

---

---

## FTIR analysis of fire effluents in cone calorimeter tests

*Analyse par FTIR des effluents du feu dans les essais au calorimètre à cône*

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[ISO/PRE TS 21397](https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397)

<https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397>

# PROOF / ÉPREUVE

---

---



## iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO/PRE TS 21397](https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397)

<https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397>



### **COPYRIGHT PROTECTED DOCUMENT**

© ISO 2021

All rights reserved. Unless otherwise specified, or required in the context of its implementation, 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  
CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

	Page
Foreword.....	iv
Introduction.....	v
<b>1 Scope.....</b>	<b>1</b>
<b>2 Normative references.....</b>	<b>1</b>
<b>3 Terms and definitions.....</b>	<b>1</b>
<b>4 Symbols.....</b>	<b>1</b>
<b>5 Principle.....</b>	<b>2</b>
<b>6 Apparatus.....</b>	<b>2</b>
<b>7 Gas measurement system.....</b>	<b>2</b>
7.1 General arrangement.....	2
7.2 Sampling probe.....	4
7.3 Primary filter.....	5
7.4 Sampling line.....	6
7.5 Gas cell.....	6
7.6 Pump and sampling flow rate.....	6
7.7 FTIR spectrometer.....	6
7.8 Detector.....	7
<b>8 Suitability of a product for testing.....</b>	<b>7</b>
<b>9 Specimen preparation.....</b>	<b>7</b>
<b>10 Test environment.....</b>	<b>7</b>
<b>11 Calibrations.....</b>	<b>7</b>
11.1 Calibration of cone calorimeter.....	7
11.2 Gas analyser calibration.....	7
<b>12 Test procedure.....</b>	<b>7</b>
12.1 General precautions.....	7
12.2 Operation before each test.....	8
12.3 Operation during a test.....	8
<b>13 Calculations.....</b>	<b>8</b>
13.1 General.....	8
13.2 Total mass loss.....	9
13.3 Mass of gas evolved.....	9
13.4 Yield of gas.....	10
13.5 Upper limits.....	10
<b>14 Test report.....</b>	<b>10</b>
<b>Annex A (informative) Examples of the use of FTIR with the cone calorimeter.....</b>	<b>11</b>
<b>Bibliography.....</b>	<b>12</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 of the voluntary nature of standards, 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 [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

The composition of the effluent from an enclosure fire is determined by the combustible items in the enclosure, such as interior furnishings and wall linings, and the fire conditions within the enclosure. To represent any product involved in any fire scenario, the ideal fire test specimen is the complete item, and the ideal test is one conducted in an enclosure of appropriate size. Unfortunately, real-scale testing of commercial products is not generally economically feasible. This document describes an indicative approach for obtaining gas yields under specific fire conditions. It involves the use of a small-scale combustor in which a small test specimen is exposed to a pre-defined radiative heat flux. The test specimen should be representative of the finished product

This document provides a method for continuous quantification of gases (i.e. time-resolved gas analysis) emitted from a test specimen exposed to irradiance in a cone calorimeter, after extraction of effluent gas to a FTIR gas analyser. It produces data as the gas volume fraction ( $\mu\text{L}/\text{L}$ ) or gas yield ( $\text{mg}/\text{kg}$ ) during the test period. Data generated allow a better understanding of gas emissions during cone calorimeter tests, which can be useful for Fire Safety Engineering and for material development. The repeatability/reproducibility results from an interlaboratory trial on the method are found in [Annex A](#). Some examples of FTIR analysers used with cone calorimeters are presented in Table A.1.

The cone calorimeter uses a well-ventilated physical fire model, so results are only relevant for this scenario. Toxicity assessment for materials or products are not covered in this document and interpretation of data is covered separately in ISO 13571 or ISO 13344.

## iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO/PRE TS 21397](#)

<https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397>

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

ISO/PRE TS 21397

<https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397>

# FTIR analysis of fire effluents in cone calorimeter tests

## 1 Scope

This document specifies a method for determining the kinetics and yields of gaseous emissions from a specimen exposed to radiant heat in a cone calorimeter. Gas yields are determined by exposing small representative specimens to an external heat flux with or without spark ignition. The concentrations of specific gases in the effluent (smoke) are measured. In combination with calculated masses of gases, their yields from the specimen mass, mass loss or mass loss rate can be determined. This document uses Fourier-Transform Infrared (FTIR) spectroscopy as described in ISO 19702, with additional information on the test apparatus and gas analyser suitable for this specific application.

