
Solid mineral fuels — Evaluation of the measurement performance of on-line analysers

Combustibles minéraux solides — Évaluation de la performance de mesure des analyseurs en ligne



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Symbols and abbreviations	4
5 Principle	6
6 Analyser installations	6
7 Evaluation techniques	8
8 Instrument stability	9
9 Calibration	12
10 Operational measurement performance	15
11 Application	20
Annex A (informative) On-line analysis techniques for solid mineral fuels	22
Annex B (informative) Sources of measurement variance	26
Annex C (normative) Comparative test methods	28
Annex D (normative) Statistical assessment procedures	36
Annex E (informative) Reference standards	47
Annex F (informative) Specimen calculations	48
Bibliography	61

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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15239 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

Introduction

There are now many instruments in use which have been developed to enable the rapid on-line measurement of solid mineral fuels for a range of parameters that indicate coal quality. The principles on which they are based differ from those currently in use for sampling and analysis and, in effect, constitute a completely different approach to the measurement of solid mineral fuel quality.

This standard has been developed to specify methods by which the measurement performance of such analysers can be evaluated.

Copyright International Organization for Standardization

.....

Solid mineral fuels — Evaluation of the measurement performance of on-line analysers

1 Scope

This International Standard sets out practices for the evaluation of the measurement performance of all types of on-line analysers for solid mineral fuel.

It presents information on the different types of analyser currently available and describes procedures for the evaluation of various aspects of measurement performance, appropriate methods of test and techniques for the statistical assessment of the data collected.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

ISO 1988, *Hard coals — Sampling*

ISO 2309, *Coke — Sampling*

ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms*

ISO 5069 (all parts), *Brown coals and lignites — Principles of sampling*

ISO 13909 (all parts):2001, *Hard coal and coke — Mechanical sampling*

3 Terms and definitions

For the purposes of this document, the definitions given in ISO 1213-2, ISO 3534-1 and ISO 13909-1 and the following apply.

3.1

accuracy

closeness of agreement between an observation and the “true” value

[ISO 1213-2:1992]

3.2

analyser dynamic precision

closeness of agreement between analyser values, obtained from solid mineral fuel interrogated by the analyser under dynamic conditions and determined by a comparative test method which eliminates random errors attributable to the reference test method

3.3

analyser test method

method of analysis which gives, for a solid mineral fuel process stream, values arising from the operation of the on-line analyser, which are estimates of the true values for specified measurands

3.4

analyser value

value of a specified measurand in a test unit that is obtained from a test carried out by an analyser test method

3.5

backscatter geometry

arrangement of an interrogation process in which a source of incident energy and a detector system are on the same, or adjacent, sides of the solid mineral fuel passing through the interrogation zone

3.6

bias

systematic error which leads to the average value of a series of results being persistently higher or persistently lower than those which are obtained using a reference test method

[ISO 13909-1]

3.7

bias of scale

bias that varies as a function of the range of values measured

3.8

bias of location

bias that is constant and independent of the range of values measured

3.9

comparative dynamic precision

closeness of agreement between analyser values obtained from solid mineral fuel interrogated by the analyser under dynamic conditions and those determined by a comparative test method, which includes random errors attributable to the reference test method

3.10

comparative test method

method of testing in which analyser values are compared with corresponding reference values

3.11

comparison period

period of time, during which a test unit is interrogated by an analyser to give an analyser value and is sampled by a reference test method to obtain a reference value, for a measurand

NOTE The period can be based on the typical time to produce a particular mass of solid mineral fuel, e.g. a trainload, or on a period which coincides with operations, e.g. a shift, or some other period that is convenient to, or preferred for, a specific evaluation procedure.

3.12

interrogation process

procedure which elicits from the solid mineral fuel process stream a measurable response related, specifically or by inference, to the quantity of the measurand

3.13

interrogation volume

volume of the solid mineral fuel process stream in which the detected response to the interrogation process originates

3.14**interrogation zone**

part of the analyser installation through which the solid mineral fuel process stream passes and in which it is subjected to the interrogation process

3.15**mainstream configuration**

configuration in which the whole of the process stream to be analysed is presented to, although not necessarily analysed by, an on-line analyser

3.16**on-line analyser**

instrument for the measurement, continuously, of one or more quality indicators of solid mineral fuel while it is undergoing processing or handling, to give data rapidly and automatically

3.17**precision**

closeness of agreement between independent results obtained under stipulated conditions

[ISO 3534-1:1993]

NOTE For the purposes of this International Standard, the index of precision used is $\pm ts$, where t is the value of Student's t (95 % confidence level, two-sided) and s is the standard deviation of the observations about the mean value.

