BRITISH STANDARD

BS ISO 13909-7:2001

*Incorporating corrigendum April 2008*

# Hard coal and coke — Mechanical sampling —

## Part 7: Methods for determining the precision of sampling, sample preparation and testing

ICS 73.040; 75.160.10

NO COPYING WITHOUT BSI PERMISSION EXCEPT AS PERMITTED BY COPYRIGHT LAW

Document Sharing Hub
British Standards

Full document details: BS ISO 13909-7:2001

# National foreword

This British Standard is the UK implementation of ISO 13909-7:2001.

Together with BS ISO 18283:2006, BS ISO 13909-1:2001, BS ISO 13909-2:2001, BS ISO 13909-3:2001, BS ISO 13909-4:2001, BS ISO 13909-5:2001, BS ISO 13909-6:2001 and BS ISO 13909-8:2001, it supersedes BS 1017-1:1989 and BS 1017-2:1994 which have been withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/16, Solid mineral fuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

**Compliance with a British Standard cannot confer immunity from legal obligations.**

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 23 January 2002

© BSI 2008

ISBN 978 0 580 61412 5

**Amendments/corrigenda issued since publication**

| Date | Comments |
|---|---|
| 30 April 2008 | Supersession details in the national foreword updated |
| | |
| | |
| | |

Documents
Document Sharing Hub

Full document details: BS ISO 13909-7:2001

INTERNATIONAL STANDARD

# ISO 13909-7

First edition
2001-12-15

# Hard coal and coke — Mechanical sampling —

## Part 7: Methods for determining the precision of sampling, sample preparation and testing

*Houille et coke — Échantillonnage mécanique —*

*Partie 7: Méthodes pour la détermination de la fidélité de l'échantillonnage, de la préparation de l'échantillon et de l'essai*

Reference number
ISO 13909-7:2001(E)

Documents
Document Sharing Hub

Full document details: BS ISO 13909-7:2001

Documents
Document Sharing Hub

Full document details: BS ISO 13909-7:2001

## Contents

Page


**1** Scope ..... **1**
**2** Normative references ..... **1**
**3** Terms and definitions ..... **1**
**4** General ..... **2**
**5** Equations relating to factors affecting precision ..... **3**
**6** Estimation of primary increment variance ..... **4**
**7** Methods for estimating precision ..... **6**
**8** Calculation of precision ..... **10**
**9** Methods of checking sample preparation and testing errors ..... **12**


## Annexes


**A** Variogram method for determining variance ..... **25**
**B** Grubbs' estimators method for determining sampling precision ..... **32**
Bibliography ..... **40**


Documents
Document Sharing Hub



Full document details: BS ISO 13909-7:2001

## Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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

Attention is drawn to the possibility that some of the elements of this part of ISO 13909 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13909-7 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

ISO 13909 cancels and replaces ISO 9411-1:1994, *Solid mineral fuels — Mechanical sampling from moving streams — Part 1: Coal* and ISO 9411-2:1993, *Solid mineral fuels — Mechanical sampling from moving streams — Part 2: Coke*, of which it constitutes a technical revision. It also supersedes the methods of mechanical sampling of coal and coke given in ISO 1988:1975, *Hard coal — Sampling* and ISO 2309:1980, *Coke — Sampling*.

ISO 13909 consists of the following parts, under the general title *Hard coal and coke — Mechanical sampling*:

— *Part 1: General introduction*

— *Part 2: Coal — Sampling from moving streams*

— *Part 3: Coal — Sampling from stationary lots*

— *Part 4: Coal — Preparation of test samples*

— *Part 5: Coke — Sampling from moving streams*

— *Part 6: Coke — Preparation of test samples*

— *Part 7: Methods for determining the precision of sampling, sample preparation and testing*

— *Part 8: Methods of testing for bias*

Annexes A and B of this part of ISO 13909 are for information only.

Documents
Document Sharing Hub



Full document details: BS ISO 13909-7:2001

## Introduction

Two different situations are considered when a measure of precision is required. In the first, an estimate is made of the precision that can be expected from an existing sampling scheme and, if this is different from that desired, adjustments are made to correct it. In the second, the precision that is achieved on a particular lot is estimated from the experimental results actually obtained using a specifically designed sampling scheme.

