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Coal — Methods for evaluating flocculants for use in coal preparation —

Part 1: **Basic parameters**

Charbon — Méthodes d'évaluation des floculants utilisés dans la préparation des charbons —

Partie 1: Paramètres de base

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 734 10 79
E-mail copyright@iso.ch
Web www.iso.ch

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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 3.

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 part of ISO 10086 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10086-1 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 1, *Coal preparation: Terminology and performance*.

ISO 10086 consists of the following parts, under the general title *Coal — Methods for evaluating flocculants for use in coal preparation*:

— *Part 1: Basic parameters*

— *Part 2: Flocculants as filter aids in vacuum filtration*

Annexes A and B of this part of ISO 10086 are for information only.

Coal — Methods for evaluating flocculants for use in coal preparation —

Part 1: Basic parameters

1 Scope

This part of ISO 10086 specifies a method for the comparative evaluation of the performances of flocculants for clarification, thickening and sedimentation applications on a given slurry. This performance can be evaluated by

a) the settling velocity in the initial period,

b) the sediment volume after compaction and consolidation,

c) the clarity of the supernatant liquid.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 10086. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 10086 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 1953, *Hard coal — Size analysis by sieving*.

3 Principle

The performance of different flocculants on a given slurry is determined by measuring the relative settling rates.

A flocculant solution is added to an aliquot of the slurry in a measuring cylinder and the formation of an interface between the supernatant liquid and the suspension is observed. An initial settling rate is calculated and is plotted against flocculant dosage to evaluate the performance of the flocculant.

4 Apparatus

Usual laboratory apparatus, and

4.1 Stirrers, two variable-speed motorized stirrers capable of 1 000 r/min (one for flocculant preparation and one for sample homogenization).

4.2 Plastic moulded cylinders, of capacity 500 ml, graduated in 5 ml scale divisions and having rubber stoppers. Where these are not available, glass cylinders, of capacity 500 ml $\pm$ 2 ml, graduated in 5 ml scale divisions and having ground-glass stoppers, may be a satisfactory alternative. However, differences in settling rates may result, because of differences in the graduated height.

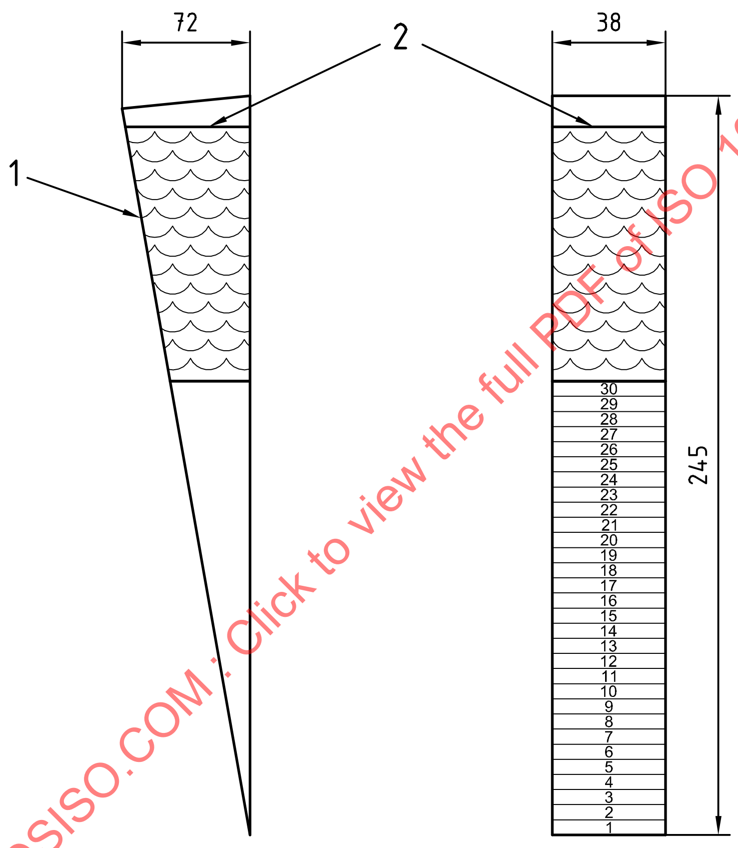
A vertical scale may be attached to or mounted beside the cylinder, with the zero point coinciding with the 5 ml mark of the cylinder.