3.18**reference test method**

method of sampling, sample preparation and analysis which is expected to give, for a solid mineral fuel process stream, values which are unbiased estimates of the true values for specified measurands

3.19**reference value**

value of a specified measurand in a test unit that is obtained from a test carried out by a reference test method and which serves as a reference for comparison with an analyser value

NOTE For the purposes of this International Standard, reference values are considered to be conventional true values.

3.20**sample**

quantity of fuel, representative of a larger mass, for which the quality is to be determined

[ISO 13909-1]

3.21**static repeatability**

closeness of agreement between replicate analyser values obtained from a reference standard in the interrogation zone of the analyser

3.22**sub-stream configuration**

configuration in which a part of the process stream to be analysed is diverted by means of a suitable sampling system for presentation to an on-line analyser

3.23**test unit**

quantity of solid mineral fuel chosen for the determination of analyser and reference values

3.24

transmission geometry

arrangement of an interrogation process in which a source of incident energy and a detector system are on opposite sides of the solid mineral fuel passing through the interrogation zone.

4 Symbols and abbreviations

4.1 Mathematical

4.1.1 Primary

- β regression coefficient (slope)
- C Cochran's criterion
- d difference between pairs of values (other than duplicates)
- D_1 duplicate 1 reference test method value
- D_2 duplicate 2 reference test method value
- $\overline{D}$ mean of duplicate reference test method values
- δ test statistic (see D.16)
- EIV errors in variables
- $E(\rho)$ expected number of runs
- F F -distribution
- f_{SDR} static/dynamic response factor
- L_C confidence level
- n number of values in a set
- P precision
- Q test statistic (see D.16)
- R reference test method value
- R_1 reference test method 1 value
- R_2 reference test method 2 value
- r linear correlation coefficient
- ρ run
- S_1 reference standard 1 value
- S_2 reference standard 2 value

- s standard deviation
- s_g the expected (guaranteed) value of precision of the analyser at one standard deviation
- $s(\rho)$ standard error of number of runs
- σ population standard deviation
- t Student's t -distribution
- V variance
- ν degrees of freedom
- X_A analyser test method value
- x any value in a set
- x_{dup} difference between pairs of duplicate values
- χ^2 chi-squared distribution
- Z test statistic (see D.16)
- z normal deviate

4.1.2 Subscripts

- A set of analyser test method values
- c critical value
- d set of differences
- dup set of duplicate differences
- D_y set of dynamic calibration values
- D_1 set of duplicate reference 1 test method values
- D_2 set of duplicate reference 2 test method values
- $\bar{D}$ set of means of duplicate reference test method values
- g guaranteed value
- i i th value
- max maximum value
- 0 time zero
- R_1 set of reference test method 1 values
- R_2 set of reference test method 2 values

- St set of static calibration values
- S1 set of reference standard 1 values
- S2 set of reference standard 2 values
- τ time
- 1 set 1
- 2 set 2

4.2 Other abbreviations

- GHz gigahertz
- keV kilo-electron volt
- MeV mega-electron volt
- RF radiofrequency

5 Principle

The performance of an on-line analyser, which has been set up and calibrated, is evaluated by procedures that address three main aspects of analyser operation. These are the stability of the instrumentation, the validity of the calibration and the precision of measurement under operational conditions. Instrument stability is assessed by static measurements made, in replicate, at operationally significant intervals of time. The installed calibration is confirmed by making simultaneous comparative measurements with the analyser and a reference method of analysis over a range of measurand values which encompasses at least the spread of values encountered in normal operations. Operational performance is evaluated by comparison of analyser values with reference values obtained from separate reference procedures.

