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Coke — Sampling

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the Member Bodies of the following countries :

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Canada	New Zealand	Turkey
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The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Belgium
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Coke — Sampling

1 SCOPE AND FIELD OF APPLICATION

This International Standard gives the practical directions for

a) the sampling of metallurgical coke from which breeze has been removed, for the determination of any property for both **routine and special purposes;**

b) preparation of a moisture sample and of a **laboratory sample** for the determination of ash and other properties.

This International Standard does not deal with the methods of analysing or testing the samples of coke obtained since these are the subjects of other ISO Recommendations and International Standards.

2 GENERAL

2.1 Guide to the reader : layout.

The following notes are given as a brief guide to the layout of this document.

Clause 3 discusses the standard of precision adopted. Clause 4 gives the numbers of increments required and clause 5 gives the minimum mass of increment. Clause 6 gives detailed instructions for sampling in various circumstances.

Clause 7 refers to the treatment of the sample and clause 8 to the report to be made.

Clause 9 gives details for the preparation of a **laboratory sample** for the determination of moisture and clause 10 for the preparation of a **laboratory sample** for general analysis.

The annex describes sampling apparatus peculiar to coke sampling.

1) At present at the stage of draft.

2.2 Theory

The general theoretical basis on which coke sampling is based is to a large extent similar to that for coal. This subject is extensively discussed in ISO 1988, *Hard coals — Sampling*[1)], and it is therefore not proposed to reproduce the relevant sections here. Reference should also be made to that International Standard for details of the methods for checking

a) the precision of sampling by means of replicate sampling;

b) the error of sample preparation;

c) the presence of bias.

As automatic sampling apparatus will, so far as is known, be common to both coal and coke, reference should also be made to ISO 1988 for details of such appliances.

Furthermore, as the problem of devising simple sampling instructions for operators is also a common feature for coal and coke, reference should be made to the suggestions given in ISO 1988 on this aspect.

While there are many theories of sampling, it is recognized that some are more suitable than others for application in particular circumstances. No theory has yet been proved to be satisfactory in all circumstances. For this reason, this International Standard is based primarily on practical experience, including a substantial volume of experimental data collected in several countries.

This International Standard gives methods of sampling which should cover all sampling problems likely to be encountered in international trade. It has been necessary, therefore, to describe a large number of alternative methods, with the result that the document is lengthy and is too complicated to be handed directly to a sampling operator. It is intended to be read by the engineer or supervisor responsible for sampling. It is important that the sampling operator should receive instructions which are simple, easily understood and capable of only one interpretation. These instructions, which should preferably be set out in writing, should be prepared by the sampling supervisor from the information given in this International Standard.

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2.3 Samples for moisture, physical tests and ash

The samples for moisture and physical tests may be collected separately or as one sample which is then halved. In this International Standard, a sample which is collected for the determination of moisture (and possibly also for general analysis) is referred to as a **moisture sample;** a sample which is collected for physical tests only is referred to as a **physical sample.** If a single sample is taken for the determination of moisture and for physical tests it is referred to as a **common sample.**

In most cases the general analysis sample will be prepared from the moisture sample. If, however, it is desired to collect a sample for the determination of ash only, such a sample is referred to as an **ash sample.** An ash sample may not be used for the determination of moisture.

2.4 Organization of sampling schemes

When the precision required for a given quantity of coke has been decided, the number of increments to be collected should be determined as described in clause 4. The mass of each increment should be determined as described in clause 5.

2.4.1 *Single consignment*

If coke is to be sampled from an isolated consignment, the required number of increments, each of the appropriate mass, should be taken from the consignment as described in clause 6, but if it is desired to confirm this beyond any doubt, the procedure of replicate sampling described should be applied.

2.4.2 *Regular consignments*

If the coke to be sampled is part of a regular series of deliveries from the same source, or part of the same production, the required precision will usually be related to a certain period; for example the weekly mean may be required to a precision of ± 1 in terms of moisture percentage. The coke handled during the period is considered to be made up of a number of units of coke, for example a shift's production, a day's production, a wagon load. The units can be fixed at will. When sampling from a stream of material, there are two possible methods of arranging the collection of the increments during the period; they can be collected either continuously or intermittently. However, when sampling from wagons, ships or stockpiles a coke which is received regularly, continuous sampling should normally be used.

2.4.3 *Continuous sampling*

In "continuous" sampling, every unit is sampled and the same number of increments should be collected from each unit. Thus the number of increments required to give the specified precision should be divided by the total number of units in the period to give the number of increments for each unit. This number of increments, each of the appropriate mass, should be taken from each unit as described in clause 6, whichever method is relevant. The increments from each unit should be pooled and a laboratory sample prepared therefrom so that one result is obtained for each unit. There are as many sample results for each period as there are units. The average should be of the required precision, but if it is desired to check that the required precision has been attained with the least possible number of increments, this can be done by using the procedure of duplicate sampling.

2.4.4 *Intermittent sampling*

It is often convenient to collect increments from some of the units of coke but not from others. Thus it may be desired to collect samples on, say, two days but not other days in a week. This is called "intermittent" sampling. The same number of increments is taken from every unit that is sampled. The number of units to be sampled should be decided and the total number of increments required should be divided by this number of units to give the number of increments to be taken from each unit sampled. The units to be sampled should be chosen at random; for example, if the sample is to be taken on only two days a week, the days for sampling should be selected at random each week (see 6.3).

The necessary number of increments, each of the appropriate mass, should be taken from each selected unit as described in clause 6. The increments from each unit are put together and a laboratory sample prepared therefrom so that there is one analysis for each unit sampled. There are therefore as many sample results per period as there are units sampled, but the number of units **available** is greater because there are some which are not sampled. In this case it is not possible to say that the average of these results will have the required precision until information about the variation between units is available. If the variation between units is too large, it may be necessary to introduce "continuous" sampling to achieve the desired precision.

"Intermittent" sampling cannot be carried out when sampling from ships or stockpiles and in such cases it is improbable that regular sampling can be carried out in any form since it is usually necessary to regard the coke in a ship or in a stockpile as a single consignment. Nevertheless, the conditions of continuous sampling might apply occasionally if coke from a single source was regularly received by ship or by barge.

3 STANDARD OF PRECISION ADOPTED

3.1 General

The limits given below apply only in the absence of bias.

The standards of precision are based on the 95 % probability level and any standard, either higher or lower than the reference standard, may be achieved by adjusting the number of increments as described in 4.3.

To ensure that the standard precision is achieved in all cases, the number of increments is based on the most difficult cases which have been observed. Consequently, the

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results may be of better precision than is required but it is not possible to reduce the number of increments in advance since the variability of the coke is not known. However, methods are given in ISO 1988 for checking the precision obtained and for reducing the number of increments in order to minimize the work involved whenever this is justified.

A check on the precision which has been obtained can be carried out by the method of replicate sampling : full details are also given in ISO 1988 and it is strongly recommended that these methods be used.

3.2 Moisture

The procedures set out in this International Standard are such that the reported moisture content of the coke should be within ± 1 % absolute of the true value at least 95 times out of 100

3.3 Ash and other chemical characteristics

It is recommended that sampling for the purpose of determining chemical characteristics be based on sampling for moisture. This characteristic is usually one of those to be determined and experience has shown that is is nearly always the most variable chemical characteristic of coke.

Sampling for a precision of ± 1 % for moisture will achieve the same or a better precision for all other chemical characteristics, but the reverse is not true.

3.4 Physical characteristics

The procedure set out in this document is based on the assumption that the coke should be sampled to give a reported mean size of the coke within ± 1/10 of the true mean size; the precisions of the cumulative percentages oversize, using the numbers and masses of increments given in this document, should not be worse than the following.

