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Coal preparation plant — Assessment of the liability to breakdown in water of materials associated with coal seams

Ateliers de préparation du charbon — Évaluation de la fiabilité de la fragmentation dans l'eau de matériaux associés aux gisements de charbon

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Contents

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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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 10753:1994), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— editorial corrections throughout the document.

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

In this document, the materials associated with coal seams are collectively referred to as “shale”, although they may contain a variety of rocks and minerals. This document describes a method of assessing the liability of these materials to breakdown on agitation with water. Future activity on this subject could involve a method for simulating the breakdown that is likely to occur, in practice, in different types of coal preparation plants, to generate samples for further testing.

The liability of the shale to breakdown is measured by the proportion of the degraded material passing through a test sieve having a nominal size of openings of 500 µm.

The method described in this document also enables the liability of the shale to form slimes to be assessed, by determining the proportion that is below a nominal particle size of 10 µm. using a simplified sedimentation technique. This technique is based on the assumption that in the size range being examined, all the particles are spherical, have a relative density of 2,5, and fall freely through the suspending liquid according to Stokes's Law. These conditions are not entirely fulfilled in practice and the particle size is, therefore, referred to as a nominal value. The extent of breakdown, as measured by this method, will be affected by whether the shale exhibits any self-flocculation. In the described test, distilled water (or the equivalent) is used to avoid possible flocculation of the shale by any soluble salts in the water. The test may be adapted to assess the relative tendencies of shales to break down in a particular plant, by using the same water as is used in that plant. However, it is emphasized that the results of such tests, using plant water, have to be treated with caution because the extent of breakdown that will occur in practice is influenced by the interaction of all the components present during the washing process, including, for example, any accumulation of soluble salts in the circuit, and by other factors, such as the residence time in the plant.

The relationship between the proportion of degraded material passing a 500 µm test sieve and the proportion of slimes provides useful information on the character of the breakdown, and is therefore included in the results that are reported.

The amount of breakdown occurring in practice and in the test will be affected by the history of the sample. For this reason, the sample is placed in a sealed container as soon as is practicable after being taken.

Coal preparation plant — Assessment of the liability to breakdown in water of materials associated with coal seams

1 Scope

This document specifies a method for assessing the liability of materials associated with coal seams to breakdown on agitation with water.

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 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

Degradation of a test portion of shale in the size range 5,6 mm to 2,8 mm, by controlled agitation in water. Determination of the proportion of degraded material passing through a 500 µm sieve, and determination of the proportion of degraded material of nominal top size less than 10 µm by sedimentation.

5 Reagent

During the test, use only distilled water or water of equivalent purity.

6 Apparatus

6.1 Rigid cylindrical jar, of non-corrodible material and of capacity 1,0 l ± 0,1 l, with an internal height of approximately 170 mm (internal diameter approximately 85 mm) and a height/diameter ratio 2,0 ± 0,1.

The jar shall have a hard lid which can be secured to form a watertight seal.

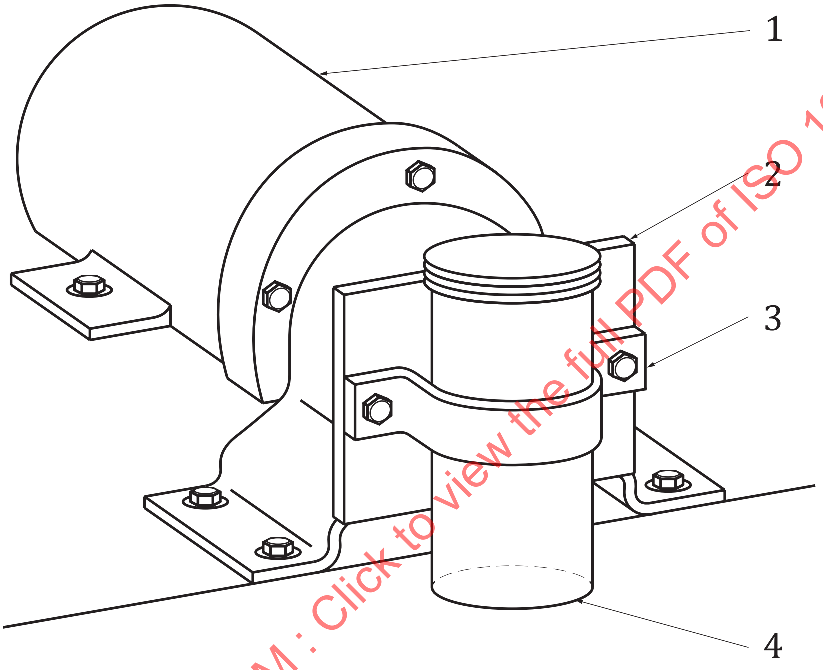
6.2 Rotation machine, capable of rotating the jar about a transverse axis at a rotational frequency of 40 min^{-1} ± 1 min^{-1} (speed of 40 r/min ± 1 r/min).

