
**Hard coal — Froth flotation testing —
Part 3:
Release evaluation**

*Houille — Essais de flottation —
Partie 3: Évaluation des émissions*



Reference number
ISO 8858-3:2004(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8858-3 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 1, *Coal preparation: Terminology and performance*.

ISO 8858 consists of the following parts, under the general title *Hard coal — Froth flotation testing*:

- *Part 1: Laboratory procedure*
- *Part 2: Sequential evaluation*
- *Part 3: Release evaluation*

Introduction

The froth flotation of coal has widespread application for the recovery of fine coal particles and their separation from unwanted mineral matter. The response of coal to the froth flotation process is measured initially by a laboratory scale test. ISO 8858-1:1990 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 potential of that coal. It is accepted that variation of the many parameters in the froth flotation process can be used to effect the beneficiation of the product. This part of ISO 8858 describes methods of applying certain test conditions to reveal the relative response of coals to the flotation process.

To facilitate the determination of how a particular coal will respond to froth flotation, a method of controlling the recovery of froth is used. This is done by inhibiting the process to a degree, determining the rate at which coal reacts and by progressively sampling the froth. This method provides a basis to vary test parameters to explore any special flotation characteristics.

This part of ISO 8858 is applicable to a wide range of coals and provides a method of comparison of flotation behaviour, and will facilitate the exchange of information relating to specific performance. It is expected that the use of this method will provide a tool for comparison of flotation characteristics.

From the results of the standard test, it is possible to draw evaluation curves similar to those obtained from the float and sinking of coal.

Because of the high reproducibility of results, the test represents a reliable means of comparing the flotation response of different samples.

Hard coal — Froth flotation testing —

Part 3: Release evaluation

1 Scope

This part of ISO 8858 describes a laboratory method for release evaluation of the froth flotation characteristics of hard coal, of particle size less than 0,5 mm. It is applicable to coal in powder form or in the form of a slurry.

2 Normative references

The following referenced documents are indispensable for the application 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 1213-1:1993, *Solid mineral fuels — Vocabulary — Part 1: Terms relating to coal preparation*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 8858-1:1990, *Hard coal — Froth flotation testing — Part 1: Laboratory procedure*

3 Terms and definitions

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

4 Principle

A test portion of coal is mixed with water to form a suspension in the flotation cell, or a test portion of slurry is added to the cell and diluted. A collector and frother are added, the suspension is conditioned and air is then introduced to the cell while agitation is maintained by means of an impeller.

To study differing flotation characteristics, the flotation operation is inhibited in two ways, relating to reagent dosage rate in the first stage of the test and to air flow rate in the second stage.

A number of froth increments are obtained which give spaced points for comparison.

5 Reagents

Use only reagents of recognized analytical grade and the following.

5.1 Water, conforming to grade 3 of ISO 3696.

5.2 Collector, undiluted *n*-dodecane.

5.3 Frother.

Prepare a solution of 0,1 % (V/V) 4-methylpentan-2-ol (formerly called methyl isobutyl carbinol or MIBC) by diluting 1 ml of MIBC to 1 litre using water (see 5.1). All dosages of MIBC stated refer to this solution.

6 Apparatus

6.1 Flotation machine

As described in 6.1 of ISO 8858-1:1990, consisting of a mechanical impeller-type flotation machine designed for laboratory-scale flotation testing, comprising the following.

- a) A flotation cell, of capacity approximately 3,5 litres, made from an inert material, such as stainless steel, glass or polymethyl methacrylate.
- b) An impeller assembly, capable of being driven so that the periphery of the impeller moves at a speed of approximately 5,7 m/s. The impeller shall be positioned centrally with a clearance of no more than 5 mm from the base of the cell.
- c) A means of supplying air to the impeller.

