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Hard coal — Froth flotation testing —

Part 1: Laboratory procedure

Houille — Essais de flottation —

Partie 1: Méthode de laboratoire

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 1, *Coal preparation: Terminology and performance*.

This second edition cancels and replaces the first edition (ISO 8858-1:1990), of which it constitutes a minor revision. It also incorporates the Technical Corrigendum ISO 8858-1:1990/Cor.1:2001.

A list of all parts in the ISO 8858 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The froth flotation of coal has widespread application for the concentration and separation of fine coal particles from mineral matter. The response of coal to the froth flotation process is initially measured by a laboratory scale test. Although the principles used for the laboratory tests are generally similar, the precise type of equipment and techniques used vary considerably.

The procedure for the laboratory froth flotation test sets out, in detail, the type of equipment to be used and the methods to be adopted. The purpose of this procedure is to provide a standard method of test by which a preliminary evaluation of the froth flotation characteristics of a coal can be compared. This need is particularly important for exploration programmes. This document also serves as an introduction for operators who are not familiar with the techniques (and problems) associated with the laboratory froth flotation of coals.

Annex A of this document is for information only.

Hard coal — Froth flotation testing —

Part 1: Laboratory procedure

1 Scope

This document sets out a laboratory procedure for the froth flotation testing of fine coal, e.g. coal of particle size less than 0,5 mm. The procedure provides a means of evaluating the general flotation characteristics of a coal under a set of specified standard 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 basic parameters such as flotation time, reagent and dosage rate. Separate flotation tests are used to assess the effect of varying these parameters to determine the best flotation conditions for a particular coal. A method of evaluating flotation response will be given in a separate standard.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1171, *Solid mineral fuels — Determination of ash*

ISO 11722, *Solid mineral fuels — Hard coal — Determination of moisture in the general analysis test sample by drying in nitrogen*

ISO 1213-1, *Solid mineral fuels — Vocabulary — Part 1: Terms relating to coal preparation*

ISO 13909-1, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*

ISO 13909-2, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*

ISO 13909-3, *Hard coal and coke — Mechanical sampling — Part 3: Coal — Sampling from stationary lots*

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 18283, *Hard coal and coke — Manual sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
collector
collecting agent
reagent added to a pulp to bring about adhesion between solid particles and air bubbles

3.2
flotation concentrate
clean product recovered in froth flotation

3.3
conditioning
preparatory stage in the flotation process in which the reagents are brought into intimate contact with the solids of the pulp

3.4
frother
frothing agent
reagent used to control the size and stability of the air bubbles in the flotation process

3.5
froth flotation
process for cleaning fine coal in which the coal, with the aid of a reagent or reagents, becomes attached to air bubbles in a liquid medium and floats as a froth

3.6
pulp
mixture of solid particles and water

3.7
flotation tailings
reject from froth flotation cells

4 Principle

Mixing of a coal sample with water to form a suspension in the flotation cell. Addition of collector and frother and introduction of air. Separate recovery of the concentrate and tailings and determination of the yield and ash of each.

NOTE 1 The use of chemical additives or heat can affect the flotation characteristics of the coal.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Collector, n-Dodecane.

5.2 Frother, Methyl iso-butyl carbinol (MIBC), (4-Methylpentan-2-ol), 0,14 % (V/V) aqueous solution.

6 Apparatus

The apparatus shall be a mechanical impeller type flotation machine (see Figure 1) with the following specifications.

6.1 Flotation cell manufactured from stainless steel.

Figure 2 shows the dimensions of the flotation cell. The capacity of the cell with the deflector block and impeller in place is approximately 3,5 l.

It has been shown that the results obtained from laboratory froth flotation tests are very dependent on the procedure used to remove the concentrate from the surface of the pulp. For this reason, a deflector block manufactured from plastics material (see Figure 3) is used to guide the concentrate to a rectangular area in front of the impeller housing. The concentrate can then be removed by means of a scraper (see Figure 4) which is designed to cover the full width of the cell to a constant depth.

In order to establish the volume of pulp which can be contained in the flotation cell, the deflector block is fitted into the cell and water is added to the flotation cell to about 20 mm to 30 mm below the required pulp level. The impeller is started (air valve off) and additional water is added to the required mark (see 8.4). The impeller is stopped, the deflector block removed, and the volume of water in the flotation cell is measured.

6.2 Impeller assembly, of stainless steel and capable of rotation at frequencies up to 1 500 revolutions per minute (equivalent to a speed of 5,7 m/s at the periphery). The dimensions of the impeller and diffuser are shown in Figures 5 and 6.

The lower face of the impeller shall be positioned no more than 5 mm from the base of the cell.

Impellers constructed of other materials may be used, provided that it can be shown that the characteristics of flotation for the cell are similar to those obtained when a stainless steel impeller is used. To achieve this, different impeller rotational frequencies may be used if necessary.

6.3 Constant level control, maintaining the level of the pulp in the cell during the flotation test.

6.4 Scraper, of the design shown in Figure 4. Its clearance at the sides of the cell shall be minimal and should not exceed 2 mm. The scraper shall be of such dimensions that it clears the pulp level (as defined in 8.4) by not more than 2 mm.

