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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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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

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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 13909-8:2001), 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

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 ought to 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. All bias tests therefore include a thorough pretest 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.

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

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

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

ISO 21398, *Hard coal and coke — Guidance to the inspection of mechanical sampling systems*

3 Terms and definitions

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

4 Principles

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 sensitivity of the statistical test is dependent on the number of pairs compared, the variability of the differences between members of the pairs, and the number of parameters used for the test.

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

— a multivariate normal distribution of the variables;

— independence of the errors of measurement for the individual parameters;

— homogeneity of the data.

5 Pretest 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 (see ISO 21398).

The party performing the test shall, however, verify performance by field measurements and observations. The operation of the sampling system shall be observed both dynamically with fuel flowing and statically with no fuel flowing. Some components will need to be actuated in static mode.

Pretest inspections of all operations and equipment, both static and under load, should be carried out by persons experienced in the sampling of segregated, heterogeneous, and 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 pretest inspection provides essential documentation of what the conditions were at the time of the test.

6 Reference methods

To test overall system bias, the use of a reference method which is known to be intrinsically unbiased is required. The preferred method is the stopped-belt method, i.e. the collection of increments from a complete cross-section of the fuel on the conveyor belt by stopping the belt at intervals. When properly collected from the primary fuel stream, the stopped-belt increment can be considered as a reference increment.

NOTE If it is not possible to collect stopped-belt increments, other reference methods can be used but, in these cases, an apparent absence of a lack of bias relative to the reference method may not be conclusive and the use of such methods may compromise the validity and authority of the findings.

With collection of stopped-belt increments, some disruption of normal operations can occur, and therefore, the plan of execution may need to be coordinated with the normal operations and organized to minimize such disruption. It should be recognized that the conveyor system involved may be used for only a few hours per day for normal operations and cannot be operated solely for bias tests unless the fuel can be diverted to another discharge point. This can extend the time necessary for completion of the field work and require special arrangements for supplying fuel to the system for testing.

Stopped-belt increments shall be taken with a sampling frame (see Figure 1), or equivalent, from a complete cross-section of the solid mineral fuel on the belt at a fixed position, for a length along the belt which is at least three times the nominal top size of the fuel.

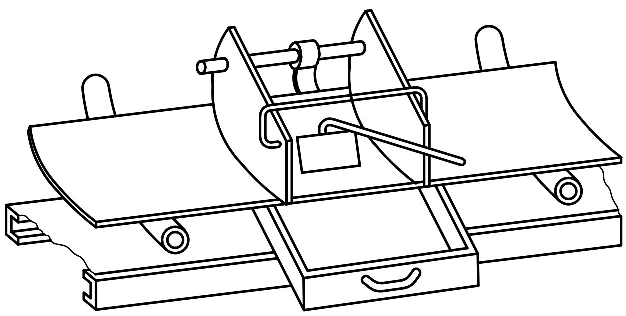

Figure 1 — Sampling frame

The sampling frame (or equivalent) shall be placed on the stationary belt at the predetermined position so that the separator plates at each end are in contact with the belt across its full width. All particles lying inside the sampling frame end plates shall be swept into the sampling container.

Particles obstructing the insertion of the end plate on the left-hand side shall be pushed into the increment, while those obstructing the insertion of the end plate on the right-hand side shall be pushed out of the increment or vice-versa. Whichever practice is used initially, this practice shall be implemented throughout the test.

Care should be taken to minimize the risk of bias being introduced in the course of preparation of the increments and samples and all sample division equipment and procedures should be checked for bias with respect to relevant test parameters.

It is recommended that all increments/samples be weighed immediately after collection. Pay close attention to minimizing unintended mass losses.

Report all observed mass losses.

7 Test design

7.1 Choice of test parameters

The standard test for general purposes shall include moisture and ash (ash on a dry basis). Use of these two parameters will generally suffice. Other variables can be included if desired. However, given a fixed number of sample pairs, the test often becomes less sensitive to detection of a bias as additional variables are included.

