BS 4603:1970

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Method for

# Determination of residue on ignition or ash of chemical products

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# Co-operating organizations

The technical committee which has been working under the supervision of the Chemical Divisional Council, and which is responsible for the preparation of this British Standard, consists of members representing the following industry standards committees:

Chemicals
Laboratory Apparatus
Pest Control Products
Petroleum
Pigments, Paints and Varnishes
Tar Products (other than Ammonia)

The committee entrusted with the preparation of this British Standard consists of members nominated by the technical committee.

This British Standard, having been endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the Executive Board on 30 June 1970

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# Foreword

This standard makes reference to the following British Standards:

BS 805, *Toluenes.*

BS 903, *Methods of testing vulcanized rubber — Part B13: Determination of ash and zinc oxide.*

BS 1416, *Methods for the sampling and analysis of rennet casein.*

BS 1417, *Methods for the sampling and analysis of acid casein.*

BS 1595, *isoPropyl alcohol.*

BS 1741, *The chemical analysis of liquid milk and cream.*

BS 1742, *Methods for the chemical analysis of condensed milk.*

BS 1743, *Methods for the chemical analysis of dried milk.*

BS 2869, *Petroleum fuels for oil engines and burners.*

BS 3235, *Test methods for bitumen.*

BS 3631, *Method for the determination of the ash content of paper.*

BS 4113, *Methods of test for sulphur.*

BS 4244, *Porcelain and silica crucibles.*

BS 4450, *Method for determination of ash from petroleum products.*

This British Standard has been prepared under the authority of the Chemical Divisional Council to provide a general method for the determination of residue on ignition or ash, applicable to a wide range of chemical products. It is hoped that this standard will provide a method that can be adopted not only by those preparing British Standard specifications for products but also by other bodies preparing methods of test and product specifications, and that the method described in the standard can eventually be adopted to replace existing standard methods that differ in detail.

Various methods published by other standardization bodies and in appendices to British Standards have been considered and a general method has been provided specifying temperatures of ignition, type and size of vessel to be used for various groups of products.

Determination of sulphated ash has not been considered.

The temperatures of ignition recommended for various types of product are wherever possible those agreed by ISO/TC 125 — Enclosures and conditioning for testing (formerly ISO/ATCO), for inclusion in an ISO draft proposal on a preferred series of temperatures. It has not been possible to reduce the number of different temperatures specified for ignition because these are based on long established practices in the industries concerned. Nevertheless, it is hoped that as new methods and specifications are prepared for these various products it will be possible to reduce the number of different temperatures specified.

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.

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**Summary of pages**

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, 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.



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## 1 Scope

This British Standard describes a method for the determination of residue on ignition or ash of chemical products. It is not suitable for samples producing ash that is fusible or volatile at the temperature of test, for which a sulphated ash procedure should be used.

## 2 Definition

For the purposes of this British Standard the following definition applies:

**residue on ignition** or **ash**

the carbon-free residue resulting from ignition

## 3 Apparatus

The following apparatus is required:

1) *Electric muffle furnace* capable of control to within ± 25 degC of the required test temperature (see Table 1).

2) *Basins or crucibles* of porcelain[1)], silica[1)] or preferably platinum of suitable size. The following sizes are recommended:

| | | |
|---|---|---|
| A. | Porcelain or silica crucible | 15 ml or 30 ml nominal capacity, as required. |
| B. | Porcelain or silica basin | 50 ml to 120 ml nominal capacity. |
| C. | Platinum crucible | 15 ml or 30 ml nominal capacity, as required. |
| D. | Platinum basin | 50 ml to 120 ml nominal capacity. |

NOTE 1 Platinum and platinum alloy vessels should not be used when the presence is suspected of lead, zinc, phosphates, or other substances that attack platinum at high temperatures. Heating the vessel in the reducing flame of a Bunsen burner should be avoided, and platinum-tipped tongs should be used for handling the vessels when they are hot.

NOTE 2 For samples expected to produce less than 0.1 % of ash, platinum vessels should be used, provided that lead or other substances liable to attack platinum are known to be absent.