6 Analyser installations

6.1 General

There are many types of analyser, based on a variety of measurement principles and possible installation configurations, which have been designed to measure one or more indicators of quality in a range of products that occur in solid mineral fuel process streams.

The measurement principles on which analysers are based may be divided into four classes, as outlined in 6.2.

6.2 Analyser types

6.2.1 Absorption/scattering processes

The majority of on-line analysers for solid mineral fuel depend upon the existence of a quantitative relationship between the measurand and the degree of absorption and/or scattering of a beam of electromagnetic radiation or neutrons incident upon the solid mineral fuel flowing through the interrogation zone of the analyser. Incident electromagnetic radiation, in the X-, gamma, microwave or optical energy regions, or neutron radiation may be used; source, sample and detector may be arranged in transmission or backscatter geometry.

6.2.2 Excitation processes

A second group depends on a quantitative relationship between the measurand and the emission of specific electromagnetic radiation, (X- or gamma rays) arising as a result of excitation by an outside source of X-, gamma or neutron radiation.

6.2.3 Natural radiation emission

In this class, the gamma radiation emitted by naturally occurring radioisotopes, present in the measurand in relatively constant proportions, is measured.

6.2.4 Property changes

A few analysers depend upon an effect of the measurand on a selected electrical or physical property that is measurable on line.

NOTE Annex A gives information on techniques for on-line analysis.

6.3 Methods of presentation

The solid mineral fuel to be analysed may be transported through or past the analyser on a conveyor belt or other supporting platform, or within the confines of a container, chute or pipe. In most designs, the analyser detection system is physically non-invasive and non-contacting with the solid mineral fuel.

The condition of the solid mineral fuel presented to the analyser varies, among the methods of analysis, from material as it occurs in the process stream, to crushed, mixed and possibly dried material which has been carefully profiled.

The solid mineral fuel may be presented to the analyser as a bulk solid or as a fuel-water slurry.

Two basic installation configurations for on-line analysers are possible (see Figure 1). The choice between the two for any particular application depends on the type of analyser appropriate to the measurand and certain parameters of the product and the plant, such as particle size and flow rate.

6.4 Installation configurations

6.4.1 Mainstream

A mainstream configuration is a system in which the whole of the process stream for which the analytical information is required is presented to the analyser. The system can contain conditioning steps, such as mixing and profiling, prior to interrogation by the analyser.

6.4.2 Sub-stream

A sub-stream configuration is a system in which a portion of the process stream is diverted to the analyser by means of a suitable sampling process. The diverted portion of the stream may be subsequently subjected to sample preparation procedures, such as crushing, dividing and conditioning before presentation to the analyser. After interrogation the sub-stream is normally returned to the main process stream.

