
Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 3: Guidance on measurement

*Essais de réaction au feu — Débit calorifique, taux de dégagement de
fumée et taux de perte de masse —
Partie 3: Lignes directrices relatives au mesurage*

ISO/TR 5660-3:2003

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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Capability and limitations of the cone calorimeter	1
4 Calibration of the cone calorimeter	2
5 Test specimen preparation and presentation	6
6 Selection of heat flux	8
7 Ignition protocols	10
8 Guidance on the testing of non-standard products	11
9 Composites and layered products	19
10 Liquids	20
11 The theory of oxygen consumption calorimetry	21
12 Start and end of test	24
13 Recommendations for presentation of data	25
Annex A (normative) Effect of additives and fillers	29
Bibliography	32

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.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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/TR 5660-3 was prepared by Technical Committee ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

ISO/TR 5660 consists of the following parts, under the general title *Reaction-to-fire tests — Heat release, smoke production and mass loss rate*:

- *Part 1: Heat release rate (cone calorimeter method)*
- *Part 2: Smoke production rate (dynamic measurement)*
- *Part 3: Guidance on measurement* [Technical Report]

Introduction

ISO 5660-1, describing a test method for rate of heat release from building products by means of a cone calorimeter, was published in 1993 (first edition) following approximately ten years of development within ISO/TC 92, *Fire safety*, Subcommittee SC1, *Fire initiation and growth*.

The cone calorimeter is a fire test instrument in which horizontal specimens are exposed to controlled levels of radiant heating by means of a truncated cone-shaped heater. Continuous spark ignition is provided and the time to ignition is recorded for specimens which ignite. The rate of heat release from the burning specimen is determined from measurements of the amount of oxygen consumed from the air flowing through the apparatus, which has been demonstrated to equate to heat release. The mass of the specimen is also measured throughout the burning period. The specimens are usually tested under well ventilated conditions.

Results are expressed in terms of peak and average rates of heat release as well as total heat released and the effective net heat of combustion. ISO 5660-1 limits the specimen type to essentially flat. Several other groups are now utilizing the cone calorimeter and a number of new parameters in addition to those defined in ISO 5660-1 and ISO 5660-2^[30] have been defined and used. Some of these, including smoke measurement, require that measurements be made from the beginning of the test rather than at the onset of ignition which is commonly used as the starting point for heat release measurement.

The cone calorimeter is also designed to allow measurement of smoke and gases such as CO and CO₂. Smoke measurement is the subject of ISO 5660-2^[30]. Further work is under way to define a quality control tool for measuring burning rates of building products. This will be published as ISO 17554^[22] and is based on mass loss measurements using the thermal decomposition model of the cone calorimeter. A similar system which measures the temperature of combustion products generated by this apparatus and has been standardized by ISO/TC 61 as ISO 13927^[23]. The cone calorimeter fire model has also been used in a corrosivity International Standard developed by ISO/TC 61 as ISO 11907-4^[24]. The effect of the evolved gases on the resistance of a printed circuit board target is used to assess corrosivity.

During development of the cone calorimeter it became apparent that there was considerable interest in the use of the instrument for products other than building products. Several standards have been developed by various national and international groups based on ISO 5660-1 and ISO 5660-2.

This part of ISO 5660 provides recommendations for the testing of products in the cone calorimeter and gives guidance on the presentation of the results. Supplementary guidance is given in documents referred to in References [1] and [2].

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Reaction-to-fire tests — Heat release, smoke production and mass loss rate —

Part 3: Guidance on measurement

1 Scope

This part of ISO 5660 examines the limitations of the cone calorimeter as currently used for building products and recommends ways in which some of these may be overcome for other types of products for other application areas. It compiles information from a large body of experience with regard to the use of the instrument into a set of guidelines which will help to standardize the use of the cone calorimeter in this wider scope.

Particular guidance is given on aspects of specimen preparation and on the behaviour, such as melting, spalling and intumescenting, of specimens exposed to radiant heat. The relevance of specimen thickness and the use of substrate, and methods of fixing to substrate, are also discussed. Advice is given on approaches to testing a variety of “non-standard” products. Recommendations are made on techniques of calibration of the apparatus, selection of appropriate heat flux levels and ignition protocols.

In addition to the guidance given to operators, the document makes recommendations on presentation of the test results.

