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Houille et coke -- Échantillonnage mécanique -- Partie 8: Méthodes de détection du biais

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Hard coal and coke — Mechanical sampling —

Part 8: Methods of testing for bias

Houille et coke — Échantillonnage mécanique —

Partie 8: Méthodes de détection du biais

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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 13909-8 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*

Annex A of this part of ISO 13909 is for information only.

Introduction

It is not possible to lay down a standard method for field work by which a sampling procedure can be tested for bias because details of the procedure will inevitably be affected by local conditions. However, certain principles can be specified which should be adhered to whenever possible and these are discussed in this part of ISO 13909.

Testing for bias can be a tedious and expensive process, especially if testing of the primary increment sampler is included. All bias tests therefore include a thorough pre-test inspection, with appropriate action taken regarding any system deficiencies likely to cause bias.

Hard coal and coke — Mechanical sampling —

Part 8:
Methods of testing for bias

1 Scope

This part of ISO 13909 sets out principles and procedures for testing the bias of test samples of hard coals or cokes, taken in accordance with other parts of ISO 13909. The use of univariate statistical methods only is addressed.

The user is cautioned that the chance of falsely concluding that there is a bias, when no bias exists in any one of several variables measured on the same set of samples, is substantially greater than for a single variable. While several variables may be measured, the single variable on which the outcome of the test will be governed shall be designated in advance.

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 only one is applicable.

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 13909-4:2001, *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 13909-7:2001, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*.

3 Terms and definitions

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

4 Principles

4.1 General

Testing for bias is not always done for a single purpose. The objectives of testing for bias may be for assessing conformity with contractual requirements, such as guarantees or purchase and acceptance specifications, or for diagnostic purposes or both, and may or may not involve multiple test parameters. This part of ISO 13909 uses univariate statistics for testing the performance of the system with respect to a single variable.

It is not possible for any scheme of sampling, or sample preparation and analysis, to be free of errors of measurement. For this reason, no statistical test can establish that there is no bias, but only that there is not likely to be a bias of more than a certain magnitude.

The testing of a sampling system for bias is based on taking a series of pairs of samples of essentially the same fuel; one member of each pair being sampled by the system or component under test, the other member being obtained by a reference method. For each pair, the difference between the analytical results is determined. The series of differences between the analytical results thus obtained are subjected to statistical analysis.

The procedure requires the sensitivity of the statistical test of significance to be such that the minimum bias that can be detected is less than or equal to the maximum tolerable bias, B. Therefore, B shall be established before the test begins.

NOTE In the absence of other information, a value of $B =$ 0,20 % to 0,30 % for ash or moisture may be appropriate, subject to commercial considerations.

The sensitivity of the statistical test used is dependent on the number of pairs compared and the variability of the differences between them.

The statistical analysis to which results will be subjected assumes three conditions:

— a normal distribution of the variable;

— independence of the errors of measurement;

— statistical homogeneity of the data.

The closeness with which these ideal conditions are achieved, in practice, governs the validity of the statistical analysis. The execution of the test, including sample reduction, division and laboratory analysis, shall be organized so as to ensure that deviations from these ideals do not invalidate the statistical analysis.

The statistical test used to make the final judgement is the t-test. A hypothesis is made that the observed mean of the differences between the two methods is drawn from a population whose mean is B. If the test shows that the observed difference is significantly less than B, then the sampler or component is declared free of bias.

In basing decisions on the outcomes of statistical tests, there is always the risk of making either one or the other of two types of error. If the hypothesis is rejected when it is true, e.g. a bias is not declared even though a bias really does exist, then an error of the first kind (Type I) has been made. On the other hand, if the hypothesis is accepted when it is false, e.g. a bias is declared even though a bias really does not exist, then an error of the second kind (Type II) has been made.

In any particular test, the probability of a Type I error can be arbitrarily set as a matter of discretion and the risk kept as small as desired. For a specific test, the probability of an error of Type II can only be quantified in relation to some other value than the original hypothesis. In this method the value of zero is used. The risk of a Type II error can be decreased at a fixed probability of an error of Type I only by increasing the number of observations. However, since the sample estimate of the population standard deviation must be used in the calculations, the risk of a Type II error is an estimated value. The final statistical test is not carried out until sufficient pairs of observations have been taken to limit both a Type I error in relation to B, and the estimated Type II error in relation to zero, to 5 %. Thus, if the observed value of the mean difference (the sample estimate of the population mean) is not significantly less than B, it shall also be significantly greater than zero.

The number of paired samples suitable for a test of the overall system relative to the maximum tolerable bias, B, may be insufficient for testing a given component. In such circumstances, if the performance of a given component is of critical importance, a separate test shall be considered. For components other than the primary increment sampler, such tests can usually be implemented with minimum disruption of normal operations and at a lesser cost than for a test for overall system bias (see 7.2 and 7.3).