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

6.2 Air flowmeter, with a needle valve to control the air flow rate to the flotation cell.

6.3 Constant level device, for maintaining the pulp at a constant level during the test.

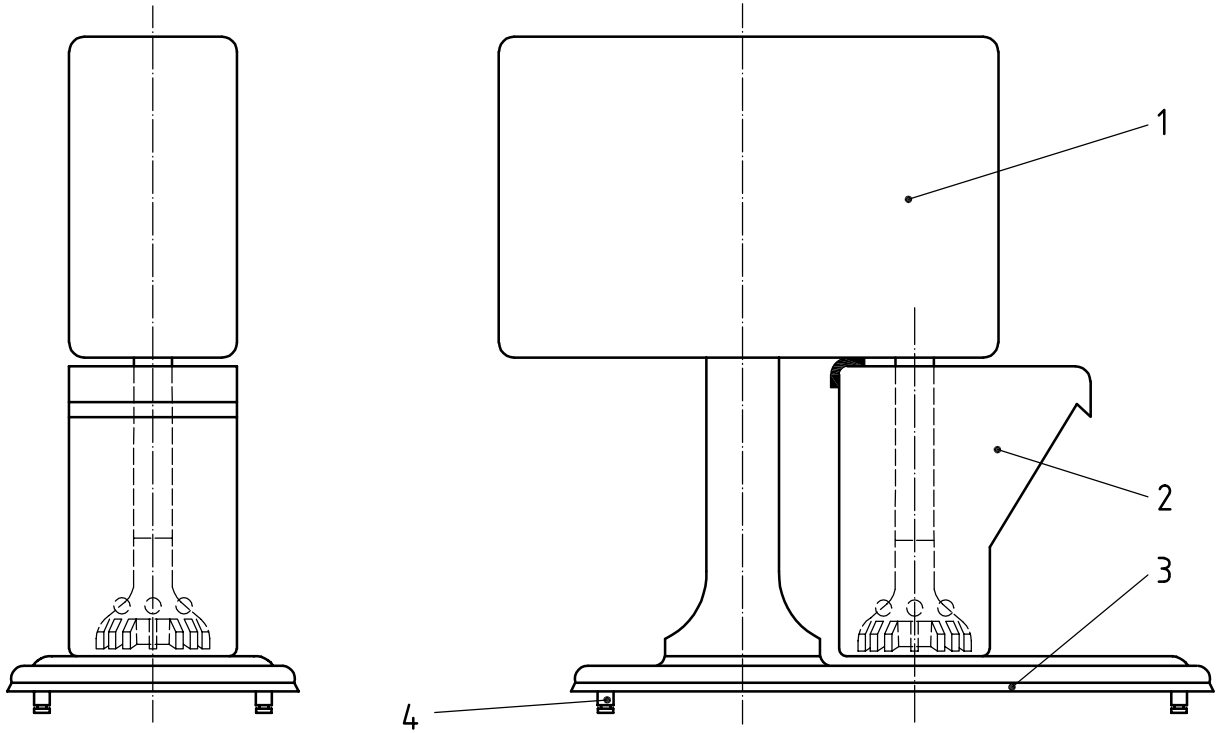
NOTE The level may be maintained by the manual addition of water.

6.4 Two scrapers, for removing the froth from the entire surface of the pulp, including the area behind the standpipe.

6.5 Calibrated micro-syringe or micro-pipette.

6.6 Containers, capable of holding individual samples of froth and tailings.

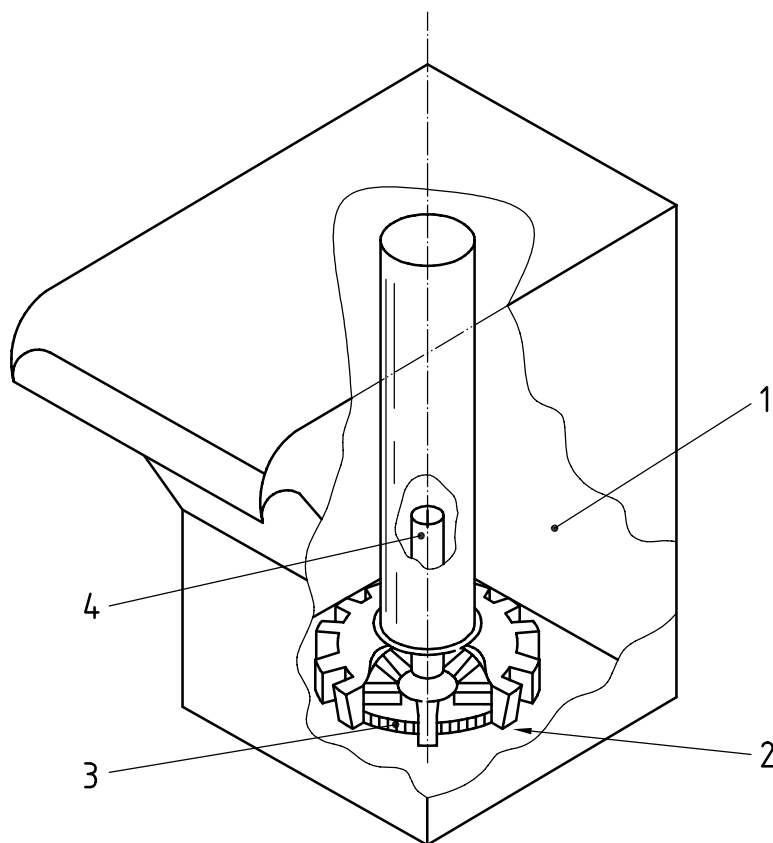
6.7 Timing device, accurate to ± 1 s and capable of being zeroed and started as required.