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)*

3 Capability and limitations of the cone calorimeter

Rate of heat release is one of the fundamental properties of fire and should almost always be taken into account in any assessment of fire hazard. Heat release significantly affects fire growth. Considerable progress has been made in methods of using rate of heat release and ignition time results from the cone calorimeter to predict full scale fire characteristics such as time to flashover in a small room lined with the tested product and exposed to a high energy fire source such as that used in ISO 9705^[3].

The design of the instrument also provides for measurement of smoke (both gravimetrically and optically) and other gaseous products of pyrolysis or combustion. The instrument may thus be applied to the assessment of real fire hazards such as smoke and toxic and corrosive gas emission in addition to heat release, particularly when the results are expressed in terms of fundamental physically-based rather than ad hoc parameters.

When functioning purely as a rate of heat release apparatus the parameter which is measured in the plume from the specimen is the concentration of oxygen. Temperature measurements are made, but these are not used to measure the heat output from the specimen in the manner of a conventional calorimeter. This is a crucial point in understanding heat release by oxygen consumption calorimetry. The theory of oxygen consumption calorimetry is discussed in more detail in Clause 11.

The instrument is limited to bench scale specimens and it uses a simple fire model which provides continuous free ventilation and removal of the products of partial and complete combustion. Specimen behaviour during the experiment such as shrinking and swelling can be tolerated if this happens within small margins, but if the specimen intumesces so that it touches the igniter or the cone, or if it exhibits spalling, this behaviour will invalidate the results generated.

4 Calibration of the cone calorimeter

4.1 General

Regular and accurate calibration of several measuring devices is essential in order for valid results to be obtained from the cone calorimeter. The following calibration procedures are outlined in ISO 5660-1:2002, Clause 10 (respectively 10.1 to 10.3):

- preliminary calibration;
- operating calibration;
- less frequent calibrations.

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Table 1 gives details of the major calibration requirements together with recommended intervals.

Calibration procedures are to some extent controlled by the apparatus and the comments below may not apply to all makes of cone calorimeter.

Some guidelines are given on actual operating experiences with these calibrations and follow the clause headings given in ISO 5660-1. In addition there are some additional comments on low orifice calibration factors and the cause thereof. The clause numbers in parentheses refer to clauses given in ISO 5660-1:2002.

4.2 Heat flux meter calibration (see 6.12 and 10.3.1 of ISO 5660-1:2002)

Early work on cross calibration of the heat flux meters showed some differences in the calibration constant as given by the supplying company. It is therefore very wise to attempt some cross calibration with another cone calorimeter user.

Great care should be taken of the heat flux meter which is in regular use and care should be taken to use this always with water cooling. It should be checked regularly against a primary meter as set out in Annex E of ISO 5660-1:2002 to ensure its continued correct working. It is false economy to omit this regular check. ISO/TS 14934 gives guidance on heat flux meter calibration.

4.3 Heater calibration (see 10.1.2 and 10.2.5 of ISO 5660-1:2002)

The setting of the required heat flux is set out in the manuals of the different instruments. Once a steady state value has been obtained (small fluctuations of the order of $\pm 0,1$ °C may occur) this value should be noted for future reference and act as an early warning of some change. In particular, users should ensure that the control thermocouples which should be situated behind and touching the heater helix (i.e. the face remote from the specimen) do not penetrate the heater helix and experience the temperature of the flame rather than that of the heater winding.

Table 1 — Frequency for various calibration procedures

Equipment item	Prior to run	Daily	Weekly	Monthly	Occasional ^a
Drying/CO ₂ removal columns	Check that unused portion is sufficient ^b	—	—	—	—
Oxygen analyser	Span	Pressure/zero	—	—	Analyser flow rates time offsets
Main filter	Check and replace if needed	—	—	—	—
Soot mass filter	Place in position	—	—	—	Controller set up
Load cell	Check loading tare and mass	Calibrate	—	—	—
Irradiance	Check temperature	Heat flux level ^c	—	Heat flux/temperature relationship Heat flux meter against reference meter	—
CO/CO ₂	—	Zero/Span	—	—	—
Methane	—	Heat release flow rate of 5 kW for methane burnt	—	Heat release flow rates of 5 kW and 10 kW for methane burnt Mass flow controller	—
Laser smoke photometer	Check adjustment and 100 % transmission	Check response with filter	—	Check photometer zero	—
Differential pressure transducer	—	Check zero	—	—	Check calibration
PMMA burn	—	—	Perform test	—	—
^a These calibrations need only be carried out very occasionally or when alterations have been made to the system. ^b Always before spanning the oxygen analyser. ^c Also when required to change irradiance level.					