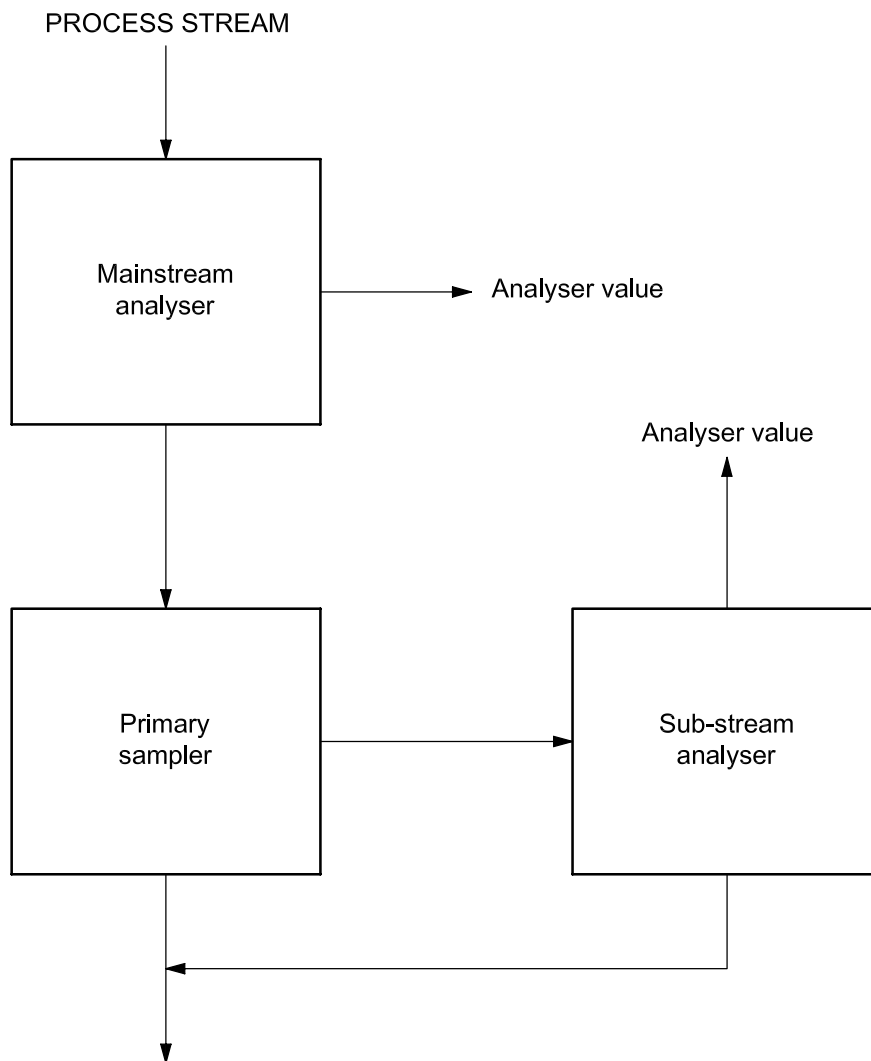


Figure 1 — Analyser configurations

7 Evaluation techniques

The procedures described in this International Standard are designed to allow the evaluation of analyser performance in a range of situations and conditions of operation.

They are intended to be applied to an analyser after it has been set up and calibrated as recommended by the manufacturer, with all instrumental parameters at their normal operational values for the particular installation.

In order to make a full evaluation of on-line analyser performance, it is necessary to address three interdependent aspects of analyser operation:

- instrument stability;
- calibration confirmation;
- operational measurement performance.

Since some of the measurement errors that are attributable to the analyser occur only as a result of operations on, or interactions with, the moving process stream, it is essential for a full evaluation of measurement performance to carry out tests under dynamic conditions.

Nevertheless, information from static tests, although more limited in its nature, is useful for monitoring some aspects of analyser performance on a routine basis.

An understanding of the sources of variance that contribute to the errors of measurement of the analyser and of any reference system with which it is compared, is necessary for the proper design of tests and the evaluation of the results. Sources of variance are discussed in Annex B.

The procedures used vary with the situation but have many features in common. General considerations for the design and operation of comparative tests are given in Annex C and techniques for the statistical analysis of the data in Annex D.

The principal steps involved in an evaluation are as follows:

- decide which aspect of analyser operation is to be evaluated (see Note);
- choose an appropriate method of test and design a scheme of operation;
- carry out the test procedure;
- apply appropriate statistical treatment to the data obtained from the test.

NOTE Frequently a situation will require more than one aspect to be considered (see Clause 11).

8 Instrument stability

8.1 General

It is a pre-requisite to accurate measurement by an on-line analyser that the instrumentation be stable and contribute as little as possible to the total error of measurement. Errors arising from the instrumentation may be random or systematic.

An estimate of random variations attributable to the instrumentation is obtained by determining the static repeatability. A significant increase in this value with time is an indicator of changes in instrumental characteristics that may need investigation and could lead to a worsening of the measurement performance of the analyser. Static repeatability is also an indicator of the limiting value of accuracy achievable (base-line performance).

Systematic instrumentation changes, which could affect the calibration if they are sufficiently large, are indicated by changes in the level of response from reference standards. These changes can provide the information needed to compensate for systematic instrumentation errors. In some analysers this process is carried out automatically at intervals and a correction applied.

Random and systematic variations originating in the instrumentation can be measured simultaneously by a relatively simple procedure that is amenable to routine use.

