BRITISH STANDARD

BS 7530-1: 1992

CONFIRMED
DECEMBER 2007

Methods for

Froth flotation testing of hard coal —

Part 1: Laboratory procedure

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Solid Mineral Fuels Standards Policy Committee (SFC/-) to Technical Committee SFC/5, upon which the following bodies were represented:

British Coal Corporation

Coal Preparation Plant Association

Minerals Engineering Society

This British Standard, having been prepared under the direction of the Solid Mineral Fuels Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on
31 January 1992

The following BSI references relate to the work on this standard:
Committee reference SFC/5
Draft for comment 87/51450 DC

ISBN 0 580 20174 0

Amendments issued since publication

Amd. No.	Date	Comments

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Foreword

This Part of BS 7530 has been prepared under the direction of the Solid Mineral Fuels Standards Policy Committee. Part 2 of the standard will specify a method of evaluating flotation response.

This Part is related to ISO 8858-1:1990, published by the International Organization for Standardization (ISO). The principal difference is that the apparatus and procedure are described in more general terms in this standard and are not based on a particular design of apparatus, as is the case in ISO 8858-1.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 6, 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.

0 Introduction

The froth flotation of coal has widespread application for the concentration of fine coal particles and their separation from mineral matter. The response of coal to the froth flotation process is measured initially by a laboratory-scale test. Although the principles on which such laboratory tests are based are generally similar, the type of equipment and the techniques used vary considerably. The purpose of this Part of BS 7530 is to provide a standard method of test by which preliminary comparisons of the froth flotation characteristics of different coals can be made. Such comparisons are particularly important for exploration programmes. This Part of BS 7530 also serves as an introduction for operators who are not familiar with the techniques (and problems) associated with the froth flotation of coals in the laboratory.

The method described provides a means of evaluating the general flotation characteristics of a coal under a set of specified conditions and will not necessarily indicate the full flotation potential of that coal. The flotation characteristics of coals are sensitive to changes in flotation conditions. These conditions can be changed by varying such basic parameters as flotation time, particle size distribution of the feed, reagent and reagent dosage. Separate flotation tests are required to assess the effect of varying these parameters to determine the best flotation conditions for a particular coal.

1 Scope

This Part of BS 7530 describes a method for the froth flotation testing of a fine coal, of particle size less than 0.5 mm, in the laboratory. It is applicable to coal in powder form or in the form of a slurry.

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

2 Definitions

For the purposes of this Part of BS 7530, the definitions given in BS 3552 apply.

3 Principle

A test portion of coal is mixed with water to form a suspension in the flotation cell, or a test portion of a slurry is added to the cell and diluted. A flotation agent is added, the suspension is conditioned, and then air is introduced to the cell while agitation is maintained by means of an impeller. The concentrate and the tailings are collected separately and the yield and ash are determined for each.

4 Reagents

4.1 *General*. During the test, use only reagents of recognized analytical grade and only water complying with grade 3 of BS 3978.

4.2 *Flotation agent*. Prepare a mixture of 80 % (*V*/*V*) of *n*-dodecane and 20 % (*V*/*V*) of 4-methylpentan-2-ol.

NOTE A stock solution of this mixture may be kept for use in several tests.

5 Apparatus

5.1 *Flotation machine*. A mechanical impeller-type flotation machine, designed for laboratory-scale flotation testing, comprising the following.

a) A flotation cell, of capacity 2.5 L to 10 L, made from an inert material such as stainless steel, glass or perspex.

b) An impeller assembly, capable of being driven so that the periphery of the impeller moves at a speed of 5.7 m/s. The impeller shall be positioned centrally with a clearance of 6 ± 1 mm above the bottom of the cell.

c) A means of supplying air to the impeller.

A typical flotation machine is shown in Figure 1 and Figure 2.

5.2 *Constant level control*, for maintaining the pulp at a constant level in the flotation cell during the test.

NOTE Alternatively, the level may be kept constant by the manual addition of water.