Cumulative percentage on one sieve	Precision %
0 – 5	3,5
5 – 10	4,0
10 – 20	4,5
20 – 40	5,0
40 – 50	4,5
50 – 60	4,0
60 – 70	3,5
70 – 80	3,0
80 – 90	2,5
90 – 95	1,5
95 – 100	1,0

3.5 Warning

In 3.1 it is stated that the standard of precision is arbitrary and that any standard, either lower or higher than the reference standard, may be obtained by suitable adjustment of the number of increments as indicated in 4.3. While this is true in principle, it is not entirely so in practice. Practical considerations limit the mass of coke that can be handled and hence the number of increments. It is generally inadvisable to attempt to attain a precision numerically less than 0,5 % for moisture, particularly with stationary coke. If a higher standard is required, it is advisable to attain this by averaging the results of several samples, so that the average results for a week or a month will have the desired "high" precision. The number of increments taken should never be less than 12.

4 NUMBER OF INCREMENTS

4.1 General

The numbers of increments given in 4.4 to 4.7 are those required to attain the standard precision.

If a precision other than the standard is required, see 4.3 and 3.5.

Where coke of consistent quality is regularly received from the same source, the procedure of duplicate sampling may be carried out and the number of increments may then be modified in subsequent tests in accordance with the results of the calculations, provided that the required precision is maintained.

In principle, the number of increments to be taken from a consignment from a single source in order to achieve a certain precision is a function of the variability of the coke in the consignment, irrespective of its mass, and the numbers of increments given in 4.4 to 4.7 would apply for any consignment. However, the segregation of material in large consignments is usually greater than in small consignments, and for this reason the number of increments given below apply only to consignments of up to 1 000 tonnes.

4.2 *Sampling large consignments*

For consignments of over 1 000 tonnes, two alternatives are permitted :

a) preferably the consignment should be divided into a number of portions, each of 1 000 tonnes or less, from each of which the specified number of increments should be taken;

b) alternatively, one sample only may be taken but the number of increments recommended for the particular case should be multiplied by the following empirical factor :

$$\sqrt{\frac{\text{mass of consignment (tonnes)}}{1\,000}}$$

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4.3 Precision different from the standard

For a precision different from the standard (for example ± 1,5 % instead of ± 1 %), the initial number of increments should be multiplied by :

$$\frac{4A_0^2}{5A_1^2 - A_0^2}$$

where

A_0 is the standard precision;

A_1 is the desired precision.

A higher precision is achieved by spreading the effect of sampling over a larger number of increments and thereby reducing the effect of sampling on the precision attained. However, the increase in the number of increments may be unduly high and unjustified unless the errors due to sample preparation and analysis can also be reduced. These errors can be reduced by grouping the increments into parts, from **each of which** one sample for analysis or testing is prepared.

In this case the following factor should be used to adjust the number of increments :

$$\frac{4nA_0^2}{5nA_1^2 - A_0^2}$$

where n is the number of gross samples.

For example, if six part samples are collected, the factor is :

$$\frac{24A_0^2}{30A_1^2 - A_0^2}$$

4.4 Moisture sample

The number of increments required depends on the method of sampling the coke in relation to its location and is shown in table 1.

TABLE 1 – **Number of increments for a precision of ± 1 % moisture**

Class	Number of increments for coke in			
	stream	wagons	ships	stockpile
Large or graded metallurgical coke from which breeze has been removed	50	75*	100	150

* Where the number of wagons to be sampled is less than 15, 5 increments should be taken from each wagon.

If the moisture content of the coke is to be determined, which is normally the case since no other "chemical" characteristic can be determined without it, the quantity handled in one crushing operation should not exceed about 70 kg, otherwise the coke would be exposed for such a time that appreciable loss of moisture would occur.

4.5 Ash sample

The number of increments should be as given in table 1.

4.6 Physical sample

The number of increments required to give the mean size within 1/10 of the true mean size depends on the nominal upper size of the coke and is shown in table 2.

TABLE 2 – **Number of increments for a precision of 1/10 of true mean size**

Nominal upper size	Number of increments for coke in			
mm	stream	wagons	ships	stockpile
< 50	50	75	100	150
51 – 100	20	30	40	60
101 – 200	10	15	20	30

4.7 Common sample

When a common sample is required, the number of increments must be adequate to give the requisite precision for both moisture and mean size. As the former requires more increments, the numbers of increments will be those shown in table 1. As these will result in the collection of more increments than are necessary for the standard precision for mean size, the precision achieved should be better than the standard, as shown in table 3.

TABLE 3 – **Precision of mean size, common sample**

Nominal upper size	Precision as fraction of true mean size
mm	
< 50	1/10
51 – 100	1/15
101 – 200	1/20

5 MINIMUM MASS OF INCREMENT

5.1 General

The masses of the increments given in table 4 below are minimum masses. Where practical considerations require the coke to be sampled by means of a mechanical sampler, for example from a fast-moving conveyor belt supplying coke to large furnaces, the masses of the increments will generally be larger than those stated in table 4.

Such a procedure is permissible and will in general ensure a higher accuracy of sampling, since the mass of the gross sample will be above that required for the recommended level of precision. The number of increments taken **must not** be reduced below that necessary for the precision required.

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5.2 Mass

The minimum mass of increment is given in table 4.

TABLE 4 – **Minimum mass of increment**

Nominal upper size*	Mass
mm	kg
⩽ 40	1
41 – 80	2
81 – 120	4
> 120	9

* The ranges of nominal upper size differ from those in table 2 because the experimental data from which these tables were derived originated from different laboratories working at different times and under different conditions. Such differences will, however, affect the mass of the gross sample to a minor and unimportant extent.

In addition, the following conditions should be satisfied :

a) when sampling from a stopped belt, the minimum width of the cross-section taken should be 2,5 times the upper size of the coke;

b) the minimum opening of the sampling implement should be 2,5 times the upper size of the coke.

6 COLLECTION OF INCREMENTS

6.1 General

The sample should 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.

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 the tops of loaded wagons is not approved.

Sampling machines should be used if possible, preferably at a point of discharge or, if this is not possible, from a moving stream. The increments should be taken at equal intervals of time or space and should be equal in mass.

It is important that the interval of time between successive increments should not coincide with any natural periodicity, either known or suspected, in the quantity or quality of the coke being sampled, since this would introduce a systematic error. Such a periodicity may arise from the cycle of operations at the coke ovens or at the colliery from which the coal is taken and particular care should be taken to avoid it.

The sampling implement should not be filled to overflowing.

The coke should be cool when sampled.

For cokes of 120 mm top size and above, manual sampling from a moving stream may be dangerous and the belt should be stopped if possible, or a sampling machine should be used.

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

The coke passing at the beginning and end of a flow should be ignored.

Any sample whose moisture is to be determined should be collected in a bin fitted with a sealed lid.

Any sample which is to be sized should be collected so that breakage is minimized.

Detailed instructions for the collection of increments are given in the following clauses.

6.2 Reference method

Sampling from a stopped belt is the most satisfactory way of ensuring that the sample is free from bias, since all the coke particles in the marked section are taken. It is therefore recommended as the reference method, which should be used for checking all other methods. Such a check is particularly important when sampling for size analysis from wagons, ships or stockpiles, where fines tend to collect at the bottom of the heap.

6.3 Random orders

A convenient way for determining random orders when sampling from wagons is as follows (the procedure is also applicable to sampling from barges and ships) :

Provide a set of discs, one disc for each position, suitably marked; for example a set of discs numbered 1 to 18. The discs should be placed in a bag close to the sampling point, together with a diagram painted on a fixed board showing the locations of the points over the surface of the wagon. On sampling from the first selected wagon, the sampling operator should remove from the bag 1, 2 or 3 discs, to correspond with the number of increments to be taken from this wagon. An increment should be collected from each area indicated by the discs. The discs should be placed in a second bag after use. For the second wagon the same procedure is used, the discs being removed from those remaining in the first bag. This process continues until all the discs are used up. The position of the bags is then reversed and the procedure continued, so that the order of the positions from which increments are taken is always different.

This procedure can also be used for selecting the wagons to be sampled, when some are sampled and some are not. For example, suppose 36 wagons are to be sampled out of a consignment of 100. A set of discs numbered 1 to 100 is placed in a bag and the sampling operator draws from the bag 36 numbered discs in succession. The selected discs may be hung on hooks on a reference board and the wagons numbered serially with chalk as they pass. The wagons corresponding to the numbers drawn should be sampled.