A typical assembly is shown in Figure 1.

NOTE A suitable drive can be provided by a 0,1 kW single-phase motor running at 1 500 r/min, geared down to 40 r/min.

6.3 Test sieve, having nominal size of openings 500 µm, which shall comply with ISO 3310-1.

This sieve is liable to suffer minor distortion when heated to 105 °C (see 8.5) and should therefore be reserved exclusively for use in this test. Furthermore, it is important to ensure that the sieve is constructed of materials that are stable at the temperature of drying.



Key

1 electric motor geared to 40 r/min
2 rotating base plane
3 clamp for jar
4 litre jar

Figure 1 — Typical apparatus for the determination of breakdown characteristics in water

6.4 Funnel, into which the 500 µm test sieve (6.3) will fit.

6.5 Heat-resistant tray, of non-corrodible material, large enough to accommodate the 500 µm test sieve.

6.6 Container, of capacity approximately 100 ml, fitted with a cover.

6.7 Two test sieves, having nominal sizes of openings 5,6 mm and 2,8 mm, which shall comply with ISO 3310-1.

6.8 Two measuring cylinders, of capacity 1 l each, fitted with stoppers.

6.9 Modified Andreasen sedimentation apparatus, comprising (see Figure 2) a measuring cylinder, having a pipette connected to a 10 ml reservoir via a three-way stopcock. The neck of the flask is fitted with a ground-glass stopper which has a small opening to allow air to enter the flask when a sample is withdrawn, by applying suction as shown in Figure 2; an outlet allows the sample to be run off. The Andreasen sedimentation apparatus is modified by reducing the length of stem of the pipette, so that the sample is withdrawn at the 100 mm mark. The apparatus shall be protected from all sources of vibration during the test.

6.10 Suction device, capable of applying a steady, controlled suction to the sedimentation pipette (6.9).

NOTE An aspirator is preferred for this purpose.

6.11 Evaporating dish, of nickel or stainless steel, having a diameter of 50 mm.

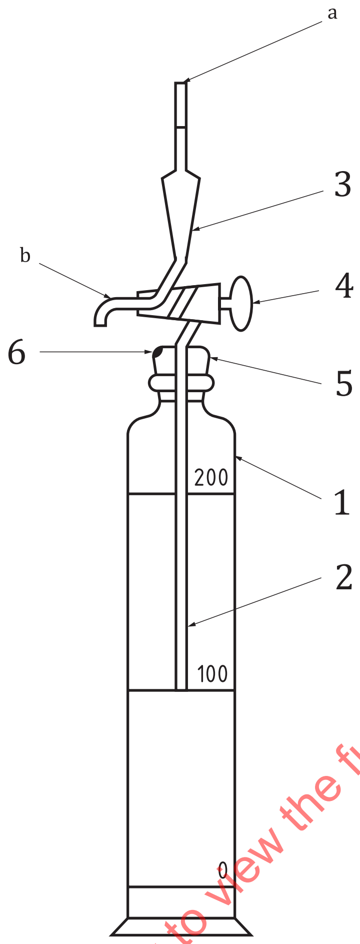
6.12 Flask, of capacity at least 2 l.

6.13 Thermostatically-controlled bath, capable of being maintained at approximately ambient temperature, to within ±0,2 °C, and of suitable dimensions to contain the sedimentation apparatus (6.9), a measuring cylinder (6.8) and the flask (6.12).

