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Second edition
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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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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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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) mm		Mass fraction %	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
-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 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
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 predetermined 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 °C at 100 kPa	Viscosity at 20 °C mPa.s	Vapour pressure at 20 °C 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.

Table 6 — Other examples of solids[a] for aqueous suspensions[c]

Material	Relative density[b]	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

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

[b] 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.

[c] 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.

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.

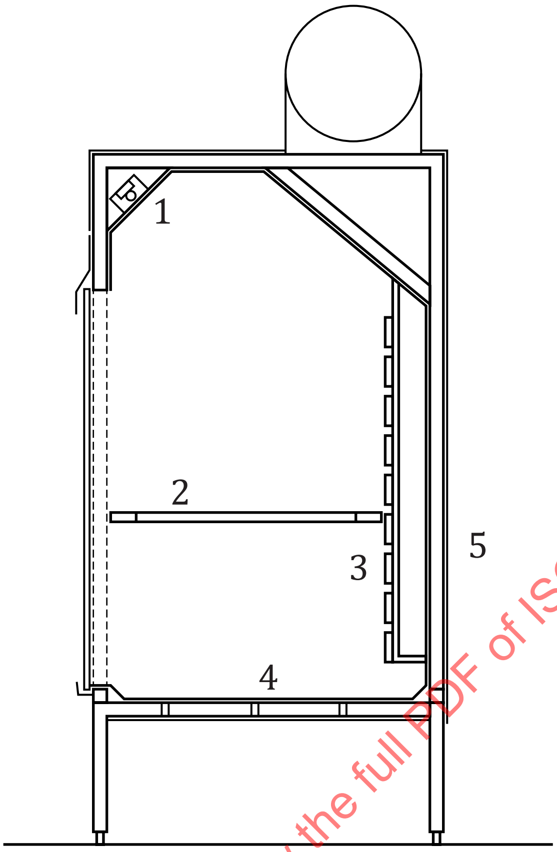
6 Apparatus

6.1 General

The testing apparatus shall be unaffected by the test media, robust, suitable for the purpose, convenient and safe to use. A variety of apparatus is used, depending primarily on the particle size distribution of samples being processed (i.e. coarse coal or fine coal).

Figure 1 and Figure 2 show common arrangements for a coarse float and sink cabinet and a typical float and sink tank arrangement. Figures 3, 4 and 5 show typical float and sink laboratory equipment.

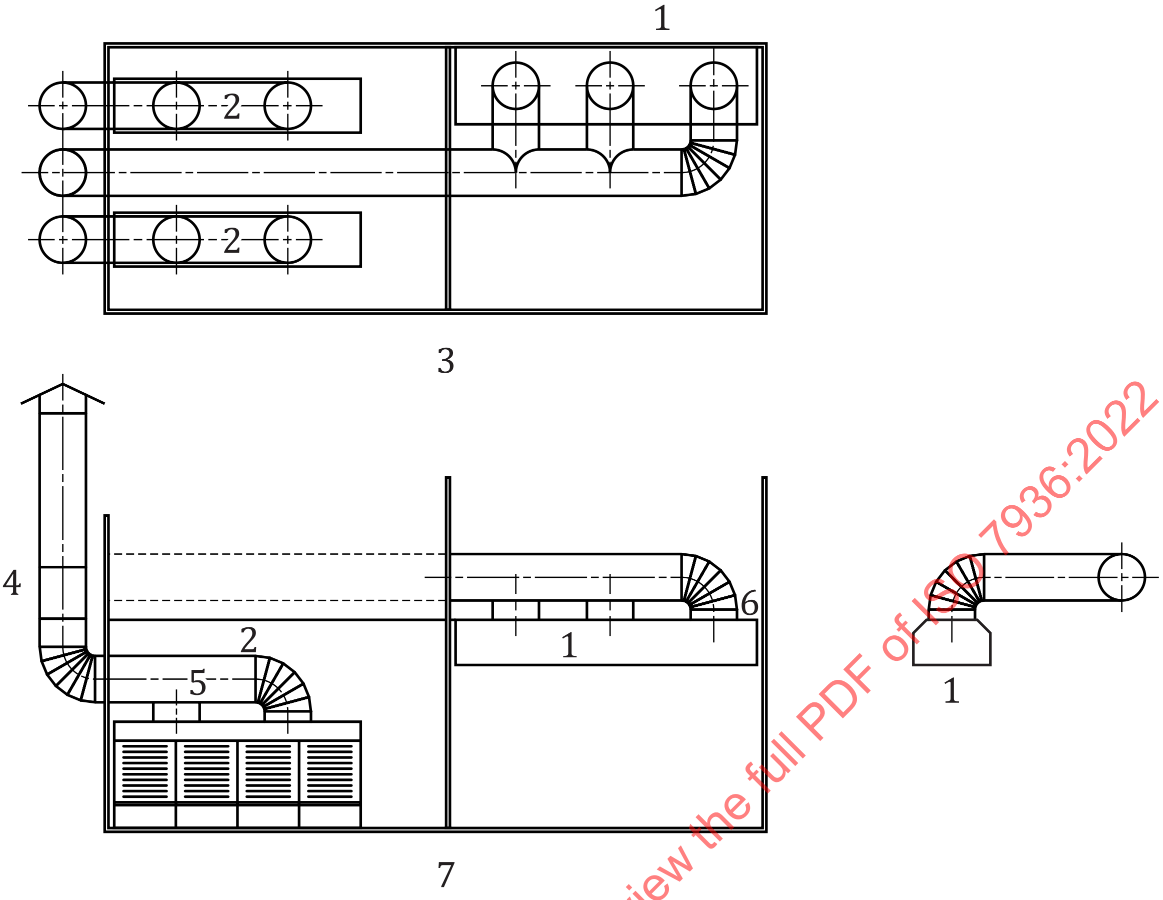
Mechanical handling is recommended where large quantities are to be treated.



Key

1	interior
2	shelf
3	slats
4	floor
5	exterior

Figure 1 — Float and sink cabinet



Key

1	drying hood	5	duct
2	rope pulleys	6	fan
3	plan view	7	elevation view
4	inline fan		

Figure 2 — Example of float and sink tank arrangement

6.2 Coarse coal apparatus

Equipment for coarse float and sink test work typically consists of the items listed in 6.2.1 to 6.2.12.

6.2.1 Rectangular or cylindrical tank, with an inner mesh basket is shown in Figure 3. This arrangement provides a convenient means of recovering floats and sinks separately.

Figure 3 — Coarse coal float and sink apparatus

6.2.2 Inner mesh basket.

Sinks are recovered by raising the inner basket which is then supported above the tank to allow liquid to drain. Baskets should be fitted with suitable lifting handles. Mesh apertures shall be suitable to contain all particles within the basket.

6.2.3 Mesh scoop of suitable aperture.

Floats are skimmed off the surface of the liquid using a mesh scoop of suitable aperture as shown in Figure 4.

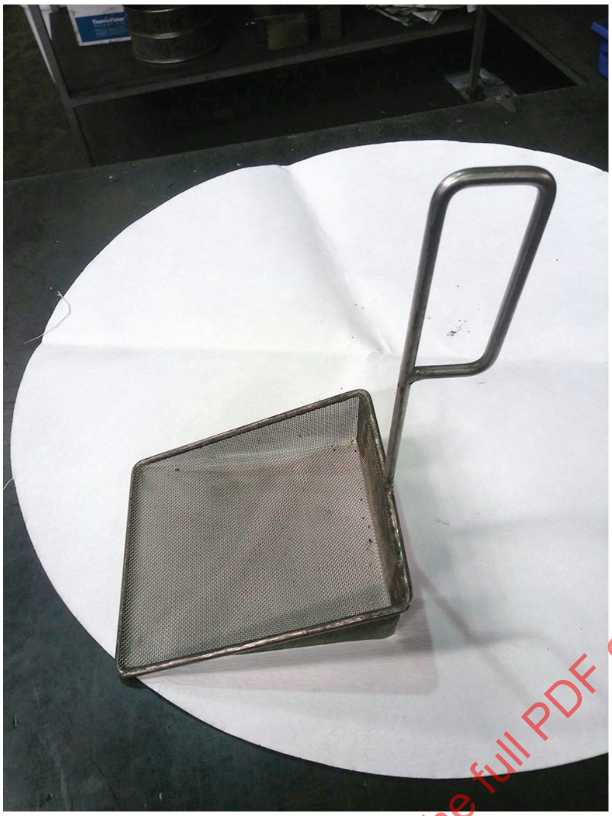


Figure 4 — Float and sink meshed scoops

6.2.4 Stirring rods, such as those shown in Figure 5, are used to agitate the contents of the separation tank. They shall be made from materials impervious to the dense media.

