INTERNATIONAL STANDARD

ISO 19702

First edition
2006-01-15

Corrected version
2006-10-15

Toxicity testing of fire effluents — Guidance for analysis of gases and vapours in fire effluents using FTIR gas analysis

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

Reference number
ISO 19702:2006(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

STANDARDSISO.COM : Click to view the full PDF of ISO 19702:2006

© ISO 2006

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 19702 was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 3, *Fire threat to people and environment*.

This corrected version of ISO 19702 incorporates the following corrections:

- 5.2.2.1: subclauses, which were numbered 4.2.2.1.1 to 4.2.2.1.4, have been renumbered to 5.2.2.1.1 to 5.2.2.1.4, respectively;
- Annex A, paragraph before Figure A.2, fourth sentence: “A coil of 0,6 cm (¼ in) od tubing...” has been changed to “A coil of 0,6 cm (¼ in) outside diameter (OD) tubing...”;
- Bibliography: The number of reference [4] reference has been changed from ISO/CD 21489 to ISO 21489.
- Bibliography: The number and title of reference [5] have been modified to the following:

  IEC/TS 60695-7-50, *Fire hazard testing — Part 7-50: Toxicity of fire effluent — Estimation of toxic potency — Apparatus and method*

Introduction

During recent years, analytical techniques have been used widely for the measurement of the concentrations of specific volatiles generated during both laboratory studies and real fires.

The analysis of gases in fire effluents, whilst occasionally needing to rely on methods perfected in other fields (e.g. atmospheric pollution), represents a very specialized field of study due to the complexity and reactivity of the gas mixtures and the possibility for a rapid change in concentration with time. This has led a number of scientists from different countries developing new, or adapting existing methods for the analysis of the gases present during combustion, in accordance with their own requirements.

In some cases, common lines of analysis have emerged, and there is now sufficient expertise and experience to define standard methods for analysing selected gases. Much of this information is provided in ISO 19701, which presents a variety of chemical methods for the determination of individual gases of toxicological importance. Typically, multiple methods are needed to determine all the species of interest for fire hazard analysis.

Fourier transform infrared (FTIR) spectroscopy is different from other techniques in that

— a single method can be used to determine a variety of gases;

— FTIR measurements can be time-resolved, enabling the monitoring of how species develop throughout the fire;

— if a new toxicant should later be identified as important, relevant data concerning the presence of that toxicant might be found in the stored FTIR spectra from previous experiments.

ISO/TC 92 SC 3 has therefore developed ISO 19702 as a separate document.

There are two distinct ways in which FTIR has been used to characterize fire effluent:

— open path analysis, in which the infrared beam is directed across the effluent within the test apparatus;

— extractive analysis, in which a fraction of the apparatus environment is continuously flowed to a sample cell for remote measurement (e.g. IMO resolution MSC 61(67), part 2 toxicity analysis).

Fire scientists have successfully applied variants of both approaches; although, the latter is the more common.

In particular, a European Union funded project (SAFIR) focused on the testing and validation of an extractive FTIR method for use in fire testing. The results of this project form the basis for ISO 19702.

In this International Standard, specific recommendations for sampling systems for use in small-scale and large-scale measurements, for spectral resolution, and for collection and use of calibration spectra are provided. Spectral information is provided for the gases studied specifically in the SAFIR project as an aid to users to determine spectral regions of interest. Finally, applications of the analytical method are discussed.

The primary purpose of the analytical method presented here is to measure the concentration of toxic species to aid in

a) characterizing physical fire models;

b) validating numerical fire models;

c) setting the conditions for exposure in biological studies;

d) monitoring of biological studies;

e) interpreting biological studies;

f) providing data for use in combustion toxicity assessment without requiring biological studies.

The methods are also generally applicable to the analysis of fire effluents in many situations including real fires.

Any chemical analysis is selective.

Chemical analytical methods are usually appropriate for accurate determination of some species, less for others. Thus, multiple methods can be needed to determine all the species of interest.

In most cases, FTIR can provide the concentration data needed to calculate the yields of the chemical species that are important as input to toxic hazard assessment.

The general recommendations given are based on work conducted using a number of small- and large-scale standard test methods. It is important that this International Standard be read together with the following standards:

- ISO 9705;
- ISO 5660-1:2002;
- ISO/CD 21489:—;
- IEC 60695-7;
- EN 13823:2002;
- ISO 5725.

Toxicity testing of fire effluents — Guidance for analysis of gases and vapours in fire effluents using FTIR gas analysis

1 Scope

This International Standard specifies methods for the individual analysis of airborne concentrations of carbon monoxide (CO), carbon dioxide (CO_2), hydrogen cyanide (HCN), hydrogen chloride (HCl), hydrogen bromide (HBr), nitric oxide (NO), nitrogen dioxide (NO_2), and acrolein (CH_2CHCHO). Although not specifically defined in this International Standard, as they were not specifically studied in the SAFIR project, the method presented is suitable for most gaseous species able to be analysed using the FTIR technique, including hydrogen fluoride (HF) and sulfur dioxide (SO_2).

2 Normative references

The following referenced documents are indispensable for the application 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 5660-1:2002, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 1: Heat release rate (cone calorimeter method)*

ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement and results*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
spectrometer
instrument used to disperse radiant energy into a spectrum and measure certain properties such as wavelength, mass, energy, or index of refraction

3.2
spectroscopy
study of spectra, especially to determine the chemical composition of substances and the physical properties or concentration of molecules, ions, and atoms

3.3
Fourier transform Infra-red spectroscopy
FTIR
technique for measuring concentrations of multiple species simultaneously using the mathematics of Fourier transforms

3.4
resolution
full width at half height of the instrument line shape function

NOTE This is expressed in units of per centimetre and is the smallest spacing between absorption peaks that can be resolved.

3.5
interferogram
modulated component of the IR beam at the detector measured versus mirror position

3.6
specimen
representative piece of the sample prepared to be tested

3.7
products of combustion
solid, liquid, and gaseous materials resulting from combustion

[ISO 13943]

3.8
minimum detection limit
MDL
theoretical lowest measurable concentration of each measured gas

4 Principle

Numerous different methods of analysis of fire gases are used internationally with acceptable results. Some of the more common alternative standard methods are outlined in ISO/TR 9122-3.

The analysis of gases in fire effluents is very complex due to the great number of different organic and inorganic chemicals potentially present in representative atmospheres. This point is discussed in more detail in conjunction with the presentation of the analysis method.

Conformance with this standard implies that

- the analysis of sampled gases has been carried out by standard procedures;
- the sampling procedure is in accordance with the general recommendations given and with due consideration to the reactive nature of the species analysed.

The sampling system is an integral part of the whole measurement methodology. Recommendations will be made separately for the various parts of the sampling system.

5 Test procedure

5.1 Principle

The effluent from the fire test or physical fire model for the FTIR analysis is taken either from a gas sampling system, such as a flue, or directly from the fire gases. The gas sample is drawn continuously through a heated sampling line to the heated IR absorption cell of the FTIR spectrometer. An interfered IR beam is directed through the gas cell and at chosen intervals interferograms are obtained and, either after the test or on-line, converted into absorption spectra. The concentrations of the gases are calculated by analysis of the absorption spectra using one or more of a variety of methods discussed in Clause 7. The concentration-time relationship of the gases can be evaluated from successive spectra at sufficiently frequent intervals.

FTIR is based on infrared absorption. Specific to FTIR is conversion of regular irradiance from an IR source into interfered irradiance and conversion of an interferogram into a conventional wavelength spectrum containing information over a wide range of wavelengths. In this method, the spectrum is usually presented as absorbance as a function of radiation in wave numbers.

Molecules with a dipole moment have absorption in the infrared region. Thus, only molecules that show sufficient asymmetry can be sensed by their infrared spectrum (e.g. symmetric molecule like oxygen cannot be studied by infrared). There is a large range of absorption strengths among IR-absorbing molecules. Thus, some weakly absorbing species can be “lost” if the user sets the scale for a species of high absorption strength. Conversely, strongly absorbing species can saturate the spectrometer if the user sets the instrument to measure weakly absorbing species. Concentrations are related to intensities of absorption. This is discussed in greater detail in Clause 7 below.

5.2 Sampling system

Recommendations for the filters, sampling line, pump speed, temperature, pressure and response of the sampling system are the same for both large-scale and small-scale testing. Thus, these are made for both small and large scale at the end of this section. The Annex A gives example of sampling systems.

The choice of volumetric sampling speed depends on a variety of parameters, including: the size of the absorption cell, the degree of turbulence within that cell, the temperature of the effluent, the rate at which the concentrations of the species changes during the experiment, the efficiency of the soot filtration system, the length of the sampling line (to control axial diffusion effects), etc. The diameter of the sampling line also should be adjusted based on the above, at least to avoid significant pressure drops in long lines. The sampling rate shall also be selected so as not to perturb the experiment. Thus, there can be differences between sampling conditions for different apparatus, even at the same scale.

The recommendations for the probe are somewhat dependent on the type of measurement and are treated separately for the small and large scale experiments.

IMPORTANT — The fire gases from the FTIR gas cell shall be returned to the exhaust duct of the fire test instrument or otherwise disposed of in a safe way that does not endanger personnel and at a location that does not disturb measurements.

