



Coal preparation

Part 1.1: Higher rank coal—Float and sink testing



AS 4156.1.1:2018

This Australian Standard® was prepared by MN-001, Coal and Coke. It was approved on behalf of the Council of Standards Australia on 31 October 2018.

This Standard was published on 23 November 2018.

The following are represented on Committee MN-001:

- Australian Coal Preparation Society
- Australian Energy Council
- Coalfield Geology Council of NSW
- CSIRO
- Minerals Council of Australia
- National Association of Testing Authorities Australia
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This Standard was issued in draft form for comment as DR AS 4156.1.1:2018.

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ISBN 978 1 76072 256 2



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Part 1.1: Higher rank coal—Float and sink testing

Originated as AS K205—1970.
Previous edition AS 4156.1—1994.
Revised and redesignated as AS 4156.1.1:2018.

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Preface

This Standard was prepared by the Standards Australia Committee MN-001, Coal and Coke, to supersede AS 4156.1, *Coal preparation, Part 1: Higher rank coal — Float and sink testing*.

The objective of this Standard is to describe the approach for determining the float and sink characteristics of higher rank coal and provide methods suitable for coarse coal (indicative size range 125 mm to 0.5 mm) and fine coal (indicative size range less than 0.5 mm).

Float and sink testing is the standard laboratory method for determining, with respect to relative density, the washability characteristics of a wide range of coal samples, from bore core to mine production and coal preparation plant products. Data are derived for a number of different purposes and forms the basis on which to —

- (a) establish limits theoretically attainable by density separations, often used as a reference base and an initial assessment of resource potential during exploration of coal deposits;
- (b) predict and compare results of simulated plant operations for various types and combinations of processing equipment, facilitating selection of optimum process arrangement;
- (c) design commercial coal preparation plant (in conjunction with appropriate non-density-based washability data); and
- (d) examine products from operating plant for routine control purposes, or to assess separation performance critically against established criteria.

In commercial practice, coal cleaning is conducted largely by processes that take advantage of a relationship between particle ash percentage and particle relative density. However, coal particles contain mineral matter not ash (ash being the residue remaining after combustion of coal), and the general relationship between ash percentage and particle relative density is therefore only an approximation.

A list of all parts in the AS 4156 series can be found in the Standards Australia online catalogue.

The terms “normative” and “informative” are used in Standards to define the application of the appendices to which they apply. A “normative” appendix is an integral part of a Standard, whereas an “informative” appendix is only for information and guidance.

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Australian Standard®

Coal preparation

Part 1.1: Higher rank coal—Float and sink testing

Section 1 Scope and general

1.1 Scope

This Standard sets out the requirements for determining the float and sink characteristics of higher rank coal (refer to AS 2096) and sets out procedures for deriving test samples and methods for presenting the washability data.

NOTE This Standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Float and sink testing is the division of a sample into relative density fractions within defined ranges. The proportions of the fractions are expressed as mass percentages of the total sample, commonly with an indication of the ash percentage (and other characteristics, if required) of each fraction.

1.2 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.

NOTE Documents for informative purposes are listed in the Bibliography.

AS 1038, *Coal and coke — Analysis and testing (all parts)*

AS 2096, *Classification and coding systems for Australian coals*

AS 2418, *Coal and coke — Glossary of terms*

AS 2519, *Guide to the technical evaluation of higher rank coal deposits*

AS 2617, *Sampling from coal seams*

AS 4156.8, *Coal preparation, Part 8: Sample pre-treatment — Drop-shatter*

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

ISO 13909, *Hard coal and coke — Mechanical sampling (all parts)*

1.3 Terms and definitions

For the purposes of this Standard the definition below and those given in AS 2418 apply.

1.3.1

measurement uncertainty

variable associated with the result of a measurement that characterizes the dispersion of the values that could be reasonably attributed to the measurand

Section 2 Sampling

2.1 Sample mass

Samples for float and sink testing shall be obtained and prepared according to the requirements of relevant Standards (AS 2519, AS 2617, ISO 13909 series) and relevant sections of this Standard. The sampling requirements stated in this Standard shall take precedence.

For samples undergoing float and sink testing, the mass of the size fraction on which float and sink testing is being conducted shall be as listed in [Table 1](#).

NOTE [Appendix B](#) provides further information and example calculations in order to determine the bulk sample mass necessary to achieve masses in individual size fractions for float and sink testing (as shown in [Table 1](#)).

As a general rule, the number of discrete particles in any size fraction shall be not less than 2000.

When coals exhibit low yields in any fraction, to obtain sufficient sample for analytical requirements, the sample mass shall not be less than 20 g and not less than 10 particles in each relative density fraction.

If these requirements cannot be met, this fact shall be noted in the test report.

NOTE 1 This is particularly relevant to float and sink testing of bore core samples.

NOTE 2 Samples with different particle sizes will contain particles with different proportions of mineral matters and organic matters, which will produce 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.

NOTE 3 It is assumed that square-mesh particle sizes are used; if wedge-wire or round-hole sizes are used, this fact should be reported.

NOTE 4 The mass of the sample to be taken for any given purpose will determine to a large extent the accuracy of the test. In turn, the sample mass will be governed by the purpose for which the test is being carried out.

