

Test Method for Chelation Value of Polyaminopolycarboxylic Acids and Their Salts: Copper PAN

1. Foreword

This test method is an alternate to AATCC TM149, Test Method for Chelation Value of Aminopolycarboxylic Acids and Their Salts: Calcium Oxalate. Titration with a calcium salt using either oxalate or a dye indicator, as in AATCC TM149, yields a value that includes partially-substituted EDTA, HEDTA, DTPA, nitrilotriacetic acid (NTA), iminodiacetic acid (IDA), glycolate and other weak chelating agents found in some commercial products. This alternative procedure excludes the effect of the partially-substituted products, providing a more stringent value.

2. Purpose and Scope

2.1 The active ingredient content of ethylenediaminetetraacetic acid (EDTA), N-hydroxyethylethylenediaminetriacetic acid (HEDTA) and diethylenetriaminepentaacetic acid (DTPA) and their salts is usually expressed by the calcium chelation value (CaCV). This value indicates the amount of calcium (as calcium carbonate) which will be chelated by a known weight of chelating agent. This method excludes the effect of partially-substituted products.

3. Principle

3.1 Calcium chelation value is determined experimentally by titrating a weighed specimen of chelating agent with a copper nitrate solution of known concentration in the presence of PAN (1-(2-pyridylazo)-2-naphthol). Initially, the color of the solution is yellow-green due to free indicator. When all of the chelating agent has reacted with copper nitrate, the color of the solution changes to a permanent purple which is the end point.

3.2 The CaCV indicates the amount of calcium (as calcium carbonate) that will be sequestered by a known weight of chelating agent and is expressed as milligrams of calcium carbonate (CaCO_3) per gram of chelating agent (mg calcium carbonate/g chelating agent).

4. Referenced Documents

NOTE: Use current versions of all publications unless otherwise specified.

4.1 AATCC 149 Test Method for Chelation Value of Aminopolycarboxylic Acids and Their Salts: Calcium Oxalate

5. Terminology

5.1 **chelating agent**, *n.*—*in textile chemistry*, a chemical capable of deactivating metal ions by formation of a water-soluble complex. SYN: *sequestering agent*.

6. Safety Precautions

6.1 The safety precautions specified in the method/procedure are ancillary to the testing procedures and are not intended to be all inclusive.

6.2 It is the user's responsibility to reference applicable safety data sheets, use safe and proper techniques, and wear appropriate personal protective equipment in handling materials in this standard.

7. Reagents

- 7.1 Acetic acid, 1.0N (CH_3COOH)
- 7.2 Cupric nitrate 2.5 hydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 2.5\text{H}_2\text{O}$)
- 7.3 Ethylene diaminetetraacetic acid (free acid) (EDTA) ($\text{C}_{10}\text{H}_{16}\text{O}_8\text{N}_2$)
- 7.4 Methanol
- 7.5 1-(2-Pyridylazo)-2-naphthol, (PAN) ($\text{C}_{15}\text{H}_{11}\text{ON}_3$)
- 7.6 Sodium acetate trihydrate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$)
- 7.7 Sodium hydroxide, 1.0N (NaOH)

8. Sampling

8.1 Conduct the test in triplicate; i.e., analyze 3 test specimens from a sample.

9. Specimens

9.1 Do not use aluminum or metallic weighing pans.

9.2 If solid chelating agent is being evaluated, dry a 2-g sample at the appropriate temperature for at least 2 h and cool in a desiccator before weighing specimens.

9.2.1 Dry free acid forms of chelating agents at 120°C.

9.2.2 Dry salt forms of chelating agents at 80°C.

9.3 For analysis of solid EDTA, HEDTA, and DTPA (either free acid or salt forms) weigh a 0.24-0.26 g specimen of the dried chelating agent to an accuracy of 0.01 g.

9.4 For analysis of commercial solution, of salts of the chelating agents, weigh a 0.49-0.51 g specimen of the solution to an accuracy of 0.01 g.

10. Preparation of Reagents

10.1 Cupric Nitrate Solution: Dissolve 23.30 g of cupric nitrate 2.5 hydrate and dilute to 1.000 L with distilled water in a volumetric flask. Calculate the molarity of the cupric nitrate solution (see 10.4).