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6.4 Sampling machines

Sampling machines, controlled by hand or automatically, are available which will traverse a falling stream of coke or sweep increments from a moving conveyor belt. They should be adjusted carefully to ensure that the whole thickness and width of stream are taken. The increment must not fill the sample container completely.

6.5 Sampling from a stream of material

6.5.1 *General*

The increments taken from a conveyor must represent the full width and depth of the stream and may be taken manually or by a sampling machine.

The loading of the belt should be controlled as far as possible to prevent undue piling, so that the increments will not be of excessive mass.

The flow should 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.

6.5.2 *Sampling from a stopped belt*

A complete section should be taken, the minimum width of which should be 2,5 times the top size of the coke. It is convenient to define a suitable position by marking the framework adjacent to the belt; all coke on the marked section must be removed. Alternatively, a suitable frame may be placed on the stationary belt so that it is in contact with the belt across its full width; all coke lying inside the frame should be swept off into a container. Any large pieces of coke obstructing the insertion of the frame are pushed

a) at the left side of the frame, into the sample;

b) at the right side of the frame, out of the sample.

6.5.3 *Sampling from a moving stream*

6.5.3.1 Sampling from a point of discharge

A scoop with a minimum opening 2,5 times the top size of the coke should be used. Care must be taken that the stream is sampled in such a way that no bias is introduced. The scoop should 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 it is impossible to sample across the whole of the width of the stream in one movement without over-filling the container, the stream should be sampled systematically, taking the increments from parts of the stream in turn.

The stream must be sampled by passing the scoop through it once and then withdrawing it in such a way that the full scoop is not passed a second time through the stream; this may be achieved by inverting the scoop, passing it to the back of the stream and withdrawing it through the stream; alternatively, the scoop may be filled in passing from front to rear provided that it can then be withdrawn away from the stream – for example by moving it sideways.

Whichever method is used, the increment should not fill the sample container after it has traversed the stream.

It may be necessary to support the handle of the sample container across a bar when it is passed into the falling stream or to erect a special gantry with adequate supports.

The combination of the requirements that each increment should represent the full width and depth of the stream, and that the minimum opening of the sampling implement should be 2,5 times the top size of the coke, may produce an increment considerably larger than the minimum mass recommended, particularly where sampling machines are used.

6.5.3.2 Sampling from a moving belt

Sampling from a moving belt may be necessary if it is impossible to sample satisfactorily at a point of discharge. This procedure demands skill and good judgement on the part of the sampler. Care should be taken to ensure that the whole thickness of the stream is sampled. The scoop should sweep the bottom of the conveyor, otherwise there will be a tendency to leave behind some of the small coke.

If it is impossible to sample the whole width of the stream from one side, increments should be taken alternately from both sides of the belt.

Belts moving at high speeds or carrying heavy loads are dangerous; manual sampling from moving belts is therefore recommended only when the speed of the belt is not greater than 1,5 m/s, the height of the coke is not greater than 0,3 m and the flow is not greater than 200 tonne/h.

Automatic sampling machines are also available which will sweep an increment from a moving belt.

6.5.4 *Sampling from discontinuously moving streams*

Such devices are discharging conveyors, bucket elevators, bucket conveyors or aerial ropeways.

Sampling may be carried out provided that the mass of the contents of a bucket is not less than the mass of increment required, a condition that is fulfilled in nearly all cases. Increments should be collected from the coke stream at the point of discharge or at any point by stopping the line. The whole contents of a bucket should be taken as an increment. With large buckets, each bucket may be divided into sections if each is larger than the specified mass of increment; one of these only is taken, but in successive buckets each section must be taken in rotation.

6.5.5 *Sampling from drag-bar conveyors*

The sample may be taken from the discharge end of the conveyor. In order to take increments representing the full width and depth of the stream, it is convenient in some cases, for example when sampling from a drag-bar conveyor

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outside a retort house, to cut a slot in the bottom plate across the conveyor. The slot is normally closed by a slide, which can be withdrawn to allow increments to fall through the bottom plate into a suitable receptacle. The slide should be fitted so as to avoid a pocket in which fines will collect between sampling times. If this cannot be effected the first runnings from the sampling slot should be rejected.

6.6 Sampling from wagons

6.6.1 *General*

The increments must be taken in such a way that they are representative of all parts of the coke in the wagon. Normally, most of the coke in a wagon is inaccessible and the methods thus involve sampling during some part of the process of unloading. The method to be adopted depends upon the method of handling wagon-borne coke and upon the types of wagons concerned.

At some plants, wagon-borne coke is tipped and then conveyed on a belt in such a way that individual makes of coke can be separately identified. The coke should then be sampled from the belt as described in 6.5.

Where the belt method cannot be applied, but it is possible to empty a wagon partially – thus making accessible two faces at rest – it is then preferable to sample in the wagon itself. If this is not possible, increments may be collected from the discharging stream of coke underneath the wagon.

One or other of the methods described below should be convenient for use in any circumstances. **Sampling from the tops of wagons for moisture or ash or physical tests is not satisfactory and should not be adopted.**

6.6.2 *Sampling from bottom-door wagons*

In wagons with eight doors, only the four centre doors are used; in those with only four doors, all are used. The wagons may be sampled either from exposed faces or during discharge.

6.6.2.1 EXPOSED FACE METHOD

Two doors side by side are opened and part of the coke is discharged. The doors are then shut and bolted, leaving two sloping faces at rest in the wagon; it is assumed that the faces are numbered in areas as shown in figure 1 and increments are taken from the numbered areas. The doors to be opened are taken at opposite ends of the wagon in alternate wagons.

6.6.2.2 DISCHARGE METHOD

Increments are collected from underneath the wagon as the stream of coke falls from the doors; the method described in 6.6.5 should be used.

6.6.3 *Sampling from side-door wagons*

The wagon is partly emptied, leaving two sloping faces. Six sample positions are allocated to each of these faces, giving 12 sampling points, as shown in figure 1.

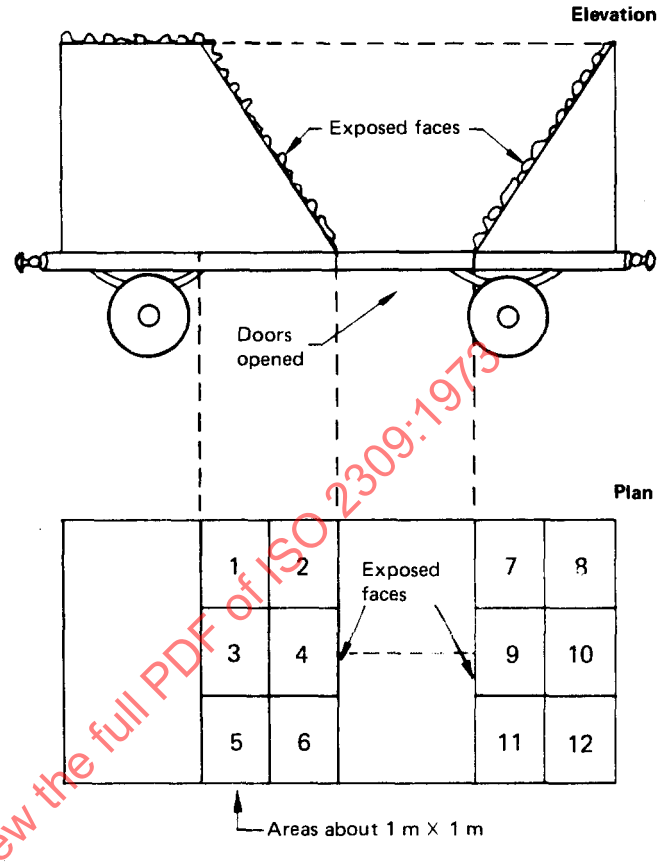

FIGURE 1 – **Exposed faces in wagon**

The method of filling the shovel is important as it should contain the correct proportion of the large and small coke; it must be held horizontally in the desired sampling position and then driven gently into the coke face to its full depth.