4.4 Oxygen analyser calibration (see 10.1.5, 10.1.6 and 10.2.3 of ISO 5660-1:2002)

Few problems should be encountered when carrying out the calibration of the oxygen analyser. When running the “zero” check using pure nitrogen with analysers equipped to measure pressure in the sensor cell, it has been found easier to set the nitrogen flow using the analyser pressure reading. The nitrogen flow is adjusted until the pressure reading is the same as when the analyser is fed from the atmosphere.

The oxygen analyser delay time should be determined from time to time (recommended frequency, once every three months) as set out in 10.1.5 of ISO 5660-1:2002. It should be remembered that because of the time offset, the amount of valid data collected would be lower than the total test time by the extent of the delay time. Thus testing should continue for at least 2 min after the last point of interest.

4.5 Determining orifice plate calibration factor

4.5.1 Calibration using methane (see 10.2.4 of ISO 5660-1:2002)

It is recommended that the calibration consisting of burning methane be carried out when the heater has been set at the required heat flux. This allows the differential pressure transducer (DPT) to warm up. The fan is shut down and the DPT re-adjusted to zero. The fan is then set to the required air flow and then the burning of methane is carried out.

ISO 5660-1 requires that at the start of each day, one heat release calibration corresponding to a heat release flow rate of 5 kW of the supplied methane be carried out. An orifice constant between 0,040 and 0,046 should be obtained with 99,9 % or 99,5 % methane at a flow rate of 8 l/min referenced to standard temperature and pressure, or 0,1 g/s (6 g/min). Daily calibration factors should agree within approximately ± 1 %.

It should be noted that the heat release calibration using methane does not constitute an absolute calibration of the instrument, but rather that it verifies the orifice plate constant, which appears in the calculations [see Equation (5) in 12.1, Equation (7) in 12.3.2 and Equation (9) in 12.4 of ISO 5660-1:2002]. It is not a direct measurement of heat release.

Black polymethylmethacrylate (PMMA) (with a thickness of 6 mm or greater) can also be used within each laboratory to check repeatability of the cone calorimeter performance.

When zeroing the differential pressure transducer (DPT), ensure that the duct fan and any "decoupled" extractor system are switched off. Air should be prevented from flowing over the open end of the stack and across the orifice plate. If necessary, a plastic bag or equivalent should be used to block the open end of the stack.

It is important to keep records of the values of X_{O_2} (oxygen analyser reading, mole fraction of oxygen), T_e (absolute temperature of gas at the orifice meter) and Δp (orifice meter pressure differential) which lead to good calibration factors which should also be noted every time the calibration is carried out. In this way any discrepancy is immediately identified and early correction can be carried out.

4.5.2 Calibration using liquids

It should be noted that when calibrating using liquids, which usually have low flash points, it is essential that calibrations be performed on a cold system (the cone heater is not powered). The liquid should be held in a stable vessel, and the vessel should be stable under the cone before ignition of the liquid. The burning liquid should not be disturbed until it is all burned.

In addition to burning methane for calibration, users have used a variety of materials such as alcohols. The heats of combustion of ethanol and propan-2-ol are 26,8 kJ/g and 30,2 kJ/g, respectively. It is desirable to use propan-2-ol with a purity $\geq 99,5$ %.

4.6 Weighing device calibration (see 10.1.3, 10.1.4 and 10.2.3 of ISO 5660-1:2002)

The weighing device is simple and generally problem free. Follow the manufacturer's instructions on recommendations for taring, calibrating and setting the damping of the weighing device.

4.7 Additional comments on the orifice calibration factor

Some variation of the orifice plate calibration factor (also known as the methane calibration factor) may be observed for various reasons. However, any large (5 %) changes in values are indicative of malfunction in the system. In the majority of cases, the problem is caused by leaks into the sampling lines, in which case the recorded factor will be higher than usual. Other items that can cause problems are

- blockages in the gas sampling line,
- connections between the orifice plate and the differential pressure transducer,

- leaks at the methane supply line,
- faulty differential pressure transducer, or
- faulty methane flow meter.

