

---

---

**Guidance for sampling and analysis  
of toxic gases and vapours in fire  
effluents using Fourier Transform  
Infrared (FTIR) spectroscopy**

*Lignes directrices pour l'analyse des gaz et des vapeur dans les effluents  
du feu par spectroscopie infrarouge à transformée de Fourier (IRTF)*





**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2015, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
copyright@iso.org  
www.iso.org

# Contents

	Page
<b>Foreword</b> .....	<b>v</b>
<b>Introduction</b> .....	<b>vi</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>2</b>
<b>3 Terms and definitions</b> .....	<b>2</b>
<b>4 Principles</b> .....	<b>3</b>
<b>5 Sampling</b> .....	<b>3</b>
5.1 General.....	3
5.2 Temperature of the sampling system.....	4
5.3 Filter systems.....	5
5.4 Sampling probes.....	6
5.4.1 General.....	6
5.4.2 Single hole probes.....	7
5.4.3 Multi hole probes.....	7
5.4.4 Probe positioning.....	7
5.5 Sampling line.....	8
5.6 Pump selection, position, and flow rate.....	9
5.7 Response time of the sampling system.....	10
5.8 Optical cell.....	10
<b>6 The FTIR spectrophotometer</b> .....	<b>11</b>
6.1 Spectrophotometer environment.....	11
6.2 Detector.....	11
6.3 IR-source.....	11
6.4 Mirror alignment and cleanliness.....	11
6.5 Spectrophotometer compartment.....	12
6.6 Spectral range limits.....	12
6.7 Resolution.....	12
<b>7 Calibration</b> .....	<b>12</b>
7.1 Background noise.....	12
7.2 Limits of detection and of quantification ( $L_D$ and $L_Q$ ).....	12
7.3 Calibration methods.....	13
7.4 Acquiring and collecting calibration standards.....	13
<b>8 Measurement procedure</b> .....	<b>13</b>
8.1 General.....	13
8.2 Daily checks and controls.....	13
8.2.1 General.....	13
8.2.2 Control of calibration method.....	13
8.2.3 Spectrophotometer sensitivity measurements.....	14
8.2.4 Sampling system tests.....	14
8.2.5 Control of the sampling flow rate.....	14
8.3 Preparation for sampling and analysis.....	15
8.4 Initial procedures immediately before a test.....	15
8.5 Procedures during sampling from a test.....	15
8.6 Procedures after a test.....	16
8.7 Data reduction.....	16
<b>9 Test report</b> .....	<b>16</b>
<b>10 Precision and accuracy</b> .....	<b>17</b>
10.1 General.....	17
10.2 $L_D$ and $L_Q$ .....	17
10.3 Repeatability and reproducibility.....	17

<b>Annex A (informative) FTIR theory</b> .....	<b>18</b>
<b>Annex B (informative) FTIR sampling systems</b> .....	<b>20</b>
<b>Annex C (informative) Analysis of filter(s), the sampling line and probe for effluent retention</b> .....	<b>25</b>
<b>Annex D (normative) Response time determination of the complete FTIR sampling system</b> .....	<b>26</b>
<b>Annex E (informative) Considerations for FTIR optical cell selection</b> .....	<b>29</b>
<b>Annex F (normative) Verification of FTIR optical cell performance</b> .....	<b>31</b>
<b>Annex G (informative) Spectrophotometer</b> .....	<b>33</b>
<b>Annex H (normative) Verification of spectrometer performance</b> .....	<b>37</b>
<b>Annex I (informative) Reference gases</b> .....	<b>41</b>
<b>Annex J (informative) Calibration methods</b> .....	<b>47</b>
<b>Annex K (informative) Recording reference spectra and building a calibration set</b> .....	<b>50</b>
<b>Annex L (informative) Repeatability and reproducibility</b> .....	<b>54</b>
<b>Annex M (informative) Examples of equipment and parameters</b> .....	<b>55</b>
<b>Bibliography</b> .....	<b>66</b>

## 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](http://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](http://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 WTO principles in the Technical Barriers to Trade (TBT), see the following URL: [Foreword — Supplementary information](#).

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

This second edition cancels and replaces the first edition (ISO 19702:2006), which has been technically revised.

## Introduction

Sampling and analysis of fire effluents is required for a variety of applications in life threat<sup>[4][11][26]</sup> and environmental impact from fires<sup>[6]</sup> assessments. The end result of these analyses is a list of chemical species and their concentrations in the effluent at a specific time or over a time interval and at a specific location, during (and possibly after), the period of generation of the effluents. Depending on the end use of these data, the requirements may range from a highly detailed, time-resolved, quantified, and validated list of chemical species to a simple estimate of a single compound or small range of compounds.

Although occasionally employing methods used in other fields (e.g. atmospheric pollution), obtaining relevant data often requires specialized sampling and analysis techniques, due to the complexity, reactivity and generally “hostile” nature of typical fire effluents, as well as the commonly observed rapid changes in concentrations with time and distance from the fire source.

The following typical properties of fire effluents render more “traditional” methods of sampling and analysis inappropriate:

- high temperatures of 1 000 °C or higher;
- presence of aerosols (i.e. solid and liquid particulates) with a wide range of particle sizes and distribution, together with adsorbed and absorbed chemical species;
- presence of condensable organic and inorganic vapours (e.g. water);
- high turbulence, with spatially and temporally variable concentrations;
- a very wide range of species and their concentrations, typically varying rapidly with time and location with respect to the fire source (or heating zone in the case of a “bench-top” physical fire model);
- presence of acidic/corrosive species;
- presence of water soluble species and/or or highly reactive species resulting in sampling losses.

