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# Coal — Determination and presentation of float and sink characteristics — General directions for apparatus and procedures

*Charbon — 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

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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, *Coal and coke*, Subcommittee SC 1, *Coal preparation: Terminology and performance*.



This second edition cancels and replaces the first edition (ISO 7936:1992), which has been technically revised.

The main changes are as follows:

— addition of new procedures for the use of inorganic solutions, such as caesium and potassium formates, and for aqueous suspensions, such as zirconium dioxide for float and sink analysis.

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 results of float and sink testing, presented in tabular and graphical form, are the basis for the provision of washability data.

The results of float and sink data from coal seam samples provide an estimation of the future quality and yield of washed coal from the area of the coal lease where the samples were taken.

The results of float and sink data from coal seams and preparation plants are also used when designing a new plant and /or redesigning an existing plant, and also in predicting, controlling and assessing the performance of an existing plant in total or in part.

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.

The following annexes provide new additional information in this revision as follows:

Annex A Drop shatter – A pre-treatment of samples for float and sink testing;

Annex B Wet tumbling – A pre-treatment of samples for float and sink and testing;

Annex C Sample masses for float and sink testing;

Annex D Validation of data from float and sink analysis;

Annex E Interpretation of data from float and sink analysis;

Annex F Guide to the safe use of organic solutions.

# Coal — Determination and presentation of float and sink characteristics — General directions for apparatus and procedures

## 1 Scope

This document specifies general directions for the apparatus and procedures, using relative density separation methods, for determining the float and sink characteristics of samples from coal seams and of feed, products and rejects from coal preparation plants.

## 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 1213-1, *Coal and coke — Vocabulary — Part 1: Terms relating to coal preparation*

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

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

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, *Coal and coke — Manual sampling*

## 3 Terms and symbols

For the purposes of this document, the terms and definitions given in ISO 1213-1 and ISO 1213-2 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/

## 4 Sampling

### 4.1 General

Samples for float and sink testing are mainly sourced from three major areas:

a) coal seams from underground and open cut mines;

b) coal preparation plants;

c) bore core.

## 4.2 Sample mass

Sampling shall be carried out in accordance with ISO 13909-1, ISO 13909-2, ISO 13909-3 or ISO 18283.

The following standard sampling guides should also be considered:

a) ISO 14180: Typical samples are bulk raw coal samples, channel samples, rotary drilled cuttings and core samples of various diameters;

b) ISO 4077: Typical samples are raw feed, clean coal and reject from the plant in total or from various parts of the plant such as cyclones.

The minimum mass of sample from coal seams (raw coal) and coal samples from a coal preparation plant required for float and sink testing are outlined in Table 1. The number of discrete particles to be aimed for in any size fraction of the sample should not be less than 2 000. The masses given in Table 1 generally ensures that the number of particles is adequate. However, these masses may not be practicable in the case of bore cores or some coal preparation plant products.

The mass of the coal seam bulk sample or large plant sample should be enough to contain the minimum quantities in each fraction as listed in Table 1. Where taking a coal seam bulk sample or a large sample from a plant, it is better to over-sample than to have insufficient material. However, in order to carry out testing on a coal seam raw coal bulk sample at the larger sizes in Table 1, the sample may have to be the order of 10 tonnes, or even greater. For example, in a newly opened mine, a trial shaft or other appropriate location, the mass of bulk sample taken should not be less than 10 tonnes.

For cores, particularly small diameter 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. If these requirements cannot be met, this fact shall be noted in the test report.

In coal preparation plants, some coals may give low yields in the intermediate relative density fractions. Consequently, 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. Refer to ISO 4077 for further guidance in this area.

This document strongly recommends that the sampling and preparation protocols and advice given in this clause, particularly those relating to the mass of sample for float and sink analysis, are followed carefully as, if not, the resultant results of any float and sink analysis can be compromised.

Samples with different particle sizes contain particles with different proportions of mineral matters and organic matters, which produces different washability (different float and sink distribution). Consequently, it is critical that a representative sample of the relevant size distribution is provided for float and sink testing.

It is assumed that square-mesh particle sizes are used; if wedge-wire or round-hole sizes are used, this fact should be reported. When a bulk sample is being taken, it is better to over-sample than to have insufficient material.

For testing on the top-sizes shown in Table 1, the bulk sample mass may be up to 20 t, and for other sizes the mass is reduced according to the decrease in nominal top-size.

NOTE The importance of enough sample mass and a method for the determination of the required mass of a bulk sample is given in Annex C. For further information on sample masses for float and sink testing and examples of calculations to determine masses needed at various size distributions, see Annex C. Refer to ISO 4077 for further guidance in this area.