6.6.4 *Sampling from wagon side-tipplers*

The wagon is half emptied so that the bottom is just visible, leaving a face at rest. The wagon is then lowered slightly to prevent the face slipping. A suitable number of sampling areas are allocated to this face as in wagon top sampling and the increments are collected from the points in the same manner as above. It may be necessary to erect special gantries on rollers which can be pushed forward or retracted; or, alternatively, to use a scoop with a long handle.

6.6.5 *Sampling from wagons during bottom discharge*

As large a container as can be handled is swung into the coke stream during discharge, the position of entry being varied from wagon to wagon. Owing to the rapid fall of coke it is difficult to obtain increments from all parts of the stream and mechanical aids are required to render the method reasonably safe. This method is difficult to operate and is dangerous. Other methods should be used if possible.

6.7 Sampling from coke feeders

Increments should be taken from the discharge point and should represent the full width and depth of the stream. A

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suitably shaped box or receptacle should be swung through the stream. If possible, increments should be taken by swinging alternately from the front and from the back. If the feeder is too wide for this, increments should be taken from the two sides and the middle of the stream in appropriate order, but great care should be taken to avoid bias.

6.8 Sampling from ships

6.8.1 *General*

Increments are collected from a number of points distributed over various layers of the coke in the hold which are exposed from time to time as the ship is unloaded.

If during loading or unloading the coke is moved by conveyor, the sample should preferably be taken from some point in the conveying system where bias can be avoided more easily. The procedure set out in 6.5 should then be followed, but in view of the segregation that occurs during the loading of a ship, the number of increments indicated for ships in table 1 should be taken.

Usually, however, it is necessary to sample from the hold of the ship, and it is essential to employ a trained and experienced sampler who works under the direction of an expert. The size distribution of the cargo should be estimated so as to ensure that the increments taken are representative.

It is essential to check that holds are well ventilated before entering and that danger from falling coke has been minimized. A safety rope should be worn.

It is important to note that segregation during loading often results in the accumulation of lumps near the walls of the hold. It is necessary to take this into account when making an estimate of the size distribution.

The sampler's skill in estimating the size distribution should be regularly checked by such means as are convenient. One suitable method is to use a grab to remove a portion of the cargo of substantial size and to check the sampler's estimate of the size distribution of the contents by a size analysis carried out on the whole of this portion. Alternatively, his estimate can be checked by that of an expert who is not actually responsible for carrying out the sampling.

When sampling from a sea-going ship the opportunities for sampling are often limited since delays during unloading must be avoided. For this reason, procedures that are not ideal may have to be adopted on occasions.

In collecting each increment, care should be taken to ensure that it represents the coke in the vicinity and, in particular, that large particles are not allowed to roll from or into the scoop when the increment is extracted.

Care should be taken to ensure that the increments are not contaminated because of prevailing atmospheric conditions. As the top layer is liable to be affected by rain, or dried by the wind, it is desirable to collect increments at least 0,2 to 0,3 m below the surface unless this has been uncovered very recently.

The position of the increments should be distributed over the surface of the coke and the procedure described in 6.3 should be used.

6.8.2 *Sampling from coasters or sea-going vessels*

Coasters or sea-going vessels should be sampled in two or more stages, there being one stage for each 4 m of the depth of coke in the hold. Figure 2 illustrates the stages at which sampling should be carried out when it is done in three stages.

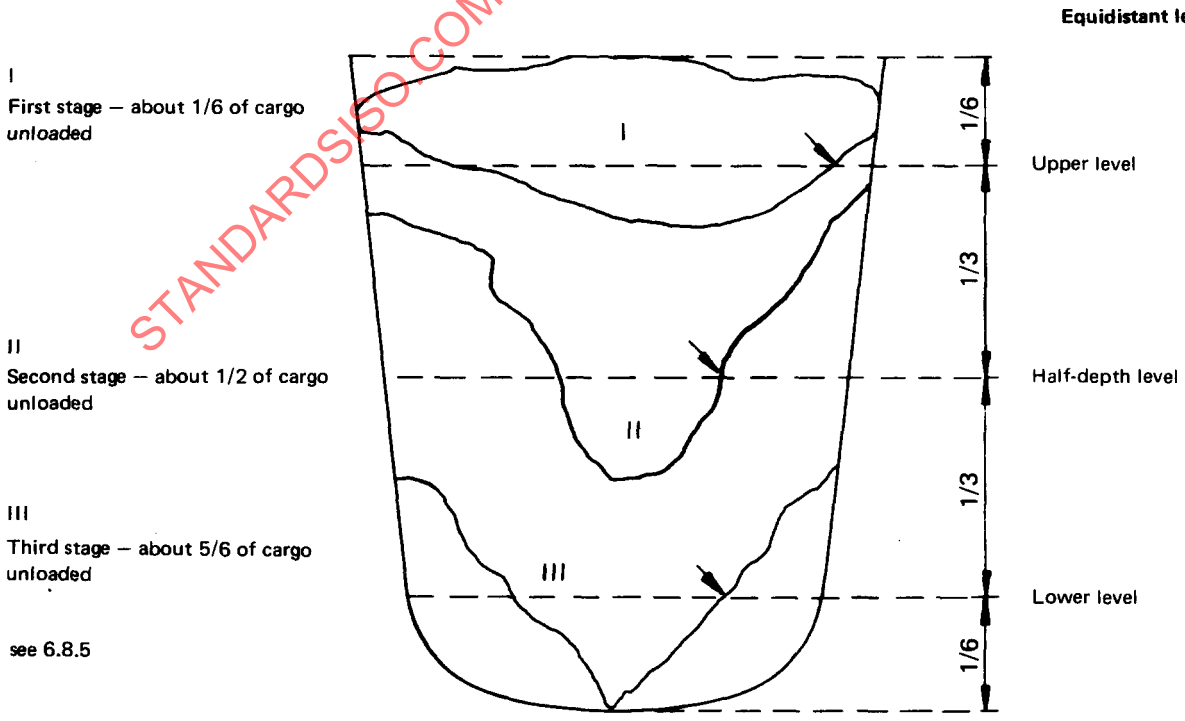

FIGURE 2 – **Sampling the hold of a vessel (moisture and ash)**

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The first stage of sampling should not take place until sufficient of the surface of the coke has been removed to permit an estimate to be made of the proportions of breeze and lump coke in the consignment.

At each stage increments should be collected from points evenly distributed over the exposed surface of the coke, taking particular care that all portions of the surface are covered satisfactorily.

6.8.3 *Sampling from barges*

If the depth of coke in the hold is less than 4 m, it should be sampled in one stage during the unloading. The sampling should be carried out when the unloading has partly uncovered the bottom of the hold.

6.8.4 *Sampling from grabs or conveyors*

When the coke in a ship or barge is unloaded by means of grabs or conveyors, it may be preferable to use the sampling scheme recommended in 6.5. However, the number of increments recommended for ships should be collected.

6.8.5 *Sampling for moisture*

If free moisture is present in the cargo this will settle towards the bottom of the hold so that a steady increase in moisture content will occur through the depth of the coke. This increase of moisture with depth makes the collection of a moisture sample particularly difficult when sampling is carried out in one stage (for example from barges). Increments should then be taken at three different levels, namely, at an upper and lower level, each equidistant from the centre level, and at half-depth (see figure 2).

Provided that the full depth of coke is exposed the upper level should be chosen 0,1 to 0,2 m from the top and the lower level at an equal distance from the bottom. If this is not possible (because the bottom has not been exposed, or the top coke has been removed), then the distance between the upper and lower levels should be as great as possible, subject to the condition that they are equidistant from the centre. Equal numbers of increments should be taken from the lower, centre and upper levels.

6.8.6 *Example*

The following example shows a method of sampling from a barge.

A sample of 48 increments is required from a barge of width 5 m, length 20 m and depth 2,5 m.

The barge should be divided into 100 sections (5 × 20), as follows, and 48 of the positions should be selected at random.