4.3 Syringes, having nominal capacities of 1 ml, 2 ml, 5 ml, 10 ml and 50 ml.

4.4 Timer, capable of reading 0,1 s to a total of 10 000 s.

4.5 Clarity wedge, as shown in Figure 1, having a scale in black numbers on a white background (or vice versa) printed inside the back.

Dimensions in millimetres



NOT TO SCALE

Key

1 Transparent plastic sides and front
Wall thickness: 4 mm

2 Suspension level

Figure 1 — Clarity wedge

5 Materials

5.1 Slurry

Flocculant evaluation shall be carried out on a slurry collected from the coal preparation plant and free from flocculant contamination. The sample shall be collected in at least 10 increments over a minimum of 2 h of stable operating conditions, to give a total sample volume of 50 litres or more.

The sample shall be collected in a container that is inert to the slurry and stored in a suitable environment that maintains a temperature of 20 °C ± 2 °C. Storage may affect the characteristics of the sample and, hence, affect the relative performance of the flocculants. Therefore, the sample shall be used as soon as possible, but not later than 24 h after collection.

A size analysis and ash determination shall be performed on the solids in the sample using the methods given in and respectively.

The concentration of solids in the slurry, in grams per litre on a dry basis, shall also be determined, by drying the sample in an oven at 105 ° C to constant mass, which is defined as the point at which two consecutive weighings do not differ by more than 5 % after 1 h intervals in the oven.

The collected sample is divided into representative 500 ml subsamples as follows.

a) Mix the slurry using a motorized stirrer (4.1) to ensure homogeneity of the sample.

b) While the sample is being stirred, dip a beaker of capacity 50 ml into the slurry and fill it. Pour this sample into the first cylinder (4.2). Repeat the procedure, adding to each cylinder in a cyclic pattern, until sufficient test cylinders for complete test work are filled to the 500 ml mark.

5.2 Water

Prepare the flocculant solution using water that is used in the plant, where this is available.

Where plant water is not available, use potable or fresh water. Collect sufficient water for all of the flocculants being tested.

If flocculants are not currently being used by the plant, careful consideration should be given to a suitable source of water.

6 Sampling

Flocculants are available in the forms of powder, emulsion, dispersion or solution. In this part of ISO 10086, emulsion, dispersion and solution flocculants are referred to as liquid flocculants. Recommendations are as follows:

a) Flocculants should be obtained as fresh samples and should be discarded after six months.

b) Store all flocculant samples at ambient temperature, away from direct sunlight and heat, and keep solid flocculant samples away from moisture.

c) Avoid unnecessarily opening and closing of the sample container.

d) Take a sufficient quantity of flocculant sample for all of the tests to be carried out.

7 Preparation of flocculant solutions

7.1 Powder flocculants

Homogenize the bulk portion of the flocculant by mixing. Using a scooped spatula, transfer a subsample of approximately 1 g of the flocculant, 0,1 g to 0,2 g at a time, into a weighing bottle.

Place 250 g ± 0,5 g of water (5.2) in a 500 ml beaker and stir the water at a speed that is sufficient to create a vortex.

Sprinkle a preweighed amount of powder (0,25 g ± 0,01 g) from the 1 g subsample onto the surface of the vortex and stir the resulting dispersion slowly until dissolution is complete, and for not less than 2 h.

Use this stock solution within 24 h.

7.2 Liquid flocculants

Homogenize the sample in its container, then fill a 2 ml syringe (4.3) with the liquid flocculant and weigh to the nearest 0,01 g. Place 200 g $\pm$ 5 g of water (5.2) in a 500 ml beaker and stir the water at a speed sufficient to create a vortex. Discharge the syringe into the vortex and reweigh the empty syringe. Continue to stir at this speed for 5 min, then stir for a further 2 h at a slower speed that is high enough to keep the solution agitated.

Use this stock solution within 24 h.

8 Procedure

8.1 Preparation of test sample

Prepare a test sample of slurry by filling the cylinder with slurry using the method described in 5.1. Stopper the cylinder and homogenize the slurry either by double-inverting the cylinder end over end for 1 min, or by mixing for 1 min with a stirrer.

Using a syringe, add the appropriate quantity (e.g. 2 ml) of flocculant solution (see 7.1) to the surface of the slurry in the cylinder, stopper and mix well by double-inverting the cylinder or by stirring, until completely homogenized.

Excessive mixing should be avoided, to prevent degradation of the flocculant.