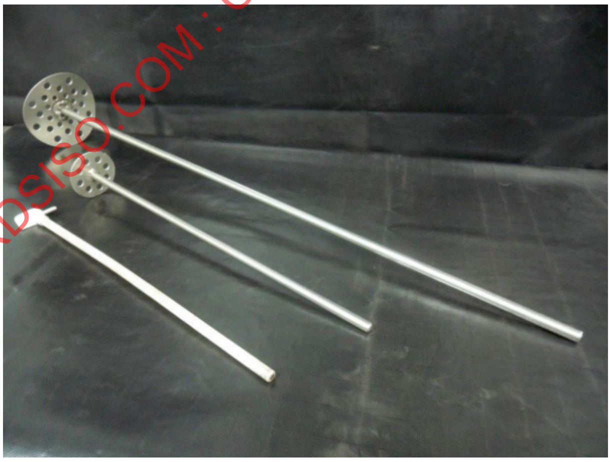


Figure 5 — Float and sink stirring apparatus

6.2.5 Hydrometers.

Maintaining the required relative densities of the media is a critical aspect of the test and requires monitoring and control throughout the whole float and sink operation. Methods for calibration of hydrometers are described in Annex G. An example of appropriate storage of hydrometers is shown in

Figure 6. Recommendations on the types of hydrometers suitable for this purpose and requirements for use are as follows:

a) types M100 or M50 having scale subdivisions of less than or equal to 0,002;

b) under normal working conditions, there is no need to apply temperature, meniscus or surface tension corrections;

c) readings on the hydrometer stem shall correspond to the bottom of the meniscus.

Figure 6 — Hydrometer storage

6.2.6 Sample containers, such as plastic buckets may be used to store samples prior to testing.

6.2.7 Drying trays, shall be made from materials impervious to the dense media used. They shall also be suitable for drying at temperatures above ambient.

6.2.8 Sample labels, may be a convenient means of identifying the various float and sink fractions.

6.2.9 Lids for media containers.

To minimize evaporation, all media containers shall be fitted with lids when not in use.

6.2.10 Balance, which shall be capable of measuring to the following sensitivity:

≥ 100 g to the nearest 0,1 %.

< 100 g to the nearest 0,1 g.

6.2.11 Air-swept drying cabinet. If appropriate, this may be heated. The cabinet shall have facilities for the safe venting of vapours.

6.2.12 Filtration apparatus. Filtration of particles after float and sink test work is recommended to remove excess medium from the samples prior to drying. This may include both gravity and vacuum equipment.

6.3 Fine coal apparatus

6.3.1 Glass separating flask.

The separating vessel is normally made from glass so that the separation can be confirmed visually. A tap at the bottom of the vessel is used to separate the float and sink material. The size of the bore shall be sufficient for the top-size of the coal being tested so that blockages are avoided.

The apparatus shown in Figure 7 has been found to be useful in separating fine particles of varying density and offers the following advantages:

a) Limited evaporation for the duration of the separation.

b) Ready means of recovering floats and sinks fractions.

c) Small volume of liquid medium needed.

d) Visual confirmation of the separation process.

Figure 7 — Fine coal separating apparatus

For additional apparatus for fine float and sink, see also 6.2.5 to 6.2.12.

6.3.2 Test sieves, in accordance with the requirements of ISO 1953.

7 Float and sink testing procedures

7.1 General

The float and sink procedures described in this clause are suitable for organic solutions (see Annex F) or aqueous solutions and suspensions. However, if aqueous liquids are used, special attention shall be given to the rinsing of particles and recovery and re-use of media.

Float and sink testing can be affected by particle degradation and hence, at all stages of the test, sample handling techniques that minimize breakage should be adopted.

7.2 Relative densities of test media

Comprehensive tests can include any number of relative densities in the range from 1,25 to 2,40.

Numerous relative densities within this range are often considered necessary for accurate interpolation of data, for example, for raw coal samples where cumulative ash is increasing rapidly in relation to cumulative yield, or for plant performance test samples at relative densities close to the plant separating density.

Formula (2) shall be used for determining the volumes of liquids required in formulating a mixture at the desired relative density:

$$V_m = \frac{V_t\left(\rho_t - \rho_p\right)}{\left(\rho_m - \rho_p\right)} \tag{2}$$

where

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

V_t is the volume of mixture desired;

ρ_t is the desired relative density of the mixture;

ρ_p is the relative density of lower relative density liquid;

ρ_m is the relative density of higher relative density liquid.

As an example, in order to make up 30 L of medium at RD 1,70, using a mixture of bromoform (RD 2,89) and perchloroethylene (RD 1,61):

$$V_m = \frac{V_t\left(\rho_t - \rho_p\right)}{\left(\rho_m - \rho_p\right)}$$

$$V_m = \frac{30(1{,}70 - 1{,}61)}{(2{,}89 - 1{,}61)}$$

$$V_m = 2{,}11$$

Consequently, 2,11 l of bromoform and 27,89 l of perchloroethylene will be required to make up 30 l of medium at 1,70.

NOTE The range and number of relative density separations to be made are generally not fixed and vary depending on the purpose of the test and individual requirements. For example, routine plant control tests can require use of only one relative density, as a guide to the level of misplaced material. Comprehensive testing of raw coals or plant products, where assessment of economics, plant design or plant performance is under investigation, can require eight or more relative densities.

Where actual requirements are uncertain, the most suitable relative densities may have to be determined by trial and error; adjustments by way of extra relative densities should be made before crushing the preliminary float or sink fractions for analysis.

Consideration should be given to developing a database that is consistent in terms of the number of and actual relative densities prescribed for washability analysis. This will facilitate blending of washability analyses for reserve calculations and ensure that all of the data can be used.

7.3 Testing of coarse size fractions

7.3.1 General

A typical distinction for the transition between coarse and fine float and sink procedures is 0,5 mm.

Because of the porous nature of the coal matter, which will be most impacted by the absorption of the dense medium, it is preferable that the test commences at the lowest relative density and proceeds in increasing relative density order.

7.3.2 Procedure

The procedure for treatment of coarse size fractions shall be as follows:

a) Ensure the separation tank is partly filled with clean medium. The tank shall be filled to approximately 85 % full by adding appropriate liquids.

b) Stir the tank contents thoroughly.

c) Conduct the hydrometer reading by taking an aliquot of the medium from the separation tank, placing this aliquot into a measuring cylinder or beaker, then carefully place the calibrated hydrometer into the medium to read the relative density.

d) Adjust the relative density in the separation tank to ± 0,002 of the required relative density by adding appropriate liquids.

e) Repeat steps b) to d) throughout test at regular intervals.

NOTE 1 Variation in the relative density of the liquids result from changes in liquid temperature, evaporation and contamination. The necessary frequency of density checking will be a function of the circumstances under which the test is conducted.

f) Place a clean, dry inner basket into the separation tank.

g) Slowly place an increment of the sample being tested into the separating liquid. The mass of this increment shall be such that only a thin layer is formed on the surface of the medium in the separation vessel.

Care should be taken not to overload the tank, as this can interfere with the separation of material. The layer of floats should not be thicker than the greater of 10 mm or the maximum particle diameter of the size fraction being tested.

h) Stir the surface layer occasionally using a suitable implement to promote unhindered separation of sinks material. Allow time for the liquid to become quiescent, and for floats and sinks to separate completely.