**Table 1 — Minimum mass for a given size fraction**

| Size fraction mm | Sample mass[a,b] kg | | |
|---|---|---|---|
| | Raw coal | Clean coal | Reject |
| −125 + 63 | 2 150 | 1 810 | 2 680 |
| −63 + 31,5 | 300 | 250 | 370 |
| −50 + 31,5 | 230 | 190 | 280 |
| −31,5 + 16 | 40 | 34 | 50 |
| −16 + 8 | 5,2 | 4,4 | 6,5 |
| −8 + 4 | 2,0 | 2,0 | 2,0 |
| −4 + 2 | 2,0 | 2,0 | 2,0 |
| −2 + 1 | 2,0 | 2,0 | 2,0 |
| −1 + 0,5 | 2,0 | 2,0 | 2,0 |
| −0,5 + 0,25 | 1,0 | 1,0 | 1,0 |

NOTE The basis for calculating the number of particles was as follows:
A Rosin and Rammler (Weibull function) size distribution was applied to the default sample, using parameters of $\bar{x}$ (size constant) = 30 mm, and $n$ (slope) = 0,60. The number of particles within each size fraction was calculated by fractionating each individual size fraction by mass into 1 mm (or smaller) sub-fractions. The volume of each particle in each sub-fraction was calculated using the particle RD stated above, and a shape factor of 1,25. Thus, if the size sub-fraction was – 60,5 + 60 mm, the particle in the sub-fraction was assumed to have the following dimensions: 60 mm × 60 mm × 75 mm.

[a] For control samples from a preparation plant as an example, where successive test results can be averaged, the mass shown in Table 1 may be reduced by approximately one-half.

[b] The sample masses in Table 1 are calculated from the required number of particles and have been calculated based on the following assumed particle relative densities (RD): Raw Coal 1,60, Clean coal 1,35, Reject 2,00 (see Annex C for calculation examples to determine bulk sample masses).



Both the size distribution and the ash mass fraction 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, $m_t$, in kg required for a float and sink test is given by Formula (1):

$$m_t = \frac{m_r}{w_s} \times 100 \tag{1}$$

where

$m_r$ is the recommended mass of coarsest size fraction (from Table 1), kg;

$w_s$ is the mass fraction of the coarsest size fraction in the sample, %.

## 4.3 Coal preparation plant products

Since the relative densities of some components, such as reject 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 be in accordance 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.

For 4.3, 4.4 and 4.5, see also ISO 4077 which provides further advice on masses required for plant products, control testing and efficiency tests and various combinations of all three items.

### 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 Table 1 should be used.

### 4.5 Comprehensive plant efficiency test

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 fractions of the raw feed, the mass and moisture fractions "as determined" of all cleaned products, discard, etc., and the volume and solids mass fractions 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 mass fractions. 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.



### 4.6 Core samples



For core samples, guidance is given in Annex C and ISO 14180.

### 4.7 Preliminary treatment

Many coal samples, such as strip and core samples require pre-treatment to better simulate the size distribution of the raw coal feed to a coal preparation plant. This pre-treatment ensures more accurate representation of fines mass fraction, which in turn affects washability results.

The pre-treatment process can involve any or a combination of the following.

a) Drop shatter — The picking up and dropping of a sample onto a steel plate under specific conditions.

b) Top-size reduction — This process requires oversize material to be reduced to pass a nominated screen, with a minimal amount of fine material being produced. Top-size reduction does not simulate the size distribution of coal preparation plant feed, because the coal particles are not selectively broken.

c) Various methods can be utilized to perform this procedure, including the following:

   1) Jaw crusher — The sample is choke-fed to the crusher with the aim of producing the nominated size;

   2) Hand knapping — The sample is broken using hand-held implements. Done carefully, this procedure can yield the least amount of fine material.

d) Hammermill type crushers shall not be used for size reduction, because of the excess amount of fine material produced apart for final crushing to minus 212 µm for analysis.

e) Dry tumbling — The sample is tumbled end over end in a drum under specified conditions.

f) Wet tumbling — The sample is tumbled end over end after the addition of water and under specified conditions.

NOTE See Annexes A and B for more information on drop shatter and wet tumbling.

## 4.8 Size analysis

The sample should be spread out on an impervious base, preferably under shelter, and allowed to dry sufficiently for screening purposes. After the sample has been dried, the sample 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.

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

NOTE Pulp and reject samples are screened promptly to avoid excessive shale breakdown.

