
**Test method for determination of
gas concentrations in ISO 5659-2
using Fourier transform infrared
spectroscopy**

*Méthode pour déterminer les concentrations des gaz émis lors de l'essai
ISO 5659-2 par spectroscopie infrarouge à transformée de Fourier*

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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).

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This document was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

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Introduction

This document describes a test method to generate and analyse effluents produced during pyrolysis and flaming combustion of samples from materials and products exposed in a single-chamber test scenario as defined in ISO 5659-2.

This document establishes a continuous measurement procedure (i.e. analysis of time-related emissions) for selected gases emitted from pyrolysis and combustion of materials exposed to ISO 5659-2 physical fire model. It produces data as gas volume ($\mu\text{L}/\text{L}$) or mass (mg/m^3) concentration versus time.

This test method is not designed to provide input data for Fire Safety Engineering, as the fire stages included in ISO 19706 are not defined according to time in the considered physical fire model and depend on product fire behaviour during the test. This test method is limited to a prescriptive approach which may be used in combination with existing reaction-to-fire tests (ignitability, spread of flame, heat release rate).

No consideration of further assessment, e.g. toxicity assessment, is proposed in this document. Other methods described in specific standards (e.g. ISO 13344, EN 45545-2) or specific codes (e.g. IMO FTP code) could be used for that kind of interpretation and to complete conditions of use of this document.

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Test method for determination of gas concentrations in ISO 5659-2 using Fourier transform infrared spectroscopy

1 Scope

This document specifies a test method suitable to analyse effluents produced during pyrolysis and combustion of samples and products tested according to ISO 5659-2. The specified test method is based on Fourier-transform infrared (FTIR) spectroscopy described in ISO 19702, with additional information on the test apparatus and analyser calibration suitable for its application to this physical fire model. This document is intended to be used in conjunction with ISO 5659-2 and ISO 19702.

The test method provides time-resolved gas concentrations during the whole of an ISO 5659-2 test.

This document does not address the accuracy of this fire model for any product application, nor does it address the accuracy of the gas concentrations relative to any real-scale fire tests or fire scenarios. For future conversion of this document into an International Standard, an interlaboratory trial is intended to be conducted to replace [Annex B](#).

This document does not include any toxicity assessment or provide input data for fire safety engineering.

As combustion conditions vary depending on the oxygen consumption rate in the enclosure during the ISO 5659-2 test, this physical fire model is not recognised as being representative of any specific fire scenario. Therefore, it is difficult to compare test results with real-scale fire conditions. As a consequence, if this test method is used for comparison among materials or products, it is intended to be done in combination with other fire tests.

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 5659-2:2017, *Plastics — Smoke generation — Part 2: Determination of optical density by a single-chamber test*

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

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

ISO 13943, *Fire Safety — Vocabulary*

ISO 19702:2015, *Guidance for sampling and analysis of toxic gases and vapours in fire effluents using Fourier Transform Infrared (FTIR) spectroscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13943, ISO 5659-2 and ISO 19702 apply.

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

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Principle

Studies that established the technical background of this document are detailed in references [1] and [2].

Fire effluents are continuously sampled from a cumulative smoke chamber (ISO 5659-2). The gas sampling flow shall be such that the sample represents the composition of the atmosphere in the chamber, and that any effect of gas sampling systems (such as filters, probes, pipes, tubes and pumps) is minimized. A filtering system prevents smoke particles from entering the cell of the gas analyser. The concentrations of specified gases in the sampled effluent flow are determined using FTIR spectroscopy following ISO 19702.

The amount of atmosphere drawn from the chamber and used for the FTIR analysis (about 1,5 L/min for 20 min) shall be kept within the limits which enable compensation of the sampling by expansion of air due to the thermal effects of the radiant cone and the combustion of the test specimen. In this way, the chamber pressure will be kept relatively constant throughout the sampling period.

The travelling time and distance of fire effluent through the gas sampling system shall be minimized.

5 Apparatus for combustion of test specimen and for cone radiator calibration

The test apparatus specified in ISO 5659-2 shall be used.

