



Preservative treatments for textiles

Part 2. Methods of test

Traitements pour la préservation des textiles
Partie 2. Méthodes d'essai

Konservierungsmaßnahmen für Textilien
Teil 2. Prüfverfahren

BS 2087 : Part 2 : 1981

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Foreword

This British Standard has been revised under the direction of the Fibres, Yarns and Fabrics Standards Committee, and, for the first time, the standard is divided into two Parts. These are as follows.

Part 1. Specification for treatments.

Part 2. Methods of test.

Attention is drawn to the foreword in Part 1 of this standard.

British Standard

Preservative treatments for textiles

Part 2. Methods of test

1. Scope

This Part of this British Standard specifies methods to be used for the quantitative analysis of the preservative agents given in Part 1 of this standard.

2. References

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

3. 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. In cases of dispute it is essential to use methods specified.

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

4. Health and safety

Attention is drawn to clause 4 of Part 1 : 1981 of this standard which contains the following statement: **Most preservative processes or chemicals have toxic properties in some measure and should be handled with care.**

It should also be noted that some of the chemicals used in the methods of test present a particular hazard. The tests should only be carried out under the supervision of an experienced analyst.

5. Reagents and apparatus

5.1 Reagents. Unless otherwise stated, all reagents shall be of analytical reagent grade. Water shall comply with the requirements of BS 3978. Specific reagents are listed for each method.

5.2 Apparatus. Apparatus is listed for each method. Appendix A gives information on British Standards for laboratory equipment. Where appropriate, apparatus shall comply with the requirements of a suitable standard.

6. 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 1000 m if the batch exceeds 1000 m.

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



7. Standard atmosphere for conditioning and testing

Freely expose the sample to the standard atmosphere for conditioning and testing as defined in BS 1051, i.e. temperature $20 \pm 2^\circ\text{C}$, relative humidity $65 \pm 2\%$ until it is in equilibrium with that atmosphere. Carry out mass determinations without removing the specimens from the standard atmosphere for testing.

8. Methods for quantitative analysis

8.1 Method 1. Determination of water soluble impurities

8.1.1 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 Part 1 of this standard, use the hot water extraction procedure.

8.1.2 Measurement of pH, conductivity, and determination of sulphates. Measure the pH and conductivity and determine the sulphate content in accordance with BS 3266.

8.1.3 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.

8.1.4 Determination of total soluble chromium content. Determine the total soluble chromium in accordance with the method described in BS Handbook No. 11 for determination of water soluble chromium in textile materials, 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 Handbook No. 11.

8.1.5 Determination of water soluble iron. Determine the water soluble iron in the filtered water extract (see 8.1.1) in accordance with the method described in BS Handbook No. 11 for the colorimetric determination of small amounts of iron using method A (mercaptoacetic acid).

8.1.6 Determination of water soluble copper. Determine the water soluble copper in the filtered water extract (see 8.1.1) in accordance with the method described in BS Handbook No. 11 for the colorimetric determination of small amounts of copper using method A (sodium diethyldithiocarbamate).



8.2 Method 2. Determination of solvent soluble matter

8.2.1 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 atmosphere for testing.

8.2.2 Reagents. The following reagents are required.

8.2.2.1 Acetone (see BS 509).

8.2.2.2 Dichloromethane (see BS 1994).

8.2.3 Apparatus. The following apparatus is required.

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

8.2.3.2 Suitable heating apparatus, temperature controlled.

8.2.3.3 Thimbles, previously extracted with solvent*.

8.2.4 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 h. 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 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.

(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.

(c) Connect the flask by means of a suitable adaptor to a suction pump. Reduce the pressure in the flask to 1333 Pa to 4000 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. The drying procedures detailed in (a), (b) and (c) are applicable to wool (BS 3582) cotton (BS 3477) and jute and related materials (BS 3845) respectively.

8.2.5 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.

