SLOVENSKI STANDARD
SIST ISO 9411-1:1998

01-februar-1998

Trdna fosilna goriva - Mehanično vzorčenje pri zveznem transportiranju - 1. del: Premog

Solid mineral fuels -- Mechanical sampling from moving streams -- Part 1: Coal

Combustibles minéraux solides -- Échantillonnage mécanique sur minéraux en mouvement -- Partie 1: Charbon

Ta slovenski standard je istoveten z: ISO 9411-1:1994

ICS:

75.160.10 Trda goriva Solid fuels

SIST ISO 9411-1:1998 **en**

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INTERNATIONAL STANDARD

ISO 9411-1

First edition
1994-11-01

Solid mineral fuels — Mechanical sampling from moving streams —

Part 1: Coal

Combustibles minéraux solides — Échantillonnage mécanique sur minéraux en mouvement —
Partie 1: Charbon

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Contents

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9411-1 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

ISO 9411 consists of the following parts, under the general title *Solid mineral fuels — Mechanical sampling from moving streams*:

— *Part 1: Coal*

— *Part 2: Coke*

Annexes A, B, C and D form an integral part of this part of ISO 9411. Annexes E and F are for information only.

Introduction

Mechanical sampling from moving streams of coal has increasingly replaced manual methods of coal sampling and, consequently, the recommended practices for mechanical sampling provided by International Standards have come under review.

Solid mineral fuels — Mechanical sampling from moving streams —

Part 1:
Coal

1 Scope

This part of ISO 9411 provides recommended practices for the mechanical sampling of solid mineral fuels from moving streams. It has been developed primarily to give guidance on falling-stream samplers. This part of ISO 9411 is applicable to the mechanical sampling and preparation of coal samples for the determination of total moisture, general analysis, including physical and chemical tests, and size analysis. ISO 9411-2 deals with the methods of sampling of coke and preparation of coke samples in a similar way.

NOTE 1 The methods of analysing or testing the test samples of coal obtained are described in other International Standards.

In this part of ISO 9411 the basic requirements, together with typical examples of applications, are described for guidance in the design, installation and operation of systems for mechanical sampling and mechanical preparation of samples.

Notes on the operation of mechanical sampling systems are given in annex A.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9411. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9411 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 3310-1:1990, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

3 Definitions

For the purposes of this part of ISO 9411, the following definitions apply.

3.1 accuracy: The closeness of agreement between an observation and the "true" value.

NOTE 2 The accuracy of a result should not be confused with its precision.

3.2 air drying: The process of partial drying of the sample to bring its moisture content near to equilibrium with the atmosphere in the area in which further reduction and division of the sample are to take place.

3.3 bias: A systematic error which leads to results which are persistently higher or persistently lower than the "true" value.

3.4 coefficient of variation: The standard deviation, s, expressed as a percentage of the absolute value of the arithmetic mean, $|\bar{x}|$.

NOTE 3 This term is usually designated as v.

$$v = \frac{s}{|\bar{x}|} \times 100$$

3.5 common sample: A sample collected for more than one intended use.

3.6 constant-mass division: The method of increment or sample division in which the portions retained from individual increments, partial samples or gross samples are of uniform mass.

3.7 correlation coefficient: A measure of the degree of correlation between the members of paired sets.

3.8 cut: An increment taken by a sample divider.

3.9 cutter: A mechanical device which takes a single increment of coal.

3.10 divided increment: The part obtained from the division of the increment in order to decrease its mass.

NOTE 4 Such division may be done with or without prior size reduction.

3.11 duplicate sampling: A particular case of replicate sampling with only two replicate samples.

3.12 error: The difference between the observation and the "true" value, which can be designated systematic (bias) or random.

NOTE 5 The procedures of sampling, sample preparation and analysis are not perfect and the observations will be dispersed about the "true" values.

3.13 fixed-rate division: The method of increment or sample division in which the portions retained from individual increments, partial samples or gross samples have a mass proportional to the mass of the increment, partial sample or gross sample.

3.14 general analysis test sample: A sample, crushed to pass a sieve of nominal size of openings 212 µm complying with ISO 3310-1, used for the determination of most physical and chemical characteristics of coal.

