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British Standard Method for

Determination of ash of textiles

Méthode de détermination de la teneur en cendres des textiles

Verfahren zur Bestimmung der Asche an Textilien

Foreword

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

This method 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 had been decided that it be revised and published as a British Standard.

The major change from the method in Handbook No. 11 is that the specimens are carbonized at a low temperature before incineration at 700 °C.

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

Compliance with a British Standard does not of itself confer immunity from legal obligations.

1 Scope

This British Standard describes a method for determination of ash of textiles.

NOTE. The title of the publication referred to in this standard is given on the inside back page.

2 Principle

An amount of the textile of known mass, previously dried at 105 ± 3 °C, is incinerated and the mass of the residual ash is determined.

3 Apparatus

3.1 *Drying oven*, controlled at 105 ± 3 °C.

3.2 *Silica or porcelain crucibles*, with fittings, of about 40 mL capacity.

3.3 *Desiccator* (vacuum type), containing a suitable desiccant, e.g. silica gel.

3.4 *Furnace*, controlled at approximately 700 °C, situated in a fume cupboard or under a fume hood.

3.5 *Balance*, having an accuracy of 0.1 mg.

3.6 *Bunsen burner.*

3.7 *Tripod.*

3.8 *Triangle*, made of pipe clay.

3.9 *Stoppered weighing bottle.*

4 Procedure

WARNING NOTE. It is important that fumes do not enter the laboratory, causing a potential build up of toxic volatile metals such as mercury, cadmium or antimony. Also acrylic fibres on ashing at 700 °C produce the highly toxic gas hydrogen cyanide.

'Determine the tared mass of a crucible with its lid, as follows. Prepare a crucible and lid (3.2) by heating in a furnace (3.4) at the requisite test temperature for at least 2 h. Allow to cool in the desiccator (3.3) for at least 1 h and determine the combined mass of the crucible and lid. Repeat the process of heating, cooling and weighing until the results of two consecutive weighings of the combined mass do not differ from each other by more than 0.5 mg, i.e. the mass is virtually constant.

Meanwhile, weigh approximately 2.5 g of the textile, cut into 5 mm squares in the case of fabrics, or 10 mm lengths in the case of fibres and yarns, in a weighing bottle (3.9) that has been similarly tared, along with its stopper, in the drying oven (3.1). Dry in the oven for 12 h to 18 h (conveniently, overnight) together with the tared stopper (see note 6).

Then, transfer the stoppered weighing bottle and its contents to the desiccator and allow to cool to ambient temperature for at least 1 h. After weighing, derive the oven-dry mass of the specimen by simple subtraction of the tare figure.

Transfer the specimen quantitatively to the tared crucible, replace the lid and pre-ash the contents of the crucible, supported by the pipe-clay triangle (3.8) and the tripod (3.7) above a low bunsen burner flame (3.6). Continue heating until the specimen is fully carbonized, taking care that ignition does not occur, as this may give rise to erratic results from indeterminate losses.

Place the covered crucible and contents at the entrance of a furnace operating at the requisite test temperature for a few minutes until all volatile matter has been evolved. Then, remove the crucible lid, put it to one side and close the door of the furnace. Allow incineration to proceed to completion (see note 4). Allow the crucible and contents to cool slightly, and replace the lid. Transfer the crucible, contents and lid to the desiccator and allow to cool for at least 1 h. Determine the mass of the crucible, lid and residue. Repeat the process of incineration, cooling and weighing until the results of two consecutive weighings do not differ from each other by more than 0.5 mg, i.e. the mass is virtually constant.'

NOTE 4. Certain metals can be lost under these conditions of heating. Mercury is lost readily, and zinc is lost at temperatures above 450 °C. Temperatures in excess of the general ashing temperature may occur locally in large samples and the general ashing temperature in such cases may have to be considerably reduced to avoid loss of certain metals. Lead is also lost under the specified conditions of heating unless sulphate ion is present.

NOTE 5. Silica and porcelain crucibles become etched during heating and cooling cycles over a prolonged period. Consequently, crucibles should be renewed fairly frequently.

'NOTE 6. The tared stopper should be kept apart from the weighing bottle throughout this part of the operation.' AMD 5720

5 Expression of results

'Express the ash as a percentage by mass, given by the following expression, AMD 5720

$$\frac{m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass of the oven-dry specimen (in g);
m_1 is the mass of the tared crucible and lid (in g);
m_2 is the mass of the crucible, lid and ash (in g).'

(c) the result obtained, expressed as in clause 5;

(d) the details of any deviation from the procedure.

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 29 November 1985.

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The following BSI references relate to the work on this standard:
Committee reference TCM/26 Draft for comment 84/39980 DC

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
Manchester Testing House
Man-made Fibres Producers' Committee
Ministry of Defence
Textile Research Council

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