
**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 vapeurs dans les effluents
du feu par spectroscopie infrarouge à transformée de Fourier (IRTF)*

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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 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^{[4][11][26]} and environmental impact from fires^[6] 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)[9]).

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[18][19] 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 [8], [15], [21], [25] and [27].

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.

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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 (CO₂), hydrogen cyanide (HCN), hydrogen chloride (HCl), hydrogen bromide (HBr), nitric oxide (NO), nitrogen dioxide (NO₂), and acrolein (CH₂CHCHO).

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, SO₂, NO_x, and HCN, and up to few per cent for CO, CO₂ and H₂O. This list is only indicative and many other species could be added.^[27] Although not specifically defined in this International Standard, as they were not specifically studied in the SAFIR project,^[18] the method presented is also suitable for analysis of other gaseous species, including e.g. hydrogen fluoride (HF) and sulfur dioxide (SO₂) 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.

- The analytical procedure has been carried out with due regard to the restraints imposed by the nature of the fire effluent atmosphere and the limitations of the FTIR methodology itself.

This International Standard only provides general recommendations for the sampling and analysis of fire effluents, based on best practice as determined from a wide variety of small- and large-scale standard and ad hoc fire test studies. The Standard may not be wholly applicable for use in specific published fire test methods where FTIR may be specified as a requirement for effluent sampling and analysis in that particular test. In these cases, the specific requirements for the sampling and analysis by FTIR may be published within the standard test procedures and should be followed. However, if such specific requirements have not been published, this edition of this International Standard may be used as a basis for acceptable results.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6286, *Molecular absorption spectrometry — Vocabulary — General — Apparatus*

ISO 6955, *Analytical spectroscopic methods — Flame emission, atomic absorption, and atomic fluorescence — Vocabulary*

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

ISO 13344, *Estimation of the lethal toxic potency of fire effluents*

ISO 13571, *Life-threatening components of fire — Guidelines for the estimation of time to compromised tenability in fires*

ISO 13943, *Fire safety — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13943, ISO 6286, and ISO 6955, as well as the following apply.

3.1 apodisation

mathematical process to correct deviations from the theoretical interferogram obtained in FTIR spectroscopy

Note 1 to entry: This process widens absorption bands and therefore lowers the resolution.

3.2 interferogram

pattern of variable radiation intensity resulting from the interaction of (usually) two beams of monochromatic electromagnetic radiation of the same wavelength when the beams have travelled over a different path length before combining

3.3 resolution

capacity of a spectrophotometer to distinguish between two adjacent frequencies (or wavenumbers), mathematically defined as that separation which produces two bands which are separated by at least half the intensity of one of them

Note 1 to entry: In infrared spectroscopy, two consecutive bands often have a different intensity. Then, resolution is generally expressed as the width at half height of a single absorption band recognized as “isolated and thin”.

Note 2 to entry: This is commonly expressed in units of cm^{-1} and is the smallest spacing between absorption peaks that can be resolved.

[SOURCE: ISO 6286]

3.4

spectrophotometer

instrument used to disperse electromagnetic radiant energy into a spectrum and measure certain properties such as wavelength, energy, or index of refraction, for example, as the basis for the qualitative and quantitative determination of chemical species which interact with the radiant energy in selective ways dependent on the species

3.5

trueness in wavelength

ability of a given spectrophotometer to provide an accurate radiant flux at a specific wavelength or wavenumber

[SOURCE: ISO 6286]

3.6

wavenumber

inverse of the wavelength (λ) of electromagnetic radiation

Note 1 to entry: $\nu = \frac{1}{\lambda}$

where ν (wavenumber) is expressed in reciprocal centimetres (cm^{-1}) and λ is normally expressed in μm . This entails a conversion factor of 10 000 in the calculation of wavenumber from wavelength.

3.7

zero filling

correction technique applied to sparse data to enable intermediate data points to be generated by a mathematical regression

4 Principles

A system for measuring concentrations of gases and vapours in fire effluent by the extractive FTIR technique consists of a sampling system, a “flow-through” optical cell, an infrared light source, an interferometer and an optical detector. Software is required to extract species identification and concentrations from the collected signals.

