

Preservative textile treatments —

Part 2: Methods of test

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Textiles and Clothing Standards Policy Committee (TCM/-) to Technical Committee TCM/22, upon which the following bodies were represented:

- Association of Heavy Textile Proofers of Great Britain
- British Narrow Fabrics Association
- British Textile Technology Group
- British Wood Preserving and Damp-proofing Association
- Chemical Industries' Association
- Health and Safety Executive
- International Wool Secretariat
- Ministry of Defence
- Society of Dyers and Colourists
- Textile Finishers' Association

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Foreword

This Part of BS 2087 has been prepared under the direction of the Textiles and Clothing Standards Policy Committee and is a revision of BS 2087-2:1981, which is withdrawn. BS 2087 is published in two Parts as follows.

— *Part 1: Specification for treatments;*

— *Part 2: Methods of test.*

The methods for determination of zinc, iron, copper and chromium are similar to those in BS 6810, but are included in full in this standard for easy reference.

CAUTION. Most preservative processes or chemicals have toxic properties in some measure and should be handled with care.

The principal change introduced in this revision is that the standard has been brought into line with BS 2087-1:1992 by the deletion of methods that are no longer required. The remaining methods have been brought up to date where necessary.

Attention is drawn to the foreword in BS 2087-1:1992.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations. In particular attention is drawn to section 6 of the Health and Safety at work etc. Act 1974, and the Environmental Protection Act 1990.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 18, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Part of BS 2087 describes methods to be used for quantitative analysis of the preservative agents listed in BS 2087-1.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Principle

The percentages of preservative chemical, impurities, etc. are calculated by mass in accordance with the appropriate methods and based upon the conditioned mass of the treated textile. The analytical methods are appropriate for textiles treated only by a single nominated preservative process.

NOTE Where treatment is associated with other textile processes, preliminary analytical procedures may be necessary in order to deal with interfering chemicals.

3 Reagents and apparatus

3.1 Reagents

3.1.1 General. Unless otherwise stated, all reagents shall be of analytical reagent grade.

3.1.2 Water, complying with BS 3978.

NOTE Specific reagents are listed for each method.

3.2 Apparatus

3.2.1 General laboratory equipment, complying with the appropriate British Standard as listed in Appendix A.

NOTE Specific apparatus is listed for each method.

4 Sampling and specimens

Laboratory test samples shall be selected so that they are representative of the bulk source. Take at least one laboratory test sample from each batch, taking at least one laboratory test sample per 1 000 m if the batch exceeds 1 000 m.

Take one specimen of the size specified in the individual test procedures from the laboratory test sample.

5 Standard atmosphere for conditioning and testing

Freely expose the sample to the standard temperate atmosphere for conditioning and testing textiles as defined in BS 1051, i.e. a test temperature of 20 ± 2 °C and a relative humidity of 65 ± 2 % until it is in equilibrium with that atmosphere. Carry out mass determinations without removing the specimens from this atmosphere.

6 Methods for quantitative analysis

6.1 Method 1. Determination of water soluble impurities

6.1.1 Preparation of extract

Obtain the aqueous extracts for the determination of water soluble impurities in the treated textile in accordance with BS 3266. Unless otherwise stated in BS 2087-1, use the hot water extraction procedure.

6.1.2 Measurement of pH, conductivity and the determination of sulfates

Measure the pH and conductivity and determine the sulfate content in accordance with BS 3266.

6.1.3 Determination of water soluble chlorides

Determine water soluble chlorides in accordance with BS 3266. If the treated textile contains chlorinated compounds normally classed as insoluble in water and soluble in organic solvents, remove these by extraction first with a suitable solvent before extracting the textile with water.

6.1.4 Determination of total soluble chromium content

Determine the total soluble chromium in accordance with the method described in BS 6810, except that the aqueous extract shall be prepared in accordance with the hot water method of BS 3266 instead of the buffer method given in BS 6810.

