

Sampling of coal and coke —

Part 2: Methods for sampling of coke

Committees responsible for this British Standard

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- Association of County Councils
- British Coal Corporation
- British Steel Industry
- Chamber of Coal Traders
- Electricity Association
- Low Temperature Coal Distillers' Association of Great Britain Ltd.
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Foreword

This revision of this Part of BS 1017 has been prepared under the direction of the Solid Mineral Fuels Standards Policy Committee. It supersedes BS 1017-2:1960 which is withdrawn.

The principal changes in this revision are as follows:

- a) “gas coke” has been excluded;
- b) sampling directly from wagons is no longer described because such operations are too dangerous;
- c) detailed descriptions of sampling equipment have been omitted because these were too restrictive and many were obsolete.

Part 1 of this standard deals with methods for the sampling and sample preparation of coal.

Attention is drawn to the provisions of the Health and Safety at Work etc. Act 1974 [1] and the need to ensure, from the initial stage of design and construction, that appropriate precautions are taken to ensure the safety of personnel carrying out the provisions required by this part of BS 1017.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 46, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Part of BS 1017 describes methods for the sampling of coke and the preparation of test samples for the determination of total moisture content and for general analysis. The preparation of samples for other purposes is described in Annex A.

2 References

2.1 Normative references

This Part of BS 1017 incorporates, by reference, provisions from specific editions of other publications. These normative references are cited at the appropriate points in the text and the publications are listed on the inside back cover. Subsequent amendments to, or revisions of, any of these publications apply to this Part of BS 1017 only when incorporated in it by updating or revision.

2.2 Informative references

This Part of BS 1017 refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Definitions

For the purposes of this Part of BS 1017, the following definitions apply.

3.1

sample

a representative portion of coke taken from a larger mass of coke the properties of which are to be determined

NOTE For a sample to be representative all particles have to have an equal chance of being included.

3.2

unit of coke

one of a series of portions of which a supply of coke is regarded as being made up and some or all of which are selected for sampling so that each sample represents a particular unit

NOTE If a sample representative of the complete consignment is taken, the consignment itself is the unit.

3.3

increment

a portion of coke collected by a single operation of the sampling instrument

3.4

test sample

a sample which is prepared to meet the requirements of a specific test

3.5

total moisture sample

a sample prepared for the determination of total moisture content and subsequently used for the preparation of the general analysis sample

3.6

general analysis sample

a sample prepared for chemical analysis

3.7

subsample

a sample consisting of a number of increments spaced evenly over the unit

3.8

replicate sampling

the taking from the unit of increments which are combined in rotation into different containers to give two or more subsamples of approximately equal mass, each being representative of the whole

3.9

duplicate sampling

a particular case of replicate sampling with only two replicate subsamples

3.10

regular sampling

the sampling of the same coke handled regularly at a given point

NOTE There are two forms of regular sampling: continuous sampling and intermittent sampling.

3.11

continuous sampling

the taking of a sample from every unit of the coke being handled

3.12

intermittent sampling

the taking of a sample from only certain units of coke (e.g. from one shift per day)

3.13

nominal top size

the smallest sieve aperture in the range included in the R20 series, square hole in accordance with Table 3 of BS 410:1986, such that not more than 5 % of the sample is retained

3.14

sample preparation

the process whereby a test sample is obtained from a sample by particle size reduction, mixing and sample division in successive stages

3.15

stage (of sample preparation)

the group of operations comprising particle size reduction, mixing and sample division

3.16

particle size reduction

the process of milling, crushing or grinding the sample to reduce the particle size

3.17

sample division

the process in sample preparation whereby a representative part of the sample is retained whilst the remainder may be rejected

3.18

pass (in sample division)

the passage of all the sample retained at a given stage through the sample division system

3.19

step

the treatment of one part of the sample in a sample divider

NOTE A pass consists of one or more steps.

3.20

precision

a measure of the extent to which observations within a set agree with each other

NOTE For the purposes of this standard the numerical value of precision is taken to be $\pm ts$, where t is the appropriate value of Student's t at the 95 % confidence limit and s is the standard deviation.

3.21

accuracy

the closeness of agreement between an observation and the true value

NOTE The accuracy of a result or method should not be confused with its precision. A determination may be made with great precision and the standard deviation of a number of determinations on the same limit may therefore be low, but the results will be accurate only if they are free from bias.

