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Standard Test Methods for Penetration of Preservatives in Wood and for Differentiating Between Heartwood and Sapwood[1]

This standard is issued under the fixed designation D 3507; 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 These test methods cover procedures for determining penetration of preservatives in wood in cases where demarcation between the treated and untreated wood is not readily visible. Included are test methods for differentiating the heartwood and the sapwood of wood samples for specific species, and a test method for differentiating the heartwoods between the red oaks and the white oaks.

1.2 The procedures appear in the following order:

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1.3 *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]

D 1193 Specification for Reagent Water

2.2 *Other Standards:*

AWPA Standard A 3-84, Methods for Determining Penetration of Preservatives and Fire Retardants[3]

AWPA Standard M 2-83, for Inspection of Treated Timber Products[3]

AWPA Standard P 9-84, Standards for Solvents and Formulations for Organic Preservative Systems[3]

3. Summary of Test Methods

3.1 A specific chemical solution or powder for each preservative is applied to a wood specimen or cross section of wood to bring out a color that is distinctive for the particular preservative.

3.2 A chemical solution, specific for a species of wood, is applied to a wood specimen or cross section of wood to bring out colors distinctive to sapwood and heartwood.

4. Significance and Use

4.1 Preservatives of the metallic series and oil soluble preservatives are not readily apparent in a cross section of wood either due to similar color to the species of wood or lack of color of the preservative itself. Chemical staining of a treated specimen of wood reveals the presence of the preservative.

4.2 The sapwood and heartwood of Douglas-Fir and the pine species can be differentiated by a chemical stain.

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[1] These test methods are under the jurisdiction of ASTM Committee D07 on Wood and are the direct responsibility of Subcommittee D07.06 on Treatments for Wood Products.

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These test methods are substantially the same as AWPA Standards A 3 and M 2.

[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] Available from American-Wood-Preservatives Assn., P.O. Box 286, Woodstock, MD 21163-0286.

5. Reagents

5.1 *Purity of Reagent*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type I or II.

PENETRATION OF ARSENIC-CONTAINING PRESERVATIVES

6. Apparatus

6.1 *Balance*.

6.2 *Atomizers (Optional)*—3.

6.3 *Medicine Dropper*.

7. Reagents

7.1 *Ammonium Molybdate* ($(NH_4)_6Mo_7O_{24}\cdot 4\ H_2O$).

7.2 *o-Anisidine*.

7.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

7.4 *Hydrochloric Acid* (1.7 %)—Mix 8.5 g of concentrated HCl (sp gr 1.19) with 491.5 g of water.

7.5 *Hydrochloric Acid* (1+1)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 1 volume of water.

7.6 *Solution 1*—Dissolve 3.5 g of ammonium molybdate in 90 mL of water, then add 9 mL of concentrated HCl.

7.7 *Solution 2*—Dissolve 1 g of *o*-anisidine in 99 g of 1.7 % HCl.

7.8 *Solution 3*—Dissolve 30 g of stannous chloride in 100 mL of HCl (1+1).

7.9 *Stannous Chloride* ($SnCl_2\cdot 2\ H_2O$).

8. Procedure

8.1 Apply Solution 1 first, followed by Solution 2 and Solution 3 in that order. Apply the solutions by dropping the solution on the wood with a medicine dropper, by pouring the solutions over the wood, or by spraying the solutions on the wood using a fine mist sprayer. Allow 2 min between application of each solution. Spraying is the preferred procedure.

8.2 After Solution 3 is applied, the areas of the wood specimen that contain arsenic will immediately turn blue. This blue color may tend to fade but may be restored by another application of Solution 3.

PENETRATION OF COPPER-CONTAINING PRESERVATIVES

9. Apparatus

9.1 *Atomizer*.

9.2 *Graduate*, 100-mL.

9.3 *Balance*.

9.4 *Medicine Dropper*.

10. Reagents

10.1 *Chrome Azurol S*.

10.2 *Chrome Azurol S–Sodium Acetate Solution*—Dissolve 0.5 g of chrome azurol S and 5.0 g of sodium acetate in 80 mL of water and dilute to 500 mL.

10.3 *Sodium Acetate*.

11. Procedure

11.1 Spray the solution over split increment cores or freshly cut surfaces of the treated sample. A deep blue color reveals the presence of copper.

PENETRATION OF FLUORIDE-CONTAININGPRESERVATIVES

12. Scope

12.1 The mixed stock solutions will not work satisfactorily after standing 1 h. Therefore, minimum quantities of the mixed stock solutions should be made up to avoid wastage.

