
**Room corner and open calorimeter —
Guidance on sampling and
measurement of effluent gas
production using FTIR technique**

*Mesurage de la production de gaz toxique à l'aide de la technique
IRTF pour l'essai en coin de salle et calorimétrie ouverte*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: [Foreword — Supplementary information](#).

The committee responsible for this document is ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

Introduction

This International Standard is intended to obtain concentrations of effluent gases produced in large-scale or simulated real-scale fire tests, such as the room corner test and open calorimeters. These tests describe the fire behaviour of a product under controlled laboratory conditions.

The test standard can be used as part of a fire hazard assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

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Room corner and open calorimeter — Guidance on sampling and measurement of effluent gas production using FTIR technique

1 Scope

This International Standard gives guidance concerning suitable apparatus and procedures to be used when applying the FTIR method to measure concentrations of effluent gases produced in large-scale or simulated real-scale fire tests. Such tests include the room corner test (see ISO 9705) and open calorimeter tests as described in ISO 24473.

This guidance and measuring method only describes the way in which the sampling of the gases and collection of FTIR spectra are performed. Analysis of spectra and calibration is part of ISO 19702.

2 Normative references

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

ISO 9705, *Fire tests — Full-scale room test for surface products*

ISO 13943, *Fire safety — Vocabulary*

ISO 19702:—¹⁾, *Guidance for sampling and analysis of toxic gases and vapours in fire effluents using Fourier Transform Infrared spectroscopy (FTIR)*

ISO 24473, *Fire tests — Open calorimetry — Measurement of the rate of production of heat and combustion products for fires of up to 40 MW*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM: 1995)*

3 Terms and definitions

For the purposes of this document, the definitions given in ISO 13943 apply.

4 Principle

By using the on-line FTIR technique, it is possible to simultaneously measure the time resolved concentration of several gases during a fire test.

The practical measurement procedure is to continuously extract a fraction of the effluents from the exhaust duct (the most common application) from the opening of the test room or, alternatively, from a position in the vicinity of a test object through a heated sampling system and into a heated optical cell. There, the specific absorption patterns of infrared-active species are recorded by a detector. This information is subsequently presented as an absorption spectrum that is used to determine the concentrations of effluent components. The frequency of collection of absorption spectra, the

1) To be published. (Revision of ISO 19702:2006)

characteristics of the flow in the exhaust duct (if applicable), and the residence time and flow pattern in the optical cell determine the time resolution of the measurements.

NOTE FTIR is based on infrared absorption. Polyatomic and heteronuclear diatomic compounds have absorption in the infrared region. Specific to FTIR is conversion of regular irradiance from a broad band infrared source into interfered irradiance by an interferometer and conversion of the recorded interferogram into a conventional wavelength spectrum. The main advantage of the FTIR technique is that information from all spectral elements is measured simultaneously and another advantage is that the measurement is made with a high optical throughput giving a high signal to noise ratio. See ISO 19702 for a more detailed background on FTIR theory.

5 Gas sampling system

5.1 General

The gas sampling system consists of a probe for sampling fire effluent gases, a filter system for removing particulates from the sampled gas, sampling tubing for transporting the gas to the FTIR gas cell, and a pump for drawing the gas. The parts of the sampling system placed before the FTIR gas cell shall be heated to avoid condensation and losses of certain water soluble compounds (e.g. HCl).

A temperature of the sampling system between 150 °C to 190 °C shall be used (see ISO 19702).

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

NOTE 1 It is important that the gas in the sampling system is heated to a temperature as close as possible to the set-temperature of the sampling system. Procedures for checking the gas temperature are given in ISO 19702.

Information on delay and response time of the overall system is necessary and shall be reported.

NOTE 2 Method for determination of the response and transport time of the measurement system is given in ISO 19702. Besides the transport time from the gas sampling probe to the gas cell, there is also a transport time from the room to the gas sampling probe.