Key

- 1 drive mechanism
- 2 cell
- 3 metal base
- 4 levelling screws

Figure 1 — Typical flotation machine



Key

- 1 cell
- 2 hood diffuser
- 3 impeller
- 4 impeller shaft

Figure 2 —Detail of flotation cell and impeller

7 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).

Where the sample for flotation testing is obtained in slurry form, allow it to settle for at least 12 h and then remove the supernatant water. Mix the resulting thickened slurry and determine its solids content.

The history and method of preparation of the sample can affect the flotation characteristics of the coal. The source 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 mixing and subdivision of the original sample. When applicable, sampling and division of samples should be carried out in accordance with ISO 13909-2.

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.

8 Flotation test conditions

8.1 Test temperature

The test temperature shall be as specified in ISO 8858-1 and shall be recorded in the test report.

8.2 Impeller operating speed

The impeller operating speed shall be such as to give an air flow rate of $2 \text{ l}\cdot\text{s}^{-1}$ per square metre of the pulp surface area with the air inlet valve fully open; an impeller tip speed of approximately 5,7 m/s is usually suitable.

8.3 Air flow rate

The air inlet valve shall be used to control the air flow rate during the test over the required range of $0,25 \text{ l}\cdot\text{s}^{-1}$ per square metre to $2,0 \text{ l}\cdot\text{s}^{-1}$ per square metre of pulp surface area (see 9.3.5).

8.4 Pulp level

This shall be $20 \text{ mm} \pm 2 \text{ mm}$ below the overflow lip of the cell, when the impeller is rotating at the operating speed and the air inlet valve is closed.

9 Procedure

9.1 Test portion

If the feed sample is in powder form, calculate the mass of the test portion required from the measured volume of the flotation cell at the pulp level (see 8.4) and the moisture content of the test sample (see ISO 8858-1) to give a solids content of 100 g (dry basis) per litre of pulp.

In the case of a slurry, calculate the required mass of thickened slurry from the volume of the flotation cell at the pulp level (see 8.4) and the solids content of the thickened slurry to give a solids content of 100 g (dry basis) per litre of pulp.

Weigh the test portion to the nearest 0,1 g.

9.2 Preparation

If the test portion is in powder form, half fill the flotation cell with water (5.1) and start the impeller with the air inlet valve closed. Gradually add the test portion and top up to the required level with water. Then agitate for 10 min at the operating impeller speed with the air inlet valve closed.

If the test portion is a thickened slurry, transfer it to the cell and add water to the required level. Agitate for 2 min at the operating impeller speed with the air inlet valve closed.

9.3 Flotation test

9.3.1 Carry out the following procedure seven times, collecting the seven froth concentrates in the same container.

Add *n*-dodecane (5.2) at a dose rate of 90 ml/t of dry solids (based on the original feed mass) and condition for 1 min without air. Add the frother (5.3) at a dose rate of 10 l/t of dry solids and condition for an additional 10 s without air. Open the air valve and control the air flow rate to $2 \text{ l}\cdot\text{s}^{-1}$ per square metre of pulp surface area.

Allow froth to build up for 30 s, then remove the froth concentrate for a further 1 min.

Close the air valve.

9.3.2 Remove any froth remaining on the surface of the pulp, the standpipe and the cell walls and add this to the froth concentrates.

9.3.3 Remove the tailings for analysis (designated tailings 1).

9.3.4 Add the combined froth concentrates to the flotation cell again and make up to the required level with water. Start the impeller with the air valve closed. Add *n*-dodecane at a dose rate of 180 ml/t, based on the original feed mass, and condition for 1 min with the air valve closed. Add the frother at a dose rate of 20 l/t and condition for an additional 10 s with the air valve closed.

9.3.5 Collect nine froth concentrates in separate containers marked 1 to 9, over consecutive times as follows.

- a) Open the air valve and adjust the flow rate to $0,25 \text{ l}\cdot\text{s}^{-1}$ per square metre of pulp surface area. Collect froth concentrate 1 in a container for 30 s. Change the container and collect froth concentrate 2 for the following 30 s.
- b) Change the container and increase the air flow rate to $0,5 \text{ l}\cdot\text{s}^{-1}$ per square metre of pulp surface area. Collect froth concentrate 3 for 30 s. Change the container and collect froth concentrate 4 for the following 30 s.
- c) Change the container and increase the air flow rate to $1 \text{ l}\cdot\text{s}^{-1}$ per square metre of pulp surface area. Collect froth concentrate 5 for 60 s. Change the container and collect froth concentrate 6 for the following 60 s.
- d) Change the container and increase the air flow rate to $2 \text{ l}\cdot\text{s}^{-1}$ per square metre of pulp surface area. Collect froth concentrate 7 for 60 s. Change the container and collect froth concentrate 8 for the following 60 s.
- e) Change the container and collect froth concentrate 9 until flotation is completed.