The identification of these factors has led to the development of new methods or the adaptation of existing methods for the sampling and analysis of the gases and vapours in the effluent from fires and physical fire tests.

Common methods have emerged in recent years, and in some cases, standards have been published for selected gases and vapours. Much of this information is provided in ISO 19701, which presents a variety of methods for the sampling and analysis of individual gases of toxicological importance. Several methods are often needed to determine all the species of interest for fire hazard analysis.

Fourier Transform Infrared (FTIR) spectroscopy offers an improved procedure, principally through:

- single-method measurements of gases and vapours relevant to fire toxicology;
- time-resolved measurements over relatively short periods (i.e. concentrations of chemical species of interest), enabling the monitoring of chemical species development and decay throughout the fire or physical fire test; and
- relevant data concerning the presence of a toxicant which may be found in the stored FTIR spectra, in case a new toxicant should later be identified as important.

Although when published, ISO 19701 summarized the technique and some applications of FTIR in fire gas analysis, the method has since undergone considerable development and the requirements for obtaining reliable results have been established, using “best practice” procedures. This International Standard is developed by ISO TC 92, SC 3 to provide the requirements, which will include additional information.

FTIR can be used to analyse fire effluents using these two methods:

- a) open path analysis, where the infrared beam is directed across the effluent within and/or outside the fire test apparatus;

- b) extractive analysis, where a fraction of the effluent from a fire test apparatus is drawn continuously through a heated sampling system through the gas cell of the FTIR instrument, enabling remote measurement (e.g. IMO Resolution MSC.307(88)<sup>[9]</sup>).

Both procedures (and variants) have been successfully applied although the extractive analysis technique is far more common in fire effluent analysis.

Of particular relevance in the development of FTIR as a practical tool in fire gas analysis is SAFIR (Smoke Gas Analysis by Fourier Infrared Spectroscopy), a European Union-funded project<sup>[18][19]</sup> which focused on the testing and validation of an extractive FTIR method when used in a variety of situations. The results of this project formed the basis for the first version of this International Standard. This revised version has been updated with more recent information, e.g. References <sup>[8]</sup>, <sup>[15]</sup>, <sup>[21]</sup>, <sup>[25]</sup> and <sup>[27]</sup>.

It should be appreciated that any chemical analysis is selective in terms of chemical species determined and the accuracy and precision of quantitative measurements. Some chemical analytical methods may be appropriate for accurate determination of some species but less appropriate for other species. Thus, despite the ability of FTIR to measure a wide range of chemical species of interest in the field of life threat from fire, additional methods may also be required to determine all the species of interest for a particular application. However the use of FTIR analysis alone can provide data of sufficient quality to identify and calculate the concentrations of many of the chemical species that are important in toxic hazard assessment.





# Guidance for sampling and analysis of toxic gases and vapours in fire effluents using Fourier Transform Infrared (FTIR) spectroscopy

## 1 Scope

This International Standard specifies requirements and makes recommendations for sampling systems for use in small and large-scale fire tests, for the selection of parameters and use of the FTIR instrument itself and for collection and use of calibration spectra.

The primary purpose of the methods is to measure the concentrations of chemical species in fire effluents which may be used to

- a) provide data for use in combustion toxicity assessment without requiring biological studies,
- b) allow the calculation of yield data in fire characterisation studies,
- c) provide data for use in mathematical modelling of hazard to life from the fire effluent by characterising the effluent composition generated by physical fire models,
- d) characterise the effluent composition of small scale physical models and larger scale fires for comparative purposes,
- e) assist in the validation of numerical fire models,
- f) set the conditions for exposure in biological studies if required,
- g) monitor biological studies where used, and
- h) assist in the interpretation of biological studies where used.

This International Standard specifies principles of sampling and methods for the individual analysis, in fire effluents, of airborne volume fractions of carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>), hydrogen cyanide (HCN), hydrogen chloride (HCl), hydrogen bromide (HBr), nitric oxide (NO), nitrogen dioxide (NO<sub>2</sub>), and acrolein (CH<sub>2</sub>CHCHO).

In most common cases, a wide concentration range may be measured by an FTIR instrument. Typically, it is in the range from few µl/l to thousands of µl/l for HCl, HBr, HF, SO<sub>2</sub>, NO<sub>x</sub>, and HCN, and up to few per cent for CO, CO<sub>2</sub> and H<sub>2</sub>O. This list is only indicative and many other species could be added.<sup>[27]</sup> Although not specifically defined in this International Standard, as they were not specifically studied in the SAFIR project,<sup>[18]</sup> the method presented is also suitable for analysis of other gaseous species, including e.g. hydrogen fluoride (HF) and sulfur dioxide (SO<sub>2</sub>) with appropriate sampling methods.

Calibration methods are provided in this International Standard. Guidance is also given on the recommended cleaning, servicing and operating checks and procedures to be carried out on the FTIR instrument and the sampling systems which are considered essential to maintain the instrument in a suitable condition for use in fire effluent analysis.

Sampling is considered to be an integral part of the whole FTIR measurement methodology and recommendations are made for the design, maintenance and operation of suitable systems.

Conformance with this International Standard implies that:

- The sampling procedure used is in accordance with current internationally accepted “best practice” for the applications described.