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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

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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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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.

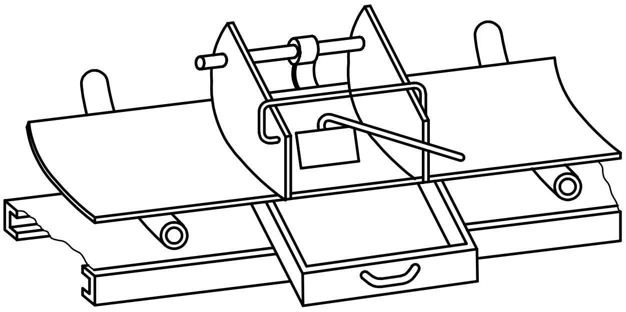

Figure 1 — Sampling frame

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.

10.2 Calculations

10.2.1 Statistical procedure for identifying outliers

Use the following procedure to identify possible outliers. This procedure is based on Cochran's maximum variance criterion. A statistic, Cochran's criterion C, is calculated using Formula (1):

$$C = \frac{d_{\max}^2}{\sum_{i=1}^{i=n} d_i^2} \tag{1}$$

where

$d_{\max}$ is the highest absolute value in the set of differences;

n is the number of pairs in the set.

Critical values for Cochran's criterion at the 1 % level are given for n = 20 to n = 120 in Table 1. If the value of C as calculated is greater than the table value, $d_{\max}$ is identified as an outlier to be investigated.

Table 1 — Critical values for Cochran's maximum variance test

n[a]	99 % Confidence level
20	0,480
21	0,465
22	0,450
23	0,437
24	0,425
25	0,413
26	0,402
27	0,391
28	0,382
29	0,372
30	0,363
31	0,355
32	0,347
33	0,339
34	0,332
35	0,325
36	0,318
37	0,312
38	0,306
39	0,300
40	0,294
60	0,215
120	0,123

NOTE This table is an extract from the table in ISO 5725-2:1994.[1]

[a] n is the number of differences in the series.

10.2.2 Disposition of outliers

An outlier may be due to

a) an extreme manifestation of the random variability inherent in the data,

b) an error in calculating or recording, or

c) the result of a gross deviation from the prescribed experimental procedures.

Statistical criteria for identifying an outlier are not a sufficient basis for discarding observations. When an outlier has been identified by a statistical test, an investigation shall be made to establish, if possible, the cause. Only if there is direct physical evidence that an outlier was caused by a gross deviation from the prescribed experimental procedure shall an observation be discarded. Whenever observations are discarded, the direct physical evidence justifying the action shall be reported together with the value(s) that are discarded.

If there is an error in the calculation of a result, it shall be corrected. If there have been deviations from prescribed experimental procedures or hard evidence that field conditions were abnormal for an observation, then it shall be discarded whether or not it agrees with the rest of the data.

If there is no rational explanation why it should be so very different from the rest of the data, then it shall be retained. When a gross deviation from prescribed experimental procedure is known to have occurred for a given observation, this observation shall be discarded whether or not it appears to be in agreement with other observations and without recourse to statistical tests for outliers.

In many cases, evidence of a deviation from prescribed procedures will consist only of the discordant value itself. When the experimenter cannot identify such observations with a gross deviation from prescribed procedures, the discordant value(s) shall be reported together with the extent to which these value(s) have been used in analysis of the data.

10.2.3 Calculation of confidence intervals and determining bias

10.2.3.1 Calculations of paired differences and sample variances and covariances of the differences

Assume that the parameters to be tested are moisture and ash content. Calculate the average moisture difference, $\bar{d}_{\mathrm{m}}$, and average ash difference, $\bar{d}_{\mathrm{a}}$, using Formulae (2) and (3).

$$\bar{d}_{\mathrm{m}} = \frac{1}{n}\sum_{n=1}^{n} d_{\mathrm{mi}} \tag{2}$$

$$\bar{d}_{\mathrm{a}} = \frac{1}{n}\sum_{i=1}^{n} d_{\mathrm{ai}} \tag{3}$$

where

n is the number of paired sets;

d_{mi} and d_{ai} are the moisture and ash differences in the respective series of differences.

Calculate the sample variances V_{mm} and V_{aa} of the moisture and ash differences using Formulae (4) and (5).

$$V_{\mathrm{mm}} = \frac{1}{n-1}\sum_{i=1}^{n} \left(d_{\mathrm{mi}} - \bar{d}_{\mathrm{m}}\right)^2 \tag{4}$$

$$V_{\mathrm{aa}} = \frac{1}{n-1}\sum_{i=1}^{n}\left(d_{\mathrm{ai}} - \bar{d}_{\mathrm{a}}\right)^2 \tag{5}$$

Calculate the sample covariance V_{ma} of the moisture and ash differences using Formula (6).

$$V_{\mathrm{ma}} = \frac{1}{n-1}\sum_{i=1}^{n}\left(d_{\mathrm{mi}} - \bar{d}_{\mathrm{m}}\right)\left(d_{\mathrm{ai}} - \bar{d}_{\mathrm{a}}\right) \tag{6}$$