Bias in ash on a dry basis is most commonly caused by errors in size distribution. Bias in moisture content may be caused by a wide variety of factors, including, but not limited to, errors in size distribution, moisture losses associated with crushers, excessive ventilation within the sampling system, less than the closest possible coupling between system components, excessive retention time in the system, or any combination of these.

Direct tests on particle size distribution are often necessary for coke. It is recommended that tests for size distribution be conducted as a separate test and that size distribution parameters not be included in the standard test for analytical parameters.

7.2 Number of paired samples

The minimum number of paired samples for the test shall be 30. However, if necessary, more sets may be collected if the variance of the quality of the fuel is suspected to influence the detection of bias. On the other hand, if use of a smaller number of paired samples results in a detection of bias, no additional samples need be collected. After completing the statistical calculations, the resulting confidence region can be evaluated. If no bias is detected, yet the confidence region covers levels of bias that are of commercial concern, more paired samples can be collected for coal originating from this same source in order to reduce the size of the confidence region.

7.3 Selection of sample pairs

7.3.1 Composition of sample pairs

The members of each pair of samples can each be comprised of portions of one or more increments. Individual increments can be paired or samples of compounded increments can be paired. 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 under test.

7.3.2 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.

NOTE 1 A final system sample consisting only of fuel collected from a single primary increment may be of insufficient mass to provide an analytical result following the requirements specified in ISO 13909-4 and ISO 13909-6 for preparation and analysis by the methods used routinely during regular operations.

NOTE 2 In causing the mechanical system to separately collect and process individual primary increments for a test for bias, the system sample may lose more moisture than while the system is operating as designed.

7.3.3 Paired-batch samples

It is often not practical to obtain single increment samples from the system. Increments taken by the system can be compounded as samples and paired with samples compounded from increments taken over the same period using the reference method. It is not necessary that the two sample members, reference and system, have the same number of increments or be of similar mass. Single stopped-belt reference increments are often used as the reference sample and paired with compounded system sample increments. In collecting paired-batch samples, the timing at which stopped-belt increments are collected from each moving batch shall be determined using a random systematic sampling scheme.

7.4 Choice of fuel for test

If more than one coal is to be sampled by the system, the coal chosen for the test shall be one that is expected to show up any bias in the sampling system. For example, bias for ash content on primary samplers and sample dividers is commonly caused by the exclusion of larger sized particles. If a coal is chosen where the ash content of such particles is similar to that of the coal as a whole, yet the particles are too large for collection, then no bias will be detected even though those particles are being excluded. If subsequently, the sampler is used to sample a coal where the large particles have an ash content which differs from the mean, the sampling system could be biased by not accurately representing those particles.

It is recommended that the complete bias test be carried out on coal from a single source.

7.5 Coke

The same requirements as those specified in 7.4 for coal with respect to its ash content shall apply when choosing a coke for test with respect to its moisture content.

8 Conduct of the test

8.1 General

Test the whole system by comparing stopped-belt reference samples taken from the primary flow with the product at the final stage of the preparation system. A bias test for the whole system is carried out by comparing the reference samples taken from the main flow with the samples collected at the final stage of the on-line system. The paired batch experimental design (see 7.3.3) is the preferred practice because it minimizes the disruption of normal operations.

Bias may be hardware induced, system logic induced, or a combination of both. It follows that routine operating conditions are best simulated by operating sampling systems under the control of system logic at routine operating condition settings. When using the paired increment methodology, do not stop the rest of the downstream system of the primary sampler via the system interlocks. This can be facilitated by use of a bias-test mode of system operation. This is not an issue in paired batch tests.

