

Test Method for Chelating Agent Content in Hydrogen Peroxide Bleach Baths: Copper PAN Indicator

1. Foreword

1.1 While several test methods exist for determining chelation value of chelating agents, this method includes an additional calculation for percent chelating agent in the bleach bath.

2. Purpose and Scope

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

2.2 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.

2.3 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.

3. Principle

3.1 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.

3.2 Results are reported as percent chelating agent. A higher value indicates more chelating agent or product in the bath.

4. Referenced Documents

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

4.1 AATCC TM168, Test Method for Chelation Value of Polyaminopolycarboxylic Acids and Their Salts: Copper PAN (see 15.1).

5. Terminology

5.1 **chelating agent**, *n.*—in *textile chemistry*, a chemical capable of deacti-

vating metal ions by formation of a water soluble complex. SYN. *sequestering agent*.

5.2 **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.

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.

6.3 Users MUST consult manufacturers for specific details such as equipment operating instructions and other recommendations. Consult and follow all applicable health and safety regulations (e.g., OSHA standards and rules).

7. Uses and Limitations

7.1 Results may be affected by any of the following:

7.1.1 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.

7.1.2 Oxidation of uncombined chelating agent by hydrogen peroxide will reduce the apparent concentration in the bleach bath.

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

7.2 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.

7.2.1 AATCC TM168 should be conducted on each new lot of the chelating agent or product to determine if the active ingredient content has changed.

7.2.2 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.

8. Reagents

8.1 Use ACS reagent grade quality chemicals. All chemicals should be handled with care. Many of the chemicals used in this procedure are corrosive or a strong irritant.

8.1.1 Acetic acid, glacial (CH_3COOH).

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

8.1.3 Methanol (CH_3OH). Methanol is a flammable liquid and should be stored in the laboratory only in small containers away from heat, open flame and sparks.

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

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

8.1.6 Sodium hydroxide, pellets (NaOH).

8.1.7 Sulfuric acid, 98% (H_2SO_4).

9. Samples

9.1 Test two samples of the chelating agent or product and two samples of the bleach bath.

10. Preparation of Reagents

10.1 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.

10.2 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. Retain remainder of Reagent A.

10.3 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.

10.4 Sodium acetate buffer solution. In preparing, dispensing and handling glacial acetic acid, 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. Dissolve 34.0 $\pm$ 0.1 g of sodium acetate trihydrate in 500 $\pm$ 1 mL of distilled water and add 15.0 $\pm$ 0.1 mL of glacial acetic acid. Mix well and store in a closed container.

10.5 Sodium hydroxide (20% by volume). In preparing, dispensing and handling sodium hydroxide, use chemical goggles, or face shield, impervious gloves and an impervious apron. Dis-

solve 200 ± 1 g of sodium hydroxide in 800 mL of distilled water with stirring. Cool, transfer to a 1 L volumetric flask and adjust to volume with distilled water.

10.6 Sulfuric acid (20% by volume). In preparing, dispensing and handling sulfuric acid (98%), 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. Slowly add 200 ± 1 g of sulfuric acid to 500 mL of distilled water. Cool, transfer to a 1 L volumetric flask and adjust to volume with distilled water.

11. Procedure

11.1 Chelation value of chelating agent or product.

11.1.1 Weigh 0.9-1.1 g of the chelating agent or product to nearest 0.01 g. Dilute to 75 mL with distilled water.

11.1.2 Add 25 mL of sodium acetate buffer solution and adjust the pH to 4.5-5.5 with glacial acetic acid (to decrease pH) or 20% sodium hydroxide (to increase pH) as required.

11.1.3 Add 1 mL of PAN indicator and titrate to a permanent purple end point with Reagent A. Record the volume used to the nearest 0.01 mL and use this volume in Eq. 1

11.1.4 A 12.5 g/L solution of copper sulfate (Reagent A) is used to titrate the chelating agent product, whereas a 2.5 g/L solution of copper sulfate (Reagent B) is used for greater accuracy to titrate the more dilute chelating agent contained in the peroxide bleach solution.

