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Chelating Agents: Chelation Value of Aminopolycarboxylic Acids and Their Salts; Calcium Oxalate Method

Developed in 1976 by AATCC Committee RR90; editorially revised and reaffirmed 1977, 1985 (new title), 1997; reaffirmed 1980, 2002, 2007, 2012; editorially revised 1984, 1986, 1988 (new title), 2010; revised 1992.

1. Purpose and Scope

1.1 The active 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.

2. Principle

2.1 The chelation value is determined experimentally by titrating a weighed sample of chelating agent with a calcium ion solution of known concentration. A precipitating anion (oxalate) is present during the titration. As the calcium solution is added, the ions are chelated by the chelating agent and no permanent precipitate is formed as long as there is free chelating agent present. Beyond this point, any excess calcium ion that is added reacts with oxalate anion to form a cloudy precipitate which is the endpoint. The calcium chelation value (CaCV) indicates the amount of calcium (as calcium carbonate) which 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).

3. Terminology

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

4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The precautions are ancillary to the testing procedures and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Manufac-

turers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

4.2 All chemicals should be handled with care.

4.3 In preparing, dispensing and handling hydrochloric acid and sodium hydroxide use chemical goggles or face shield, impervious gloves and an impervious apron. Concentrated acids should be handled only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

4.4 Diethylamine hydrochloride is an eye, skin and respiratory irritant. It should be handled only in an adequately ventilated hood.

4.5 An eyewash/safety shower should be located nearby and a self-contained breathing apparatus should be readily available for emergency use.

4.6 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities (e.g., Occupational Safety and Health Administration's [OSHA] permissible exposure limits [PEL] as found in 29 CFR 1910.1000; see web site: www.osha.gov for latest version). In addition, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) comprised of time weighted averages (TLV-TWA), short term exposure limits (TLV-STEL) and ceiling limits (TLV-C) are recommended as a general guide for air contaminant exposure which should be met (see 14.1).

5. Uses and Limitations

5.1 Analysis by this method will yield a CaCV which includes partially substituted EDTA, HEDTA and DTPA as well as nitrilotriacetic acid (NTA), iminodiacetic acid (IDA), glycolate and other weak chelating agents. These compounds may be present in some commercial products.

5.2 AATCC Method 168, Chelating Agents: Active Ingredient Content of Polyaminopolycarboxylic Acids and Their Salts: Copper PAN Method, is an alternative procedure which yields a CaCV that does not include the partially substituted products and weaker chelating agents.

6. Reagents

- 6.1 Calcium carbonate (CaCO_3).
- 6.2 Sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$).
- 6.3 Diethylamine hydrochloride [$(\text{C}_2\text{H}_5)_2\text{NH} \cdot \text{HCl}$]
- 6.4 Sodium hydroxide (NaOH) solution, 50% W/W.
- 6.5 Hydrochloric acid (HCl) conc.

7. Sampling

7.1 The test should be conducted in triplicate; i.e., three test specimens from a sample should be analyzed.

8. Conditioning

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

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

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

9. Specimens

9.1 Do not use aluminum or metallic weighing pans.

9.2 For analysis of solid EDTA, HEDTA and DTPA (either free acid or salt forms) weigh a 0.49-0.51 g specimen of the dried chelating agent to an accuracy of 0.01 g.

9.3 For analysis of commercial solutions of salts of the chelating agents in 9.2 weigh a 1.00-1.20 g specimen of the solution to an accuracy of 0.01 g.

10. Preparation of Reagents

10.1 Calcium Titrant Solution (0.250 M): Weigh 25.0 g of calcium carbonate to an accuracy of 0.1 g and add it to 300 mL of water in a 600 mL beaker with stirring on a magnetic stirrer. Dissolve by adding 43 mL of concentrated hydrochloric acid. Add the hydrochloric acid slowly and carefully to prevent bubbling over and excessive splattering. After the calcium carbonate has dissolved (the solution becomes clear), heat the solution to boiling and allow it to boil for at least 5 min to expel carbon dioxide. Cool the solution to room temperature, transfer it to a 1 L volumetric flask and dilute it to 1 L. Prepare a fresh solution monthly.

10.2 Sodium Oxalate Indicator: Dissolve 5 g of sodium oxalate in 250 mL of