3.15 gross sample: The quantity of coal consisting of all the increments or partial samples taken from a sampling unit, either in the condition as taken or after the increments have been individually reduced and/or divided.

3.16 increment: A portion of coal collected in a single operation of the sampling instrument.

3.17 laboratory sample: A sample prepared from the gross or partial sample as delivered to the laboratory, and from which further samples are prepared for test purposes.

3.18 lot: A discrete quantity of coal for which the overall quality to a particular precision needs to be determined.

3.19 manual sampling: The collection of increments by human effort.

3.20 mass-basis sampling: The taking of increments in uniform mass intervals throughout the sampling unit.

NOTE 6 Each increment or divided increment constituting the partial sample or the gross sample should be of almost uniform mass.

3.21 mechanical sampling: The collection of increments by mechanical means.

3.22 nominal top size: The smallest sieve in the range included in the R 20 series (see ISO 565, square hole) on which not more than 5 % of the sample is retained.

3.23 off-line sample preparation: Sample preparation performed manually or by mechanical equipment not integral with the mechanical sampling system.

3.24 on-line sample preparation: Sample preparation by mechanical equipment integral with the sampling system.

3.25 outlier: A result which appears to be in disagreement with others from the same coal and which arouses suspicion that there has been a mistake in the sampling, sample preparation or analysis.

3.26 partial sample: A sample representative of a part of the whole sampling unit, constituted in order to prepare laboratory samples or test samples.

NOTE 7 Partial samples may be obtained by combining all increments from a sampling unit into two or more sets, each set being composed of consecutive increments, the number of which need not be the same in all sets (see figure 1).

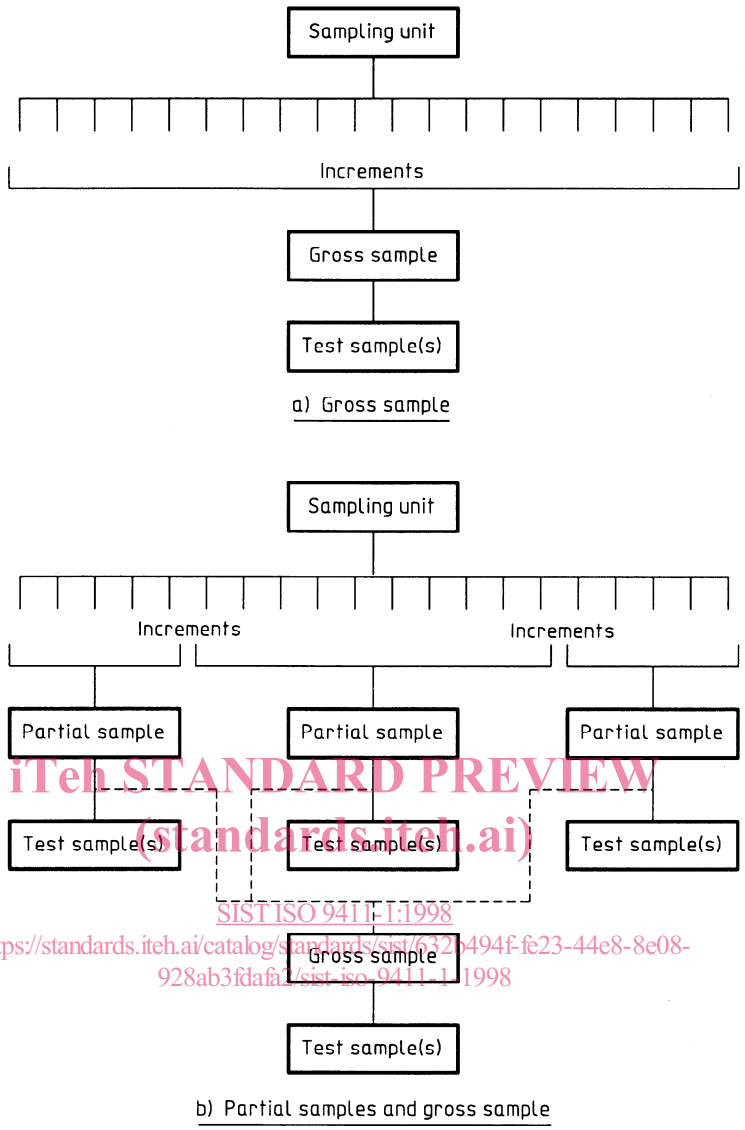

Figure 1 — Examples of the constitution of gross sample and/or partial samples

3.27 precision: A measure of the extent to which observations within a set agree with each other.