The sampling system is designed to deliver a gas flow to the FTIR optical cell that is as true a representation as possible of the effluent in a known region from which the sample is extracted. Inaccuracies, caused for example, by condensation or adsorption losses in the sampling tubing must be understood, quantified and applied to the final results.

A detailed description of the principles of infrared spectroscopy and FTIR is provided in many texts including References [12], [16] and [28]. [Annex A](#) gives the general outline of this principle.

The mathematics by which an FTIR spectrophotometer generates species concentrations is typically proprietary and varies among manufacturers and models. Obtaining accurate concentrations requires calibration, examination of the spectroscopic peak shapes and separations, and perhaps verification of some concentrations using a second analytical technique.

5 Sampling

5.1 General

The requirements for the sampling systems given in this International Standard are in most cases generally applicable both for large-scale and small-scale testing but details of sampling requirements

for specific tests are not included. This detail is to be provided with the Standards which describe these specific tests. Parts of the sampling system to be designed for a specific test apparatus or sampling location within that apparatus include the sampling probe and the filter, the length and maintained temperature of the sampling line and the sample flow. These sampling parameters are normally specified to match the volume and geometry of the optical cell in the FTIR spectrometer.[18][19][25][27] Examples of sampling systems are given in [Annex B](#).

The sampling probe is designed to extract fully representative samples from the test apparatus and/or the fire effluent stream and be constructed from material/s which will be inert to the effluents being measured. Guidance for probe design is provided in [5.4](#).

The filter in the sampling train shall capture particles to avoid deposits on mirrors in the FTIR cell. Guidance for the selection of filters is provided in [5.3](#).

The design of the sampling line and the flow characteristics affect the time of the concentration measurements. Guidance for sampling line design and sampling flow selection are provided in [5.5](#) to [5.7](#).

The cell volume and geometry set restrictions on the sample flow used and thus on the time resolution of the measurements and uniformity of the gas in the optical cell. Guidance for cell selection is provided in [5.8](#).

For safety reasons, the extracted and analysed gases are to be returned to the exhaust duct of the fire test fixture or otherwise disposed of in a safe way that does not endanger personnel and to a location that does not disturb measurements. With a standard test which has a requirement or option to use FTIR gas analysis, it is anticipated that this International Standard will provide specific requirements for the safe disposal of the effluent stream after measurement (e.g. whether the effluents will be recirculated or safely discharged elsewhere).

The requirement for each part of the sampling system is described separately in the following subclauses. [Annex M](#) gives examples of proprietary FTIR equipment and sampling systems that have been proved to be suitable for fire effluent analysis.

NOTE Sampling in large-scale tests such as in ISO 9705 and in large open calorimeters (ISO 24473) is described in Reference [3].

5.2 Temperature of the sampling system

5.2.1 The sampling system shall be heated to a temperature which shall be set between 150 °C and the temperature of the gas cell (recommended maximum 190 °C).

5.2.2 The temperature of the entire sampling system shall be kept at an even temperature and not be higher than the gas cell temperature to avoid condensation in the cell.

NOTE 1 It is important that the gas in the sampling system is heated to a temperature as close as possible to the set-temperature of the sampling system. A procedure for checking the gas temperature in the optical cell is given in [F.1](#).

The temperature of the system should optimally be even but the most important condition is that the temperature of the gas cell is the highest in the sampling system.

The temperature shall be high enough to prevent condensation and losses due to the dissolving of soluble fire gases from the sample. The whole sampling system shall be kept at a minimum temperature of 150 °C, although the recommended temperature is 180 °C.[18][19]

NOTE 2 The temperature must not exceed 200 °C at any point between sampling point and gas cell, as chemical reactions between components of the sampled fire effluents could lead to a modification of gas composition above this temperature. Nitrogen oxides are particularly sensitive to this parameter.

5.3 Filter systems

5.3.1 A gas cell with internal mirrors shall be protected from soot/particle deposition from the sampled effluent, with a particulate filter.

5.3.2 The filter shall normally be placed directly after the sampling probe and prior to the heated sampling line.

5.3.3 The filter shall be uniformly heated in a housing to prevent condensation of liquids

NOTE 1 Correct placement of filters in the sample train is important, since incorrect filter placement can result in significant sample losses (e.g. through gas adsorption on particles) which leads to distorted concentration/time profiles of the measured species.