10.2.3.2 Calculation of Hotelling's T^2 from the bias test sample data (in matrix form)

In matrix form, the mean differences are expressed by:

$$\bar{\boldsymbol{d}} = \begin{pmatrix} \bar{d}_{\mathrm{m}} \\ \bar{d}_{\mathrm{a}} \end{pmatrix} \tag{7}$$

The transpose of $\bar{\boldsymbol{d}}$ is:

$$\bar{\boldsymbol{d}}' = \begin{pmatrix} \bar{d}_{\mathrm{m}} & \bar{d}_{\mathrm{a}} \end{pmatrix} \tag{8}$$

The two parameter sample covariance matrix is calculated as:

$$\boldsymbol{S} = \begin{pmatrix} V_{\mathrm{mm}} & V_{\mathrm{ma}} \\ V_{\mathrm{ma}} & V_{\mathrm{aa}} \end{pmatrix} \tag{9}$$

and the inverse $\boldsymbol{S}^{-1}$ of $\boldsymbol{S}$ is:

$$\boldsymbol{S}^{-1} = \begin{pmatrix} \dfrac{V_{\mathrm{aa}}}{|\boldsymbol{S}|} & -\dfrac{V_{\mathrm{ma}}}{|\boldsymbol{S}|} \\ -\dfrac{V_{\mathrm{ma}}}{|\boldsymbol{S}|} & \dfrac{V_{\mathrm{mm}}}{|\boldsymbol{S}|} \end{pmatrix} \tag{10}$$

where $|\boldsymbol{S}|$ is the determinant of $\boldsymbol{S}$ calculated as:

$$|\boldsymbol{S}| = V_{\mathrm{mm}}V_{\mathrm{aa}} - \left(V_{\mathrm{ma}}\right)^2 \tag{11}$$

The value of T^2 is then calculated from the bias test data as:

$$T^2 = n\bar{\boldsymbol{d}}'\boldsymbol{S}^{-1}\bar{\boldsymbol{d}} \tag{12}$$

10.2.3.3 Calculation of Hotelling's T^2 from the bias test sample data (in algebraic form)

For two parameters (p = 2), as an alternative to the matrix format, use the following relationship for calculating the value of T^2:

$$T^2 = \frac{n}{\left(V_{\mathrm{mm}}V_{\mathrm{aa}} - V_{\mathrm{ma}}{}^2\right)}\left[\left(\bar{d}_{\mathrm{a}}\right)^2 V_{\mathrm{mm}} - 2\bar{d}_{\mathrm{m}}\bar{d}_{\mathrm{a}}V_{\mathrm{ma}} + \left(\bar{d}_{\mathrm{m}}\right)^2 V_{\mathrm{aa}}\right] \tag{13}$$

For one parameter (p = 1), the value of T^2 is calculated as:

$$T^2 = \frac{n\left(\bar{d}_x\right)^2}{V_{xx}} \tag{14}$$

where x represents the single parameter used for the test.

10.2.3.4 Determination of bias

Compare the value of T^2 calculated from the bias test data to the value T_0^2 shown in Table 2 for the number n of test sets and the number p of parameters. If the calculated value T^2 is greater than the table value T_0^2, conclude that the system is biased. If the calculated value T^2 is less than the table value, no bias has been detected.

Table 2 — Values of T_0^2 at 95 % confidence level (*n* = number of data sets and *p* = number of parameters)

n-1	*p* = 1	*p* = 2	*p* = 3	*p* = 4	*p* = 5
15	4,543	8,012	11,806	16,296	21,845
16	4,494	7,856	11,465	15,651	20,706
17	4,451	7,722	11,177	15,117	19,782
18	4,414	7,606	10,931	14,667	19,017
19	4,381	7,504	10,719	14,283	18,375
20	4,351	7,415	10,533	13,952	17,828
21	4,325	7,335	10,370	13,663	17,356
22	4,301	7,264	10,225	13,409	16,945
23	4,279	7,200	10,095	13,184	16,585
24	4,260	7,142	9,979	12,983	16,265
25	4,242	7,089	9,874	12,803	15,981
26	4,225	7,041	9,779	12,641	15,726
27	4,210	6,997	9,692	12,493	15,496
28	4,196	6,957	9,612	12,359	15,287
29	4,183	6,919	9,539	12,236	15,097
30	4,171	6,885	9,471	12,123	14,924
31	4,160	6,853	9,409	12,019	14,765
32	4,149	6,823	9,351	11,923	14,618
33	4,139	6,795	9,297	11,834	14,482
34	4,130	6,769	9,247	11,752	14,357
35	4,121	6,744	9,200	11,674	14,240
36	4,113	6,722	9,157	11,602	14,131
37	4,105	6,700	9,115	11,535	14,030
38	4,098	6,680	9,077	11,472	13,935
39	4,091	6,660	9,040	11,412	13,846
40	4,085	6,642	9,005	11,356	13,762
45	4,057	6,564	8,859	11,118	13,409
50	4,034	6,503	8,744	10,934	13,138
55	4,016	6,454	8,652	10,787	12,923
60	4,001	6,413	8,577	10,668	12,748
70	3,978	6,350	8,460	10,484	12,482
80	3,960	6,303	8,375	10,350	12,289
90	3,947	6,267	8,309	10,248	12,142
100	3,936	6,239	8,257	10,167	12,027
110	3,927	6,216	8,215	10,102	11,934
120	3,920	6,196	8,181	10,048	11,858

