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**Solid mineral fuels — Mechanical sampling  
from moving streams —**

**Part 2:  
Coke**

**STANDARD PREVIEW**  
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*Combustibles minéraux solides — Échantillonnage mécanique sur  
minéraux en mouvement —*  
*Partie 2: Coke*



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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 9411-2 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Sub-Committee SC 4, *Sampling*.

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

- Part 1: *Coal*
- Part 2: *Coke*

Annexes A and B form an integral part of this part of ISO 9411. Annex C is for information only.

## Introduction

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

Whilst mechanical sampling is based on theoretical principles, it also relies heavily on practical experience. The information given in this International Standard is drawn from this experience in several countries.

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# Solid mineral fuels — Mechanical sampling from moving streams —

## Part 2: Coke

### 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 of coke from moving streams for physical testing, general analysis and the determination of total moisture in the coke. Methods for the preparation of these samples are also described. It also outlines the special problems encountered and the limitations imposed in the preparation of samples for the determination of physical properties. ISO 9411-1 deals with the methods of sampling of coal and preparation of coal samples in a similar way.

Apart from the taking of primary increments and primary increment division, this part of ISO 9411 describes the on-line single stage crushing and division of the moisture sample.

Methods for checking overall precision of sampling, testing for bias and general statistical methods are included in annexes A, B and C respectively. Methods for checking sample preparation errors, if required, may be found in ISO 9411-1.

### 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 2309:1980, *Coke — Sampling.*

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

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

ISO 9411-1:—<sup>1)</sup>, *Solid mineral fuels — Mechanical sampling from moving streams — Part 1: Coal.*

### 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 1 The accuracy of a result should not be confused with its precision.

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

1) To be published.

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

NOTE 2 This term is usually designated as  $v$ .

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

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

**3.5 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.

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

**3.7 cut:** An increment taken by a sample divider.

**3.8 cutter:** A mechanical device which takes a single increment of coke.

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

NOTE 3 Such division may be done with or without prior size reduction.

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

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

NOTE 4 The procedures of sampling, sample preparation and analysis are not necessarily perfect and the observations will be dispersed about the "true" values.

**3.12 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.

**3.13 general analysis test sample:** A sample, crushed to pass a sieve of nominal size of openings 212  $\mu\text{m}$  complying with ISO 3310-1, used for the determination of most chemical characteristics of coke.

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

**3.15 increment:** A portion of coke collected in a single operation of the sampling instrument.

**3.16 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.

**3.17 lot:** A discrete quantity of coke for which the overall quality to a particular precision needs to be determined.

**3.18 manual sampling:** The collection of increments by human effort.

**3.19 mass-basis sampling:** The taking of increments in uniform mass intervals throughout the sampling units.

NOTE 5 Each increment or divided increment constituting the partial sample or the gross sample should be of almost uniform mass.

**3.20 mechanical sampling:** The collection of increments by mechanical means.

**3.21 moisture sample:** A sample taken specifically for the purpose of determining total moisture and, where required, general analysis.

**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.

NOTE 6 Whilst square-hole sieve sizes are referred to throughout this part of ISO 9411, the approximate equivalent in a round-hole sieve may be obtained by multiplying the square aperture by 1,17.

**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 coke 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.

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**3.27 physical sample:** A sample taken specifically for the determination of physical characteristics, e.g. physical strength indices or size analysis.

**3.28 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.

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

**3.30 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.

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

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

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

**3.34 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.

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

**3.36 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.

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

**3.38 sampling unit:** A quantity of coke, the sampling of which results in one gross sample.

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

**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 coke stream at the time of taking the increment.

**3.43 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

The general procedure for sampling shall be as follows.

- 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 coke (see 4.4.2) and establish the number of sampling units  $u$  (see 4.4.4) required to attain the desired precision and the minimum number of increments  $n$  (see 4.4.5). Methods for determining variability are given in annex A.
- e) Decide whether to use time basis or mass basis sampling (see clause 5), and define the sampling intervals in minutes for time basis sampling or in tonnes for mass basis sampling.
- f) Ascertain the nominal top size of coke for the purpose of determining the reference increment mass.
- g) Determine the method of combining the increments into gross samples or partial samples and the method of sample preparation (see clause 6).