6.1.5 Determination of water soluble iron

Determine the water soluble iron in the filtered water extract (see 6.1.1) in accordance with the method described in BS 6810.

6.1.6 Determination of water soluble copper

Determine the water soluble copper in the filtered water extract (see 6.1.1) in accordance with the method described in BS 6810.

6.2 Method 2. Determination of solvent soluble matter

6.2.1 General

The procedure describes a method for the removal and determination of oil, fat and added wax in textile materials treated with inorganic proofing agents. All organic solvents, however, extract some of the natural constituents of textile materials, and blanks of the untreated materials may have to be determined. The mass of the solvent extractable matter is expressed as a percentage of the sample conditioned in the standard temperate atmosphere for testing textiles (see clause 5).

6.2.2 Reagents

6.2.2.1 Acetone, complying with BS 509.

6.2.2.2 Dichloromethane, complying with BS 1994.

6.2.3 Apparatus

6.2.3.1 Soxhlet extraction apparatus, of suitable capacity with interchangeable ground glass joints.

6.2.3.2 Suitable heating apparatus, temperature controlled.

6.2.3.3 Cellulose extraction thimbles, previously extracted with solvent¹⁾.

6.2.4 Procedure

Extract a convenient quantity (e.g. 5 g to 10 g) of the conditioned sample, diced, loosely rolled or folded in a filter thimble in a Soxhlet extractor with dichloromethane. Extract for 3 h with a minimum siphon rate of at least six times per hour. Disconnect the extractor and remove the bulk of the solvent in the flask by distillation. If droplets of water are present in the residual solvent add 2 ml to 3 ml of acetone and heat on a water bath repeating the process if necessary until no water is present. Remove the remainder of the solvent by one of the following methods.

- a) Heat the flask in a drying oven at 90 °C to 95 °C for 30 min. Place it in a vacuum desiccator, evacuate the air and allow to cool for 30 min before weighing.

NOTE This method is applicable to wool (see BS 3582).

- b) Evaporate the solvent with a jet of clean air and dry in an oven at 95 °C to 100 °C for 2 h. Cool in a desiccator and weigh.

NOTE This method is applicable to cotton (see BS 3477).

- c) Connect the flask by means of a suitable adaptor to a suction pump. Reduce the pressure in the flask to 1 333 Pa to 4 000 Pa for not less than 12 min and not more than 15 min whilst warming the flask to 60 °C to 65 °C. Release the vacuum through a side tube with a cotton wool filter before disconnecting the adaptor. Cool in a desiccator and weigh.

NOTE This method is applicable to jute and related materials (see BS 3845).

6.2.5 Calculation of results

Calculate the solvent soluble matter as a percentage of the conditioned sample mass corrected for the blank where required. Where a stitch finish is required the solvent soluble matter shall be in accordance with the limits given in the respective treatments.

6.3 Method 3. Determination of zinc

6.3.1 General

Two methods are given for the determination of the total zinc content but neither method is specific for zinc naphthenate or zinc pyrron. These are:

- a) volumetric;
- b) atomic absorption.

If method a), volumetric, is used, then jute and similar materials which contain metal impurities forming oxinates at pH 5 to pH 6 will be determined as zinc and it is necessary therefore to carry out a blank determination on the unproofed material. Method b), atomic absorption, is specific for zinc.

NOTE Where the determination of zinc relates solely to assessing the content of zinc naphthenate or zinc pyrron it is expected that the proofer or finisher will guarantee that the zinc is present solely as naphthenate or pyrron and is not mixed with other zinc salts or compounds.

6.3.2 Volumetric method

6.3.2.1 General

The material is subjected to wet oxidation. The zinc is precipitated as zinc oxinate from a buffered solution, the precipitate dissolved in acid and the oxine content determined after bromination by an iodometric solution.

6.3.2.2 Reagents

6.3.2.2.1 Sodium acetate solution,
 $c(\text{CH}_3\text{COONa}) = 1 \text{ mol/l}$.

6.3.2.2.2 Acetic acid, $c(\text{CH}_3\text{COOH}) = 1 \text{ mol/l}$. Dilute acetic acid $c(\text{CH}_3\text{COOH}) = 5 \text{ mol/l}$, 1 + 4.