NOTE 3 The response and delay time can be obtained at the same time as burner calibrations are performed (see ISO 9705). It also allows an overall check of the system.

5.2 Gas sampling probe

5.2.1 Sampling position

In enclosure tests, the normal sampling position is in the duct of the smoke collection system. This sampling position represents cooled and diluted fire effluents.

NOTE 1 This sampling position is preferred in many cases as matrix effects from the fire effluents are minimized by the dilution. A further advantage is that when fire effluents are quantitatively collected with the hood system, the production of toxic gases can be quantitatively measured.

NOTE 2 In the early stages of an enclosure fire or when sampling from a small fire in an enclosure, the dilution of the fire effluents can result in concentrations below practical detection limits.

An alternative sampling position in an enclosure test, which can be preferred in certain cases, is in the top of the doorway (i.e. sampling from the undiluted out-flowing fire effluents).

NOTE 3 For sampling from the doorway, it is important to ascertain that a representative sample is taken over the out-flowing area. It is further important to consider that hot fire gases sampled from the opening might not be sufficiently oxidized and will continue to react outside of the opening.

NOTE 4 For quantitative measurement of toxic gas production, the flow rate out from the room has to be quantified.

Another alternative sampling position in an enclosure test is at various local positions within the enclosure.

NOTE 5 The results from measurements at specific positions within an enclosure are, however, only relevant for the specific test scenario.

In open tests where the fire effluents are collected by a hood/smoke gas collection system, the normal sampling position is in the duct of the smoke collection system.

Alternative sampling positions in open tests are at various local positions in the vicinity of the fire.

NOTE 6 The results from point measurements in the vicinity of the fire are, however, only relevant for the specific test scenario.

5.2.2 Exhaust duct sampling application

The gas sampling probe shall extract a representative sample of gases from the exhaust duct.

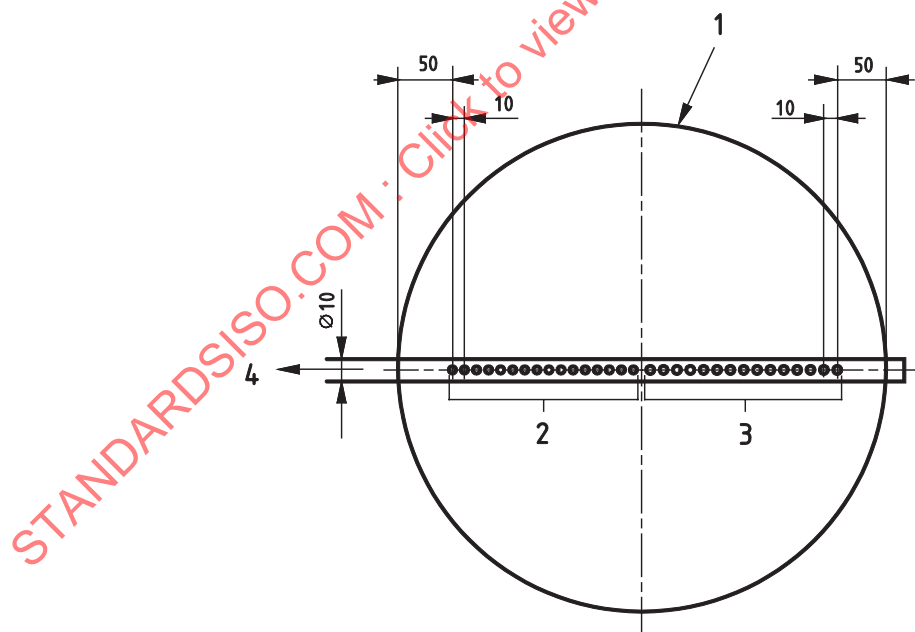
The probe shall be mounted at a position in the smoke gas duct where the diluted fire effluents are uniformly mixed.

NOTE 1 This is normally the case at a distance of 5 diameters to 10 diameters from the bend of the duct after the collection hood.