NOTE 3 High results for ash are often obtained when substances containing phosphates are ashed in silica or porcelain vessels, owing to attack on the vessel. A suitable method for determining the ash of organic materials containing phosphorus is to destroy the organic matter by wet oxidation (e.g. with nitric and sulphuric acids in a glass flask) and then to evaporate the solution and heat the residue to constant mass in a platinum vessel. Evaporation of the residual sulphuric or phosphoric acids and heating to constant mass might cause some attack on the platinum, but this is usually slight.

3) *Desiccator* of appropriate size, containing suitable desiccant (see Table 1).

## 4 Materials

The following materials are required with certain samples (see **5.2**, Notes 1 and 2). When used they shall be tested to ensure freedom from ash:

1) *Toluene* complying with the requirements of BS 805[2)].

2) *isoPropyl alcohol*, complying with the requirements of BS 1595[3)].

3) *Paraffin wax*.

## 5 Procedure

**5.1** According to the product to be tested (see Table 1), select a suitable vessel from the list of recommended sizes given in **3** 2). Heat the vessel in the muffle furnace at the temperature of test for 10-15 minutes, allow it to cool sufficiently, transfer it to the desiccator and when cold weigh it to the nearest 0.1 mg. Weigh, to within ± 1 %, the quantity of sample required by the specification for the product being tested (or if not specified, see Table 1) into the vessel.

**5.2** Ignite the sample or, if necessary, heat the vessel and its contents gently over a bunsen flame *in a fume cupboard*, and ignite any flammable vapours.

**CAUTION. When testing highly flammable or explosive liquids, add the sample to the vessel in small portions, and burn each portion completely and cool the vessel before adding the next portion.**

NOTE 1 The burning of some highly flammable materials can be moderated by the addition of molten paraffin wax.

NOTE 2 If the sample contains sufficient moisture to cause foaming and loss of material when heated, the test should be discontinued and repeated on a fresh sample to which 1-2 ml of 99 % *iso*propyl alcohol is added before heating. If this procedure does not give satisfactory results, add 10-20 ml of an equivolume mixture of ash-free toluene and *iso*propyl alcohol, to a fresh sample in a basin of 125 ml capacity, and mix thoroughly. Place several strips of ashless filter paper in the mixture and heat *in a fume cupboard* and ignite any flammable vapours. When the paper begins to burn the greater part of the water will have been removed. When determining very low ash values it may be necessary to carry out a blank determination with this solvent mixture.

NOTE 3 All fine powders should be heated very slowly to drive off initial gases including moisture.

[1)] See BS 4244, "*Porcelain and silica crucibles*".

[2)] BS 805, "*Toluenes*".

[3)] BS 1595, "*isoPropyl alcohol*".

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**5.3** Continue heating until any vapours go on burning when the flame is withdrawn, and allow the sample to burn itself out or to evaporate to a residue. When the sample burns itself out, and provided that it can be clearly seen that no undecomposed organic matter remains, the vessel should be heated gently over a bunsen burner until all organic matter has either been driven off or been decomposed to carbon. It is important to see that the upper parts of the vessel are heated, otherwise a ring of undecomposed material may remain round the top of the vessel. Finally, ignite the residue in the muffle furnace at the temperature given in Table 1 until the ash is free from carbon.

NOTE In testing foodstuffs, if the carbon formed is difficult to ignite the ash may be moistened with water, dried and re-ignited.

**5.4** Allow the vessel to cool sufficiently, transfer it to the desiccator and, when cold, weigh it to the nearest 0.1 mg. Repeat the heating in the muffle furnace for 15 minute periods followed by cooling and weighing until two successive weighings do not differ by more than 0.5 mg.

If the mass of ash obtained is greater than 20 mg, the determination should be repeated on a smaller portion of the sample.

NOTE For certain products the ashes of which are chemically reactive or take up water preferentially, e.g. lime, the desiccant should be omitted.

## 6 Calculation

Calculate the mass of the residue as a percentage by mass of the original sample from the following formula:

$$\frac{100 \times A}{M}$$

where $A$ = mass, in grammes, of residue found

and $M$ = mass, in grammes, of sample taken.

## 7 Test report

Report the result as a percentage by mass or as required by the specification for the product being tested, indicating in the report the particular test conditions employed.