1					96
2					97
3					98
4					99
5					100

6.9 Sampling from stockpiles

6.9.1 *General*

Increments are spaced as evenly as possible over the surface and layers of the stockpiles. The usual method of sampling stationary material, digging holes to traverse the layers, is not adequate to fulfil the basic requirement of sampling so that the whole of the consignment shall be equally accessible. Moreover, the coke in the top layer of a stockpile is almost always different in quality from the rest, due to exposure, segregation and other causes. Consequently, if it is possible, in the course of stocking or lifting, to take a representative sample from a conveyor or from a falling stream, this method of sampling is to be preferred and the procedure described in 6.5 should be used, except that the number of increments should be as indicated for stockpiles.

The positions of the increments should be spaced as evenly as possible over the surface of the stockpile. It is advisable, especially in the case of large stockpiles, to work to a plan, to indicate the sampling positions on a scale drawing or map of the area and to mark the positions before sampling.

Occasionally, the stockpile may consist of a mixture of coke of different sizes, piled in separate areas of the total pile. Under these conditions, the masses and numbers of increments taken should be appropriate to the size of coke being sampled and a separate gross sample should be collected for each size fraction. Each fraction should be analysed separately and the value for the whole pile obtained by taking a weighted average of the analyses for the individual components.

In all cases the sample can only represent that part and that depth of the coke from which it is collected. It is essential to use trained and experienced samplers.

6.9.2 *Sampling for ash or physical tests*

The sample should contain the same proportions of large pieces and small pieces as are estimated for the stockpile.

In collecting each increment, care should be taken to ensure that it represents the coke in the vicinity and, in particular, that large particles are not allowed to roll from or into the scoop when the increment is extracted.

It is necessary to dig holes, by means of a shovel or a grabdredger. The angle of the sides should be less than the angle of rest of the coke so that no particles will trickle down the sides. Holes should be dug to different depths and from the bottom of each hole an increment should be taken by shovel so that samples of approximately the same mass are collected from different layers.

6.9.3 *Sampling for moisture*

The following additional points should be considered when sampling for moisture.

Stored coke gradually loses water by drainage until equilibrium is reached. After rainfall or snow, the moisture content will not change below a certain depth. This depth

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depends on the size distribution and the arrangements made during stocking, covering with breeze, or rolling the breeze.

The moisture content of samples collected from the surface of the stockpile largely depends on the weather. It will always be too low unless the samples are collected after rainfall or snow. Hence, it is necessary to dig holes to such a depth as will avoid the surface layer.

7 TREATMENT OF SAMPLE

7.1 Moisture sample

If the moisture content of the sample is to be determined, the increments should be placed as quickly as possible into metal or other impermeable containers provided with well-fitting lids, which should be replaced after each increment has been inserted. The sample should be kept in a cool place during storage.

A label giving a clear and sufficient description of the sample should be attached to the sample container.

The sample shall be prepared for the determination of moisture as described in clause 10.

Where samples of small mass are being taken each day, the samples may (if necessary) be retained in the container for two or three days or until about 50 kg has been collected, but it is strongly recommended that the coke from each day's increments should have the moisture content determined not later than the following morning.

7.2 Physical sample

If the sample is to have any physical property determined, the increments must be collected so that breakage is minimized.

A label giving a clear and sufficient description of the sample should be attached to the sample container.

7.3 Common sample

7.3.1 *General*

A common sample is collected so that the conditions of both 7.1 and 7.2 are satisfied.

The sample is halved as described below; the moisture half is treated as described in 7.1 and the physical half as described in 7.2.

7.3.2 *Halving the sample*

Prepare a container of such a size that it will hold the whole of one sample. The container should have two dividers crossing in the centre at right angles so that four equal compartments are made. The width of each compartment should be at least 2,5 times the maximum particle size. The dimensions of the container should be sufficient to allow for the space occupied by the dividers. An alternative arrangement which may be more convenient is to provide four containers which can be joined to form a cross and later separated.

Each sample which has to be halved should be carefully and quickly poured into the container over the centre cross so that moisture is not lost and breakage is not caused. Two diagonally opposite portions should then be removed and put together for the physical sample.

7.4 Ash sample

An ash sample may not be used for the determination of moisture.

The ash sample should be treated as described in section 10.

8 REPORT

The sampler should prepare a report stating the number and size of increments, details of the sampling procedure, full details of the origin and condition of the coke and the precision adopted. This report should be attached to the sample or otherwise made available to the recipient of the final results. A sampling plan may also be included, if required.

9 PREPARATION OF SAMPLE FOR DETERMINATION OF TOTAL MOISTURE

9.1 General

The procedure for the preparation of the 1 kg sample for the determination of total moisture is shown in diagram A.

Samples which are visibly wet and those with moisture likely to exceed 15 % are partially air-dried as described in 9.2 before reduction and division[1].

Precautions must be taken to prevent loss of moisture during these operations, which should be carried out in an enclosed space, roofed over and made free from draughts. The total time taken to crush and divide the coke should not exceed 15 min, and it is therefore essential to crush the coke quickly and mechanically.

9.2 Visibly wet samples

Samples which are visibly wet, or with moisture likely to exceed 15 %, should be partially air-dried in the following manner :

The whole of the sample is weighed in its container on a weighing machine which can be read to an accuracy of 100 g or better. It is then spread out in a thin layer on a steel plate in a warm, well-ventilated room and left until there is no visible moisture on either the coke or the steel plate. The minimum time of exposure should be 12 h. In the meantime the container is dried and weighed. After air-drying, the sample is collected in the original container and weighed. The percentage loss in mass is recorded, and

1) In this International Standard, "reduction" means diminution of particle size, and "division" means diminution of mass of sample.

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DIAGRAM A

PREPARATION OF 1 kg SAMPLE OF COKE FOR TOTAL MOISTURE

1) More than one pass may be necessary.

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details written on the label of the sample container. Alternatively, it may be partially dried in a large oven at 200 °C for 4 h, or overnight if more convenient. The sample is then treated as described in 9.3 and 9.4.

After the moisture has been determined (see 9.5), the total moisture, T.M., will be given by :

$$\text{T.M.} = X + M\left(1 - \frac{X}{100}\right)$$

where

X is the percentage loss on partial drying (taken from the label);

M is the percentage of moisture determined as described in 9.5.

9.3 Sample reduction

The whole of the sample should be broken down to the required size in a mechanical jaw crusher of a size and type which will allow the sample to be crushed to a nominal size of 20 mm, i.e. so that it contains not more than 5 % above 20 mm. The time taken to crush 50 kg should not exceed 5 min. The process should be timed occasionally to ensure that this time is not exceeded. The crusher used must be capable of crushing the largest sizes of coke being sampled in the specified time. Details of a typical apparatus are given in the annex and in figure 3.

The jaw crusher should be installed conveniently near to the sampling points to minimize carriage, and should be placed on a smooth concrete floor or manganese steel floor

plate which can be swept clean. Precautions should be taken against loss of moisture by undue ventilation or loss of sample as dust.

9.4 Sample division

Mechanical or hand division should be adopted as appropriate, the former being preferred.

9.4.1 *Mechanical division*

The collection of the 1 kg sample (see also 9.4.3) from the crushed sample may be carried out mechanically. The two types of divider described in the annex and illustrated in figures 4 and 5 have been found suitable. For both machines two passes[1)] are necessary to divide about 70 kg of sample to 1 kg. The number of passes may be increased, if this is found necessary in order to achieve the desired precision.

For the container type (figure 4), the first pass consists of two or three steps, in each of which some 20 kg are sample divided to 1 kg, then in the second pass some 2 to 3 kg are sample divided to give a product of 1 kg.

For the cone type (figure 5), the feed bin is inverted onto the upper hopper, which will hold about 35 kg of coke. With the aperture set at about 1/8 to 1/10, some 3 to 5 kg of coke falls into the sample bin and the remainder into the reject bin whence it is discharged. The second 35 kg bin is inverted onto the top of the divider so that after two steps the sample bin holds about 8 kg. This quantity of coke is returned to the feed, and after the second pass the sample bin holds about 1 kg of coke. By adjusting the width of the variable sector on the cone, it is possible to use the same setting for both passes so as to give the required sample of 1 kg.

For both types of divider the whole operation should be completed in 15 min, or less, to prevent loss of moisture.