NOTE This table is an extract from *Methods of Multivariate Analysis, Third Edition,*[5] Table A.7

10.2.4 Review of the 95 % confidence region for the bias

The 95 % confidence region for the bias is defined by the inequality:

$$n\,\bar{\boldsymbol{d}}'\,\boldsymbol{S}^{-1}\,\bar{\boldsymbol{d}} \le T_0^2 \tag{15}$$

where T_0^2 is the table value for n-1 and p (see Table 2).

Formula 15 gives the interior boundary of an ellipsoid in p-dimensional space with centre at $\bar{\boldsymbol{d}}$.

For $p = 2$, e.g. the parameters moisture and dry ash content, the extremums for the ellipsoid are given by:

$$\text{for moisture,}\quad \bar{d}_\mathrm{m} \pm \left[\frac{T^2_{p,n-1}}{n}\left(\frac{V_\mathrm{mm}V_\mathrm{aa}-V_\mathrm{ma}}{V_\mathrm{aa}-V_\mathrm{ma}/V_\mathrm{mm}}\right)\right]^{1/2} \tag{16}$$

$$\text{for dry ash,}\quad \bar{d}_\mathrm{a} \pm \left[\frac{T^2_{p,n-1}}{n}\left(\frac{V_\mathrm{mm}V_\mathrm{aa}-V_\mathrm{ma}}{V_\mathrm{mm}-V_\mathrm{ma}/V_\mathrm{aa}}\right)\right]^{1/2} \tag{17}$$

For one parameter ($p = 1$), the confidence intervals are given by:

$$\bar{d}_x \pm \left(\frac{T^2_{1,n-1}}{n}V_{xx}\right)^{1/2} \tag{18}$$

The confidence region should be examined to determine if it covers a bias of commercial significance. If so, the taking of additional sets of data should be seriously considered. The decrease in area of the conference region to be obtained by taking more sets of data can be estimated by multiplying the existing confidence region by $\sqrt{n/n'}$, where n' is the new total number of data sets.

11 Test report

The format and detail included in the test report will depend on the purpose of the test. It is recommended that the report should include the following:

a) statement of the purpose of the test;

b) summary statement of the findings;

c) description and technical specification of the sampling system;

d) findings of the pretest inspection together with corrective action taken if the test is not to determine existing performance;

e) characterization of the fuel by preparation, size and quality;

f) specification of test conditions, flow rate, sampler operating parameters, etc.;

g) details of procedures adopted for the conduct of the test and tabulation of analysis values obtained;

h) explanation and discussion of any deviations from normal operating conditions and test specifications or of events occurring during the conduct of the test that could affect results;

i) statistical analyses and interpretations of results.

An annex should include all field logs, analytical results and mass data.

Where this method is used for certification of performance of systems and components, it is likely that all of the above items will be required.

Annex A
(informative)

Example calculations

A.1 Example calculation 1

A.1.1 General

This example illustrates the standard procedure used for determination of bias. Moisture and dry-basis ash content are the two parameters used in the test.

A.1.2 Data

The data used for the example calculations in A.1 and given in Tables A.1 and A.2 are taken from analyses for moisture and dry-basis ash content in coal samples.

Table A.1 — Raw data for moisture (%) and dry-basis ash (%) contents

	% Moisture		% Dry-basis ash	
Set no.	**System**	**Reference**	**System**	**Reference**
1	6,72	6,44	12,39	12,44
2	7,03	7,19	12,23	12,16
3	6,59	7,08	12,10	12,41
4	6,85	7,06	12,04	12,65
5	7,08	7,00	12,99	11,91
6	7,36	7,70	13,35	14,59
7	7,80	7,77	13,18	12,57
8	7,39	7,28	13,64	12,89
9	7,70	7,71	13,16	12,35
10	7,85	7,75	13,39	13,88
11	7,42	7,48	13,23	12,98
12	7,01	6,98	13,40	13,14
13	6,73	6,68	12,31	12,84
14	6,74	7,17	13,33	14,18
15	6,72	6,70	13,73	13,78
16	6,80	6,61	15,30	15,60
17	7,42	7,98	14,79	15,00
18	6,92	6,92	15,98	15,79
19	6,48	6,32	14,17	14,58
20	6,38	6,38	14,02	13,20
21	7,08	7,08	14,33	15,09
22	7,05	7,49	14,45	14,45
23	6,88	6,93	13,77	13,24
24	6,10	6,23	13,08	13,34
25	6,21	6,94	13,36	13,29
26	6,33	6,35	13,49	13,91
27	7,16	7,14	13,70	14,73
28	6,53	6,54	13,33	13,55
29	6,39	6,18	12,68	11,73
30	6,88	6,55	14,04	13,36