5.2.1 Temperature

The temperature of the sampling system shall be kept as high as practically possible to reduce the risk of condensation and subsequent washing of soluble fire gases out of the sample. The whole system shall be kept at a minimum temperature of 150 °C, although the recommended temperature is 180 °C.

The temperature throughout the sampling system should be homogeneous, or slightly increasing along the sampling system from the probe to the gas cell, to avoid cold points that could act as a condensation point for water and soluble gases.

5.2.2 Probe

5.2.2.1 Small scale

Sampling the gases from small-scale tests depends on the fire model (e.g. smoke box as in ISO 5659, tubular furnace, cone calorimeter as in ISO 5660-1). The best choice of a probe and its position are closely related with the fire model. Only recommendations are given in this International Standard.

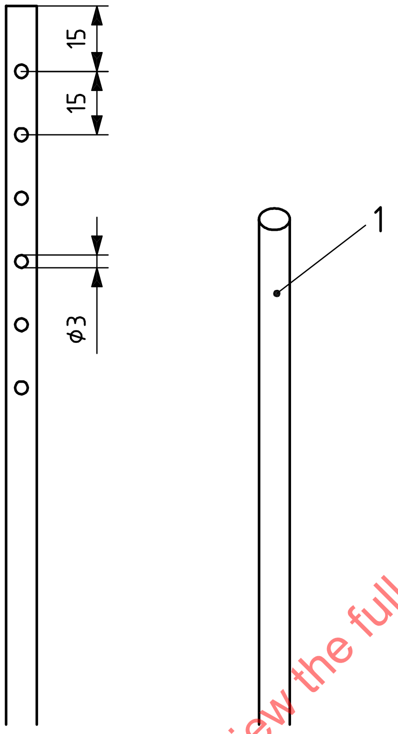
5.2.2.1.1 Different types of probes can be used, i.e. a single-hole or multi-hole probes. The choice will depend directly on the fire model considered. In order to obtain a representative mixture of effluent; when stratification effects are suspected, a multi-hole probe is preferred. An example of the different types of probes is shown in Figure 1.

5.2.2.1.2 The probe shall be made of material resistant to corrosion, stainless steel is recommended, with a minimum hole size of 3 mm in diameter.

5.2.2.1.3 In the case of a tube furnace type of test, the extraction probe takes a portion of the total flow from the furnace to the FTIR (generally by dividing the exhaust flow). In this case, the internal diameter of the extraction probe should not be less than 3 mm, in the case of a single-hole probe.

5.2.2.1.4 Samples from the exhaust duct of small-scale tests can be extracted using a probe placed in the exhaust duct of the particular test. In this type of situation, a multi-hole probe, inserted perpendicular to the flow of the fire gases, with the holes facing downstream from the gas flow, is recommended.

Dimensions in millimetres



Key

1 inner diameter

Figure 1 — Schematic of probes for sampling in exhaust duct

Care shall be taken to ensure representative sampling. In order to avoid perturbation in the effluent, volumetric sampling shall be a small fraction of the total flow through the duct. The probe shall be positioned such that the flow of effluent is not turbulent (effluents well mixed) and as near as possible to the fire source (in order to limit the condensation or secondary reaction of gases). Positioning of probe shall be considered for each fire model: taking into account the effect of stratification and/or the velocity profile in flow tubes.

The probe shall be cleaned regularly to ensure that the holes do not become blocked, as this will reduce the reliability of the sampling.

5.2.2.2 Large scale

The type of probe recommended above for duct extraction in small scale tests is suitable for measurements in the duct of a large-scale test such as the ISO 9705 room or SBI apparatus.

When sampling directly from inhomogeneous fire gases, however, a representative sample should be taken over the whole of the fire gases being monitored. In order to accomplish this, it is necessary to have a gradation of hole sizes along the length of the probe and to place the probe such that it crosses the fire gases monitored. An example of a suitable probe is given in Table 1. This probe has been successfully applied to measurements taken in the door of the ISO 9705 room, where the top of the probe (closest to the pump) was placed at the top of the ISO 9705 door and the probe traversed the door diagonally, finishing 30 cm below the top of the doorway.

Table 1 — Example of a probe suitable for sampling fire gases from the door of the ISO 9705 room

Probe characteristics [a]	
Hole number	**Hole diameter** mm
1 (closest to end)	5
2	3,2
3	2,5
4	2,1
5	1,8
6	1,6
7 (closest to pump)	1,5

[a] This probe has an internal diameter of 6 mm and the holes are placed 10 cm apart. The end of the probe is sealed.

This probe is placed such that it crosses the top of the door opening with the lowermost hole 30 cm below the top of the door. The holes are facing downstream from the fire gases.

5.2.3 Gas cell

The gas cell should be composed of inert material that is resistant to the fire gases measured (e.g. nickel coated aluminium or synthetic silica are often used).

It has been shown that nickel-coated aluminium can fail rapidly unless it is ensured that the coating is of sufficient thickness. Caution shall be taken using that type of equipment.

The volume and the path length of the gas cell are critical for the response time of the total measurement system, and a gas cell volume of between 0,2 to 2 l is normally recommended in fire testing. The path length could vary with the volume of gas cell. A path length between 2 m and 10 m has been found suitable for fire effluent analysis. The volume and the path length of the gas cell shall be adapted with regard to the desired detection limit and the fire model considered.

The gas cell should be heated to a minimum of 150 °C and both the temperature and pressure in the cell should be monitored throughout the fire test. It is recommended that both the pressure and temperature be kept constant as this effects the intensity of the spectral band (see Annex A). If the temperature or pressure in the fire test cannot be maintained at the same value as that used when collecting calibration spectra, then a correction should be made for this difference when calculating species concentrations. However, the correction is valid only for limited variation in pressure and temperature. This correction can be made using the universal gas law. This correction might not be completely effective in every case due to non-linear changes in the shape of spectra with pressure and temperature.

5.2.4 Filter

The gas cell (internal mirrors) shall be protected from the intrusion of soot from the fire gases.

NOTE This is always true unless a so called “dirty cell” is being used. In this case, soot is allowed to enter the gas cell. These cells are, however, only suitable when small amounts of soot are expected and are not recommended for the study of fire tests.

A filter or filters may be placed either between the probe and sample line or after the sample line. Both positions have advantages and disadvantages.

Placing the filter between the probe and sample line protects the sample line from build-up of soot, which may adsorb acid gases. The disadvantage of this filter position is that during the early stages of a fire, the filter medium may be cooled by the sample gas stream. A large temperature differential may develop between the geometric centre of the filter medium and the heated filter housing. This creates a condition favourable for the retention of water and water-soluble acid gases in the filter medium. Care shall be taken to use a sufficiently long heated probe to preheat the sample gases prior to contact with the filter medium, and/or a heated filter housing of sufficiently small diameter to allow the filter housing to more effectively heat the core of the filter medium. Decreasing the diameter of a membrane filter has the benefit of lowering the absorption of acid gases on the filter.

Placing the filters downstream of the heated sample line preheats the sample gas prior to reaching the filters, ensuring that the filter medium is too hot to contain moisture. This minimizes the loss of acid gases on the filter medium.

Either planar membrane filters or cylindrical filters are recommended. The planar filters should optimally have a porosity of 5 µm and a diameter of 47 mm, although other porosity and diameters may be used. Placing a high-capacity, high-porosity filter upstream of this low-porosity filter prolongs the service life of the downstream filter. Glass fibre, ceramic, and PTFE coated filters have been found suitable. Care shall be taken when working with heated filters, in particular close to the high working temperature limit, as the filter is required to function at temperatures above 150 °C for a long period of time.

Glass wool filters cannot be used for fluorine analysis. The porosity of the filter shall be chosen as a compromise between sufficient entrapment of soot without premature clogging of the filter.

The filter housing should be made of stainless steel (although in certain cases PTFE can be used) and the filter temperature should not be less than 150 °C.

More important than the specific choice of filter material and porosity is that the filters used are analysed for the presence of acid gases if these are measured. This is due to the fact that acid gases can adsorb onto the soot particles in the fire plume or onto the soot retained by the filter in an unpredictable way. This adsorption behaviour is especially important when measuring low concentrations of acid gases as some saturation behaviour is expected, i.e. the amount adsorbed onto the filter decreases as the filter becomes saturated with acid gases. An example of a procedure to analyse the gases retained on the filter is given in Annex B.

It should be noted that some filters contain chlorine. Care shall be taken if the filter is washed to collect the gases retained on soot or condensed with the water coming from combustion to determine the chlorine content without fire gases to provide a baseline for the determination of the level of adsorbed chlorine from the fire gases.

The kinetics of absorption onto the filter has not been characterized. However, when the total amount of gas produced during the combustion is needed, the quantity measured on the filter should be added to the total amount measured by FTIR. It shall be noted 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 needed, information on gases analysed on the filter shall be considered in order to identify the potential effect on the concentration/time curves.

Soot deposition on the cell optics can be minimized by selecting a FTIR system with the gas cell mirrors and cell windows mounted in a vertical position.

5.2.5 Sampling line

A gas sampling line of PTFE offers the best performance. This line should be kept at a constant temperature of at least 150 °C. An inner diameter of 3 mm to 4 mm is suitable. The length should be as short as possible and 4 m has been found to work well. Problems due to the adsorption of gases, particularly acid gases, onto the walls of the sampling line become more pronounced for the longer sampling line.