NOTE 2 A suitable probe construction and arrangement for the ISO 9705 test is shown in [Figure 1](#) (information on probe positioning is available in ISO 9705, Annex E).

NOTE 3 General information on sampling probes for FTIR measurement is available in ISO 19702.

Dimensions in mm



Key

- 1 exhaust duct
- 2 16 Ø 2 mm holes on downstream side of flow
- 3 15 Ø 3 mm holes on downstream side of flow
- 4 connection to the sampling line

Figure 1 — Gas sampling probe for the ISO 9705 room exhaust duct

5.2.3 Alternative sampling applications

When sampling directly from poorly mixed and inhomogeneous fire gases (for example, in the door opening of a test enclosure), one shall ascertain representative sampling.

NOTE 1 The flow patterns through the opening of a test enclosure are complicated and it varies during the course of a fire test. Typically, the upper part of the exit flow is well-mixed but this is surrounded by a relatively slow-moving boundary layer between effluent and air that can be partly diluted by the incoming air. Ideally, it is useful to observe the flow shape and characteristics for the particular test before selecting a probe type and its position in the effluent stream. It is likely that a multi-hole probe will be necessary.

One method to accomplish representative sampling is to use a multi-hole probe with gradation of hole sizes along the length of the probe and to place the probe such that it crosses the fire gases monitored.

NOTE 2 An example of a suitable multi-hole 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.^[3]

A simpler but less precise method is to place a single-port probe in a well-mixed location; in a door opening, the probe shall be placed in the relatively well-mixed upper layers of the out-flowing effluents.

Measurement of the volumetric exit flow rate from a door opening is necessary whatever probe type is used in order to convert measured concentrations to total flow of gas species out from the test enclosure.

NOTE 3 The exit flow in enclosure fire tests varies during a test. The rate of the exit flow grows with increasing fire intensity and the position of the neutral plane stabilises first at "steady-state" where the fire growth is relatively stable (e.g. due to ventilation control of burning rate rather than fuel involvement control of burning rate).

NOTE 4 The exit flow can be measured using traditional velocity probes such as pitot tubes, McCaffrey probes, or more advanced optical methods such as particle image velocimetry. It is, however, not always possible to directly measure the exit flow using velocity probes as the pressure difference over the opening is relatively small and the often turbulent outflows in the opening have velocity components in directions other than the normal direction to the exit plane.

NOTE 5 An alternative method is to calculate the exit flow based on the pressure difference between the room and outside and the height of the neutral plane (i.e. a virtual horizontal plane separating the outgoing effluent from the incoming air) in the stratified case. An example of this method is given in Reference [4] and more details can be found in Reference [5].

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.	

5.3 Filter

A heated filter shall be placed directly after the probe to remove soot and other particulates.

NOTE 1 This is necessary as soot, otherwise, might accumulate in the sampling tubing and obstruct the sampling flow. It is further necessary to remove any particulates before entering the gas cell where mirrors and windows can become contaminated with a reduced optical throughput of the instrument as a result.

The type of filter recommended is a high capacity cylindrical filter.

NOTE 2 The choice of filter porosity and filter area is often a compromise. A fine porosity gives a high degree of soot removal but often leads to relatively fast blocking of the filter. A large filter area increases the capacity of the filter but can increase losses of e.g. HCl in the filter (possibly due to insufficient heating of the larger filter or due to the faster saturation behaviour of a smaller filter).

NOTE 3 Details on the selection of filters are given in ISO 19702.

The filter characteristics shall be reported (see [Clause 9](#)).

The filter housing shall be heated to at least 150 °C (the maximum is 190 °C).

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

NOTE 4 ISO 19702 provides information for the analysis procedure.

5.4 Tubing

Tubing for transportation of the sampled gas to the gas cell of the FTIR shall be made of a non-reactive heat resistant material.

Sampling tubing made of PTFE with an internal diameter of 3 mm to 4 mm is suitable. The length shall be kept as short as practically possible (by preference less than 3 m but lengths of up to 10 m can be needed in some installations).