The machines may be installed so that the product from the jaw crusher is discharged directly into the feed bin of the sample divider, so that reduction and division may proceed concurrently. Precautions should be taken to prevent undue ventilation.

9.4.2 *Hand division*

The crushed sample should be quickly mixed on the floor with a shovel, then scraped into a heap and flattened or laid out in a strip. Care should be taken to avoid loss of moisture from prolonged exposure to air, or by undue ventilation.

At least 20 increments, of approximately equal mass, should be taken from various points of the heap or strip to make up 1 kg. The increments should be taken uniformly from all parts of the sample, including the more finely divided material of the lower layers, by digging the whole width of the shovel to the bottom of the coke each time and withdrawing material from the whole depth of the coke; in the case of a strip, increments should be taken across the whole width of the strip.

The moisture sample should be placed in a closed container and the moisture content determined as soon as possible.

9.4.3 *Reserve samples*

If a second sample is needed as a reserve or check sample, duplicate 1 kg samples should be taken at the same time, either by suitably modifying the procedure described in 9.4.1 or by taking alternate increments when carrying out the procedure described in 9.4.2. The collection of a 2 kg sample which is subsequently halved is not satisfactory.

9.5 Determination of moisture

The 1 kg moisture sample, taken as described above, should be removed to the laboratory in a closed container and the moisture determined in accordance with the relevant ISO document.

When air-drying has been used in the preparation of the sample in accordance with 9.2, the percentage loss of moisture in this operation should be recorded on the label with a reference to the method of sampling and preparation used.

9.6 Further reduction of sample for general analysis

After the determination of moisture, the dried samples may be used for the preparation of samples for general analysis, as described in clause 10.

10 PREPARATION OF SAMPLE FOR GENERAL ANALYSIS

10.1 General

The procedure for the preparation of the 60 g sample for general analysis is shown in schematic form in diagram B.

The material used may be the 1 kg sample of coke which has been dried in the determination of total moisture (see 9.6).

Alternatively, a separate 1 kg sample may be prepared as described in clause 9 and dried.

The process of sample preparation, which involves two distinct operations, namely reduction and sample division, may lead to errors due to contamination, segregation and loss.

10.2 Contamination

The contamination error arises from the abrasive nature of coke, which tends to wear the surface of mild steel, cast iron, porcelain and – if a rubbing action is used – most of the surfaces used for grinding equipment. This error may be

1) Definitions of the terms "stage", and "pass" are given in ISO/R 1213/II, *Vocabulary of terms relating to solid mineral fuels – Part II : Terms relating to coal sampling and analysis.*

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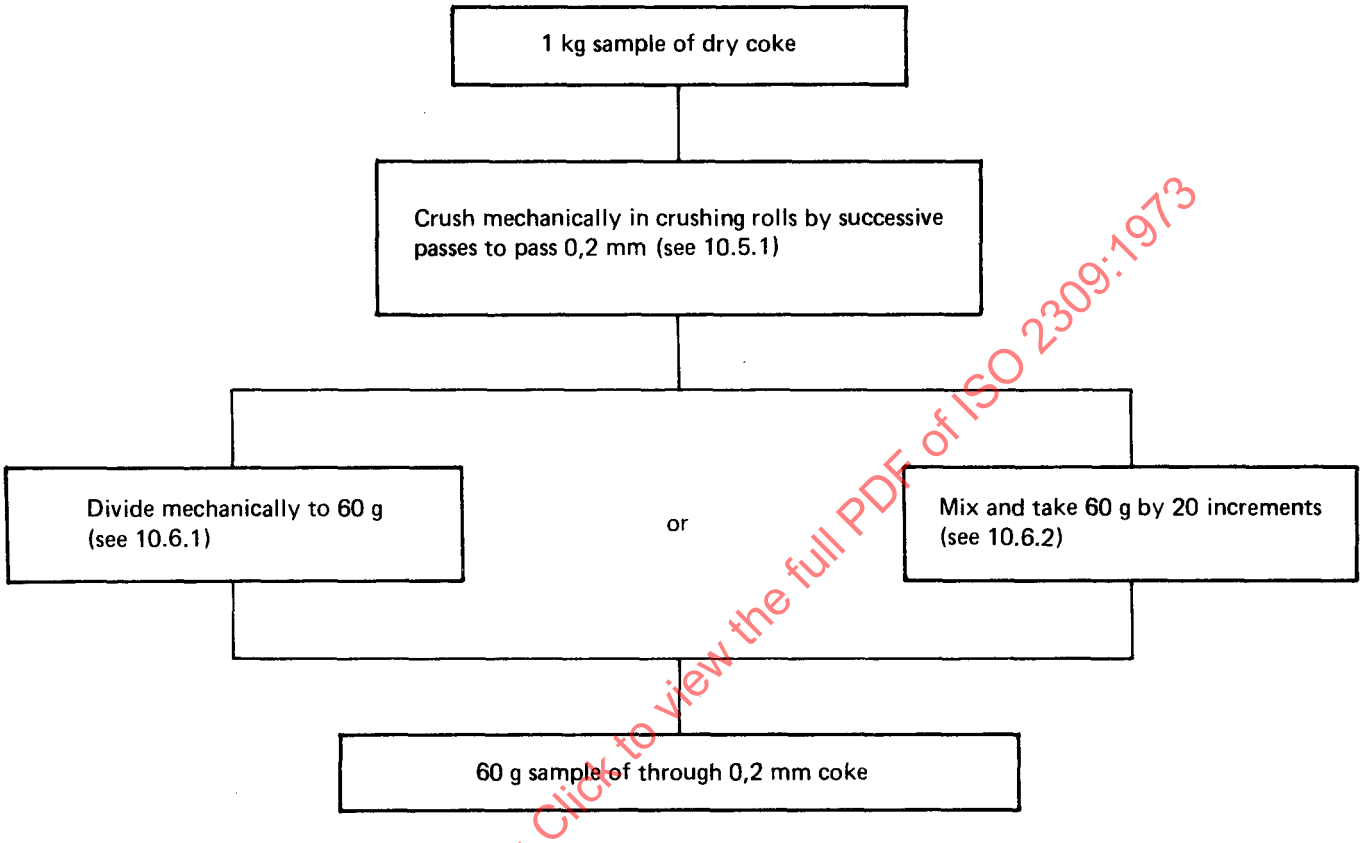
DIAGRAM B

PREPARATION OF 60 g SAMPLE OF THROUGH 0,2 mm COKE FOR GENERAL ANALYSIS



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as much as 3 % of ash if the dried moisture sample is reduced to 0,2 mm by rubbing on a cast iron surface. Although contamination usually implies the presence in the ash of iron oxide derived from a ferrous grinding surface, it may also involve an increase in the phosphorus content if cast iron surfaces are used. The contamination error can never be entirely eliminated, but it can be reduced to negligible proportions if the recommended methods and equipment are used. Methods of size reduction other than those recommended, such as rubbing against metal or other surfaces, are unsatisfactory and should never be used.

10.3 Segregation

The segregation error arises from the heterogeneous nature of coke and because of the difficulty of mixing uniformly any coke in which there is a wide range of sizes. It is therefore essential to mix the coke thoroughly in order to obtain a representative sample.

The segregation error increases with both the ash in the coke and the size at which division occurs. It can be made negligible – even for high-ash cokes – if the particle size is reduced to 0,2 mm, the size required for general analysis, before division occurs.

10.4 Loss

Precautions should be taken to prevent loss of fine material during the operations.

10.5 Particle size reduction

The size reduction of the dried 1 kg sample of coke (see 9.3) to pass an 0,2 mm sieve should be carried out by any suitable means, such as by crushing in chromium-steel crushing rolls.

The coke should be crushed in successive passes until it is all smaller than 0,2 mm. The rolls should be capable of crushing to this size without causing contamination. An example of suitable crushing rolls is described in the annex and illustrated in figure 6. The hand wheel should be adjusted to that the gap between the rolls or the pressure on them suits the degree of reduction at each pass of the sample. The settings of the rolls for the first three passes should be such as to give 95 % through 4 mm, 2 mm and 1 mm respectively. For further passes the setting for the third pass should be used.