NOTE 8 A determination may be made with great precision and the standard deviation of a number of determinations on the same sampling unit may therefore be low, but the results will be accurate only if they are free from bias.

3.28 primary increment: The increment taken at the first stage of sampling, prior to any sample division and/or sample reduction.

3.29 random error: Error that has an equal probability of being positive or negative.

NOTE 9 The mean of the random errors resulting from a series of observations tends towards zero as the number of observations increases.

3.30 range: The difference between the greatest and least values of a number of observations.

3.31 reference increment mass: The minimum average mass of an increment which should be collected when taking primary or divided increments from a coal stream with a mechanical sampler.

3.32 relevant bias: Bias that is of practical importance, whether economic, scientific, legal or social.

3.33 replicate sampling: The taking from the sampling unit of increments which are combined in rotation into different containers to give two or more samples of approximately equal mass, each being representative of the whole sampling unit.

3.34 sample division: The process in sample preparation whereby the sample is divided into separate portions, one or more of which is retained.

3.35 sample preparation: The process of bringing samples to the condition required for analysis or testing.

NOTE 10 Sample preparation covers mixing, sample division, particle size reduction and sometimes air drying of the sample, and may be performed in several stages.

3.36 sample reduction: The process in sample preparation whereby the particle size of the sample is reduced by crushing or grinding.

3.37 sampling unit: A quantity of coal, the sampling of which results in one gross sample.

NOTE 11 There may be one or more sampling units per lot.

3.38 size analysis sample: A sample taken specifically for particle size analysis.

3.39 standard deviation: The positive square root of the variance.

NOTE 12 This term is usually designated as s.

3.40 stratified random sampling: The taking of an increment at random within the mass interval or time interval determined for mass-basis sampling or time-basis sampling respectively.

3.41 test sample: A sample which is prepared to meet the requirements of a specific test.

3.42 time-basis sampling: The taking of increments in uniform time intervals throughout the sampling unit.

NOTE 13 Each increment or divided increment constituting the partial sample or the gross sample should be of a mass proportional to the flow rate of the coal stream at the time of taking the increment.

3.43 total moisture sample: A sample taken specifically for the determination of total moisture.

3.44 variance: The mean square of deviations from the mean value of a set of observations.

NOTE 14 This term is usually designated as V.

4 Establishing a sampling scheme

4.1 General procedure

The following shall be the general procedure for establishing a sampling scheme.

a) Define the quality parameters to be determined and the types of samples required.

b) Define the lot.

c) Define the precision required.

d) Determine the variability of the coal (see 4.5.2) and establish the number of sampling units u (see 4.5.4) required to attain the desired precision and the minimum number of increments n (see 4.5.5). Methods for determining variability are given in annex B.

e) Decide whether to use time-basis or mass-basis sampling (see clause 5) and define the sampling intervals in minutes or tonnes respectively.

f) Ascertain the nominal top size of coal for the purpose of determining the reference increment mass.

g) Determine the method of combining the increments into gross samples or partial samples (see 6.2) and the method of sample preparation.

h) Determine the minimum mass of increments at each stage of division (see 6.5.2).

4.2 Types of samples

It is permitted to collect either separate samples or a common sample for the determination of total moisture, for size analysis and for general analysis and other tests.

4.3 Division of lots

A lot may be a single sampling unit or a series of sampling units, for example, coal despatched or delivered over a period of time, a ship load, a train load, a wagon load, or coal produced during a certain period, e.g. a shift.

A sampling unit may be represented by a gross sample or by two or more partial samples.

A division of a lot into a number of sampling units or the representation of a sampling unit by a number of

partial samples may be necessary, in order to improve the precision of the results.

For lots sampled over long periods, it may be convenient to divide the lot into a series of sampling units, obtaining a gross sample for each sampling unit.

If the quality is to be determined for parts of a lot in addition to that of the lot as a whole, the lot shall be sampled as two or more part-lots and each part-lot shall be treated as a lot in its own right. The resulting samples shall conform to the requirements for gross samples. The quality for the lot as a whole shall be calculated on the weighted average of the quality of the part-lots.