NOTE 5 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 weigh up to 20 t, and for other sizes the amounts should be reduced according to the decrease in nominal topline. The importance of sufficient sample mass and a method for the determination of the required mass of a bulk sample is given in [Appendix B](#).

Table 1 — Sample mass^a

Size fraction mm	Sample mass ^b kg		
	Raw coal	Clean coal	Reject
– 125 + 63	2150	1810	2680
– 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

^a For control samples, 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: Raw Coal 1.60, Clean Coal 1.35, Reject 2.00 (see [Appendix B](#) for calculation examples to determine bulk sample masses).

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.

2.2 Coal preparation plant samples

Coal preparation plant samples shall be collected in accordance with the ISO 13909 series of Standards. The minimum quantities for each sample shall be in accordance with [Table 1](#).

NOTE The masses in [Table 1](#) generate very large bulk samples for 50 mm top size coal (typically more than 1200 kg). While processing these large samples may be impractical, a method to reduce the bulk sample mass is shown in [Appendix B](#).

In the sampling of slurry, the quantity of the (dried) solids shall conform with [Table 1](#).

2.3 Borecore samples

For bore core samples, guidance is given in [Appendix B](#).

Section 3 Sample pre-treatment

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

The pre-treatment process shall 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. Refer to AS 4156.8.
- (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.

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

- (i) *Jaw crusher* — The sample is choke-fed to the crusher with the aim of producing the nominated size.
- (ii) *Hand knapping* — The sample is broken using hand-held implements. Done carefully, this procedure can yield the least amount of fine material.

Hammermill type crushers shall not be used for size reduction, due to the excess amount of fine material produced.

- (c) *Dry tumbling* — The sample is tumbled end over end in a drum under specified conditions.
- (d) *Wet tumbling* — The sample is tumbled end over end after the addition of water and under specified conditions.

NOTE See [Appendix E](#) for more information.

Section 4 Sample preparation for analysis after float and sink testing

Where possible, sample preparation of relative density fractions from a float sink procedure shall be carried out according to ISO 13909-4. 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, 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 4mm top size as possible to allow subsequent testing. Sample preparation instructions showing sub-division requirements at differing mass ranges shall be documented.

[Table 2](#) provides an example of sample preparation instructions for – 4 mm top size fractions from a bore core project:

Table 2 — 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 < 1000 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 topsize. Otherwise, errors attributable to sample preparation will be reduced if the entire RD fraction is prepared for general analysis.			

Section 5 Analysis and testing

Where chemical analysis and testing are required, these shall be carried out in accordance with the various parts of the AS 1038 series.

Section 6 Separation media

6.1 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.

6.2 Organic solutions

6.2.1 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, AS WELL AS ENSURING COMPLIANCE TO 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, sym-tetrabromoethane 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.

NOTE 1 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 sym-tetrabromoethane, shall be rinsed with a more rapidly evaporating compatible organic liquid, such as white spirit.

NOTE 2 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
Sym-tetrabromoethane (acetylene tetrabromide)	2.96	239.0	12.00	0.01	No
Tetrachloroethylene (perchloroethylene)	1.61	121	0.89	1.83	No
NOTE Refer to vendor Safety Data Sheet for current information regarding physical properties.					

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

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

6.3 Inorganic solutions

6.3.1 General

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

6.3.2 Formate solutions

6.3.2.1 General

The float sink procedure for water soluble salt solutions shall be the same as that outlined in [Section 9](#) of this Standard. 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 vendor Safety Data Sheet for current information regarding physical properties.

6.3.2.2 Limitations on accuracy

The use of inorganic solutions 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 may limit the use of caesium formate:

- Caesium formate may contaminate coal particles even after rinsing. This could affect subsequent properties to be tested, in particular ash.
- Some coal or mineral matter particles may disintegrate in a water-based solution.
- Density stability — Formate solutions with densities above RD1.70 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.

6.3.2.3 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.

6.3.2.4 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.

6.3.2.5 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.

6.4 Aqueous suspensions

6.4.1 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.

An example of an aqueous suspension would be zirconium dioxide.

6.4.2 Zirconium dioxide

6.4.2.1 General

The float sink procedure for aqueous suspensions shall be the same as that outlined in [Section 9](#) of this Standard. 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](#).

6.4.2.2 Limitations on accuracy

The use of aqueous suspensions such as zirconium dioxide 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 may limit the use of zirconium dioxide:

- (a) Zirconium dioxide may contaminate particles even after rinsing. This could affect subsequent properties to be tested, in particular ash.
- (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 °C at 100 kPa	Viscosity at 20 °C mPa.s	Vapour pressure at 20 °C kPa	Flammable
Zirconium dioxide	1.30 – 2.20	—	11.2	—	No
NOTE Refer to vendor Safety Data Sheet for current information regarding physical properties.					

6.4.2.3 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:

- Prepare a water solution containing the stabilization agent.
- 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.
- Pass the suspension prepared in Step (b) twice through a colloid mill.
- 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.

6.4.2.4 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 may fail to drain completely.

6.4.2.5 Recovery of media

Dilute suspension media shall be recovered by concentration using one of the options below:

- Recovering zirconium dioxide particles from a dilute suspension by reducing its pH to 6 or less to allow fast settling; or
- Using a ceramic membrane to re-concentrate diluted suspensions; or
- Leaving dilute suspensions in a container for a long period of time to allow slow settling.