Table A.2 — Paired differences (system–reference) for moisture and dry-basis ash contents (%)

Set no.	% Moisture	% Dry-basis ash
1	0,28	−0,05
2	−0,16	0,07
3	−0,49	−0,31
4	−0,21	−0,61
5	0,08	1,08
6	−0,34	−1,24
7	0,03	0,61
8	0,11	0,75
9	−0,01	0,81
10	0,10	−0,49
11	−0,06	0,25
12	0,03	0,26
13	0,05	−0,53
14	−0,43	−0,85
15	0,02	−0,05
16	0,19	−0,30
17	−0,56	−0,21
18	0,00	0,19
19	0,16	−0,41
20	0,00	0,82
21	0,00	−0,76
22	−0,44	0,00
23	−0,05	0,53
24	−0,13	−0,26
25	−0,73	0,07
26	−0,02	−0,42
27	0,02	−1,03
28	−0,01	−0,22
29	0,21	0,95
30	0,33	0,68

A.1.3 Cochran’s test for outliers (p = 2)

n = 30 pairs

Parameter	**Moisture**	**Dry-basis ash**
Sum of squared values $\sum_{i=1}^{n} d_i^2$	2,001	10,739
Maximum squared difference	0,533	1,538
Ratio $\max d_i^2 / \sum_{i=1}^{n} d_i^2$	0,266	0,143
Cochran’s criterion C for n = 30 (see Table 1)	0,363	0,363

Conclusion: No outlier is detected.

A.1.4 Calculation of paired differences, sample variances and sample covariance (p = 2)

Average moisture difference $\bar{d}_{\mathrm{m}}$:

$$\bar{d}_{\mathrm{m}} = \frac{1}{n}\sum_{n=1}^{n} d_{\mathrm{m}i}$$

$$= -0{,}0677$$

Average dry-basis ash difference $\bar{d}_{\mathrm{a}}$:

$$\bar{d}_{\mathrm{a}} = \frac{1}{n}\sum_{i=1}^{n} d_{\mathrm{a}i}$$

$$= -0{,}0223$$

Sample variance for moisture differences V_{mm}:

$$V_{\mathrm{mm}} = \frac{1}{n-1}\sum_{i=1}^{n}\left(d_{\mathrm{m}i} - \bar{d}_{\mathrm{m}}\right)^2$$

$$= 0{,}0643$$

Sample variance for dry-basis ash differences V_{aa}:

$$V_{\mathrm{aa}} = \frac{1}{n-1}\sum_{i=1}^{n}\left(d_{\mathrm{a}i} - \bar{d}_{\mathrm{a}}\right)^2$$

$$= 0{,}3698$$

Sample covariance for moisture and dry-basis ash differences V_{ma}:

$$V_{\mathrm{ma}} = \frac{1}{n-1}\sum_{i=1}^{n}\left(d_{\mathrm{m}i} - \bar{d}_{\mathrm{m}}\right)\left(d_{\mathrm{a}i} - \bar{d}_{\mathrm{a}}\right)$$

$$= 0{,}0502$$

Calculation of T^2 for determination of bias (p = 2):

$$T^2 = \frac{n}{\left(V_{\mathrm{mm}}V_{\mathrm{aa}} - V_{\mathrm{ma}}{}^2\right)}\left[\left(\bar{d}_{\mathrm{a}}\right)^2 V_{\mathrm{mm}} - 2\bar{d}_{\mathrm{m}}\bar{d}_{\mathrm{a}}V_{\mathrm{ma}} + \left(\bar{d}_{\mathrm{m}}\right)^2 V_{\mathrm{aa}}\right]$$

$$= \frac{30}{\left(0{,}0643\right)\left(0{,}3698\right) - 0{,}0502^2}\left[\begin{array}{l}\left(-0{,}0223\right)^2\left(0{,}0643\right) - 2\left(-0{,}0677\right)\left(-0{,}0223\right)\left(0{,}0502\right)\\ +\left(-0{,}0677\right)^2\left(0{,}3698\right)\end{array}\right]$$

$$= 2{,}22$$

For p = 2 parameters and n = 30:

$$T^2_{p,n-1}(1-a) = T^2_{2,29}(0{,}95) = 6{,}919 \text{ (from Table 2)}$$

The value of T^2 calculated from the bias test data (2,22) is less than the table value $(T^2_{p\ ,n-1} = 6,919)$, so no bias is detected.