4.4 Basis of sampling

Sampling may be carried out on either a time basis or a mass basis. In time-basis sampling, the sampling interval is defined in minutes and the increment mass shall be proportional to the flow rate at the time of taking the increment. In mass-basis sampling, the sampling interval is defined in tonnes and the mass of increments added to the gross or partial sample shall be uniform.

4.5 Precision of sampling

4.5.1 General

In all methods of sampling, sample preparation and analysis, errors are incurred, and the experimental results obtained from such methods for any given parameter will deviate from the true value of that parameter. As the true value cannot be known exactly, it is not possible to assess the accuracy of the experimental results, i.e. the closeness with which they agree with the true value. However, it is possible to make an estimate of the precision of the experimental results, i.e. the closeness with which the results of a series of experiments made on the same coal agree among themselves.

It is possible to design a sampling scheme by which, in principle, an arbitrary level of precision can be achieved. However, limits will be imposed on the precision that should be aimed for because of practical considerations.

NOTE 15 The required overall precision on a lot is agreed upon by the parties concerned.

The theory of the estimation of precision is given in annex B. The following equation is derived from equation (B.5):

$$P_L = \pm 2 \times \left(\frac{\frac{V_I}{n} + V_{PT}}{u} \right) \quad \ldots (1)$$

where

P_L is the overall precision of sampling, sample preparation and testing for the lot at 95 % confidence level expressed as percentage absolute;

V_I is the primary increment variance;

V_{PT} is the preparation and testing variance;

n is the number of increments to be taken from each sampling unit;

u is the number of sampling units in the lot.

If the quality of a coal of a type not previously sampled is required, then in order to devise a sampling scheme assumptions will have to be made about the variability (see 4.5.2). The precision actually achieved for a particular lot by the scheme devised can be measured by the procedures given in annex B.

If the same type of coal is sampled regularly, sampling schemes can be laid down using data derived from previous sampling. The procedures given in 4.5.5 can be used to devise the optimum scheme, thus keeping the sampling costs to a minimum.

4.5.2 Primary increment variance

The primary increment variance, V_I, depends upon the type and nominal top size of coal, the degree of pretreatment and mixing, the absolute value of the parameter to be determined and the mass of increment taken.

For many coals, the variability for ash is higher than that for moisture and hence, for the same precision, the number of increments for ash will be adequate for moisture. If, however, a higher precision is required for moisture, the relevant primary increment variance shall be applied for each sample.

If at all possible, the primary increment variance, V_I, should be determined on the coal to be sampled using one of the methods described in annex B. If this cannot be done, V_I should be determined for a similar coal from a similar sampling system. If neither of these procedures is possible, assume an initial value of 20 for V_I and check this, after the sampling has been carried out, using one of the methods described in annex B.

NOTE 16 A method using the variogram technique is described in annex F.

4.5.3 Variance of preparation and testing

If at all possible, the variance of preparation and testing, V_{PT}, should be determined on the coal to be sampled using one of the methods described in annex C. If this cannot be done, V_{PT} should be determined for a similar coal from a similar sampling system. If neither of these procedures is possible, assume an initial value of 0,2 for V_{PT} and check this, after the preparation and testing has been carried out, using one of the methods described in annex C.

4.5.4 Number of sampling units

The number of increments taken from a lot in order to attain a certain precision is a function of the variability of the quality of coal in the lot, irrespective of the mass of the lot. When designing sampling schemes, the measure of variability, i.e. the primary increment variance, often has to be determined from the results of sampling relatively small sampling units. This may be a serious underestimation of the variability of the whole lot, for example, when segregation occurs during transport of very large masses of coal, during stockpiling, or when coal is despatched or received over extended periods during which long-term changes in quality may occur. Therefore, lots should be divided into a convenient number of sampling units.

It is recommended that a lot be divided into a number of sampling units, u, not less than the number given in table 1.

