

Nederlandse norm

NEN-ISO 13909-7

(en)

Steenkool en cokes - Mechanische monsterneming
- Deel 7: Methoden voor de bepaling van de
precisie van monsterneming, monsterpreparatie
en analyse (ISO 13909-7:2016,IDT)

Hard coal and coke - Mechanical sampling - Part 7:
Methods for determining the precision of
sampling, sample preparation and testing (ISO
13909-7:2016,IDT)

Vervangt NEN-ISO 13909-7:2002

ICS 73.040; 75.160.10
september 2016

Als Nederlandse norm is aanvaard:

- ISO 13909-7:2016, IDT

Normcommissie 310027 "Vaste Minerale Brandstoffen"



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Preview

**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*



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ISO 13909-7:2016(E)**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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

This second edition cancels and replaces the first edition (ISO 13909-7:2001), which has been technically revised.

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*

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 formulae 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.

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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, formulae are developed which link the variables that 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

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

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

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

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

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

3 Terms and definitions

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

4 General

When designing a sampling scheme in order to meet a required precision of results, formulae 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 formulae are derived in [Clause 5](#). Methods for estimating the parameters used in those formulae 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_I , the numbers of increments, n , and sub-lots, m , (see [Clause 5](#)) and the preparation and testing variance, V_{PT} . The preparation component of V_{PT} is made up of on-line sample processing and off-line sample preparation.

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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_I and V_{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_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 should 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 formulae used in [Clause 5](#).

5 Formulae 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 the method is. 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 [Formula \(1\)](#):

$$P = 2s = 2\sqrt{V_{\text{SPT}}} \quad (1)$$

where

s is the sample estimate of the population standard deviation;

V_{SPT} is the total variance.

The total variance, V_{SPT} , in [Formula \(1\)](#) 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 [Formula \(2\)](#):

$$V_{\text{SPT}} = \frac{V_1}{n} + V_{\text{PT}} \quad (2)$$

where

V_1 is the primary increment variance;

V_{PT} is the preparation and testing variance;

n is the number of primary increments in the sample.

5.2 Sampling

Where the test 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, [Formula \(2\)](#) becomes:

$$V_{\text{SPT}} = \frac{V_1}{mn} + \frac{V_{\text{PT}}}{m} \quad (3)$$

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 [Formulae \(1\)](#) and [\(3\)](#), it can be shown that:

$$P = 2\sqrt{\frac{V_1}{mn} + \frac{V_{\text{PT}}}{m}} \quad (4)$$

[Formula \(4\)](#) 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, [Formula \(4\)](#) 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,

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it is more convenient to use either or both [Formulae \(5\)](#) and [\(6\)](#), both of which have been derived by rearranging [Formula \(4\)](#).

$$n = \frac{4V_I}{mP^2 - 4V_{PT}} \quad (5)$$

$$m = \frac{4(V_I + nV_{PT})}{nP^2} \quad (6)$$

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](#).

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 are calculated for each pair.

It is recommended that at least 30 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_{PT} .

$$V_{PT} = \frac{\sum d^2}{2n_p} \quad (7)$$

where

d is the difference between pair members;

n_p is the number of pairs.

- b) Calculate the primary increment variance, V_I .

$$V_I = \frac{\sum x^2 - \frac{1}{n_p} (\sum x)^2}{(n_p - 1)} - \frac{V_{PT}}{2} \quad (8)$$

where

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

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