Saturation behaviour is expected for the sampling line as described for the filters above. This implies that a pre-aged line will adsorb less acid gases than a new line. This behaviour has not been characterized and thus a recommended method of ageing the line is not available.

Various materials such as synthetic, high-purity silica, PTFE and stainless steel have been found suitable for sampling lines. Their use depends on the type of gases to be analysed.

5.2.6 Pump speed

The sampling flow rate should be as high as possible without incurring problems due to clogging of filters. A minimum pump flow of 3,5 l/min is recommended, although this does depend on the cell volume and required response time. In the case of tubular furnace, for example, a lower flow rate is usually sufficient.

5.2.7 Response time of the sampling system

It is difficult to recommend a single experimental set-up for FTIR measurements, as different laboratories will have different equipment. In order to correctly interpret the FTIR results and compare them to other measurements in the same experiment, it is important that the response time for the system be known. A protocol for the determination of the response time for the whole sampling system is summarized in Annex C.

In general, the FTIR gas cell is responsible for most of the response time. An approximate response time can be calculated using an estimate of the total volume of your sampling system plus gas cell to divide by the flow rate in your system. This does not take into account the effect of the geometry of the sampling system, but is generally greater than 70 % of the full response time

6 Spectrometer parameters

The spectrometer should be located in a suitable environment to ensure correct function of the equipment, keeping in mind that some parts of the equipment are susceptible to moisture.

The spectrometer parameters are described in more detail in Annex E.

6.1 Detectors

Different type of detectors can be used, e.g. DTGS or MCT (see Annex E).

6.2 Resolution

A resolution in the range 0,5 cm^{-1} to 4 cm^{-1} is recommended for most requirements in fire experiments. This allows the collection of several measurements every minute while still retaining an acceptable sensitivity.

The resolution should be the same for calibration spectra and testing. Indeed, some software does not allow calibration spectra and spectra from tests to have different resolution.

6.3 Minimum detection limit (MDL)

Table 2 shows some examples of how the combination of resolution and number of scans that are being averaged effects the minimum detection limit of CO.

Table 2 — The effect of resolution and number of scans on the MDL of CO

Resolution cm^{-1}	**Number of scans**	**MDL** µl/l
1	1	16
	4	8
4	1	15
	4	7

It should be noted that the concentration values in Table 2 relate to the performance of a specific FTIR instrument. Further, it should be noted that other compounds absorbing in the same region, as the required compound, will increase the MDL.

6.4 Mirror alignment

The throughput of the spectrometer should be as high as possible. In order to achieve this, the mirrors shall be well aligned and the mirrors and optical window allowing the entrance of the IR beam should be kept clean and in good condition. Mirror alignment is also important for wavenumber reproducibility.

6.5 Spectral range

The spectral range will, to a certain degree, determine which species can be quantified. A wavenumber range of 4 500 cm^{-1} to 400 cm^{-1} is recommended (see Annex G).

It is recommended that calibration spectra are collected with the same spectral range as experimental spectra. This is not necessary for all quantification methods, but is worthwhile should it be necessary to employ a method which requires full calibration spectra at a later date.

Annex G gives examples of FTIR equipment and sampling systems that have been proved to be suitable for fire effluent analysis.

7 Calibration and analysis procedure

Calibration spectra shall be obtained with as little interference from unknown species as possible. It is, therefore, important that the calibration gases are dry and free from contaminants, unless the exact concentration of the contaminant is known. Gas mixtures of known concentrations may be used as reference gases in calibration with a precision consistent with requirements. These are usually composed of a single or dual gas mixture in nitrogen, although in some cases it can be useful to obtain calibration spectra containing fire gases from a well defined fire scenario. Gas mixtures can also be made using “diffusion tubes” or a “closed loop” calibration system; see Annex F. In some cases, mixtures of several gases must be used with certain more refined methods, especially in matrix based analysis methods such as PLS type methods. In some cases, gases supplied in cylinders (e.g. hydrogen chloride and hydrogen bromide) can incur significant losses over relatively short time periods.

Calibration spectra should be collected using the same FTIR set-up as in the subsequent fire experiments. Any difference in cell path length, resolution, temperature, pressure or spectrometer throughput can significantly reduce the reliability of any quantitative evaluation.

Details concerning the collection of a calibration set are included in Annex D. Once a calibration set has been obtained, several methods can be used to create a calibration model depending on the analysis method chosen.

It is important to acknowledge that FTIR determines the infrared absorption of a gas or mixture of gases over a wide range of wave numbers (typically 400 cm^{-1} to 4 500 cm^{-1}). The infrared absorption bands for the main fire gases are given in Annex F. Although it should be noted that many other gases found in fire atmospheres, e.g. hydrocarbons, also absorb infrared radiation in this region. It is, therefore, rare for a fire gas to absorb infrared within a given wave number region without interference from other fire gases in the same region.

Fire gases are identified by the characteristic wave numbers where they absorb the infrared beam and quantified by the intensity of the infrared absorption. The reliability and accuracy of the identification and quantification depend directly on the ability of the analysis method to identify and separate the spectrum of the specified fire gas from that of the spectra of other unspecified gases in the mixture.

The methods used to analyse FTIR spectra may be defined as univariate and multivariate types and are available as commercial software packages.

The analysis methods and calibration requirements for the analysis of fire gases relate to the type of analysis used.

7.1 Univariate

These methods include “peak height” and “peak area”.

Specific regions of the spectra of the specific gas are selected to be as free as possible of interference from other gases. It may be necessary to select more than one region. The fire gas spectra are then compared to that of the specified gas and, where necessary, the spectrum of interfering gases can be subtracted from that of the fire gas spectrum. The specified fire gas is then quantified using either the height or area of a selected peak or peaks and corrected for any non-linearity of the absorption/concentration characteristics of the gas.

These methods are simple and can be applied only for simple cases, i.e. cases with minimal interference between different fire gases. This is often not the case for fire effluents where there are numerous gases present in the fire effluents. For complex mixture of gases, the univariate methods can provide erroneous results. To identify errors from univariate methods, it is important to manually inspect selected spectra to identify possible interfering gases and erroneous concentration calculations.

7.2 Multivariate analysis

These methods include the following:

- multilinear regression (MLR);
- classical least squares (CLS);
- ridged regression (RR);
- partial least squares (PLS);
- implicit non-linear latent regression;
- target factor analysis (TFA).

It is not proposed to discuss these techniques, as they are extremely complex. In essence, they use a number of mathematical models and processes to combine the spectra of known or suspected interferences to match the fire gas spectrum. The spectrum of the specified gas is then identified and the gas quantified using correction curves to allow for any non-linearity in the response of the FTIR.

These methods require the interferences to be identified within the analysis method or selected from a large database of spectra by the analytical method.

7.2.1 General observations

It is possible to develop FTIR analysis methods to provide good levels of accuracy, especially for a single gas or for a simple mixture of known gases; but it must be acknowledged that as the complexity of the gas mixture increases and the number of known and unknown interferences are increased, then the accuracy of the FTIR analysis can decrease. In practice, it may be desirable to use a combination of both univariate and multivariate methods to analyse fire gas samples which inherently contain a large number of gases, many of which may be unknown or even unexpected.

It is increasingly common for most spectroscopic software packages to include most of the methods listed above. The calibration procedure will therefore depend on the requirements of the method selected and the software package being used.

8 Test start-up

Before a test, the sampling system and gas sampling cell are heated to their chosen working temperatures. Ample time should be allowed for temperature equilibration of the whole sampling system. A new or clean filter should be used when determining acid gases. The throughput of the IR beam shall be checked to ensure correct performance of the spectrometer and suitable wave number reproducibility. A background reference spectrum is obtained.

9 Presentation of results

The way results are presented is dictated by how the results are to be used. Typically, one would expect results to be presented graphically as time resolved concentrations or yields, and tabulated as peak concentrations. In addition, the following parameters should be reported for each gas:

- wavenumber(s) or region(s) used for calibration;
- calibration method(s) employed;
- estimated number of overlapping gases in the selected wave number region;
- time of measurement of maximum concentration during the test;
- spectral resolution of references and test data;
- time resolution between each spectrum acquired;
- type of detector used;
- whether evaluated concentrations lie in the range defined by calibration data or are based on extrapolation outside of this range.

10 Precision

The precision of FTIR smoke gas data is influenced by several factors, including the gas flow and ensuing gas cell pressure control, sampling system temperature, filter and sampling system efficiency, and calibration model quality.

10.1 Repeatability and reproducibility

An interlaboratory trial has been conducted in connection with the SAFIR project. The materials tested in this trial were 12 mm particle board ($\rho = 700$ kg/m^3), 3 mm fire-retarded PVC ($\rho = 1\,180$ kg/m^3), and 35 mm PUR foam panel ($\rho = 40$ kg/m^3). Three replicates of each material were tested by eight laboratories using the cone calorimeter method at the irradiance level of 50 kW/m^2 in accordance with ISO 5660-1. The FTIR protocol used was functionally the same as described in this part of ISO 19702.