11.2 Chelating agent in bleach bath.

11.2.1 Weigh 90-110 g of the hydrogen peroxide bleach solution to the nearest 0.01 g. If the specimen is taken from the saturator, ensure that it is free of debris and foam.

11.2.2 Adjust the pH to 7.0-9.0 with 20% sulfuric acid.

11.2.3 Add 35 mL sodium acetate buffer solution. Adjust the pH to 4.5-5.5 with glacial acetic acid or 20% sodium hydroxide if necessary.

11.2.4 Add 1 mL of PAN indicator and titrate to a permanent purple end point with Reagent B. Record the volume used to nearest 0.01 mL and use this volume in Eq. 2.

12. Calculations

12.1 Calculate the copper chelation value of the chelating agent or product to two decimal places using Eq. 1:

$$\text{CuCV} = 12.5 (V)/W \quad (\text{Eq. 1})$$

where:

CuCV = Copper chelation value

V = volume of Reagent A used, mL

W = weight specimen, g

12.2 Calculate the mean copper chelation value of two samples.

12.3 Calculate the chelating agent content in the bleach bath to the nearest 0.01% using Eq. 2:

$$\text{CA} = 2.5(100)(V_2)/(W_2)(S) \quad (\text{Eq. 2})$$

where:

CA = chelating agent content, %

V_2 = volume Reagent B used, mL

W_2 = weight of specimen, g

S = average CuCV (from 12.2)

12.4 Calculate the average percent chelating agent content in the bleach bath.

13. Report

13.1 Describe or identify the samples/products tested.

13.2 Report that the sample was tested using AATCC TM185-2021.

13.3 Report the test results:

13.4.1 Report the average chelating agent value.

13.4.2 Report the average percent chelating agent content in the bleach bath.

13.5 Describe any modification(s) of the published standard.

14. Precision and Bias

14.1 Precision.

14.1.1 In 1990, an interlaboratory study was completed which included seven laboratories, one operator in each, running two determinations per sample on three samples in Step 11.1 of the procedure, and running two determinations per bath on three baths for Step 11.2 of the procedure. No prior assessment was made of the relative level of the participating laboratories in performance of the test method. Results from one lab were not included in the analysis.

14.1.2 Analysis of the data sets yielded components of variance and critical differences as displayed in Tables I, II and III. Differences between two averages of N determinations, for the appropriate precision parameter, should reach or exceed the table value to be statistically significant at the 95% confidence level.

14.2 Bias.

14.2.1 The values for CuCV can only

Table I—Components of Variance

Component	Chelation Value	% Chelating Agent
Laboratory V(L)	2.61	0.000111
Interaction V(SL)	0.76	0.000157
Within V	0.81	0.000030

Table II—Chelating Agent or Product Critical Differences (95% Confidence)

Det. in Avg (N)	Within Laboratory	Between Laboratory
(Single Sample Comparisons)		
1	2.49	5.13
2	1.76	4.81
3	1.44	4.71
(Multiple Sample Comparisons)		
1	2.49	5.67
2	1.76	5.39
3	1.44	5.29

Table III—Bleach Bath Critical Differences (95% Confidence)

Det. in Avg (N)	Within Laboratory	Between Laboratory
(Single Bath Comparisons)		
1	0.015	0.033
2	0.011	0.031
3	0.009	0.031
(Multiple Bath Comparisons)		
1	0.015	0.048
2	0.011	0.047
3	0.009	0.046

be defined in terms of a test method. There is no independent, referee method for determining the true values. The method has no known bias.

15. Notes

15.1 Available from AATCC, PO Box 12215, Research Triangle Park NC 27709, USA; +1.919.549.8141; ordering@aatcc.org; www.aatcc.org.

16. History

16.1 Revised in 2021 to clarify and align with prescribed AATCC style guidelines.

16.2 Editorially revised 2019. Reaffirmed 2016, 2011. Editorially revised 2010. Reaffirmed 2006, 2000, 1999.

16.3 Developed in 1998 by AATCC Committee RR90. Maintained by RA99.