The tubing shall be maintained at a constant temperature of at least 150 °C (the maximum is 190 °C).

If long sampling lines (longer than 3 m) are used, a consequence analysis should be done and information on the increase of delay time should be given in the report.

NOTE 3 If there are hot spots in the sampling system, PTFE can distort. ISO 19702 provides more information.

5.5 Pump

The pump shall transport the sampled gases through the FTIR gas cell preferable at a constant volume flow rate during the fire test. The required volume flow rate depends on the volume of the gas cell and of the desired time resolution of the measurement.

NOTE 1 The required flow rate of the combustion gases drawn into the system must be high enough to achieve the desired time to 90 percent response. This is a function of the flow rate and the internal volumes of the filters, tubing, and gas cell. See ISO 19702 for the procedure to determine response times.

The pump can be positioned before or after the FTIR gas cell.

NOTE 2 Positioning of the pump after the gas cell and letting the pump pull the gases through the cell is the most common technical solution. The advantage is that an unheated pump can be used (removal of water before the fire effluent gases entering the pump is, however, often necessary). The disadvantage is that the pressure in the gas cell can be affected by the pump.

For a pump positioned after the gas cell, the pressure in the FTIR gas cell shall be monitored and the pump speed should be adjusted, if needed, to keep a constant pressure. If this is not possible, corrections of the results shall be made based on the pressure measurement in the cell.

NOTE 3 Positioning of the pump before the gas cell and letting the pump push the gases through the cell is a possible technical solution. This requires a specially designed heated pump ($>150\text{ }^{\circ}\text{C}$, max $190\text{ }^{\circ}\text{C}$) and a thorough investigation of possible risks for losses in the pump. The advantage is that the pressure is maintained constant in the gas cell as it works against the atmosphere. The risk of dilution of the sample gas from any leakages in the sampling system is further reduced.

NOTE 4 Pulsation of pressure at the outlet of the pump positioned before the gas cell can affect the measurement at the cell.

NOTE 5 The high flow rate in the fire effluent duct of the large-scale and simulated real-scale fire tests sets no practical upper restriction on the pump flow rate for the FTIR measurements.

6 FTIR instrument

6.1 General

The main parts of an FTIR instrument suitable for measurements of fire gases are a heated corrosion-resistant multi-path gas cell, an IR source, a spectrometer, and a detector. A data acquisition and processing unit and software for spectral evaluation are also vital parts of the instrumentation.

Since the measurements are performed in a large-scale or simulated real-scale fire test, stability of the FTIR instrument should be ensured (i.e. protection against e.g. heat, vibration, extinguishing media).

6.2 Gas cell

The gas cell shall have an internal volume that gives an acceptable response time and an optical path length that allows acceptable detection limits.

NOTE 1 The volume of the gas cell determines to a large extent the response time of the measurement. Cell volumes of between $0,2\text{ dm}^3$ and $2,0\text{ dm}^3$ have successfully been used for fire testing. The optical path length of the cell determines the strength of the signal measured by the detector (c.f. Lambert-Beer's law). The possible choice of path length varies with different cells (make and volume), but normal path lengths used are between 2 m and 10 m.

NOTE 2 For the large-scale applications that are covered in this International Standard, it is possible to use a larger cell volume and a longer path length from the ranges given in Note 1. An acceptable response time can still be met as there generally are no restrictions in sampling flow rate in large-scale applications.

NOTE 3 ISO 19702 gives detailed guidance on gas cell construction and considerations for optical cell performance.

The gas cell shall be heated to a minimum of 150°C .

NOTE 4 Recommended maximum cell temperature is $190\text{ }^{\circ}\text{C}$ as fittings and gaskets can be degraded over time at higher temperatures.

Both the temperature and pressure in the cell shall be monitored throughout the fire test.

