Designation: D 5655 – 95 (Reapproved 2000)

Standard Test Method for Analysis of Copper Dimethyldithiocarbamate (CDDC) Treated Wood by Colorimetry 1[1]

This standard is issued under the fixed designation D 5655; 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 test method covers colorimetric analysis of CDDC in treated wood.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

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:*

D 147 Methods of Testing Bituminous Mastics, Grouts, and Like Mixtures[2]

2.2 *AWPA Standards:*

A10 Methods of Analysis of CCA Treating Solutions and CCA Treated Wood by Colorimetry[3]

M2 Method for Sampling Wood[3]

P5-14 Method of Analysis of Copper Dimethyldithiocarbamate (CDDC) Treated Wood by Colorimetry[3]

2.3 *AOAC International Standard:*

Method 972.29 Spectrophotometric Method—Thiram and Thiram Residues[4]

3. Summary of Test Method

3.1 Wood treated with CDDC is ground and extracted with chloroform/methanol azeotrope. CDDC, a colored compound, is determined colorimetrically by use of a suitable spectrophotometer at a wavelength of 435 nm.

4. Significance and Use

4.1 Copper dimethyldithiocarbamate (CDDC) for use in the preservative treatment of wood must conform with this test method.

5. Apparatus

5.1 *Spectrophotometer*—Any good spectrophotometer can be used.[5]

5.2 *Heated Magnetic Stir Plate*.

5.3 *Increment Borer and Grinder*—A typical Wiley mill is suitable to grind samples. The device should be capable of reducing the wood to a small particle size (30 mesh).

5.4 *Reflux Apparatus*, (Methods D 147, or equivalent).

5.5 *Buchner Vacuum Filter/Flask*.[5]

5.6 *Laboratory Glassware*, volumetric flasks, pipettes, cuvettes, and other commonly available glassware.

5.7 *Filter Paper*, Whatman #42[5] (sized to fit Buchner Funnel).

6. Reagents

6.1 *Chloroform*, Spectro Grade CH_3 Cl.

6.2 *Copper Dimethyldithiocarbamate*, analytical standard >99 %.[6]

6.3 *Methyl Alcohol*, Spectro Grade CH_3 OH.

6.4 *Extraction Azeotrope*, 12.6 % MeOH/87.4 % CH_3 Cl (w/w).

7. Sampling

7.1 Sample treated wood as described in Paragraph 5 of AWPA Standard M 2 except that the increment borings must be oven dried (60°C or less).

8. Preparation of CDDC Standards

8.1 Produce a standard curve by dissolving analytical grade copper dimethyldithiocarbamate in azeotrope to produce

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[1] This test method is under the jurisdiction of ASTM Committee D-7 on Wood and is the direct responsibility of Subcommittee D07.06 on Treatments for Wood Products.

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[2] Discontinued; see *1988 Annual Book of ASTM Standards*, Vol 04.04.

[3] Available from the American Wood-Preservers' Association, P.O. Box 286, Woodstock, MD 21163.

[4] Available from the Association of Official Analytical Chemists, Suite 400, 2200 Wilson Blvd., Arlington, VA 22201-3301.

[5] Use a visual spectrophotometer, such as Beckman Model, 35, B/L Spectronic 20, or equivalent. Available from Fisher Scientific, Box 405, Pittsburgh, PA 15230; or CMS, P.O. Box 98944, Chicago, IL 60693.

[6] Available from Pflatz and Bauer, P. O. Box 3723, New Hyde Park, NY 11040.

known amounts of CDDC in the range from 0 to 25 ppm. Plot these known, weighed, prepared standards against the absorbance measured by the spectrophotometer at a wavelength of 435 nm on standard quadrille graph paper.

9. Procedure

9.1 When using 20 borings (0.6 in. (15.24 mm)) in length (approximately 3.2 g of wood) use 200 g of chloroform azeotrope for extraction.

9.2 *Preparation of Standard Graph for CDDC in Treated Wood*:

9.2.1 Place analytical grade CDDC standard in azeotrope in dosage range from 3.55 to 21.31 µg/mL CDDC. Read in the spectrophotometer at 435 nm against chloroform/methanol azeotrope as the reference. The absorbance found will be 0.08 to 1.00. Plot the data for microgram per millilitre (or alternatively kilogram per cubic metre CDDC) against absorbance. Determine linearity and best fit for the respective curve.