6.3.2.2.3 Ammonia solution, 5 mol/l.

6.3.2.2.4 Ammonium chloride solution,
 $c(\text{NH}_4\text{Cl}) = 2 \text{ mol/l}$.

6.3.2.2.5 8-hydroxyquinoline, 20 g/l solution, prepared by dissolving 2 g of 8-hydroxyquinoline in 100 ml of acetic acid (6.3.2.2.2).

6.3.2.2.6 Hydrochloric acid, $c(\text{HCl}) = 2 \text{ mol/l}$. Dilute hydrochloric acid $c(\text{HCl}) = 5 \text{ mol/l}$, 2 + 3.

6.3.2.2.7 Nitric acid, concentrated, 70 % (m/m).

6.3.2.2.8 Sulfuric acid, concentrated, 98 % (m/m).

6.3.2.2.9 Potassium bromate standard solution,
 $c(\text{KBrO}_3) = 0.02 \text{ mol/l}$. Dissolve 2.784 g potassium bromate and 12.0 g potassium bromide in water and make up to 1 000 ml with water.

6.3.2.2.10 Sodium thiosulfate standard solution,
 $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1 \text{ mol/l}$.

6.3.2.2.11 Potassium iodide non-standardized solution, $c(\text{KI}) = 0.5 \text{ mol/l}$.

¹⁾ For information on the availability of this material, write to Customer Information, BSI, Linford Wood, Milton Keynes MK14 6LE.

6.3.2.2.12 Methyl red 0.05 g/l indicator solution, prepared by dissolving 0.05 g of methyl red in 1 000 ml of a solution consisting of 800 ml 95 % (V/V) ethanol and 200 ml of water.

6.3.2.2.13 Soluble starch indicator solution, 10 g/l.

6.3.2.3 Procedure

Weigh accurately approximately 2 g of the material, transfer to a 200 ml Kjeldahl flask. Add 10 ml of sulfuric acid followed by gradual addition of nitric acid until there is no reaction on further addition of acid.

NOTE This volume can vary and may be as much as 30 ml.

Apply heat gradually and digest with the further addition of nitric acid, if necessary until organic matter is completely destroyed. Evaporate down to fuming, cool, add 5 ml to 10 ml of water and boil to remove nitric acid. Cool, dilute to approximately 50 ml with water and filter if necessary through a hardened ashless grade filter paper with high retention efficiency and high wet strength²⁾.

To the solution add a few drops of methyl red, make just alkaline with ammonia solution, add 120 ml of ammonium chloride solution, 25 ml of sodium acetate solution, 6 ml of acetic acid and dilute the solution to approximately 200 ml with water. Adjust the pH value of the solution using a pH meter to approximately 5.3 by adding acetic acid or ammonia solution as appropriate. Heat the solution to 70 °C, add 10 ml of 8-hydroxyquinoline reagent solution, bring to the boil and place on a heated steam bath for 30 min. Check that there is excess of reagent solution then filter through a suitable sintered glass crucible (e.g. G3 or G4) and wash the precipitate with hot water to remove excess 8-hydroxyquinoline, i.e. until the washings are free from colour. Transfer the precipitate to a beaker, and extract the crucible with hot hydrochloric acid; then transfer the washings to the beaker, bulk up to approximately 100 ml with hydrochloric acid and boil solution to dissolve precipitate. Transfer the solution to a 500 ml conical flask, cool, add 1 ml of methyl red indicator solution and titrate with potassium bromate until the colour of the solution becomes sulfur yellow. Add a further 1 ml of indicator solution. Continue the process until a 1 ml portion of the indicator is decolourized immediately.

NOTE This second stage of the titration should not take more than a few millilitres of the potassium bromate solution.

