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Validation method for fire gas analysis —

Part 3: Considerations related to interlaboratory trials

Méthode de validation pour l'analyse des effluents du feu — Partie 3: Considerations relatives aux essais inter laboratoires avec les analyses chimiques des effluents du feu

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 3, *Fire threat to people and environment*.

A list of all parts in the ISO 12828 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The reduction of human tenability from fire effluent has long been recognized as a major cause of injury and death in fire. The composition and concentration of the effluent from a large fire are also clearly key factors in determining the potential for harm to the environment. The harmful components of fire effluent can be determined from both large-and small-scale tests of materials and finished products. Equations have been developed for quantifying the effects of the effluent components for example to estimate the available safe escape time (ASET)[1]. Related documents are also being developed in ISO/TC 92/SC 3 which deal with environmental threats from fire effluent.

These advances in fire science and fire safety engineering have led to an increasing demand for quantitative measurements of the chemical components of the fire effluent. Characterizing these measurements is described in ISO 12828-2. Comparing results from one laboratory to another and giving a global confidence in any measurement technique, independently from the user and the conditions of use, are described in this document.

Validation method for fire gas analysis —

Part 3:
Considerations related to interlaboratory trials

1 Scope

This document describes tools and gives guidance concerning interlaboratory trials related to fire effluent analyses. It explains the relative contributions from the physical fire model and analytical techniques to evaluate trueness and fidelity. It also explains the difficulties involved with the interpretation of round-robin data and with the evaluation of trueness in fire effluent analyses.

This document complements ISO 12828-1, which deals with limits of quantification and detection and ISO 12828-2, which deals with interlaboratory validation of analytical methods. It is a toolbox useful in the framework of ISO/IEC 17025 assessment of any fire laboratory.

Examples of existing standards where the information contained in this document can be used are the analytical chemical methods in ISO 19701[2], ISO 19702[3], ISO 5660-1[4], and the chemical measurements in the methods discussed in ISO/TR 16312-2, ISO 16405[6], ISO/TS 19021[7], or their application to fire toxicity assessment using ISO 13571[1] and ISO 13344[8].

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 12828-1, *Validation method for fire gas analysis — Part 1: Limits of detection and quantification*

ISO 12828-2, *Validation method for fire gas analysis — Part 2: Intralaboratory validation of quantification methods*

ISO 13943, *Fire safety — Vocabulary*

ISO 19706, *Guidelines for assessing the fire threat to people*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5725-1, ISO 13943, and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
accuracy
closeness of agreement between a measured quantity value and a true quantity value of a measurand

[SOURCE: ISO/IEC Guide 99:2007, 2.13, modified — NOTES 1 to 3 have been removed.]

3.2
k-score
score that characterizes the fidelity of a laboratory, defined by:

$$k_i = \frac{s_w}{s_r}$$

3.3
precision
closeness of agreement between indications or measured quantity values obtained by replicate measurements on the same or similar objects under specified conditions

[SOURCE: ISO/IEC Guide 99:2007, 2.15, modified — NOTES 1 to 4 have been removed.]

3.4
trueness
closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value

[SOURCE: ISO/IEC Guide 99:2007, 2.14, modified — NOTES 1 to 3 have been removed.]

3.5
z-score
score that characterizes the bias and thus the trueness of a laboratory, assuming the real value is the general mean and the real dispersion is the overall standard deviation *s*, defined by:

$$z_i = \frac{\bar{y}_i - m}{s}$$

4 Symbols

b	laboratory component of bias under repeatability conditions
e	random error occurring in repeatability conditions
m	general mean, sometimes expressed as the level of the test
n_i	number of laboratories
s_L	between-laboratory standard deviation
s_r	estimate of the repeatability standard deviation
s_R	estimate of the reproducibility standard deviation
s_w	within-laboratory standard deviation for the laboratory i
$\bar{y}_i$	mean value from laboratory i
$\bar{\bar{y}}_i$	mean value from all laboratories

5 General considerations

5.1 Trueness and fidelity

A test result is described by the model $y = m + b + e$. In this expression, the measured value is the real value affected by the bias (trueness error) and the random error (fidelity error).

5.1.1 Trueness

In the context of fire effluent analysis, trueness is the correspondence between the real (theoretical) value of an analyte and the measured value (see ISO 19703). Depending on the existence and knowledge of the real value, bias *b*, characterizing trueness is sometimes partially characterized by Z-score. Bias expresses the deviation to a real value, where Z-score supposes that the general mean corresponds to the real value; this last assumption is questionable in several cases for fire gases analysis. The Z-score could be interpreted as follows:

— $|z_i| \leq 2$ means that the trueness performance of the laboratory is in the 95 % range of more probable values;

— $2 < |z_i| \leq 3$ means that the trueness performance of the laboratory is questionable, in the range of the next 4,7 % less probable values;

— $|z_i| > 3$ means that the trueness performance of the laboratory is unsatisfactory, in the range of the remaining 0,3 % of the least probable values.

There are several ways to determine trueness in fire gas analysis:

— Case 1): Physical fire model included. General principle is combustion of standard materials then mass balance. A real value could be assumed and bias calculated for several mass balances, including:
  — Halogenated acids, assuming 100 % mol/mol of X (often chloride) in the initial material is converted into HX;
  — Carbon, considering CO_2, CO and other carbonaceous compounds represent the large majority of carbon initially present, preferably in well ventilated conditions;
  — Sulfur released as SO_2 in well ventilated conditions (stage 2 according to ISO 19706).

  This kind of mass balance corresponds to a global validation of trueness and fidelity due to the fire model itself plus the analysis as the related error sources cannot be separated. It is not possible to do so with some other elements such as nitrogen.

— Case 2): Physical fire model excluded
  — Sub-case 2a): Use of standard gases injected at the point of emission in normal use, e.g. at the location of the material in combustion tests. This checks the sampling and analysis trueness and fidelity, but not the possible variation due to combustion process itself.
  — Sub-case 2b): Use of standard gases or standard solutions (see ISO 12828-1) in realistic matrix. This checks the trueness and fidelity of the analysis.

EXAMPLE 1 Cases where the theoretical value is known.

The analyte studied is hydrogen chloride. The analytical method is high performance ion chromatography according to ISO 19701:2013, 5.5.2. To determine the trueness of the method:

— Case 1): Unmodified PVC is burnt according to an appropriate fire model. Suitable solution traps are used to capture the hydrogen chloride gas from the effluent. The solution is then analyzed. Chlorine comprises 56,8 % by mass of PVC and the theoretical yield of HCl is 0,584 g/g.