8.3 Method 3. Determination of zinc

8.3.1 General. Two methods are given for the determination of the total zinc content but neither method is specific for zinc naphthenate. 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 it is expected that the proofer or finisher will guarantee that the zinc is present solely as naphthenate and is not mixed with other zinc soaps or compounds.

8.3.2 Volumetric method. 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 titration.

8.3.2.1 Reagents. The following reagents are required.

8.3.2.1.1 Sodium acetate solution M.

8.3.2.1.2 Acetic acid M, dilute 5M reagent solution 1 + 4.

8.3.2.1.3 Ammonia solution, 5M reagent solution.

8.3.2.1.4 Ammonium chloride, 2M reagent solution.

8.3.2.1.5 8-hydroxyquinolin 20 g/l, reagent solution prepared by dissolving 2 g of 8-hydroxyquinolin in 100 ml of M acetic acid.

8.3.2.1.6 Hydrochloric acid, 2M, dilute 5M reagent solution 2 + 3.

8.3.2.1.7 Nitric acid, concentrated 70 % (m/m) (16M).

8.3.2.1.8 Sulphuric acid, concentrated, 98 % (m/m) (approximately 18M).

8.3.2.1.9 Potassium bromate 0.02M standard volumetric solution prepared by dissolving 2.784 g potassium bromate and 12.0 g potassium bromide in water and making up to 1000 ml with water.

8.3.2.1.10 Sodium thiosulphate 0.1M standard volumetric solution.

8.3.2.1.11 Potassium iodide 0.5M non-standardized volumetric solution.

8.3.2.1.12 Methyl red 0.05 g/l indicator solution prepared by dissolving 0.05 g of methyl red in 1000 ml of a solution consisting of 800 ml 95 % (V/V) ethanol and 200 ml of water.

8.3.2.1.13 Soluble starch indicator solution 10 g/l.

8.3.2.2 Procedure. Weigh accurately about 2 g of the material, transfer to a 200 ml Kjeldahl flask. Add 10 ml of sulphuric acid followed by gradual addition of nitric acid until there is no reaction on further addition of acid (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 suitable filter paper.†

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 to approximately 5.3 by adding acetic acid or

*Whatman filter paper thimbles are suitable.

†Whatman 542 is a suitable paper.

ammonia solution as appropriate using a pH meter. Heat the solution to 70 °C, add 10 ml of 8-hydroxyquinolin 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-hydroxyquinolin 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 sulphur yellow. Add a further 1 ml of indicator solution. Continue the process until a 1 ml portion of the indicator is decolorized immediately. 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-hydroxyquinolin, add 5 ml of the potassium iodide solution and titrate the liberated iodine with the sodium thiosulphate solution using soluble starch as indicator. 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 0.2M potassium bromate solution $\equiv$ 0.000 980 6 g zinc.

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

8.3.3.1 Instrumental conditions. Instrumental conditions should be as follows.

Wavelength : 213.9 nm

Fuel : Acetylene-air.

8.3.3.2 Reagents. The following reagents are required.

8.3.3.2.1 Zinc oxide.

8.3.3.2.2 Nitric acid, concentrated, 70 % (m/m) (16M).

8.3.3.2.3 Sulphuric acid, concentrated, 98 % (m/m) (approximately 18M)

8.3.3.2.4 Sulphuric acid 0.5M, dilute 2.5M reagent solution 1 + 4.

8.3.3.2.5 Zinc solution 100 mg/l zinc, standard reference solution, prepared by heating approximately 1 g of zinc oxide at 600 °C for 1 h, and cooling. Dissolve 0.1245 g in 10 ml of the 0.5M sulphuric acid solution and make up to 1000 ml.

8.3.3.3 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 sulphuric acid 0.5M reagent solution 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 Zn/litre in 0.015M sulphuric acid. Run the solution containing no added zinc as a blank.

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

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

8.4 Method 4. Determination of iron, chromium and copper

8.4.1 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.