A.1.5 Determination of extremums for moisture and dry-basis ash bias estimates (p = 2)

Using Formula (16), the lower and upper extremums for moisture content are given by the lower and upper values of:

$$\text{Interval} = \bar{d}_{\text{m}} \pm \left[\frac{T^2_{2,29}}{n} \left(\frac{V_{\text{mm}} V_{\text{aa}} - V_{\text{ma}}{}^2}{V_{\text{aa}} - V_{\text{ma}}{}^2 / V_{\text{mm}}} \right) \right]^{1/2}$$

$$\text{Interval} = -0,0677 \pm \left\{ \frac{6,90}{30} \left[\frac{0,0643(0,3698) - 0,0502^2)}{0,3698 - 0,0502^2 \ / \ 0,0643} \right] \right\}^{1/2}$$

$$\text{Interval} = -0,0677 \pm 0,1215$$

$$\text{Lower extremum} = -0,189; \ \ \text{Upper extremum} = 0,054$$

Using Formula (17), the lower and upper extremums for dry-basis ash content are given by the lower and upper values of:

$$\text{Interval} = \bar{d}_{\text{a}} \pm \left[\frac{T^2_{2,29}}{n} \left(\frac{V_{\text{mm}} V_{\text{aa}} - V_{\text{ma}}{}^2}{V_{\text{mm}} - V_{\text{ma}}{}^2 / V_{\text{aa}}} \right) \right]^{1/2}$$

$$\text{Interval} = -0,0223 \pm \left\{ \frac{6,90}{30} \left[\frac{0,0643(0,3698) - 0,0502^2)}{0,0643 - 0,0502^2 \ / \ 0,3698} \right] \right\}^{1/2}$$

$$\text{Interval} = -0,0223 \pm 0,2916$$

$$\text{Lower extremum} = -0,314; \ \ \text{Upper extremum} = 0,269$$

The confidence ellipse given by Formula (15) is shown in Figure A.1.

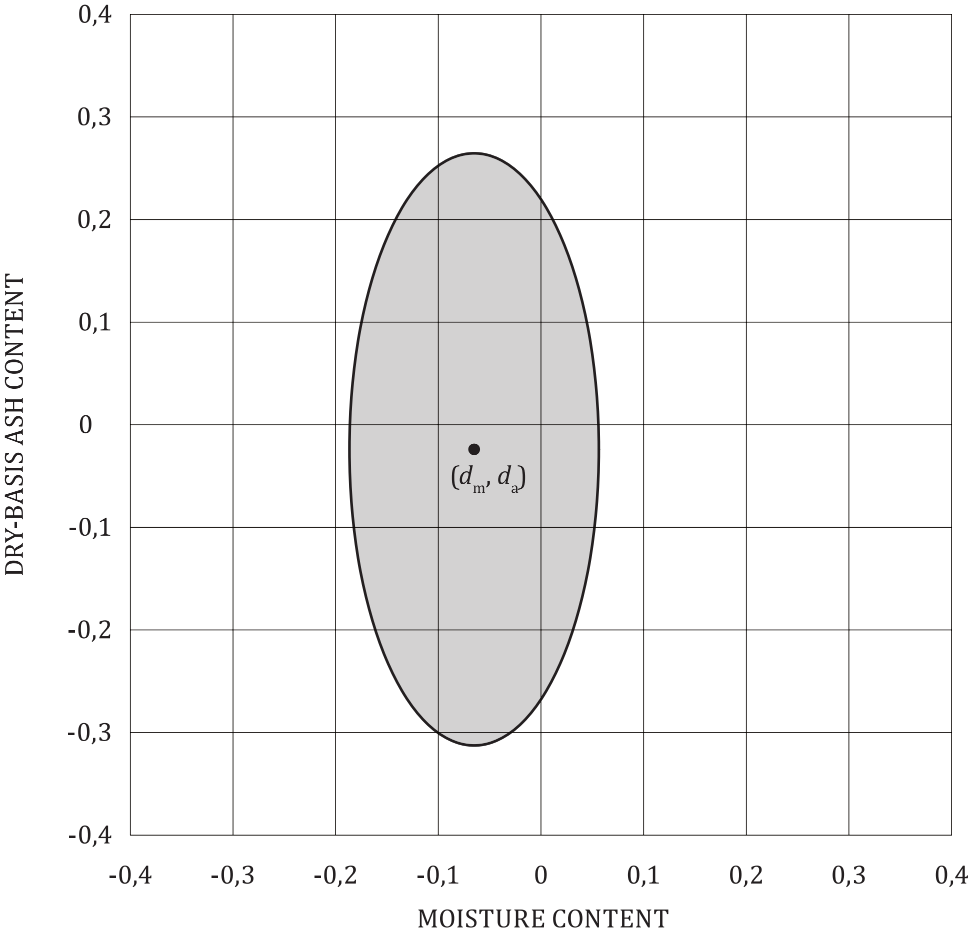

Figure A.1 — Confidence region for moisture and dry-basis ash biases

A.2 Example calculation 2

A.2.1 General ($p = 1$)

On occasions, for special purposes, only one parameter needs to be tested. For this example, only moisture content is used for the test of bias. The data are given in Table A.3.

