BRITISH STANDARD

BS 6648:1986

Incorporating Amendment No. 1

Methods for

Wet ashing of textile materials for subsequent determination of metal content

UDC 677.074.014.27:[543.832:54 – 32]:542.4

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Textiles and Clothing Standards Committee (TCM/-) to Technical Committee TCM/26, upon which the following bodies were represented:

British Carpet Manufacturers' Association Ltd.
Department of Trade and Industry (Laboratory of the Government Chemist)
International Wool Secretariat
Man-made Fibres Producers' Committee
Manchester Testing House
Ministry of Defence
Textile Research Council

This British Standard, having been prepared under the direction of the Textiles and Clothing Standards Committee, was published under the authority of the Board of BSI and comes into effect on 30 April 1986

The following BSI references relate to the work on this standard:
Committee reference TCM/26
Draft for comment 84/39977 DC

ISBN 0 580 15099 2

Amendments issued since publication

Amd. No.	Date of issue	Comments
7007	July 1992	Indicated by a sideline in the margin

Contents

Foreword

This British Standard has been prepared under the direction of the Textiles and Clothing Standards Committee.

A method for wet ashing of textile materials was included in the 1974 edition of BS Handbook No. 11 "*Methods of test for textiles*" as a recommended method. However, due to the wide usage of this method it has been decided that it be revised and published as a British Standard. The revised method is similar to method 3 of BS 2087-2.

The major changes from the method in Handbook No. 11 are that perchloric acid is only used if treatment with sulphuric acid and nitric acid fails to oxidize the specimen completely. An additional method has been included for determination of mercury which involves a special apparatus to minimize losses of mercury.

Where destruction of organic matter in a sample of textile material is required, and the ash is not to be determined, wet combustion possesses many advantages over dry ashing.

At the time of publication of this British Standard, no corresponding International Standard exists.

NOTE 1 The wet ashing method for the determination of mercury is based on method IV A published in *Official, Standardised and Recommended Methods of Analysis second edition,* 1973, edited by N. W. Hanson and published by the Society for Analytical Chemistry, and is included by permission of the Royal Society of Chemistry. Copies are available from the Royal Society of Chemistry, Burlington House, London W1V 0BN.

This British Standard calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

NOTE 2 See the Health and Safety at Work etc. Act 1974 and the Control of Substances Hazardous to Health Regulations 1988 (COSHH).

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 and 2, 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 British Standard describes two methods for wet ashing of textile materials for subsequent determination of metal content. Method A is a general method while method B is applicable for the determination of mercury.

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

CAUTION. To avoid hazard it is essential that the conditions described be carefully followed and carried out only by trained operators.

It is essential to wear safety goggles and to carry out the procedures in a fume cupboard.

2 Principle

The sample is heated under controlled conditions with a mixture of nitric acid and sulphuric acid and, if this fails to oxidize the specimen completely, perchloric acid.

3 Apparatus and reagents

3.1 *Kjeldahl flasks,* capacity 200 mL.

3.2 *Means for heating Kjeldahl flasks*

3.3 *Safety glass screen*

3.4 *Fume cupboard*

3.5 *Balance,* capable of weighing to an accuracy of 0.1 mg.

3.6 *Digestion apparatus* for the determination of mercury, as shown in Figure 1, comprising a flask of capacity 250 mL and a reservoir B having a capacity of 150 mL to 200 mL. The condenser is a double surface or spiral surface reflux type. The thermometer is calibrated for temperatures up to 200 °C. All the connections are made through standard ground glass joints.

3.7 *Nitric acid,* concentrated, relative density 1.42.

3.8 *Sulphuric acid,* concentrated, relative density 1.84.

3.9 *Perchloric acid,* 60 %, relative density 1.54.

3.10 *Water,* complying with BS 3978.

4 Method A. General wet ashing

Using the balance (**3.5**), weigh about 2 g of the textile and transfer it to a 200 mL Kjeldahl flask (**3.1**). Add 10 mL of sulphuric acid (**3.8**) followed by gradual addition of nitric acid (**3.7**) until there is no reaction on further addition of nitric 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 the organic matter is completely destroyed.

If the sample cannot be oxidized with nitric acid, allow the flask and contents to cool. Add 2 mL of perchloric acid (**3.9**) and continue to apply heat gradually.

Evaporate down to fuming, cool, then add 5 mL to 10 mL of distilled water and boil to remove nitric acid. Cool. The solution at this stage is ready for analysis.

5 Method B. Wet ashing for the determination of mercury

Transfer an accurately weighed amount of the textile (see note 1) to the flask of the digestion apparatus (**3.6**). Add a cooled mixture of 20 mL of water (**3.10**), 5 mL of concentrated sulphuric acid (**3.8**) and 50 mL of concentrated nitric acid (**3.7**) (see note 2) and a few anti-bumping granules or glass beads. Assemble the apparatus (see Figure 1) immediately and allow any initial reaction to subside. With tap A closed, heat the flask gently at first, collecting the distillate in reservoir B. When the temperature indicated by the thermometer reaches 116 °C (see note 3), run off the distillate through the drain tube C and collect it in a 500 mL measuring cylinder. Close tap A again, and continue to heat the flask and collect the distillate in the reservoir; when the oxidation mixture darkens, run a little of the distillate from the reservoir into the flask. Continue this procedure, maintaining a slight excess of nitric acid in the oxidation mixture, until the solution in the flask ceases to darken and fumes of sulphuric acid are evolved. Allow the solution in the flask to cool, run the distillate from the reservoir into the flask, then add the mixture to the reserved distillate in the measuring cylinder (see note 4), rinsing in with a small volume of water, and mix. Cool. The solution at this stage is ready for analysis.

NOTE 1 The mass of sample taken should be such that it contains between 0.5 µg and 5 µg of mercury.

NOTE 2 Sample masses up to about 10 g of dry solid can be oxidized by the procedure with 50 mL of nitric acid.

NOTE 3 The temperature of 116 °C is close to the boiling point of nitric acid.

NOTE 4 The volume of the mixed liquids is usually about 80 mL to 90 mL.

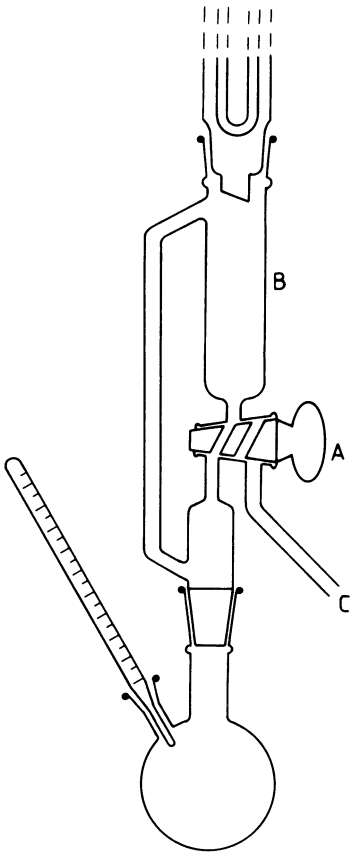

Figure 1 — Apparatus for wet ashing of textile material for the determination of mercury

Publications referred to

BS 2087, *Preservative treatments for textiles*[1)].
BS 2087-2, *Methods of test.*
BS 3978, *Water for laboratory use.*

[1)] Referred to in the foreword only.

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