Conveyor belt systems for handling fuel are often not designed for repeated starting and stopping under load. The paired increment experimental design does not necessarily preclude collection of

stopped-belt increments, provided arrangements are made to stop the feed to the belt from which the increment is to be collected before the belt is stopped, so that the belt will be only partially loaded on restart. Increments can then be collected off the belt from the points in the stream where conditions that prevailed before the feed was stopped still exist.

8.2 Collection and preparation of test samples

Special care is required in planning the duties of each member of the sampling team and exactly how such duties will be performed. At this stage of planning, detailed operating protocols should be established to ensure uniform and consistent collection, weighing and processing of samples. This includes the preparation of facilities and assembly of all equipment necessary for collection of samples, processing and packaging them, and transporting them from collection and weighing sites to processing facilities and to the laboratory for analysis.

Special safety precautions are necessary for personnel working around the fuel handling and sampling machinery. Attention is drawn to the need to comply with all relevant safety regulations, especially the blocking of drive power to the conveyor from which the reference samples are collected.

The efficiency of the matched-pairs experimental design depends on the closeness with which reference and system samples are physically paired (correlated) to minimize the impact of variations in product quality within pairs. The extent to which within-pair variances are smaller than between-pair variances is an indication that the objective of the matched-pairs experimental design is being fulfilled. Prepare member pairs of samples together (even though through different steps) and analyse them in the same batch to avoid the introduction of systematic error resulting from variations in treatment during preparation and analysis. Be certain to preserve the identity of all samples.

Check the nominal top size of the fuel and the product of any sample crusher that is an integral part of the system or subsystem under test. Include this information in the test report. Samples for this purpose shall be separate from the matched pairs taken for the bias test.

In conducting bias tests where moisture content is a test parameter, care shall be taken to minimize moisture change from the test samples by following the procedures herein (see 9.2). Prepare and analyse the reference samples using the methods specified in ISO 13909-4, ISO 13909-6 and ISO 13909-7 for sample preparation and analysis with respect to precision and bias. Prepare and analyse the system samples by the methods used routinely during regular operations.

9 Outline of test procedure

9.1 General

The order of operations is as follows:

a) conduct a pretest inspection (see ISO 21398);

b) determine the physical location where the stopped-belt reference samples will be collected;

c) determine the parameters for the test;

d) determine the number of paired samples (minimum 30);

e) determine the composition of the sample pairs, and, if the paired batch method is to be used, determine the random or systematic random scheme for collection of reference increments for each batch;

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

9.2 Special precautions for moisture-test samples

Bias testing for total moisture content requires care to ensure that there is no change in the moisture content of samples collected. Consequently, the following precautions shall be taken.

a) Avoid conditions that may cause changes in moisture content, such as rain, snow, excessive heat, and wind.

b) Avoid contamination from free water carried by conveyor belts.

c) Ensure that sufficient labour is available to collect system and reference samples without delay.

d) Seal samples immediately after collection.

e) Carry out preparation and testing without delay to avoid moisture changes, if necessary, by weighing samples at the collection point.

f) Take account of any moisture condensation on sample containers.

NOTE Coal that has been treated to eliminate freezing or dust generation is not suitable for testing for moisture bias.

9.3 Documentation

Keep a record of the mass of each reference and non-reference sample. Also, keep a record of flow rates on the main belt if the system is equipped with a flow-rate indicator or retain charts from flow-rate recorders, if available.

A detailed log should be kept during the test showing the clock time at the beginning and end of collection of each sample, the occurrence of any deviations from operating protocols or unusual events and delays, and the reasons for them.

10 Statistical analysis and interpretation

10.1 Outline of statistical procedure

An outline of the procedure is as follows:

a) calculate all pair differences for each test set and each test parameter;

b) check the pair differences for outliers (see 10.2.1) and, if found, determine the appropriate disposition of each outlier (see 10.2.2);

c) calculate the statistics and determine if a bias is detected;

d) if a bias is not detected, examine the confidence region. If the region covers levels of bias of commercial concern, take more paired data sets and repeat the calculations.