8.2 Objectives

The test methods and methods of data analysis described in 8.3, 8.4 and 8.5 are designed to achieve three objectives:

- to establish benchmarks against which subsequent tests for variations due to instrument instability may be compared;

- to determine and monitor the contribution made to the overall measurement performance of the analyser by random variations in response originating in the instrumentation;
- to monitor systematic changes in response, originating in the instrumentation, which may affect calibration.

8.3 Test conditions

Test conditions require

- a measurement period that will return an adequate precision to allow the testing of the significance of any changes from previous measurements,
- a minimum of 10 periods (see note).

NOTE A larger number of periods will increase the precision, but with diminishing returns; a practical maximum is probably about 20.

8.4 Test procedure

Make an initial set of consecutive replicate measurements under the chosen test conditions with a reference standard (see Notes 1 and 2) in the interrogation zone of the analyser. If it is required to detect systematic changes, repeat with a second reference standard that offers a different level of response from the first one. Designate these as measurements made at time 0. The levels of response from the standards chosen for the detection of systematic changes should be representative of those which are obtained from values of measurands close to each end of the calibration range. This will ensure that any shift of the calibration line and/or change in slope is signalled.

After an interval, repeat the above procedure. Designate these as measurements made at time τ . A suitable interval depends upon the usual operating programme of the analyser. The repeat test should normally be part of a regular check routine at intervals of a shift or a day, for example. A special check should be undertaken after any system changes (see Clause 11).

NOTE 1 Information on suitable reference standards is given in Annex E.

NOTE 2 Evaluation of instrument stability might not be possible with some designs of analysers that cannot accept a reference standard. In others, a suitable response might be available from the empty interrogation zone.

8.5 Data analysis

8.5.1 General

Consider measurements made at time 0 to be benchmarks against which subsequent measurements made at time τ are compared.

If, at time τ , the instrumental parameters in use when the benchmark performance was established have changed, normalize the data obtained for the test at time τ with respect to those changes.

8.5.2 Measurement precision

8.5.2.1 For each set of observations calculate the following values:

- variance, V_{S1} and V_{S2} (see D.2);
- standard deviation, s_{S1} and s_{S2} (see D.3);
- precision, P_{S1} and P_{S2} (see D.4).

8.5.2.2 Test the following for significance:

- ratio of the variances, $V_{S1,0}$ and $V_{S1,\tau}$ (see D.5);
- ratio of the variances, $V_{S2,0}$ and $V_{S2,\tau}$ (see D.5).

8.5.3 Changes in response level**8.5.3.1** For each set of observations, calculate the following:

- mean values, $\overline{S_1}$ and $\overline{S_2}$ (see D.6);
- differences of the means, $\overline{S_{1,0}}$ and $\overline{S_{1,\tau}}$, $\overline{S_{2,0}}$ and $\overline{S_{2,\tau}}$.

8.5.3.2 Test the following for significance:

- difference of the means, $\overline{S_{1,0}}$ and $\overline{S_{1,\tau}}$ (see D.7);
- difference of the means, $\overline{S_{2,0}}$ and $\overline{S_{2,\tau}}$ (see D.7).

8.6 Results and interpretation

Record the following information:

- the date of the test;
- the identity of the reference standard(s) used;
- relevant instrument parameters;
- the conditions of the test;
- the values of the individual observations;
- the values calculated in 8.5;
- the results of the significance tests.

The calculated value of P indicates the base-line performance of the analyser, i.e. a lower limiting value for operational accuracy over a similar measurement period. In practice, this value cannot be achieved since other sources of variation, outside the analyser instrumentation, also contribute to operational accuracy.

When an acceptable initial value for variance has been determined (see 11.2) or re-established (see 11.4), designate it $V_{S1,0}$. If a subsequent determination of variance, $V_{S1,\tau}$, is not significantly different from $V_{S1,0}$, then the instrumentation may be considered to be stable with respect to its contribution to random errors in the measurement of operational accuracy. If this is not the case, examine the instrumentation for the cause of the change.