6.5 Calibrated micro-syringe, for the addition of the collector (5.1).

6.6 Pipette, of capacity 25 ml, complying with ISO 648, for the addition of the frother solution (5.2).

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

6.8 Test sieves, complying with ISO 565, for the particle size analysis of the feed.

7 Sampling

The history and method of preparation of samples can affect considerably the flotation characteristics of the coal. The origin of the sample should be recorded and care should be taken to ensure that samples for comparison purposes are prepared in a similar manner. Since repeat tests are required together with sub-sampling for particle size and moisture determination, great care should be taken in mixing and subdivision of the original sample. Where applicable, all sampling and subdivision of samples shall be carried out in accordance with the procedures specified in ISO 13909-1 to ISO 13909-4 and ISO 18283 as appropriate.

The quantity of gross sample required is at least five times the quantity required for a single flotation test. The latter quantity can be determined by reference to 8.3.

Where the sample for flotation testing is obtained in pulp (or slurry) form, it will be necessary to separate the solids from the water before the procedures laid down in this document can be applied. Dewatering shall be by natural settlement followed by decantation and filtration. No chemicals or heat

shall be used in the dewatering process as they may affect the surface properties of the materials and change their flotation characteristics.

The results of the flotation test are influenced by the particle size and a complete particle size analysis of the coal, carried out according to ISO 1953, should be reported (see Annex A). A sub-sample should also be retained for the determination of ash and other parameters as required (see 9.2).

8 Flotation conditions

8.1 Operating temperature

The operating temperature shall be 25 °C ± 10 °C.

8.2 Water

Water complying with Clause 5 shall be used.

8.3 Solids content

The solids content of the sample to be tested shall be 100 g of solids (on a dry basis) per litre of pulp. For example, when the effective cell volume is 3,5 l the sample mass is 350 g of dry solids plus any associated moisture as previously determined, see 9.1.

8.4 Pulp level

The pulp level shall be 20 mm ± 2 mm below the overflow lip of the cell when the impeller is rotating at 1 500 revolutions per minute and the air is off.

8.5 Collector

The quantity of collector (5.1) added shall be equivalent to 1 ml/kg of dry solids. For example, for a cell volume of 3,5 litres, a volume of 0,35 ml of collector will be required.

8.6 Frother

The quantity of frother (5.2) to be added shall be 0,1 ml of MIBC per kilogram of dry solids. For example, for a cell volume of 3,5 l, a volume of 25 ml of the solution (5.2) will be required.

8.7 Air flow rate

The air flow rate shall be 4 l/min ± 0,4 l/min. It is recommended that this flow rate be controlled by the use of a needle valve and measured by means of a flowmeter, and that a separate on/off valve be provided.

9 Procedure

9.1 From a representative sub-sample, determine the moisture content and ash percentage of the coal sample (Clause 7) by the procedures specified in ISO 11722 and ISO 1171 respectively. Calculate the mass of coal required for the flotation test from the measured cell volume (6.1) and the specified solids content of the sample to be tested (8.3).

9.2 From the coal sample (Clause 7), take representative sub-samples of appropriate mass for particle size analysis (and determination of other parameters as required), and for the individual flotation tests.

External factors such as exposure of a coal sample to heat or prolonged exposure to atmosphere can severely affect the flotation characteristics of coal. In order to assess the reliability and repeatability of flotation tests, at least one repeat test should be carried out on any one coal sample.

9.3 Gradually transfer a flotation test sub-sample into the cell containing 1 000 ml of water, with the impeller rotating at 1 000 revolutions per minute and the air inlet closed. Mix for 2 min, slowly add more water and increase the rotational frequency until the required level and a frequency of 1 500 revolutions per minute are reached. Agitate for a further 2 min.

NOTE The purpose of this procedure is to ensure thorough wetting of the sample. Variations in nominated conditions are permissible.

9.4 Add the collector (5.1), beneath the surface of the pulp by means of a calibrated micro-syringe (6.5) and start the timing device. Condition for 1 min, then add the frother solution (5.2) beneath the sur face of the pulp using a pipette (6.6). Open the air valve after an additional 10 s of conditioning. Open the tap connecting the cell to the constant level control as concentrate is removed.

A significant amount of floated coal may adhere to the sides of the cell above the water level. Since this coal has been floated it should be washed back into the concentrate so that it may be removed from the cell by the scraper. A minimal amount of water should be used.

9.5 Every 15 s move the scraper through the concentrate and collect a froth increment. The recessed edge of the scraper shall rest on the top edge of the cell and the movement of the scraper should be gradual, to ensure that pulp is not collected with the froth.

9.6 Collect the total froth produced over a period of 3 min frothing time.

9.7 At the end of 3 min, stop the impeller and restore to the concentrate any particles adhering to the sides of the cell and the impeller housing. A small paint brush may be useful for this purpose. Then remove the concentrate from the surface of the pulp by means of the scraper.

Turn off the valve to the constant level control tank. Any material remaining on the scraper or on the cell lip should be washed into the concentrate using water. Any material remaining on the impeller or deflector block should be washed into the tailings.

9.8 Dewater and dry the concentrate and tailings separately to constant mass [see a) below] and weigh. Determine the percentage of ash (and any other parameters required) of each in accordance with the appropriate International Standard.

a) Where special tests are to be made on the concentrate, it may be necessary for it to be air-dried. In such cases the moisture content of the concentrate should be determined to enable the results to be calculated to a dry basis.

b) Do not oven-dry any sample on which caking or coking tests are to be carried out or samples which are prone to excessive oxidation. In these cases, air-dry the sample and determine the moisture content of the air-dried product on a sub-sample.

c) 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.

9.9 The procedures described in 9.3 to 9.8 should be repeated at least once (see 9.2, second paragraph).