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**Table 1 — Recommended vessels and conditions of test**

Reference should be made to the specification for the product being tested, and where reference is made in this table to other British Standards, details specific to individual products or groups of products may be found therein. In the absence of such specific details, the table below may serve as a guide.

*Key to type of vessel* [see **3** 2) for further details]: A — Porcelain or silica crucible; B — Porcelain or silica basin; C — Platinum crucible; D — Platinum basin.

| Type of product to be tested | Vessel [see 3 2) and notes thereto] | Mass of sample | Temperature of ignition | Preferred desiccant |
|---|---|---|---|---|
| | | g | °C | |
| Antifreeze solutions | D | 20 | 630 ± 25 | |
| Bitumen (see BS 3235[a]) | A(30 ml), B(50 ml) C(30 ml) or D(50 ml) | Up to 100 g[b] | 775 ± 25 | none required |
| Carbon black | A | 2 | 500 ± 25[c] | anhydrous calcium chloride |
| Cellulose acetate | A or C(200 ml) | 10 | 600 ± 15 | |
| Cellulose acetate derivatives | D | 10 to 50 | 500 ± 25 | |
| Cereal products | | | 500 to 630 | |
| Crude tar/refined tar/pitch | A (30 ml silica) | 2 | 800 ± 25 | |
| Dairy products[d] | B or D | 10 | 500 to 550 | |
| Ethyl cellulose | C(30 ml) | 2 to 5 | 900 ± 25 | |
| Extenders and pigments | A(silica) | 2 to 5 | 1 000 ± 25 | |
| Fatty oils and fatty acids | B | 20 | 500 to 630 | |
| Fatty quaternary ammonium chlorides | B | 20 | 500 ± 25 | |
| General chemicals and rubber chemicals[e] | B or D | 5 | 800 ± 25 | silica gel |
| Glycols and polyglycols | B or D | 100 | 800 ± 25 | silica gel |



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**Table 1 — Recommended vessels and conditions of test**

| Type of product to be tested | Vessel [see 3 2) and notes thereto] | Mass of sample | Temperature of ignition | Preferred desiccant |
|---|---|---|---|---|
| | | g | °C | |
| Lac resin | A | 2 to 5 | 500 ± 25 | |
| Lubricating grease | A | 2 to 5 | 500 ± 25 | |
| Paper and board (see also BS 3631[e]) | A or C | 1 | 800 ± 25 | |
| Petroleum oils[f] | B or D | Up to 100[b] | 775 ± 25 | none required |
| Pharmaceutical products | B or D | 2 to 3 | 500 ± 25 | |
| Pigments | A | 5 | 1 000 ± 25 | |
| Protein products[g] | A | 2 to 5 | 500 to 550 | |
| Pyridine bases | B | 10 | 630 ± 25 | |
| Rosin and tall oil | D | 20 | 500 ± 25 | |
| Solvents | D | 50 | 630 ± 25 | |
| Sulphur (see BS 4113[h]) | B(silica) | 25 | 850 ± 50 | |
| Vulcanized rubber (see BS 903-B13[i]) | A(30 ml silica) | 2 | 600 ± 25 | |

[a] BS 3235, "*Test methods for bitumen*".

[b] According to expected yield.

[c] A temperature of up to 950 °C may be required for some types of carbon black, according to specification.

[d] See also BS 1741, "*The chemical analysis of liquid milk and cream*", BS 1742, "*Methods for the chemical analysis of condensed milk*" and BS 1743, "*Methods for the chemical analysis of dried milk*".

[e] BS 3631, "*Method for the determination of the ash content of paper*".

[f] Distillate and residual fuel oils, crude oils, lubricating oils, waxes, and other petroleum products free from ash-forming derivatives (see also BS 2869, "*Petroleum fuels for oil engines and burners*" and BS 4450, "*Method for determination of ash from petroleum products*").

[g] See also BS 1416, "*Methods for the sampling and analysis of rennet casein*" and BS 1417, "*Methods for the sampling and analysis of acid casein*". It may be necessary to add magnesium acetate to phosphoroproteins containing insufficient calcium or magnesium to bind the phosphorus.

[h] BS 4113, "*Methods of test for sulphur*".

[i] BS 903, "*Methods of testing vulcanized rubber*", Part B13: "*Determination of ash and zinc oxide*".



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