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Standard Practice for Use of Qualitative Chemical Spot Test Kits for Detection of Lead in Dry Paint Films[1]

This standard is issued under the fixed designation E1753; 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 practice covers the use of commercial spot test kits based on either sulfide or rhodizonate for the qualitative determination of the presence of lead in dry paint films.

1.2 This practice may also be used as a qualitative procedure for other dry coating films such as varnishes.

1.3 This practice provides a list of the advantages and limitations of chemical spot test kits based on sulfide and rhodizonate to allow the user to choose the appropriate spot test for a given circumstance.

1.4 This practice contains notes which are explanatory and not part of mandatory requirements.

1.5 Methods described in this practice may not meet or be allowed by requirements or regulations established by local authorities having jurisdiction. It is the responsibility of the user of this standard to comply with all such requirements and regulations.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

E1605 Terminology Relating to Lead in Buildings

[1] This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.12 on Sampling and ~~Analysis,~~Analysis of ~~Lead,~~Lead for Exposure and Risk Assessment.

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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. Terminology

3.1 For definitions of terms relating to this practice that do not appear here, refer to ~~Terminology~~Terminologies D1356 and E1605.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *core sample*—a fragment of the entire dry paint film removed from the substrate with a coring tool which is designed to remove a specified area (for example, a square centimetre) of dry paint film.

3.2.2 *negative screen*—a spot test for which a negative result indicates a low probability of lead being present in the test specimen above a predetermined level; for example, a ~~regulated federal or state abatement~~ regulatory action level.

3.2.3 *negative test*—the absence of the characteristic color change within a specified time limit, usually within a few minutes.

3.2.4 *paint chip sample*—a fragment of a dry paint film removed from the substrate.

3.2.5 *positive test*—the observation of the characteristic color change within a specified time limit, usually within a few minutes, although specific procedures for some test kits include observing the characteristic color change after an overnight waiting period.

3.2.6 *rhodizonate spot test method—for lead detection*, the use of a dilute solution of rhodizonate ion to test a painted surface or paint chip for the qualitative presence of lead **(1)**.[3]

3.2.6.1 *Discussion—*

A characteristic color change of the reagent from yellow/orange to pink or red indicates the presence of lead above the level of detection of the test kit.

3.2.7 *sulfide spot test method—for lead detection*, the use of a dilute solution of sulfide ion to test a painted surface or paint chip for the qualitative presence of lead **(2)**.

3.2.7.1 *Discussion—*

A characteristic color change of the reagent from colorless to grey or black indicates the presence of lead above the level of detection of the spot test.

4. Summary of Practice

4.1 A dry paint film sample (a painted surface, paint chip, ground paint powder, or core sample) is tested for lead qualitatively through the use of a spot test. Spot tests kits are based on the reaction of Lead II (Pb^{2+}) ion with either sulfide ion (S^{2-}) or rhodizonate ion [$C_6O_6^{2-}$], resulting in the characteristic color change (See 3.2.6 and 3.2.7).

4.1.1 Prior to performing the spot test, the dry paint film surface is first cleaned. Except for surface tests, the film is then prepared by either cutting a notch or an angular cut *in situ* or by removing a paint chip or core sample.

4.1.2 The test is performed by applying the spot test reagents (directly or with the use of an adsorbent applicator) to the prepared dry paint sample and observing the characteristic color change after a specified time, usually within a few minutes.

4.1.3 An interpretation of the presence or absence of lead in the dry paint sample is made based on the observation of the presence or absence of the characteristic color change.

5. Significance and Use

5.1 This technique is applicable to dry paint films and varnishes in a variety of forms including the intact dry paint film surface, a notched or other angular cut surface that exposes a cross section of all paint layers, a paint chip, and ground paint film.

5.2 The response of the spot test method varies depending on the extractability of lead from a coating matrix, which may differ depending on the test kit used, the coating type tested, and the type of lead pigment **(3)**.

[3] The boldface numbers in parentheses refer to a list of references at the end of this practice.

5.3 In some situations, metals and other chemical species interfere with the spot tests causing false negative or false positive results (see Section 8).

5.4 A spot test result may be used as a negative screen for the presence of lead in paints and varnishes provided the response of the test kit is sensitive to detecting lead reliably at a given predetermined level, for example, a ~~federal or state regulated abatement~~ regulatory action level **(4)**.

5.5 This practice may be used in conjunction with quantitative and semi-quantitative analytical methods for lead such as ~~portable X-ray fluorescence,~~ anodic stripping ~~voltammetry,~~voltammetry or ~~fixed-site~~spectroscopic laboratory analysis of paint chip ~~samples.~~samples, or portable X-ray fluorescence testing of in situ paint films.

5.6 Colorblind individuals (protanomalous viewers) who are deficient in viewing red colors may have difficulty in discerning the pink or red color of a positive rhodizonate test.

6. Apparatus and Materials

6.1 *For Sulfide Spot Tests:*

6.1.1 *Sulfide Based Spot Test Kit* (usually consists of a ~~5~~5 % to 8 % solution of sodium sulfide in a dropper bottle).