8.2 Determination of settling rate

8.2.1 Free settling rate

Stand the cylinder upright. Observe the definite interface that forms between the clear supernatant liquid and the bulk of the flocculated suspension. Record the time (t s) for the interface to descend from the 450 ml mark to the 250 ml mark on the cylinder. Measure the distance (d), in millimetres, between these two marks.

The free settling rate V_{f}, in metres per hour, is then calculated from the following equation:

$$V_{\mathrm{f}} = \frac{d}{t} \times 3{,}6$$

where

d is the distance between the marks, in millimetres;

t is the time, in seconds, for the interface to descend from 450 ml to 250 ml.

Repeat the procedure using different volumes of the same flocculant solution.

Where it is required to compare flocculants, repeat the test(s) for each flocculant being evaluated.

8.2.2 Full settling curve

Where it is required to construct the full settling curve, it is necessary to monitor the position of the interface throughout its descent down the measuring cylinder. Start the timer immediately after the flocculant has been added to the slurry and position the cylinder upright after the agitation.

Record the time for the interface to pass each 25 ml subdivision.

From these measurements, calculate a series of settling rates, in metres per hour, and from them calculate the settling rates by the maximum average procedure (see the note).

NOTE The maximum average procedure averages the settling rates progressively and chooses the maximum average (A_{max}). During the flocculation process, an induction period is usually observed which can cause the initial appearance of the interface between supernatant liquid and flocculated solids to be delayed. The induction period is followed by a period of free settling of solids and then by a period in which compaction takes place. The “maximum average procedure” takes into account this behaviour of the slurry. A worked example is given for information in annex A.

Repeat the procedure with different volumes of the same flocculant solution.

Where it is required to compare flocculants, the test should be repeated on each flocculant being evaluated.

8.3 Determination of sediment height and supernatant clarity

Thirty minutes after standing the cylinder upright, measure the settled volume of the sediment, in millilitres. Then fill the clarity wedge (4.5) with the supernatant liquid by decantation. Determine the clarity using the clarity wedge, by observing the highest value visible through the liquid.

9 Calculation of results

9.1 Flocculant dosage rate

The concentration of the flocculant solution, in grams of flocculant per gram of solution, is given by the equation:

$$C = \frac{m_F}{m_W} \qquad (1)$$

where

m_W is the mass of water in the beaker;

m_F is the mass of flocculant dissolved in the water;

For the purpose of this calculation, it is assumed that a volume of 1 ml of flocculant solution weighs 1 g.

The mass of the solids (m_S), in grams, in 500 ml of slurry is given by the equation:

$$m_S = 0{,}5 \times \rho_S \qquad (2)$$

where

ρ_S is the concentration of solids in the slurry, in grams per litre.

The flocculant dosage rate (D_F), in kilograms of flocculant per tonne (which is equivalent to grams per kilogram), is given by the equation:

$$D_F = 2\,000 \times \frac{m_F \times V_F}{m_W \times \rho_S} \qquad (3)$$

where

V_F is the volume of flocculant solution used to dose a slurry suspension in the cylinder, in millilitres.

The units of the dosage rate may also be expressed in kilograms of flocculant per tonne of dry solids.

9.2 Worked example

Mass of water (m_W), in grams = 201,2

Mass of flocculant (m_F), in grams = 0,23

Concentration of solids in the slurry (ρ_S), in grams per litre = 50,6

Volume of flocculant solution used (V_F), in millilitres = 3,0

Therefore, the flocculant dosage rate, in kilograms of flocculant per tonne which is equivalent to grams per kilogram

$$= 2\,000 \times \frac{m_F \times V_F}{m_W \times \rho_S}$$

$$= \frac{2\,000 \times 0{,}23 \times 3}{201{,}2 \times 50{,}6}$$

$$= 136$$

The procedure is repeated for other volumes of flocculant solution (V_F) used.

10 Recording of results

Record the results for the free settling rate on a data sheet.

NOTE An example of a data sheet is shown in annex B.

The characteristics of the slurry and flocculant shall also be recorded on this sheet.

Plot the free settling rate as a function of flocculant dosage for each flocculant, as illustrated in Figure A.2.

Where the position of the interface has been recorded, the full settling curve shall be recorded on the data sheet in Table B.2.

Draw a horizontal line at the required free-settling rate. The intercepts at the settling rate curves give the flocculant dosage/settling rate relationship. The optimum flocculant achieves the desired settling rate at the lowest dosage.