10.6 Sample division

Mechanical or hand division should be adopted as appropriate, the former being preferred.

10.6.1 *Mechanical division*

A suitable sample divider to give 60 g of the through 0,2 mm coke is described in the annex and illustrated in figure 7.

10.6.2 *Hand division*

If a mechanical sample divider is not available, the sample should be mixed, spread out in a flat heap and a sample of 60 g taken by hand by not less than 20 increments from various parts of the heap.

10.7 Mixing the analysis sample

Before being placed in the storage container the sample should be well mixed.

10.8 Storing the sample

The sample for general analysis should be stored in a suitable container with an air-tight cover. A glass jar with a screw-on lid is a convenient container. A description of the sample should be affixed to the container.

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ANNEX

APPARATUS

A.1 JAW CRUSHER

The example shown (figure 3) has replaceable serrated jaws, size 0,25 m, made of chilled cast or alloy iron, and is capable of crushing large coke to pass 15 mm at the rate of 50 kg in 5 min.

Such a machine requires a 5 hp motor and is operated at about 250 rev/min. The crushed material can be collected either in a metal container set below the machine or else discharged by means of a steel chute directly onto a smooth floor. The jaw opening must not be set too narrow, otherwise the coke is crushed too finely and the rate of crushing will be reduced below the standard of about 50 kg in 5 min.

The jaw opening of the machine illustrated is adjustable by varying the length of visible thread and should be set to give a nominal 15 mm.

A.2 LARGE ROTARY SAMPLE DIVIDERS

A.2.1 Container type

A container type sample divider is illustrated in figure 4. It consists of a hopper, an inverted truncated cone with a suitable aperture to give the required rate of output, a fixed cone to distribute the coke, and a receiver with several containers on a rotary base, revolving at 60 rev/min. The receiver will hold 25 kg of through 15 mm coke but, to make it easier to reject the waste, three 1/4 containers are used (each of area 1/4 of that of the receiver) and the remaining 1/4 segment is filled by two 1/12 and two 1/24 containers. Each 1/12 and 1/24 container has one side extended and formed into a lip which engages the adjacent container. One 1/24 container should preferably be made of stainless steel. With 25 kg of coke, the 1/24 container will hold approximately 1 kg so that, with 15 mm coke, this is the required sample and the rest is waste. It has been found to be desirable to collect the coke in about 1 min. The hopper is counterbalanced by weights in the hollow side members so that it can be lifted for removal of the containers.

The fraction retained after the first pass depends on the mass of the sub-sample; the fraction retained after the second pass is such as to give a mass of just over 1 kg.

A.2.2 Cone type

A rotating cone type of sample divider is illustrated in figure 5. It consists of a hopper for holding the coke, an inverted cone with an aperture and two collecting nozzles, discharging into bins to hold the sample and the reject coke. The coke feeds from the reservoir through a cylindrical tube onto the surface of the cone. This has an aperture in it forming a sector of the cone which can be adjusted so that its area varies from 5 to 20 % of the cone's surface. The cone is rotated at a speed of about 20 rev/min by a 0,1 hp motor. When the coke falls onto the cone it is deflected outwards into the large distributing chute and then falls into the reject hopper. When the aperture in the cone is below the feed, the coke falls into the smaller circular receptacle and thence into the collecting bin. The slots are adjusted to give a fraction of 1/8 to 1/10 in the first pass and a product of about 1 kg in the second.

All bins are sealed to prevent loss of moisture.

The parts of the sample divider can be adjusted vertically relative to one another on the four corner posts of the apparatus and, where convenient, the discharge can be turned to waste and, possibly, the feed received directly from the jaw crusher on a platform at a higher level. Alternatively, if the discharge can be arranged to go to waste at a low level, the feed point can be reduced to a more convenient height of operation.

A.3 CRUSHING ROLLS

The crushing rolls illustrated in figure 6 are of 0,2 m diameter and 80 mm face; they are made of chromium steel and are operated at about 150 rev/min. The gap between the rolls, or the pressure on the rolls, is controlled by spring-loading of the bearings of the rear roll and is adjusted by varying the position of the front roll by hand wheels.

Above the rolls is a feed box with a distributor which is mechanically operated for most of the pass but which must be hand operated finally to charge all the feed. The rolls are mounted on suitable side supports so that a box may be inserted to collect the product. The box must be made substantially dust-tight to prevent loss of sample. By sealing possible dust exits,

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the loss of sample can be reduced to about 5 %. For preference, the rolls should be kept in an atmosphere of less than 80 % relative humidity, to avoid corrosion.

After continuous use for a long period, the rolls will wear and, with the sides of the rolls touching, a 1,5 mm gap in the centre is sufficient to justify regrinding.

A.4 SMALL ROTARY SAMPLE DIVIDER

A suitable sample divider for the sample division of up to 1 kg of material finer than 0,2 mm is illustrated in figure 7.

It consists of a hopper with steep sides to give a continuous flow, from which the coke passes through an aperture which is variable according to the size of the coke and is set to pass 1 kg in 1 min onto a fixed cone; thence it passes into a rotating cylindrical receiver, a suitable fraction (1/16, 1/8 or 1/2) being collected in a container as the sample; the remainder is waste.

The container of the required fraction of the cross-sectional area is clipped onto a fixed diameter.

The cylindrical receiver is rotated at about 64 rev/min through a 0,1 hp motor and reduction gear. A clutch enables the hopper to be fixed above the cylinder during use and to be swung to one side for emptying.

A.5 SAMPLING IMPLEMENTS

A.5.1 Scoops

Sampling scoops should be made of about 2,5 mm sheet steel according to the dimensions given in figure 8. They should be fitted with a handle of appropriate length.

A.5.2 Shovels

The standard sizes of the numbered shovels are as given in table 5.

TABLE 5 – Sizes of shovels

Shovel No.	Dimensions of blade	
	Length m	Width m
1	0,32	0,25
2	0,40	0,33

For sampling from side-tipped wagons it is necessary to use a shovel with a built-up back as shown in figure 9.

A.5.3 Two-handled scoop

The two-handled scoop shown in figure 10 consists of a light steel frame-work 0,3 m square carrying a shallow container. This is made of a piece of canvas or other flexible material fixed on a stout 10 mm wire, 0,3 m square, and is fitted to the underside of the framework. Wooden shafts, respectively 1,2 m and 1,5 m long, are fitted to the ends of the framework, and the shorter shaft bears a V-shaped handle.

A.5.4 Sampling device

In sampling for moisture from side-tipped wagons it is advisable to use a sampling device selected to ensure that an increment of the right size is obtained. A suitable sampling device is shown in figure 11.

A.6 GANTRIES FOR WAGON-TIPPLERS

In sampling from side-tipped wagons, it will probably be necessary to erect one or two special gantries to gain easy access to the wall of coke in the partly emptied wagon. These gantries, or their protective rails, may have to be movable so that they are not fouled by the wagon when it is tipped.