6.1.2 *Disposable Plastic or Latex Gloves*.

6.2 *For Rhodizonate Spot Tests:*

6.2.1 *Rhodizonate Based Spot Test Kit* (usually consists of rhodizonate reagent and an acid extraction solution).

6.2.2 *Absorbent Applicators,* (that is, swabs, filter papers) for applying spot test reagents or for extracting and collecting the lead from the painted ~~surface. Absorbent applicators (that is, swabs, filter paper)~~ surface, which may or may not be provided with a purchased test kit.

6.3 *For Both Sulfide and Rhodizonate Spot Tests:*

6.3.1 *Non-Abrasive Cleaning Solution*.

6.3.2 *Towels, Towelette, or Sponge*.

6.3.3 *Cutting Tool,* used to cut into the dry paint film. A cutting tool may or may not be provided with the kit. Acceptable cutting tools include a clean cutting knife with a fine, sharp edge, razor knife, thin scalpel blade, or coring tool.

6.3.4 *Mortar and Pestle,* for grinding paint chip sample, if necessary.

6.3.5 *Magnifying Glass* (at least 4× power).

6.3.6 *Flashlight,* to examine color change under incandescent white light in dimly lit areas.

7. Reagents

7.1 *Reagents as Provided by the Spot Test Kit*—Reagents and materials kept beyond the preparer's expiration date or recommended shelf life shall be discarded. Store spot test kits at room temperature away from direct sunlight or room light. Freshly prepared rhodizonate reagents require storage in a refrigerator to retard the rate of hydrolysis of the rhodizonate dye.

7.2 Dispose of reagents according to applicable regulations promulgated by authorities having jurisdiction.

8. Advantages and Disadvantages of Different Chemical Spot Tests

8.1 *Sulfide Test*—A clear solution of sodium sulfide reacts with lead *in situ* or on paint chips to produce a grey or black color (lead sulfide, PbS).

8.1.1 *Sulfide Test Advantages:*

8.1.1.1 Sulfide-based tests are rapid, easy, and relatively inexpensive to use.

8.1.1.2 Sodium sulfide reacts with most lead containing pigments in paint, including lead chromate pigments, within ~~1 or~~ 2 min.

8.1.2 *Sulfide Test Disadvantages:*

8.1.2.1 Sulfide solutions emit a toxic, potentially hazardous gas (H_2S) which has an unpleasant odor (rotten eggs). The generation of hazardous levels of H_2S in the field, however, can be minimized by good ventilation, by using low concentrations of sodium sulfide (not to exceed ~~5~~5 % to 8 %), by restricting the volume of sodium sulfide solution used per test to a drop, and by carrying only small amounts of sodium sulfide solutions (30 mL or less). Also, since acid conditions increase the release of H_2S gas, do not use a strong acid or an acidic solution to clean the test location either before or after the sulfide test is performed. The user is cautioned to minimize breathing in the H_2S fumes. In addition, since sulfide solutions are alkaline, it is recommended that the user consider wearing gloves and eye protection. Sulfide solutions are poisonous and are to be kept out of the reach of children.

8.1.2.2 Sulfide based tests are not specific for lead. In addition to lead, sulfide ion reacts with several other metal ions to give a black color including iron, nickel, cobalt, copper, mercury, and molybdenum (**1**, **5**). Except for iron, the concentration of these metals in paint is usually less than 1 %, which is too low to be detected by a ~~5~~5 % to 8 % solution of sodium sulfide (**2**). Iron-containing pigments, oxides, and iron blue are found in paints. Oxides are used primarily in exterior paints to provide deep earth-toned colors. Although sodium sulfide solution does not cause a color change with all iron oxide pigments, positive sulfide spot test results on deep earth-toned or blue colored paints are to be considered suspect. In these cases, the use of other test procedures for lead is recommended.

8.1.2.3 Testing paint directly on metal surfaces composed of iron, copper, or nickel (for example, pipes and radiators) is discouraged as it may lead to false positive results. An immediate dark color is formed on the surface of some treated steels, for example, phosphate treatment.

8.1.2.4 Metals other than lead and iron found in large quantities in paint include zinc, titanium, and barium, but none of these metals produces a black color with sulfide (**2**). Zinc sulfides are colorless; titanium sulfides can be red or even grey, and barium sulfides are yellow/green (**1**). However, these sulfides are rarely observed when testing paint with sodium sulfide spot tests because of the low solubility of the metal species in the sodium sulfide solution.

8.1.2.5 Sulfide can react with some non-lead containing paints used today to produce a grey color; however, most non-lead containing paints do not react with the ~~5~~5 % to 8 % solutions of sodium sulfide to give a grey color.

8.1.2.6 It is difficult to discern a black color against dark paints such as black, brown, dark green, or dark blue. For dark paints other than black, the use of a magnifying glass, white tissue, or commercial cotton swab to take up the black color may aid in the determination of the test result. Results observed at the test location shall be compared to a similarly prepared control test location prepared a few centimetres away from the test location and wetted with water. If there is no difference in appearance between the control test location and the test location, the test result is considered negative. Alternatively, for black paint and other dark colored paints, the use of a rhodizonate based test kit that uses absorbent applicators or other acceptable procedures is recommended.