3.22**deviation**

the difference between an experimental result and an arbitrary central value, usually the experimental mean value

3.23**variance**

the sum of the squares of deviations from the mean divided by one less than the number of results, i.e.:

$$\text{variance} = \frac{\sum (x - \bar{x})^2}{n - 1}$$

where

x is the sample value;

$\bar{x}$ is the mean sample value;

n is the number of results.

3.24**standard deviation (s)**

the positive square root of the variance i.e:

$$s = \left\{ \frac{\sum (x - \bar{x})^2}{n - 1} \right\}^{1/2}$$

NOTE Also,

$$s = \left[\frac{1}{n - 1} \left\{ \sum x^2 - \frac{(\sum x)^2}{n} \right\} \right]^{1/2}$$

3.25**bias**

- 1) The tendency to obtain a value which is either persistently higher or persistently lower than the true value.
- 2) The numerical value of the difference between the true value and the mean result obtained from a large number of determinations using a biased method.

3.26**error**

the difference between the observation and the true value, which can be designated systematic (bias) or random

NOTE The procedures of sampling, sample preparation and analysis are necessarily imperfect and the observations will be dispersed about the true values.

3.27**random error**

error that has an equal probability of being positive or negative

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

3.28**range**

the difference between the greatest and least values of a number of observations

3.29**rogue result**

a result which appears to be in disagreement with others from the same material and which arouses suspicion that there has been a mistake in the sampling, sample preparation, or analysis

3.30

screening

the commercial operation of separating the smaller pieces of coke from the larger by shaking or rolling the coke over a surface provided with appropriately sized apertures

3.31

sieving

the laboratory or test operation of size analysis

3.32

grading

the commercial operation of sorting coke between two screens such that the ratio of the larger to the smaller screen aperture does not exceed 2.5 : 1

3.33

large coke

the oversize which is removed before separating the largest size of graded coke

3.34

breeze

the undersize remaining after separating the smallest size of graded coke

4 Sampling scheme

4.1 Types of sampling scheme

Decide, first, which is the appropriate type of sampling scheme to operate. Three types may be distinguished as follows:

- a) continuous sampling;
- b) intermittent sampling;
- c) consignment sampling.

With continuous sampling, increments are spread uniformly through the whole supply of coke, but are grouped together in samples for analysis and testing.

With intermittent sampling, the increments are not spread uniformly through the whole supply of coke. Instead, the coke is regarded as a series of units and certain units are selected for sampling. The selected units are spread uniformly through the whole supply of coke and the increments are spread uniformly through each unit selected for sampling.

In consignment sampling, as in continuous sampling, increments are spread uniformly through the whole supply of coke. The distinction is that in this case the particular consignment is to be considered separately. The distinction between the first two schemes and the third is quite clear. Consignment sampling relates to the problem of extracting a sample from a single consignment which is to be considered in isolation from any other consignments of the same coke. Such a case may arise, for instance, if a single consignment is received from a new source, or if a particular consignment is the subject of dispute or is being used for test purposes. Consignment sampling usually takes place in a strictly defined short period.

The other two schemes both come under the heading of regular sampling and apply where coke is passing in a stream so that the whole of it is accessible and all parts have an equal chance of being sampled. This condition arises at the point of production of coke, or at some consumers' works such as a blast-furnace plant where supplies of the same coke are regularly and frequently received.

At such places, either continuous or intermittent sampling will almost always be adopted since it is seldom necessary to have a high precision for the daily figure, the average at the end of a week giving the information required. Nevertheless, it is often desirable to have daily or shift figures of less precision for control purposes.

For practical reasons, a decision may be made not to sample every unit of coke. For instance, a consumer may not be able to sample every consignment of coke received or may take samples from only certain groups of wagons; or a producer may sample a particular grade of coke only every third day or on only three days per week. Such schemes are examples of intermittent sampling. More interest attaches to the average value for a number of samples than to any single sample. Intermittent sampling is not possible unless it is known, from previous experience, that the units sampled do not differ in quality from those that are neglected. It may sometimes happen that coke quality (especially in relation to moisture) is different on night shifts, or at the weekend, when there may be less control. If it is known that the coke produced on the night shift differs in moisture content from that produced on the morning shift, it would not be legitimate to sample the morning shift only. It would be legitimate to sample only six days a week if the coke quality on the seventh day was known not to differ from that on the other days. Consequently it is recommended that unless the sampled units are picked at random, the quality of the coke in the units not usually sampled should be checked from time to time.