Table 1 — Recommended minimum number of sampling units in a lot

Mass of lot tonne	Number of sampling units
< 5 000	1
5 001 to 20 000	2
20 001 to 45 000	3
45 001 to 80 000	4
80 001 to 125 000	5
125 001 to 180 000	6
180 001 to 245 000	7

This number may be increased so that the sampling unit coincides with a convenient mass or time, or because it is necessary in order to achieve the desired precision (see 4.5.5). It may also be necessary to increase the number of sampling units, if ambient conditions make it necessary to limit the time over which the sample is collected.

The quality of the lot shall be calculated as the weighted average of the values found for the sampling units.

4.5.5 Number of increments per sampling unit

As stated in 4.5.1, the precision is determined by the variability of the coal, the number of increments and sampling units and the preparation and testing variance. By transposing equation (1) it can be shown that the number of increments for a desired precision in a lot can be estimated from the following equation:

$$n = \frac{4V_I}{uP_L^2 - 4V_{PT}} \qquad \ldots (2)$$

If n is a practicable number, the initial scheme is established. However, if n is less than 10, take 10 increments per sampling unit.

If n is impractically large, increase the number of sampling units either by

a) increasing u to a number corresponding to a convenient mass or time, recalculating n and continuing this until n is a practicable number, or

b) deciding on the maximum practicable number of increments per sampling unit, n_1, and calculating u from equation (3) [derived from equation (B.7)]:

$$u = \frac{4V_I + 4n_1V_{PT}}{n_1P_L^2} \qquad \ldots (3)$$

If necessary, adjust u upwards to a convenient number and recalculate n.

This method of calculating the number of increments required per sampling unit for a certain precision from the primary increment variance and the preparation and testing variance will generally give an overestimate of the required number. This is because it is based on the assumption that the quality of coal varies in a random manner. In addition, because a certain amount of preparation and testing is required when measuring the increment variance, the preparation and testing errors are included more than once.

The designer of a sampling system shall cater for the worst case anticipated, and will use higher values for V_I than may actually occur when the system is in operation. On implementing a new sampling scheme, it is therefore recommended that a check on the actual precision being achieved be carried out using the methods described in annex B. The number of increments/sampling units for the type of coal in

question can then be adjusted according to that procedure, so that the required precision can be achieved with the minimum sampling cost.

4.5.6 Examples

EXAMPLE 1

The lot is 80 000 tonnes delivered in 1 000 tonne train loads and the required precision, P_L, is ± 0,25 %.

The quality variation is known and the following values have been determined:

$$V_I = 0,5, \quad V_{PT} = 0,05.$$

a) Initial number of sampling units

From table 1, $u = 4$

Therefore, take 4 sampling units of 20 000 tonnes each.

b) Number of increments per sampling unit

Using equation (2)

$$n = \frac{4 \times 0,5}{\left(4 \times 0,25^2\right) - (4 \times 0,05)} = 40$$

Therefore, take 4 sampling units of 40 increments each.

EXAMPLE 2

The lot is 100 000 tonnes delivered as 5 000 tonnes/day over two shifts and the required precision, P_L, is ± 0,25 %.

As the quality variation is unknown, assume $V_I = 20$ and $V_{PT} = 0,20$.

From table 1, $u = 5$, but for convenience take daily samples i.e. $u = 20$, in order to avoid storage of samples overnight.

Using equation (2)

$$n = \frac{4 \times 20}{\left(20 \times 0,25^2\right) - (4 \times 0,2)} = 178$$

This number is too large for a single sample, therefore try taking shift samples, e.g. $u = 40$.

$$n = \frac{4 \times 20}{\left(40 \times 0,25^2\right) - (4 \times 0,2)} = 47$$

It would then be sensible to take 48 increments per shift (1 every 10 min).

EXAMPLE 3

The lot is 8 000 tonnes in a single load and the required precision, P_L, is ± 0,5 %.

The quality variation is known and the following values have been determined:

$$V_I = 15, \quad V_{PT} = 0,20.$$

From table 1, $u = 2$

$$n = \frac{4 \times 15}{\left(2 \times 0,5^2\right) - (4 \times 0,2)} = \frac{60}{-0,3} = -200$$

A value of infinity or a negative number indicates that the errors of preparation and testing are such that the required precision cannot be achieved with this number of sampling units.

It could be decided that 50 increments is the maximum practicable number in a sampling unit, and from equation (3):

$$u = \frac{(4 \times 15) + (4 \times 50 \times 0,2)}{50 \times 0,5^2} = 8$$

This gives a practical sampling method of dividing the lot into 8 sampling units and taking 50 increments from each.