The bath shall operate without causing significant vibration or disturbance of the measuring cylinder and its contents, when placed in the bath.

NOTE If a thermostatically controlled bath is not available, a large container filled with water that has been allowed to attain room temperature can be used instead.

Dimensions in millimetres



Key

1	graduated cylindrical flask
2	pipette
3	10 ml reservoir
4	three-way stopcock
5	ground-glass stopper
6	small opening in stopper (to allow air to enter)
a	Suction application point.
b	Outlet.

Figure 2 — Modified Andreasen sedimentation apparatus

6.14 Drying oven, well ventilated and capable of being maintained in the temperature range 105 °C to 110 °C.

6.15 Stop clock.

6.16 Analytical balance(s), capable of weighing up to 20 g to the nearest 0,000 1 g and up to 200 g to the nearest 0,01 g.

6.17 Wash bottle, of capacity at least 400 ml, fitted with a fine spray-jet outlet.

6.18 Desiccator.

6.19 Vibration-free bench.

7 Sampling and preparation of test sample

If sufficient material is available, take a representative sample containing a minimum of 1 kg in the 5,6 mm to 2,8 mm size fraction. If there is insufficient material to provide the required amount in this size fraction, crush all the larger shale and add the 5,6 mm to 2,8 mm size fraction of the crushed product to that size fraction in the original material. If neither of these measures provides sufficient material, a size fraction of 5,6 mm to 1,0 mm shall be used instead. Full details of the sample taken shall be included in the test report (see Clause 11).

NOTE 1 It can be useful to prepare and test separate representative samples from the floor, roof and dirt bands for each seam.

NOTE 2 If the test sample is obtained from raw coal by means of float and sink separation, this can affect the results of the test.

Remove the dust from the test sample by gentle blowing with air and store the test sample, until required for testing, in a secure, sealed container.

NOTE 3 If the sample needs to be crushed use either a roll crusher or jaw crusher to minimize the amount of fine material generated.

8 Procedure

8.1 Determination of initial moisture content

Determine the moisture content of the shale "as-received" (see Clause 6) by weighing a 10 g portion, to an accuracy of ±0,000 1 g, drying in the oven (6.14) for 2 h at 105 °C to 110 °C and then reweighing.

8.2 Preparation of test portion

Dry for 2 h in the oven (6.14), at 105 °C to 110 °C enough of the test sample to provide a mass of 120 g, when dried, and place it in a sealed container.

8.3 Determination of residual moisture content

Determine the residual moisture in the test portion by taking 10 g of the dried test sample (see 8.2), weighed to an accuracy of ±0,000 1 g, drying in the oven (6.14) to constant mass at 105 °C to 110 °C and then reweighing.

8.4 Agitation of test portion

Weigh, to an accuracy of ±0,01 g, 100 g of the dried test sample (8.2), place in the cylindrical jar (6.1) and add 500 ml of water (Clause 5). Seal the jar securely, place it in the rotation machine (6.2) and rotate it about a transverse axis (i.e. "end over end") at a rotational frequency of 40 min^{-1} ± 1 min^{-1} (speed of 40 r/min ± 1 r/min) for 30 min.

8.5 Sieving of suspension

Locate the funnel (6.4) in the neck of one of the measuring cylinders (6.8) and mount the 500 µm test sieve (6.3) in the funnel. Sieve the suspension resulting from the agitation (8.4) through the test sieve, taking care to ensure that the solids are distributed evenly over the surface of the sieve. Use 250 ml of water (Clause 5) to rinse out the jar and wash the residue on the sieve, applying the water by means of the wash bottle (6.17).

Remove the sieve from the funnel, place it on the tray (6.5) and dry in the oven (6.14) at 105 °C to 110 °C for 1 h. Remove the tray containing the sieve from the oven. Brush off the dried material into the tared container (6.6) and dry in the oven to constant mass (see next paragraph). Cool in the desiccator (6.18), to prevent absorption of moisture, and weigh to an accuracy of ±0,01 g.

If the operation of drying to constant mass is likely to take more than 30 min, the mass measured at that time should be recorded and used to calculate the concentration of solids in the suspension passing through the sieve (see 8.8). This procedure is used in order that the proportion of material that is below a nominal particle size of 10 µm may be determined with the minimum of delay.