NOTE 2 The time needed for complete separation varies with particle size, viscosity of liquid and the differential in relative density between individual particles and the separating medium. Typically, +11,2 mm particles separate within one minute, whereas 0,5 mm particles can take as much as 30 min.

i) When the floats and sinks have separated completely, remove the floats using a suitable mesh scoop, taking care not to disturb the sinks, and deposit them on a draining platform or inclined tray.

NOTE 3 The float particles are generally considered to be in the top 25 % of the liquid depth.

j) Subject to adequate sinks capacity remaining in the separating vessel, repeat the above steps until all of the test sample is treated at this one relative density.

Accumulated sinks should not occupy any more than 25 % of the liquid depth. If sinks reach this level, the test should be interrupted and sinks removed according to step k), before recommencing at step g);

k) When all the sample has been processed (or at an earlier stage if sinks capacity is reached), agitate the sinks to release any floats that might have become entrapped during the test. Allow sinks to re-settle and recover any floats.

NOTE 4 Agitation can be achieved by partially raising the inner basket two or three times in quick succession and plunging it firmly downwards without splashing the medium. Alternately, stirring of the sinks can be used if practical.

l) Remove the inner basket and support it over the tank, allowing the sinks to substantially drain. Then transfer the sinks for drying.

m) On recovery of the floats and sinks material from the separation tank, label the floats and sinks fractions as appropriate (see NOTE 5), and record the relative density at which the separation was conducted.

NOTE 5 As an example, products from a separation at 1,40 relative density will be labelled “F 1,40” and “S 1,40”; if the sample being tested is a product from a previous float and sink operation, labelling takes the following form:

For sinks 1,40 re-separated at 1,50: “S 1,40 - F 1,50” and “S 1,50”.

Materials used for labelling should not be rendered illegible by the action of the medium being used.

Where floats or sinks are not to be re-separated at another relative density, allow to air-dry, and determine the mass using a balance as described in 6.2.10.

Where floats or sinks are to be re-separated at other relative densities, re-commence the procedure at step a) for succeeding relative densities as required.

Immediately after completion of the test at all required relative densities, check the calculated recombined mass of the air-dried fraction against the starting mass to ensure the absence of any significant gain or loss. Annex D shall be used for validation of data if the variation in mass is greater than 2 %.

Extract subsamples for analysis if required, noting any requirements for subsequently preparing composite analysis samples. See 4.2.

7.3.3 Air drying

Unless air-drying at elevated temperatures is specifically permissible for the sample, air drying shall be conducted at an ambient temperature not exceeding 40 °C. Air-swept drying cabinets will accelerate this process. Temperatures above ambient shall not be used where detrimental effects may result for the sample, or where the possibility of spontaneous combustion exists. (Refer to ISO 13909-4 or ISO 18283 for a guide to drying conditions.) Drying temperatures of 110 °C shall not be exceeded. Where samples are dried at temperatures above ambient, this shall be recorded.

If any form of oven is used, attention shall be given to continuous, safe exhausting of fumes.

7.4 Testing of fine size fractions

7.4.1 General

The mass of fine coal samples (typically sizes less than 0,5 mm and greater than 0,038 mm) for float and sink analysis are usually comparatively small. Separations can take less than 10 min, but can often be difficult and may take several hours to reach completion at each relative density, particularly where large proportions of near-density material are present.

Because of the porous nature of the coal matter it will be most impacted by the absorption of the dense medium. It is preferable that the test commences at the lowest relative density and proceeds in increasing relative density order.

7.4.2 Procedure

The procedure for treatment of fine size fractions shall be as follows.

a) Close the drain valve. Pour a sufficient quantity of medium into the separating vessel. The separation vessel shall be filled to approximately 85 % full.

b) Stir the vessel contents thoroughly.

c) Conduct the hydrometer reading by taking an aliquot of the medium from the separation vessel, placing this aliquot into a measuring cylinder or beaker, then reading the relative density using a calibrated hydrometer.

d) Adjust the relative density in the separation vessel to± 0,002 of the required relative density by adding appropriate liquids.

e) Repeat steps b) to d) throughout the test at regular intervals (see Note).

NOTE 1 Variation in the relative density of the liquids result from changes in liquid temperature, evaporation and contamination. The necessary frequency of density checking will be a function of the circumstances under which the test is conducted.

f) Carefully place an increment of the sample being tested into the separating medium. The mass of this increment shall be such that only a thin layer is formed on the surface of the medium in the separation vessel.

Care should be taken not to overload the vessel, as this may interfere with the separation of material. The layer of floats should not be thicker than about 10 mm.

g) Stir the sample within the separation vessel, using a suitable implement to ensure that all sample is wetted with the medium. Allow sufficient time for separation, re-stir, then allow time for the liquid to become quiescent, and allow time for floats and sinks to separate.

h) When the float and sink separation has been confirmed visually, open the outlet valve to discharge sinks for filtration and recovery of associated medium.

i) Close the outlet valve and change the filter papers.

j) Discharge the floats through the outlet valve directly onto filter papers and recover the associated undiluted medium.

NOTE 2 The floats are typically considered to be contained within the top 25 % of the medium volume.

On discharge of the floats and sinks material from the separation vessel, label the floats and sinks fractions as appropriate (see NOTE 3), and record the relative density at which the separation was conducted.

NOTE 3 As an example, products from a separation at 1,40 relative density would be labelled “F 1,40” and “S 1,40”; if the sample being tested is a product from a previous float and sink operation, labelling would take the following form: For sinks 1,40 re-separated at 1,50: “S 1,40 - F 1,50” and “S 1,50”.

Materials used for labelling should not be rendered illegible by the action of the medium being used.

Where floats or sinks are not to be re-separated at another relative density, allow to air-dry, and determine the mass using a balance as described in 6.2.10.

Where floats or sinks are to be re-separated at other relative densities, re-commence the procedure at step a). Repeat at succeeding relative densities as required.

Immediately after completion of the test at all required relative densities, check the calculated recombined mass against the starting mass to ensure the absence of any significant gain or loss in mass. Annex D shall be used for validation of data if the variation in mass is greater than 2 %.

Extract subsamples for analysis if required, noting any requirements for subsequently preparing composite analysis samples.

7.4.2.1 Air drying

The air drying of samples shall be conducted as outlined in 7.3.3.

7.4.2.2 Cleaning of liquid media and apparatus

Fine particles may tend to accumulate in the test media, and sludges may deposit in separating vessels. These shall be removed at regular intervals by drainage and filtration, to prevent masking of hydrometer scale readings and to avoid contamination of test samples.

7.4.2.3 Sample preparation for analysis after float and sink testing

Where possible, sample preparation of relative density fractions from a float and sink procedure shall be carried out according to ISO 13909-4 or ISO 18283. However, masses of relative density fractions from float and sink testing are often smaller than the minimum required for subdivision in accordance with ISO 13909-4 or ISO 18283, but subdivision prior to sample milling is still required to generate samples for caking tests, physical tests, or product composite testing.

Consequently, consideration shall be given to preserving as much sample at 4 mm top-size as possible to allow subsequent testing. Sample preparation instructions showing sub-division requirements at differing mass ranges shall be documented.

Table 7 provides an example of sample preparation instructions for 4 mm top-size fractions from a core project.

Table 7 — Example of sample preparation instructions

Mass range	Procedure	Prepare to −0,212 mm	Reserve
< 40 g	—	All	Nil
≥ 40 and < 200 g	RSD[a]	1/2	1/2
≥ 200 and < 400 g	RSD	1/4	3/4
≥ 400 and < 1 000 g	RSD	1/8	7/8

[a] RSD = Rotary Sample Division.

NOTE: The example sample preparation instructions take account of the limited mass that is specifically available for bore core samples. Samples for reserve are only required to be retained in the circumstance that subsequent analysis composites will be required at 4 mm top-size. Otherwise, errors attributable to sample preparation will be reduced if the entire RD fraction is prepared for general analysis.

