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Hard coal — Determination and presentation of float and sink characteristics — General directions for apparatus and procedures

Houille — Détermination et présentation des caractéristiques de flottation et d'enfoncement — Principes directeurs relatifs à l'appareillage et aux modes opératoires

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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.

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.

International Standard ISO 7936 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Sub-Committee SC 1, *Coal preparation. Terminology and performance*.

Annexes A and B form an integral part of this International Standard. Annex C is for information only.

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Introduction

The results of float and sink testing, presented in tabular and graphical form, are the basis for the provision of washability data. These results are useful when designing and redesigning a plant, and in predicting, controlling and assessing the performance of a plant.

Where tests other than those for routine control purposes are carried out, it is essential that there is precise instruction regarding size ranges and relative density fractions to establish the scope of information and accuracy required.

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Hard coal — Determination and presentation of float and sink characteristics — General directions for apparatus and procedures

1 Scope

This International Standard describes general directions for the apparatus and procedures, using relative density separation methods, for determining the float and sink characteristics of raw coal and of products from coal preparation plants.

A general procedure for a centrifugal float and sink test is given in annex A. A typical procedure for treating and testing a sample of raw coal is described in annex B. Some practical hints on float and sink testing are given in annex C.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 923:1975, *Coal cleaning tests — Expression and presentation of results.*

ISO 1213-1:1982, *Solid mineral fuels — Vocabulary Part 1: Terms relating to coal preparation.*

ISO 1213-2:1971, *Vocabulary of terms relating to solid mineral fuels — Part 2: Terms relating to coal sampling and analysis.*

ISO 1988:1975, *Hard coal — Sampling.*

ISO 9411-1:—[1], *Solid mineral fuels — Mechanical sampling from moving streams — Part 1: Coal.*

ISO 1953:1972, *Hard coals — Size analysis.*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 1213-1 and ISO 1213-2 apply.

4 Sampling

4.1 General

Sampling shall be carried out in accordance with ISO 1988.

NOTE 1 A method for the mechanical sampling of coal from moving streams will be covered in ISO 9411-1.

The quantity of sample, and consequently the degree of accuracy obtained in a float and sink test, may be varied according to the purpose for which the test is being carried out. The three main categories are

a) investigation of the characteristics of raw coal;

b) comprehensive plant efficiency tests;

c) plant control tests.

4.2 Raw coal

The mass of the bulk sample should be sufficient to contain the minimum quantities in each fraction as listed in table 1, which is for guidance only for use with an unknown sample. These masses may not be practicable in the case of some plant products or bore core samples.

1) To be published.

The number of discrete particles to be aimed for in any size fraction should not be less than 2 000. The masses given in table 1 will generally ensure that the number of particles is adequate for raw coal.

Because some coals give low yields in the intermediate relative density fractions, there may be insufficient material for analytical requirements. In addition, the recommended mass of the sample may have to be substantially increased to meet the following requirements: not less than 20 g and not less than 10 particles in each relative density fraction.

Where taking a bulk sample, it is better to oversample than to have insufficient material. In order to carry out testing on the larger sizes in table 1, the sample may have to be of the order of 10 t, or even greater.

In a newly opened mine, a trial shaft or other appropriate location, the mass of bulk sample taken should not be less than 10 t.

For bore cores, the masses recommended in table 1 are not often obtained. For this reason, core plies or sections should be selected as large as possible, and subdivision of the crushed ply or section prior to float and sink testing should be avoided.

Table 1 — Minimum mass for a given size fraction

Size fraction[1)] (square hole) mm	Typical recommended masses for raw coal kg
− 250 + 125	1 000
− 125 + 63	350
− 63 + 31,5	180
− 31,5 + 16,0	90
− 16,0 + 8,0	33
− 8,0 + 4,0	7
− 4,0 + 2,0	3
− 2,0 + 1,0	1,5
− 1,0 + 0,5	1,0
− 0,5 + 0,063	0,5

1) The sizes shown in this table may be supplemented or replaced by other sizes. Quantities within specified size ranges will be determined by the number of separations to be made and the quantitative distribution of the components in terms of relative density.

Both the size distribution and the ash percentage of the raw coal coming from a working face or mine will vary during a shift, as well as from day to day. It is essential that the duration of sampling be long enough to cover such variations.