## 4.2 Types of sampling

### 4.2.1 Single lots

If the quality of coke of a type not previously sampled, delivered as a single large consignment, is required, then in order to devise a sampling scheme assumptions will have to be made about the coke variability. The precision actually achieved by the scheme devised can be measured by the procedure of replicate sampling (see annex A).

### 4.2.2 Regular sampling

If the same type of coke is sampled regularly, sampling schemes can be laid down using data derived from previous sampling. The procedure of duplicate sampling (see annex A) can be used to obtain the optimum scheme, thus keeping the sampling costs to the minimum necessary.

## 4.3 Design of the sampling scheme

### 4.3.1 Material to be sampled

The first stage in the design of the scheme is to identify the cokes to be sampled. Samples may be required for technical evaluation, process control, quality control and for commercial reasons by both the producer and the customer. It is essential to ascertain exactly at what stage in the coke handling process the sample is required, and as far as practicable to design the system accordingly.

In some instances, however, it may prove impracticable to obtain samples at the preferred points, and in such cases a more practicable alternative should be sought.

### 4.3.2 Parameters to be determined on samples

The samples for moisture and physical tests may be collected separately or as one sample which is then divided. In this part of ISO 9411 a sample which is collected for the determination of moisture (and possibly also for general analysis) is referred to as the moisture sample; a sample which is collected for physical tests only is referred to as the physical sample. If a gross sample is used for the determination of moisture and for physical tests, it is referred to as a common sample.

In the mechanical sampling of coke the only sample which can, in certain circumstances (see 4.3.6), be processed automatically beyond the divided increment stage is the moisture sample.

In order to achieve desired precisions, it may be necessary to take different numbers of increments for the moisture and physical samples. Where a common sample is taken, the greater number of increments shall be used.

### 4.3.3 Division of lots

A lot may be a single sampling unit or a series of sampling units, for example, coke despatched or delivered over a period of time, a ship load, a train load, a wagon load, or coke 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.

The 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.

### 4.3.4 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.

### 4.3.5 Precision of results

For each parameter to be measured, the required precision for a lot shall be decided. The minimum number of increments collected shall then be determined as described in 4.4.5, and the average mass of each increment shall be determined as described in 4.6.

For single lots, the quality variation shall be assumed as the worst case (see 4.4.2). The precision of sampling achieved may be measured using the procedure of replicate sampling (see annex A).

At the start of regular sampling of unknown cokes, the worst-case quality variation shall be assumed. When the system is in operation, a check shall be carried out to confirm that the desired precision has been achieved using the procedure of duplicate sampling as described in annex A.

If any subsequent change in precision is required, the number of increments shall be changed as deter-



mined in 4.4.5 and the attained precision rechecked. The precision shall also be checked if there is any reason to suppose that the variability of the coke being sampled has increased. The number of increments determined in 4.4.5 applies to the precision of the result when the sampling errors are large relative to the testing errors, e.g. moisture. However, in some tests, e.g. Micum Index, the testing errors are themselves large. In order to obtain a better precision for the result for the gross sample than that indicated by the test method, it will be necessary either to form two or more partial samples and prepare a test portion from each, or to prepare two or more test portions from the gross sample (see 4.4.5.3). The average of the results will then have an improved precision.

#### 4.3.6 Minimization of bias

In mechanical sampling, it is of particular importance to ensure as far as possible that the parameter to be measured is not altered by the sampling and sample preparation process or by subsequent storage prior to testing. For example, care should be taken to avoid breakage of coke intended for physical testing and loss of moisture from the moisture sample during storage.

In particular, it will be necessary when collecting large samples for moisture determination to combine the increments as a series of partial samples, determining the moisture content of each and combining the results to give the moisture content for the gross sample.

The use of on-line crushing and division of the moisture sample for moisture determination should be treated with caution because of the risk of bias caused by loss of moisture in the processing (see 7.2.2). In particular, the crushing of hot coke is not recommended. If the bias is unacceptable, the sample shall be left in the uncrushed state and the sample preparation carried out by manual methods. It has to be accepted, however, that some bias is inevitable, either due to breakage or loss of moisture from hot coke. The object, therefore, shall be to restrict such degradation to a minimum.