Values for the relative repeatability and reproducibility standard deviations (s_r / m and s_R / m, respectively) were calculated for the data set in accordance with the principles of ISO 5725. The FTIR quantities studied were the maximum concentrations and yields of CO_2, CO, HCl and HCN. The results are presented in Table 3.

Table 3 — Ranges and average values of relative repeatability and reproducibility standard deviations for FTIR quantities

Quantity	s_r / m Range %	 Average %	s_R / m Range %	 Average %
$CO_{2,max}$	4,9 to 16,2	9,2	14,9 to 23,8	19,7
CO_2 yield	3,8 to 15,6	8,3	15,6 to 39,1	24,2
CO_{max}	9,9 to 16,5	13,8	19,8 to 29,8	25,3
CO yield	5,7 to 17,0	12,2	34,0 to 46,7	38,8
HCl_{max} [a]	—	13,0	—	28,5
HCl yield [a]	—	8,6	—	26,0
HCN_{max} [a]	—	11,7	—	39,3
HCN yield [a]	—	12,2	—	28,7

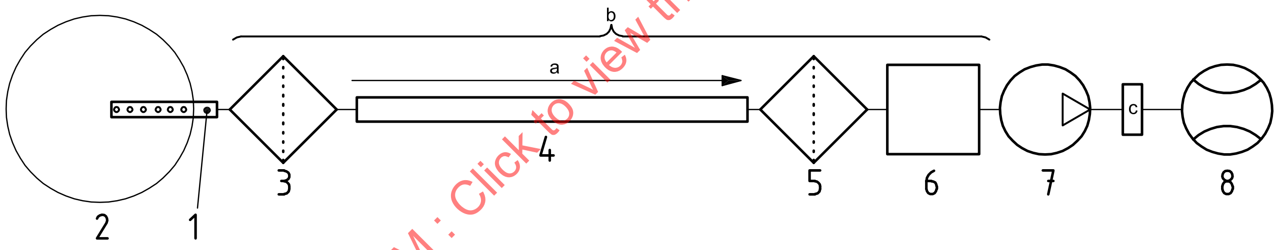
[a] Statistical analysis of HCl and HCN results was performed only for one material. These single repeatability and reproducibility values are reported in the "Average" column for HCl and HCN.

Annex A
(informative)

Example of sampling device

An optimized sampling device has been proposed in the SAFIR project. The Figure A.1 gives a schematic of the optimized sampling device.

- The point for gas collection in the exhaust duct shall be placed at a distance where the gaseous mixture is homogeneous and the gas flow is not disturbed.
- The distance between the combustion area and the probe should be as short as possible to avoid condensation.
- The filter shall be placed between the probe and the transfer line. Another filter may be used between the line and the gas cell to protect the cell from fine soot particles.
- The flow meter shall be placed after the pump and it is recommended to trap the water between the pump and the flow meter.
- The temperatures of the filter, sampling line and gas cell should be as similar as possible.



Key

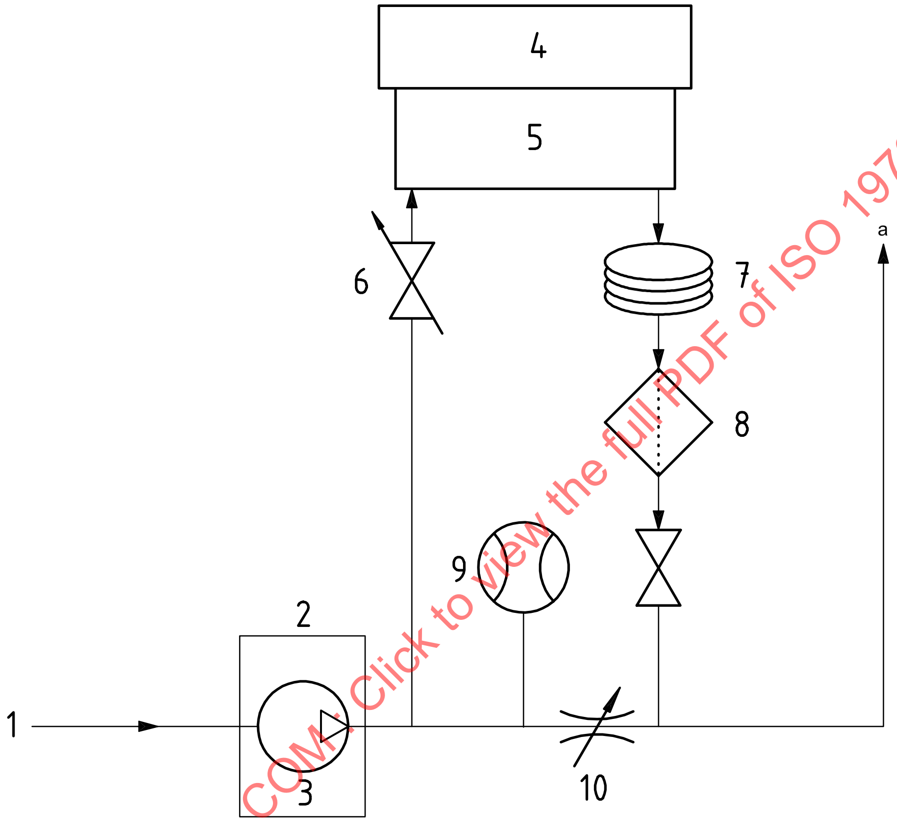
1 probe
2 exhaust duct (fire model)
3 filter 1
4 transfer line
5 filter 2
6 FTIR gas cell
7 pump
8 flow meter

[a] Minimum flow rate, 3,5 l/min.
[b] Part of the sampling system heated to ⩾ 150 °C.
[c] System to trap water.

Figure A.1 — Diagram of the optimized sampling device

Other sampling system has been used with the objective to maintain a constant pressure.

The gas cell pressure can be kept constant for systems with the pump upstream of the sample cell by teeing a back-pressure regulator to the analyser inlet (Figure A.1). A needle valve is positioned upstream of the heated cell. This configuration provides a constant cell pressure very slightly above atmospheric pressure. A coil of 0,6 cm (¼ in) od outside diameter (OD) tubing downstream of the heated cell cools the sample gas to room temperature prior to entering the filter. Low-molecular-weight organic gases are trapped by a high capacity 0,3 µ filter with a gas filtration area of 625 cm^2 to protect the flowmeter from build-up of condensates. The high surface area of the filter media provides a long filter life without affecting the cell pressure.



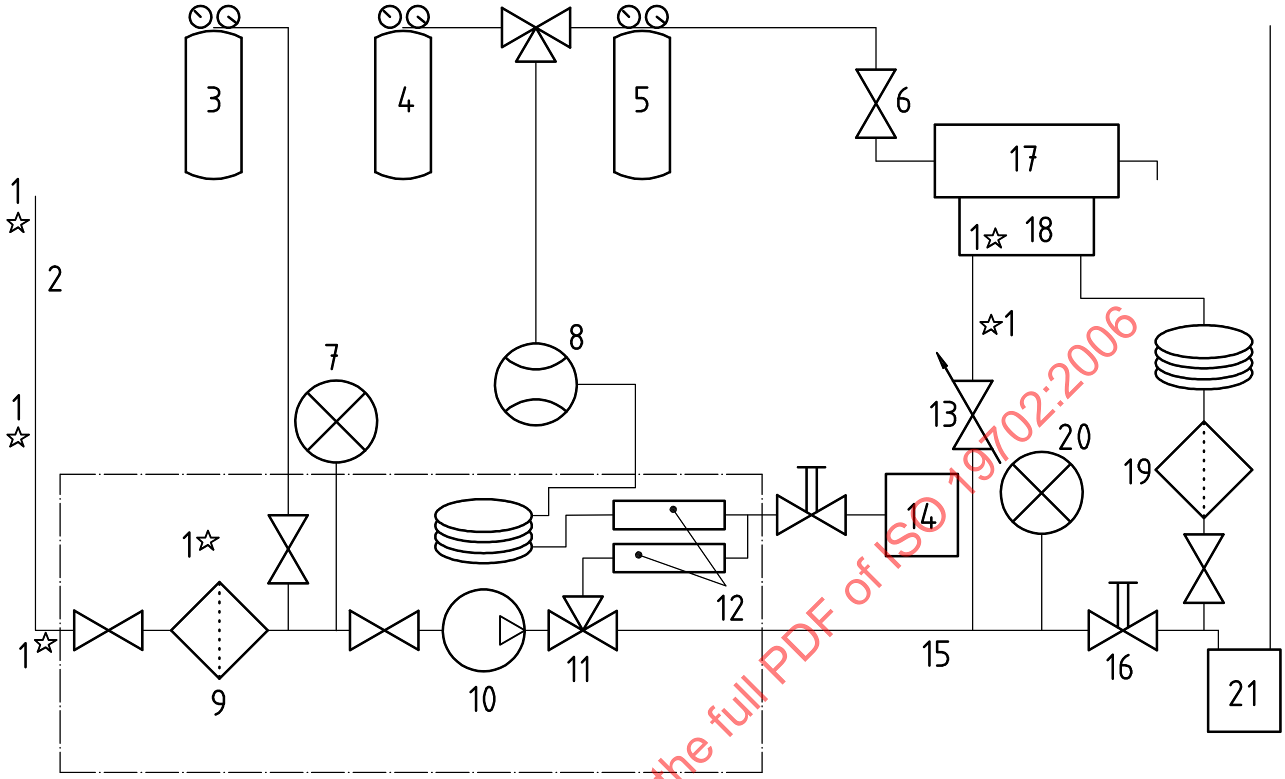
Key

1 heated sample line
2 oven
3 pump head
4 FTIR
5 heated cell
6 needle valve
7 cooling coil
8 filter
9 flow meter
10 back-pressure regulator

[a] Flow out.

