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Validation method for fire gas analysis —

Part 1:
Limits of detection and quantification

Méthode de validation des analyses de gaz d'incendie —

Partie 1: Limites de détection et de quantification

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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 12828-1 was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 3, *Fire threat to people and environment*.

ISO 12828 consists of the following parts, under the general title *Validation method for fire gas analysis*:

— *Part 1: Limits of detection and quantification*

The validation of the quantification method will be covered in a future Part 2.

Introduction

A major cause of injury and death in fire is exposure to the mobile fire effluent, which typically contains many toxic and irritant chemical species such as gases and vapours in addition to solid and liquid particulates (aerosols) such as visible smoke. In addition, fire effluents, especially those released from fires which are large and relatively prolonged, have the potential to contaminate a wider environment, both through the airborne smoke plume and the residues remaining on the ground which can affect the soil and watercourses.

Clearly, a knowledge of the composition and concentration of fire effluents and how they change during a fire is a vital requirement for assessing the potential for injury, death and environmental impact from fires.

Chemical and physical measurements of the harmful components of fire effluents are obtained from a wide variety of standard and ad-hoc fire tests on materials and finished products, often with the capability of varying the combustion conditions (e.g. temperature and air availability). Such tests can range in size from those using small-scale bench-top apparatus to those utilizing full-scale structures, often simulating a specific real-fire scenario.

When used for the assessment of hazards to life from fire, these data have been increasingly applied through the use of equations (e.g. fractional effective dose) developed specifically for quantifying the effects of the effluent on humans and, in particular, for an estimation of the times before specific hazards in a fire (ISO 13571).

Procedures are also currently being developed within ISO/TC 92 SC 3 for dealing with the environmental threats from fire effluent.

Recent advances in fire-safety engineering, including the calculation of time available for escape, have led to an increased demand for accurate detailed quantitative measurements of the chemical components of the fire effluent. It is clearly important, therefore, that the methods used to obtain these data be suitably validated for use in the specific application required. It is also important to define the required limits of detection and quantification (L_D and L_Q) values for a given analysis and application to avoid setting unnecessarily low limits which could prove expensive, time consuming and impose undue technical restraints, with little or no effect on the accuracy and precision of the end-use of the data.

This part of ISO 12828 provides guidance on methods for ensuring that any chemical or physical method of analysis for specific chemical species in fire effluents is suitably validated for correct use of limits of detection and limits of quantification for a given application of the data. It provides information to assist compliance with general requirements for the competence of testing and calibration laboratories (ISO/IEC 17025).

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Validation method for fire gas analysis —

Part 1:
Limits of detection and quantification

1 Scope

In this part of ISO 12828, limits of detection (L_D) and limits of quantification (L_Q) are defined and calculated. It provides methods for determining suitable values for these two parameters for a specific analytical procedure and for a specific chemical species. It does not provide detailed guidance on methods of sampling and analysis of specific species which might be present in fire effluents. This guidance is contained in ISO 19701 and ISO 19702. The use of this part of ISO 12828 fulfils the requirement in ISO/IEC 17025 that a laboratory carrying out chemical analysis (e.g. of fire effluents) is able to characterize and evaluate a method by such parameters as L_D, L_Q and uncertainty. Examples of where the information contained in this part of ISO 12828 can be applied are:

a) Method validation: The parameters L_D and L_Q are required for all chemical analytical methods; they are as important as measurements of accuracy and precision.

b) Classifications based on toxicity indexes: Methods selected for analysis of effluents must have a minimum limit of quantification, consistent with the critical concentration used to calculate the contribution of each effluent to toxicity index. Furthermore, a toxicity index is not considered as zero when concentrations of toxic species are detected but not quantified (as they are below the limit of quantification). In this case, a contribution at least equal to the limit of detection for each measured species can be registered. Examples are shown in Annex B.

c) Round-robin comparison between two analytical methods: For a given working range, two methods can be compared only if the limits of these methods (calculated by using this part of ISO 12828) are similar for the lower range of concentrations to be measured. For example, if one laboratory provides values near its own limit of detection, and another laboratory gives results well above its own limit of detection, the reproducibility R assessment of the round robin can be artificially overestimated. In many round-robin tests, bad reproducibility R values can be found if some values are close to the limit of quantification and/or limit of detection. In such cases, no conclusion on the round robin can be given without an assessment of the limit of quantification value and the expression of results as described in this part of ISO 12828.

This part of ISO 12828 is intended for use by operatives familiar with chemical and physical analysis of fire effluents.

Examples of existing standards where the information contained in this part of ISO 12828 can be used are the analytical chemical methods in ISO 19701, ISO 19702, ISO 5660-1, and the chemical measurements in the methods discussed in ISO/TR 16312-2.

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 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 13571:2007, *Life-threatening components of fire — Guidelines for the estimation of time available for escape using fire data*

ISO 13943, *Fire safety — Vocabulary*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 19701, *Methods for sampling and analysis of fire effluents*

ISO 19702, *Toxicity testing of fire effluents — Guidance for analysis of gases and vapours in fire effluents using FTIR gas analysis*

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 13943, ISO 5725-1 and the following apply.