Table A.3 — Raw data and paired differences (system–reference) for moisture content (%)

Set no.	System	Reference	Difference
1	9,73	10,03	−0,30
2	9,65	10,16	−0,51
3	9,84	10,26	−0,42
4	10,00	10,69	−0,69
5	10,49	11,11	−0,62
6	10,49	11,33	−0,84
7	10,85	10,87	−0,02
8	11,33	10,76	0,57
9	11,87	12,48	−0,61
10	12,20	11,17	1,03
11	11,81	12,23	−0,42
12	11,93	11,28	0,65
13	9,86	9,91	−0,05
14	9,99	9,91	0,08
15	9,63	10,12	−0,49
16	11,23	11,36	−0,13
17	10,87	12,04	–1,17
18	9,64	9,72	−0,08
19	9,66	9,91	−0,25
20	9,85	9,89	−0,04
21	10,04	9,63	0,41
22	9,87	9,65	0,22
23	9,58	9,85	−0,27
24	9,81	10,40	−0,59
25	9,75	10,27	−0,52
26	9,15	10,40	–1,25
27	10,01	10,01	0,00
28	9,81	9,95	−0,14
29	10,63	11,24	−0,61
30	10,59	10,48	0,11
31	10,46	10,82	−0,36

A.2.2 Cochran’s test for outliers (*p* = 1)

n = 31 pairs

Parameter	Moisture
Sum of squared values $\sum_{i=1}^{n} d_i^2$	9,159
Maximum squared difference	1,563
Ratio $\max d_i^2 / \sum_{i=1}^{n} d_i^2$	0,171
Cochran’s criterion *C* for *n* = 31 (see Table 1)	0,355

Conclusion: No outlier is detected.

A.2.3 Calculation of paired differences and sample variance (*p* = 1)

Average moisture difference $\bar{d}_\text{m}$:

$$\bar{d}_\text{m} = \frac{1}{n} \sum_{n=1}^{n} d_\text{mi}$$

$$= -0{,}2358$$

Sample variance for moisture differences V_mm:

$$V_\text{mm} = \frac{1}{n-1} \sum_{i=1}^{n} \left(d_\text{mi} - \bar{d}_\text{m} \right)^2$$

$$= 0{,}2478$$

A.2.4 Calculation for determination of bias (*p* = 1)

From Formula (14):

$$T^2 = \frac{n\left(\bar{d}_\text{m}\right)^2}{V_\text{mm}} = \frac{31\left(-0{,}2358\right)^2}{0{,}2478}$$

$$= 6{,}956$$

For one parameter and *n* = 31:

$T_{p,n-1}^2 (1-\alpha) = T_{1,30}^2 (0{,}95) = 4{,}171$ (from Table 2)

The value of T^2 calculated from the bias test data (6,956) is greater than the table value ($T_{p,n-1}^2 = 4{,}171$), so a bias is detected.

A.2.5 Determination of confidence intervals for moisture bias estimate ($p = 1$)

Using Formula (18), the lower (LCL) and upper (UCL) confidence limits on the moisture bias are given by the lower and upper values of:

$$\text{Interval} = \bar{d}_{\text{m}} \pm \left(\frac{T_{1,30}^{2}}{n} V_{\text{mm}} \right)^{1/2}$$

$$\text{Interval} = -0{,}235\,8 \pm \left[\frac{4{,}171(0{,}247\,8)}{31} \right]^{1/2}$$

$$\text{Interval} = -0{,}235\,8 \pm 0{,}182\,6$$

$$\text{Lower extremum} = -0{,}418\,4; \quad \text{Upper extremum} = 0{,}053\,2$$

A.3 Example calculation 3

A.3.1 General

This example illustrates the standard procedure used for determination of bias. In this case, sulfur, on a dry basis, and gross calorific value, on an as-received basis, are the two parameters used in the test.

A.3.2 Data

The data used for the example calculations in A.3 and given in Tables A.4 and A.5 are taken from analyses for the sulfur content and gross calorific value (J/g) of coal samples.

Table A.4 — Raw data for dry-basis sulfur (%) and as-received gross calorific value (J/g)

	Dry-basis sulphur (%)		**As-received gross calorific value** (J/g)	
Set no.	**System**	**Reference**	**System**	**Reference**
1	1,34	1,40	15 731	15 724
2	1,65	1,36	15 159	14 887
3	1,49	1,05	14 808	15 004
4	1,55	1,32	15 728	15 226
5	1,66	1,48	14 656	14 823
6	1,28	2,19	14 908	14 838
7	1,39	2,02	15 307	15 063
8	1,48	2,27	14 818	14 526
9	1,43	2,32	14 272	14 651
10	1,27	1,56	14 806	14 915
11	1,28	2,07	14 996	14 856
12	1,30	1,52	15 564	15 720
13	1,38	2,15	14 568	14 899
14	1,46	1,43	14 938	14 951
15	1,34	1,10	14 097	13 971
16	1,22	1,98	15 113	14 523
17	1,36	1,40	14 996	14 856
18	1,17	2,11	15 032	15 002
19	1,54	1,55	15 581	15 965
20	1,60	2,04	15 750	15 800
21	1,28	1,06	15 712	15 888
22	1,71	2,43	14 269	14 207
23	1,72	1,96	14 506	14 285
24	1,63	2,24	14 888	15 035
25	1,83	2,14	15 954	15 809
26	1,70	1,66	15 591	15 831
27	1,58	1,66	15 541	15 756
28	1,83	1,91	16 150	16 011
29	1,45	1,60	15 494	15 445
30	1,82	2,40	15 160	15 351