If a value representing coke quality is required for every shift or every day, continuous sampling will have to be adopted, and it is better always to adopt this scheme. The increments are spread uniformly through the whole production, though they are grouped for analysis and testing purposes into samples, each representing one unit of coke.

In choosing between continuous and intermittent sampling, considerations of cost and convenience enter. Intermittent sampling is more flexible, since both the number of increments and the proportion of units sampled can be varied, but it introduces a risk that the sampled units may be unrepresentative.

In continuous sampling any increase in the number of increments per sample will give a greater precision for each sample and hence for the mean of a number of samples.

In intermittent sampling, on the other hand, the precision of the mean figure is influenced by the variation from unit to unit. Consequently although the collection of more increments will increase the precision of each sample value, the precision of the mean figure can be improved substantially only by collecting samples from more units. It may then be found in certain cases that to achieve a stated precision it is necessary to sample every unit, so that continuous sampling will have to be adopted.

At a point of production, sampling is continuous or intermittent depending on whether or not the units sampled represent the whole of the output. This difference is illustrated by the following examples.

- a) If a sample is composed of increments taken from one shift only on each day, then since the other shifts are not sampled, the sampling is intermittent; if samples are collected on only three days per week, the sampling is intermittent.
- b) If a producer takes increments throughout the whole of one day for a particular grade of coke and on the second day he takes a similar sample for another grade and so on throughout the week, then each grade is sampled only once per week and consequently the sampling is intermittent.

At a point of consumption, it is essential to establish whether the increments are spread over the whole of the receipts, or whether they are restricted to some units, others being omitted. The issue is complicated by the fact that when sampling from wagons the increments may be concentrated into certain wagons because of the difficulty caused by interrupting the unloading process, e.g. two or four increments may be taken from each wagon sampled.

The points of difference between continuous and intermittent sampling under such circumstances may best be illustrated by the following examples.

- 1) A consumer receiving wagon-borne coke from a given source takes one increment from every sixth wagon irrespective of its time of arrival or discharge. The increments are spread evenly over the whole of the receipts; the sampling is therefore continuous.
- 2) A consumer receiving wagon-borne coke takes a complete sample of 12 increments from a group of six wagons, two such groups being chosen from every consignment of coke received. Certain groups of wagon are not sampled; the sampling is therefore intermittent.

4.2 Establishing a sampling scheme

4.2.1 Continuous sampling

The first step is to decide on the desired level of precision and the period of sampling to which this applies, e.g. the weekly average may be needed to $\pm 1\%$. The number of increments to be collected during the period to give a precision of $\pm 1\%$ shall be as given in Table 1. If a different precision is required this number of increments shall be multiplied by the appropriate factor from Table 2.

Table 1 — Number of increments for precision of $\pm 1\%$ ^a

Class	Moisture content %(m/m)	Increments
1	3 or less	48
	3 to 5	72
	over 5	108
2	3 or less	100
	3 to 5	150
	over 5	225
3	All moisture contents	16

^a An outline of the assumptions made in deriving this table and a summary of the available evidence are given in Annex F.

Table 2 — Factor for number of increments

Precision of period mean %	Factor
0.25	16
0.5	4
1.0	1
1.5	0.43
2.0	0.25

This number of increments shall be collected by taking an equal number from each unit handled in the period. The increments shall be spread evenly over the unit, and the precision of each sample value shall be as given in Table 3. If samples greater than 70 kg are required sampling shall be carried out in accordance with 6.3.

NOTE 1 It is strongly recommended that in the first period this number of increments should be collected from each unit by duplicate sampling and the results examined by the methods given in Annex B.

NOTE 2 The use of the factor given in Table 2 presupposes that the errors due to sample preparation and analysis are small in comparison with the sampling errors; the factor has been calculated on the assumption that if more increments are required, more samples will have to be prepared and analysed separately, as described in 6.3. The period average may then be obtained to any desired precision by changes in the number of increments.