NOTE   There is no consensus for an exact definition of the following two limits, especially for the limit of detection. However, two references have been used as guidance for the definitions cited here: ISO 11843-1 and ISO 11843-2.

3.1
limit of detection
L_D

smallest quantity of an analyte in a sample that can be detected and considered with a stated probability as different from the detector output from a blank sample

NOTE   It should be noted that the actual quantity of the analyte need not be stated and that the symbol "y_{L_D}" is used to express the limit of detection in terms of a detector signal value, converted (via a calibration technique) into a mass, volume or concentration term.

3.2
limit of quantification
L_Q

smallest quantity of an analyte which is possible to quantify under the specific experimental conditions described in the chosen method, where the variability of the method has been defined (i.e. a variation coefficient has been determined)

NOTE   The symbol "y_{L_Q}" is used to express the limit of quantification in terms of a detector signal value, converted (via a calibration technique) into a mass, volume or concentration term.

4 Symbols

u Actual analyte concentration or terms which use this

y Value of the analyte concentration as measured by the analytical system (detector output as "raw data")

$U(x)$ Enlarged absolute uncertainty on measurement of x

σ_i Standard deviation for i

5 General considerations

5.1 Limit of detection: table of risks

There are two contingencies or risks associated with L_D:

— a risk designated "α" where the substance may be detected in the sample even though the substance is not actually present;

— a risk designated "β" where the substance is not detected in the sample even though the substance is actually present.

These risks can be illustrated using a simple table of analysis result versus reality as in Table 1.

**Table 1 — Table of risks: analysis result versus reality**

		Analysis result	
		Not present	**Present**
Reality	**Not present**	Good decision Probability $1 - \alpha$	Probability α FALSE POSITIVE
	Present	Probability β FALSE NEGATIVE	Good decision Probability $1 - \beta$

It is important that false negatives be eliminated. Failing to observe the presence of a particular toxicant, especially if it is present at a toxicologically important level, can lead to a false sense of safety in an engineering calculation. False positives may indicate a hazard that is not actually present. This conservative outcome is less harmful than the outcome from a false negative. It can be considered as a "fail safe" result.

5.2 Limit of quantification: effect on repeatability r

Near the limit of quantification, the accuracy of measurement is lower than in the region over which an analysis system has been calibrated. The limit of quantification, however, may be substantially lower than the lowest extremity of the calibrated region for an analytical system and is essentially the lowest point where the analytical method may give an acceptable quantified measurement.
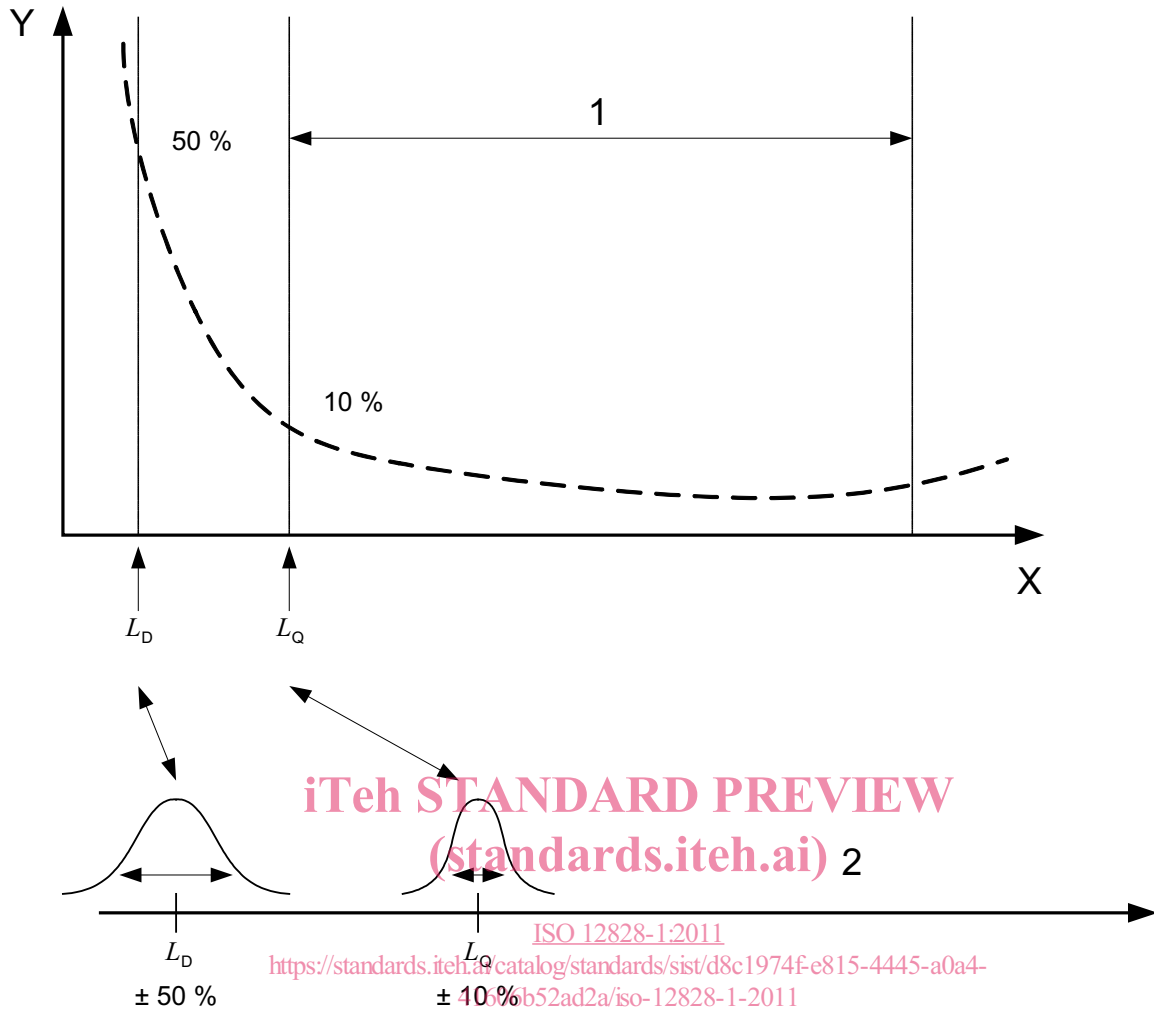
Key