### 4.4 Precision of sampling

#### 4.4.1 Precision and total variance

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 re-

sults of a series of experiments made on the same coke 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 A. The following equation is derived from equation (A.5).

$$P_L = \pm 2 \times \left( \frac{V_1}{n} + V_{PT} \right)^{1/2} \quad \dots (1)$$

where

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

$V_1$  is the primary increment variance;

$V_{PT}$  is the preparation and testing variance;

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

$u$  is the number of sampling units in the lot.

If the quality of a coke 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 4.4.2). The precision actually achieved for a particular lot by the scheme devised can be measured by the procedures given in annex A.

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

#### 4.4.2 Primary increment variance

The primary increment variance,  $V_1$ , depends upon the type and nominal top size of coke, the degree of pre-treatment and mixing, the absolute value of the parameter to be determined and the mass of increment taken.

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

If at all possible, the primary increment variance,  $V_1$ , should be determined on the coke to be sampled using one of the methods described in annex A. If this cannot be done,  $V_1$  should be determined for a similar coke from a similar sampling system. If neither of these procedures is possible, assume an initial value of 25 for  $V_1$  and check this, after the sampling has been carried out, using one of the methods described in annex A.

**4.4.3 Preparation and testing variance**

If at all possible, the variance of preparation and testing,  $V_{PT}$ , should be determined on the coke to be sampled using one of the methods described in ISO 9411-1. If this cannot be done,  $V_{PT}$  should be determined for a similar coke from a similar sampling system. If neither of these procedures is possible, assume an initial value of 0,5 for  $V_{PT}$  and check this, after the preparation and testing has been carried out, using one of the methods described in ISO 9411-1.

**4.4.4 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 coke in the lot, irrespective of the mass of the lot. When designing sampling schemes, the measure of variability of the lot, 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 coke, during stockpiling, or when coke 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 — Number of sampling units in a lot**

Mass of lot tonne × 10 <sup>3</sup>	Number of sampling units
< 5	1
5 to 20	2
20 to 45	3
45 to 80	4
> 80	5

This number may be increased so that the sampling unit coincides with a convenient mass or time.

If, with this number of sampling units, in order to achieve the desired precision the number of increments in a sampling unit is too high for convenient handling, this minimum number of sampling units will need to be increased (see 4.4.5.1). It may also be necessary to increase the number of sampling units, if ambient conditions or the condition of the coke 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.

**4.4.5 Number of increments per sampling unit**

**4.4.5.1 General**

As stated in 4.4.1, the precision is determined by the variability of the coke, 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 single lot can be estimated from the following equation:

$$n = \frac{4V_1}{u \cdot P_L^2 - 4V_{PT}} \dots (2)$$

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 impracticably 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 (A.7)].

$$u = \frac{4V_1 + 4n_1 V_{PT}}{n_1 P_L^2} \dots (3)$$

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 coke 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  $V_1$  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 A. The number of increments/sampling units for the type of coke in question can then be adjusted according to that procedure, so that the required precision can be achieved with the minimum sampling cost.

#### 4.4.5.2 Moisture sample

The sampling variance for moisture may vary in the range 0,2 to 25, depending on the absolute value of the moisture content, the size range of the coke and the extent of cutting, screening and mixing it has undergone prior to sampling. For example, a closely graded, highly cut small sized industrial coke sampled on delivery to the customer would have a much lower variance than an uncut coke sampled at the wharf or a very large coke on despatch from the producer's works. It may be known from experience what level of variance is to be expected.

The variance for ash and other chemical properties is usually less than for moisture. However, it is often desired to obtain a higher precision for the ash result, and hence the number of increments should be calculated for each and the greater number taken for the moisture sample.

#### 4.4.5.3 Physical sample

The cokes to be sampled within the scope of this part of ISO 9411 will exhibit large differences in physical strength, size, size range, and size distribution. In addition, many different parameters, e.g. Micum test, porosity, percentage retained on a particular sieve, mean size etc., can be determined on the samples. Sample preparation errors may be zero when the test is done on the whole gross sample, or large when division of the gross sample takes place.

Furthermore, it is usually not possible to determine the individual increment variances for tests such as the Micum test because the increment mass is too small.

Because of these factors, it is not possible to draw up a simple table for the number of increments for the physical sample. Instead, it is recommended that the number of increments initially required be sufficient to give a mass of gross sample greater than the mass given in table 3 subject to a minimum of 10 increments.