NOTE 2 For certain designs of filter and housing, placing the filter directly between the probe and the sample line can in some cases cause problems in the early stages of a compartment fire test. The filter medium, even though heated externally may be cooled internally by the cool sample gas stream. A significant temperature differential may then develop between the internal filter medium and the heated filter housing. This creates conditions favourable for the condensation of water, water-soluble acid gases and as well as high boiling point vapours within the filter medium. The trapped gases may be released as the filter medium temperature increases, further distorting the concentration histories. To avoid this, a sufficiently long sample line, with the appropriate number of heated segments can be used to preheat the sample gases to the intended sample line temperature prior to contact with the filter medium.

5.3.4 The filter shall be heated to a temperature which shall be set between 150 °C and the temperature of the spectrometer gas cell.

NOTE The recommended maximum temperature of the spectrometer gas cell is 190 °C.

5.3.5 The type of filter recommended is a cylindrical filter as opposed to a plane filter.

NOTE A cylindrical filter has a large area which gives a high filtration capacity and this is especially important for the primary or pre-filter. Cylindrical ceramic filters with a length of 75 mm, a diameter of 31 mm and a porosity of 2 µm have been successfully used in various types of fire tests.^{[13][14][18]} Alternative materials for cylindrical filters are polytetrafluoroethylene (PTFE), glass fibre, and stainless steel.

Glass wool, ceramic, and stainless steel filters could induce losses of hydrogen fluoride and should be avoided in the sampling train when hydrogen fluoride is likely to be present in the effluent stream.

The porosity and size of the filter shall be chosen as a compromise between sufficient entrapment of soot and other particles without premature clogging of the filter, using the above recommendations as a guide.

5.3.6 Plane surface filters may alternatively be used.

NOTE 1 Plane surface filters generally have a lower filtration capacity compared to cylindrical filters with more likelihood of early blockage occurring. These can, however, be used effectively as secondary filters following a cylindrical pre-filter.

NOTE 2 Where no pre-filter is used, a 1 µm porosity, 47 mm to 75 mm diameter filter is the best compromise for plane surface filters (compared to 0,45 µm and/or 5 µm for PTFE), although other porosities and diameters may be successfully used.^[18] Alternatively, 1µm or 2 µm stainless steel filter has been reported to be successful. Stainless steel filters can be cleaned and regenerated by calcinations. References ^[15] and ^[21] provide details on filters selection.

5.3.7 Filters shall be analysed for the presence of acid gases if these are to be measured and losses in the filter can be expected.

NOTE 1 Acid gas losses in the filter are due to adsorption onto the soot and or liquid particles in the fire plume or onto the soot retained by the filter. The adsorption losses are especially important when measuring low concentrations of acid gases or when sampling over a short time period. Under these conditions acid gases may initially be removed from the sampled effluent stream until the adsorption sites in the filter become saturated. After this stage most of the acid gas component will be passed on to the sampling line. This results in a spurious release of acid gas from the filter over time which does not accurately follow the release of the acid gases from the sampled atmosphere. To estimate these losses, a procedure to analyse the gases retained on the filter is given in [Annex C](#).

NOTE 2 Analysis of losses of hydrogen chloride (HCl) in filters was made in the SAFIR project.^[18] The chemical analysis of the filters in door opening measurements in ISO 9705 test chamber experiments showed that there were low losses (losses of between 0,4 % and 1,3 % relative to the total sampled amount) of HCl in cylindrical ceramic filters. A slightly greater retention of HCl was seen for a planar glass fibre filter (losses of between 1,1 and 5,8 relative per cent). In both cases the higher relative losses of HCl were found when the measured concentration of HCl was low.

The kinetics of absorption onto the filter has not been characterized and it is therefore difficult to establish a true time/concentration profile of the acidic species in the sampled fire atmosphere. However, when the total amount of gas produced during the combustion is required, the quantity measured on the filter should be added to the total amount measured by FTIR (see [Annex C](#)). It is stressed that the adsorption on the filter has an effect on the accuracy of the time-dependent measurement of the gases evolved during combustion. When a time-dependent measurement is required, information on gases analysed on the filter should be considered in order to estimate the potential effect on the concentration/time data.