The equations developed in this part of ISO 13909 are based on the assumption that the quality of the fuel varies in a random manner throughout the mass being sampled and that the observations will follow a normal distribution. Neither of these assumptions is strictly correct. Although the assumption that observations will follow a normal distribution is not strictly correct for some fuel parameters, this deviation from assumed conditions will not materially affect the validity of the formulae developed for precision checking since the statistics used are not very sensitive to non-normality. Strictly speaking, however, confidence limits will not always be symmetrically distributed about the mean. For most practical uses of precision, however, the errors are not significant.

NOTE In the text, the term “fuel” is used where both coal and coke would be applicable in the context, and either “coal” or “coke” where that term is exclusively applicable.

Documents
Document Sharing Hub



Full document details: BS ISO 13909-7:2001

*blank*

Documents
Document Sharing Hub

# Hard coal and coke — Mechanical sampling —

## Part 7:
## Methods for determining the precision of sampling, sample preparation and testing

## 1 Scope

In this part of ISO 13909, equations are developed which link the variables which contribute to overall sampling precision. Methods are described for estimating overall precision and for deriving values for primary increment variance which can be used to modify the sampling scheme to change the precision. Methods for checking the variance of sample preparation and testing are also described.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13909. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13909 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 13909-1:2001, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*.

ISO 13909-2:2001, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*.

ISO 13909-3:2001, *Hard coal and coke — Mechanical sampling — Part 3: Coal — Sampling from stationary lots*.

ISO 13909-4:2001, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*.

ISO 13909-5:2001, *Hard coal and coke — Mechanical sampling — Part 5: Coke — Sampling from moving streams*.

ISO 13909-6:2001, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*.

ISO 13909-8:2001, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*.

## 3 Terms and definitions

For the purposes of this part of ISO 13909, the terms and definitions given in ISO 13909-1 apply.

Documents
Document Sharing Hub



Full document details: BS ISO 13909-7:2001

## 4 General

When designing a sampling scheme in order to meet a required precision of results, equations are necessary that link certain fuel and sampling characteristics to that precision. The main factors to be considered are the variability of primary increments, preparation and testing errors, the number of increments and samples taken to represent the lot, and the mass of the samples. These equations, for both continuous and intermittent sampling, are derived in clause 5. Methods for estimating the parameters used in those equations are given in clause 6.

Once a sampling system has been designed and installed, the precision which is being achieved on a routine basis should be checked. An estimate of the precision can be obtained from the primary increment variance, $V_{\mathrm{I}}$, the numbers of increments, $n$, and sub-lots, $u$, (see clause 5) and the preparation and testing variance, $V_{\mathrm{PT}}$.

Sampling variance is a function of product variability, so the same number of increments, sub-lots, and preparation and testing errors will yield different precision with fuels that exhibit different product variability.

Depending on the extent to which serial correlation exists and which method of estimating primary increment variance is used, such an estimate could represent a considerable overestimate of the numerical value of the precision (i.e. indicate that it is worse than is really the case). In addition, in order for the results to be meaningful, large numbers of increments (in duplicate) would need to be prepared and analysed for the estimation of $V_{\mathrm{I}}$ and $V_{\mathrm{PT}}$.

Quality variations obtained in the form of primary increment variances on existing systems are not absolute and therefore designers should exercise caution when using such results in a different situation. The estimated value of the primary increment variance, $V_{\mathrm{I}}$, should be derived experimentally for each fuel and at each sampling location.

Whenever a sampling scheme is used for determining increment variance, the operating conditions should be as similar as possible to the conditions known, or anticipated, to prevail during the sampling for which the increment variance is needed, whether it be carried out by the same or by a different sampling system.

An estimate of the precision actually achieved can be obtained by taking the sample in a number of parts and comparing the results obtained from these parts. There are several methods of doing this, depending on

a) the purpose of the test, and

b) the practical limitations imposed by the available sampling procedures and equipment.

Where a sampling system is in existence, the purpose of the test is to check that the scheme is in fact achieving the desired precision (see clause 7). If it is not, it may need to be modified and rechecked until it meets the precision required. In order to do this, a special check scheme will have to be devised which may be different from the regular scheme but which measures the precision of the regular scheme.

For regular sampling schemes, the most rigorous approach is that of duplicate sampling of sub-lots. In many existing mechanical sampling systems, however, the capacity of individual components, and the interval between increments in the regular scheme is insufficient to allow the taking of extra increments. In such cases, duplicate samples can be constituted from the normal number of increments, and the result adjusted for the smaller number of increments in each sample (see 7.3).