7.4.2.4 Analysis and testing

Where chemical analysis and testing are required, these shall be carried out in accordance with the relevant ISO coal analysis standards. Preparation for some tests such as total moisture is found in ISO 13909-4 or ISO 18283. However, some individual standards, for example Hardgrove grindability index, stipulate some preparation to form the test sample. Examples of analysis carried out on the minus 212 µm test sample are as follows:

a) ISO 11722 to determine moisture in the analysis sample;

b) ISO 1171 to determine the level of ash;

c) ISO 1928 to determine the calorific value;

d) ISO 501 to determine the crucible swelling number (CSN).

7.4.2.5 Calculation and expression of results

Calculate the air-dried mass of each density fraction as a mass fraction of the initial mass. Report the mass fraction to the nearest 0,1 %, fractionally and cumulatively. The loss or gain in mass as a mass fraction of the air-dried starting mass should be reported to the nearest 0,1 %.

If ash or other properties have been determined on each density fraction, include these in a table with mass fraction percentage. Table 8 below shows an example for reporting results in tabular form.

NOTE For interpretation of results and calculation of washability curves, see Annex E.

Measurement uncertainty may be determined based on individual laboratory sample preparation and testing practices. A suitable method for determining measurement uncertainty is described in ISO/IEC Guide 98-3. It is difficult to assess the reproducibility of larger top-size material, due to the difficulty in procuring representative splits in the quantities that would be needed. ASTM D4371 provides information for one size fraction only (−6,3 mm + 1,18 mm), using starting masses of 4 400 g to 6 000 g and data are based on testing at two RDs only: 1,40 and 1,65 (see ASTM D4371).

Table 8 — Example for presenting float and sink results

Relative density fraction	**Sample mass** g	**Fractional**		**Cumulative**	
	Raw coal	**Mass (ad) fraction** %	**Ash (ad)** %	**Mass (ad)fraction** %	**Ash (ad)** %
F1,40	1 172,4	55,3	8,4	55,3	8,4
S1,40 - F1,50	498,22	23,5	16,7	78,8	10,9
S1,50 - F1,55	78,42	3,7	23,5	82,5	11,4
S1,55 - F1,60	63,63	3,0	26,7	85,5	12,0
S1,60 - F1,65	50,89	2,4	32,6	87,9	12,5
S1,65 - F1,70	12,71	0,6	34,4	88,5	12,7
S1,70 - F1,75	21,23	1,0	38,5	89,5	13,0
S1,75 - F1,80	19,07	0,9	41,0	90,4	13,3
S1,80 - F1,90	25,44	1,2	45,4	91,6	13,7
S1,90 - F2,00	29,69	1,4	51,8	93,0	14,3
S2,00 - F2,20	44,51	2,1	59,1	95,1	15,2
S2,20	103,90	4,9	75,5	100,0	18,2
Size fraction = −4,0 + 2,0 mm Starting mass (g) = 2 127,6 Total mass after float and sink = 2 120,1 g Sample loss = 0,4 %					

8 Test report

The test report shall contain the following information:

a) sample identification details;

b) the size fraction of the sample tested;

c) the sample mass tested (at commencement and the recombined total mass after testing);

d) the density order of the test if descending order has been used;

e) the yield for each density fraction, to the nearest 0,1 %;

f) the moisture basis of the mass (e.g. air-dried);

g) any other relevant data (e.g. ash %);

h) a reference to this document, i.e. ISO 7936:2022;

i) record in the test report all liquids (medium) used whether organic or inorganic for separation.

Annex A
(informative)

Drop shatter

A.1 General

As coal is processed through materials handling systems from mining to preparation plants, the size distribution will change as a result of breakage of coal particles. The breakage is considered to be random. This method describes a procedure to simulate the breakage and produce sample that has a similar size distribution to coal after it has passed through a materials handling system. In the case of raw coal reporting to a coal preparation plant, the size distribution may simulate the plant feed size distribution, and the resultant sample is able to be treated by the wet-pre-treatment method (see Annex B) to simulate further breakdown of coal during processing and provide the sample for float and sink testing. This method, in conjunction with the wet-pre-treatment procedure, aims to provide accurate size and washability information for coal preparation process design, and prediction of yields and quality of products made from the raw coal.

The method may also be used to estimate changes in size distribution of product coal caused by handling processes.

A.2 Sampling

A sample of the coal shall be taken in accordance with ISO 13909-2 or ISO 18283.

Examples of samples from different locations are as follows:

a) large diameter cores crushed to pass 50 mm;

b) uncrushed large diameter cores (hand broken to fit inside the sample container);

c) coal from a channel or strip sample;

d) run of mine sample;

e) underground mine and open cut samples can also include coal material and non-coal material (e.g. roof, floor and interburden).

A.3 Preparation

A test sample of the coal needs to be extracted from the gross sample by the sample reduction methods described in ISO 13909-4 or ISO 18283. The test sample may be crushed or broken to a pre-determined top-size before the drop shatter procedure in order to simulate a specific materials handling process. Alternatively, sample may be used at the received top-size if it can be contained in the sample holder and can pass through the opened bottom of the container.

A size distribution of the sample can be determined at this stage and should be referred to as the initial size distribution.

A.4 Apparatus — Manual

The apparatus is as follows:

a) A typical apparatus is shown in Figure A.1 and is composed of a steel box (drop shatter box) that is raised off ground level, with a solid base impact plate of 12 mm thick steel. The box is supported by steel legs of sufficient strength to withstand the effects of multiple impacts of 50 kg of coal falling from heights of up to three metres, inside the box;

b) A second box (sample container) is fitted inside the drop shatter box. This sample container is designed to hold 50 kg of coal sample and has a drop bottom arrangement that is activated when the sample container is raised to the specified height within the drop shatter box, dropping the coal sample onto the impact plate. The sample container is raised with the aid of a pulley system. The base of the container is opened by a trigger device when the container reaches the specified height;

c) Balance: able to determine at least 60 kg to the nearest 100 g;

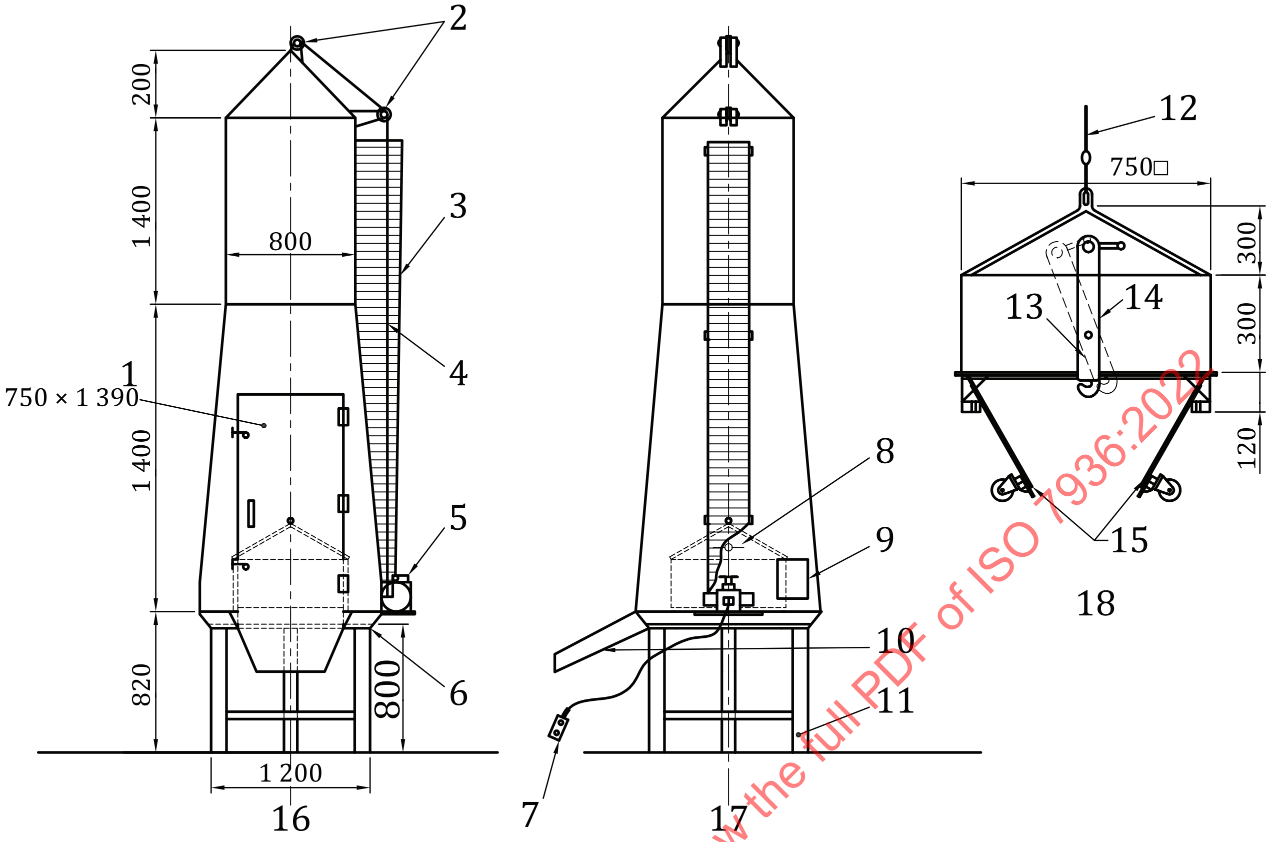
d) Sample containers required to collect sample from the drop shatter apparatus;