8.6 Treatment of fines

While the material retained on the sieve is being dried, rinse any fine particles adhering to the funnel into the measuring cylinder containing the suspension that has passed through the sieve, using 100 ml of water (Clause 5). Dilute to 1 l and place the measuring cylinder in the thermostatically-controlled bath (6.13).

8.7 Preparation of water and apparatus for sedimentation

Fill the flask (6.12) with water (Clause 5) and place it, together with the sedimentation apparatus (6.9), in the thermostatically-controlled bath (6.13), maintained, to within ±0,2 °C, at a temperature close to ambient temperature. Allow at least 1 h to reach temperature equilibrium before proceeding with the procedure described in 8.8. Use this water whenever water is required for subsequent steps in the procedure.

8.8 Adjustment of concentration of suspension

Calculate the percentage of material passing through the 500 µm test sieve in accordance with 9.1, using the approximate mass of material retained on the sieve if necessary (see 8.5, last paragraph). From this percentage, and the mass of the test portion (see 8.4), determine the concentration of solids in the suspension obtained as described in 8.6. If this concentration does not exceed 20 g/l, use the suspension directly for the sedimentation (see 8.10). Otherwise, invert the cylinder six times to mix its contents, transfer an appropriate volume to the second measuring cylinder (6.8) and dilute to volume with water maintained as described in 8.7. Keep the measuring cylinder, containing the suspension to be used for the next step, in the thermostatically-controlled bath (6.13).

The concentration of the solids in the suspension is adjusted to a level at which the particles may be assumed to be in free settlement, without mutual hindrance. The actual volume taken from the first measuring cylinder for dilution to 1 litre is not critical, provided that it is measured accurately. If the suspension in the first measuring cylinder contains m g of solids, a measured volume of not more than $\frac{2{,}0\times10^4}{m}$ ml should be transferred to the second measuring cylinder.

8.9 Determination of settling time

Calculate the time t, in seconds, taken for particles of 10 µm nominal diameter to fall 100 mm. from the following Formula:

$$t = 1{,}223\times10^6\eta \tag{1}$$

where η is the dynamic viscosity of water, in pascal seconds, at the temperature of the test1).

NOTE The derivation of Formula (1) is given in Annex A.

Alternatively, the settling time may be determined from Table 1, by interpolation if necessary.

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1) The dynamic viscosity of water at different temperatures can be obtained from reference tables, e.g. Tables of physical and chemical constants, G.W.C. Kaye and T.H. Laby.

Table 1 — Settling time for 10 µm particles

Temperature °C	**Settling time** min	 s
10	26	35
15	23	10
20	20	25
25	18	10
30	16	15

8.10 Determination of particles below 10 µm nominal size (slimes) by sedimentation

NOTE 1 An alternative sedimentation technique can be used, provided that it can be demonstrated that it will produce results comparable with those obtained using the technique described below.

Carry out this part of the procedure on a vibration-free bench (6.19).

Fit the suction device (6.10) to the sedimentation apparatus (6.9).

NOTE 2 It is important to fit the suction device before the settling is commenced, so that the withdrawal of a portion of the suspension can take place smoothly and the apparatus is not disturbed while settling is in progress.

Mix the suspension in the measuring cylinder, diluted if necessary as described in 8.8, by inverting it six times. Pour the suspension into the sedimentation apparatus, up to the 200 mm mark. Fit the pipette and replace the apparatus in the thermostatically controlled bath. After allowing a few minutes for the apparatus to return to the temperature of the bath, remove it from the bath, place a finger over hole F (see Figure 2) and, while holding the pipette in place, agitate the suspension by inverting the apparatus six times. Immediately replace the apparatus in the bath and start the stop clock (6.15).

After the time interval calculated in accordance with 8.9 has elapsed, withdraw a 10 ml portion of the suspension by connecting the pipette stem to the reservoir via the stopcock and applying steady suction in the point shown in Figure 2.

This withdrawal should take between 25 s and 30 s.