The most common cause for this is some leakage in the system. However, in one instance a user encountered low values of the order indicated above. After extensive testing indicating that no leaks were present in the system and everything else was functioning correctly, the refrigeration system was suspected and it was flushed with propanone. As the effluent was heavily discoloured, it was thought that the instability was caused by a severely clogged refrigeration column. Subsequent re-calibration gave a satisfactory calibration factor. A low calibration factor may also be the result of inactive CO₂ removal agent. If CO₂ is not removed from the gas stream entering the oxygen analyser, the heat release determined using the standard equations will be higher than expected, hence the calibration factor will be lower.

4.8 Calibration of smoke measurement system

Calibration using filters assumes that the system used to calibrate the filter is superior to the optical system in the cone calorimeter. The photodiodes used in the cone calorimeter specify a high degree of linearity. The optical density quoted for a commercially supplied filter is usually the average over a range of wavelengths and the value at the frequency of the monochromatic laser used in the cone may not be this average value. Therefore, the use of the filter is better confined for daily routine checking of the proper functioning of the system rather than as a primary calibration.

The user may therefore calibrate by checking zero and 100 % values and utilizing the linearity of the photodiode.

If filters calibrated at the correct wavelength are used, the following routine may be used. The smoke measurement system should be checked weekly using neutral density glass filters of 0,3 nominal optical density. This procedure assumes that the smoke system is the conventional split beam laser described in ISO 5660-2^[30].

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Place the filter in the beam between the duct and the detector. Collect data for a period of 60 s. The measured calibration extinction coefficient, k_{mc} , is obtained from the equation:

$$k_{mc} = \ln(I_0/I)/d$$

where d is the duct diameter.

The correct k value, k_c , is given by the equation:

$$k_c = 2,303D'/d$$

where D' is the optical density of the calibration filter.

A correction factor, f , is calculated from these two k values and is used to correct all subsequent measured k values

$$f = k_c / k_{mc}$$

thus

$$k' = k_m f$$

where

k' is the corrected value;

k_m is the measured value.

Where a calibration factor, F , is used, it is calculated as follows:

$$F = f/d$$

and subsequent k' values are calculated using the equation:

$$k' = \ln(I_0/I) \times F$$

The filter used should be of the doped type because coated filters can give rise to interference effects with laser light and can deteriorate with time. The filter should have a reliable calibration covering the wavelength of the measurement.

4.9 Precautions in relation to water/CO₂ removal

Where carbon monoxide and carbon dioxide are analysed in the gas stream it is important to select the correct drying agent. Some drying agents (e.g. silica gel) lead to tailing of the carbon dioxide peaks due to absorption on the drying agent. Anhydrous calcium sulphate has been found to be the most reliable drying agent and is recommended when carbon dioxide is to be analysed.

4.10 Routine maintenance

It should be noted that all safety precautions regarding potentially toxic or carcinogenic dusts should be carefully observed when cleaning the ductwork and traps. Particular precautions should be taken when dealing with fluorinated or other compounds with high toxic potencies. The study of fluorine-containing compounds needs to be conducted with care as the generation of hydrogen fluoride can result in chemical attack on the glass beads of the refrigeration column as well as on human tissue due to the highly corrosive nature of this compound.

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The equipment will always collect a certain amount of soot. Some will inevitably be deposited on the inside of the ductwork. This should be removed regularly by brushing and vacuum cleaning.

Performance will not normally be affected unless the gas sampling ring holes are blocked. The gas sampling probe and the associated tubing, which connect to the oxygen analyser, require periodic cleaning. One indication of blocking is the need to adjust the waste regulator repeatedly to maintain the proper flow to the oxygen analyser. Cleaning of the interconnecting tubes consists of disassembling the various sections of tubing and blowing them through with compressed air [0,70 MPa (100 psi)].

Never direct high pressure air into the analyser and remember to vent the dirt and soot to a safe place. The pump situated in the gas sampling train should be cleaned or serviced following the manufacturer's instructions.