NOTE 3 If it is impossible to carry out the statistical check, then sampling should be carried out using the initial number of increments, but it should be understood that there is a risk of oversampling with excessive use of manpower and waste of coke.

Table 3 — Precision of single sample

Number of units in period	Precision of period mean (%)				
	0.25	0.5	1.0	1.5	2.0
	Precision of single sample (%)				
2	0.4	0.7	1.4	2.1	2.8
3	0.4	0.9	1.7	2.6	3.5
4	0.5	1.0	2.0	3.0	4.0
5	0.6	1.1	2.2	3.4	4.5
6	0.6	1.2	2.4	3.7	4.9
7	0.7	1.3	2.6	4.0	5.3
14	0.9	1.9	3.7	5.6	7.5
21	1.1	2.3	4.6	6.9	9.2
28	1.3	2.6	5.3	7.9	10.6
31	1.4	2.8	5.6	8.4	11.1
70	2.1	4.2	8.4	12.5	16.7
90	2.4	4.7	9.5	14.2	19.0

4.2.2 Intermittent sampling

With this type of scheme it is necessary to allow for the fact that the period average will be influenced by the variation between units, which cannot be predicted. More increments will be required over the period to achieve a given precision than if continuous sampling had been carried out, since the error of sampling a unit is now only a portion of the total error.

The first step is to decide the desired level of precision for the period average. Two aspects of the sampling shall then be considered as follows.

a) *Number of units to be sampled in the given period*

In order to determine the number of units to be sampled in a given period, information is required as to the variability of the coke in question. If no information is available from previous sampling, at least 10 units shall be sampled as soon as possible to obtain the necessary information. The results shall then be examined by the test given in Annex C to estimate the number of units to be sampled in the given period. It may be necessary to make adjustments and further batches of 10 shall then be sampled to confirm that the required precision has been achieved.

b) *Number of increments to be taken during the period*

Initially the total number of increments taken during the period shall be twice the number that would be required if continuous sampling were to be carried out. Thus, if a period average is wanted to $\pm 1\%$, the total number of increments during the period shall be double that given in Table 1. If a different precision is required this number shall then be multiplied by the appropriate factor from Table 2. This number of increments shall be collected by taking an equal number from each unit sampled in the period. The increments shall be spread evenly over each unit sampled.

NOTE 1 It is strongly recommended that at the outset the specified number of increments should be collected by duplicate sampling and the results examined as in Annex C. If duplicate sampling is not carried out there is considerable risk that more increments are being taken from each unit than is really necessary for the precision required.

NOTE 2 With this sampling scheme, a larger number of increments will not increase the sampling precision if relatively large errors are being introduced by the variation between units. To increase the precision of the period average it may be necessary to increase the number of units sampled, possibly until every unit is sampled. Continuous sampling has then to be adopted.

4.2.3 Consignment sampling

An assessment shall be made of the level of precision required for the consignment. The procedure adopted for fixing the number of increments depends on whether a consignment of the same coke has been sampled before.

If a consignment has been sampled before and the results have been analysed statistically, the number of increments to be taken shall be calculated from the previous results (see Annex D).

If the same coke has not been sampled before or was sampled before but not by subsamples, then the number of increments to be taken shall be calculated. The number of increments needed to give a precision of 1 % shall be determined in accordance with 6.1. If the desired precision is different from 1 %, then the number of increments to give this precision will be given by N/P^2 where N is the number of increments to give a precision of 1 % and $\pm P$ is the precision desired.

Provided that the sample is collected in at least six separate replicate samples the mean value may be determined to almost any desired precision by increasing the number of increments, but the question of variance due to sample preparation and analysis should be borne in mind (see E.9).

NOTE 1 It is strongly recommended that the necessary number of increments should always be taken in at least six separate samples (replicate sampling), successive increments being placed into bins labelled A, B, C, etc., and that each sample should be analysed separately.

When the sampling by this method is completed the results shall be examined by the method given in Annex D. The actual method of examining the results depends on the circumstances, as follows.

- If the sampling was arranged to obtain a specified precision the statistical check is used to determine whether this was obtained or not.
- If the sampling was not arranged to obtain a specified precision or if this check shows that the desired precision was not obtained, the precision that was actually obtained is calculated from the experimental results.