## 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 13943, *Fire safety — Vocabulary*

ISO 5660-1:2015, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 1: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)*

ISO 19702, *Guidance for sampling and analysis of toxic gases and vapours in fire effluents using Fourier Transform Infrared (FTIR) spectroscopy* ISO/PRF TS 21397

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

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

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13943, ISO 5660-1, ISO 19702, ISO 12828-1 and ISO 12828-2 apply.

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

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

## 4 Symbols

Symbol	Designations	Unit
$\Delta m$	total mass loss of the test specimen	g
$\dot{m}_g$	mass flow rate of sampled gas species of interest	g s <sup>-1</sup>
$m_g$	total mass produced of sampled gas species of interest	g
$m_{te}$	mass at test end	g
$M_g$	molecular weight of gas of interest	g mol <sup>-1</sup>
$t$	is the time from ignition( $t_i$ ) to extinction( $t_e$ )	s

Symbol	Designations	Unit
$t_i$	time to ignition (onset of sustained flaming)	s
$t_e$	time of extinction	s
$\dot{V}_{298}$	volume flow rate in exhaust duct at 0,1 MPa and 25 °C	m <sup>3</sup> s <sup>-1</sup>
$\dot{V}_g$	volume flow rate of sampled gas species at 0,1 MPa and 25 °C	m <sup>3</sup> s <sup>-1</sup>
$V_g$	total volume of sampled gas species at 0,1 MPa and 25 °C	m <sup>3</sup>
$Y_g$	species yield of gas	g g <sup>-1</sup>
$\phi_g$	volume fraction of gas	dimensionless
$A$	gas density	kg m <sup>-3</sup>

## 5 Principle

This document specifies a method for measurement of gas yields from small test specimens by exposing them to an incident heat flux in a cone calorimeter. The concentrations ( $\mu\text{L/L}$ ) of specific gases presented in the effluents are determined via FTIR gas analysis, and their yields are determined in combination with the specimen mass loss.

## 6 Apparatus

The test apparatus is specified in ISO 5660-1 and the gas measurement system described in [Clause 7](#) shall be used.

iTeh STANDARD PREVIEW  
(standards.iteh.ai)

## 7 Gas measurement system

[ISO/PRE TS 21397](#)

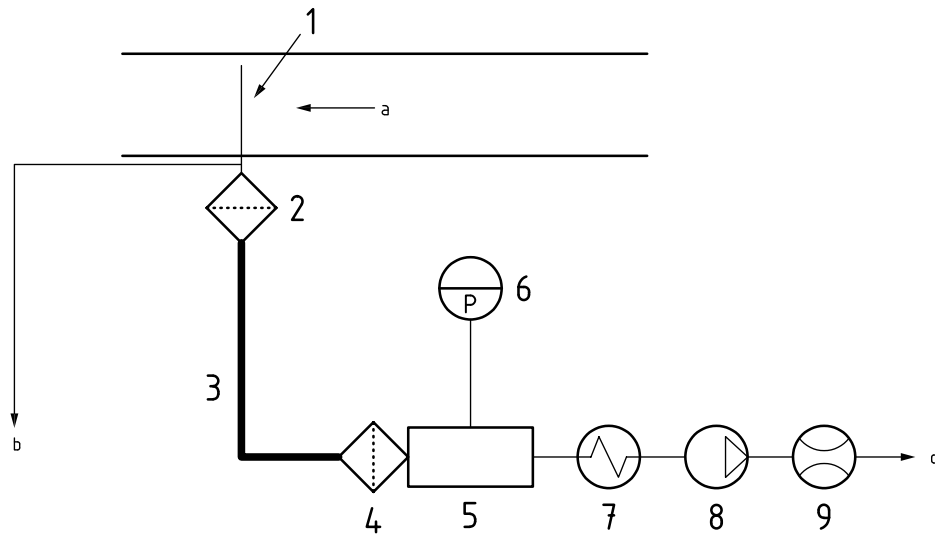
<https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397>

### 7.1 General arrangement

The gas sampling system shall consist of a sampling probe, a heated primary filter, a heated gas sampling line, a heated gas cell fitted with a gas pressure transducer, a pump and flow meter. An optional heated secondary filter and an optional gas cooler may also be fitted. The sampling system shall be heated to an even temperature which shall not be greater than the gas cell temperature in order to avoid condensation in the cell. Other conditions not described in this document shall be in accordance with ISO 19702. Examples of alternative sampling system schematics are provided in [Figure 1](#) and [Figure 2](#) respectively.