The need may arise to sample a particular lot and to know the precision of the result obtained (see clause 8). Once again, a special check scheme needs to be devised, but in this case it is the precision achieved by that scheme on that lot which is required. For the measurement of the precision achieved for a particular lot, replicate sampling is the best method.

Methods for detailed checking of preparation and testing errors are given in clause 9. The results may also be used to provide data for the equations used in clause 5.



Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2001

## 5 Equations relating to factors affecting precision

### 5.1 General

Precision is a measure of the closeness of agreement between the results obtained by repeating a measurement procedure several times under specified conditions, and is a characteristic of the method used. The smaller the random errors of a method, the more precise is the method. A commonly accepted index of precision is two times the sample estimate of the population standard deviation, and this index of precision is used throughout this part of ISO 13909.

If a large number of replicate samples, $j$, are taken from a sub-lot of fuel and are prepared and analysed separately, the estimated precision, $P$ , of a single observation is given by equation (1):

$$P = 2s = 2\sqrt{V_{\text{SPT}}} \tag{1}$$

where

$s$ is the sample estimate of the population standard deviation;

$V_{\text{SPT}}$ is the total variance.

The total variance, $V_{\text{SPT}}$, in equation (1) is a function of the primary increment variance, the number of increments, and the errors associated with sample preparation and testing.

NOTE The components of primary increment variance are the variance of sample extraction and the variance contributed by product variability. The variance contributed by product variability is generally, but not always, the largest source of variance in sampling.

For a single sample, this relationship is expressed by equation (2):

$$V_{\text{SPT}} = \frac{V_{\text{I}}}{n} + V_{\text{PT}} \tag{2}$$

where

$V_{\text{I}}$ is the primary increment variance;

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

$n$ is the number of primary increments in the sample.

### 5.2 Continuous sampling

Where the result is the arithmetic mean of a number of samples, resulting from dividing the lot into a series of sub-lots and taking a sample from each, equation (2) becomes:

$$V_{\text{SPT}} = \frac{V_{\text{I}}}{mn} + \frac{V_{\text{PT}}}{m} \tag{3}$$

where $m$ is the number of sample results used to obtain the mean.

Since a sample is equivalent to one member of a set of replicate samples, by combining equations 1 and 3 it can be shown that:

$$P = 2\sqrt{\frac{V_{\text{I}}}{mn} + \frac{V_{\text{PT}}}{m}} \tag{4}$$

AVA Documents
Document Sharing Hub



Full document details: BS ISO 13909-7:2001

Equation (4) gives an estimate of the precision that can be expected to be achieved when a given sampling scheme is used for testing a given fuel, the variability of which is known or can somehow be estimated. In addition, equation (4) enables the designer of the sampling scheme to determine, for the desired precision and with fuel of known or estimated variability, the combination of numbers of increments and samples, respectively, which will be most favourable considering the relative merits of the sampling equipment and the laboratory facilities in question. For the latter purpose, however, it is more convenient to use either or both equations (5) and (6), both of which have been derived by rearranging equation (4).

$$n = \frac{4V_{\mathrm{I}}}{mP^2 - 4V_{\mathrm{PT}}} \tag{5}$$

$$m = \frac{4\left(V_{\mathrm{I}} + nV_{\mathrm{PT}}\right)}{nP^2} \tag{6}$$

NOTE Results obtained from solid mineral fuels flowing in a stream will frequently display serial correlation, i.e. immediately adjoining fuel tends to be of similar composition, while fuel further apart tends to be of dissimilar composition. When this is so, the estimates of precision of the result of a single sample based on primary increment variance and the variance of preparation and testing would indicate precision that is worse, i.e. numerically higher, than the precision actually achieved. The effect of serial correlation can be taken into account using the variographic method of determining variance given in informative annex A.

### 5.3 Intermittent sampling

Whilst the value used for the primary increment variance is assumed to be consistent for all the sub-lots in a lot, there may be variations between the means of sub-lots. Providing all sub-lots are sampled and tested, this is not a source of additional variance. However, if only some sub-lots are sampled and tested, (i.e. intermittent sampling) then a term to correct for sub-lot variance should be included in equation (3) and the equations derived from it, as follows:

$$V_{\mathrm{SPT}} = \frac{V_{\mathrm{I}}}{un} + \frac{V_{\mathrm{PT}}}{u} + \left(1 - \frac{u}{m}\right) V_{\mathrm{m}} \tag{7}$$

where

$m$ is the total number of sub-lots;

$u$ is the number of sub-lots sampled;

$V_{\mathrm{m}}$ is the sub-lot variance.