5.3 *Two scrapers,* for removing the froth from the entire surface of the pulp.

5.4 *Graduated micro-pipette or micro-syringe,* for adding the flotation agent.

5.5 *Timing device*, accurate to 1 s and capable of being zeroed and started as required.

5.6 *Balance*, capable of weighing to the nearest 0.1 g.

5.7 *Oven*, capable of maintaining a temperature within the range 105 °C to 110 °C.

5.8 *Set of test sieves*, complying with BS 410, for carrying out a size analysis of the test sample.

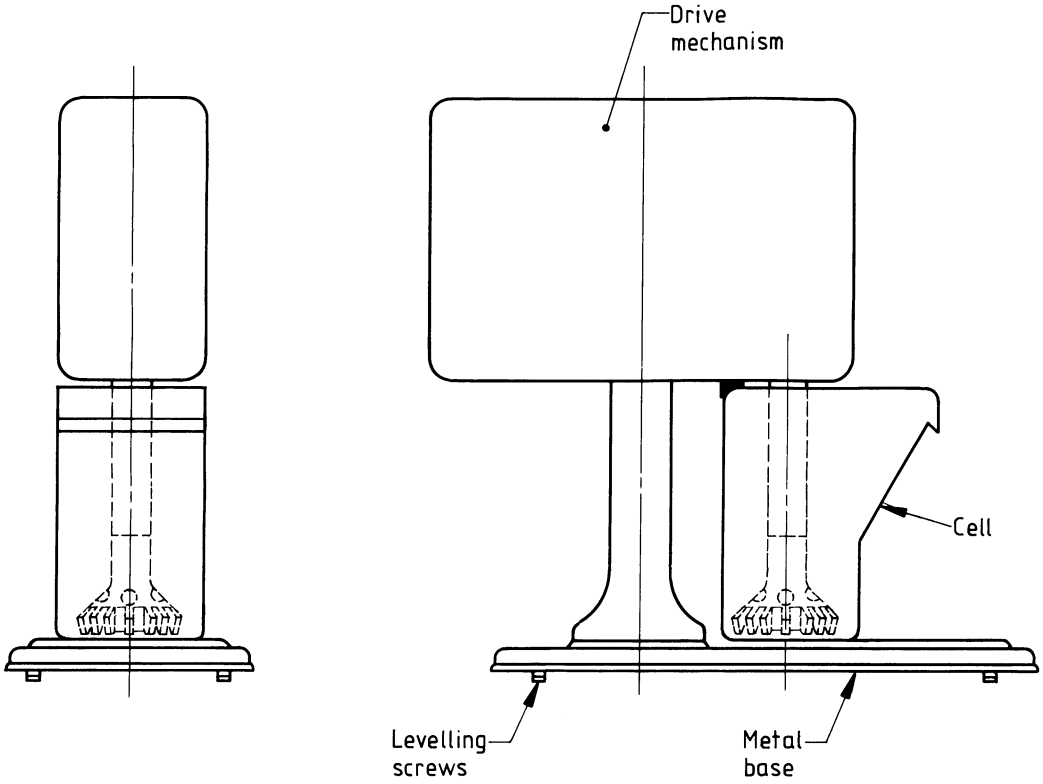

Figure 1 — Typical flotation machine

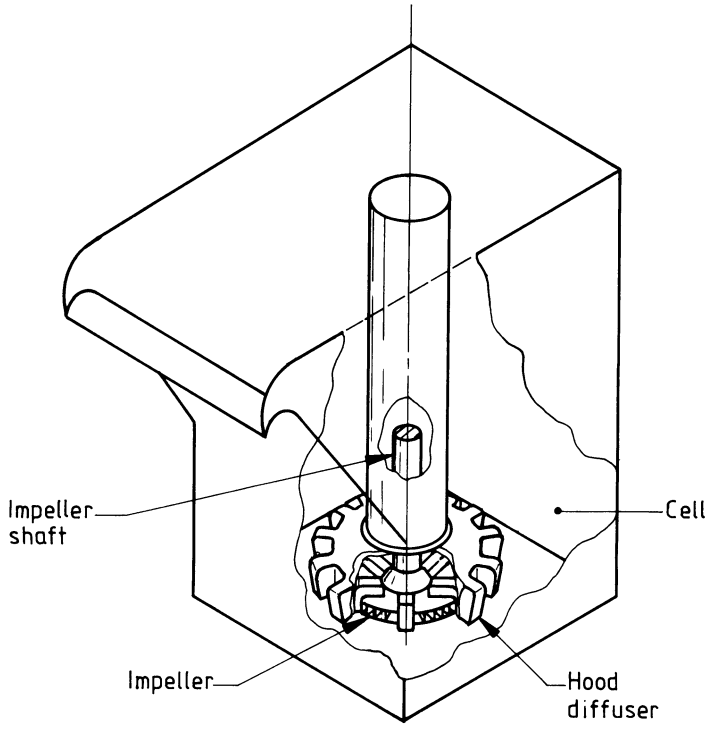

Figure 2 — Detail of flotation cell and impeller

6 Sampling and preparation of test sample

Take a gross sample which is at least five times the mass required for a single flotation test (see **9.1**).

If the gross sample is in powder form, take representative subsamples for carrying out preliminary tests (see **7.2** to **7.4**).

If the gross sample is in the form of a slurry, allow it to settle for at least 12 h and then decant the supernatant water. Thoroughly mix the thickened slurry and take representative subsamples for the determination of its solids content and other tests (see clause **7**).

NOTE 1 The history and method of preparation of the samples can affect the flotation characteristics of the coal. The origin of the sample should be recorded and care should be taken to ensure that samples for comparative tests are prepared in the same manner. Since replicate tests are required together with subsampling for size analysis and other tests, care should be taken in the mixing and subdivision of the original sample. When applicable, sampling and division of samples should be carried out in accordance with BS 1017-1.

NOTE 2 The use of chemical additives to enhance the settlement of a slurry and the use of heat to drive off water should not be employed because such practices can affect the flotation characteristics of the coal.