NOTE 2 If it is impossible to carry out replicate sampling then it is impossible to confirm that the specified precision has been achieved or to measure the sampling precision. Moreover, for high precisions, the variance due to sample preparation and analysis has to be taken into account and it may be impossible to achieve a very high precision (see Annex D).

4.3 Dummy runs

It will be seen that a high precision for a single consignment requires a very large number of increments, and it may well be desirable to reduce the number of increments as much as possible. The procedure given in Annex D may indicate that the required precision has not been achieved or that too many increments may have been taken, but this information will not be available until the trial is over.

If it is necessary to ensure that the specified precision is obtained or to reduce the number of increments on account of the work involved, consideration should be given to the possibility of carrying out a dummy run before the trial proper. This may be done by taking increments in six subsamples and analysing the results in accordance with the statistical procedure of Annex D. The number of increments taken may be half of the specified number. The increments shall be collected in such a way that they are spread throughout the whole bulk of the coke to be used for the trial.

Alternatively, it may be possible to carry out the dummy run on a bulk of the same coke which can be used for some other purpose before the trial proper begins. Then if this dummy run indicates that the number of increments needs to be increased or reduced, this change can be introduced for the proper trial, though a factor of safety should be allowed.

5 Precision

NOTE The statistical background to various requirements and procedures in this standard is given in Annex E.

5.1 Probability level

In describing the degree of precision obtained in sampling the results are referred to a probability of 95 % in accordance with the convention generally adopted. This means that if sampling is carried out correctly, in the long run the errors should not exceed the given figure more than once in 20 samples. Thus if coke is sampled to ± 1 % moisture content, then the moisture content determined will, on average, lie within ± 1 % of the true moisture content 95 times out of 100.

5.2 Precision of period mean

When a coke is received regularly, samples are usually taken regularly and it is necessary to distinguish between the precision of a result obtained from a single sample and the precision of the mean value obtained from several samples. The former expresses the closeness with which the result represents the true value for the unit of coke from which it is taken; the latter expresses the closeness with which the mean value represents all the coke passing the point of sampling including units which may not have been sampled at all.

5.3 Precision of continuous sampling

The two precisions are directly related only if the sampling is continuous, i.e. if every unit of coke has a sample extracted from it. In this case the relation is:

$$\begin{aligned} &\text{Precision of mean of } n \text{ samples} \\ &= \text{Precision of single sample} \div \sqrt{n}. \end{aligned}$$

The precisions applicable to a single sample are given in Table 3, where each column corresponds to a certain standard precision for the period mean. Thus if the period mean is to be determined to a precision of ± 1 % and there are seven units in the period all of which are sampled, then the precision of each unit value needs to be 2.6 % only.

5.4 Precision of intermittent sampling

When sampling is carried out intermittently, further considerations arise, since the samples which are taken are assumed to represent all units whether sampled or not. There will thus be errors due to the variation between units, and the mean of the sample values will be subject to a greater error than if the same number of units had been sampled by continuous sampling.

Thus with intermittent sampling it is necessary to consider the number of units to be sampled in a given period as well as the number of increments to be taken from each unit. The number of increments is chosen in the first place so that the variance due to sampling is a specified fraction of the variance between units. Then the number of units to be sampled is chosen so that the period mean has the desired precision.

5.5 Duplicate sampling

The recommendations given below for the number of increments required for a given precision are based on the most difficult cases which have been observed in the experimental evidence. With continuous sampling the number of increments specified should ensure that the specified precision is always obtained provided that the sampling is carried out strictly in accordance with the specification. Since this number is based on the most difficult cases it follows that in other cases the number of increments will be more than is really necessary.

With intermittent sampling it is not possible to specify in advance the number of units to be sampled to ensure that the average figure has the specified precision, since the variation between units has to be taken into account as well as the variation due to sampling. Thus an assurance as to the precision of the period average can be given only after some sampling has been carried out. Even when the number of units to be sampled has been checked, it is possible that more increments will be taken than are necessary for the precision required. In either of the above instances, oversampling is inevitable unless the number of increments for the required precision is determined in each particular case.

Thus with both intermittent and continuous sampling, it is advisable to calculate the sampling precision which has been obtained in the course of routine sampling. This can be done by using the procedure known as duplicate sampling: each sample is taken in two subsamples by putting alternate increments into two different containers. When a number of samples have been taken in this way, it is possible by comparison of the results of subsamples to estimate the sampling precision, provided that the sampling is unbiased. The number of increments may be adjusted to give the specified precision and in addition a check should be kept on the sampling to ensure that this precision is maintained.