The total sample mass m_t required for a float and sink test is given by the following equation:

$$m_t = \frac{m_r}{m_s} \times 100 \qquad \ldots (1)$$

where

m_r is the recommended mass of coarsest size fraction (from table 1);

m_s is the mass percent of the coarsest size fraction in the sample.

4.3 Plant products

For clean coal, the minimum mass of sample required is normally 50 % greater than that required for raw coal, to ensure that adequate amounts of misplaced material are available.

Since the relative densities of some components, such as discard and middlings, are greater than that of clean coal, the minimum masses of samples containing these components should be increased proportionately. This ensures that these samples contain approximately the same number of particles as the corresponding clean coal sample, and consequently a similar degree of accuracy will be obtained in the test.

Samples should be taken as soon as practicable after the material leaves the cleaning unit, in order to minimize breakage. Testing should then commence as soon as possible.

In sampling pulp, the mass of the (dried) solids should comply with the requirements of table 1. Increments shall be taken at regular time intervals over the total cross-section of the pulp stream, either manually or by mechanical means, using a sampling device having a capacity equal to at least twice that of the recommended minimum mass of increment. Care should be taken to ensure that none of the sample is lost by splashing.

4.4 Plant control testing

Routine samples are taken regularly for the purpose of determining the average efficiency of a cleaning plant. They may represent daily, weekly or longer periods of running. The mass taken may be less than that given in table 1, depending on the reason for the test. However, if any dispute arises over the accuracy of the results, sample masses in accordance with 4.2 and 4.3 should be used.

4.5 Comprehensive plant efficiency tests

A comprehensive cleaning plant efficiency test involves a systematic mass balance of all materials entering and leaving the plant. In this case, the mass and moisture content of the raw feed, the mass and

moisture content "as weighed" of all cleaned products, discard etc., and the volume and solids content of the effluent will be required. The mass of all materials is calculated to a uniform moisture basis, and the feed entering and products leaving the plant are balanced against each other. The efficiency of the cleaning plant is assessed from the actual and theoretical yields and ash percentages. The analysis of the raw feed by computation from the masses and analyses of all the products is more reliable than that obtained by direct examination, and it is therefore used for the calculation of the theoretical yields.

When a screen analysis of a plant product is made in connection with a cleaning plant efficiency test, it will be found that there is some material below the nominal bottom size being treated in the cleaning unit. The mass and particle size range of this undersize material should be recorded.

Table 2 — Size analysis

Size fraction (square hole) mm		Mass %	Material retained %	Material passing %
+ 125,0		Nil	Nil	100,0
− 125,0	+ 63,0	11,9	11,9	88,1
− 63,0	+ 31,5	12,1	24,0	76,0
− 31,5	+ 16,0	12,8	36,8	63,2
− 16,0	+ 8,0	15,7	52,5	47,5
− 8,0	+ 4,0	12,5	65,0	35,0
− 4,0	+ 2,0	10,2	75,2	24,8
− 2,0	+ 1,0	7,5	82,7	17,3
− 1,0	+ 0,5	5,6	88,3	11,7
− 0,5		11,7	100,0	Nil
Total 100,0				

5 Preliminary treatment

5.1 Size analysis

The sample should be spread out on an impervious base, preferably under shelter, and allowed to dry sufficiently for screening purposes. It should then be screened using a suitable range of apertures (typical sizes are given in table 2). Oversize material may be broken by hand or machine-crushed according to the nominal top size required. If applicable, the relevant part of the crusher circuit may be simulated.

The quantity of material passing the 63 mm screen is usually more than the amount required and it can be divided before proceeding to the next size of screen. Further division may be necessary at lower sizes.

NOTE 2 The screening process described in this subclause may be preceded by a sample treatment operation designed to simulate the particle breakdown which may occur in a coal preparation plant.

Wet screening should be used, to ensure that fine particles adhering to larger particles are included in the proper size fraction.

NOTE 3 Pulp and discard samples should be screened promptly to avoid excessive shale breakdown.

5.2 Pilot testing

Pilot testing is frequently carried out on a representative sample, in order to determine how the bulk material will behave. This knowledge enables the operator to plan the actual test in such a way that unnecessary operations are avoided, so that the test is carried out more expeditiously and with less effort. The pilot test, or previous experience, may indicate that it is advantageous to commence the separation at either the highest or the lowest relative density.

A sample which will give a high yield at either of these densities should be separated at that density, so that the bulk of the sample can be removed in one operation.