If obtaining the number of pairs required is found to be impracticable, changes will need to be made to reduce the within-set variance. Investigate what improvements can be made in the closeness of members of pairs and/or what reduction can be achieved in the preparation and testing errors. If such improvements or reductions are not possible, give consideration to increasing the number of increments in the samples, taking into account the practical problems associated with taking increments and the relative costs and errors involved in sampling, sample preparation and testing.

If the required number of pairs of samples is still excessive, the maximum tolerable bias, B, may be reviewed.

When samples of more than one increment are compared, it is necessary that the reference samples and the samples from the system under test be constituted on the same basis, i.e. for a time-basis system, the individual increment masses shall be proportional to the flow rate and, for mass-basis systems, the individual increment mass shall be uniform (see ISO 13909-2 and ISO 13909-5).

4.2 Selection of sample pairs

4.2.1 Composition of sample pairs

The members of each pair of samples can each comprise one or more increments. Individual increments can be compared or samples compounded from increments taken by the two methods. The test shall be structured so that the expected mean of the differences of the result would be zero if no systematic error is present in the system or component under test.

4.2.2 Paired-increment samples

Paired-increment experimental design is the comparing of individual primary increments after being processed by the system, with the reference samples collected from the stopped belt.

For a given parameter, the variance of the differences between paired samples will normally be smaller than the variance of either of the two series of samples, taken by the system or component under test and the reference method respectively, except for fuels that are very homogeneous. For this reason, if the increments taken by the two methods are taken in close proximity to each other in the fuel stream (without overlapping), the variance of the differences between them will be minimized and the sensitivity of the test improved.

4.2.3 Paired-batch samples

It is often not practicable to obtain single increment samples from the system. Increments taken by the system can be compounded as samples, and compared with samples compounded from increments taken over the same period using the reference method. It is not necessary that the two samples have the same number of increments or that they are of similar mass. In the extreme, a single stopped-belt reference increment could be used as the reference sample.

4.3 Location of sampling points

For a test of the overall system, the reference sample shall be taken from the primary fuel stream using the stopped-belt reference method (see clause 7). The system sample shall be the final sample.

The primary sampler shall be tested by examining the differences between members of each pair consisting of the samples taken by the primary sampler and the reference method.

NOTE 1 For high volume, high capacity, conveyor systems, a test of a primary increment sampler as an individual component will require the collection and processing of increments of large mass in a short time interval. Before such a test is undertaken, therefore, it is necessary to consider carefully whether such a test can be justified.

With the exception of the primary increment sampler, when testing individual components and subsystems, the test compares the differences between the sample feed stream and the discharge stream of that component or subsystem.

NOTE 2 In some cases, the quality of the feed stream can only be obtained indirectly, for example, by calculation from the results of the divided sample and the corresponding reject-stream sample, weighted according to the division ratio.

For all crushing equipment, differences between samples taken from the fuel both before and after the crusher are used.

For subsystems and sample dividers, pairs obtained by one of the following methods shall be tested:

a) by taking samples from the feed stream and from the sample discharge stream;

b) by taking samples from the sample discharge stream and the reject stream;

c) by collecting the entire sample discharge stream and the entire reject stream.

When using either method a) or method b), great care shall be exercised to obtain unbiased samples; in the case of method a) being used, care shall be taken to minimize disturbance of the feed flow, as such disturbances may introduce bias or distort normal operating conditions.

5 Outline of procedure

The order of operations is as follows:

a) carry out a pre-test inspection (see clause 6);

b) for the overall system, determine where the stopped-belt reference sample will be collected (see 7.1); for diagnostic testing of the system components, see 7.2 or 7.3;

c) determine the variable for test (see clause 8);

d) choose the fuel to be used for the test (see clause 9);

e) decide on the maximum tolerable bias, B (see 4.1);

f) decide on the composition of the sample pairs, i.e. whether to compare sample pairs of one increment or more than one increment;

g) proceed with collection of samples and carry out the tests according to clauses 10 and 11.

6 Pre-test inspection

The primary sources of information regarding compliance with the sampling standard are the equipment specifications and drawings.

A thorough examination of the sampling system and a review of its component specification shall be made.

The party performing the test shall, however, verify performance by field measurements and observations. The operation of the sampling system shall be observed both with fuel flowing and with no fuel.

Pre-test inspections of all operations and equipment, both static and under load, should be carried out by persons experienced in the sampling of segregated, heterogeneous, lumpy bulk materials. It is recommended that operation under normal conditions be observed for an entire lot.

Do not execute a test for bias until all conditions known to cause bias are corrected, unless it is necessary to establish the performance of a system or component as it stands. In the latter case, the pre-test inspection provides essential documentation of what the conditions were at the time of the test.