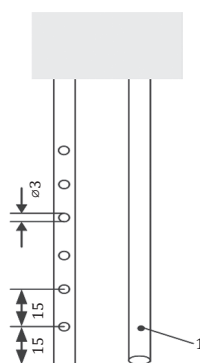
5.3.8 It should be noted that some filter materials can contain chlorine. The chlorine content of a clean unused filter should be measured to provide a baseline for the determination of the level of adsorbed chlorine from the fire gases.

5.4 Sampling probes

5.4.1 General

Two types of probes can be distinguished: “single-hole” and “multi-hole”. The choice depends on the physical fire model from which the sample is being extracted in order to obtain a representative mixture of effluent. Examples of the two types of probes are shown in [Figure 1](#).

Dimensions in millimetres



Key

1 inner diameter

NOTE An inner diameter of a single-hole probe of 4 mm has been shown to work effectively.^[18]

Figure 1 — Schematic of multi-hole and single-hole probes for sampling in an exhaust duct

5.4.1.1 The probe shall be made of material resistant to corrosion and must not otherwise react significantly with the gases to be analysed.^{[18][19]}

NOTE 1 PTFE or perfluoroalkoxy polymer (PFA) is recommended as the contact material for the effluent stream.

NOTE 2 A probe of PTFE is generally inert and would have a low potential for causing losses. The flexibility of the material could be a disadvantage in certain applications, for example where precise positioning is necessary. A further disadvantage is that PTFE is not suitable for higher temperatures, as high temperatures influences the material properties and at sufficiently high temperatures thermal degradation occurs.

NOTE 3 Quartz and/or stainless steels can be used for some effluents, but react with hydrogen fluoride (HF).

NOTE 4 A stainless steel probe placed in a position with initially a temperature lower than the sampled effluent may cause condensation of products including water. This will lead to sample losses, an important example of a gas species prone to losses being hydrogen chloride (HCl).

NOTE 5 A practical probe can be constructed using stainless steel tubing lined with a narrower-bore PTFE tube or lined with epoxy resin. The stainless steel tube will facilitate direct ohmic heating (i.e. passing a low voltage electric current through the metal tube) and the PTFE or epoxy lining will further reduce losses on the probe surfaces.

5.4.1.2 The probe shall be cleaned before each test to ensure that the holes do not become partially or wholly blocked

5.4.2 Single hole probes

5.4.2.1 Where the effluent stream is likely to be well mixed/homogenous a single-hole probe is appropriate

5.4.2.2 In the case of a flow-through type of physical fire model (e.g. a tube furnace^[7]), the extraction probe takes a proportion of the total homogenous flow from the apparatus and directs it to the FTIR spectrometer. A single-hole probe is therefore appropriate for such applications

5.4.2.3 The internal diameter of the extraction probe shall not be less than 3 mm

NOTE A single-hole probe is used in many standardized test procedures where the effluent stream is assumed well mixed and homogenous e.g. Reference ^[9]. However, due to stratification effects in fire chambers and in extract ducts where laminar flow is present, (i.e. leading to poor mixing) the gas concentration may not be uniform over the chamber or duct volume and the measured gas concentration is thus dependent on the position of the single-hole probe. In these situations the use of a multi-hole probe is appropriate where the non-uniform gases can be sampled over a more representative area.

5.4.3 Multi hole probes

5.4.3.1 When stratification is present in the effluents from the fire, a multi-hole probe is preferred to obtain a representative sample from across the stratified layer.

5.4.3.2 A minimum hole diameter of 3 mm is recommended for multi-hole probes for most applications.

5.4.3.3 The holes in a multi-hole probe shall be directed downstream in the effluent stream to avoid direct impingement of soot particles and thus reducing the possibility of blocking of the holes.

5.4.4 Probe positioning

5.4.4.1 In order to avoid perturbation in the effluent, the rate of volumetric sampling shall be a small fraction (<25 %) of the total flow rate in flow-through apparatus or the total volume in a fixed volume apparatus.