12.2 The surface of the wood should be well dried before testing. Results are not reliable on wet surfaces.

13. Apparatus

13.1 *Atomizer*.

13.2 *Balance*.

13.3 *Medicine Dropper*.

14. Reagents

14.1 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

14.2 *Sodium Alizarin Sulfonate*.

14.3 *Solution 1*—Mix 0.5 g of sodium alizarin sulfonate in 100.0 g of water.

14.4 *Solution 2*—Mix 0.5 g of zirconyl chloride in 30.0 g of HCl and 70.0 g of water.

14.5 *Solution, Mixed*—Mix equal quantities of Solution 1 and Solution 2 by adding Solution 1 to 2 (red to white); otherwise, a proper reaction will not take place.

14.6 *Zirconyl Chloride*. [5]

15. Procedure

15.1 Spray or drop the mixed solution on the surface of the split increment core or cross section of wood to be tested. The atomizer should be held about 75 mm (3 in.) from the surface under test and the spraying continued until the surface can be seen to be wetted. No running of the condensed spray must

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[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[5] Fisher Z-80 or an equivalent have been found suitable and is available from Fisher Scientific Co., Pittsburgh, PA.

take place. As the wood dries, the red color of the sprayed surface will change to yellow where fluoride is present. The reaction is somewhat slow but will be developed fully after 15 min. An infrared lamp or laboratory oven set at no higher than 105°C will help accelerate the reaction. Make a second application of spray when the surface has dried again in order to obtain clearer definition.

PENETRATION OF PENTACHLOROPHENOL USING 4,4′-*bis*-DIMETHYLAMINO-TRIPHENYLMETHANE (DMTM)

16. Scope

16.1 The test may become less selective as a solution of DMTM in acetone ages. Therefore, only freshly prepared solutions should be used.

16.2 The method is *not* suitable for determining penetration in wood treated with pentachlorophenol dissolved in conventional Type A hydrocarbon solvents (see AWPA Standard P 9).

16.3 Since the solution is basically a stain, any unused portion should be disposed of with care. If kept in the laboratory, the solution will dry, becoming a greenish dust which may stain clothing and equipment.

17. Apparatus

17.1 *Balance*, accurate to 0.1 g.
17.2 *Eye Dropper*.
17.3 *Erlenmeyer Flask*, 125-mL.
17.4 *Graduated Cylinder*, 100-mL.
17.5 *Infrared Lamp*, 115 to 125-V, 250-W.[6]
17.6 *Spatula*.
17.7 *Support for Lamp*.

18. Reagents

18.1 *Acetone*.
18.2 *4,4′-bis-Dimethylamino-Triphenylmethane* (DMTM).
18.3 *Solution*—The stain consists of 0.5 g of DMTM dissolved in 75 mL of acetone. In making the solution, the solid dye is weighed on the balance into a 125-mL Erlenmeyer flask. Acetone is poured into the flask and the dye is dissolved by swirling gently. Solution should be made up fresh each day unless stored under refrigeration.

19. Procedure

19.1 With an eyedropper (Note 1), place several drops of freshly prepared stain on a cross section or the end grain of an increment core. Usually 4 to 6 drops per 25.4-mm (1.0-in.) length of core is sufficient to soak the surface thoroughly.

NOTE 1—An eye dropper is recommended rather than dipping the sample in stain solution. If samples are dipped, there is a possibility of solution contamination by pentachlorophenol from one sample to the next. This will not occur when a dropper is used.

19.2 Allow the surface of the samples to dry for 10 to 15 s; then place them under the infrared lamp at 75 to 100 mm (3 to 4 in.) distance beneath the lamp. The test may be conducted, however, without the use of a lamp.

19.3 After an exposure of 2 to 3 min under the lamp, maximum reaction will have taken place. If no lamp is used, maximum reaction will take place in 5 to 10 min.

19.4 The presence of pentachlorophenol is indicated by the development of a green color on the surface of the wood.

NOTE 2—The reaction is positive when freshly prepared solutions are used. When it is necessary to use old solutions (24 h or older), a faint greenish cast appears even on the surface of untreated wood.

20. Precision

20.1 This test method will detect quantities of 0.8 kg/m^3 (0.05 lb/ft^3) readily, and frequently will detect 0.16 kg/m^3 (0.01 lb/ft^3).

PENETRATION OF PENTACHLOROPHENOL USING A SILVER-COPPER COMPLEX KNOWN AS "PENTA-CHECK"

21. Scope

21.1 This test method uses a blend of copper and silver ions, and reddish colored copper pentachlorophenate is formed where pentachlorophenol is present. Untreated earlywood (springwood) and heartwood of southern pine and ponderosa pine are colored green. Untreated Douglas fir is colored yellowish to olive.