Note the volume of titrant added. Allow the solution to stand for 2 min to ensure complete bromination of the 8-hydroxyquinoline, add 5 ml of the potassium iodide solution and titrate the liberated iodine with the sodium thiosulfate solution using soluble starch as indicator.

NOTE The back titration should not exceed 2 ml.

Carry out a reagent blank determination using the same amount of indicator solution as used in the determination.

1 ml of 0.2 mol/l potassium bromate solution = 0.000 980 6 g zinc.

6.3.3 Atomic absorption method

6.3.3.1 General

The material is subjected to wet oxidation and the solution is examined for zinc content by an atomic absorption spectrophotometer.

6.3.3.2 Instrumental conditions

Instrumental conditions are as follows:

Wavelength: 213.9 nm

Fuel: Acetylene-air

6.3.3.3 Reagents

6.3.3.3.1 Zinc oxide

6.3.3.3.2 Nitric acid, concentrated, 70 % (m/m).

6.3.3.3.3 Sulfuric acid, concentrated, 98 % (m/m).

6.3.3.3.4 Sulfuric acid, $c(\text{H}_2\text{SO}_4) = 0.5 \text{ mol/l}$. Dilute sulfuric acid $c(\text{H}_2\text{SO}_4) = 2.5 \text{ mol/l}$, 1 + 4.

6.3.3.3.5 Zinc, 100 mg/l, *standard solution*, prepared by dissolving 0.1245 g of zinc oxide, previously heated to 600 °C for 1 h, in sulfuric acid solution (6.3.3.3.4) and diluting to 1 000 ml in a volumetric flask.

6.3.3.4 Calibration

Standard zinc solutions. Into five 100 ml volumetric flasks run from a burette 0.0, 2.0, 4.0, 6.0 and 8.0 ml of standard zinc solution. Pipette 3 ml of sulfuric acid (6.3.3.3.4) into each flask and make up to the mark with water. These solutions contain 0.0, 2.0, 4.0, 6.0 and 8.0 mg of zinc per litre in 0.015 mol/l sulfuric acid. Run the solution containing no added zinc as blank.

6.3.3.5 Procedure

Weigh accurately 2.0 g of the material, transfer to a 200 ml Kjeldahl flask; add 10 ml of concentrated sulfuric acid followed by gradual addition of nitric acid until there is no reaction on further addition of acid.

NOTE This volume can vary and may be as much as 30 ml.

²⁾ For information on the availability of this material, write to Customer Information, BSI, Linford Wood, Milton Keynes MK14 6LE.

Apply heat gradually and digest with the further addition of nitric acid if necessary until organic matter is completely destroyed. Evaporate down to fuming, cool, add 5 ml to 10 ml of water and boil to remove nitric acid. Cool, dilute to approximately 50 ml with water, filter if necessary and make up to 500 ml with water in a volumetric flask. Pipette 10 ml of this solution into a 100 ml volumetric flask and make up to the mark with water.

Select optimum instrument working conditions then aspirate in turn the blank, standard and test solutions. Check the most concentrated standard solution after the test solution has been run and then recheck all the standard solutions.

6.3.3.6 Calculation of results

Plot a calibration graph of zinc concentration in the standard solutions against absorption. Read off the zinc concentration in the test solution and hence the zinc content of the sample.

6.4 Method 4. Determination of iron, chromium and copper

6.4.1 General

Where the determination of copper relates solely to assessing the content of copper naphthenate it is to be expected that the proofer or finisher will guarantee that the copper is present solely as naphthenate and is not adulterated with other copper soaps or compounds.

These methods are intended to be used for the determination of appreciable quantities of iron, chromium and copper. Alternative methods using atomic absorption spectrophotometry are given.

6.4.2 Reagents

6.4.2.1 Ammonium thiocyanate

6.4.2.2 Potassium chlorate

6.4.2.3 Hydrochloric acid, concentrated, 36 % (m/m), $c(\text{HCl}) = 1 \text{ mol/l}$.

6.4.2.4 Hydrochloric acid, $c(\text{HCl}) = 1 \text{ mol/l}$. Dilute hydrochloric acid $c(\text{HCl}) = 5 \text{ mol/l}$, 1 + 4.