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Dimensions in metres

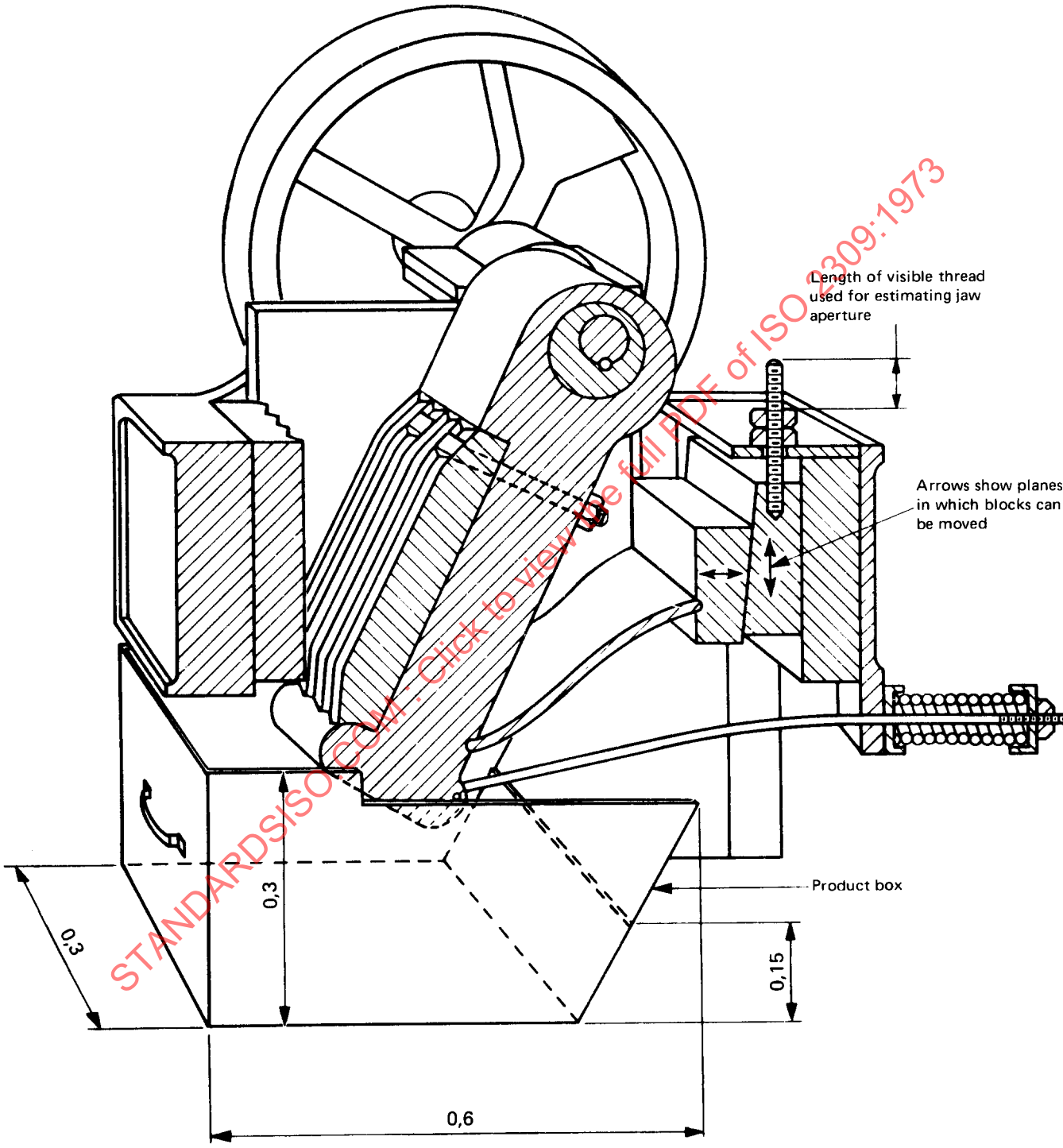

FIGURE 3 – **Jaw crusher with product box**

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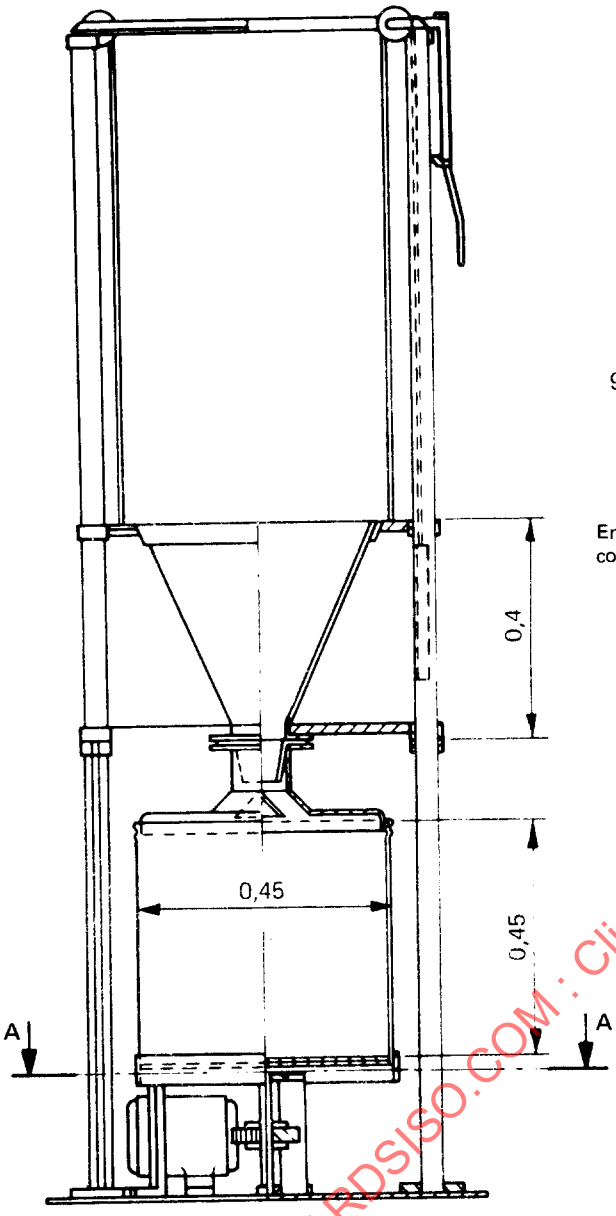
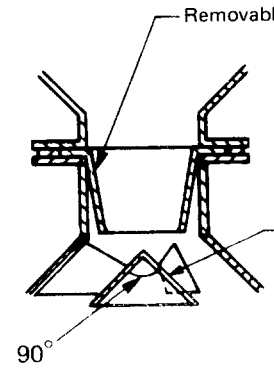
Dimensions in metres



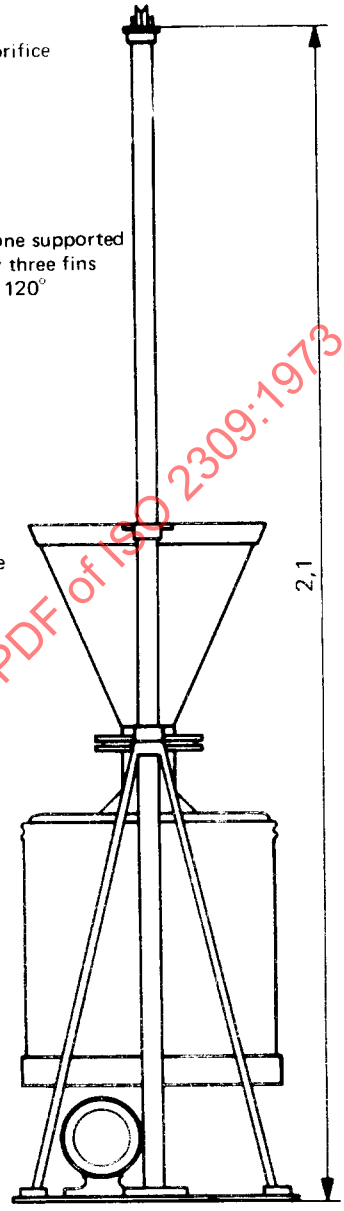
Half-sectional front elevation



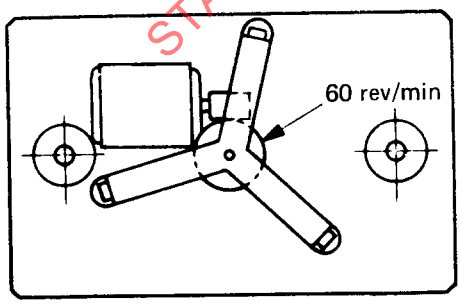
0 50 100 150 mm

Scale

Enlarged detail of distributing cone and orifice for 15 mm coke

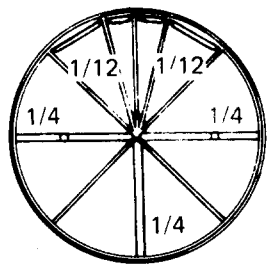


End elevation



Plan below section A-A

End view of a 1/24 container showing clip



Plan of receiver and containers

0,05 0,15
0 0,1 0,3 0,45 m

Scale

FIGURE 4 — **Sample divider — Container type**

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