It will be found with many physical tests that the only way to achieve the required precision will be either to

divide the lot into sampling units or to prepare two or more test portions from the gross sample, taking the mean result for the sampling unit. The precision for the particular parameter required should then be checked and the number of increments adjusted according to the procedure laid down in annex B.

## 4.5 Examples

### EXAMPLE 1

The lot is 35,000 tonnes of 40 mm × 20 mm coke in a single consignment.

Primary increment variance, $V_1$ , for moisture	5
Required precision, $P_L$	± 1,0 %
Preparation and testing variance, $V_{PT} = 0,02 \times V_1 =$	0,10

a) Initial number of sampling units

From table 1,  $u = 3$

b) Number of increments per sampling unit

using equation (2)

$$n = \frac{4 \times 5}{3 \times 1^2 - 4 \times 0,10} = 7,7$$

Therefore, split the lot into 3 sampling units and take 10 increments from each.

### EXAMPLE 2

The lot is 100,000 tonnes of 100 mm × 25 mm coke delivered as 5,000 tonnes/day over two shifts.

Primary increment variance unknown $V_1$ , initially assume	25
Required precision, $P_L$	± 0,25 %
Preparation and testing variance, $V_{PT}$ , maximum	0,20

a) Initial number of sampling units

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

b) Number of increments per sampling unit

Using equation (2)

$$n = \frac{4 \times 25}{(20 \times 0,25^2) - (4 \times 0,2)} = 222,2$$

This number will result in an unmanageable sample mass. Therefore, increase the number of sampling units to 40, i.e. 1 per shift.

$$n = \frac{4 \times 25}{(40 \times 0,25^2) - (4 \times 0,2)} = 58,8 \approx 59$$

This may still be considered too large a mass to crush as a single moisture sample. Therefore, increase  $u$  to 80, i.e. 2 per shift.

$$n = \frac{4 \times 25}{(80 \times 0,25^2) - (4 \times 0,2)} = 23,8 \approx 24$$

Take 24 increments per half shift i.e. one every 10 min.

EXAMPLE 3

The lot is 10,000 tonnes of 40 mm × 25 mm coke in a single consignment.

Primary increment variance, $V_1$	15
Preparation and testing variance, $V_{PT}$	0,2
Desired precision	± 0,5 %

a) Number of sampling units

From table 1,  $u = 2$

b) Number of increments per lot

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

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 40 increments is the maximum practicable number in a sampling unit, and from equation (3).

$$u = \frac{(4 \times 15) + (4 \times 40 \times 0,2)}{40 \times 0,5^2} = 9,2 \approx 10$$

This gives a practical sampling method of dividing the lot into 10 sampling units of 1,000 tonnes each, taking 40 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:

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

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 comply with the requirements of 7.4, whatever the flow rate at the time of taking an increment, its extraction from the coke stream will be unbiased. The mass of increment, of itself, is irrelevant when considering extraction bias.

Within the constraints of nominal top size, cutter speed/aperture and flow rate, mechanical sampling implements/systems are normally designed in terms of the mass of coke they will sample, i.e. increment 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 A.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 A), 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 nominal top sizes. Reference increment masses corresponding to nominal top sizes falling between those given in table 2 may be estimated by interpolation.

Table 2 — Reference increment mass

Nominal top size	Reference increment mass
mm	kg
> 125	20
125	10
63	5
31,5	2
16,0	1
< 16,0	1

NOTES

16 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 in-

crement mass, very small increments may suffer disproportionate changes in quality, e.g. loss in moisture. Precautions should be taken to prevent this. If such losses cannot be prevented and are found to cause relevant bias, either the flow of coke can be made more regular by 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.

17 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 6.6.3 (sample).

**Table 3 — Mass of gross sample/mass of sample after division**

Nominal top size	Minimum mass
mm	kg
> 125	2 000
125	1 000
90	500
63	250
45	125
31,5	60
22,4	30
16,0	15

NOTE 18 For moisture samples, there is a restriction of 700 kg on the maximum mass of sample to be crushed at one time during off-line preparation.

## 5 Methods of sampling

### 5.1 General

#### 4.7 Minimum mass of gross sample

For most parameters, particularly size grading and those that 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 coke being sampled.

The minimum mass of gross sample is dependent on the nominal top size of the coke, 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 4.4.5).