It is important that both the cell pressure and temperature are kept constant as this affects the intensity and shape of the spectra. If the temperature or pressure in the cell during measurements in a fire test cannot be maintained at the same value as those used when collecting calibration spectra, then a correction should be made for this difference when calculating species concentrations. This correction can be made using the universal gas law. However, the correction is valid only for limited variation in pressure and temperature. This correction might not be completely effective in every case due to non-linear changes in the shape of spectra with pressure and temperature. More information is available in ISO 19702.

The gas cell shall be well maintained to keep the optical performance.

6.3 Spectrometer parameters

A suitable resolution of the spectrometer is between 0,5 cm⁻¹ and 4 cm⁻¹.

NOTE 1 The advantage of a higher resolution (i.e. a lower cm⁻¹ value) is that more of the spectral fine structure is resolved. However, a scan at a higher resolution takes more time. It is further advantageous to average a number of scans for each spectrum, as the noise is reduced as a function of measurement time (the noise is inversely proportional to the square root of the number of scans). The scanning time (and noise level) varies for different FTIR instruments and the specific settings have thus to be individually made for each type of instrument to achieve enough sensitivity and a satisfying time resolution.

NOTE 2 A resolution of 0,5 cm⁻¹ is recommended for elimination of interferences in fire gas analysis. More information is available in ISO 19702.

The spectral range of the measurement determines the amount of information acquired. In order to collect information of all compounds that are generally of interest in a fire test, a wave number range from 4 500 cm⁻¹ to 650 cm⁻¹ is appropriate.

NOTE 3 A collection rate of a minimum 4 spectra/min is recommended in order to capture the dynamic fire behaviour of e.g. a room-fire.

NOTE 4 Depending on the desired output from the fire test, 4 spectra/min might not be sufficient to capture peak values and an integrated area, reflective of the total yield of key gases. The fire stages of some room-scale fire tests might last only 3 min or less and might need a higher collection rate of spectra.

6.4 Detector

Guidance on type of detectors shall follow ISO 19702.

7 Measurement

7.1 Requirements

To make quantitative measurements of fire effluent gases, special calibration of the instrument is mandatory. Calibration has to be made for the gaseous species that are to be measured. Calibration shall additionally be made for species interfering in the spectra which, most importantly, are water (H₂O) and carbon dioxide (CO₂).

7.2 Calibration

Calibration shall be made with the same settings of the instrument and the same physical conditions (i.e. cell pressure and cell temperature) that will be present during fire test measurements.

The absorbance of a gas shows, in most cases, a non-linear relationship against concentration in practical FTIR measurements. The calibration curve shall thus be based on several data points.

Detailed recommendations and procedures for calibration are given in ISO 19702.

7.3 Test procedure

The measurement shall be made with the same settings of the instrument and the same physical conditions (i.e. cell pressure and cell temperature) that were present during calibration.

An appropriate test procedure is as follows.

- Change to a clean filter in the sampling system.
- Ensure that all parts of the sampling system have stabilised at the desired temperature (≥150 °C).

- Ensure that the temperature of the FTIR gas cell is stable at the desired temperature (≥ 150 °C).
- Record a reference spectrum with N₂ flushed through the gas cell.
- Start the pump of the sampling system. For systems where the sampling pump is run continuously, change the line from purging system to the line from the gas sampling probe.
- Start the on-line data acquisition software of the FTIR instrument at the starting point of the fire test or at a defined earlier time to record a baseline. Collect averaged FTIR spectra at a minimum frequency of one averaged spectrum per 15 s.
- Record the gas cell temperature and pressure during the test.
- Make notes of important occurrences in the fire test such as ignition, flash-over, and any other events that influence the production and collection of fire effluent gases.
- Stop the on-line data acquisition software of the FTIR instrument at the end point of the fire test.
- Stop the pump of the sampling system. For systems where the sampling pump is run continuously, change the line from gas sampling probe to the line of purging system.
- Flush the gas cell with nitrogen to clean the cell.
- Store the data safely.