e) Broom: A suitable broom is required for sweeping coal fines from the base of the drop shatter apparatus.

A.5 Apparatus — Automatic

The drop shatter tower and box used in this test can be automated with examples shown in Figures A.1, A.2 and A.3.

Dimensions in millimetres



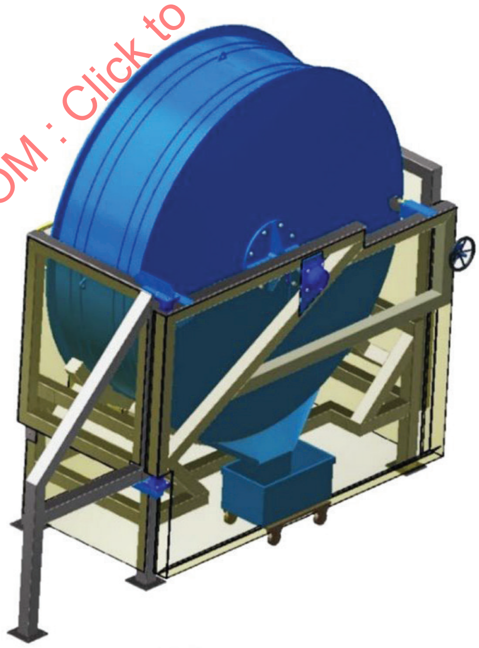
Key

1	door
2	rope pulleys
3	mesh guard
4	winch cable
5	adjustable cut-out light
6	12 mm thick plate
7	winch control
8	adjustable light striker
9	electrical controls
10	discharge shute
11	plate support legs
12	winch cable
13	unlocked position
14	locked position
15	drop doors in open positions
16	front view
17	side view
18	side view drop box

Figure A.1 — Drop shatter tower

NOTE Courtesy of Wallerby Mining Products.

Figure A.2 — Example of automatic drop shatter machine



NOTE Courtesy of SGS Australia Pty Ltd.

Figure A.3 — Example (CAD) of automatic drop shatter machine

A.6 Procedure

Each sample lot shall be processed separately, so that at the end of the procedure all lots of the coal sample have been dropped in the apparatus the same number of times.

The procedure shall be as follows:

a) Ensure that the inside of the drop shatter apparatus and sample container is clean and free of contamination.

b) Ensure that the trigger for the drop-bottom of the sample container is set at the required position, usually so that the bottom opens when it is 2 000 mm ± 5 mm above the impact plate.

c) Latch the drop-bottom of the sample container shut.

d) With the sample container resting on the impact plate (base of apparatus) place a sample lot inside the container to a uniform depth across the area of the box.

e) Close and securely latch and lock the door to prevent personal ingress to the apparatus.

f) Using the pulley system (electric or manual), raise the sample container (and coal sample) inside the box to the point where the trigger automatically unlatches the drop-bottom box and the coal sample discharges from the container and falls onto the impact plate. Mechanically isolate the sample container.

g) Wait two minutes after the sample has dropped to allow dust to settle inside the box and open the door to the apparatus.

h) Extract the coal sample, now resting on the impact plate. At this stage, the sample container is still suspended approximately 2 metres above the impact plate, allow unrestricted access to the coal on the impact plate. The coal is to be extracted by scraping it onto the discharge chute that directs it to a 60 l sample drum/container. Sweep fine coal and dust into the plastic sample container.

i) Repeat the procedure for all sample lots.

j) Repeat the process from items (a) to (j) until the sample has been dropped the specified number of times;

   The number of drops is usually dependent on the nature of the coal and the proposed coal handling system. The number of drops may be nominated prior to testing.

k) Composite the sample lots and determine the size distribution according to ISO 1953 using nominated screens.

l) Repeat the process from steps a) to j) until the sample has been dropped the specified number of times.

   The number of drops is usually dependent on the nature of the coal and the proposed coal handling system. The number of drops may be nominated prior to testing. When the drop shatter procedure has been deemed complete, a final analysis of the sample is performed and may include extra sizes not included previously.

A.7 Test report

A test report shall be prepared. The test report should include the following:

a) sample identification details;

b) total number of drop shatters performed;

c) cumulative percent (air-dry basis) passing each size for each drop shatter;

d) final size distribution after the final drop;

e) total starting sample mass (air-dry basis);

f) total final sample mass as air-dry basis;

g) percent gain (or loss) of sample mass—i.e. 100 × (initial mass–final mass) / initial mass;

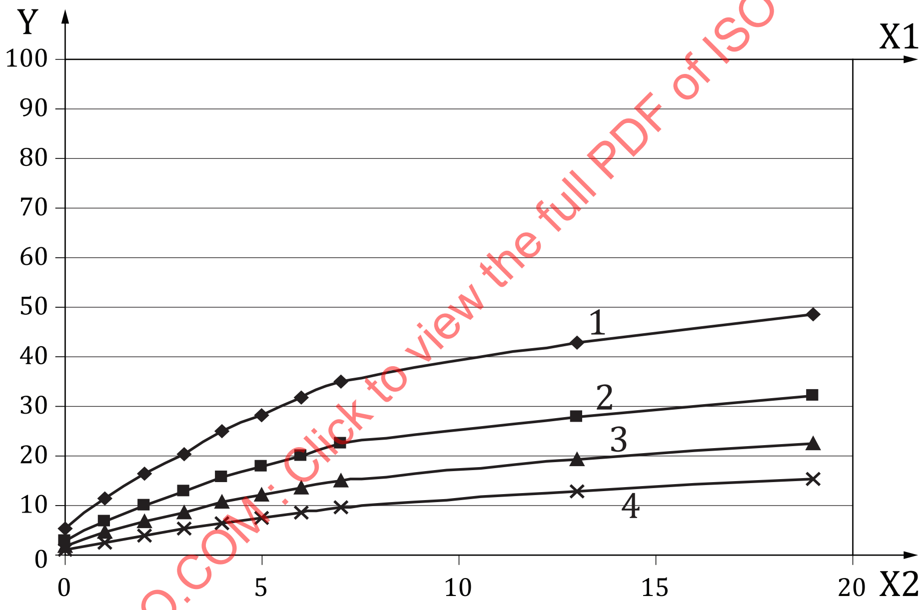
h) sample lot masses (air-dry basis);

i) plot a graph of sample drop number versus mass fraction passing for each size fraction;

j) plot a Rosin Rammler size distribution graph of initial size distribution (if available) or the size distribution after the first drop shatter, and the final size distribution on the same graph.

