

Standard Practice for Use of Qualitative Chemical Spot Test Kits for Detection of Lead in Dry Paint Films¹

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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 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 and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

E1605 Terminology Relating to Lead in Buildings

3. Terminology

- 3.1 For definitions of terms relating to this practice that do not appear here, refer to Terminology 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.
- ¹ This practice is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Lead Hazards Associated with Buildings.
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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.

- 3.2.2 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 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).
- 3.2.6.1 *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.

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



- 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).
- 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 action level (4).
- 5.5 This practice may be used in conjunction with quantitative analytical methods for lead such as portable X-ray fluorescence, anodic stripping voltammetry, or fixed-site laboratory analysis of paint chip samples.
- 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 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 extraction solution).
- 6.2.2 Absorbent Applicators, for applying spot test reagents or for extracting and collecting the lead from the painted surface. Absorbent applicators (that is, swabs, filter paper) 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 4x power).
- 6.3.6 *Flashlight*, to examine color change under incandescent 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 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 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 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 to 8 % solutions of sodium sulfide have a limited shelf life. Do not use sodium sulfide solutions beyond their expiration date.

 $\mbox{\it Note}\mbox{\it 1}\mbox{\it --}\mbox{\it Shelf}$ 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 (PbC₆O₆) 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 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—To determine if red or pink paint bleeds, dip a cotton swab into the leaching solution supplied by the test kit or, if none is provided, dip the cotton swab into household vinegar and rub the cotton swab over the surface of the red paint. If a red or pink color is observed on the cotton swab, rhodizonate based tests shall not be used to test these painted surfaces. Instead, the use of a sodium sulfide spot test or other acceptable test method to determine the presence of lead is recommended.
- 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).
- 8.2.2.3 Sulfates can interfere with the pink color development of lead rhodizonate and cause a false negative result. Substrate materials such as plaster, gypsum wallboard (sheet rock), some cement blocks, and some bricks may contain soluble sulfates.

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 dust generated while cutting a notch (or angular cut) is brushed from the notched (or angular cut) test location or extricated paint chip before the test is conducted. Dust from the substrate may be removed by brushing or blowing it out of the notch (or angular cut) or by washing it off the paint chip. Always confirm a negative result by touching the reagent soaked adsorbent applicator to a lead containing positive control.

8.2.2.4 The development of a pink/red color at the test location or on the adsorbent applicator 30 to 60 min or even 18 to 24 h later indicates the presence of lead chromate pigments. Rhodizonate reacts slowly with lead chromate pigmented paint. 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 to 60 min later or even up to 18 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 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.

8.2.2.5 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.

8.2.2.6 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.

9. Evaluation of Performance Characteristics

- 9.1 Use of spot test kits as negative screens.
- 9.1.1 A criterion for use of spot test kits as negative screens is: Upon evaluation of the test kit performance characteristics, the probability of a negative response (with 95 % confidence) at the predetermined lead (for example, a regulated abatement action level promulgated by authorities having jurisdiction) level is $\leq 5\%$ (4).
- 9.1.2 Equivalently, the lead level at which there is a 95 % probability of a positive response (with 95 % confidence) is less than or equal to the predetermined lead level.

10. Procedure

- 10.1 Quality Control of Test Kit Reagents:
- 10.1.1 For kits with bulk reagents (for example, large quantities in bottles), perform a test on a positive lead control (for example, lead containing positive control supplied with the kit or a sample of dry leaded paint film previously demonstrated to contain a hazardous level of lead near the level of interest) to assure reagents are active before a series of tests is initiated. If the test on the positive control does not result in the expected color change within the specified time limit, the solution shall not be used for testing.
- 10.1.2 For kits whose reagents are packaged for a single use or test, a quality control test shall be performed following each negative result. Following a negative result, apply a drop of the same reagent(s) or reagent soaked adsorbent applicator, or both, used in the test to a positive lead control (for example, a lead containing positive control supplied with the kit or a sample of dry leaded paint film previously demonstrated to contain a hazardous level of lead near the level of interest) in order to assure the activity and sensitivity of the reagents used and, hence, to verify the negative result. If the expected color change is not obtained on the positive control, the test result shall not be considered valid.

10.2 Preparation of Test Locations:

- 10.2.1 Clean a painted test location with a nonabrasive cleaning solution to remove dirt and grease from the surface. Thoroughly rinse with water to remove the cleaning solution and dry with a paper towel or clean cloth. Examine the surface to ensure all visible dirt and grease have been removed.
- 10.2.2 When the test location is the top surface of the dry paint film, the surface shall be lightly sanded or otherwise scratched before conducting the spot test.
- Note 6—Occasionally, information about the lead content of the outermost surface is desired. However, it is emphasized that testing the top surface of the dry paint film does not provide information about the total lead content of the layers of paint.
- 10.2.3 For Testing All Layers of Paint In Situ in the Field—Using a cutting tool, cut a small notch or angular cut

about 6 mm long into the painted surface. Cut diagonally through all the layers of paint down to the substrate in order to expose a cross section of all layers of paint.