A procedure to determine the response time for the complete FTIR gas measurement system is given in ISO 19702:2015, Annex D.



**Key**

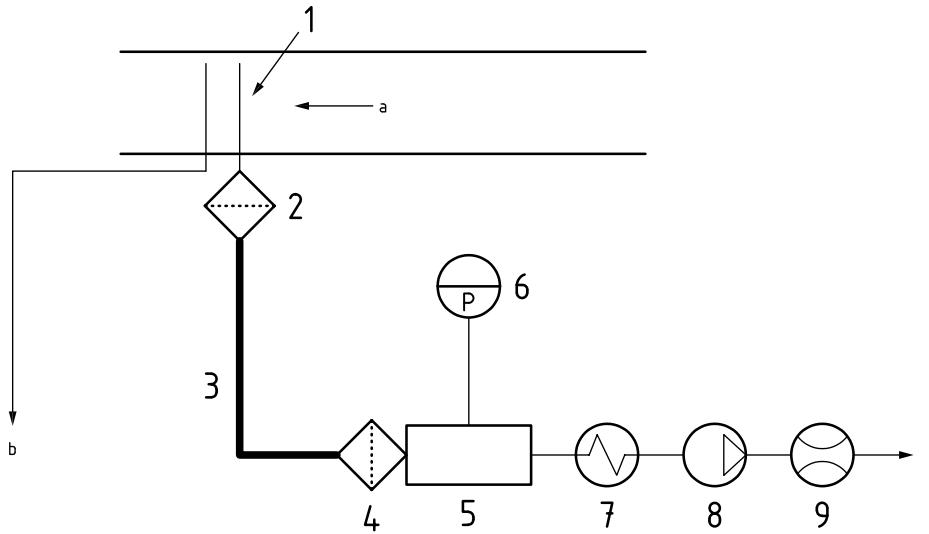
- 1 ISO 5660-1 gas sampling ring probe
- 2 heated primary filter
- 3 heated line for gas sample transport
- 4 heated secondary filter
- 5 heated FTIR gas cell
- 6 pressure transducer
- 7 optional gas cooler
- 8 sampling pump (here located downstream of the gas cell; upstream location is also allowed but requires a heated pump head)
- 9 flow meter
- a Direction of Combustion gas flow.
- b To O<sub>2</sub>, CO<sub>2</sub> and CO analyser.
- c To exhaust.

iTeh STANDARD PREVIEW  
(standards.iteh.ai)

[ISO/PREF TS 21397](https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397)

<https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397>

**Figure 1 — Example schematic diagram of sampling system when using the ISO 5660-1 ring probe fitted with a T-connection for sampling to FTIR analysis**



**Key**

- 1 linear multi-hole sampling probe fitted in the cone calorimeter exhaust duct
- 2 heated primary filter
- 3 heated line for gas sample transport
- 4 heated secondary filter
- 5 heated FTIR gas cell
- 6 pressure transducer
- 7 optional gas cooler
- 8 sampling pump (here located downstream of the gas cell; upstream location is also allowed but requires a heated pump head)
- 9 flow meter
- a Direction of Combustion gas flow.
- b To O<sub>2</sub>, CO<sub>2</sub> and CO analyser.
- c To exhaust.

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

<https://standards.iteh.ai/catalog/standards/sist/f4d4dcc2-9d72-42ad-a56f-fbb83552a9cc/iso-prf-ts-21397>