9.3 *Testing Borings from Treated Wood*:

9.3.1 Grind whole borings prior to analysis. For this extraction technique, it is necessary to use an azeotrope, that has the capability of swelling the wood, to assist in extraction efficiency. Use an azeotrope consisting of 87.4 % chloroform, 12.6 % methanol.

9.3.2 Place ground borings into the extraction flask.

9.3.3 Place 200.0 mL of azeotrope into extraction flask.

9.3.4 Heat the extraction flask to 45°C and allow influx to cycle several times over a 30-min period.

9.3.5 After cooling, filter sawdust and extraction solvent containing CDDC by vacuum filtration through Whatman 42[6] (or equivalent) filter paper.

9.3.6 Rinse sawdust with two successive 100.0-mL washings of azeotrope.

9.3.7 Take exactly 3.0 mL of filtrate and dilute with 200.0 mL of azeotrope.

9.3.8 Read this final dilution at 435 nm on spectrophotometer.

9.4 *General Information*:

9.4.1 Analyze all diluted samples within 24 h.

9.4.2 Excess copper or dithiocarbamate used to form the chelate in the wood will not be detected. No spectral response is found for the individual components at 435 nm.

10. Test Procedures for Spectrophotometer

10.1 Turn on the spectrophotometer, and allow to warm up 10 min or longer to stabilize.

10.2 Obtain two test tube cells and clean them before using them, rinse each twice with acetone. Wipe off the outside of each with a tissue rather than cloth or anything that might scratch the cells.

10.3 Fill one cell with chloroform or azeotrope for the blank. Fill the other with the solution of CDDC after rinsing the cell at least once with the CDDC solution. Wipe off any liquid on the outside of each cell with a tissue.

10.4 *Setting 0 % Transmittance (Absorbance = 100 %)*—The 0 % transmittance setting corresponds to no light passing through the solution; it is made without inserting the cell into the instrument on the spectrophotometer. Simply adjust the left-hand knob so that the meter needle on the scale reads 0 on the percent transmittance scale. Be sure the reading is stable. Check it periodically throughout the experiment and readjust it if necessary.

10.5 *Setting the Wavelength*—Adjust the wavelength to the desired setting of 435 nm. For the Spectronic 20[8], adjust the wavelength using the knob on the top of the instrument.

10.6 *Setting 0 Absorbance (100 % Transmittance)*—Turn the right-hand knob of the Spectronic 20[8] counter-clockwise almost to its limit. Insert the test tube cell containing the sample into the cell holder. Match the line on the test tube cell with the index line on the holder. Close the top of the holder. Adjust the right-hand knob clockwise until the meter needle on the scale reads 0 absorbance. Remove the test tube to avoid instrument fatigue. Check the 0 % transmittance setting to be sure it has not changed. If it has, both the 0 % transmittance and 0 absorbance settings must be readjusted.

10.7 *Measurement of Absorbance of the CDDC Solution*—Insert the test tube cell containing the CDDC solution into the holder, again matching the marks. Allow the needle to stabilize, and read the absorbance of the solution to two significant figures; for example, 0.05, 0.10, 0.21. (Recall that for log terms such as absorbance, all digits, including zeroes to the right of the decimal point, are significant.) Remove the test tube to avoid instrument fatigue.

11. Interpretation of Results

11.1 *Extraction Efficiency*—Duplicate determinations of extraction efficiency run by the same operator on the same apparatus yielded extraction efficiencies of 88, 97, and 101 % for retentions of 0.48, 0.72, and 0.96 lb/ft^3, respectively.

11.2 Least squares regression was plotted and determined for all retention levels evaluated and found to have a correlation coefficient (R^2) of >0.95.

12. Report

12.1 Report values of sample mass, absorbance, and determination of instrument drift, and calculate (from regression line equation) or interpret (from linear graph) retention in kilograms per cubic metre (pound per cubic foot).

13. Precision and Bias

13.1 *Precision*:

13.1.1 *Repeatability*—Intralaboratory results have indicated the same operator using the same equipment on duplicate samples has agreed within the 95 % confidence limits.

13.1.2 *Reproducibility*—The precision of the procedure in this test method is being determined.

13.2 *Bias*—Duplicate determinations of extraction efficiency run by the same operator on the same apparatus yielded extraction efficiencies of 88, 97, and 101 % for retentions of 0.48, 0.72, and 0.96 lb/ft^3, respectively.

13.3 Least squares regression was plotted and determined for all retention levels evaluated and found to have a correlation coefficient (R^2) of >0.95.

14. Keywords

14.1 colorimetry; copper dimethyldithiocarbamate