- Note 7—The chemistry of the outermost paint film often provides a protective barrier through which the water-based solutions of spot tests and a leaded species cannot quickly penetrate. Thus, a cut shall be made through the layer or layers of paint to expose the interior paint matrices so that the Lead II (Pb²⁺) ion can be extracted from the lead pigments and dryers and react with the spot test chemicals.
- 10.2.4 For testing a paint chip or core sample, carefully remove the paint chip or core sample with the appropriate cutting tool, being careful to include all layers of paint but to exclude any substrate. Alternatively, in the case of actively peeling paint, a sizable paint chip is removed easily by hand or with minimal use of cutting tools. This paint chip is suitable for testing provided all layers of paint are included in the collected chip.
- 10.3 Performance of the Test—A chemical spot test is performed in the field on either the notch or angular cut prepared at the test location or on a paint chip or core sample removed from the test location. Adsorbent applicators used to perform spot tests shall be used only once.
- 10.3.1 *In Situ in a Notch or Angular Cut*—Apply the spot test reagents to all layers exposed in the notch or angular cut prepared in situ. In general, apply one or two drops of the spot test reagent or extraction solution to the notched or angular cut test location. Look for the appearance of the characteristic color change in the notch (or angular cut) or on the adsorbent applicator, or both, used to perform the test. If the test kit is purchased from a commercial source, the user shall follow the manufacturer's detailed instructions for use.
- 10.3.2 On a Paint Chip or Core Sample Collected in the Field—To prepare the paint chip or core sample for the test, either (1) crush the paint chip or core sample and place in a small lead-free container or (2) cut the paint chip to expose a fresh cross section of all layers of paint. Apply the spot test directly on the crushed paint sample or along the fresh cross section of the paint chip or core sample. Alternatively, a mortar and pestle is suitable for grinding a paint chip or core sample to a fine powder prior to testing. When a sufficiently large paint chip is obtained, the top and bottom surfaces may be tested individually.
- 10.3.3 Examine the test location, the paint chip or the absorbent applicator, or both, used to perform the test for a characteristic color change. Usually the color change occurs within a few seconds. If an immediate color change is not observed, wait for 1 to 2 min before making a determination. (See also 8.2.2.4.)
- 10.3.4 Testing shall not be conducted under conditions that cause freezing of the test solutions before chemical reactions can occur.

Note 8—When temperatures are well below freezing, the few drops of solution may freeze upon contact with the surface. Also, diffusion of test solution and lead ions through a paint film slow, as the temperature decreases.

10.4 Cleanup:

10.4.1 Clean the in situ test location with a household cleaning solution to remove the test reagents and any associated color. Also, when cutting the notch (or angular cut) into

the painted surface or when removing the paint chip, clean up any small paint chips with a paper towel moistened with a household cleaning solution.

10.4.2 Clean the cutting or coring tool used to cut the notch (or angular cut) or core sample before moving on to the next test location. Use a paper or cloth towel moistened with a household cleaning solution to clean the blade of a cutting tool. Use the cleaning brush provided with the coring device to clean the inside of the coring tool. If no brush is available, wash both the inside and outside of the coring device thoroughly and dry before using again.

10.4.3 Dispose of the used materials according to applicable regulations promulgated by authorities having jurisdiction.

11. Interpretation of Results

11.1 Test specimens shall be examined for a characteristic color change under daylight or incandescent light usually within a few seconds but sometimes up to 1 to 2 min later or longer. (See also 8.2.2.4.)

11.1.1 If the characteristic color change is observed at the test location or on the absorbent applicator, or both, used to perform the test, then the test is positive. For sodium sulfide based test kits, the characteristic color change of the reagent is

from colorless to grey or black. For rhodizonate based test kits, the characteristic color change of the reagent is from yellow/ orange to pink or red under low pH (acidic) conditions (see Note 1). Usually the intensity of the color is an indication of the lead concentration; that is, the greater the intensity of the color, the higher the lead concentration.

11.1.2 If no color change or a change to a noncharacteristic color is observed at the test location or on the absorbent applicator used to perform the test, then the test is negative.

Note 9—If a color other than the characteristic color is observed, the possibility exists that the noncharacteristic color change may be masking or interfering with a weak reaction with lead. In such a case, retesting the dry paint film with an alternative chemical spot test or other test method is recommended.

11.2 If no characteristic color change occurs using a spot test kit that has been evaluated for use as a negative screen, the negative result can be interpreted as indicating that lead, if present, is below the predetermined level, for example, a regulated abatement action level promulgated by authorities having jurisdiction.

12. Keywords

12.1 lead; rhodizonate test; spot test; sulfide test; test kit

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