Figure A.2 — Schematic diagram for maintaining constant pressure to sample cell using a back pressure regulator

A schematic diagram of a pressurized system with a back-pressure regulator is illustrated in Figure A.3.



Key

1	thermocouples	12	bottles
2	heated sampling line	13	needle valve
3	nitrogen	14	syringe pump
4	calibration gas	15	heated line
5	liquid nitrogen	16	back-pressure regulator
6	FTIR purge gas	17	FTIR
7	pressure/vacuum gauge	18	sample cell
8	flow meter	19	filter
9	filters	20	flow meter
10	pump head	21	water trap
11	selection valve		

Figure A.3 — Schematic diagram of FTIR sampling system with a back-pressure regulator

Annex B
(normative)

Analysis of filter materials and sampling line for gas retention evaluation

B.1 Washing procedures for filters

Acid gases can be absorbed on soot retained by the filter. When the total amount of one specific acid gas has to be measured, the quantity retained on the filter should be added to the total amount of acid gases analysed by the FTIR.

The washing procedures for filters is as follows:

a) Procedure for planar filters:

- After each test, the filter is removed and placed in a minimum volume of water (analytical quality).
- The solution (water and filter) is placed in an ultrasonic bath for at least 10 min.
- The solution is gauged to a known volume before analysis. (Analytical methods such as titrimetry or ion chromatography can be used.)

b) For cylindrical filters:

- Use the method described above for planar filters; or
- Wash the filter with hot water in a Soxhlet for approximately 20 min. Then the solution is gauged to a known volume before analysis. (Analytical methods such as titrimetry or ion chromatography could be used.)

The same protocol (with a Soxhlet extractor) can be used to wash new filters. The filters should be dried before use (250 °C in an oven is acceptable).

B.2 Washing procedures for transfer line and probe

The transfer line and the probe should be rinsed with water (analytical quality). Before rinsing the systems, their temperatures must be approximately 70 °C to avoid the vaporization of water. For each part of the sampling system, the washing solutions are collected in volumetric flasks and the solutions are gauged to a known volume before analysis by appropriate analytical methods (see above).

B.3 Adjusting total concentration

When the total amount of gas evolved during combustion is measured, adjustment may need to be made for the gases adsorbed on the filter to include the total amount of gas analysed by FTIR (area under the curve concentration versus time) and the total amount of “trapped” gas analysed in the “washing” solution.

Annex C
(informative)

Response time determination of FTIR cell and sampling device

C.1 General

The purpose of the following procedure is to determine the response time of the sampling device and the gas cell.

C.2 Factors influencing the response time

The following factors influence the response time:

- heated sampling line length and internal diameter of various pipes;
- internal volume of measuring cell and filtering element(s);
- geometry of the cell and filtering element(s);
- sampling flow rate in the line.

The configuration and position of the sampling line, filters and other devices usually used during real tests shall be kept for this determination as should the temperature usually set for heated elements and sampling flow rate in the circuit.

Make the determination setting using a setting of e.g. 4 cm^{-1} resolution and 1 scan per spectrum on the acquisition software. Set an acquisition rate as short as possible between spectra.

C.3 Principle of determination

A bottle containing CO_2 in nitrogen shall be used; see Figure C.2.

Set the CO_2 flow pressure to approximately 0,1 MPa (1 bar). It is essential to maintain a constant flow rate during spectra acquisition. To obtain a new flow cut when the introduction of the gas stopped, use tongs to pinch tube A instead of closing the bottle pressure reduced. Both tube A (from the bottle to the T) and B (from the T to the heated sampling line's entry) shall be as short as possible. Use a T to introduce the CO_2 into the heated sampling line. The dilution that occurs on the CO_2 has no influence on the determination.

Once the whole FTIR device is equilibrated in terms of temperature, pressure and flow rate, start the acquisition of spectra. After a baseline determination of at least 1 min, introduce the gas into the heated sampling line (see Figure C.1). Collect spectra for at least 3 min. Stop the CO_2 supply by closing the shutoff valve at the outlet tube of the CO_2 bottle. Continue the acquisition of the spectra for at least 2 min after the end of CO_2 supply.

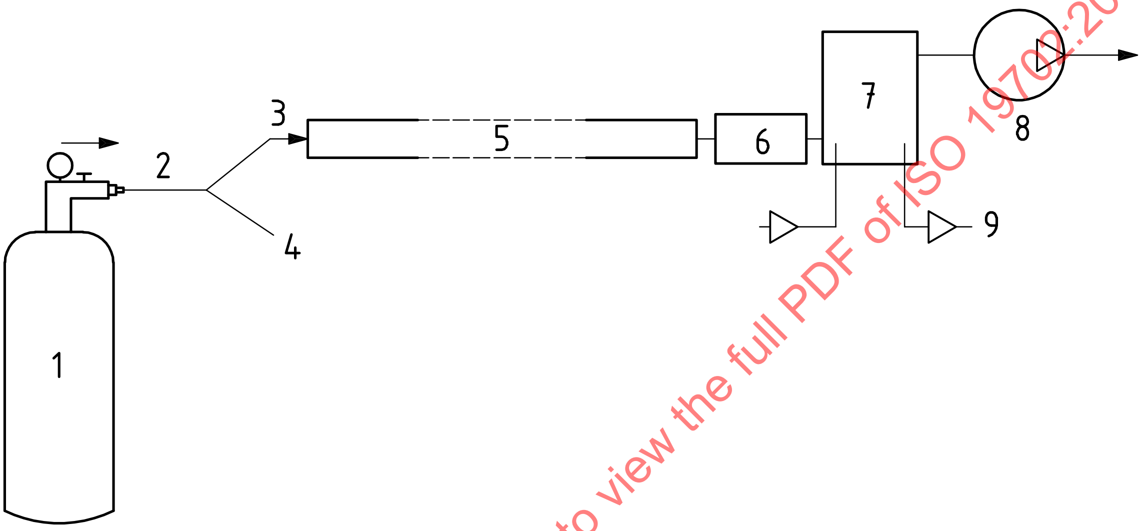
To record the evolution of CO_2 concentration, the spectral region of 710 cm^{-1} to 752 cm^{-1} can be used. Plot the concentration as a function of time. Determine the average concentration on the plateau of the curve, i.e. the concentration value of the ultimate deflection.

Calculate the 90 % rise time ($t_{90\%,\ up}$) and 10 % fall time ($t_{10\%,\ down}$) as follows:

$t_{90\%,\ up}$ is the time at which the CO_2 concentration exceeds the 90 % level of the plateau concentration minus the time of the last point of zero concentration in the beginning of the curve;

$t_{10\%,\ down}$ is the time at which the CO_2 concentration goes below the 10 % level of the plateau concentration minus the time of the last point of the plateau concentration in the end of the curve.

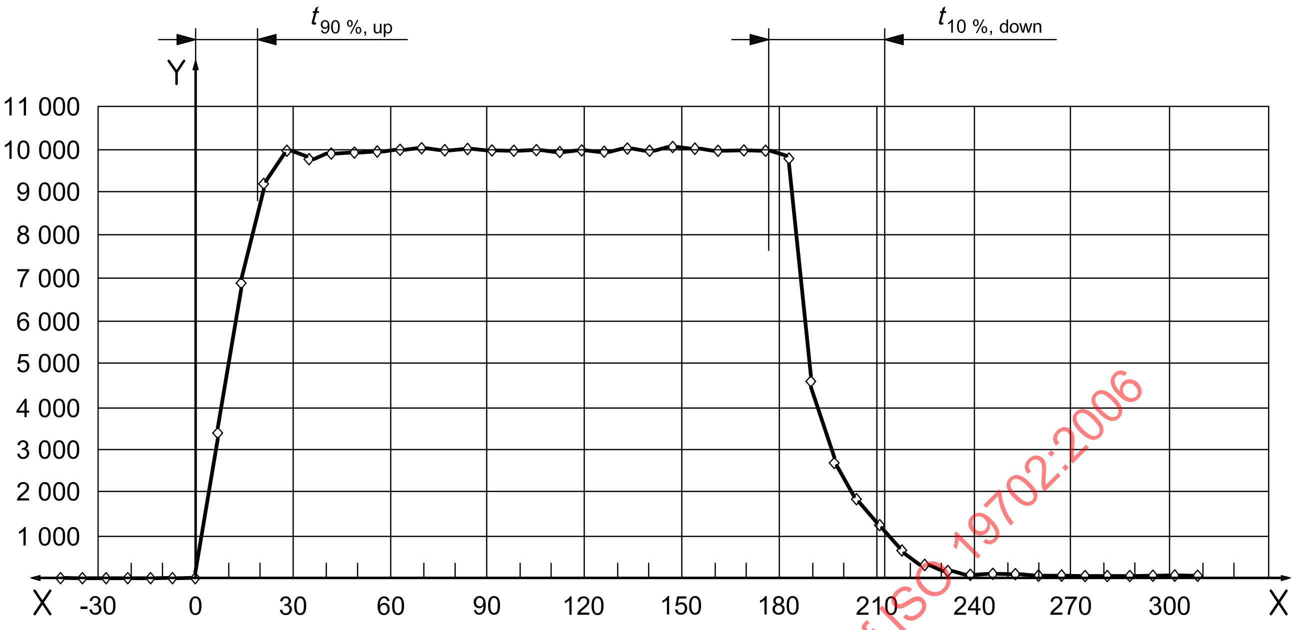
An example of the response time determination is shown in Figure C.2.