**Table 2 — Size analysis**

| Size fraction (square hole) | | Mass fraction | Material retained | Material passing |
|---|---|---|---|---|
| mm | | % | % | % |
| +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 |
| -10,0 | +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 | | | | |



## 4.9 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 that 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.

# 5 Separation media

## 5.1 General

The medium which is to be used for the separation is generally a mixture of organic liquids as described in Table 3. Aqueous solutions of inorganic salts (see Table 4), or solids in aqueous suspensions (see Table 5) are acceptable but shall be validated prior to use.

The most suitable type of medium is determined by the type of testing required, particle size of the sample and relative densities required.

Where tests are conducted in exposed situations, samples should be protected from airborne contaminants and wind loss.

## 5.2 Organic solutions

### 5.2.1 General

**WARNING — Particular attention is drawn to the fact that organic liquids and their vapours are toxic and present a danger to health. The user of such liquids shall conform to the relevant safety data sheet, and be aware of any statutory regulations.**

Where relative densities of 1,6 and less are required, a mixture of perchloroethylene and white spirit shall be used.

Where relative densities in the range of 1,6 to 2,9 are needed, tetrabromoethane (TBE), acetylene tetrabromide or bromoform may be mixed with perchloroethylene.

Organic liquids shall be used sparingly, and liquid recovery shall be undertaken, particularly by drainage, after removal of the sample from the separation tank.

Organic liquids are hazardous but are preferred to alternative media because of their low viscosity, ease of use and the fact that they are inert towards shales. Prolonged washing and drying times are unnecessary for the products of the separation owing to the generally high volatility of these liquids.

To accelerate drying and to minimize contamination, float and sink fractions separated by mixtures containing TBE, shall be rinsed with a more rapidly evaporating compatible organic liquid, such as white spirit.

NOTE The physical properties of the organic liquids used in float and sink testing are shown in Table 3.

**Table 3 — Physical properties of organic liquids**

| Organic liquid | Relative density<br>at 20 °C | Distillation range or boiling point<br>°C at 100 kPa | Viscosity at 20 °C<br>mPa.s | Vapour pressure at 20 °C<br>kPa | Flammable |
|---|---|---|---|---|---|
| White spirit | 0,77 | 30,0 to 200,0 | — | — | Yes |
| Bromoform (tribromomethane) | 2,89 | 149,5 | 2,152 (15 °C) | 0,70 | No |
| Tetrabromoethane (TBE, acetylene tetrabromide) | 2,96 | 239,0 | 12,00 | 0,01 | No |
| Perchloroethylene | 1,61 | 121 | 0,89 | 1,83 | No |
| NOTE Refer to the vendor's Safety Data Sheet for current information regarding physical properties. | | | | | |

### 5.2.2 Limitations on accuracy

Organic liquids may react with the coal and despite thorough drying after float and sink testing, some component of organic liquids could remain. This retention may influence subsequent analyses

and tests, for example, chlorine, trace elements, fly-ash precipitability, moisture-holding capacity and caking properties such as fluidity.

Absorption of organic liquid into the coal may also affect the apparent relative density of the particle, which can lead to inaccuracies in the separation.

## 5.3 Inorganic solutions

### 5.3.1 General

A water-soluble salt (e.g. caesium or potassium formate) shall be used as a dense medium for float sink testing because of the lower density of potassium formate, its application is restricted to low density separation or its use in a blend with caesium formate.

### 5.3.2 Formate solutions

#### 5.3.2.1 General

The float sink procedure for water soluble salt solutions shall be the same as that outlined in Clause 6. The major differences compared to using organic liquids are the preparation of the dense media, sample rinsing and recovery of dilute media.

NOTE The physical properties of the inorganic solutions used in float and sink testing are shown in Table 4.



**Table 4 — Physical properties of inorganic solutions**

| Formate solution | Relative density at 20 °C | Distillation range or boiling point (°C at 100 kPa) | Viscosity at 20 °C mPa.s | Vapour pressure at 20 °C kPa | Flammable |
|---|---|---|---|---|---|
| Caesium formate | 2,20 | 137 | 2,30 | 0,63 | No |
| Potassium formate | 1,57 | 142 | 13,2 | 0,62 | No |
| NOTE Refer to the vendor's Safety Data Sheet for current information regarding physical properties. | | | | | |



#### 5.3.2.2 Limitations on accuracy

The use of an inorganic solution such as caesium formate shall be thoroughly validated before its use to ensure washability results are accurate and any subsequent laboratory testing of float sink fractions is not compromised.