Table A.5 — Paired differences (system–reference) for dry-basis sulfur (%) and as-received gross calorific value (J/g)

Set no.	Dry-basis sulfur (%)	As-received gross calorific value (J/g)
1	−0,06	7
2	0,29	272
3	0,44	–196
4	0,23	502
5	0,18	–167
6	−0,91	70
7	−0,63	244
8	−0,79	292
9	−0,89	–379
10	−0,29	–109
11	−0,79	140
12	−0,22	–156
13	−0,77	–331
14	0,03	–13
15	0,24	126
16	−0,76	590
17	−0,04	140
18	−0,94	30
19	−0,01	–384
20	−0,44	–50
21	0,22	–176
22	−0,72	62
23	−0,24	221
24	−0,61	–147
25	−0,31	145
26	0,04	–240
27	−0,08	–215
28	−0,08	139
29	−0,15	49
30	−0,58	–191

A.3.3 Cochran's test for outliers (p = 2)

n = 30 pairs

Parameter	% Dry-basis sulfur	As-received gross calorific value (J/g)
Sum of squared values $\sum_{i=1}^{n} d_i^2$	7,538	1 673 985
Maximum squared difference	0,883 6	348 100
Ratio $\max d_i^2 \, / \sum_{i=1}^{n} d_i^2$	0,117	0,208
Cochran's criterion C for n = 30 (see Table 1)	0,363	0,363

Conclusion: No outlier is detected.

A.3.4 Calculation of paired differences, sample variances and sample covariance (p = 2)

Average dry-basis sulfur difference $\bar{d}_s$:

$$\bar{d}_s = \frac{1}{n}\sum_{n=1}^{n} d_{si}$$

$$= -0{,}288\,0$$

Average as-received gross calorific value difference $\bar{d}_c$:

$$\bar{d}_c = \frac{1}{n}\sum_{n=1}^{n} d_{ci}$$

$$= 9{,}167$$

Sample variance for dry-basis sulfur differences V_{ss}:

$$V_{ss} = \frac{1}{n-1}\sum_{i=1}^{n}\left(d_{si} - \bar{d}_s\right)^2$$

$$= 0{,}174\,1$$

Sample variance for as-received gross calorific value differences V_{cc}:

$$V_{cc} = \frac{1}{n-1}\sum_{i=1}^{n}\left(d_{ci} - \bar{d}_c\right)^2$$

$$= 57\,637$$

Sample covariance for dry-basis sulfur and as-received gross calorific value differences V_{sc}:

$$V_{sc} = \frac{1}{n-1}\sum_{i=1}^{n}\left(d_{si} - \bar{d}_s\right)\left(d_{ci} - \bar{d}_c\right)$$

$$= -5{,}587\,9$$

Calculation of T^2 for determination of bias (p = 2):

$$T^2 = \frac{n}{\left(V_{ss}V_{cc} - V_{sc}{}^2\right)}\left[\left(\bar{d}_c\right)^2 V_{ss} - 2\bar{d}_s\bar{d}_c V_{sc} + \left(\bar{d}_s\right)^2 V_{cc}\right]$$

$$= \frac{30}{\left(0{,}174\,1\right)\left(57\,637\right) - \left(-5{,}587\,9\right)^2}\left[\begin{array}{l}\left(9{,}167\right)^2\left(0{,}174\,1\right) - 2\left(-0{,}288\,0\right)\left(9{,}167\right)\left(-5{,}587\,9\right)\\ +\left(-0{,}288\,0\right)^2\left(57\,637\right)\end{array}\right]$$

$$= 14{,}29$$

For p = 2 parameters and n = 30:

$$T^2_{p\,,\,n-1}\left(1-\alpha\right) = T^2_{2\,,29}\left(0{,}95\right) = 6{,}919 \text{ (from Table 2)}$$

The value of T^2 calculated from the bias test data (14,29) is greater than the table value $(T^2_{(p,n-1)} = 6{,}919)$, so a bias is detected.