X   amount of a substance
Y   repeatability, r, (%)
1   calibration range
2   value

Figure 1 — Position of L_D and L_Q on calibration range
(based on the work of Horwitz[8], and Brown *et al* [9])

5.3   Typically accepted values for limits of detection and quantification

The limit of quantification is usually considered to be 10 % of the measurement repeatability r, and the limit of detection is usually considered to be three times the standard deviation of a matrix of blank sample results, σ_b. If the standard deviation is constant between zero and the limit of quantification, the limit of quantification is equal to 10 times the standard deviation of a matrix blank result. The limit of detection is therefore usually considered as 50 % of the repeatability r.

The value of 10 % is derived from Equation (1)

$$L_Q = 10 \times \sigma_b \qquad\qquad \frac{\sigma_b}{L_Q} = 10\,\% \qquad\qquad\qquad (1)$$

6 Methods for determining limits of detection and quantification

6.1 Principles and summary of methods

Validation of a chemical or physical method of analysis for a specific species in the fire effluent can be considered as a four-step process:

a) Step 1. Define the final objective/end-use of the data.

When undertaking a chemical or physical analysis of a fire effluent, the objective/end-use of the analytical data can be considered. For example, the objective might be to contribute to the fire-safety engineering design of a building (e.g. through a fractional effective dose calculation of Available Safe Egress Time – ASET), to determine the accuracy of a numerical fire model, the relevance of a small-scale physical fire model or the determination of the toxic potency of the effluent from a particular combustible item.

b) Step 2. Determine the lowest concentrations and degree of accuracy and precision required.

Having established the end-use of the data, the lowest concentrations and the appropriate degree of accuracy and precision required in the chemical analysis can be determined. For example, in a fractional effective dose calculation (where the cumulative effects over fixed time intervals of reducing tenability due to a specified range of species is considered), interest might range from concentrations which could incapacitate people of average sensitivity to the species measured, to concentrations which show negligible toxic effect over a long exposure period. It is also important to appreciate that it is not normally necessary to attempt such measurements with any greater precision than that resulting from the precision of the end-use of these data. This can avoid undue technical and economic restraints in obtaining measurements.

c) Step 3. Select an appropriate sampling and analytical method.

The ultimate requirement of any chemical analysis of a species in the fire effluent is to obtain mass, volume or concentration data for the species which is as close as practicable (given the considerations of step 2) to the actual mass, volume or concentration of the species in the effluent being measured. The two main stages to consider are the sampling procedures and the analytical methodology. Sampling may be continuous or take place over discrete time intervals but either procedure may be subject to potential losses through a variety of effects. Analysis of a species may be carried out continuously or intermittently during the fire or from stored samples.

d) Step 4. Evaluate the specific methodology chosen.

For chemical analyses, as with any other measurement, it is important to evaluate a specific methodology for its ability to provide appropriate, sufficient and adequate data for a particular application. This evaluation normally has to consider a range of factors, including repeatability r, reproducibility R, and a measurement of uncertainty, especially for laboratories working under the rules in ISO/IEC 17025. For fire-effluent toxicity, these requirements are discussed in ISO 19706.

Two key parameters in the evaluation of a method (e.g. when it is required to compare different methods for a particular application) are

1) the lower concentrations of particular species which are able to be detected adequately (limits of detection), L_D, and

2) the lower concentrations of particular species which are able to be quantified adequately (limits of quantification), L_Q.

Knowledge of the L_Q value is essential when comparing small concentrations of fire effluent gases measured by different methods. Both the L_D and L_Q parameters in specific analytical methods are relevant to the assessment of the contribution of gases to a fractional effective dose (FED) or fractional effective concentration (FEC) calculation, as set out in ISO 13571. Both parameters are also important in the evaluation