NOTE 1 An example of a test report is shown in A.8. Graphs drawn from the example data are given in Figures A.4 and A.5.

NOTE 2 For an end point result the initial size distribution is not required.



Key

X1	after drop shatter
X2	drop number
Y	cumulative % passing
1	−31,5 mm
2	−16,0 mm
3	−8,0 mm
4	−4,4 mm

Figure A.4 — Graph showing cumulative passing for each size fraction after each drop shatter

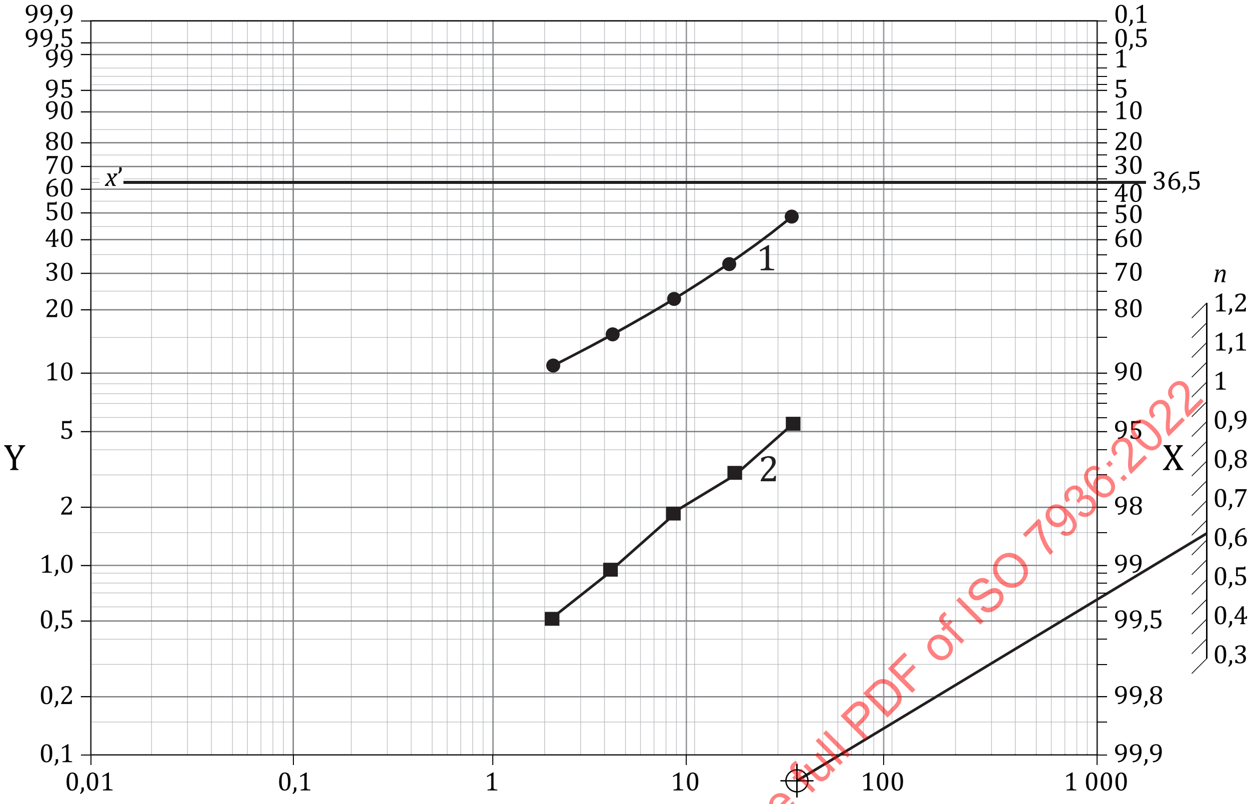


Figure A.5 — Rosin Rammler plot of size distribution before and after drop shatter

Key

X cumulative % retained

Y cumulative % passing

1 after drop shatter – after 0 drops

2 after drop shatter – after 19 drops

A.8 Example of a test report

	Analysis and Testing Report **Coal Mining Company** **Sample: 9771** **Hole: LDH 123**		**Report No.: XYZ 1234** **Date: 01/06/2005** **Page: 1**
Size analysis: After drop shatter — after 0 drops			
Size fraction **(mm square aperture)**	**Fractional mass** **%**	**Cumulative mass** **%** **(Retained)**	**Cumulative mass** **%** **(Passing)**
+31,5	94,6	94,6	—
−31,5 + 16,0	2,4	97,0	5,4
−16,0 + 8,0	1,2	98,2	3,0
−8,0 + 4,0	0,9	99,1	1,8
−4,0 + 2,0	0,4	99,5	0,9
−2,0	0,5	100,0	0,5
Size analysis: After drop shatter — after 1 drop			
Size fraction **(mm square aperture)**	**Fractional mass** **%**	**Cumulative mass** **%** **(Retained)**	**Cumulative mass** **%** **(Passing)**
+31,5	88,4	88,4	—
−31,5 +16,0	4,6	93,0	11,6
−16,0 +8,0	2,4	95,4	7,0
−8,0 +4,0	2,0	97,4	4,6
−4,0 +2,0	1,0	98,4	2,6
−2,0	1,6	100,0	1,6

Analysis and Testing Report			**Report No.: XYZ 1234** **Date: 01/06/2005** **Page: 2**
Size analysis: After drop shatter — after 2 drops			
Size fraction (mm square aperture)	**Fractional mass %**	**Cumulative mass % (Retained)**	**Cumulative mass % (Passing)**
+31,5	83,6	83,6	—
−31,5 +16,0	6,6	90,2	16,4
−16,0 +8,0	3,2	93,4	9,8
−8,0 +4,0	2,7	96,1	6,6
−4,0 +2,0	1,5	97,6	3,9
−2,0	2,4	100,0	2,4
Size analysis: After drop shatter — after 3 drops			
Size fraction (mm square aperture)	**Fractional mass %**	**Cumulative mass % (Retained)**	**Cumulative mass % (Passing)**
+31,5	79,6	79,6	—
−31,5 +16,0	7,5	87,1	20,4
−16,0 +8,0	4,2	91,3	12,9
−8,0 +4,0	3,5	94,8	8,7
−4,0 +2,0	1,9	96,7	5,2
−2,0	3,3	100,0	2,4

	Analysis and Testing Report **Coal Mining Company**		**Report No.: XYZ 1234** **Date: 01/06/2005** **Page: 3**
Size analysis: After drop shatter — after 4 drops			
Size fraction (mm square aperture)	**Fractional mass %**	**Cumulative mass % (Retained)**	**Cumulative mass % (Passing)**
+31,5	75,1	75,1	—
−31,5 +16,0	9,4	84,5	24,9
−16,0 +8,0	5,0	89,5	15,5
−8,0 +4,0	4,1	93,6	10,5
−4,0 +2,0	2,2	95,8	6,4
−2,0	4,2	100,0	4,2
Size analysis: After drop shatter — after 5 drops			
Size fraction (mm square aperture)	**Fractional mass %**	**Cumulative mass % (Retained)**	**Cumulative mass % (Passing)**
+31,5	71,8	71,8	—
−31,5 +16,0	10,2	82,0	28,2
−16,0 +8,0	5,9	87,9	18,0
−8,0 +4,0	4,6	92,5	12,1
−4,0 +2,0	2,6	95,1	7,5
−2,0	4,9	100,0	4,9