8.4.2 Reagents. The following reagents are required.

8.4.2.1 Ammonium thiocyanate.

8.4.2.2 Potassium chlorate.

8.4.2.3 Hydrochloric acid, concentrated (36 % m/m) 11M.

8.4.2.4 Hydrochloric acid, M dilute 5M reagent solution 1 + 4.

8.4.2.5 Hydrochloric acid, 0.2M dilute 5M reagent solution 1 + 24.

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

8.4.2.7 Hydrogen sulphide, saturated solution, 4.4 g/l at 20 °C.

8.4.2.8 Nickel sulphate, 50 g/l reagent solution.

8.4.2.9 Nitric acid concentrated, (70 % m/m) 16M.

8.4.2.10 Nitric acid, 20 g/l reagent solution.

8.4.2.11 Potassium iodide, 100 g/l reagent solution.

8.4.2.12 Sodium hydroxide, 2M, dilute 5M reagent solution 2 + 3.

8.4.2.13 Sulphuric acid, concentrated (98 % m/m) 18M.

8.4.2.14 Sulphuric acid, 1M, dilute 2.5M reagent solution 2 + 3.

8.4.2.15 Ceric sulphate, 0.2M standard volumetric solution.

8.4.2.16 Ammonium ferrous sulphate, 0.1M standard volumetric solution.

8.4.2.17 Sodium thiosulphate, 0.1M standard volumetric solution.

8.4.2.18 Ferrouin, 15 g/l, indicator solution prepared by dissolving 1.5 g of 1,10-phenanthroline hydrate and 0.7 g of ferrous sulphate heptahydrate in 100 ml of water.

8.4.2.19 *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.

8.4.2.20 Soluble starch, indicator solution, 5 g/l.

8.4.3 Apparatus. The following apparatus is required.

8.4.3.1 Jones reductor, silver reductor.*

8.4.4 Preliminary separation methods

8.4.4.1 Copper present: iron and chromium absent. Weigh accurately about 5 g of textile cut into 5 mm squares and transfer to a silica or porcelain crucible. Ash at about 500 °C. Dissolve the ash by warming in 25 ml of sulphuric acid, 1 M, filter if necessary and determine the copper content as described in 8.4.7.

8.4.4.2 Chromium present: iron and copper absent. Weigh accurately about 2.5 g of the textile cut into 5 mm squares and transfer to a silica or porcelain crucible. Ash at about 500 °C. Add to the ash about 0.3 g of potassium chlorate and 5 ml nitric acid, concentrated, 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 sulphuric acid, 1 M filter if necessary, and transfer to a suitable flask, diluting to about 100 ml with sulphuric acid, 1 M. Determine the chromium content as described in 8.4.6.

8.4.4.3 Iron and chromium present: copper absent. Weigh accurately about 2.5 g of the textile cut into 5 mm squares and transfer to a silica or porcelain crucible. Ash at about 500 °C. Add to the ash about 0.3 g of potassium chlorate and 5 ml concentrated nitric acid, 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 sulphuric acid, 1 M, and again evaporate to dryness. Finally dissolve the residue in water, filter and dilute with water to about 50 ml. Bring the filtrate to the boil and add sodium hydroxide, 2 M, to precipitate the iron as ferric hydroxide, then add about 10 ml excess of sodium hydroxide, 2 M. Boil the contents of the beaker for one to two min then filter through a suitable filter paper.† Wash the precipitate with hot water and add the washings to the filtrate. When the precipitate of ferric hydroxide indicates other than a negligible iron content, dissolve the precipitate in hot hydrochloric acid, M, and precipitate the iron as previously described. Filter, wash the precipitate with hot water, and add the filtrate to the filtrate from the first precipitation.

Acidify the combined filtrates with sulphuric acid, 1 M, and add sufficient sulphuric acid, concentrated, to give the solution approximately molar concentration with respect to free acid and cool. Determine the chromium content as described in 8.4.6.

Dissolve the ferric hydroxide precipitate in hot hydrochloric acid, M, and then cool. Determine the iron content as described in 8.4.5.