4.6 Mass of primary increment

The mass m, in kilograms, of an increment taken by a mechanical cutter, with cutting edges normal to the stream, at the discharge of a moving stream, can be calculated from the following equation:

$$m = \frac{CA}{3,6S} \times 10^{-3} \qquad \ldots (4)$$

where

C is the flow rate, in tonnes per hour;

A is the cutting aperture, in millimetres;

S is the cutter speed, in metres per second.

Since the design of the cutter shall conform to the requirements of 8.7 and 8.8, whatever the flow rate at the time of taking an increment, its extraction from the coal stream will be unbiased. The mass of increment, of itself, is irrelevant when considering extraction bias.

Within the constraints of coal nominal top size, cutter speed/aperture and coal flow rate, mechanical sampling increments/systems are normally designed in terms of the mass of coal they will sample, i.e. in-

crement mass. In order to give guidance to system designers, this part of ISO 9411 specifies a reference increment mass.

In most mechanical systems, the mass of primary increment collected [see equation (4)] will greatly exceed that necessary to make up a sample of the required mass. In some systems, the primary increments are therefore divided, either as taken or after reduction, in order to prevent the mass of the sample becoming excessive. The reference increment mass should be used as a guideline for the minimum average mass for primary increments or divided increments, but not as an absolute lower limit. In addition, when measuring primary increment variance (see B.2) at preliminary stages in the system design, these are examples of masses that can be used.

When the system is commissioned, the precision of the result can be estimated and adjusted (see annex B), by increasing or decreasing the number of increments in the sample, while keeping the same increment mass.

Table 2 gives values for the reference increment mass for a series of coal nominal top sizes. Reference increment masses corresponding to nominal top sizes between those given in table 2 may be estimated by interpolation.

NOTES

17 The comments on bias apply to increment extraction only. In systems with widely varying flow rates and hence, for flow-proportional increments, wide variations in increment mass, very small increments may suffer disproportionate changes in quality, e.g. loss in moisture. Precautions can be taken to prevent this. If such losses cannot be prevented and are found to cause relevant bias, either the flow of coal can be made more regular by such means such as buffer hoppers or a variable speed cutter (mass-basis sampling) should be used. Alternatively, increments can themselves be retained temporarily in a buffer hopper until there is sufficient mass to ensure bias-free passage through an on-line preparation system. On no account should a primary sampler, in a time-basis system or a mass-basis system, be switched off at low flow rates to avoid low mass increments.

18 The reference increment masses given in table 2 apply only to primary increments or to the divided increments resulting from their subsequent preparation. They should not be applied to the masses of individual cuts of sample dividers, either on individual increments or samples. Mass restrictions applying to division are given in 6.5.2 (divided increment) and 6.8 (sample).

Table 2 — Reference increment mass

Nominal top size of coal mm	Reference increment mass kg
300	100
200	25
150	15
125	10
90	5
63	3
45	2
31,5	1
22,4	0,75
16,0	0,50
11,2	0,25
8,0	0,15
5,6	0,10
4,0	0,10
2,8	0,10

4.7 Minimum mass of gross sample for general analysis/moisture determination

For most parameters, particularly those which are related to particle size, the precision of the result is limited by the ability of the gross sample to represent all the particle sizes in the mass of coal being sampled.

The minimum mass of gross sample is dependent on the nominal top size of the coal, the precision required for the parameter concerned and the relationship of that parameter to particle size. Such a relationship applies at all stages of preparation. The attainment of this mass will not, in itself, guarantee the required precision. This is also dependent on the number of increments in the sample and their variability (see 4.5.5).

Values for the minimum mass of gross sample or of the sample after division are given in table 3.

NOTE 19 The values in table 3 are based on a division variance of 0,01 with respect to ash. These values are generally suitable for off-line division, but for nominal top sizes of 16 mm and below the masses may not be sufficient to maintain the integrity of the sample when performing on-line division. When a coal is regularly sampled under the same circumstances, the precision obtained should be checked (see annexes B and C) and the masses adjusted accordingly. However, the masses should not be reduced below the minimum specified in the relevant standards for methods of analysis.