The term $(1 - u/m)\, V_{\mathrm{m}}$ compensates for the fact that, as the proportion of sub-lots sampled and tested increases, the influence of sub-lot variance decreases, until it disappears when $u = m$.

The equivalent to equation (4) can be derived by combining equations (1) and (7) from which the equations equivalent to equations (5) and (6) can be obtained by rearrangement.

## 6 Estimation of primary increment variance

### 6.1 Direct determination of individual primary increments

The direct estimation of primary increment variance can be accomplished with a duplicate sampling scheme comprised of several hierarchical levels which allows both the overall variance and the variance of preparation and testing to be estimated. The estimated variance of primary increments can then be obtained by subtraction of the variance of preparation and testing from the estimated overall variance. A number of primary increments is taken systematically and either divided into two parts or prepared so that duplicate samples are taken at the first division stage. Each part is prepared and tested for the quality characteristic of interest, using the same methods that are expected to be used in routine operations. The mean of the two results and the difference between the two results is calculated for each pair.

AVA Documents
Document Sharing Hub



Full document details: BS ISO 13909-7:2001

It is recommended that at least 50 increments be taken, spread if possible over an entire lot or even over several lots of the same type of fuel.

The procedure is as follows:

a) Calculate the preparation and testing variance, $V_{\mathrm{PT}}$.

$$V_{\mathrm{PT}} = \frac{\sum d^2}{2n_{\mathrm{P}}} \tag{8}$$

where

$d$ is the difference between pair members;

$n_{\mathrm{P}}$ is the number of pairs.

b) Calculate the primary increment variance, $V_{\mathrm{I}}$.

$$V_{\mathrm{I}} = \frac{\sum x^2 - \frac{1}{n_{\mathrm{P}}}\left(\sum x\right)^2}{(n_{\mathrm{P}} - 1)} - \frac{V_{\mathrm{PT}}}{2} \tag{9}$$

where $x$ is the mean of the two measurements for each increment.

An alternative method for estimating primary increment variance, $V_{\mathrm{I}}$, is as follows:

$$V_{\mathrm{I}} = \frac{\sum D^2}{2h} - \frac{V_{\mathrm{PT}}}{2} \tag{10}$$

where

$D$ is the difference between the means of successive pairs;

$h$ is the number of successive pairs.

This method avoids the overestimation of sampling variance when there is serial correlation (see Note in 5.2), but can only be used if the primary-increment sampling interval at which the increments are taken is more than or approximately equal to the primary-increment sampling interval used when the scheme is implemented in routine sampling operations.

The most rigorous treatment of serial correlation is to use the variographic method given in annex A. This takes into account both serial correlation and sampling interval effects, thereby avoiding overestimation of sampling variance and number of primary increments due to these factors.

## 6.2 Determination using the estimate of precision

The primary increment variance can be calculated from the estimate of precision obtained either using the method of duplicate sampling given in 7.2 or replicate sampling given in clause 8 according to equation (11) which is derived by rearranging the terms of equation (4).

$$V_{\mathrm{I}} = \frac{mnP^2}{4} - nV_{\mathrm{PT}} \tag{11}$$

This value can then be used to adjust the sampling scheme if necessary.

Documents
Document Sharing Hub



Full document details: BS ISO 13909-7:2001

# 7 Methods for estimating precision

## 7.1 General

For all the methods given in this clause, the following symbols and definitions apply:

$n_0$ is the number of increments in a sub-lot for the regular scheme;

$m_0$ is the number of sub-lots in a lot for the regular scheme;

$P_0$ is the desired precision for the regular scheme;

$P_W$ is the worst (highest absolute value) precision to be permitted.

In all cases, the same methods of sample preparation shall be used as for the regular scheme.

## 7.2 Duplicate sampling with twice the number of increments

Twice the normal number of increments ($2n_0$) are taken from each sub-lot and combined as duplicate samples (see Figure 1), each containing $n_0$ increments. This process is repeated, if necessary, over several lots of the same fuel, until at least ten pairs of duplicate samples have been taken.

A parameter of the fuel is chosen to be analysed, e.g. ash (dry basis) for coal, or Micum 40 Index for coke. The standard deviation within duplicate samples for the test parameter is then calculated using equation (12):

$$s = \sqrt{\frac{\sum d^2}{2n_p}} \tag{12}$$

where

$d$ is the difference between duplicates;

$n_p$ is the number of pairs of duplicates being examined.