7 Preliminary tests

7.1 Determination of solids content of a thickened slurry

Take a subsample of about 500 mL of the thickened slurry (see clause **6**) and weigh it, to the nearest 0.1 g, using the balance (**5.6**).

Dry the slurry in the oven (**5.7**) at 105 °C to 110 °C until the difference between successive weighings, at an interval of at least 15 min drying time, does not exceed 0.1 g.

NOTE 1 Drying in the oven for 24 h is usually adequate.

NOTE 2 The thickened slurry may be flocculated and filtered first, prior to drying in the oven, provided that all the solids are recovered.

Weigh the dried solids, to the nearest 0.1 g, and calculate the solids content of the thickened slurry as a percentage by mass.

7.2 Size analysis

Carry out a full size analysis of a subsample (see clause **6**) by the wet sieving method described in BS 1016-17, using the test sieves (**5.8**).

7.3 Determination of moisture content

If the test sample is in powder form, determine its moisture content by the method described in BS 1016-104.1. Also determine the moisture content of each of the size fractions (see **7.2**), so that results can be calculated on a dry basis.

7.4 Determination of ash

Determine the ash of the test sample and of each of the size fractions (see **7.2**) by the method described in BS 1016-104.4.

8 Flotation test conditions

NOTE Some coals may not react under the flotation conditions specified in this standard. This does not necessarily mean that the coal is not amenable to froth flotation. In such a case further experimental work would be necessary to determine suitable flotation conditions.

8.1 Test temperature

The test temperature shall be 20 ± 10 °C.

8.2 Solids content of the pulp

The pulp in the flotation cell shall contain 100 ± 5 g/L of solids, on a dry basis.

8.3 Pulp level

The level of the pulp shall be 20 ± 2 mm below the overflow lip of the flotation cell when the impeller is rotating at the operating speed and the air valve is closed.