8.4.4.4 Copper and chromium present: iron absent. Weigh accurately about 5 g of the textile cut into 5 mm squares and transfer to a silica or porcelain crucible. Ash at about 500 °C. Add to the ash about 0.3 g of potassium chlorate and 5 ml nitric acid, concentrated, 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. To the residue add 1 ml hydrochloric acid, concentrated and again evaporate to dryness. Treat the residue with hydrochloric acid, 0.2 M, transfer to a beaker, filtering if necessary. Add hydrogen sulphide solution or pass hydrogen sulphide gas through the solution in order to precipitate copper. Filter through a suitable paper.†

Boil the filtrate containing chromium until free from hydrogen sulphide, cool and add 3 ml hydrogen peroxide and an excess of sodium hydroxide, 2 M. Heat on a water bath until effervescence ceases. Add 5 ml nickel sulphate reagent solution and boil for 5 min, cool and acidify by the addition of 10 ml sulphuric acid, concentrated, and adjust solution volume so that it is approximately molar in respect of free acid and cool. Determine the chromium content as described in 8.4.6.

Wash the precipitate of copper sulphide with a small volume of hydrogen sulphide solution. Dissolve the precipitate in hot nitric acid, 20 g/l solution, and boil the solution nearly to dryness, then add 25 ml of sulphuric acid, 1 M, subsequently evaporating until fumes appear. Cool, make up to 25 ml with water, filter if necessary, and determine the copper as described in 8.4.7.

8.4.4.5 Copper and iron present: chromium absent. Weigh accurately about 5 g of the textile cut into 5 mm squares and transfer to a silica or porcelain crucible. Ash at about 500 °C. Add to the ash about 0.3 g of potassium chlorate and 5 ml nitric acid, concentrated, 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. To the residue add 1 ml hydrochloric acid, concentrated, and again evaporate to dryness. Treat the residue with hydrochloric acid, 0.2 M, transfer to a beaker, filtering if necessary. Add hydrogen sulphide solution or pass hydrogen sulphide gas through the solution in order to remove copper and filter through a suitable filter paper. Boil the filtrate containing iron until free of hydrogen sulphide, cool and add hydrochloric acid, concentrated, until the solution is approximately molar with respect to free acid. Determine the iron content as described in 8.4.5.

*See Walden, Hammett & Edmonds, *J. Amer. Chem. Soc.* **56**, (1934), 350.

†Whatman 54 is a suitable paper.

Wash the precipitate of copper sulphide with a small volume of hydrogen sulphide solution. Dissolve the precipitate in hot nitric acid, 20 g/l solution, and boil the solution nearly to dryness then add 25 ml of sulphuric acid, 1M, and subsequently evaporate until fumes appear. Cool, make up to 25 ml with water, filter if necessary and determine the copper content as described in 8.4.7.

8.4.5 Determination of iron. Pass the solution of iron in hydrochloric acid through ferrion reductor* and collect in a suitable flask. Wash the reductor with 150 ml of hydrochloric acid, M reagent, and add the washings to the flask. Titrate the solution with ceric sulphate standard volumetric solution, using a suitable oxidation reduction indicator.

1 ml ceric sulphate 0.02M solution $\equiv$ 1.12 mg Fe.

8.4.6 Determination of chromium. Titrate the solution containing chromium as dichromate with ammonium ferrous sulphate, standard volumetric solution using *N*-phenylanthranilic acid as an oxidation reduction indicator.

1 ml ammonium ferrous sulphate 0.1M solution $\equiv$ 1.73 mg Cr.

8.4.7 Determination of copper. Add to the solution containing copper, 3 ml to 5 ml potassium iodide solution followed by approximately 2 g ammonium thiocyanate. Titrate immediately with sodium thiosulphate standard volumetric solution. Towards the end point dilute with water to approximately 100 ml and add 2 ml starch indicator solution.

8.4.8 Determination of iron, chromium and copper by atomic absorption spectrophotometry

8.4.8.1 General. This procedure is included as an alternative to the wet/volumetric procedures, within the limit of up to 2.4 % of iron as Fe, up to 3.8 % of chromium as Cr and up to 2 % of copper as Cu, provided that the iron to chromium ratio does not exceed 12:1. The method is suitable for all treated textiles which have been proofed by normal iron, chromium and copper procedures.