Typical results for coal ash are given in Table 1.

The number of pairs, $n_p$ is 10. The variance of the ash is therefore:

$$s^2 = \frac{\sum d^2}{2n_p} = \frac{2{,}78}{20} = 0{,}139$$

and the standard deviation is

$$s = \sqrt{0{,}139\,0} = 0{,}373$$



Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2001

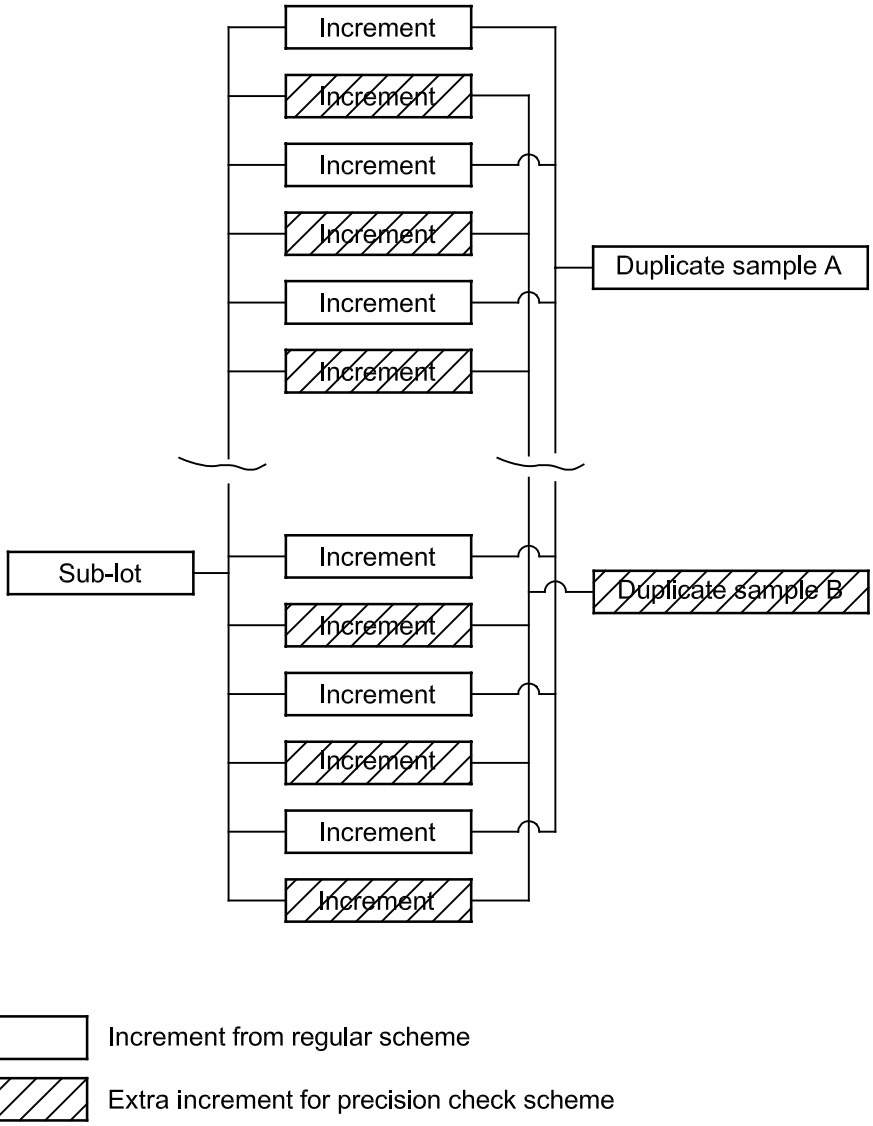


**Figure 1 — Example of a plan of duplicate sampling**

The precision of the result for a single sub-lot is therefore:

$$P = 2s$$
$$= 2\,(0{,}373) = 0{,}75\ \%$$

The precision achieved for the mean ash of a normal lot sampled as $m$ sub-lots is given by $\frac{2s}{\sqrt{m}}$. For example, if $m = 10$, then:

$$P = \frac{2\,(0{,}373)}{\sqrt{10}} = 0{,}235\,9\ \%$$

These values of $P$ have been calculated using point estimates for the standard deviation, and represent the best estimate for precision.

Documents
Document Sharing Hub



Full document details: BS ISO 13909-7:2001