8.4 Volume of flotation agent

The volume of flotation agent added to the pulp shall be 1 mL/kg of dry solids (see **8.2**).

8.5 Air flow rate

The air flow rate shall be 120 ± 12 L/min per square metre of surface area of the pulp.

NOTE It is recommended that the air flow be controlled by means of a needle valve coupled to a flowmeter and that a separate on/off valve be provided.

9 Procedure

9.1 Test portion

If the test sample is in powder form, calculate the mass of test portion required from the measured volume of the flotation cell (**5.1**), the moisture content of the test sample (see **7.3**) and the specified solids content of the pulp (see **8.2**).

In the case of a slurry, calculate the required mass of thickened slurry from the volume of the flotation cell, the solids content of the slurry (see **7.1**) and the specified solids content of the pulp (see **8.2**).

Weigh the test portion, to the nearest 0.1 g, using the balance (**5.6**).

9.2 Flotation test

If the test portion (see **9.1**) is in powder form, half-fill the flotation cell (**5.1**) with water and start the impeller rotating at its operating speed, so that the periphery of the impeller moves at 5.7 m/s, with the air inlet closed. Gradually add the test portion to the flotation cell and top up with water to the required level (see **8.3**). Continue mixing for 10 min.

If the test portion is a thickened slurry, transfer it to the flotation cell and add water to the required level. With the air inlet closed, start the impeller rotating at its operating speed and mix the pulp for 2 min.

Add the required volume (see **8.4**) of the flotation agent (**4.2**), by means of the micro-pipette or micro-syringe (**5.4**), beneath the surface of the pulp and start the timing device (**5.5**). Condition for 1 min with the air inlet closed.

Open the air inlet and pass air through the flotation cell at the required rate (see **8.5**).

Every 5 s, move the scrapers (**5.3**) at a steady rate across the surface of the pulp and collect the froth, ensuring that none of the pulp is collected with it.

NOTE 1 As flotation progresses, the frequency of froth removal may be reduced.

NOTE 2 A significant amount of floated coal may adhere to the sides of the flotation cell above the level of the pulp. This coal should be washed on to the surface of the pulp, using a minimal amount of water, so that it can be removed by the scrapers.

Maintain the pulp at the required level manually or by means of the constant level control (**5.2**). Collect the total froth produced over a flotation period of 3 min and then stop the impeller. Remove any particles adhering to the sides of the cell and the impeller standpipe with a small brush and wash them into the concentrate of coal particles collected with the froth. Remove any particles remaining on the surface of the pulp by means of the scrapers and add them to the concentrate. Then wash any material remaining on the scrapers or on the cell lip into the concentrate with water. Collect the pulp as the tailings and wash any material remaining on the impeller into the tailings. Dewater and dry the concentrate and the tailings separately. Weigh the concentrate and the tailings, to the nearest 0.1 g, using the balance (**5.6**) and repeat the operations of drying and weighing until the difference between successive weighings, at an interval of at least 15 min drying time, does not exceed 0.1 g. Record the final mass, to the nearest 0.1 g, in each case.

NOTE 3 Flocculants may be added as a filtration aid, provided that the products are not required for additional flotation tests. In general, flocculants have an adverse effect on flotation.

NOTE 4 Drying in an oven should be avoided if caking tests, cooking tests or certain other special tests are to be carried out subsequently, or if the products are prone to excessive oxidation. In these cases, the material should be air-dried and the moisture content of the air-dried product should be determined by the method described in BS 1016-104.1 so that its mass can be calculated on a dry basis.

Determine the ash of both the concentrate and the tailings by the method described in BS 1016-104.4.

NOTE 5 Other properties of the concentrate and tailings may be determined as required.

9.3 Validity of the test

For a flotation test to be valid, the sum of the mass of the concentrate and the mass of the tailings, both on a dry basis, should not differ by more than 3 % (*m*/*m*) from the original mass of solids in the test portion, also on a dry basis. Otherwise, disregard the results and carry out a further test.