11 Repeatability

Duplicate results of tests carried out by the same operator should not differ by more than 10 %.

12 Test report

The test report shall include the following information:

a) date on which the test was carried out;

b) description of the flocculant under test;

c) details of the slurry used;

d) reference to this part of ISO 10086 and its year of publication;

e) the free-settling rate;

f) the clarity of the supernatant liquid;

g) the sediment height after 30 min;

h) the flocculant dosage rate.

Annex A
(informative)

Worked example

A test was performed on a slurry with a commercial flocculant at a dosage of 50 mg of flocculant per litre of slurry. The distance between the 25 ml scale marks of the 500 ml cylinder being used for the test was measured as 14 mm. The test procedure was carried out according to this part of ISO 10086. The times, in seconds, for the interface between supernatant liquid and settling solids to pass each 25 ml subdivision (475 ml, 450 ml, etc.) were: 85 s, 124 s, 150 s, 194 s, 251 s, 340 s and 630 s. The time/velocity data were then calculated as shown in Table A.1.

Table A.1 — Calculation of settling rate

Interval travelled	Time h	Velocity[a] m/h
From 500 ml to 475 ml mark	$t_1 = 85/3\,600 = 2{,}36 \times 10^{-2}$	$v_1 = 0{,}014/t_1 = 0{,}59$
From 475 ml to 450 ml mark	$t_2 = (124 - 85)/3\,600 = 1{,}08 \times 10^{-2}$	$v_2 = 0{,}014/t_2 = 1{,}30$
From 450 ml to 425 ml mark	$t_3 = (150 - 124)/3\,600 = 0{,}72 \times 10^{-2}$	$v_3 = 0{,}014/t_3 = 1{,}94$
From 425 ml to 400 ml mark	$t_4 = (194 - 150)/3\,600 = 1{,}22 \times 10^{-2}$	$v_4 = 0{,}014/t_4 = 1{,}15$
From 400 ml to 375 ml mark	$t_5 = (251 - 194)/3\,600 = 1{,}58 \times 10^{-2}$	$v_5 = 0{,}014/t_5 = 0{,}89$
From 375 ml to 350 ml mark	$t_6 = (340 - 251)/3\,600 = 2{,}47 \times 10^{-2}$	$v_6 = 0{,}014/t_6 = 0{,}57$
From 350 ml to 325 ml mark	$t_7 = (630 - 340)/3\,600 = 8{,}06 \times 10^{-2}$	$v_7 = 0{,}014/t_7 = 0{,}17$

[a] v_n is the average velocity of the interface over each interval.

Velocity (v) increases with time, passes through a maximum and decreases thereafter, indicating the existence of the induction, free settling and compaction zones indicated in Figure A.1. According to the "maximum average" expression, a series of averages, in metres per hour, are calculated as follows:

$$A_1 = (v_1 + v_2)/2 = 0{,}95$$

$$A_2 = (v_1 + v_2 + v_3)/3 = 1{,}28$$

$$A_3 = (v_1 + v_2 + v_3 + v_4)/4 = 1{,}25$$

In the same manner:

$$A_4 = 1{,}17$$

$$A_5 = 1{,}07$$

$$A_6 = 0{,}94$$

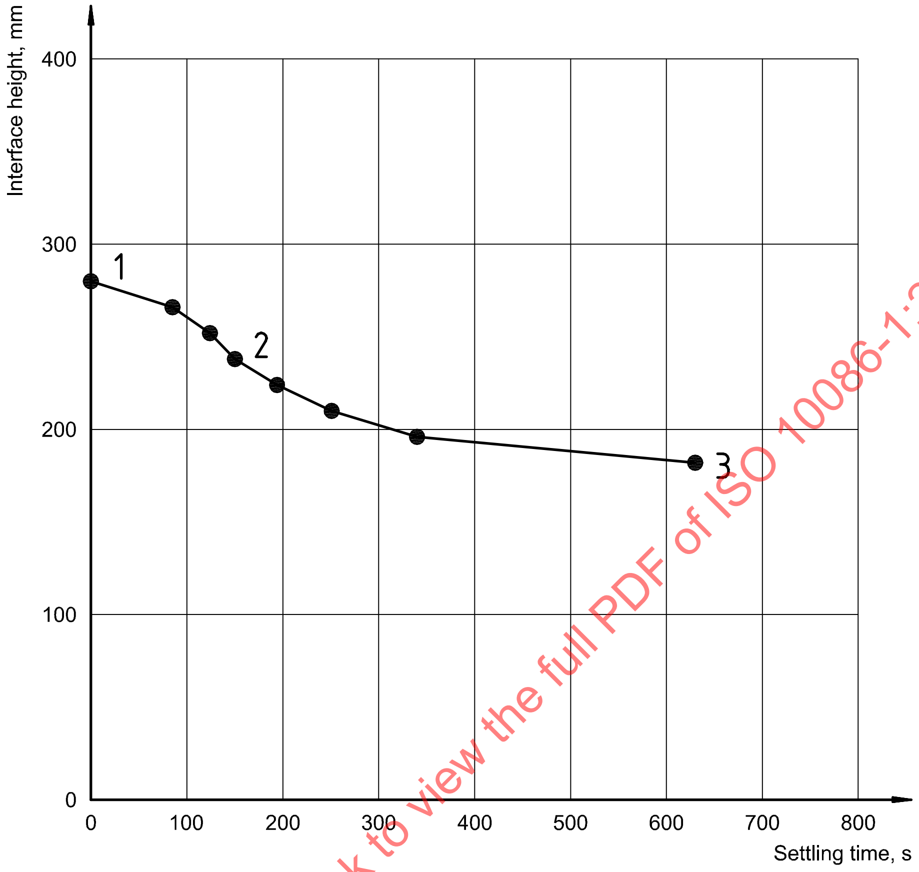
Thus, the maximum A_{max}, is equal to 1,28 m/h.

This was taken as the free-settling rate from this test for this flocculant and dosage.

A series of tests was carried out at different dosages, enabling the free-settling rate/dosage plot (see Figure A.2) to be prepared.

In Figure A.2, for a desired settling rate of 3,0 m/h (0,83 mm/s), this is achieved at a lower dosage with flocculant C than with flocculant A or B. However, only flocculant B is suitable if a free-settling rate of 7,0 m/h (1,94 mm/s) is required.

Supernatant clarity and/or degree (or even rate) of solid compaction is taken into consideration for cases where clarification and/or thickening are of importance.



Key

1 Induction

2 Free settling

3 Compaction

Figure A.1 — Plot of interface height as a function of settling time

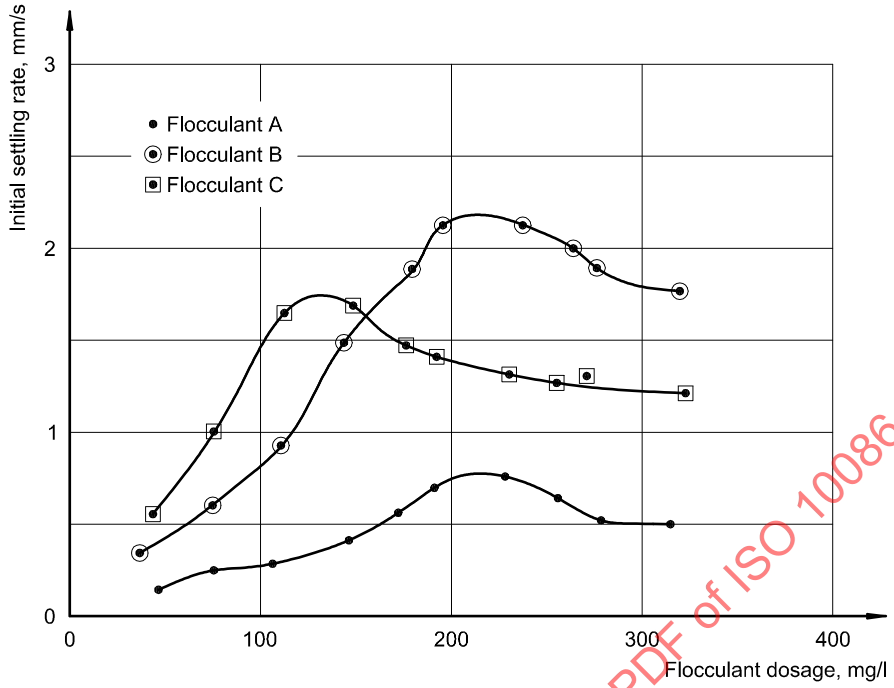


Figure A.2 — Plots of free-settling rate as a function of flocculant dosage for different flocculants