5 Test specimen preparation and presentation

5.1 General

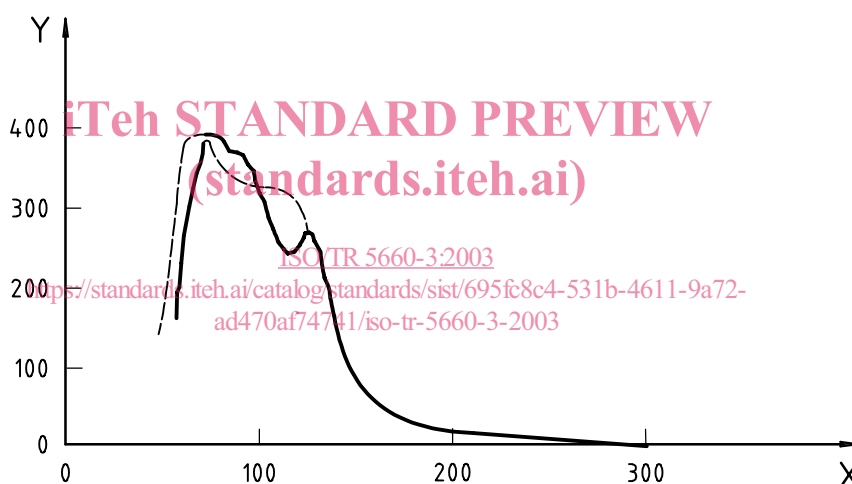
Specimen preparation is described in Clause 8 of ISO 5660-1:2002 and the advice given therein is generally recommended. Products used at thicknesses between 6 mm and 50 mm should be tested at the finished thickness. For products thicker than 50 mm the advice in 8.1.4 and 8.1.5 of ISO 5660-1:2002 recommends that the product is cut down to 50 mm from the unexposed face. Care should be taken when reducing the total thickness to ensure that the resulting product is representative of the original specimen. Products used at thicknesses thinner than 6 mm should be tested at the finished thickness as in end-use or fixed to a typical substrate. Guidance on the selection and use of suitable substrates is given in ISO/TR 14697^[28]. Systems using air gaps should be studied carefully since this can influence the results and special protocols should be determined. Figure 1 shows that the same piece of material can give very different results when tested:

- a) flat without an air gap;
- b) with an air gap that allows the specimen to burn on both sides;
- c) with an air gap that allows pyrolysis products to escape from the irradiation zone unburnt.

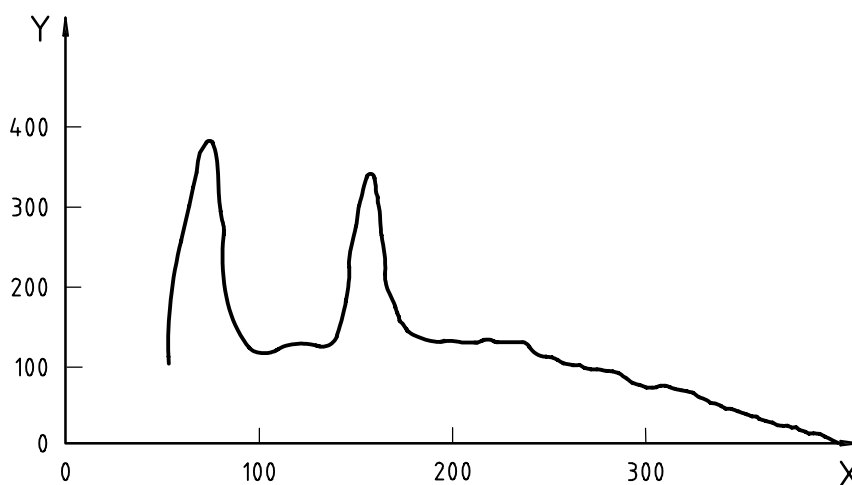
In all cases, it is important that specimen construction enable pyrolysis products that are generated behind surface layers to be vented from composite specimens in a similar way to that which they would be released in the burning end product. Combustion products can either be vented from the top of the specimen holder where the flames are and contribute to the heat release or may be vented from the base of the specimen holder. Pyrolysis products escaping around the base of the specimen holder may also burn but this is not necessarily the case.

It is possible that the upper part of the specimen may have been completely combusted and that during the latter stages of the test, the only combustion may be due to pyrolysis from the inside of the specimen escaping around the specimen edge.

In other circumstances, pyrolysis can take place so rapidly that the combustion concentration limit is exceeded before the pyrolysis fumes ignite. The use of the specimen shield (6.2 of ISO 5660-1:2002) or positioning the igniter before the test may be of assistance. It is important that specifiers and users ensure that the data are relevant and consistent in testing sets of products (see also 8.4 and 11.3).



a) Composite over mineral fibre pad



b) Composite over air gap (two-sided burning)