9.4 Number of tests

Repeat the procedure described in **9.2** at least once, using a further test portion.

10 Expression of results

10.1 Preliminary tests

Report the results of the size analysis (see **7.2**) as the percentage by mass of each size fraction, calculated on a dry basis, using the moisture contents determined as described in **7.3**. For calculation of results on a dry basis see BS 1016-16. Report the ash of the test sample and of each size fraction (see **7.4**) as percentages by mass on a dry basis.

10.2 Flotation test

Calculate the amount of coal reporting to the concentrate (i.e. the yield) as a percentage by mass, from the following formula

$$\frac{m_C}{(m_C + m_T)} \times 100$$

where

m_C is the mass of the dried concentrate (in g);

m_T is the mass of the dried tailings (in g).

Calculate the amount of material reporting to the tailings, as a percentage by mass, from the following formula

$$\frac{m_T}{(m_C + m_T)} \times 100$$

Report each of these results, to the nearest 0.1 %, as the arithmetic mean and the range of the values determined in the individual tests.

Calculate the ash in the concentrate and the tailings as percentages by mass on a dry basis and report each of these results as the arithmetic mean and the range of the values determined in the individual tests.

11 Precision

11.1 Repeatability

The results of duplicate tests, carried out at different times in the same laboratory by the same operator with the same apparatus on representative portions taken from the same test sample, should not differ by more than 5 % (*m*/*m*) for the yield and 0.5 % (*m*/*m*) for the ash in the concentrate.

11.2 Reproducibility

No value for reproducibility can be quoted for tests carried out in different laboratories because insufficient data are available.

12 Test report

The test report shall include the following:

a) the date of the test;

b) the complete identification of the sample;

c) the history of the sample;

d) the reference to the method used, i.e. BS 7530-1:1992;

e) the mass of solids in the test portion expressed on a dry basis (in g);

f) the results of the size analysis expressed in accordance with **10.1**;

g) the ash of the test sample and of each size fraction expressed in accordance with **10.1**;

h) the number of flotation tests carried out;

i) the mass distribution between the concentrate and the tailings expressed in accordance with **10.2**;

j) the ash of the concentrate and the tailings expressed in accordance with **10.2**;

k) the results for any other properties determined;

l) any unusual features noted during the tests;

m) any operation not specified in this standard, or the standards to which reference is made, or regarded as optional.

NOTE A recommended layout of a form for a test report is given in Appendix A.

Appendix A Recommended layout of a form for a test report

Froth flotation testing

Date of report: ..... Operator:.....

Date of test:.....

Sample identification:.....

.....

Sample history:.....

Sample size analysis

Particle size square aperture	Mass	Ash
µm	% (*m*/*m*)	% (*m*/*m*)
≥ 500		
< 500 ≥ 250		
< 250 ≥ 125		
< 125 ≥ 63		
< 63		

Mass of test portion =

Ash of test portion (% [*m*/*m*)] =

Results

	Concentrate	Tailings
Mass distribution, % (*m*/*m*)		
Ash, % (*m*/*m*)		
Other properties		

All results and analyses expressed on dry basis

Comment: .....

.....

.....

.....

Publication(s) referred to

BS 410, *Specification for test sieves.*
BS 1016, *Methods for analysis and testing of coal and coke.*
BS 1016-16, *Methods for reporting results.*
BS 1016-17, *Size analysis of coal.*
BS 1016-104, *Proximate analysis.*
BS 1016-104.1, *Determination of moisture content of the general analysis sample of coal.*
BS 1016-104.4, *Determination of ash.*
BS 1017, *Sampling of coal and coke.*
BS 1017-1, *Method for sampling of coal.*
BS 3552, *Glossary of coal preparation terms.*
BS 3978, *Specification for water for laboratory use.*
ISO 8858-1, *Hard coal — Froth flotation testing — Part 1: Laboratory procedure*[1)].

[1)] Referred to in the foreword only.

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