Key

1 CO_2 bottle
2 tube from the bottle to the “T”
3 tube from the “T” to the entry to the heated sampling line
4 ambient air
5 heated sampling line
6 filtering device
7 cell
8 pump; 8 l/min
9 IR beam

Figure C.1 — System set-up to define response time



Key

X time, expressed in seconds

Y concentration, expressed in millilitres per litre

Figure C.2 — Determination of FTIR response time for CO_2

Annex D
(informative)

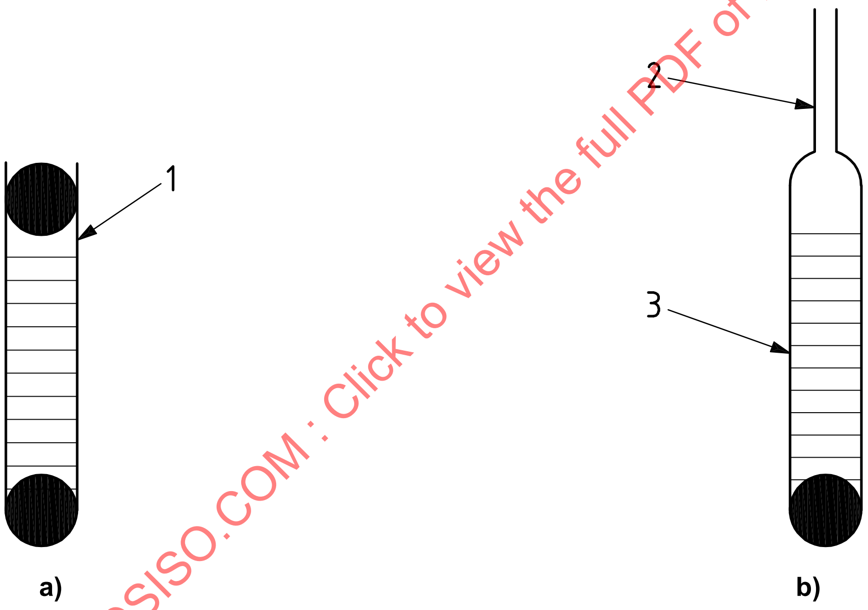
Calibration methods and the “diffusion tube” and “closed loop” methods

Calibration gases may be prepared in a number of ways:

a) Calibration gas mixtures, including water, may be purchased ready made in cylinders. These calibration gases may be further mixed or diluted using suitable equipment to extend the calibration range.

NOTE Experience has shown that particularly for reactive gases, the stated concentration given by the supplier are sometimes not be reliable.

b) Gas mixtures may be made in a dynamic system using permeation or diffusion tubes to produce a range of concentrations (Figure D.1). Gas concentrations are calculated from the mass loss of the diffusion or permeation tube. Subsequent analyses for verification may be carried out if needed.



a) b)

Key

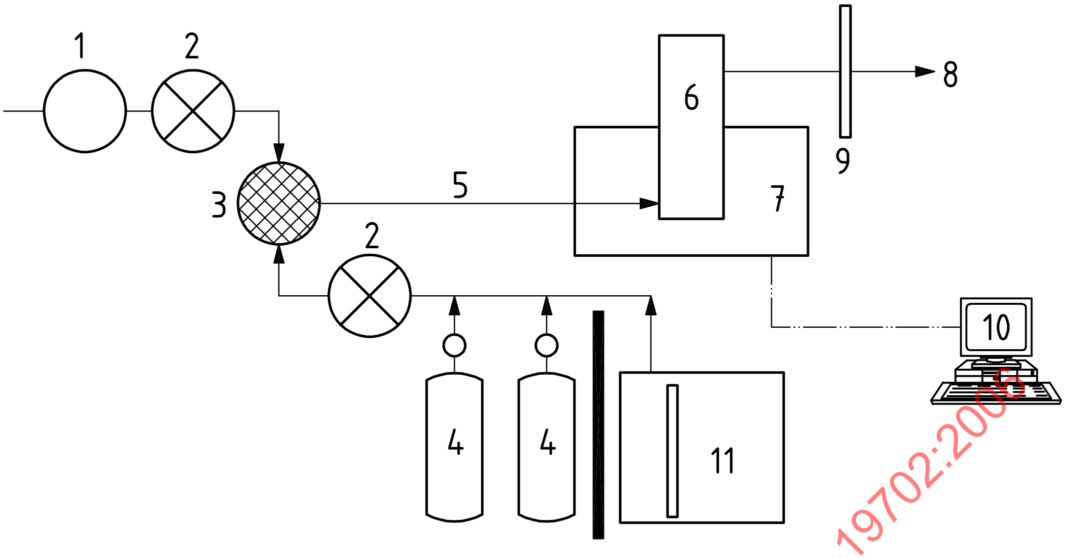
1 PFTE tube

2 capillary tube

3 glass

Figure D.1 — Schematic of prepared (a) permeation tube and (b) diffusion tube

A schematic of the calibration set-up when using gas cylinders or permeation tubes is shown in Figure D.2.

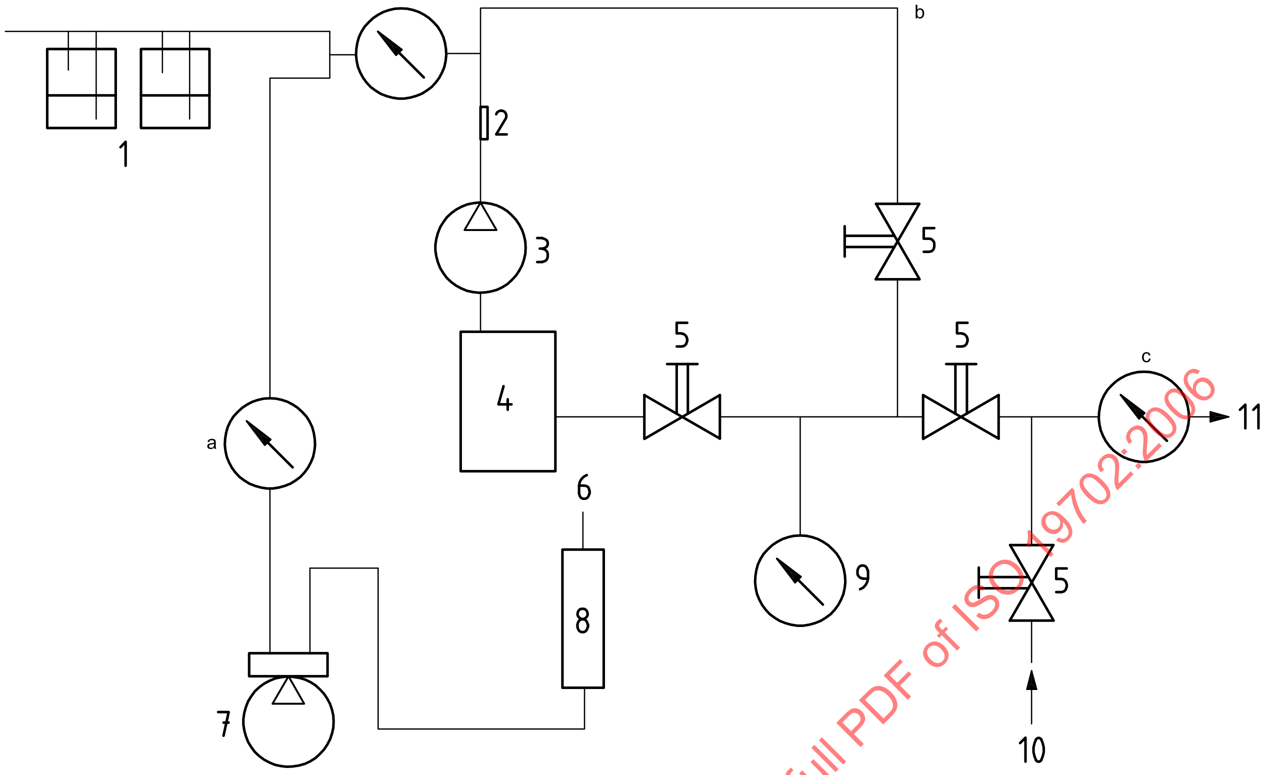


Key

1	diluent in
2	flowmeter
3	mixer
4	reference cylinder gases
5	heated line
6	heated cell
7	FTIR
8	flow out
9	total flow
10	computer
11	diffusion or permeation tubes

Figure D.2 — Schematic flow diagram for calibration by gas cylinder and permeation tubes

A heated, pumped, closed loop system attached to the FTIR cell may also be used to generate known gas concentrations for calibration. If the system is of known volume, then injecting known amounts of materials gives known concentrations (see Figure D.3). After the calibration spectra have been obtained, the contents of the cell and loop may be swept into an appropriate trapping medium for subsequent analysis to determine concentrations.