8 Analysis of spectra

Analysis of smoke gas spectra is necessary for extracting quantitative data of the gas species calibrated for. The analysis of a spectrum could be based on peak height or the area of a limited region of the spectrum. It is advisable to avoid regions where other gases overlap, if possible. A calculation routine is used to simplify the analysis. There are a number of possible mathematical methods that can be used for the analysis. It is beyond the scope of this International Standard to describe these methods.

More details can be found in ISO 19702.

9 Expression of results

The report shall include the following for each gas measured.

- Calculated concentrations as measured in the gases from the exhaust duct can be presented as time dependent concentrations in microliter/litre or as integrated values.
- As concentrations in the gases from the exhaust duct are directly dependent on the mass flow rate in the exhaust system, a more general way is to express time dependent results as gas production rates in g/s.
- Total production of each gas (typically reported in units of g). This is obtained by integration over the relevant time period.
- Total yield of each gas (typically reported in units of g/g). This can be calculated when the total mass loss of the test specimen is known.
- Detailed information on the characteristics of the whole measuring system (see ISO 19702). Such information will include e.g. delay time, response time, filter information, detection limits, measuring range, calibration routines and results, reference spectra, and information about analysis procedures.
- An uncertainty analysis of the measurements. Guidance can be found in ISO/IEC Guide 98-3.

Calculation procedures are given in [Annex A](#).

NOTE More guidance, detailed information, and examples on calculation of yields and equivalence ratios can be found in ISO 19703.

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Annex A (normative)

Calculation

A.1 Volume flow

For the instrumentation in the duct described in ISO 9705, Annex E or ISO 24473, the volume flow in the exhaust duct, $\dot{V}_{298}$, expressed in cubic metres per second, related to atmospheric pressure and an ambient temperature of 25 °C, is given by Formula (A.1).

$$\dot{V}_{298} = (Ak_t / k_p) \frac{1}{\rho_{298}} (2\Delta p T_o \rho_o / T_s)^{1/2} = 22,59 (Ak_t / k_p) (\Delta p / T_s)^{1/2} \quad (\text{A.1})$$

where

T_s is the gas temperature in the exhaust duct, expressed in Kelvin (K);

T_o is 273,15 K;

Δp is the pressure difference measured by the bi-directional probe, expressed in Pascal (Pa);

ρ_{298} is the air density at 25 °C and atmospheric pressure, expressed in kilograms per cubic metre (kg m^{-3});

ρ_o is the air density at 0 °C and 0,1 MPa, expressed in kilograms per cubic metre (kg m^{-3});

A is the cross-sectional area of exhaust duct, expressed in square metres (m^2);

k_t is the ratio of the average mass flow per unit area to mass flow per unit area in the centre of the exhaust duct;

k_p is the Reynolds number correction for the bi-directional probe suggested by McCaffrey and Heskestad. In the exhaust, duct conditions are such that Re is usually larger than 3 800 hence, k_p can be taken as constant and equal to 1,08;

22,59 is a factor based on $\rho_{298} = 1,168 \text{ 4 kg}\cdot\text{m}^{-3}$ and $\rho_o = 1,275 \text{ 3 kg}\cdot\text{m}^{-3}$ ($\text{m}^{1.5}\cdot\text{K}^{0.5}\cdot\text{kg}^{-0.5}$).

Formula (A.1) assumes that density changes in the combustion gases (related to air) are caused solely by the temperature increase. Corrections due to a changed chemical composition or humidity content may be ignored except in studies of the extinguishment process with water. The calibration constant k_t is determined by measuring the temperature and flow profile inside the exhaust duct along a cross-sectional diameter. Several series of measurements shall be made with representative mass flows and with both warm and cold gas flows. The difference between different measurements when determining the k_t factor shall not exceed $\pm 3 \%$.

A.2 Combustion gases

By measuring the mole fraction of a specified gas in the collection duct or in the doorway of a room test, it is possible to calculate the instantaneous rate of gas flow and the total production of the gas. The calculations for measurements made in the collection duct of an open calorimetry tests are equivalent.