21.2 The exact mechanism of the silver ions is not known but in their presence a redder color is formed than with copper alone.

21.3 This test method is recommended for determining the penetration in wood of pentachlorophenol dissolved in light-colored hydrocarbon solvents of all conventional types, that is, Types A, B, C, and D (see AWPA Standard P 9). Excessively dark treatments tend to obscure the color. The penetration of pentachlorophenol in dark-colored Type A hydrocarbon solvent should be judged by visual examination without staining. This method is *not* suitable for determining penetration in the heartwood of redwood (*Sequoia sempervirens*).

22. Apparatus

22.1 *Atomizer*.
22.2 *Balance*.
22.3 *Medicine Dropper*.

23. Reagents

23.1 *Cupric Acetate*.
23.2 *Isopropyl Alcohol* (99 %), commercial grade.
23.3 *Silver Acetate*.
23.4 *Solution 1*—Stir 4.0 g of cupric acetate in 100.0 g of water until dissolved, and then add 0.5 g of Tergitol XD. The XD is semisolid at normal temperatures and it is best to heat this until liquid and then add to the blend with mixing until the solution clears.
23.5 *Solution 2*—Stir 0.4 g of silver acetate in 100.0 g of water until dissolved.
23.6 *Solution, Mixed*—Prepare the stain solution in the following preparations:

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[6] Westinghouse clean glass heat lamp or an equivalent have been found satisfactory.

	Volume, %
Solution 1	25
Solution 2	25
Water	25
Isopropyl alcohol	25
	100

23.7 *Tergitol XD.*[7]

24. Procedure

24.1 Apply the mixed solution to cross sections of wood or split increment cores and observe the rapid formation of red copper pentachlorophenate. Application may be by small hand spray or by medicine dropper.

PENETRATION OF SOLVENT USED WITH OIL-SOLUBLE PRESERVATIVES

25. Scope

25.1 This test method is designed exclusively for determining penetration of pentachlorophenol dissolved in light-colored hydrocarbon solvent Type A (see AWPA Standard P 9). The penetration of pentachlorophenol in dark-colored Type A hydrocarbon solvent should be judged by visual examination without staining.

26. Apparatus

26.1 *Atomizer or Small Brush*.
26.2 *Balance*.
26.3 *Mortar and Pestle*.

27. Reagents

27.1 *Red Dyestuff Powder.*[8]
27.2 *Speedex Filter-Aid Powder or Calcium Carbonate*.
27.3 *Mixed Powders*—Weigh 20 parts of Speedex filter-aid powder, or calcium carbonate, and 1 part of red dyestuff powder; then grind together with the mortar and pestle.

28. Procedure

28.1 Use freshly made wood cross sections or fresh split increment cores.

28.2 Apply very light (invisible) coatings of the mixed powders to the wood surface with the small brush or atomizer. The portions containing the hydrocarbon solvent in which the pentachlorophenol is carried will turn bright red within 5 min. Untreated portions of the wood sample will retain their normal color.

28.3 Penetration must be noted immediately as the samples do not retain their color. The hydrocarbon solvent tends to bleed along freshly exposed surfaces; however, the line of demarcation between treated and untreated wood is very sharp for the first few minutes.

NOTE 3—**Caution:** Keep the brush and the powder that is to be used for test dry and free of oils and hydrocarbon solvent.

PENETRATION OF ZINC-CONTAINING PRESERVATIVES

29. Scope

29.1 Starch indicator solution is subject to decomposition and, therefore, should not be used longer than 3 days, unless a stabilized starch solution is used.

30. Apparatus

30.1 *Atomizer*.
30.2 *Balance*.
30.3 *Medicine Dropper*.

31. Reagents

31.1 *Potassium Ferricyanide*.
31.2 *Potassium Iodide*.
31.3 *Starch*, soluble.
31.4 *Solution*—Dissolve 1 g of potassium ferricyanide in 100 mL of water and dissolve 1 g of potassium iodide in 100 mL of water. Make a paste of 1 g of soluble starch in about 5 mL of water and boil 1 min with constant stirring. Cool. Mix 10 mL of each of the three stock solutions and pour into a clean atomizer or bottle.

32. Procedure

32.1 Spray the solution evenly over the reasonably dry split increment core or cross section of wood. The reaction between the zinc and the indicator solution will cause the treated wood to turn a deep blue instantly, while the untreated part will retain its original color. The method is positive. Should the color fade, repeat the procedure.