The masses specified in table 3 are for guidance on the minimum mass for unknown or heterogeneous cokes. Whilst they can usually be reduced for the moisture sample, they may be inadequate for the determination of, say, oversize to  $\pm 1\%$  precision, particularly on very large cokes. When a coke is regularly sampled under the same circumstances, the precision obtained for all the required quality parameters should be checked (see annex A) and the masses adjusted accordingly. However, the masses should not be reduced below the minimum requirements laid down in the relevant analysis standards.

The uses to which the sample is to be put, the numbers, masses and size distribution of the test samples required shall also be taken into account when deciding on the minimum mass of the gross sample.

Sampling should be carried out by systematic sampling either on time basis or on mass basis, or by stratified random sampling. For operational and economic reasons, time-basis sampling is the preferred method. The procedures of sample preparation vary in accordance with the type of sampling employed (see clause 6).

It is essential that the increments taken from a stream represent the full width and depth of the stream. The consistency of loading of the belt shall be controlled, as far as possible, so that sampling is as efficient as possible.

As far as possible, the flow shall be made reasonably uniform over the whole cross-section of the stream at all times, by means of controlled loading or suitable devices such as feed hoppers, ploughs etc.

Whichever method of primary increment collection is used, it is essential that the increment does not completely fill or overflow the sample container. With mechanized sampling devices, the increment mass may be considerably larger than the reference increment mass. Hence, a system of primary increment division may be necessary to allow division of the increment to a manageable mass.

All processes and operations upstream of the sampling location shall be examined for characteristics which could produce periodic variations in belt loading or quality, and which may coincide with the operation of the primary samplers. Such periodicity may arise from the cycle of operations or feeder systems in use. If it is not possible to eliminate coincidence between the plant operation cycle and the sampling cycle, stratified random sampling within fixed mass or time intervals shall be adopted.

## 5.2 Time-basis sampling

### 5.2.1 Method of taking primary increments

Each increment shall be taken by a single motion or by a complete cycle of the sampling device, so that a full cross-section of the coke stream is taken. The speed of the cutter shall be constant throughout the sampling of the entire sampling unit (see 7.4.2.2).

Primary increments shall be taken at pre-set equal time intervals throughout the lot or sampling unit. If the calculated number of increments has been taken before the handling has been completed, additional increments shall be taken at the same interval until the handling operation is completed.

### 5.2.2 Sampling interval

The time interval  $T$ , in minutes, between taking primary increments by time-basis sampling shall be calculated from the following equation:

$$T \leq \frac{60 Q}{Gn} \quad (5)$$

where

- $Q$  is the mass, in tonnes, of sampling unit;
- $G$  is the maximum flow rate on the conveyor belt, in tonnes per hour;
- $n$  is the number of primary increments in the gross sample (see clause 4).

The designer should ensure that the time taken for the sampling or processing of an increment is less than the time between increments under normal operating conditions. For a sampling unit of abnormally low mass, a reduction in feed rate may be necessary to allow processing of the required number of increments.

In order to minimize the possibility of introduction of bias, a random start within the first sampling interval is recommended.

### 5.2.3 Mass of increment

The mass of the primary increment corresponding to the average flow rate (total mass/operating time) of the coke stream shall be not less than the reference increment mass (see table 2).

The mass of the increment shall be proportional to the flow rate of the coke stream at the time it is taken.

## 5.3 Mass-basis sampling

### 5.3.1 Method of taking primary increments

Each increment shall be taken by a single motion or by a complete cycle of the sampling device, so that a full cross-section of the coke stream shall be taken.

Either a fixed or variable speed cutter may be used (see 5.3.3).

The required number of increments shall be taken by sampling at a preset mass interval. This interval shall not be changed during the sampling of the sampling unit.

If the calculated number of increments has been taken before the handling has been completed, additional increments shall be taken at the same interval until the handling operation is completed.

### 5.3.2 Sampling interval

The increments shall be spread uniformly on a tonnage basis throughout the mass of the lot or the sampling unit.

The mass interval  $m_i$ , in tonnes, between taking increments by mass-basis sampling shall be determined from the following equation:

$$m_i \leq \frac{Q}{n} \quad \dots (6)$$

- where
- $Q$  is the mass, in tonnes, of the sampling unit;
- $n$  is the number of primary increments in the gross sample.

The designer should ensure that the time taken for the sampling or processing of an increment is less than the time between increments under normal operating conditions. For a sampling unit of abnormally low mass, a reduction in feed rate may be necessary to allow processing of the required number of increments.