	Analysis and Testing Report Coal Mining Company		Report No.: XYZ 1234 Date: 01/06/2005 Page: 4
Size analysis: After drop shatter — after 6 drops			
Size fraction (mm square aperture)	**Fractional mass %**	**Cumulative mass % (Retained)**	**Cumulative mass % (Passing)**
+31,5	68,3	68,3	—
−31,5 +16,0	11,7	80,0	31,7
−16,0 +8,0	6,4	86,4	20,0
−8,0 +4,0	5,1	91,5	13,6
−4,0 +2,0	2,8	94,3	8,5
−2,0	5,7	100,0	5,7
Size analysis: After drop shatter — after 7 drops			
Size fraction (mm square aperture)	**Fractional mass %**	**Cumulative mass % (Retained)**	**Cumulative mass % (Passing)**
+31,5	64,9	64,9	—
−31,5 +16,0	12,6	77,5	35,1
−16,0 +8,0	7,4	84,9	22,5
−8,0 +4,0	5,5	90,4	15,1
−4,0 +2,0	3,2	93,6	9,6
−2,0	6,4	100,0	6,4

	Analysis and Testing Report **Coal Mining Company**		**Report No.: XYZ 1234** **Date: 01/06/2005** **Page: 5**
Size analysis: After drop shatter — after 13 drops			
Size fraction **(mm square aperture)**	**Fractional mass** **%**	**Cumulative mass** **%** **(Retained)**	**Cumulative mass** **%** **(Passing)**
+31,5	57,0	57,0	—
−31,5 +16,0	15,2	72,2	43,0
−16,0 +8,0	8,5	80,7	27,8
−8,0 +4,0	6,6	87,3	19,3
−4,0 +2,0	4,0	91,3	12,7
−2,0	8,7	100,0	8,7
Size analysis: After drop shatter — after 19 drops			
Size fraction **(mm square aperture)**	**Fractional mass** **%**	**Cumulative mass** **%** **(Retained)**	**Cumulative mass** **%** **(Passing)**
+31,5	51,5	51,5	—
−31,5 +16,0	16,4	67,9	48,5
−16,0 +8,0	9,5	77,4	32,1
−8,0 +4,0	7,3	84,7	22,6
−4,0 +2,0	4,4	89,1	15,3
−2,0	10,9	100,0	10,9

Annex B
(informative)

Wet tumbling

B.1 General

In the drum tumbler method, the sample of coal, water, and steel cubes are rotated for a predetermined time, to simulate conditions that would be experienced by coal being pumped through a dense medium cyclone/froth flotation process.

B.2 Sampling

A test sample of the coal needs to be extracted from the gross sample by the sample reduction methods described in ISO 13909-4.

B.3 Apparatus

The apparatus is as follows:

a) A tumbler steel drum, having a capacity of 200 l, capable of being turned end-over-end operating at a speed of 20 r/min. The drum should have provision for adding and removing the sample. Typical apparatus is shown in Figure B.1.

b) 18 steel cubes each having an edge length of 50 mm.

B.4 Procedure

The breakdown of shales in water is determined as follows:

a) Place 50 kg of the raw coal into the drum tumbler and add cubes and 150 l of water.

b) Secure the lid firmly and rotate for the time cycle required.

c) When tumbling is complete, screen the contents of the drum on a screen deck.

   NOTE A suitable screen deck is a 0,50 mm wedge-wire screen arranged as a lid of a clean, empty 200 l drum.

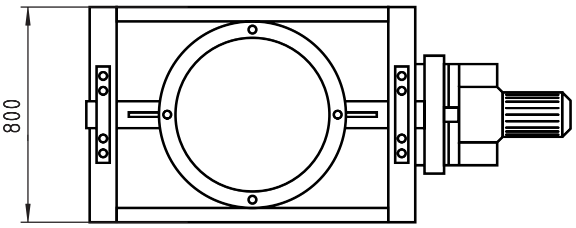
d) Air-dry the + 0,50 mm fraction and retain it for size and float and sink testing;

e) Recover the – 0,50 mm fraction, either by filtration or by decantation after the addition of a flocculant;

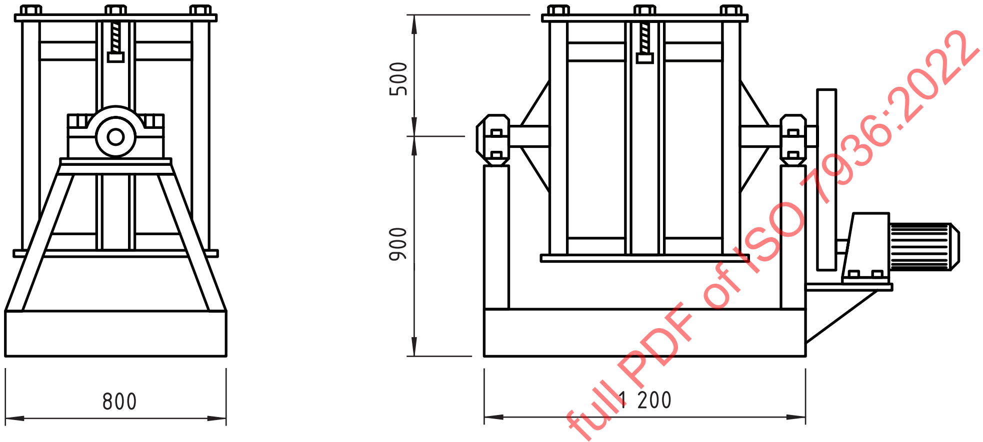
   The use of a flocculant is not recommended if froth flotation studies are to be carried out on the – 0,50 mm material, or if caking tests are required on the sample.

f) Air-dry the residual – 0,50 mm fraction and retain it for size analysis and other testing as required (e.g. float and sink testing, flotation, filtration and settling rates).

Dimensions in millimetres



a) Top view



b) Side view

c) Front view

Figure B.1 — Typical drum tumbler

Annex C
(informative)

Sample masses for float and sink testing

C.1 General

Particles in a coal mass, especially raw coal, may take many different forms. Figure C.1 shows the range in type of lump particles that may be present in raw coal. It is necessary to have at least 2 000 particles in any standard size fraction so that when the sample is separated into relative density fractions in a float and sink test, the resulting mass distribution can be considered to be representative of the material.

Figure C.1 — Example of raw coal particles

For size fractions where Table C.1 recommends a very large gross sample mass (e.g. 20 t for 125 mm top-size material), it is necessary to consider the practicality of whether that amount of material may be accurately and reliably handled by a well-resourced laboratory. In such circumstances, it is more appropriate to accept a smaller (more practical) sample mass and accept an associated lower level of precision in the result. Precision in the float and sink result will reduce as sample mass reduces below that required in Table C.1. In general terms, precision (interpreted here as the standard error of the mean), is proportional to $1/\sqrt{n}$, where n is the number of particles in the gross sample. For a given material, n is proportional to the sample mass.

Assume that Table C.1 provides a resulting precision of 0,a % in terms of mass fraction percent in any one RD fraction. If in the example above, the gross sample mass was reduced from 20 t to 1,2 t, the precision would increase (worsen) to $(1/\sqrt{1,2})/(1/\sqrt{20}) \times 0,a$ %, or $4,1 \times 0,a$ %. So, if the initial actual precision was 0,5 %, then the precision using the reduced sample mass would increase (worsen) to $4,1 \times 0,5 = 2,05$ %. In many cases, that worsening of precision may be deemed acceptable, but it needs to be recognized and the results qualified accordingly.

NOTE Where “a” is variable precision.

If the heterogeneity of a critical parameter (e.g. ash % ad) is known, the minimum sample mass may be calculated for a required level of precision.

C.2 General mass calculation examples

Invariably, the largest size fraction for float and sink testing determines what the gross sample mass needs to be. In this case if testing up to 125 mm top-size is required, then a gross sample mass of 21,5 t may be required to generate sufficient mass in the −125 + 31,5 mm size fraction. Table C.1 shows an example calculation.

It is particularly critical to note that the minimum mass for float and sink of the +31,5 mm fraction in this example is 6 450 kg, not 2 150 kg + 300 kg = 2 450 kg.

