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Validation methods for fire gas analyses —

Part 2: Intralaboratory validation of quantification methods

Méthode de validation des analyses de gaz d'incendie —

Partie 2: Validation intralaboratoire des méthode de d'analyse

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 92, *Fire safety*, Subcommittee SC 3, *Fire threat to people and the environment*.

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

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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 egress time (ASET). Related documents are also being developed in ISO TC92 SC3 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 a key factor in evaluating the quality of the quantitative data produced. Such a characterization is developed over four items.

Item 1: Define the objective of the analysis. Before undertaking a chemical analysis of fire effluent, the final objective of the analysis should be established. For example, the objective might be part of a fire safety engineering design of a building, validation of a numerical fire model, or determination of the toxic potency of the effluent from a particular combustible item.

Item 2: Determine the degree of accuracy and precision required from the analysis. Accuracy is dependent on a combination of the physical fire model being used, the sampling of the effluent and the analytical chemical technique. Precision means the tolerable uncertainty in the measured result. For example, in an FED (Fractional Effective Dose) calculation, where the individual contribution of a range of different species to the overall toxic potency of a fire effluent is estimated, interest might range from concentrations which might incapacitate people of average sensitivity to the effluent, to concentrations which show negligible toxic effect over a long exposure period.

Item 3: Select the appropriate chemical analytical methods, considering specificity, i.e. the other gases present. Guidance on options for measuring a wide variety of chemical species is provided in ISO 19701 and ISO 19702.

Item 4: Evaluate the suitability of the chosen method considering specificity. 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, reproducibility, and a measurement of uncertainty, especially for laboratories working under ISO 17025 rules. For fire effluent toxicity, these properties are discussed in ISO 19706.

Different methods may be deemed suitable for the particular application and for consistency in the interpretation of results from these different methods, it is also important to be able to compare the validity of the analytical technique used. In the field of fire effluents, many factors can affect the trueness and the fidelity of a measurement technique.

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Validation methods for fire gas analyses —

Part 2:
Intralaboratory validation of quantification methods

1 Scope

This document describes tools and techniques for use in validating the analysis of fire gases when an analytical method is developed in a laboratory. It complements ISO 12828-1, which deals with limits of quantification and detection.

The tools and techniques described can be applied to the measurement of quantities, concentrations (molar and mass), volume fractions, and concentration or volume fraction versus time analyses. Fire effluents are often a complex matrix of chemical species, strongly dependent on the materials involved in the fire, but also dependent on fire scenario parameters (see ISO 19706). With such a wide variety of conditions, the analytical techniques available will differ in terms of the influence of the matrix on the methods and on the concentration ranges which can be measured. The analytical techniques available are likely to differ significantly in several respects, such as their sensitivity to the matrix and the range of concentrations/volume fractions which can be reliably measured. For these reasons, a unique reference analytical technique for every fire effluent of interest is, in practical terms, difficult or impossible to achieve. The tools in this document allow verification of the reliable measurement ranges and conditions for the analysis of fire effluents, thereby enabling a comparison among various analytical techniques.

Examples of existing International Standards where the information contained in this document 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, ISO 16405, or their application to fire toxicity assessment using ISO 13571 and ISO 13344.

NOTE 1 The variable “concentration” is used throughout this document, but it can be replaced in all places with “volume fraction” without altering the meaning. This does not apply to the Annexes.

NOTE 2 Concentration can be calculated from volume fraction by multiplying by the density of the relevant gas at the relevant temperature and pressure.

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 12828-1:2011, *Validation method for fire gas analysis — Part 1: Limits of detection and quantification*

ISO 5479, *Statistical interpretation of data — Tests for departure from the normal distribution*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13943, ISO 5725-1, ISO 2854, ISO 2602, ISO 13571 and the following apply.

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

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

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

3.1
matrix (of fire effluents)
mixture of fire effluents in which the analyte of interest is present

Note 1 to entry: This includes all other species, solid, liquid and gas phases. It constitutes all components that could affect analysis, such as interfering species.

4 Symbols and abbreviated terms

y_0	Actual concentration of an analyte in a fire effluent
y_1	Concentration just after extraction by the sampling probe
y_2	Concentration after transportation to the conditioning system
y_3	Concentration at the entrance of the sensor
y_4	Concentration read by the sensing apparatus
$X_1 = y_0/y_1$	Sampling ratio; Because of the effectiveness of the sampling probe, X_1 might be more than 1 (see 6.2 for details)
$X_2 = y_1/y_2$	Transportation ratio (see 6.3 for details)
$X_3 = y_2/y_3$	Conditioning ratio; X_3 might be more than 1 (see 6.4 for details)
$X_4 = y_3/y_4$	Analysis ratio; X_4 might be more than 1 (see 6.5 for details)
y_m	Reported concentration of an analyte in the gas phase
y_i	One of a number of y_m values in a group
b_0	Zero order coefficient term in a regression; For a linear regression, b_0 is the intercept
b_1	First order coefficient term in a regression; For a linear regression, b_1 is the slope
b_2	Second order coefficient term in a regression.
$\hat{y}_i$	Predicted value for y_i, given by application of a regression model
$\bar{y}_i$	Mean value for y_i
p	Total number of measurements
df	Degrees of freedom; According to the context, several degrees of freedom could be defined
SCE	Sum of squares of deviations between measured values y_i and mean value $\bar{y}_i$
MS	Median square, corresponding to SCE divided by df

5 General considerations

5.1 Actual concentration and measured concentration

The objective of every chemical analysis used in fire science is to approach the actual concentration of an analyte, y_0, in fire effluents. The value of y_0 is unknown, as the only value measured is the concentration

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