In cases where there is only a small yield at one or two consecutive relative density fractions, it is better to combine these fractions before going through a full treatment process. Within these limits it is possible to vary the procedure without affecting the outcome of the test; in many cases its accuracy will be improved and the time and labour involved will be reduced.

6 Float and sink testing

6.1 Float and sink medium

6.1.1 Basis of selection

The medium which is to be used for the separation may be a mixture of organic liquids, aqueous solutions of inorganic salts, or solids in aqueous suspensions. The choice of medium is also governed to some extent by the bulk and particle size of the coal

being tested, by its rank and relative density, and by the purpose for which the separation is being carried out. The most suitable range of relative densities will have to be determined by trial and error, but would normally include 1,3; 1,4; 1,5; 1,6; 1,7; 1,8; 1,9 and 2,0. Relative densities less than 1,3 and above 2,0 may also be required.

Additional separation at intermediate relative densities will be found useful where cumulative ash is increasing rapidly in relation to the cumulative yield. As stated in 4.2, each relative density fraction should weigh at least 20 g and should contain at least 10 discrete particles.

Where it is known or suspected that the sample will disintegrate or otherwise react on contact with water or aqueous solutions, separations are to be carried out using organic liquids. However, the fact that the raw coal will react with water will affect its behaviour in the cleaning process, and any information which will provide guidance should be obtained for reference purposes.

NOTE 4 Where water quality problems are suspected, water of at least "potable" grade should be used to prepare aqueous suspensions and inorganic solutions.

6.1.2 Organic liquids

Where the separation is critical, particularly in finer sizes, the use of organic liquids is preferred (see note 5) because of their low viscosity, low volatility and inertness towards shales. Some organic liquids and their physical properties are listed in table 3.

NOTE 5 Some organic liquids may influence subsequent analyses.

Table 3 — Typical physical properties of organic liquids used in float and sink analysis

Organic liquid	Relative density	Distillation range or boiling point at 100 kN/m (100 kPa) °C	Viscosity at 20 °C mPa·s (mN·s/m²)	Vapour pressure at 20 °C kPa (kN/m²)	Flammable
White spirit	0,77	30 to 200	—	—	Yes
Petroleum spirit[1) 2)]	0,73	37 to 185	0,548	25,33	Yes
Toluene	0,87	110,7	0,588	2,93	Yes
Kerosene	0,75	165 to 230	1,365	0,11	Yes
o-Xylene	0,88	144,4	0,810	0,68	Yes
m-Xylene	0,86	139,0	0,620	0,85	Yes
p-Xylene	0,86	138,4	0,648	0,92	Yes
Bromoform (tribromomethane)	2,79	150,0	2,152 (at 15 °C)	0,60	No
Sym tetrabromoethane (acetylene tetrabromide)	2,96	239	12,0	0,01	No
Tetrachloroethylene (perchloroethylene)	1,61	120,8	1,0	1,83	No

SAFETY PRECAUTIONS

Particular attention is drawn to the fact that many solvent vapours present a serious health hazard and hence adequate ventilation, preferably down-draught, is essential.

As the use of some organic liquids is governed by safety regulations, the user is urged to ensure compliance with the relevant statutory regulations.

1) **Mixtures of air and petroleum spirit vapour are highly explosive.**
2) If petroleum spirit is used it should be lead free.

Where relative densities of 1,6 and less are required, mixtures of perchloroethylene and one of the less dense liquids may be used. Where relative densities of 1,6 to 2,9 are required, mixtures of perchloroethylene and one or more of the more dense liquids may be used.

NOTE 6 Tetrabromoethane and bromoform have extremely low vapour pressures. After use, it is therefore necessary to rinse them from the sample by a more rapidly evaporating solvent.

Organic liquids are costly but are frequently preferred to aqueous solutions, since the products of the separation are easier to deal with and prolonged washing and drying times are unnecessary because of the volatility of the solvents. They should be used sparingly and it is recommended that solvent recovery be practised, particularly by drainage, after the coal is removed from the separation tank.

SAFETY PRECAUTIONS

Particular attention is drawn to the fact that many solvent vapours are toxic and present a serious health hazard, and hence adequate ventilation is essential, preferably down-draught (see figure 3). Suitable protection to avoid contact with the skin is also required. Many countries have statutory requirements concerning the use of organic liquids with respect to toxicity and fire; these should be observed.