NOTE It is strongly recommended that duplicate sampling should be carried out periodically whenever a coke is sampled regularly, because of the added security and the possibility of economizing by taking fewer increments if the results warrant it.

5.6 Precision for single consignments

A procedure similar to that of 5.5 is possible when sampling single consignments, but in this case the sample is collected in at least six subsamples.

6 Collection of sample

6.1 Number of increments

The number of increments required to give a specified precision will depend on the type of coke, its moisture content, the degree of precision chosen and, in certain circumstances, the conditions under which the sampling is to be carried out. In general, the number is independent of the total mass of coke sampled.

It is convenient to allocate the various types of coke to one of two main classes of increasing sampling difficulty, with an additional class for breeze, in accordance with Table 4. These coke classes require, therefore, an increasing number of increments and are subdivided further, according to the estimated moisture content of the coke, as in Table 1.

Table 4 — Coke sampling classification

Class	Description
1	Large or graded cokes from which breeze has been removed
2	Cokes from which breeze has not been removed.
3	Breeze

If there is any doubt as to the type of coke, it shall be assumed that it falls within the class which requires a greater number of increments to be collected.

If the numbers of increments in Table 1 are collected the sample value should give the percentage moisture content of the coke within $\pm 1\%$, at the 95 % probability level. For other levels of precision the number of increments shall be multiplied by a factor, as given in Table 2.

NOTE The numbers of increments given in Table 1 should be taken as an initial estimate. A reduction may be possible after tests for the precision achieved have been carried out (see 5.5).

6.2 Mass of increment

The sampling scoop shall be sufficiently large to take a representative sample of all the sizes present in the coke so that as little bias as possible is introduced.

The critical scoop dimension is the width, which shall be at least 2.5 times the nominal top size of the coke.

In addition, experiments have shown that, for the number of increments given in Table 1, in order to achieve the required precision, increments of 1.25 kg mass are adequate for cokes up to about 45 mm size, and increments of 2 kg mass for cokes up to about 63 mm size. For larger cokes, which are mainly encountered in the metallurgical industries, much larger scoops (nos. 3 and 4) are required. For extremely large coke the largest scoop (no. 4) may not be wide enough. Scoops large enough to sample this coke may be unmanageable and stopped-belt sampling is recommended. This relationship is set out in Table 5. The suggested scoops are described in Annex G. The masses referred to are nominal and the mass of coke collected is usually somewhat less than the nominal mass.

Table 5 — Sampling implements

Nominal top size of coke mm	Sampling implement
45	1.25 kg scoop (no. 1 scoop)
63	2 kg scoop (no. 2 scoop)
90	5 kg scoop (no. 3 scoop) or no. 2 shovel
125	10 kg scoop (no. 4 scoop)
Over 125	Stopped-belt sampling

If the statistical tests referred to in 4.2 and 4.3 show that an adjustment in sampling is necessary this shall be made by adjusting the number of increments: the scoop or shovel size need be changed only if it has been shown that the scoop in use gives biased results (see E.8). A larger scoop may always be used in place of a smaller one, but the number of increments shall not be reduced unless the method of duplicate sampling shows it is safe to do so. In cases of doubt, a larger scoop shall be used.

6.3 Collection of separate subsamples

The quantity of coke which is handled in one reducing operation shall not exceed 70 kg in mass, otherwise the coke would be exposed for such a time that appreciable loss of moisture would occur.

If the number of increments to be taken from each unit will result in a total mass of coke which is less than 70 kg, no difficulty arises.

Where samples of mass greater than 70 kg are needed, they shall be collected in separate subsamples which are reduced and divided separately. These subsamples shall be formed by placing successive increments in different containers to provide a permanent check on the sampling precision.

The same requirement applies to sampling a single consignment. If, on account of its mass, a sample has to be collected in subsamples not greater than 70 kg each, they shall be formed by putting increments into different bins in rotation since this procedure will enable an assessment to be made of the precision (see Annex D).