6 Gas sampling system

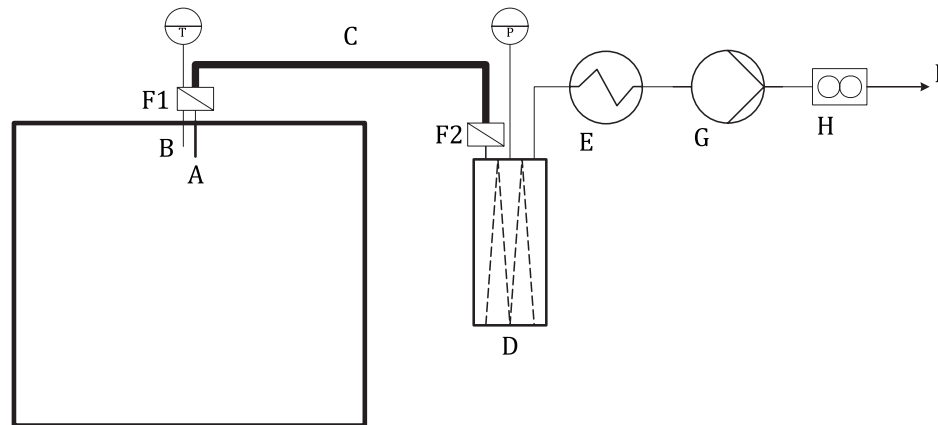
6.1 General arrangement

General arrangement of gas sampling system is specified hereafter in addition to ISO 19702 requirements. The gas sampling system shall consist of a sampling probe, a main filter, a gas sampling line, a secondary filter, a gas cell, a pressure transducer, an optional cooler, a pump and a flowmeter. The main filter shall be located directly after the probe. The gas analyser shall be located after the end of the sampling line and up-stream of the pump. An example of suitable sampling system is shown in [Figure 1](#). Other arrangements are possible, if they respect key points of this standard: flow rate conditions, main heated filter just after the sampling probe and a heated zone from outlet of the smoke density chamber to the outlet of the FTIR gas cell.

The smoke density chamber shall be equipped with a pressure transducer, which allows recording internal pressure P_{chamber} as function of time.

A system which uses a higher extraction rate and re-circulation of the extracted gases back into the test apparatus has been experimented by some laboratories successfully when the pressure of the box is kept relatively constant inside the box. For such systems, the user shall estimate the deviation from the general arrangement described in this document. The ISO 12828 series may be used for such assessment.

NOTE A valve can be added upstream or downstream of the pump, to facilitate the pressure regulation in the gas cell.

**Key**

A	ISO 5659-2 smoke chamber and sampling probe, see 6.2	E	gas cooler
B	thermocouple extremity, see 6.2	G	pump
F1	3-way valve and main heated filter, see 6.3	H	flowmeter
C	heated sampling line, see 6.4	I	to exhaust, at atmospheric pressure
F2	secondary heated filter, see 6.5	P	pressure transducer
D	FTIR heated gas cell, see 6.6	T	thermocouple transducer

Figure 1 — Schematic of an example layout of sampling system

6.2 Sampling probe

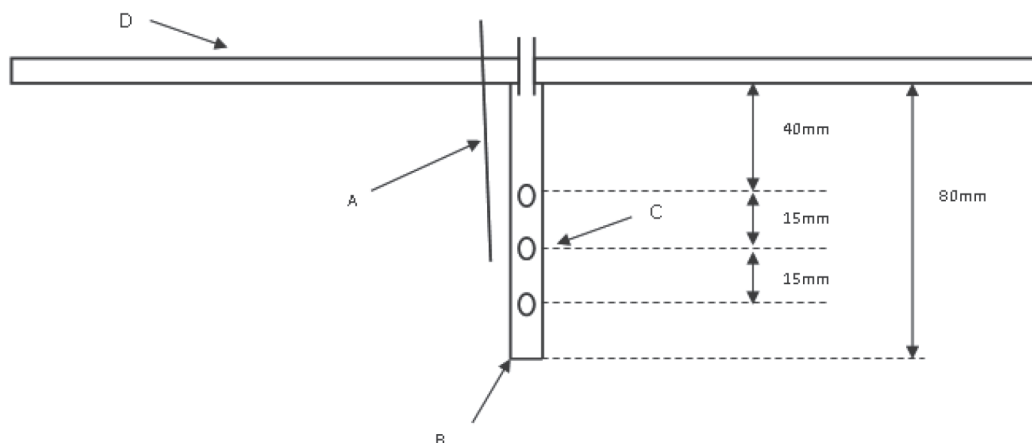
The internal probe shall be made from a 5 mm internal diameter stainless steel tube with a closed end, as shown in Figure 2. It shall be fixed in the central point of the chamber roof and projected into the chamber by 80 mm from the chamber ceiling.

The probe shall have 3 sampling holes of 2 mm diameter, facing toward the rear of the chamber, as shown in Figure 2, positioned at 40 mm, 55 mm and 70 mm measured from the internal ceiling of the chamber.

NOTE Some acid gases can react with the interior surface of the probe, resulting in loss of these gases. This is especially important (in proportion) for low concentrations.

Close to the central hole on the internal probe, a shielded thermocouple (K type, maximum diameter 2 mm) shall be placed at a distance of (8 ± 2) mm from the hole, to measure the temperature of the gas being sampled.