Key

1	bubblers
2	septum
3	pump
4	3,6 m path length FTIR cell
5	ON/OFF valve
6	exhaust
7	pump
8	rotometer
9	manometer
10	purge air
11	FTIR sampling line

[a] C2: flow and cell pressure control with 6 mm S/S bellows valves.

[b] Calibration loop system.

[c] C1: flow and cell pressure control with 6 mm S/S bellows valves.

Figure D.3 — Schematic flow diagram for closed loop calibration set-up

Annex E
(informative)

Spectrometer parameters

E.1 Detectors

Two types of detectors are commonly used in FTIR measurements. The first one is the so-called deuterated triGlycine sulfate, or DTGS, detector, which operates at room-temperature, and the second is the mercury-cadmium telluride detector, or MCT. The MCT detector is cooled by nitrogen and has a superior signal-to-noise ratio. A drawback with the MCT detector is that extra-equipment is needed to supply the FTIR apparatus with liquid nitrogen. Also, more time is needed to stabilize the MCT detector. Both detectors can be used for smoke gas analysis.

E.2 Resolution

The resolution defines the detail collected in the spectra. A low-resolution means that less species-specific information can be extracted from the spectra. However, the higher the resolution the longer one needs to obtain spectral information with a good signal-to-noise ratio to allow acceptable lower detection levels or sensitivity. Further, as fire tests often exhibit turbulent behaviour, it is important that measurements are made as often as possible in order to obtain good time resolution.

E.3 Minimum detection limit

The path length and signal-to-noise ratio determine the minimum detection limit (MDL). To estimate the MDL of a particular gas component, it is assumed that a spectral line can be detected when it is approximately as high as the noise level. It is also assumed that a linear relationship exists between the absorbance and the concentration for low concentrations. Under these assumption, the following procedure can be used.

a) From at least 10 different real smoke spectra, determine the peak-to-peak noise level of the spectra in a region free from peaks. Calculate the average $\overline{x}_{\text{pp}}$;

b) obtain the spectrum of the reference gas with the lowest concentration (c) and determine the highest absorbance value, α_{max}, of the spectrum in the region you use for building your calibration model (see Clause 6);

c) the minimum detection level, x_{MDL}, is the ratio of the peak-to-peak noise level to the highest absorbance value times the concentration of the reference gas, as given in Equation (E.1):

$$x_{\text{MDL}} = \frac{\overline{x}_{\text{pp}}}{\alpha_{\text{max}}} c \tag{E.1}$$

The greater the number of scans, the greater the signal-to-noise ratio. This effects the sensitivity of the spectrometer for different gases.

Annex F
(normative)

Recording the reference spectra and building the calibration set

F.1 Spectral properties of reference gases

The gases specifically studied within the SAFIR project are H_2O, CO_2, CO, NO, NO_2, HCN, HBr, HCl, and acrolein. Each gas has a unique FTIR spectrum which exhibits specific absorption peaks. In Table F.1, the spectral regions where these absorption peaks are observed are summarized. The maximum absorbance is given as an indication of the importance of this region.

NOTE These values relate to the specific spectrometer set up used when the spectra were collected and are cited here in order to show the relative importance of the various absorbances. The specific numbers will be dependent on the choice of spectral resolution, gas cell pathlength, detector, etc.

Table F.1 — Summary of spectral regions where absorption peaks are observed

Reference gas	Concentration µl/l	Spectral region		Maximum absorbance
		start cm^{-1}	end cm^{-1}	
H_2O	12 000	4 000	3 400	
		2 000	1 170	
		500	—	
CO_2	15 100	3 764	3 480	0,63
		2 400	2 200	$> 6,0$
		800	520	2,46
CO	3 005	2 264	1 975	0,17
Acrolein	322	3 457	3 374	0,01
		3 160	2 600	0,08
		1 783	1 584	0,55
		1 452	1 336	0,04
		1 200	1 100	0,08
		1 054	872	0,12
		670	500	0,02
NO	510	2 000	1 775	0,05
NO_2	470	2 939	2 815	0,08
		1 667	1 518	1,16
SO_2	960	2 525	2 442	0,04
		1 410	1 290	1,5
		1 253	1 029	0,13
		640	437	0,14
HCN	566	3 400	3 200	0,11
		1 550	1 300	0,04
		833	533	> 6
HCl	5 420	3 150	2 500	0,28
HBr	1 000	2 744	2 290	0,04
HF	148	4 200	4 000	0,01

This information can be more easily visualized in Figure F.1, where the spectral absorption regions are plotted against the wave number of absorption. Using this figure, one can easily verify which spectral regions overlap.

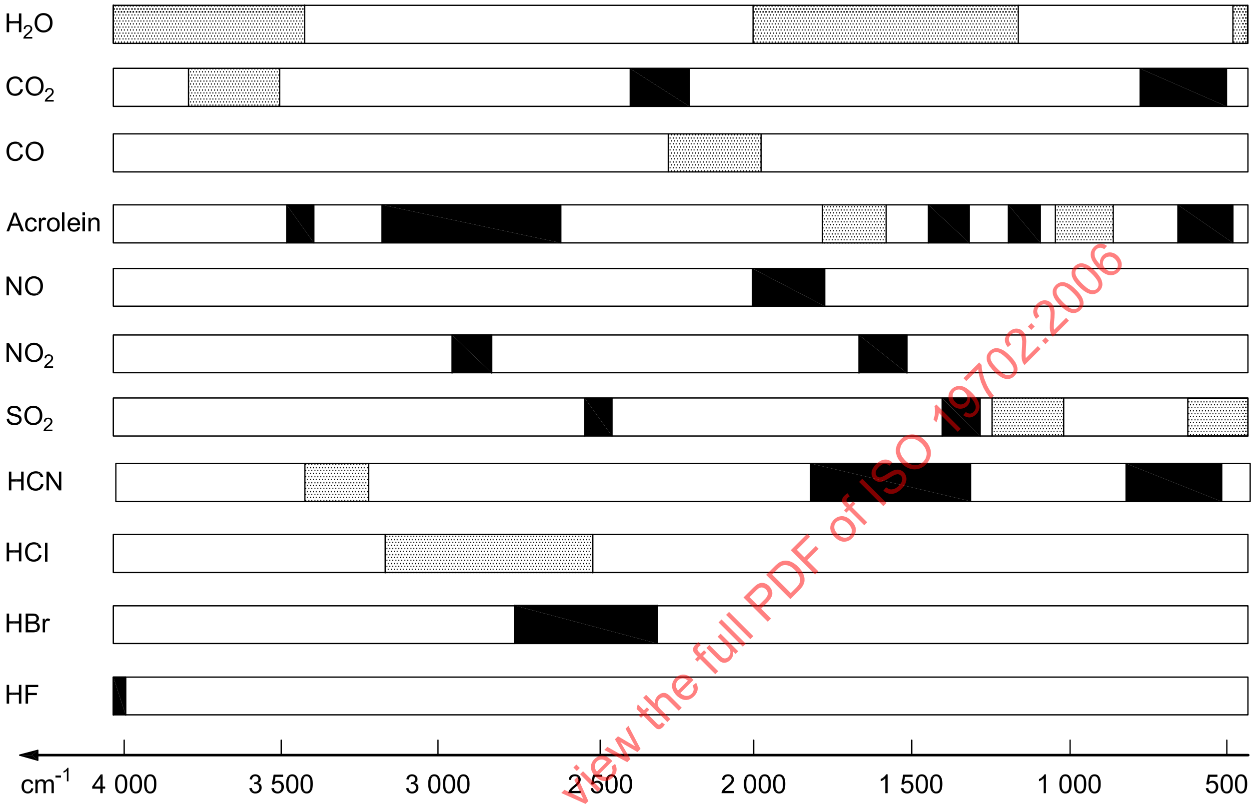


Figure F.1 — Spectral absorption regions for each gas component

F.2 Collecting the reference spectra

The calibration curve for a particular gas is usually built upon a set of reference spectra obtained on pure components of different concentrations.

NOTE The reference spectra may be composed of known combinations of gases in certain cases (e.g. when creating a calibration model for PLS analysis).

The number of concentrations which are necessary to obtain a calibration curve of sufficient reliability depends on the degree of linearity between absorbance and concentration (Beer's Law). When the absorbance-concentration relationship is linear then two concentrations are theoretically sufficient, although three are recommended. When deviation from linearity is not strong then 3 concentrations are sufficient, but 4 are recommended; and when there is strong non-linear behaviour, 5 concentrations are recommended. The concentration ranges normally expected in fire gases for each species (except water) and the recommended number of concentration points used to create the calibration model are summarized in Table F.2.