8.4.8.2 Reagents. The following reagents are required.

8.4.8.2.1 Mixed acid. Spectroscopically pure, prepared by diluting 15 ml sulphuric acid (relative density 1.84) and 15 ml of orthophosphoric acid (relative density 1.75) to 100 ml.

8.4.8.2.2 Nitric acid. (relative density 1.42).

8.4.8.2.3 Stock iron solution. (4 mg/ml). Dissolve 1.000 g of pure iron wire in a minimum amount of mixed acid by boiling and the addition of a small amount of nitric acid (relative density 1.42). Continue the boiling until the appearance of a white precipitate. Dissolve in water and dilute to 250 ml in a standard flask.

8.4.8.2.4 Standard iron solution. (0.4 mg/ml) Dilute 50 ml of the stock iron solution to 500 ml in a standard flask.

8.4.8.2.5 Stock chromium solution. (4 mg/ml) Dissolve 1.000 g of pure chromium metal in a minimum amount of mixed acid by boiling and the addition of a small amount of nitric acid (relative density 1.42). Evaporate the solution down to a small volume and dilute to 250 ml in a standard flask.

8.4.8.2.6 Standard chromium solution. (0.4 mg/ml) Dilute 50 ml of the stock chromium solution to 500 ml in a standard flask.

8.4.8.2.7 Stock copper solution. (1000 mg/l). Dissolve 3.930 g of AR copper sulphate in water and dilute to 1 litre. Store in a polyethylene container.

8.4.8.2.8 Standard copper solution. (100 mg/l). Pipette 10 ml of the 1000 mg/l copper solution into a 100 ml volumetric flask and make up to the mark with water. Store in a polyethylene container.

8.4.8.3 Instrumental conditions. Instrumental conditions shall be as follows.

Wavelength: iron 248.33 nm; chromium 428.97 nm; copper 324.8 nm

Fuel: acetylene - air

8.4.8.4 Calibration solution

(a) *Iron and chromium.* Pipette into seven 500 ml standard flasks the following volumes of the standard iron and chromium solutions.

Standard iron solution (ml)
5 10 20 30 40 50 60

Standard chromium solution (ml)
10 20 30 40 50 60 70

To each flask add 10 ml mixed acid and make up to the mark with water. These solutions represent the following percentages by mass of iron and chromium.

Iron in sample	0.20	0.40	0.80	1.20	1.60	2.00	2.40
Chromium in sample	0.80	1.60	2.40	3.20	4.00	4.80	5.60

(b) *Copper.* To six 100 ml volumetric flasks, add the following by burette.

Standard copper solution (ml)
0.0 2.0 4.0 6.0 8.0 10.0

Make up to the mark with water. These solutions represent the following percentages by mass of copper in sample.

0.0 0.2 0.4 0.6 0.8 1.0.

8.4.8.5 Procedure

8.4.8.5.1 Iron and chromium. Weigh accurately about 2.5 g of the textile cut into 5 mm squares, transfer to a silica or porcelain crucible and partially cover with a lid. Ash over a low bunsen flame. Remove the lid and place the crucible in an electric furnace maintained at 580 °C to 600 °C. After 1 h, remove the crucible from the furnace and allow it to cool. Using a flat ended glass rod carefully break down the residue and replace the crucible in the furnace for a further period of one h, then allow it to cool in a desiccator. Transfer the residue into a 400 ml conical beaker.

Add 20 ml of mixed acid, using 3 ml to 4 ml of the reagent to wash out the crucible. Heat the contents of the flask on a hot plate to dissolve the ash, then evaporate to fuming. Add 5 ml nitric acid (relative density 1.42) dropwise, ensuring that brown fumes are evolved. Continue heating to remove all excess nitric acid, add 5 ml of water and again evaporate to fuming.

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