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Chelating Agents: Percent Content in Hydrogen Peroxide Bleach Baths; Copper PAN Indicator Method

Developed in 1998 by AATCC Committee RR90; reaffirmed 1999, 2000, 2006, 2011; editorially revised 2010.

1. Purpose and Scope

1.1 This test method measures the chelating agent content in a hydrogen peroxide bleach solution.

1.2 The method is designed to permit routine or periodic titration of the peroxide saturator section of a plant preparation range, or other prepared peroxide bleach solutions, to determine the concentration of chelating agent present.

1.3 The method is restricted to chelating agents based on ethylenediaminetetraacetic acid (EDTA), N-hydroxyethyl-ethylenediaminetriacetic acid (HEDTA), and diethylenetriaminepentaacetic acid (DTPA). This includes any proprietary products (referred to herein as "product") containing possible multiple ingredients, one or more of which may be chelating agents.

2. Principle

2.1 The percent chelating agent or product in the hydrogen peroxide bleach bath is determined in two steps: first, a direct titration of the chelating agent or product using a known concentration of copper sulfate in the presence of an indicator (PAN, 1-(2-pyridylazo)-2-naphthol), and second, a similar titration of the bleach solution containing the chelating agent using a more dilute concentration of the copper sulfate as the titrant.

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*.

3.2 **copper chelation value (CuCV)**, *n.*—the milligrams of copper sulfate pentahydrate chelated by one gram of a chelating agent or product containing a chelating 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. Manufacturers 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. Many of the chemicals used in this procedure are corrosive or a strong irritant.

4.3 In preparing, dispensing and handling sulfuric acid (98%), glacial acetic 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 hood. CAUTION: Always add acid to water.

4.4 Methanol is a flammable liquid and should be stored in the laboratory only in small containers away from heat, open flame and sparks.

4.5 An eyewash/safety shower should be located nearby and a high efficiency particulate respirator with organic vapor cartridges and a full face piece 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 13.1).

5. Uses and Limitations

5.1 Results may be affected by any of the following:

5.1.1 Pre-chelation of iron or copper ions contained in the bleach solution will reduce the apparent, calculated concentration of chelating agent originally present in the bath.

5.1.2 Oxidation of uncombined chelating agent by hydrogen peroxide will reduce the apparent concentration in the

bleach bath.

5.1.3 Cyanide ions, ammonia, and most amines will form molecular compounds with copper ions without affecting the PAN indicator, resulting in false positive results.

5.2 The percent chelating agent in the bleach bath will not be affected by changes in the active ingredient content of the chelating agent or product.

5.2.1 AATCC Method 168, Chelating Agents: Active Ingredient Content of Polyaminopolycarboxylic Acids, should be conducted on each new lot of the chelating agent or product to determine if the active ingredient content has changed.

5.2.2 The copper chelation value (CuCV) determined in this method is a direct indication of the active ingredient content of the chelating agent or product and can be monitored for this purpose.

6. Reagents

6.1 The reagents used in this method should be ACS reagent grade quality.

6.1.1 Acetic acid, glacial (CH_3COOH)

6.1.2 Copper sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)

6.1.3 Methanol (CH_3OH)

6.1.4 PAN, 1-(2-pyridylazo)-2-naphthol ($\text{C}_{15}\text{H}_{11}\text{ON}_3$)

6.1.5 Sodium acetate trihydrate ($\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$)

6.1.6 Sodium hydroxide, pellets (NaOH)

6.1.7 Sulfuric acid, 98% (H_2SO_4)

7. Sampling

7.1 Test two specimens of the chelating agent or product and two specimens of the bleach bath.

8. Preparation of Reagents

8.1 Reagent A (12.500 g/L copper sulfate). Dissolve 12.500 g $\pm$ 0.002 g of copper sulfate pentahydrate in distilled water and dilute to 1 L in a volumetric flask.

8.2 Reagent B (2.500 g/L copper sulfate). Transfer 200 mL of Reagent A to a 1 L volumetric flask and dilute to volume with distilled water.

8.3 PAN indicator. Dissolve 0.025 $\pm$ 0.001 g of PAN in 50.0 mL of methanol. Store in a stoppered bottle in a refrigerator. Prepare fresh weekly.

8.4 Sodium acetate buffer solution. Dissolve 34.0 $\pm$ 0.1 g of sodium acetate