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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

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Foreword

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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.

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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.

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

— Dy set of dynamic calibration values

— D1 set of duplicate reference 1 test method values

— D2 set of duplicate reference 2 test method values

— $\overline{\text{D}}$ set of means of duplicate reference test method values

— g guaranteed value

— i *i*th value

— max maximum value

— 0 time zero

— R1 set of reference test method 1 values

— R2 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.

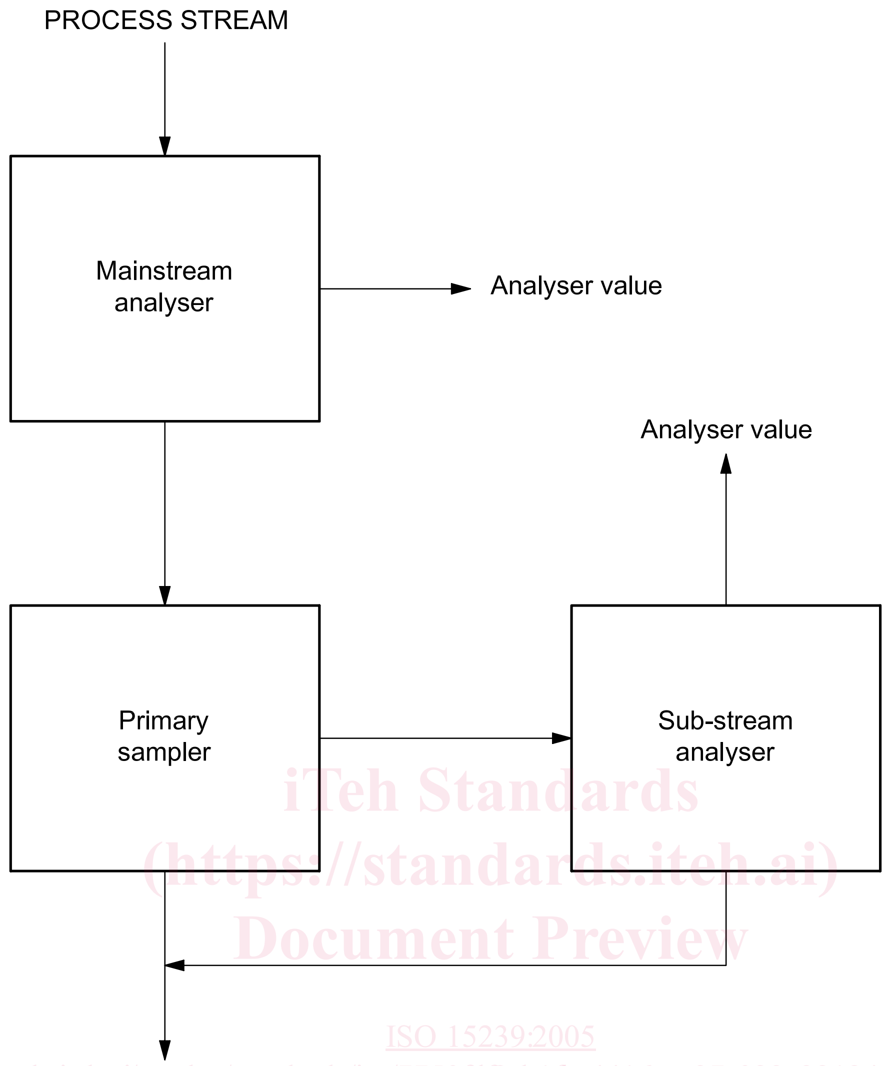

Figure 1 — Analyser configurations

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.