NOTE Where samples of much smaller mass are being taken each day, the samples may (if necessary) be retained in a container¹⁾ with a closely fitting lid for two or three days or until 70 kg has been collected, but it is strongly recommended that the moisture content of the coke from each day's increments be determined not later than the following morning.

6.4 Sampling for ash determination

Usually the moisture sample will be used for the determination of ash and other characteristics. If the sampling is sufficiently precise for moisture, it is usually sufficiently precise for these other properties. For low moisture cokes, or in some cokes which have a particularly high variance for ash, the ash may be a more difficult characteristic to determine than the moisture content. However, the limiting values of the variance, which are used to determine the number of increments given in Table 1, are large enough to cover all cases which have been noted in the experimental evidence.

The results for ash and for other analytical properties may be treated statistically in exactly the same way as the moisture results. If either the moisture or the ash result indicates that the number of increments should be increased, such an increase shall be made. The number shall be decreased only if both ash and moisture results indicate a decrease.

¹⁾ Dustbins conforming to BS 792 are suitable containers.

7 Method of taking increments

7.1 General principles

The sample shall be taken by increments of approximately equal mass, spread evenly over the unit to be sampled in such a way as to avoid bias due to size segregation.

NOTE Preferably, the increments should be taken while the coke is in motion, or from a stopped belt, or during loading into or unloading from wagons or lorries. Sampling from stationary material in heaps or piles, or from the tops of loaded wagons, is not recommended for moisture or ash determinations and is best avoided altogether.

When the coke is in motion the increments shall represent the full width and depth of the stream of material being sampled.

The sampling implement shall not be filled to overflowing.

To avoid loss of moisture in the sample the increments shall be placed directly in metal containers, such as dustbins, with close fitting covers and, preferably, the coke shall be cool when sampled.

7.2 Procedure of duplicate sampling

The two subsamples shall be taken by placing increments alternately in two bins labelled A and B. After 10 samples have been collected in duplicate, the results shall be examined by the methods in Annex B or Annex C, as appropriate. The results will show whether too many or too few increments are being taken in each sample and, in the case of intermittent sampling, whether too many or too few units are being sampled. The number of increments or the number of units sampled shall be amended in accordance with 6.1 or 4.2.2, as appropriate.

NOTE 1 This procedure does not increase the work involved in taking the sample, as no more increments are required when duplicate sampling is being carried out. In fact, after carrying out this procedure, it will generally be found that fewer increments can be taken in future than were at first estimated.

It is not necessary to continue to take every sample in duplicate: if an examination of two successive sets of ten samples does not indicate that any change in procedure is necessary, duplicate sampling need then only be used as a check. This may be done by taking one sample in five in duplicate or a run of 10 samples in duplicate after every 40 ordinary samples. With either method it is impossible to assess whether there has been a change in sampling behaviour until two sets of 10 duplicate results have been obtained. If at any time there is reason to believe that conditions have changed, a further set of 10 duplicate samples shall be collected at once.

NOTE 2 It is important that the check samples are taken with no more than ordinary care. One way of ensuring this is always to sample in duplicate, putting the two subsamples together, and then preparing the whole sample when duplicate results are not required.

7.3 Sampling from belt conveyors and loading booms

The increments taken from a conveyor shall represent the full width and depth of the stream and so the best method for sampling is to stop the conveyor. A complete section shall then be taken from such a length as will give the required mass.

NOTE 1 It is convenient to define a suitable position by marking the framework adjacent to the belt.

If it is not possible to stop the conveyor the coke has to be sampled whilst it is in motion. If physically practicable, the sample shall be taken as the coke passes from the loading booms into the wagons; if this is not possible, the coke shall be sampled as it passes from one conveyor to another, if there is sufficient fall for the insertion of the sampling scoop.

NOTE 2 A suitable point may be found where the coke falls from a belt on to the loading boom and a platform should be erected to make access easy and sampling safe.

A scoop (see Annex G) shall be used for sampling a moving stream. Care shall be taken that the stream is sampled in such a way that no bias is introduced. The scoop shall be inserted, for alternate increments, from the left and from the right and passed entirely through the stream to ensure that the coke from the full width is included in the sample. If the size of the stream is too great to be sampled as a whole, successive increments may be taken from adjacent parts of the stream.

Whether the belt is stopped or not, the loading shall be controlled as far as possible to prevent undue piling so that the increments will not be of excessive mass.