9.3.6 Remove the tailings for analysis (designated tailings 2).

9.3.7 Dry, weigh and analyse all products in accordance with the relevant International Standards.

All reagent additions should be made below the surface of the pulp.

All air flow rates should be $\pm 10 \%$ of the stated value and adjustments to air flow rates made within 10 s.

The recommended procedure for froth removal is as follows:

- use two froth scrapers;
- wipe the cell walls and standpipes at least once during each concentrate removal;
- remove froth from the surface approximately once every 15 s.

10 Presentation of results

Tabulate the percentage by mass of froth concentrate and tailings together with the results of ash determinations and/or other tests and calculate the results as shown in the example given in Table 1.

Present the results graphically by plotting the cumulative percentage by mass of froth concentrate/tailings versus cumulative percentage by mass of ash as shown in Figure 3 and/or by plotting the 'Mayer curve' as shown in Figure 4.

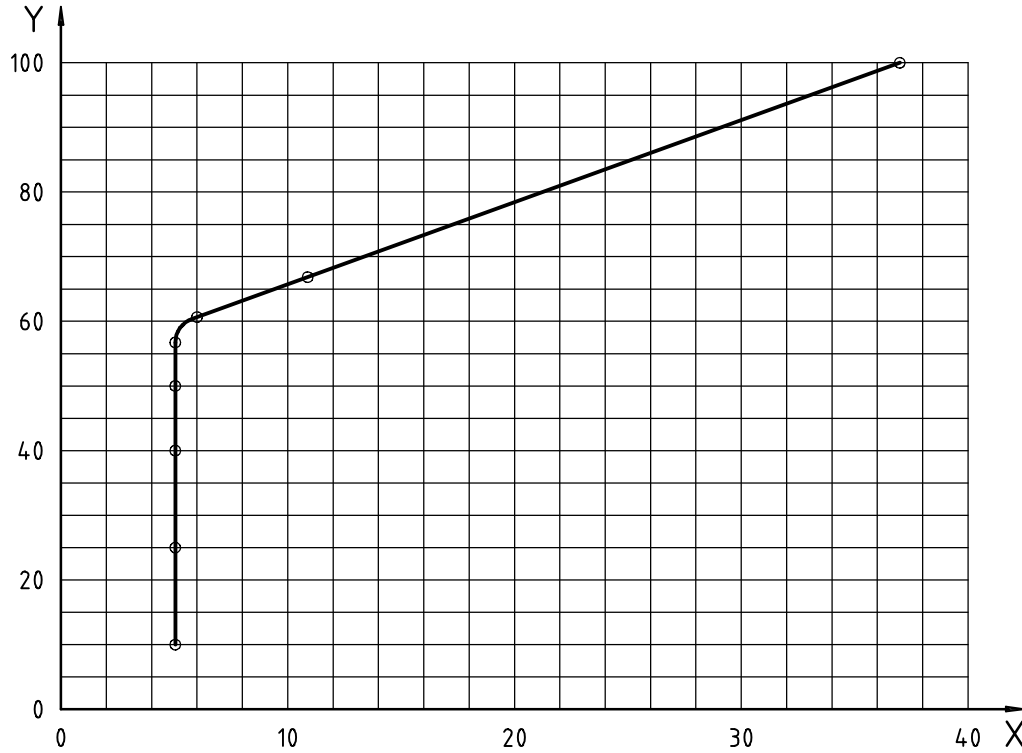
11 Test report

The test report shall include the following information:

- a) a reference to this part of ISO 8858, i.e. ISO 8858-3;
- b) complete identification of the sample;
- c) history of the sample;
- d) date on which the test was carried out;
- e) the percentages by mass of froth concentrates and tailings;
- f) the results for any other properties determined, including temperature;
- g) any operation not specified in this part of ISO 8858 or the standards to which reference is made, or regarded as optional.