Table F.2 — Recommended concentrations ranges and number of points for calibration

Reference gas	Recommended number of concentrations	Minimum concentration µl/l	Maximum concentration µl/l
CO_2	5	100	15 000
CO	5	50	3 000
SO_2	2 to 3	50	1 000
Acrolein	2 to 3	50	300
NO	3	50	500
NO_2	3	50	500
HCl	5	100	5 000
HCN	4	50	500
HBr	4	50	1 000
HF	4	50	1 000

F.3 Collecting the spectra of the reference gases

When collecting the spectra of reference gases, the FTIR settings need to be identical to the settings during a test (as far as practically possible). Generally, it is sufficient to buy one or two concentrations of the reference gases in nitrogen and dilute these further to obtain a number of reference gas concentrations. In some cases, it can be necessary to create reference gases in the testing laboratory as they can be difficult to buy ready-made. In such cases, a variety of methods can be used depending on the gas to be manufactured (see Annex D). In all cases, the most important thing is that the reference gas be free from unspecified contaminants and that the concentration of the reference gas is well known.

F.4 Water spectra

Water spectra are necessary for building the calibration model as water interferes with the absorptions of many of the other gases one wishes to quantify. This means that, although the exact concentration of water produced in a fire is generally not important, it is important to quantify this in FTIR measurements in order to correct the spectra for the presence of water.

Annex G
(informative)

Example of equipment

Laboratory	1	2	3	4	5
Probe					
Probe type	linear multi-hole (CC); open-ended (smoke ch.)	open-ended	open-ended	open-ended	linear multi-hole (CC); open-ended (smoke ch.); none (tube furn.)
Material	stainless steel	stainless steel	stainless steel	stainless steel	stainless steel (CC); glass (smoke ch.)
Diameter of hole(s)	2,5 mm or 2,0 mm (CC); 4 mm (smoke ch.)	10 mm	8 mm	10 mm	2 mm (CC); 5 mm (smoke ch.)
Filtering system					
Filter type / model	S&S GF 50 or Whatman GF/A	filter syst.:M&C SP 2000 H; filters: S 2K150 (ceramic), F-0.1GF150 (glass fibre)	Unique Products Int., S-P-4-032-025-063-0	Glass (o.); BAISTON 95T-1/4 (n.)	white cylinder
Filter material	glass fibre	ceramic or glass fibre	glass, inorganic binder	glass fibre (o.); PTFE (n.)	microfibreglass, teflon coating
Filter porosity	—	ceramic: 2 µm; glass fibre: 0,1 µm	—	—	2 µm
Filter size	circular, $\varnothing = 47$ mm	ceramic: $\varnothing_{int} = 15{,}5$ mm, $\varnothing_{ext} = 29{,}5$ mm, $l = 150$ mm; glass fibre: $\varnothing_{int} = 25$ mm, $\varnothing_{ext} = 32$ mm, $l = 150$ mm	cylindrical: $\varnothing_{int} = 25{,}4$ mm, $\varnothing_{ext} = 31{,}8$ mm, $l = 63{,}5$ mm	circular, $\varnothing = 50$ mm (o.); cylindrical, $l = 58$ mm, $\varnothing = 18$ mm (n.)	cylindrical, $l = 175$ mm, $\varnothing = 32$ mm, thickness 3 mm to 4 mm
Replacement interval	each test	ceramic: cleaned every day (after about 9 tests); glass fibre: each test	2 to 3 tests for CC	each test	4 to 6 tests for CC, > 10 tests for smoke ch. and tubular furn.
Working temperature	RT	150 °C	185 °C	130 °C	150 °C
Temperature range	no regulation	⩽ 180 °C	⩽ 200 °C	⩽ 180 °C	⩽ 450 °C
Sampling line					
Length	3,8 m	4 m	10 m	variable, max. 5 m	5 m
Material	PTFE	PTFE	PTFE	PTFE	PTFE
Inner diameter	4 mm	6 mm	8 mm	10 mm	4 mm
Working temperature	130 °C (o.); 180 °C (n.)	150 °C	185 °C	130 °C	150 °C
Temperature range	⩽ 200 °C	⩽ 250°C	⩽ 200 °C	⩽ 200 °C	⩽ 380 °C
Pump location	after gas cell	after gas cell	after gas cell	after gas cell	after gas cell
Pressure	unknown	unknown	88 kPa (0,86 atm)	≈ 102 kPa (≈ 1 atm)	88 kPa (0,86 atm)
Flow rate	6 l/min (o.); 1 l/min (n.)	6 l/min to 10 l/min	20 l/min	5 l/min	10 l/min [for 88 kPa (0,86 atm)]
Flow rate range	max. 10 l/min (n.)	0 l/min to 10 l/min	—	0 l/min to 20 l/min	pressure dependent
Flow rate meas. syst.	flowmeter after pump	mass flow controller in front of pump	flow gauge before pump	flowmeter	absolute pressure gauge

Laboratory	6	7	8	9	10	11
Probe						
Probe type	linear multi-hole (CC); open-ended (dual smoke ch., NBS ch.)	open-ended	linear multi-hole (ISO room duct or door); open-ended (small scale)	linear multi-hole	open-ended	circular multi-hole
Material	stainless steel (CC, dual smoke ch.); PTFE (NBS ch.)	stainless steel or PTFE (CC); PTFE (tube furn.)	stainless steel	stainless steel	stainless steel	stainless steel
Diameter of holes	2,5 mm or 2,0 mm (CC); 5,0 mm (dual smoke ch., NBS ch.)	4 mm (CC); 3 mm (tube furn.)	2 mm or 3 mm (duct);	—	10 mm	2 mm
Filtering system						
Filter type / model	Whatman HGS, stainless steel filter unit, circular plate	Unique Products Inc., FLT 1584-B-B-6-A-AI-000	M&C PSP4000H (large scale); planar filter holder (small scale)	filtering syst.: M&C SP2000, filter: M&C S 2K 150	Glass (o.); BAISTON 95T-1/4 (n.)	Stainless steel cylinder Stainless steel filter unit
Filter material	glass fibre membrane	micro bonded element ceramic fibre	Cylindrical ceramic (M&CSP-2K) (large scale) Planar glass fiber + PTFE (small scale)	ceramic	glass fibre (o.); PTFE (n.)	layers glass wool/ glass beads, 3 mm o.d. glass fiber membrane Whatman GF/C
Filter porosity	1 µm	“micro pore”	Ceramic 2 µm PTFE 5 µm	2 µm	—	— 1 µm
Filter size	$\varnothing$ = 47 mm	cylindrical, l = 63 mm, $\varnothing_{int}$ = 26 mm, $\varnothing_{ext}$ = 31 mm	cylindrical, l = 75 mm, $\varnothing_{int}$ = 16 mm, $\varnothing_{ext}$ = 30 mm planar 47 mm $\varnothing$	cylindrical	circular, $\varnothing$ = 50 mm (o.); cylindrical, l = 58 mm, $\varnothing$ = 18 mm (n.)	circular layers, Φ_{Int} = 35 mm, l = 80 mm, circular, Φ_{NT} = 70 mm
Replacement interval	each test	each test	each test	each test	each test	When vacuum gauge downstream of filters decreases by 13,78 kPa (2 psi) (after about 10 tests) replaced daily (after about 2 to 5 tests)
Working temperature	(150 ± 10) °C	185 °C	180 °C	150 °C	130 °C	170 °C
Temperature range	$\leqslant$ 200 °C	$\leqslant$ 185 °C	$\leqslant$ 180 °C	$\leqslant$ 180 °C	$\leqslant$ 180 °C	$\leqslant$ 200 °C
Sampling line						
Length	2 m	4 m (CC); 1 m (tube furn.)	1,5 to 7 m	4 m	variable, max. 5 m	4 m
Material	PTFE	PTFE	PTFE	PTFE	PTFE	PTFE-lined stainless steel
Inner diameter	5 mm	3 mm	4 mm	6 mm	10 mm	4 mm
Working temperature	(150 ± 10) °C	185 °C	200 °C	160 °C	130 °C	170 °C
Temperature range	$\leqslant$ 200 °C	$\leqslant$ 185 °C	$\leqslant$ 250 °C	$\leqslant$ 250 °C	$\leqslant$ 200 °C	$\leqslant$ 200 °C

Pump location	after gas cell, end of line	after gas cell, end of line	after gas cell	after gas cell	after gas cell	Before gas cell
Pressure	105,8 kPa (1,033 atm) (dual smoke ch.); 112,0 kPa (1,094 atm) (CC, NBS ch.)	88 kPa (0,86 atm)	—	—	≈ 102 kPa (1 atm)	~102 kPa (1 atm)
Flow rate	4 l/min (dual smoke ch.); 7 l/min (CC, NBS ch.)	10 l/min to 14 l/min	4 l/min	7 l/min	5 l/min	11 l/min (probe) 5,0 l/min (cell)
Flow rate range	—	2 l/min to 15 l/min	1 l/min to 10 l/min	1 l/min to 10 l/min	0 l/min to 20 l/min	8 l/min to 11 l/min (probe) (pressure dependent) 5,0 l/min (cell) (pressure regulator maintains constant flow through cell and a constant cell pressure)(see Figures 1 and 2)
Flow rate meas. syst.	flowmeter before gas cell	calibrated flow meter	flow meter before pump, calibrated diaphragm gas meter after pump	mass flow controller in front of pump	flow meter	Sampling mode: Flowmeter after cell Calibration mode: Mass flowmeter before cell and flowmeter after cell