Table C.1 — Example of mass calculation for −125 + 31,5 mm float and sink

Size fraction mm	**Fractional mass** %	**Mass fraction** %	**Minimum fractional mass per Table 1** kg	**Minimum gross sample mass prior to sizing and float and sink testing** kg	**Resulting mass in fraction subjected to float and sink testing** kg
	Raw coal (A)	**Factored to relevant size fraction**	**(B)**	**(C) = (B)*100/(A)**	**(E) =(D)*(A)/100**
−125 + 63	10	33,3	2 150	21 500	2 150
−63 + 31,5	20	66,7	300	1 500	4 300
−31,5 + 16	14				
−16 + 8	16				
−8 + 4	12				
−4 + 2	8				
−2	20				
RESULTING MINIMUM GROSS SAMPLE MASS (D) = Maximum of Column (C)				21 500	
RESULTING MINIMUM Float and Sink SAMPLE MASS = Sum of Column (E)					6 450

Another example of this calculation is shown in Table C.2 for − 50 + 4 mm material (e.g. DMC process samples). It is particularly critical to note that the minimum mass for float and sink of the +4 mm fraction in this example is 1 380 kg, not 230 kg + 40 kg + 5,2 kg + 2 kg = 277 kg.

Table C.2 — Example of mass calculation for −50 mm + 4 mm float and sink

Size fraction mm	Fractional mass %	Mass fraction %	Minimum fractional mass per Table 1 kg	Minimum gross sample mass prior to sizing and float and sink testing kg	Resulting mass in fraction subjected to float and sink testing kg
	Raw coal (A)	**Factored to relevant size fraction**	**(B)**	**(C) = (B)*100/(A)**	**(E) = (A)/100 *(D)**
−50 + 31,5	10	16,7	230	2 300	230
−31,5 + 16	18	30	40	222	414
−16 + 8	18	30	5,2	29	414
−8 + 4	14	23,3	2	14	322
−4 + 2	12				
−2	10				
RESULTING MINIMUM GROSS SAMPLE MASS (D) = Maximum of Column (C)				2 300	
RESULTING MINIMUM Float and Sink SAMPLE MASS = Sum of Column (E)					1 380

A third example for calculating the appropriate mass for float and sink testing is shown in Table C.3 for – 31,5 mm + 2 mm material [e.g. Dense media cyclone (DMC) process samples]. This example shows that the sample mass subjected to float and sink may be substantially reduced if the larger particles are removed from the sample to be tested, and each size fraction is tested separately.

Table C.3 — Example of mass calculation for −31,5 + 2 mm float and sink

Size fraction mm	Fractional mass %	Mass fraction %	Minimum fractional mass per Table 1 kg	Minimum gross sample mass prior to sizing and float and sink testing kg	Resulting mass in fraction subjected to float and sink testing kg
	Raw Coal (A)	**Factored to relevant size fraction**	**(B)**	**(C) = (B)*100/(A)**	**(E) = (A)/100 *(D)**
−50 + 31,5	10				
−31,5 + 16	18	29,0	40	222	40
−16 + 8	18	29,0	5,2	29	40
−8 + 4	14	22,6	2	14	31
−4 + 2	12	19,4	2	17	27
−2	28				
RESULTING MINIMUM GROSS SAMPLE MASS (D) = Maximum of Column (C)				222	
RESULTING MINIMUM Float and Sink SAMPLE MASS = Sum of Column (E)					138

C.3 Impact of lower masses

Figure C.2 shows an example where the – 50 mm + 16 mm fraction, sourced from a large diameter (LD) core, has been subjected to float and sink testing. In this case, the starting mass was around 16 kg. It is clearly evident that the float and sink result needs to be heavily qualified in this circumstance. One middling relative density fraction shows no particles at all, while 7 of the 12 relative density fractions have only 1 to 3 particles.

For a small starting mass of this nature, any misplacement of individual particles during the testing process results in a significant change to the float and sink mass distribution.

a) Floats 1,30

b) Sinks 1,30 Floats 1,35

c) Sinks 1,35 Floats 1,40

d) Sinks 1,40 Floats 1,45

e) Sinks 1,45 Floats 1,50

f) Sinks 1,50 Floats 1,55

g) No sample, Sinks 1,55 Floats 1,60

h) Sinks 1,60 Floats 1,70

i) Sinks 1,70 Floats 1,80

j) Sinks 1,80 Floats 2,00

k) Sinks 2,00 Floats 2,20

l) Sinks 2,20

Figure C.2 — Example, PS – 50 mm + 16 mm float and sink distribution from a core sample

C.4 Core samples

Slim core samples (63 mm diameter) will almost always need to be crushed to -8 mm in order to satisfy the minimum number of particles requirement for float and sink testing. As a guide, if a topsize of

11,2 mm is chosen, a sample mass of at least 5 kg will be required which is approximately a 1 m section at a relative density of 1,60.

For larger diameter core samples, the largest particle size that can be legitimately tested under this Standard will vary depending on the core diameter and length of intersection. Considering a typical section, say 2 m deep for a 100 mm diameter core, that will yield approximately 25 kg of raw coal assuming 1,6 relative density, and that will only allow a testing top-size of the order 16 mm to 20 mm if this standard is to be conformed with.

For a 200 mm diameter core (2 m intersection), any float and sink work undertaken on particles larger than 25 mm is likely to be misleading. It is therefore necessary to qualify core float and sink results where the masses employed clearly do not conform with the requirements of this Standard. Core samples present specific challenges for float and sink testing.

The main reasons for this are:

a) Washability distribution will vary with particle size distribution, so this will impact the results depending on how the particle size distribution of the sample is generated.

b) Sample masses from cores are small, even from large diameter cores.

c) The results from float and sink testing may be used in statutory interpretation of coal reserves (e.g. Australasian Code for Reporting of Exploration Results, Mineral Resources and Ore Reserves (JORC), and so any known shortcomings of the testing need to be considered in that context.

While not recommended, in some circumstances resource samples are required to be tested “by zero”. The term “by zero” refers to the size fraction (e.g. 11,2 mm × 0 mm) and means that the entire range of size fractions is to be separated in a float and sink test. Samples should be separated inside a container with no basket. The coarser float material should be removed using a fine mesh scoop, the top 25 % of the solution layer should be decanted and filtered to recover the fine floats. This is added to the coarse material to form a combined floats. The remaining liquid should then be filtered to recover the sink material.

Alternately, the ultrafine size fraction (e.g. −0,038 mm) may be removed from the rest of the sample by sizing, and then separated within a separating flask using the fine coal float and sink test procedure. The air drying of samples should be conducted as outlined in 6.4.3.2.

Annex D
(normative)

Validation of data

D.1 General

Float and sink test results shall be validated by undertaking some basic assessments of the raw data.

D.2 Before commencing the float and sink test

Ensure the sample is fully air-dried; record the total starting sample mass immediately prior to testing; and record the air temperature and humidity. This is most important in more humid regions where sample masses may drift up and down with humidity.

All observations shall be recorded.

D.3 Immediately following the float and sink test and before sample preparation

After the fractional samples have been air-dried and their mass determined, check the recombined mass against the starting mass and re-record the humidity. Normally, the expectation is that the recombined mass will be slightly lower than the starting mass, because of small remnants of sample that remain dispersed in the solutions. However, sometimes the recombined mass may be higher than the starting mass. If that occurs:

a) the result, if a minor discrepancy, may be explained by increased humidity; or

b) the fractional samples may require further air drying to ensure that negligible solution remains absorbed in or adsorbed on the samples; or

c) an inadvertent error was made regarding recording of the starting mass; or

d) there is sample contamination.

If the recombined mass is significantly lower than the starting mass:

1) the result, if a minor discrepancy, may be explained by decreased humidity; or

2) the starting sample may not have been fully air-dried prior to commencement; or

3) an error was made regarding recording of the starting mass; or

4) elements of the sample may have broken down and dispersed in the float sink media. This dispersion should however be visually obvious and recorded at the time of conducting the test.

Provided an assessment of starting and recombined mass is undertaken prior to sample preparation, it provides the laboratory with the opportunity to re-run aspects of the test (at the correct particle size) if necessary.

Further to mass balance checks, plot the relative density curve, refer to Annex D, and check for any abnormalities.