DIFFERENTIATING BETWEEN SAPWOOD AND HEARTWOOD IN PINE SPECIES (*Pinus sp.*)

33. Scope

33.1 This test method is applicable to pine species only.

33.2 For maximum shelf-life, both Solution 1 and 2 should be stored in a refrigerator or in a cool, dark location. Under such conditions the storage life will exceed 1 month. The mix of the two solutions can be used over a period of several days, but filtering before use is necessary.

34. Apparatus

34.1 *Atomizer*.
34.2 *Balance*.
34.3 *Small Brush*.
34.4 *Medicine Dropper*.

35. Reagents

35.1 *o-Anisidine Hydrochloride*.
35.2 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).
35.3 *Kodak Photo-Flo or Equivalent*.
35.4 *Sodium Nitrite*, ($NaNO_2$).
35.5 *Solution 1*—Mix 8.5 g of concentrated HCl in water to make up 495.0 g. Add 5.0 g of *o*-anisidine to the HCl water solution and stir until the *o*-anisidine is completely dissolved.

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[7] Tergitol XD has been found suitable and is available from Union Carbide Chemical Co., 270 Park Ave., New York, NY 10017.

[8] Oil Red-O dyestuff available from National Aniline Allied Chemical Co. Specialty Chemical Div., P.O. Box 1087R, Morristown, NJ, and Red N-1700, available from American Cyanamid Co., Organic Chemicals Div., Boundbrook, NJ 08805, have been found suitable.

35.6 *Solution 2*—Mix 50.0 g of sodium nitrite in 450 g of water. Wettability can be improved by adding a few drops of Kodak Photo-Flo, or equivalent, to each 100 mL of mixed indicator.

36. Procedure

36.1 Apply the mixture of equal volumes of the two solutions by brush, atomizer, or medicine dropper. After several minutes the heartwood will develop a bright reddish-orange color while the sapwood will remain a pale orange. Smooth surfaces (for example, split increment cores, or cross sections made with sharp saws) give better results than rough surfaces.

DIFFERENTIATING BETWEEN SAPWOOD AND HEARTWOOD IN DOUGLAS FIR (*Pseudotsuga menziesii*)

37. Scope

37.1 This test method works best on a freshly cut wood surface. It is applicable to both split increment cores or freshly exposed cross sections, and works on both dry and green wood.

38. Apparatus

38.1 *Atomizer*.
38.2 *Balance*.
38.3 *Small Brush*.
38.4 *Medicine Dropper*.

39. Reagents

39.1 *Alizarin Sulfonate Solution*—Prepare a 0.75 weight % water solution.

39.2 *Methyl Alcohol*.

39.3 *Methyl Orange Solution*—Prepare a 0.1 weight % water solution.

NOTE 4—A little alcohol added to either solution will make wet wood dry more rapidly.

40. Procedure

40.1 Regardless of the solution selected, apply by atomizer, brush, or medicine dropper. The methyl orange solution stains the heartwood a reddish color and the sapwood yellowish. The alizarin sulfonate solution stains the heartwood and one or two adjacent annual rings in the sapwood yellow and the sapwood pink or some shade of red. These colors are comparatively permanent.

DIFFERENTIATING BETWEEN SAPWOOD AND HEARTWOOD IN WHITE FIR (*Abies concolor*)

41. Scope

41.1 This test method is applicable to white fir (*Abies concolor*) only.

42. Apparatus

42.1 *Atomizer, Medicine Dropper, or Squeeze Bottle*.

43. Reagents

43.1 *Perchloric Acid Solution* (Note 5) (70 %)—Dilute the 70 % perchloric acid solution with an equal volume of water in glass apparatus only, before use.

NOTE 5—A 5 % water solution of ferric chloride may be used in place of the diluted perchloric acid, in which case the sapwood becomes yellow and the heartwood becomes yellowish red.

44. Procedure

44.1 Apply the solution as a fine spray, or by means of a medicine dropper or squeeze bottle. Heartwood will turn greenish while wetted sapwood will be unchanged in color.

DIFFERENTIATING BETWEEN WOODS OF THE RED OAK AND THE WHITE OAK SPECIES

45. Scope

45.1 This test method is especially applicable for use on oak crossties.

46. Apparatus

46.1 *Atomizer*.
46.2 *Balance*.
46.3 *Small Brush*.
46.4 *Medicine Dropper*.

47. Reagents

47.1 See Section 35.

48. Procedure

48.1 Proceed as directed in Section 36, except for color reaction. The heartwood of the red oaks remains the color of the indicator while the heartwood of the white oaks turns a blackish green.