Turn stopcock so that the 10 ml portion is discharged through outlet into the tared evaporating dish (6.11). Wash out reservoir with a few millilitres of water and add the washings to the contents of the evaporating dish. Determine the mass of the solids by evaporating to dryness and then drying to constant mass in the oven (6.14) at 105 °C to 110 °C. Cool in the desiccator (6.18) and weigh to an accuracy of ±0,000 1 g.

NOTE 3 A check on the results of the determination of slimes may be carried out by shaking the suspension remaining in the sedimentation apparatus immediately after the withdrawal of the 10 ml portion, and then starting the stop clock and repeating the settling as described above. In this case, the settling time will have to be calculated from Formula (A.1), making an appropriate correction to the value of *h* to allow for the portion of suspension removed.

8.11 Number of tests

Carry out the entire procedure in duplicate.

9 Expression of results

9.1 Material passing through the 500 µm test sieve

The proportion (w_{500}) of material passing through the test sieve, expressed as a percentage by mass on a dry basis, is given by the following Formula (2):

$$w_{500} = 100\left[1 - \frac{100 m_1}{m_0\left(100 - M\right)}\right] \tag{2}$$

where

m_1 is the mass, in grams, retained on the 500 µm test sieve;

m_0 is the mass, in grams, of the test portion;

M is the residual moisture in the test portion, expressed as a percentage by mass.

9.2 Particles below 10 µm nominal size (slimes)

9.2.1 No further dilution

If the suspension obtained as described in 8.6 did not require further dilution (see 8.8), the proportion (w_{10}) of solid material below 10 µm nominal size in the test sample, expressed as a percentage by mass on a dry basis, is given by the following Formula (3):

$$w_{10} = \frac{10^6 m_2}{m_0\left(100 - M\right)} \tag{3}$$

where

m_2 is the mass, in grams, of solids in the 10 ml portion withdrawn from the sedimentation apparatus after the settling period;

m_0 is the mass, in grams, of the test portion;

M is the residual moisture in the test portion, expressed as a percentage by mass.

9.2.2 Further dilution

If the suspension did require further dilution in accordance with 8.8, the proportion (w_{10}) of solid material below 10 µm nominal size, expressed as a percentage by mass on a dry basis, is given by the following Formula (4):

$$w_{10} = \frac{10^9 m_2}{V m_0\left(100 - M\right)} \tag{4}$$

where

m_2 is the mass, in grams, of solids in the 10 ml portion withdrawn from the sedimentation apparatus after the settling period;

V is the volume, in millilitres, taken from the first measuring cylinder and diluted to 1 l in the second measuring cylinder;

m_0 is the mass, in grams, of the test portion;

M is the residual moisture in the test portion, expressed as a percentage by mass.

9.3 Breakdown ratio

The breakdown ratio B, expressed as a percentage by mass, is given by the following Formula (5):

$$B = \frac{w_{10}}{w_{500}} \times 100 \tag{5}$$

where

w_{10} is the proportion of slimes in the test sample, expressed as a percentage by mass;

w_{500} is the proportion of material passing through the 500 µm test sieve, expressed as a percentage by mass.

10 Precision

10.1 Repeatability limit

The difference between two single results found on test portions, taken from the same test sample, by one operator using the same apparatus within the shortest feasible time interval will exceed 5 %, relative, on average not more that once in 20 cases during the normal and correct operation of the method.

10.2 Reproducibility limit

The difference between two single results found on identical test material by two different operators using different apparatus at different times will exceed 10 %, relative, on average not more than once in 20 cases during the normal and correct operation of the method.

11 Test method

The test report shall include the following information:

a) the identification and history of the sample under test;

b) whether the test sample contained material other than that naturally occurring in the 5,6 mm to 2,8 mm size fraction;

c) the initial moisture content of the shale “as received” and the residual moisture content in the test portions after drying;

d) the sedimentation technique used, if different from that described;

e) the sedimentation test conditions, i.e.

- the volume of suspension diluted,
- the settling time,

  - the test temperature;

f) any other variations in the described procedure;

g) the individual results of the duplicate tests, and the average values.

NOTE An example of a suitable format for the test report is given in Annex B. The results can also be presented in graphical form, as illustrated in Figure B.1.