TECHNICAL SPECIFICATION

ISO/TS 21397

First edition
2021-03

FTIR analysis of fire effluents in cone calorimeter tests

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

Reference number
ISO/TS 21397:2021(E)

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Foreword

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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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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.

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.

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 (μL/L) or gas yield (mg/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. 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.

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 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
Δm	total mass loss of the test specimen	g
$\dot{m}_g$	mass flow rate of sampled gas species of interest	$g\ s^{-1}$
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	$g\ mol^{-1}$
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	$m^3\ s^{-1}$
$\dot{V}_g$	volume flow rate of sampled gas species at 0,1 MPa and 25 °C	$m^3\ s^{-1}$
V_g	total volume of sampled gas species at 0,1 MPa and 25 °C	m^3
y_g	species yield of gas	$g\ g^{-1}$
$Æ_g$	volume fraction of gas	dimensionless
ρ	gas density	$kg\ m^{-3}$

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 (μL/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

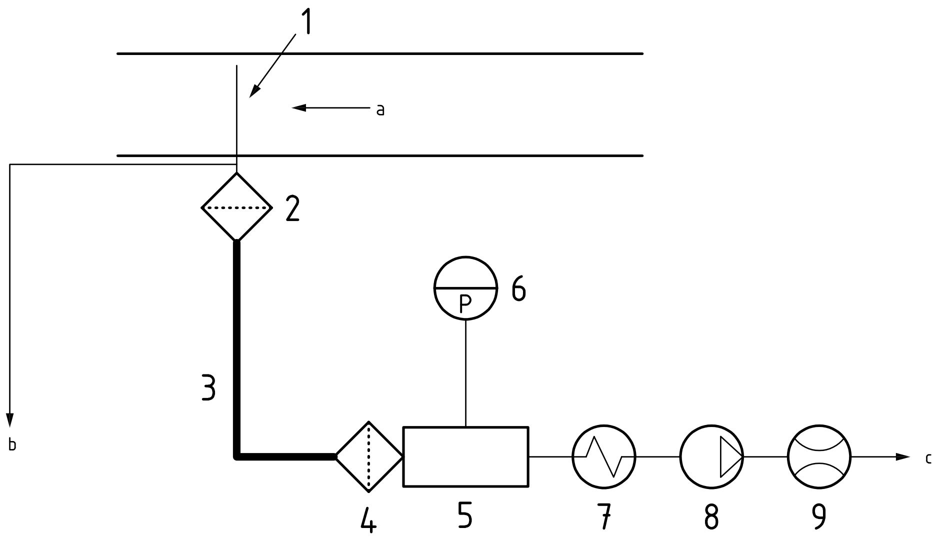
The test apparatus is specified in ISO 5660-1 and the gas measurement system described in Clause 7 shall be used.

7 Gas measurement system

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)

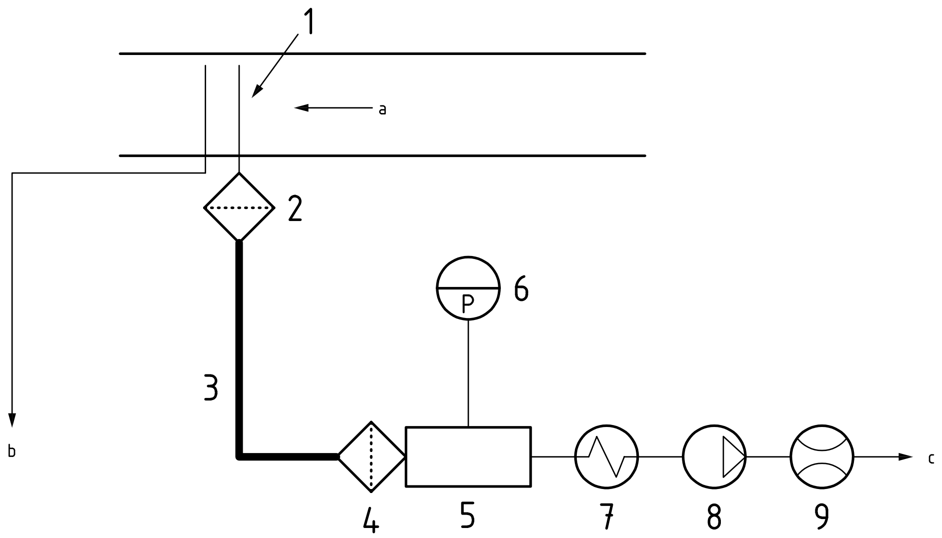
9 flow meter

a Direction of Combustion gas flow.

b To O_2, CO_2 and CO analyser.

c To exhaust.

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_2, CO_2 and CO analyser.
c To exhaust.

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

7.2 Sampling probe

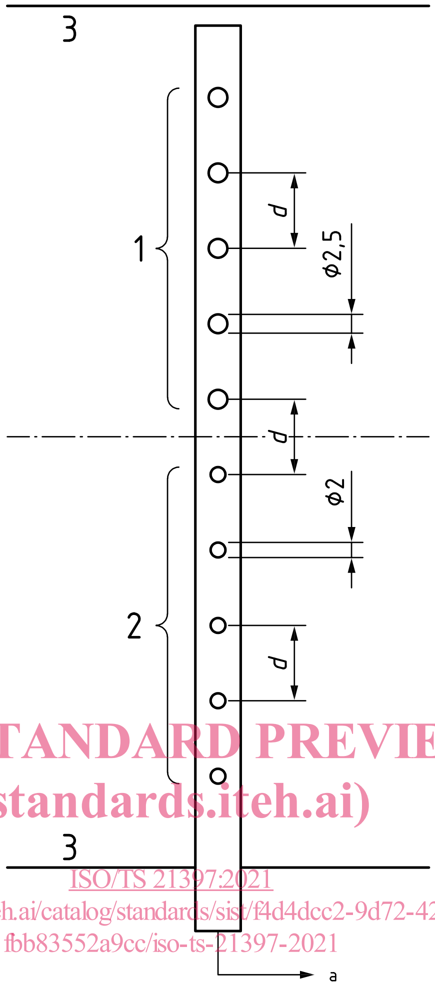
The ISO 5660-1 gas sampling ring probe can be used. A heated tee-connection/heated gas line is required to sample from the main gas sampling line from the ring probe (connecting the sampling ring to the cone calorimeter oxygen analyser) for transfer to the FTIR analyser. The tee-connection shall be located 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[3],[4] 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 dimeter

2 five evenly-distributed sampling holes with 2,0 mm dimeter

3 cone calorimeter exhaust duct wall

d hole spacing, (10,0 ± 0,2) mm

a 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 ± 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 filters with high particle capture capacity have been found to be most effective[6]. A cylindrical filter with a porosity of 2 µm is recommended in ISO 19702.