8.1.2.7 Once exposed to air, ~~5~~5 % to 8 % solutions of sodium sulfide have a limited shelf life. Do not use sodium sulfide solutions beyond their expiration date.

NOTE 1—Shelf life depends upon storage, temperature, light conditions, and other use factors.

8.1.2.8 If a sulfide spot test that is first conducted on a notch cut into the test surface yields a negative results, re-conduct the test on a chip removed from the surface. Research has indicated that some sulfide test kits have increased sensitivity when used on a chip removed from the surface than on a notch cut into the surface (**4**).

8.2 *Rhodizonate Test*—A yellow/orange solution of rhodizonate reacts with lead to produce a pink or red complex (PbC_6O_6) under acid conditions.

8.2.1 *Rhodizonate Test Advantages:*

8.2.1.1 Rhodizonate is more specific for lead in paint than sulfide. Under acid conditions, only lead reacts with the yellow/orange rhodizonate solution to give a pink to red color (**1**, **6**).

NOTE 2—If the pH of the rhodizonate reagent is neutral or basic, the characteristic color change for lead is from yellow/orange to blue or violet. However, under neutral or basic conditions, ~~a~~the blue or violet color change is not specific for lead (**1**, **6**).

8.2.1.2 Rhodizonate based tests are rapid, easy, and relatively inexpensive to use.

8.2.2 *Rhodizonate Disadvantages:*

8.2.2.1 Rhodizonate based spot tests shall not be used on red or pink paint that rubs off or bleeds color. However, rhodizonate based spot tests that use absorbent applicators to apply the test reagents may be used on red or pink paint that does not bleed color.

NOTE 3—It may be determined if red or pink paint does bleed by dipping a cotton swab into the leaching solution supplied by the test kit or, if none is provided, dipping a cotton swab into household vinegar and rubbing the cotton swab over the surface of the red or pink paint. If a red or pink color is observed on the cotton swab, rhodizonate based tests should not be used to test these painted surfaces. Instead, it is recommended that a sodium sulfide spot test or other acceptable test method be used to determine the presence of lead.

8.2.2.2 Rhodizonate ion will react with barium ion to form an orange color. Barium sulfate was sometimes added to household paints as an extender, but because of the insolubility of barium sulfate, barium ion and, hence, the orange color, is only rarely detected. However, since it is possible for a strong orange color to mask a weak pink color due to lead, it is recommended that paints providing an orange color with rhodizonate be further tested with a sodium sulfide test kit or other acceptable lead detection method (for example, laboratory analysis of a paint chip sample).

NOTE 4—A rhodizonate test can be performed at a notched or angular cut test location on wallboard (sheet rock) or on a paint chip removed from the wallboard (sheet rock), etc., provided any debris and dust generated while cutting a notch (or angular cut) is collected from the notched (or angular cut) test location or extricated paint chip before the test is conducted. Debris and dust from the substrate may be collected and removed from the notch (or angular cut) using a wetted towel, towelette, or sponge, or by washing it off the paint chip. See 10.4.3.

8.2.2.3 The development of a pink/red color at the test location or on the adsorbent applicator ~~30~~30 min to 60 min or even ~~18~~18 h to 24 h later indicates the presence of lead chromate pigments. Rhodizonate reacts slowly with lead chromate pigmented paint (**7**). Paint containing lead chromate pigments (usually bright colors such as red, orange, yellow, and some greens) are found primarily in marine and industrial settings. Nevertheless, lead chromate-containing pigments as well as other colored pigments were used in consumer paints prior to 1972 to provide colors ranging from blue-green across the spectrum to red. Since lead chromate is insoluble in water-based media, the lead is only slowly extracted from these paints by the aqueous media provided by most field test kits. If lead chromate is suspected (due to testing of red, orange, yellow, or green household paints), a test location or the adsorbent applicator, or both, which gives an initial negative result should be reexamined ~~30~~30 min to 60 min later or even up to ~~18~~18 h to 24 h later for the formation of the characteristic pink/red color. Alternatively, an additional test can be performed in which a paint chip is keep in contact with the rhodizonate reagent for up to ~~18~~18 h to 24 h after which time it is examined for the characteristic color change.

NOTE 5—Since it is unlikely that light colored paints contain lead chromate pigments, a negative result for a light colored paint can be interpreted as a negative result within a few minutes following a test.

8.2.2.4 Colorblind individuals (protanomalous viewers) who are deficient in viewing red colors may have difficulty in discerning the pink or red color of a positive rhodizonate test.

8.2.2.5 Once mixed with water or acid solutions, rhodizonate has a limited shelf life. In water, rhodizonate slowly deteriorates and may remain reactive towards lead a few hours to a few weeks depending on its concentration. In acid solution, the activity of rhodizonate degrades within a few minutes. When provided, follow test kit instructions for mixing and time limitations for test performance.