When acceptable initial values for variance have been determined or re-established, designate the mean values of the observations as $\overline{S_{1,0}}$ and $\overline{S_{2,0}}$. If, in a subsequent determination, at time τ , there is no significant difference between $\overline{S_{1,0}}$ and $\overline{S_{1,\tau}}$, or between $\overline{S_{2,0}}$ and $\overline{S_{2,\tau}}$, then it can be concluded that the instrumentation response has not changed in a way that will affect the calibration. If one or both differences are significant, then there has been a change that could affect the calibration. Whether it will translate to a detectable change in the calibration depends on the size of other sources of error outside the instrumentation and the precision with which the calibration is defined. Consider confirmation of the calibration (see Clause 9).

9 Calibration

9.1 General

Since on-line analysers can only make measurements by reference to values obtained by some other method, the establishment and maintenance of a sound calibration is vital to accurate measurements.

The procedures described under 8.3 and 8.4 provide information that can be used to make corrections to the calibration line for errors due to instrumentation variations. However they offer no help in detecting systematic changes due to other sources, such as changes in the quality of the fuel, in its presentation or in the reference sampling and analysis scheme. This can be achieved only by undertaking a test described in this section.

A particular problem may occur with a well-controlled system that maintains product quality within a narrow range of values. It can be very inconvenient or even difficult to obtain, under dynamic conditions, a range of values that is wide enough to define a calibration with adequate precision. In that case, this International Standard describes a procedure (the static calibration), that is based on samples obtained other than under dynamic conditions.

9.2 Objective

To confirm that a previously installed calibration remains valid and appropriate to the current operating conditions.

9.3 Dynamic calibration

9.3.1 Test conditions

Test conditions include

- a two-instrument test scheme set up in accordance with the principles outlined in C.2,
- a comparison period that is of sufficient duration to allow enough reference sample increments to be taken to achieve a high degree of reference sample precision (see C.5 and C.6),
- a set of reference samples that adequately covers the full range of values to be expected under operating conditions,
- a minimum of 15 comparison periods (see C.5).

It might not always be possible to obtain a full range of expected values within a continuous test period. If this is the case, data obtained from further test periods may be added to the initial data provided that stability of the instrumentation has been shown to be satisfactory (see Clause 8) and the new data are consistent with it. (See ISO 13909-8:2001, 11.5, for tests of homogeneity of data).

9.3.2 Test procedure

For each comparison period defined in the conditions of test, collect duplicate reference samples as described in ISO 13909-7:2001, Clause 7 and log the corresponding data, X_{Ai}) from the analyser. Ensure that the collection of samples and data are properly synchronised and that sets of data all relate properly to their respective comparison periods. Prepare and analyse the duplicate test samples to give reference duplicate values (D_{1i} and D_{2i}).

9.4 Static calibration

9.4.1 General

The response of an analyser to solid mineral fuel presented under static conditions is unlikely to be the same as that presented dynamically. In order to check the calibration using static samples, it is necessary first to compare the analyser response under static and dynamic conditions and determine a correction factor designated the static/dynamic response. This correction can then be applied to estimate the relationship between analyser and reference values under dynamic conditions.

9.4.2 Test conditions

Test conditions include

- a two-instrument test scheme set up in accordance with the principles outlined in C.2,
- a measurement period that will minimize variations due to instrumentation,
- a minimum of 10 reference samples for static/dynamic response,
- a minimum of 15 samples of the product that adequately cover the full range of values to be expected under operating conditions for static test.

9.4.3 Test procedure

9.4.3.1 Static/dynamic response

Collect reference samples under normal operating conditions. Log the corresponding analyser readings, $X_{ADy,i}$. Prepare a static calibration sample from each reference sample. The quantity and condition of the static calibration samples will depend on the design of the on-line analyser. Typically, for an on-belt ash analyser for instance, about 5 kg of air-dried coal crushed to a top size of 1 mm is suitable. It is important that all samples in a set be prepared in the same manner using the same equipment. Present the static calibration samples to the analyser and log the analyser readings, X_{Ai} . Determine the static/dynamic response factor (f_{SDR} ; see D.8).

9.4.3.2 Static test

Collect a set of samples, not necessarily under dynamic conditions. From each sample prepare a static calibration sample and duplicate analysis test samples.

Present each static calibration sample to the analyser, under static conditions, for the chosen measurement period. Log the data, X_{Ai} , and analyse each duplicate test sample, D_{1i} and D_{2i} . Correct the results by adding the f_{SDR} (see 9.4.3.1) to each static analyser value.