Table 1 — Example of presentation of froth flotation data and calculations for flotation characteristics curves

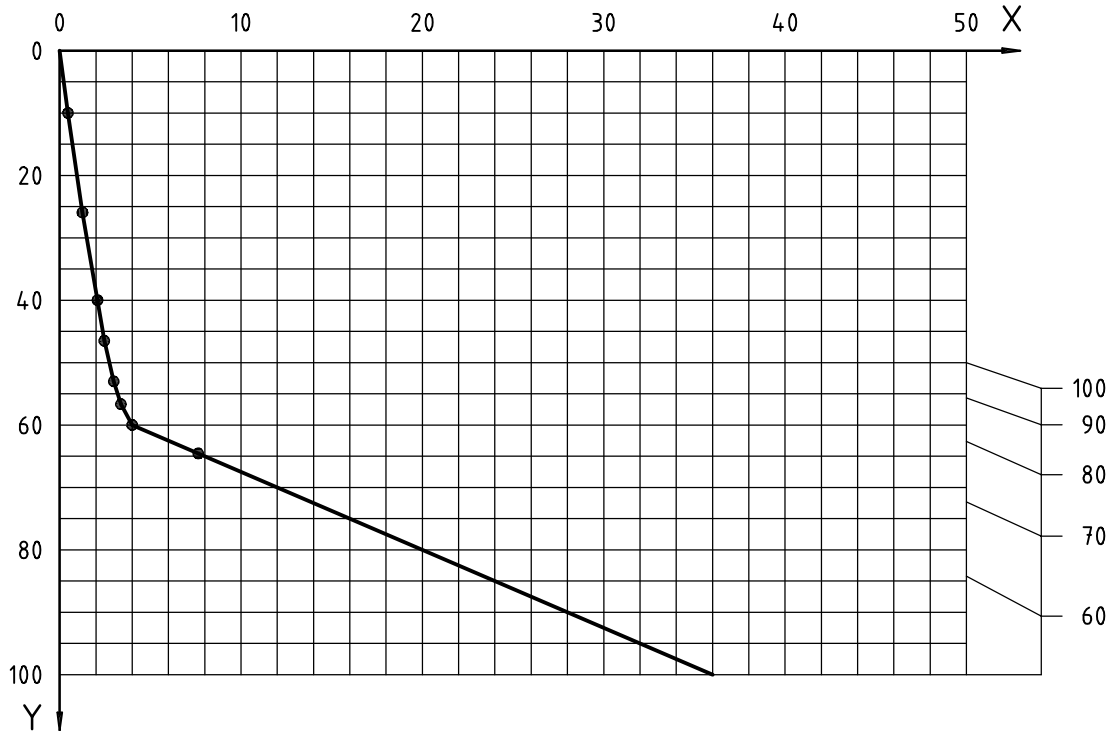
SOURCE: Mansfield			SAMPLE DATE: 24 March 2000			
SIZE FRACTION: < 0,5 mm						
TEST TEMPERATURE: 22,3 °C						
1	2	3	4	5	6	7
Froth flotation product	Percentage by mass of concentrate/tailings	Percentage by mass of ash	Quantity of ash, (2) × (3) ^a	Cumulative percentage by mass of concentrate/tailings, Σ (2)	Cumulative quantity of ash, Σ (4)	Cumulative percentage of ash, (6) ÷ (5)
	%	%	g × 100	%	g × 100	%
Froth 1	9,9	4,9	48,51	9,9	48,51	4,9
Froth 2	16,1	5,0	80,50	26,0	129,01	5,0
Froth 3	14,3	5,6	80,08	40,3	209,09	5,2
Froth 4	7,4	6,2	45,88	47,7	254,97	5,3
Froth 5	6,6	7,4	48,84	54,3	303,81	5,6
Froth 6	2,5	9,4	23,50	56,8	327,31	5,8
Froth 7	3,6	18,4	66,24	60,4	393,55	6,5
Froth 8	Nil	—				
Froth 9	Nil	—				
Tailings 2	5,0	69,2	346,00	65,4	739,55	11,3
Tailings 1	34,6	85,8	2 968,68	100,0	3 708,23	37,1
Totals	1 000	37,1	3 708,23			
NOTE Flotation is complete after froth 7.						
^a Numbers in parentheses refer to column numbers in the table.						



Key

- X cumulative ash, % (column 7 in Table 1)
- Y cumulative mass, % (column 5 in Table 1)

Figure 3 — Example of plot of cumulative percentage by mass of froth concentrate/tailings versus cumulative percentage of ash using data from Table 1



Key

- X cumulative quantity of ash, % (column 6/100 in Table 1)
- Y cumulative mass, % (column 5)

Figure 4 — Example of plot of “Mayer curve” using data from Table 1

Bibliography

- [1] ISO 13909-2:2001, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*
- [2] ISO 13909-4:2001, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test sample*

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