10.2 PAN Indicator: Dissolve 0.025 g of 1-(2-pyridylazo)-2-naphthol in 50 mL of methanol. Store in a refrigerator in a stoppered bottled. Prepare fresh weekly.

10.3 Acetate Buffer Solution: (pH = 4.65). Dissolve 34.0 g of sodium acetate trihydrate in 500 mL of distilled water and add 15 mL of glacial acetic acid. Mix well and store in a closed container.

10.4 Standardization of Copper-Nitrate Solution: Weigh to the nearest 0.0001 g approximately 0.5 g of pure reagent grade EDTA acid powder into each of the three 250-mL Erlenmeyer flasks. Add 150 mL of distilled water and 6.5 mL of 1.0N sodium hydroxide (or five drops of 50% sodium hydroxide) and stir the mixture until the EDTA is dissolved. Add 25 mL of sodium acetate buffer. Adjust to pH 4.5-5.5 with 1.0N acetic acid or 1.0N sodium hydroxide, if necessary. Add 1 mL PAN indicator and titrate immediately with copper nitrate to a permanent purple end point. Calculate the molarity of copper using Equation 1:

$$M = \frac{(W)(P)(K)}{V} \quad (\text{Eq. 1})$$

where:

M = Molarity of copper nitrate, expressed in moles/L

W = Weight of EDTA acid in grams

P = Percent purity of EDTA acid

V = Volume of copper nitrate titrant in mL

K = 0.042994

$$= \frac{(1000) \text{ mL/L}}{(100)(232.59 \text{ g/mole})}$$

$$= 0.042994 \frac{\text{mole} - \text{mL}}{\text{g} - \text{L}}$$

11. Procedure

11.1 Transfer the specimen to a 500-mL Erlenmeyer flask and add 150 mL of distilled water (see 9.2, 9.3 and 9.4). Wash any residual chelating agent from the weighing paper or dish into the flask, and swirl to mix (dissolve).

11.2 Add 25 mL of pH 4.65 acetate

buffer solution to the flask and swirl to mix. Check the pH and adjust to 4.5-5.5 with 1.0*N* acetic acid or 1.0*N* sodium hydroxide, if necessary.

11.3 Add 1 mL of PAN indicator to the flask and swirl to mix.

11.4 Titrate with 0.1000 M cupric nitrate solution to a purple end point that persists for at least 1 min.

12. Calculation

12.1 Calculate the CaCV for each specimen to 3 significant digits for each titration using the Equation 2:

$$\text{CaCV} = \frac{(100.1)(V)(M)}{W} \quad (\text{Eq. 2})$$

where:

CaCV = Calcium chelation value, expressed as mg CaCO₃/g chelating agent

V = Volume of cupric nitrate titrant in mL

M = Molarity of cupric nitrate solution, expressed in mmol/mL

100.1 = Molecular weight of calcium carbonate, expressed in mg/mmol

12.2 Average the CaCV values for the 3 specimens tested.

13. Report

13.1 Report for each sample tested:

13.1.1 A description or identification of the sample.

13.1.2 Evaluation was performed using AATCC TM168-2022.

13.1.3 Average CaCV, to 3 significant digits.

13.1.4 Any modifications of the test method.

14. Precision and Bias

14.1 *Precision*. The interlaboratory,

single operator, single apparatus, same day precision of the test method is ± 2 (1.60) standard deviations. The interlaboratory precision of the test method (over five participating laboratories) is ± 1 (0.54) standard deviations.

14.2 *Bias*. Chelation value can be defined only in terms of a test method. There is no independent method for determining the true value. As a means of estimating this property, the method has no known bias.

15. History

15.1 Revised in 2023 to clarify and align with prescribed AATCC style guidelines.

15.2 Editorially revised 2019; revised 2018; reaffirmed 2012; editorially revised 2010; editorially revised and reaffirmed 2007; reaffirmed 2002; editorially revised and reaffirmed 1997; revised 1992; editorially revised and reaffirmed 1989; reaffirmed 1988.

15.3 Developed in 1987 by AATCC Committee RR90. Maintained by RA99.