The temperature shall be recorded when the sampling has been made in order to calculate the mass concentration of gas species.



Key

- A thermocouple
- B close ended pipe
- C 3 holes of 2 mm diameter, directed to the back side
- D ceiling of the chamber

Figure 2 — Schematic of an example layout of sampling system

6.3 Main filter

The FTIR cell shall be protected by a filter unit from contamination of soot and other solid particles that are often contained in fire effluents. The filter unit shall be such that the filter element can be changed. ISO 19702:2015, 5.3 describes characteristics of suitable filter units.

The main filter unit shall be placed between the chamber and the sampling line, immediately after the 3-way valve placed after the sampling probe (See F1 in Figure 1). The temperature of the filtering system shall be set to $(180 \pm 10) ^\circ\text{C}$.

NOTE A filtering system constituted of a cylindrical PTFE cartridge of 30 mm diameter and 75 mm length with porosity of $2 \mu\text{m}$ inside a heated housing has been found suitable for the purpose of this analysis.

The use of PTFE is recommended as it is not reactive with fire effluents. Fibre glass is often inappropriate as it is known to react with HF, and ceramic wool is often inappropriate as it is known to absorb hydrogen halides even at high temperature.

6.4 Sampling line before gas cell

The sampling line used between the main filter and the FTIR gas cell shall be made of a heated flexible PTFE tube. The sampling line shall have an inner diameter of $(4,0 \pm 0,2) \text{ mm}$ and a maximum length of 3 m. The temperature of the sampling line shall be $(180 \pm 10) ^\circ\text{C}$. The sampling line shall be manufactured so that the PTFE tube is able to be replaced as needed.

6.5 Secondary filter

To increase the level of protection of internal mirrors, a secondary filter shall be placed just before FTIR gas cell. This secondary filter shall be heated to the same temperature as the sampling line and gas cell.

NOTE A small circular planar filter (47 mm diameter) using a $1 \mu\text{m}$ porosity PTFE membrane has been found suitable as secondary filter.

6.6 FTIR gas cell

The gas cell used shall have a volume not greater than 0,5 L. The temperature of the gas cell shall be (180 ± 10) °C. Pressure shall be monitored and corrected, in order to maintain pressure conditions during the test identical to the calibration pressure with a maximum deviation of $\pm 1,33$ kPa (10 torr), as specified in ISO 19702.

The renewal of gas in the cell is at least 3 renewals per minute. The response time of the analysis, determined according ISO 19702, shall be short enough to permit at least acquisition of 3 spectra per minute so that the interval between spectra is less than or equal to 20 s. Other systems shall be allowed if they demonstrate the compliance with these performance requirements.

NOTE 1 Gas cells with a volume from 0,2 L to 0,4 L and an optical path length from 2 m up to 5 m have been found suitable for the purpose of this document.

NOTE 2 torr is a non-SI unit frequently used in spectroscopy. 1 atm = 101 325 Pa = 1 torr.

6.7 Conditioning of sampling flow and pump capacity

The flow rate shall be maintained at $(1,5 \pm 0,1)$ L/min, using a flowmeter connected to an outlet at ambient pressure. The flow rate shall be maintained using a manual regulation valve or an automatic flow control system. The temperature of the gas entering in the flowmeter shall be less than 30 °C.

NOTE A gas cooler followed by a pump with a capacity of at least four times the inner volume of the gas cell plus gas sampling line per minute has been found suitable.

6.8 Sampling flow rate

The sampling flow rate shall be maintained to $(1,5 \pm 0,1)$ L/min during the test.

NOTE A sampling flow rate of $(1,5 \pm 0,1)$ L/min has been found suitable for the following reasons^[2]:

- 1) No influence has been found on smoke density measurement according to ISO 5659-2.
- 2) The volume drawn out of the chamber during 20 min test period is limited so as to avoid any under-pressure effects inside the smoke chamber.

6.9 FTIR Spectrometer

The following FTIR spectrometer parameters are required for the application of this document:

- an IR source stabilized at high intensity and temperature;
- a resolution better than or equal to 4 cm^{-1} over a range between 600 cm^{-1} and $4\,400 \text{ cm}^{-1}$;
A resolution of $0,5 \text{ cm}^{-1}$ is recommended to correct from interference.
- a measurement interval (interval between spectra) ≤ 20 s.

The Minimum Detection Limit (MDL) for gas species of interest depends on the type of gas, and shall be determined according to ISO 12828-1.

A MDL $\leq 15 \times 10^{-6}$ L/L ($\leq 15 \mu\text{L/L}$) is suggested for the majority of species, except for carbon dioxide for which MDL is usually $< 300 \times 10^{-6}$ L/L ($< 300 \mu\text{L/L}$).