6.4.2.5 Hydrochloric acid, $c(\text{HCl}) = 0.2 \text{ mol/l}$. Dilute hydrochloric acid $c(\text{HCl}) = 5 \text{ mol/l}$, 1 + 24.

6.4.2.6 Hydrogen peroxide, 300 g/l (100 volumes) solution.

6.4.2.7 Hydrogen sulfide, saturated solution, 4.4 g/l at 20 °C.

6.4.2.8 Nickel sulfate, 50 g/l solution.

6.4.2.9 Nitric acid, concentrated, 70 % (m/m).

6.4.2.10 Nitric acid, 20 g/l solution.

6.4.2.11 Potassium iodide, 100 g/l solution.

6.4.2.12 Sodium hydroxide, $c(\text{NaOH}) = 2 \text{ mol/l}$.

Dilute sodium hydroxide $c(\text{NaOH}) = 5 \text{ mol/l}$, 2 + 3.

6.4.2.13 Sulfuric acid, concentrated 98 % (m/m).

6.4.2.14 Sulfuric acid, $c(\text{H}_2\text{SO}_4) = 1.0 \text{ mol/l}$. Dilute sulfuric acid $c(\text{H}_2\text{SO}_4) = 2.5 \text{ mol/l}$, 2 + 3.

6.4.2.15 Cerium(IV) sulfate standard volumetric solution, $c(\text{Ce}(\text{SO}_4)_2) = 2 \text{ mol/l}$.

6.4.2.16 Ammonium iron(II) sulfate standard solution, $c(\text{FeSO}_4(\text{NH}_4)_5\text{SO}_4 \cdot 6\text{H}_2\text{O}) = 0.1 \text{ mol/l}$.

6.4.2.17 Sodium thiosulfate standard volumetric solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1 \text{ mol/l}$.

6.4.2.18 Ferroin, 15 g/l indicator solution, prepared by dissolving 1.5 g of 1,10-phenanthroline hydrate and 0.7 g of iron (II) sulfate heptahydrate in 100 ml of water.

6.4.2.19 N-phenylanthranilic acid, 1 g/l indicator solution, prepared by dissolving 0.1 g of N-phenylanthranilic acid and 0.05 g of anhydrous sodium carbonate in a little hot water and diluting to 100 ml with water.

6.4.2.20 Soluble starch indicator solution, 5 g/l.

6.4.3 Apparatus

6.4.3.1 Jones reductor, silver reductor³⁾

6.4.4 Preliminary separation methods

6.4.4.1 Copper present: iron and chromium absent

Weigh accurately approximately 5 g of material cut into 5 mm squares and transfer to a silica or porcelain crucible. Ash at $500 \pm 25^\circ\text{C}$. Dissolve the ash by warming in 25 ml of sulfuric acid (6.4.2.14), filter if necessary and determine the copper content as described in 6.4.7.

6.4.4.2 Chromium present: iron and copper absent

Weigh accurately approximately 2.5 g of the material cut into 5 mm squares and transfer to a silica or porcelain crucible. Ash at $500 \pm 25^\circ\text{C}$. Add to the ash 0.3 g of potassium chlorate and 5 ml nitric acid (6.4.2.9) and heat on a steam bath keeping the crucible covered with a watch glass during the process. When the reaction has ceased, remove the watch glass and rinse it with a little concentrated nitric acid if necessary. Evaporate the contents of the crucible to dryness. If any dark coloured material remains, repeat the treatment with potassium chlorate and nitric acid. Dissolve the residue in 5 ml sulfuric acid (6.4.2.14), filter if necessary and transfer to a suitable flask, diluting to about 100 ml with sulfuric acid (6.4.2.14). Determine the chromium content as described in 6.4.6.

³⁾ See Walden, Hamlett and Edmonds, *J. Amer. Chem. Soc.* **56** (1934) 350.