:29-39.
- [6] ASTM [2002]. ASTM E 1728-02, Standard practice for field collection of settled dust samples using wipe sampling methods for lead determination. In: Annual Book of ASTM Standards, Vol. 04.11. West Conshohocken, PA: American Society for Testing and Materials.

METHOD WRITTEN BY:

Eric J. Esswein, NIOSH/DSHEFS, and Kevin Ashley, NIOSH/DART.