Particular attention shall be given to the following concerns, which can limit the use of caesium formate.

a) Caesium formate may contaminate coal particles even after rinsing. This can affect subsequent properties to be tested, in particular ash.

b) Some coal or mineral matter particles can disintegrate in a water-based solution.

c) Density stability — Formate solutions with densities above RD 1,70 absorb moisture from the air resulting in a decrease in the density. Solutions with densities below RD 1,70 lose moisture resulting in a gradual increase in density.

#### 5.3.2.3 Preparation of solutions

Caesium formate is readily soluble in water and therefore the solution can be diluted with potable tap water to give densities in the normal working range of float and sink testing.

#### 5.3.2.4 Sample rinsing

Particles shall be rinsed with water while vacuum filtering to remove caesium formate. The dilute rinsings may then be processed to recover the caesium formate.

#### 5.3.2.5 Recovery of media

If required, the recovery of caesium formate from dilute washings can be achieved by either distillation (e.g. using an immersion heater), vacuum evaporation or reverse osmosis.

### 5.4 Aqueous suspensions

#### 5.4.1 General

An insoluble material with a high relative density and correct particle size distribution shall be used to give a relatively stable suspension of low viscosity.

Zirconium dioxide is an example of an aqueous suspension that is suitable.

#### 5.4.2 Zirconium dioxide

##### 5.4.2.1 General

The float and sink procedure for aqueous suspensions shall be the same as that outlined in Clause 6. The major differences compared to using organic liquids or inorganic solutions are the preparation of the dense media, sample rinsing and recovery of dilute media. Zirconium dioxide is chemically inert and has a particle density of 5,75 allowing a range of aqueous suspensions to be prepared.

NOTE The physical properties of the aqueous suspensions used in float and sink testing are shown in Table 5.



##### 5.4.2.2 Limitations on accuracy

The use of an aqueous suspension such as zirconium dioxide shall be thoroughly validated before its use to ensure washability results are accurate and any subsequent laboratory testing of float and sink fractions is not compromised.

Particular attention shall be given to the following concerns, which may limit the use of zirconium dioxide.

a) Zirconium dioxide may contaminate particles even after rinsing. This contamination could affect subsequent properties to be tested, in particular the ash in the sample.

b) Some coal or mineral matter particles may disintegrate in a water-based solution.

c) Zirconium dioxide suspensions are opaque. Therefore, the time required for the completion of the separation of floats from sinks cannot be judged by visual observation and has to be pre-determined by experiments combined with theoretical calculations.

d) The stability of the aqueous suspension should be known and monitored. Note that lower density suspensions are the least stable.

e) Zirconium dioxide is not a suitable media for separation of fine particles (< 1 mm).

**Table 5 — Physical properties of aqueous suspensions**

| Aqueous suspension | Relative density at 20 °C | Distillation range or boiling point<br>°C at 100 kPa | Viscosity at 20 °C<br>mPa.s | Vapour pressure at 20 °C<br>kPa | Flammable |
|---|---|---|---|---|---|
| Zirconium dioxide | 1,30 to 2,20 | — | 11,2 | — | No |
| NOTE Refer to the vendor's Safety Data Sheet for current information regarding physical properties. | | | | | |

#### 5.4.2.3 Preparation of suspension

A very fine zirconium dioxide powder shall be prepared with water and electrostatically stabilized using a suitable additive, such as a polyelectrolyte. The preparation procedure shall be as follows.

a) Prepare a water solution containing the stabilization agent.

b) Add sufficient zirconium dioxide powder to the agitated solution prepared in step a) to produce suspensions with a relative density of 2,00 to 2,20.

c) Pass the suspension prepared in Step (b) twice through a colloid mill.

d) Measure the density of the stock suspension and store in a sealed container to avoid the evaporation of water.

NOTE Zirconium dioxide suspension can be diluted with potable tap water to give densities in the normal working range of float and sink testing.



#### 5.4.2.4 Sample rinsing

Particles shall be washed by spraying with water on a vibrating sieve or screen.



NOTE The amount of suspension retained on the surface of the particles increases as the particle size decreases because of the higher specific surface area. Further, the amount of suspension retained on the particles increases as the viscosity increases, as the suspension can fail to drain completely.

#### 5.4.2.5 Recovery of media

Dilute suspension media shall be recovered by concentration using one of the following options.

a) Recovering zirconium dioxide particles from a dilute suspension by reducing its pH to 6 or less to allow fast settling; or

b) Using a ceramic membrane to re-concentrate diluted suspensions; or

c) Leaving dilute suspensions in a container for a long period of time to allow slow settling.