**Figure 2 — Example of schematic diagram of sampling system when using a linear multi-hole probe for sampling to FTIR**

**7.2 Sampling probe**

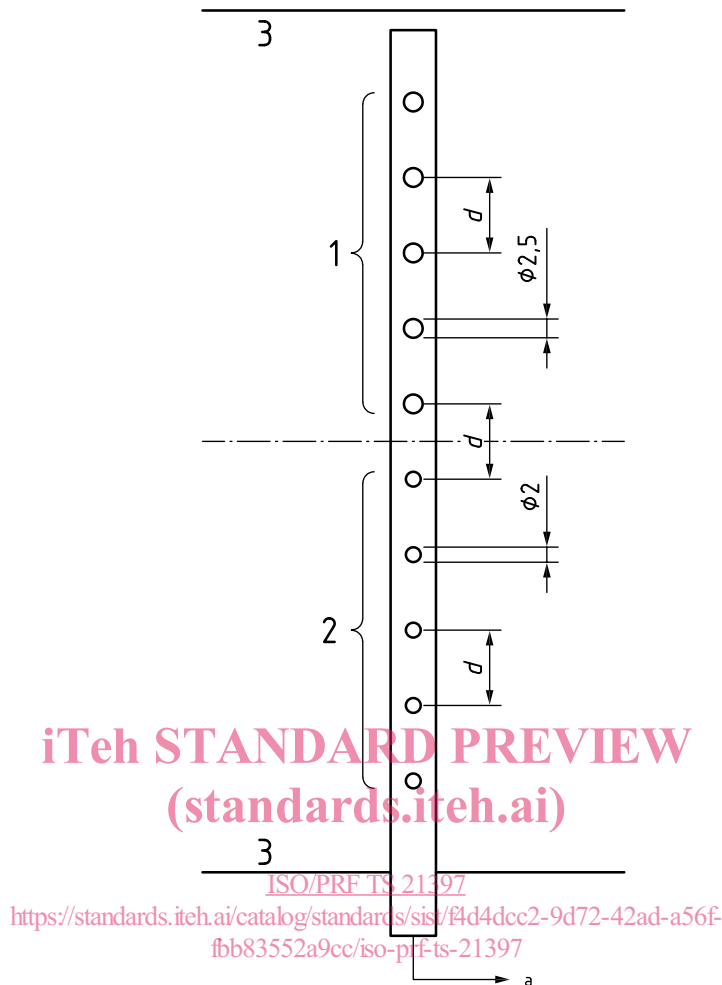
The ISO 5660-1 gas sampling ring probe can be used. A heated tee-connection/heated gas line is required to sample from the main gas sampling line from the ring probe (connecting the sampling ring to the cone calorimeter oxygen analyser) for transfer to the FTIR analyser. The tee-connection shall be located as close as possible to the sampling ring and shall as a minimum be efficiently insulated in order not to cool the sampled gases.

**NOTE** When using the ring probe for sampling to the FTIR it can be necessary to adjust the sampling flow rate to the oxygen analyser.

Alternatively, a separate linear multi-hole probe may be used for gas sampling. This is positioned vertically in the duct either 100 mm upstream<sup>[3][4]</sup> or at 50 mm downstream of the ISO 5660-1 gas sampling ring probe. The probe shall be made from 6 mm (OD) stainless steel tubing, closed at the sampling end, and shall have 10 holes evenly distributed along the length of the tube. The 10 holes on the probe shall face downstream to prevent clogging due to the soot deposition.

The five holes furthest from the pump shall have a diameter of 2,5 mm, while the five holes closest to the pump shall have a diameter of 2,0 mm. The probe shall be long enough to sample over the whole inner diameter of the duct. A Schematic diagram of the linear multi-hole probe is provided in [Figure 3](#).

The probe shall be connected directly to the primary heated filter without any cold-trap between.



#### Key

- 1 five evenly-distributed sampling holes with 2,5 mm diameter
- 2 five evenly-distributed sampling holes with 2,0 mm diameter
- 3 cone calorimeter exhaust duct wall
- $d$  hole spacing,  $(10,0 \pm 0,2)$  mm
- <sup>a</sup> To gas sampling system.

**Figure 3 — Schematic diagram of the linear multi-hole probe**

### 7.3 Primary filter

A primary filter unit shall be connected directly to the tee-connection (or multi-hole probe) to protect the FTIR gas cell from soot and other solid particles, and its temperature shall be maintained at  $(180 \pm 10)$  °C. The filter element shall be easy to change and inert to fire effluents. The dimensions and materials of the filter are described in ISO 19702. The replacement interval shall be determined from considerations of the sampling rate, test duration and soot production in the specific test. The filter shall normally be replaced before tests.

NOTE Cylindrical filter with high particle capture capacity have been found to be most effective<sup>[6]</sup>. A cylindrical filter with a porosity of 2 µm is recommended in ISO 19702.