SLOVENSKI STANDARD
SIST ISO 2309:1998

01-februar-1998

Koks - Vzorļenje

Coke -- Sampling

Coke -- Échantillonnage

Ta slovenski standard je istoveten z: **ISO 2309:1980**

ICS:

75.160.10 Trda goriva Solid fuels

SIST ISO 2309:1998 **en**

International Standard 2309

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Coke — Sampling

Coke — Échantillonnage

Second edition — 1980-02-01

UDC 662.749.2 : 620.113 **Ref. No. ISO 2309-1980 (E)**

Descriptors : coke, sampling, preparation of test pieces.

Price based on 23 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

International Standard ISO 2309 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels.* The first edition (ISO 2309-1973) had been approved by the member bodies of the following countries :

Australia	Ireland	Switzerland
Canada	New Zealand	Turkey
Czechoslovakia	Poland	United Kingdom
Denmark	Portugal	USA
Egypt, Arab Rep. of	Romania	USSR
Germany , F.R.	South Africa, Rep. of	Yugoslavia
India	Spain	
Iran	Sweden	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Belgium
France

This second edition, which supersedes ISO 2309-1973, incorporates draft addendum 1, which was circulated to the member bodies in April 1978. This draft addendum has been approved by the member bodies of the following countries :

Australia	India	Spain
Austria	Iran	Turkey
Belgium	Japan	United Kingdom
Canada	Mexico	USA
Egypt, Arab Rep. of	Poland	USSR
France	Romania	Yugoslavia
Germany, F.R.	South Africa, Rep. of	

No member body expressed disapproval of the document.

Printed in Switzerland

CONTENTS

TABLES

FIGURES

DIAGRAMS

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) the sampling of breeze, or small coke of nominal top size 20 mm or less;

c) 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 International Standards.

2 REFERENCES

ISO 579, *Coke — Determination of total moisture.*

ISO 687, *Coke — Determination of moisture in the analysis sample.*

ISO 1213/2, *Vocabulary of terms relating to solid mineral fuels — Part 2 : Terms relating to coal sampling and analysis.*[1)]

ISO 1213/3, *Vocabulary of terms relating to solid mineral fuels — Part 3 : Terms relating to coke.*[1)]

ISO 1988, *Hard coal — Sampling.*

3 GENERAL

3.1 Theory

The contents list provides a brief guide to the layout of this document. 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 and the relevant sections are therefore not reproduced. Reference should also be made to ISO 1988 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 specifies 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.

1) In course of revision.

3.2 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 shall not be used for the determination of moisture.

3.3 Organization of sampling schemes

When the precision required for a given quantity of coke has been decided, the number of increments to be collected shall be determined as described in clause 5. The mass of each increment shall be determined as described in clause 6.

3.3.1 *Single consignment*

If coke is to be sampled from an isolated consignment, the required number of increments, each of the appropriate mass, shall be taken from the consignment as described in clause 7 but if it is desired to confirm this beyond any doubt, the procedure of replicate sampling described shall be applied.

3.3.2 *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 shall normally be used.

3.3.3 *Continuous sampling*

In "continuous" sampling, every unit is sampled and the same number of increments collected from each unit. Thus the number of increments required to give the specified precision shall 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, shall be taken from each unit as described in clause 7, whichever method is relevant. The increments from each unit shall 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.

3.3.4 *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. (See ISO 1213/2.) The same number of increments is taken from every unit that is sampled. The number of units to be sampled shall be decided and the total number of increments required shall 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 shall be chosen at random; for example, if the sample is to be taken on only two days a week, the days for sampling shall be selected at random each week (see 7.3).

The necessary number of increments, each of the appropriate mass, shall be taken from each selected unit as described in clause 7. 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 were regularly received by ship or by barge.

3.3.5 *Sampling of breeze and small coke*

It is generally assumed that small coke, produced as the result of breakage of larger coke, should be sampled during the transfer operation from the point of generation and screening. In such cases samples may be taken from a falling stream, a moving belt or a stopped belt. It is difficult to extract representative samples from static consignments such as road or rail wagons or ground stockpiles; in such cases the above sampling systems (see 3.3.1 and 3.3.2) are not recommended.

4 STANDARD OF PRECISION ADOPTED

4.1 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 5.3.

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 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.

4.2 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.

4.3 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 it 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.

4.4 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 to 5	3,5
5 to 10	4,0
10 to 20	4,5
20 to 40	5,0
40 to 50	4,5
50 to 60	4,0
60 to 70	3,5
70 to 80	3,0
80 to 90	2,5
90 to 95	1,5
95 to 100	1,0

4.5 Warning

In 4.1 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 5.3. 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 shall never be less than 12.

5 NUMBER OF INCREMENTS

5.1 General

The numbers of increments given in 5.4 to 5.7 are those required to attain the standard precision.

If a precision other than the standard is required, see 5.3 and 4.5.

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 5.4 to 5.7 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 t.

5.2 Sampling large consignments

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

a) preferably, the consignment should be divided into a number of portions, each of 1 000 t 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 :

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

5.3 Precision different from the standard

For a precision different from the standard (for example ± 1,5 % instead of ± 1 %), the initial number of increments shall 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 such cases use the following factor 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

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

5.4 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 coke	50	75[1)]	100	150
Breeze or small coke of nominal top size 20 mm or less	20	—	—	—

1) Where the number of wagons to be sampled is less than 15, five 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 shall not exceed about 70 kg, otherwise the coke would be exposed for such a time that appreciable loss of moisture would occur.

5.5 Ash sample

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

5.6 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 mm	Number of increments for coke in			
	stream	wagons	ships	stockpiles
⩽ 20	20	—	—	—
21 to 50	50	75	100	150
51 to 100	20	30	40	60
101 to 200	10	15	20	30

5.7 Common sample

When a common sample is required, it is essential that the number of increments taken is 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 mm	Precision as fraction of true mean size
⩽ 50	1/10
51 to 100	1/15
101 to 200	1/20

6 MINIMUM MASS OF INCREMENT

6.1 General

The masses of the increments given in table 4 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. It is essential that the number of increments taken **is not reduced** below that necessary for the precision required.

6.2 Mass

The minimum mass of increment is given in table 4.

TABLE 4 — **Minimum mass of increment**

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

1) 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, it is essential that the following conditions be satisified :

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

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

7 COLLECTION OF INCREMENTS

7.1 General

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.

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

It is important that the interval of time between successive increments does 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 shall not be filled to overflowing.

The coke shall 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 shall be ignored.

Any sample whose moisture is to be determined shall 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.

7.2 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 shall 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.

7.3 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. Place the discs 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 one, two or three 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