DETERMINATION OF COPPER IN WOOD USING RUBEANIC ACID

49. Apparatus

49.1 *Atomizers.*
49.2 *Graduate*, 100-mL.
49.3 *Balance.*
49.4 *Medicine Dropper.*

50. Reagents

50.1 *Rubeanic Acid Solution*—Dissolve 0.5 g of rubeanic acid (dithio oxamide) in 100 mL ethanol.

50.2 *Sodium Acetate Solution*—Dissolve 5 g of sodium acetate in 100 mL distilled water.

51. Procedure

51.1 Spray the core or wood section with rubeanic acid solution, then the sodium acetate solution. A dark green color indicates the presence of copper, the untreated wood being colored pale yellow.

52. Comments

52.1 The rubeanic acid has approximately the same sensitivity as Chrome Azurol S, at approximately 25 ppm copper. Chrome Azurol S is the preferred reagent for freshly treated wood because of its stronger reaction. Rubeanic acid is, however, more specific to copper and is less subject to interference reactions. Therefore, it is the preferred reagent for measuring CCA penetration in wood that has been in service for some time in ground contact.

METHOD FOR DETERMINING PENETRATION OF COPPER-CONTAINING PRESERVATIVES

53. Scope

53.1 By using PAN indicator, it is possible to detect the presence of copper in treated wood. The test will readily detect quantities of 0.01 pcf and frequently will detect 0.002 pcf. The test may become less selective as the solution in methanol ages. Therefore, only freshly prepared solutions should be used. The method is not suitable for determining penetration in wood treated with oxine copper (copper 8-quinolinolate) dissolved in darkly colored P-9 heavy solvents.

54. Apparatus and Chemicals

54.1 *Balance*, accurate to 0.1 g.

54.2 *125-mL Flask.*

55. Reagents

55.1 1-(2-pyridylazo)-2-naphthol, indicator grade from Aldrich Chemical, No. 10, 103-6 CAS# 85-85-8.

55.2 *Methanol*, reagent grade.

55.3 Add 0.05 % by weight of the PAN indicator to the methanol. Mix until uniform.

NOTE 6—Prepare and store the indicator solution in an area free from open flames. Follow normal safety procedures for handling volatile alcohols. Solution can be maintained for extended periods under refrigeration.

56. Procedure

56.1 Spray the solution over split borings or freshly cut surfaces of treated wood. A deep red/magneta color reveals the presence of copper. Untreated areas remain orange in color.

NOTE 7—This method will detect copper-8-quinolinolate to 5 µg/cm^2 or 0.002 pcf.

METHOD FOR DETERMINATION OF THE PRESENCE OF HEXAVALENT CHROMIUM IN TREATED WOOD

57. Scope

57.1 When chromotropic acid is complexed with chromium (VI), a pink to purple color is formed. Such visible color does not occur with chromium (III). This method indicates, by the absence of the colored chromotropic acid complex, the presence of 15 ppm or less chromium (VI).

58. Apparatus

58.1 *Filter Paper.*

58.2 *Medicine Dropper.*

58.3 *Balance.*

58.4 *Graduate,* 100-mL.

59. Reagent

59.1 *Chromotropic Acid.*

59.2 *1N Sulfuric Acid.*

59.3 *Solution*—Dissolve 0.5 g of chromotropic acid (4,5-dihydroxy-2,7-naphthalene disulfonic acid) or its disodium salt in 100 mL of 1N (approx, 5 wgt. %) sulfuric acid. This solution should have a shelf life of at least 2 months.

60. Procedure

60.1 Borings for testing should be taken by hand, split and the bit thoroughly rinsed in water between borings. Borings should be allowed to come to room temperature before testing.

60.2 Place the freshly cut boring on a white blotter paper surface, such as filter paper or a white index card. With a medicine dropper, apply several drops of the chromotropic acid solution to wet the core. Usually 5 to 7 drops per one-in. length of core is sufficient for a core still wet from treatment. If the core is dry, it is helpful to apply 2 or 3 drops of the solution carefully and allow the surface of the core to become saturated before applying the remaining drops.

60.3 Allow the reaction to continue for at least 10 min. Then remove the core and observe the colored complex that has leached onto the blotter; if the blotter is not wet, insufficient solution was applied and the test must be repeated. Any pink to purple color on the blotter indicates the incomplete conversion of chromium (VI) to chromium (III).

61. Precision and Bias

61.1 The minimum detection limit of the method is 15 ppm chromium (VI).

PRECISION AND BIAS

62. Precision and Bias

62.1 A precision and bias statement is inappropriate for these test methods.

63. Keywords

63.1 heartwood; penetration; preservatives; sapwood; test