A.3.5 Determination of extremums for dry-basis sulfur and as-received gross calorific value bias estimates (p = 2)

The lower and upper extremums for dry-basis sulfur content are given by the lower and upper values of:

$$\text{Interval} = \bar{d}_{\text{s}} \pm \left[\frac{T^2_{2,29}}{n} \left(\frac{V_{\text{ss}} V_{\text{cc}} - V_{\text{sc}}{}^2}{V_{\text{cc}} - V_{\text{sc}}{}^2 / V_{\text{ss}}} \right) \right]^{1/2}$$

$$\text{Interval} = -0{,}288\,0 \pm \left\{ \frac{6{,}919}{30} \left[\frac{(0{,}174\,1)(57\,637) - (-5{,}587\,9)^2}{57\,637 - \left(-5{,}587\,9\right)^2 / (0{,}174\,1)} \right] \right\}^{1/2}$$

$$\text{Interval} = -0{,}288\,0 \pm 0{,}200\,4$$

$$\text{Lower extremum} = -0{,}49; \quad \text{Upper extremum} = -\,0{,}09$$

The lower and upper extremums for as-received gross calorific value are given by the lower and upper values of:

$$\text{Interval} = \bar{d}_{\text{c}} \pm \left[\frac{T^2_{2,29}}{n} \left(\frac{V_{\text{ss}} V_{\text{cc}} - V_{\text{sc}}{}^2}{V_{\text{ss}} - V_{\text{sc}}{}^2 / V_{\text{cc}}} \right) \right]^{1/2}$$

$$\text{Interval} = 9{,}167 \pm \left\{ \frac{6{,}919}{30} \left[\frac{(0{,}174\,1)(57\,637) - (-5{,}587\,9)^2}{0{,}174\,1 - \left(-5{,}587\,9\right)^2 / (57\,637)} \right] \right\}^{1/2}$$

$$\text{Interval} = 9{,}167 \pm 115{,}28$$

$$\text{Lower extremum} = -106{,}1; \quad \text{Upper extremum} = 124{,}4$$

The confidence ellipse given by Formula (15) is shown in Figure A.2. It is noted that the confidence ellipse does not cross the y-axis (dry-basis sulfur content) but does cross the x-axis (as-received gross calorific value). Thus, it is clear from the data that the bias found is in the dry-basis sulfur content parameter but not in the as-received gross calorific value parameter.

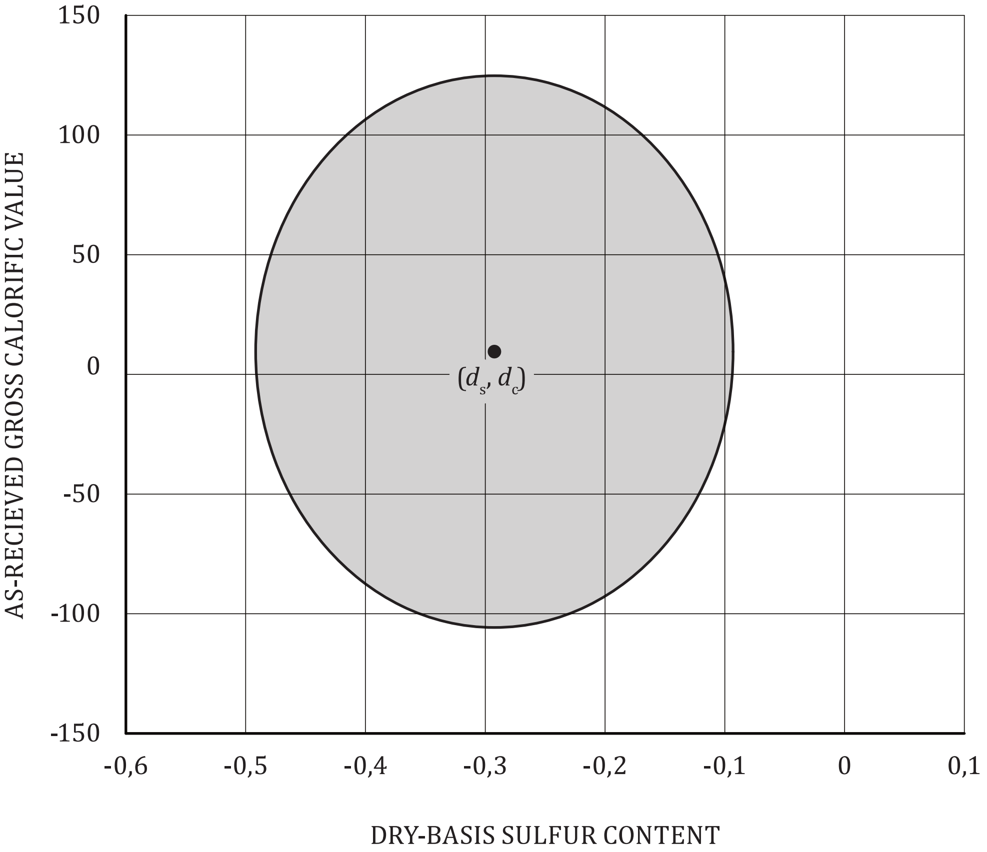

Figure A.2 — Confidence region for dry-basis sulfur content and as-received gross calorific value

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