The mass interval between increments shall be equal to or smaller than that calculated from the number of increments specified in 4.4.5, in order to ensure that the number of increments will be at least the minimum number specified.

In order to minimize the possibility of introduction of bias, a random start within the first sampling interval is recommended.

### 5.3.3 Mass of increment

The masses of the individual increments added to the gross sample or partial sample shall be almost uniform, i.e. the coefficient of variation shall be less than

20 %, and there should be no correlation between the flow rate at the time of taking the increment and the mass of the increment. The method for determining whether or not these criteria have been met is given in annex C. These criteria may be achieved by either of the following procedures.

- a) Taking primary increments of almost uniform mass using a variable speed cutter whose cutter speed is constant while cutting the stream but can be regulated, increment by increment, in proportion to the flow rate of the coke at the point of sampling. The average increment mass shall be not less than the reference increment mass (see table 2). This is the preferred procedure.
- b) Taking primary increments using a fixed speed cutter, with subsequent division of the individual increments, if necessary, to almost uniform mass at a practical stage prior to the constitution of the partial or gross sample. The mass of the primary increment taken at the average flow rate of the coke stream shall be not less than the reference increment mass (see table 2).

## 5.4 Stratified random sampling

### 5.4.1 General

Cyclical variations in coke quality may occur during systematic sampling, i.e. time-basis or mass-basis sampling. Whilst every effort should be made to avoid such variations, it is not always practicable to eliminate the possibility that they may coincide with the taking of increments. In the case of coincidence, a bias will invariably be introduced that may be of unacceptable proportions. In such circumstances, stratified random sampling may be adopted in which, for each time or mass interval (as the case may be), the actual taking of the increment is displaced by a random amount of time or mass respectively, subject to the limitation that it shall be taken before that interval has expired.

NOTE 19 During stratified random sampling, it is possible that two increments will be collected very close together even though they are collected in different mass or time intervals. It is therefore necessary that the discharge bin of the primary sampler is large enough to accept a minimum of two primary increments at the maximum flow rate.

### 5.4.2 Time-basis stratified random sampling

The sampling interval shall be determined as in 5.2.2.

Prior to the start of each sampling interval, a random number between zero and the sampling interval, in seconds or minutes, shall be generated. The increments shall then be taken after the time indicated by the random number. The mass of the increment shall be proportional to the flow rate of the coke (see 5.2.3).

### 5.4.3 Mass-basis stratified random sampling

The sampling interval shall be determined as in 5.3.2. Prior to the start of each sampling interval, a random number between zero and the mass, in tonnes, of the sampling interval, shall be generated. The increment shall be taken after the passage of the mass of coke indicated by the random number. The mass of the increment shall be independent of the flow rate of the coke (see 5.3.3).

## 5.5 Reference sampling

Reference samples should be taken by the stopped-belt method to enable checking for bias (see B.5).

## 6 Methods of sample preparation

### 6.1 General

Since the sampling cut should have a width of at least 2,5 times the nominal top size of the coke, in many cases this will result in very large increment masses. The handling and preparation of such large increments would be either manpower or equipment intensive. Hence, division of primary increments prior to further treatment may be necessary to provide a manageable sample mass. However, any handling of coke will cause size degradation and moisture loss and hence bias. Any such bias should be kept to a minimum (see clause 8). Where samples are for moisture or general analysis, it is permissible to install on-line crushing to a nominal top size of 16 mm followed by sample division.

### 6.2 Type of division

When increments are prepared individually and constituted into partial samples or a gross sample, the division of increments shall be conducted either by constant-mass division or by fixed-rate division subject to the conditions set out in 6.3.1 and 6.3.2.

Constant-mass division is a method of obtaining divided samples of almost uniform mass (see 5.3.3) regardless of the variation in masses of samples to be divided.

Fixed-rate division is a method of obtaining divided samples of mass proportional to the mass of sample before division.

NOTE 20 The procedure recommended for fixed-rate division (see 6.4.2.2) is the simplest to implement. However, other procedures are permitted provided that the mass of the divided sample is proportional to the mass of the feed. For example, the number of cuts could be kept constant by making the feed rate for each division proportional to the mass of coke to be divided.

Examples of types of sample divider are shown in figure 1. In addition, most types of primary sampler