Equation (2) may be used to calculate the volumes of liquids required in formulating a mixture at the desired relative density. It is important that the relative density of the resultant mixture be checked, for example by means of a hydrometer with maximum scale divisions of 0,002.

$$V_m = V_t \frac{\rho_t - \rho_p}{\rho_m - \rho_p} \qquad \ldots (2)$$

where

V_m is the volume of the liquid with higher relative density;

V_t is the volume of mixture desired;

ρ_m is the relative density of the denser liquid;

ρ_t is the desired relative density of the mixture;

ρ_p is the relative density of the less dense liquid.

6.1.3 Inorganic solutions

Inorganic solutions may often be used in place of organic liquids, but not where any portion of the sample is subject to disintegration in water to an extent which will influence the accuracy of the test.

For samples of less than 8 mm particle size, because of the effect of viscosity on the separation process, longer times of separation are required. The quantity of sample immersed at any one time therefore has to be controlled to achieve complete separation.

Zinc chloride is a commonly used inorganic salt, but it has several disadvantages which should be considered carefully. Zinc chloride solutions are corrosive and hence care should be exercised in the choice of the container used in the test. Furthermore, the pores of the sample frequently become permeated by the zinc chloride solution which is difficult to remove even with prolonged washing with fresh water. The presence of residual zinc chloride may introduce errors in mass and may also affect the analysis of the ash.

WARNING — Zinc chloride must not be allowed to contact the skin.

Where contamination by zinc chloride is likely to affect any of the coal analysis results, a measure of the level of zinc chloride in the wash water should be established.

6.1.4 Solids in aqueous suspension

Insoluble material with a medium to high relative density and the correct particle size distribution may be used to give a relatively stable suspension of low viscosity. Examples of suitable materials are given in table 4.

Table 4 — Suitable solids for aqueous suspensions

Material	Relative density	Nominal top size µm	Comments
Finely ground shale	2,4 to 2,6	250	Discard from a coal preparation plant Brickwork shales
Froth flotation tailings	2,4 to 2,6	250	
Barytes	3,7 to 4,1	63	Commercial barium sulfate
Magnetite	5,0	38	As used in coal preparation plants
Ferrosilicon	6,0	38	Ground or atomized An alloy containing 85 % iron and 15 % silicon

NOTES

1 All of these solids can be used separately or in mixtures. Bentonite may be used to stabilize a suspension.

2 For separations with relative densities above 1,5, ground shale or froth flotation tailings will require an addition of higher relative density materials to avoid viscosity problems.

3 Aqueous suspensions require continuous mixing to keep the solids from settling and to keep the mixture homogeneous, which in turn can affect the gravimetric separation of the coal particles.

The procedure for using aqueous suspensions is similar to that recommended for use with inorganic solutions. The separated fractions are thoroughly washed with water to remove adhering medium. It is important to have a device for accurately determining the relative density of the suspension. A suitable apparatus, which should be calibrated with water at 20 °C, is shown in figure 1.

Aqueous suspensions are non-toxic and non-volatile, and therefore fume extraction is not required. Provided that products are washed free of medium, no adverse effects on product quality occur. Aqueous suspensions are not recommended for testing particles below 4 mm.

6.2 Apparatus

The apparatus should be unaffected by the liquids or suspensions involved and should be convenient for use. Types of apparatus which have been found to be suitable are shown in figures 2 to 5.

The apparatus for separating the float fraction from the sink fraction may consist of a basket with a movable partition which allows the float and sink fractions to be dealt with simultaneously while keeping them separate. Alternatively, a tank with a fine mesh base made to fit inside another tank, forms a useful means of recovering floats and sinks separately. In this case, the floats are skimmed off the liquid surface using a fine mesh scoop and the sinks are recovered by raising the inner perforated base tank and draining off the liquid.

NOTE 7 The mesh used for both the scoop and the basket should have openings small enough to ensure that solid particles are retained.

6.3 Test procedure

6.3.1 Basic method

The separation tank is partly filled with the required medium, the relative density of which is then checked using a suitable hydrometer. The actual relative density should be adjusted to the correct reading, and frequent checking should be carried out to ensure that it remains within the range of ±0,002 of the desired relative density throughout the test.