9.5 Data analysis

9.5.1 General

Calculate the following:

- mean duplicate reference values $\bar{D}_i$;
- differences, d_i , between the analyser values X_{Ai} (corrected in the case of static test) and the mean duplicate reference values $\bar{D}_i$.

9.5.2 Visual assessment

Plot a graph of the analyser values, X_{Ai} , against the mean duplicate reference sample values $\bar{D}_i$, and assess visually for the presence of possible outliers and other problems (see D.9.2).

It is recommended that other graphical procedures outlined in D.9 also be carried out before a full statistical analysis of the data is undertaken.

9.5.3 Outliers

Check for outliers by the statistical procedure of Cochran's criterion (see D.10.2). Consider possible outliers identified in this way for removal in accordance with the criteria defined in D.10.3. Remove those values that meet the criteria.

9.5.4 Independence of differences

Test the list of differences for independence between observations: (see D.11).

If the test is satisfactory, continue with the test for bias. Otherwise, discard the data and examine the scheme for possible causes. Modify the test scheme if appropriate and repeat the test.

9.5.5 Tests for bias

Calculate the slope of the linear regression of the analyser values, X_{Ai} , on the mean duplicate reference values $\bar{D}_i$ using an "errors-in-variables" (EIV) method (see D.12.1)

Calculate the variance of this slope (see D.12.2)

Test for existence of significant bias of scale (see D.13)

If there is no significant bias of scale, test for bias of location (see D.14)

9.6 Results and interpretation

Record the following information:

- date of the test;
- identity of the product;
- relevant instrument parameters;
- conditions of the test;
- values of the individual observations;
- results of the tests in 9.5.

Tests for bias of scale and total bias are made with reference to the current calibration that is described in this test by the relationship $X_{Ai} = \bar{D}_i$. In the case of commissioning tests (see 11.2), the current calibration is the preliminary calibration installed in accordance with the manufacturer's instructions. Otherwise it is a calibration installed following a previous test of this kind.

If there is a significant change in bias of scale or of bias of location, which cannot be attributed to systematic instrumentation variations (see 8.6), examine the reference method scheme for possible sources of variation which have arisen since the previous confirmation of the calibration. Changes in solid mineral fuel quality are

also potential sources of systematic error to be considered. If the additional source of error cannot be identified and eliminated, install a new calibration based on the relationship found between analyser and reference values in the current test.

10 Operational measurement performance

10.1 General

When the analyser calibration has been established and the absence of significant bias demonstrated, the precision of the measurement under operational (dynamic) conditions becomes the manifestation of the accuracy of measurement of the analyser.

Depending on the particular application, one or both of two performance indicators may be considered for its evaluation:

- analyser dynamic precision (initial performance testing);
- comparative dynamic precision (maintenance of measurement performance).

10.2 Determination of analyser dynamic precision

10.2.1 Test methods

The analyser dynamic precision is the definitive measure of analyser performance and is determined by either of two comparative test methods (see C.2) that eliminate the errors due to the reference test method from the result.

- The two-instrument test: this is the less complex test but it is susceptible to error if there is any calibration bias present. Such errors will report to the analyser precision.
- The three-instrument test: this test requires a second, independent reference sampling system and eliminates bias errors. However, care must be taken to ensure that all the relevant constraints on the test procedure are fully met.

NOTE Provided that one of the reference test methods used is that which will also be used in subsequent routine operations, it will also allow the measurement of comparative dynamic precision (see 10.3).

10.2.2 Objectives

The test methods described in 10.2.1 are designed to achieve two objectives:

- determination of the definitive measurement performance of the analyser;
- its comparison with a guaranteed value.

10.2.3 Test conditions

Test conditions include the following:

- two- or three-instrument test scheme set up in accordance with the principles outlined in C.2;
- comparison period chosen with reference to the conditions of the specified guaranteed performance. (Note that if this application is required and if the performance guarantee is to be checked, the period should be chosen with reference to the conditions of the specified performance. However, due regard should be paid to the need to constrain the precision of the reference test method to be similar to the expected value for the analyser; see C.5 and C.6);