An increment of the size fraction under test, which is sufficient to form a thin layer, is introduced into the tank containing the medium and gently agitated. Care needs to be taken not to overload the tank, as this is liable to interfere with the separation of entrained near-density material. After allowing sufficient time for separation, the float material is removed and collected on a draining platform or tray. The settled sinks material is agitated to release any entrained float material. This process is repeated until all of the test sample has been separated. The time for separation will vary according to the type of separating medium used and the particle size.

NOTE 8 To reduce the time required for gravimetric separation of coals finer than 1 mm, a centrifugal float and sink procedure may be employed, as described in annex A, provided that the requirements of table 1 are met. The centrifugal float and sink procedure is recommended for minus 0,063 mm material, for improved separation efficiency and reduced separation times. This method gives reliable results only if certain precautions are taken and the liquids are correctly selected.

6.3.2 Testing in ascending order of relative density

This sequence is used where it is known from pilot testing or previous experience that the majority of the sample is of low relative density. The sample is introduced into the medium of lowest relative density. All float material is washed free of medium if necessary, dried in air, weighed and prepared for analysis when required. The sink material is well

drained, care being taken to maintain the relative density of the succeeding liquid, by introducing only completely drained material from the previous test. It is then introduced into the medium of next higher relative density. The float material from this separation is also washed free of medium if necessary, dried in air, weighed and prepared for analysis when required.

This procedure is repeated until all the size fractions have been tested at all relative densities.

In this way, a large proportion of the sample is removed from the test at the first separation, thereby reducing handling and breakage.

6.3.3 Testing in descending order of relative density

This sequence is used where it is known from pilot testing or previous experience that most of the sample is of high relative density. The procedure for testing is similar to that described in 6.3.2, except that the test sample is introduced firstly into the medium of highest relative density. The sinks fraction is washed free of adhering medium if necessary, dried in air, weighed and prepared for analysis when required. The float fraction is thoroughly drained and introduced into the medium of the next lower relative density. Incompletely drained material may alter the relative density of the next liquid. As described already, the sink material is washed free of medium if necessary, dried in air, weighed and prepared for analysis when required. This process is repeated until separation takes place in the medium of lowest relative density.

As with testing in ascending sequence, a large proportion of the sample is removed from the test at the first separation, thereby reducing handling and breakage.

6.3.4 Testing procedure for samples containing small proportions of material with intermediate relative density

Where it is known from pilot testing or previous experience that the majority of the test sample is a mixture of materials of both low and high relative density with very little material of intermediate relative density, the sample is introduced firstly into the medium of low relative density. The float fraction is drained, washed if necessary, air dried, weighed and prepared for analysis when required.

The sink fraction is drained and transferred to the medium of highest relative density. The sink fraction from this separation is drained, washed if necessary, air dried, weighed and prepared for analysis when required.

The float fraction, now of relatively small mass, can be introduced into a medium in either ascending or descending order of relative density and treated as in 6.3.2 or 6.3.3.

This method eliminates the need to move large masses of material through the intermediate relative densities.

6.3.5 Testing procedure for samples containing a large proportion of material with intermediate relative density

Where it is known from pilot testing or previous experience that the sample contains a large proportion of material of intermediate relative density, the sample is introduced into a medium of intermediate relative density which will give an approximately even split in the mass to be further treated. The floats fraction is then tested in ascending order of relative density as in 6.3.2, and the sinks fraction is tested in descending order of relative density as in 6.3.3.

7 Presentation of results

7.1 Size analysis

The results of a size analysis may be presented as shown in table 2. If a graphical representation is necessary, it should be prepared in accordance with ISO 1953.

7.2 Float and sink data

The results of float and sink testing of each size fraction may be recorded as shown in table 5. Initially, the results are recorded on the basis of 100 % recovery for each size fraction. The cumulative float and sink data for that size fraction are then calculated from these values. The cumulative data may be presented graphically in the form of washability curves (see figure 6) or a Mayer curve (M-curve, see figure 7).

Similar tables may be prepared and presented graphically for combinations of size fractions, on the size analysis proportional basis. Other determined parameters, such as the total sulfur content, may also be included in table 5. The results of float and sink analysis of the products of a cleaning process should be presented in accordance with ISO 923.

7.3 Washability curves

In order to produce full washability curves, it will be necessary to determine the ash percentage of each relative density fraction in each size range. Other parameters such as sulfur content and calorific value may be assessed, recorded and presented using the same technique.