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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

Reference number
ISO/TR 5660-3:2003(E)

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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.

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is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.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 CO_2. 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].

TECHNICAL REPORT **ISO/TR 5660-3:2003(E)**

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 intumescing, 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.

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 ± 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 $\geqslant$ 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 CO_2 removal agent. If CO_2 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].

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:

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

where d is the duct diameter.

The correct k value, k_{c}, is given by the equation:

$$k_{\mathrm{c}} = 2{,}303D'/d$$

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

$$f = k_{\mathrm{c}} / k_{\mathrm{mc}}$$

thus

$$k' = k_{\mathrm{m}} f$$

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/CO_2 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.

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;

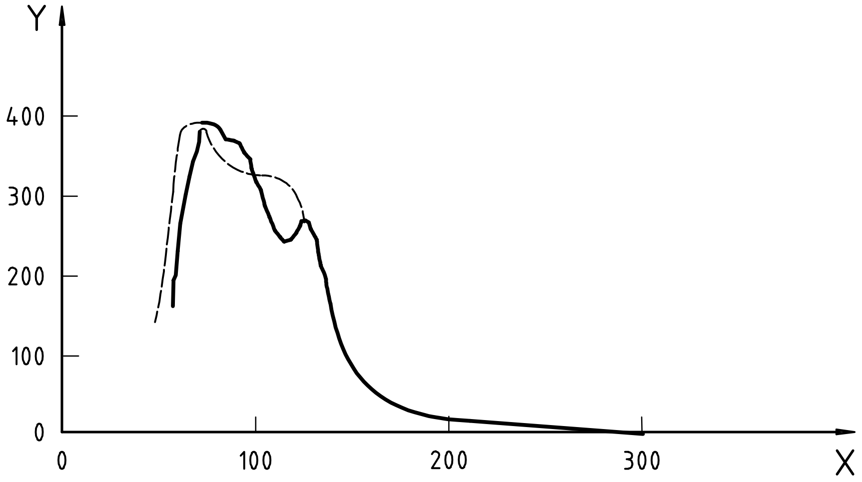
b) with an air gap that allows the specimen to burn on both sides;

c) with an air gap that allows pyrolysis products to escape from the irradiation zone unburnt.

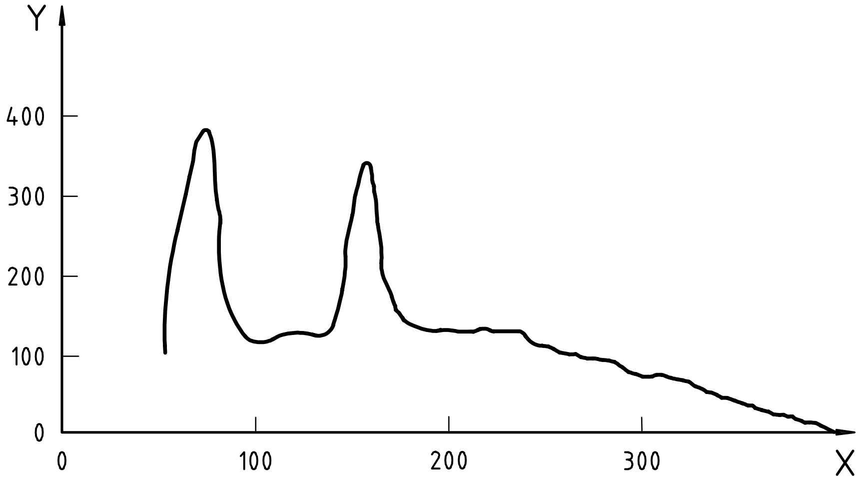
In all cases, it is important that specimen construction enable pyrolysis products that are generated behind surface layers to be vented from composite specimens in a similar way to that which they would be released in the burning end product. Combustion products can either be vented from the top of 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.

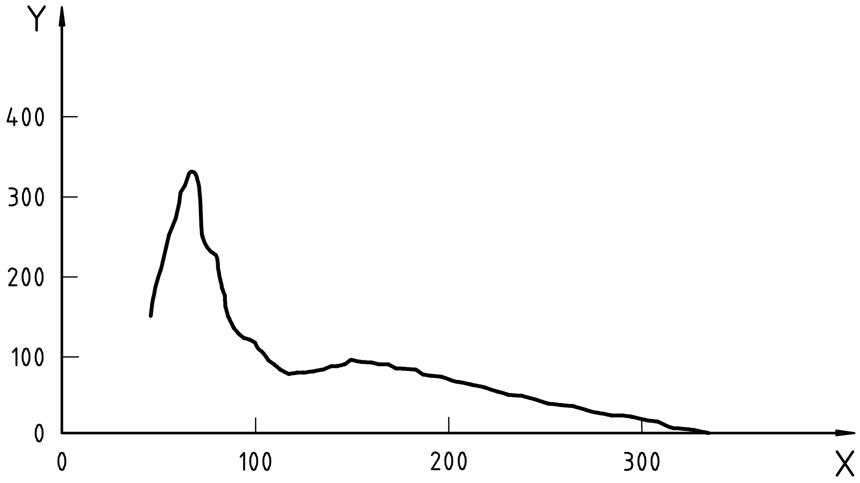
In other circumstances, pyrolysis can take place so rapidly that the combustion concentration limit is exceeded before the pyrolysis fumes ignite. The use of the specimen shield (6.2 of ISO 5660-1:2002) or positioning the igniter before the test may be of assistance. It is important that specifiers and 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)



c) Composite over air gap (one-sided burning)

Key

X time (s)

Y heat release rate (kW/m^2)

Figure 1 — Rate of heat release against time curves for 3 mm thick polyester resin/woven glass roving composites tested at 50 kW/m^2

5.2 Specimen trays and edge retainer frame

The 25 mm metal trays and associated retainer frames used for testing should be constructed from stainless steel as specified in ISO 5660-1. Use of incorrect steel may mean that there is a large mass range for the trays that would then have differing thermal inertia and lead to different results being obtained.

ISO 5660-1 requires that an edge retainer frame be used when testing in the horizontal orientation. For composite products edge coverage is important to be consistent with the end use application and reduce any edge burning effects. However, some industry sectors may find that the use of the edge retainer frame is unnecessary for their applications. Babrauskas[4] has studied this extensively. This may be the case where low heat release rates from low thermal inertia materials are being masked because of the high thermal capacity of comparatively bulky specimen hardware. The CBUF study[5] examined furniture composites without using any edge frames. The CBUF test recommendations were accepted by the ASTM committee E5 in 1995 when it replaced the 1993 protocol in ASTM E1474[31].

If an edge retainer frame is used, the surface area will be less than the 0,01 m^2. The exposed area should be clearly stated in the reports.

6 Selection of heat flux

ISO 5660-1 does not prescribe the level of irradiance to be used in testing. It is the responsibility of the end user or the product committees to ensure that the recommended heat flux levels are selected appropriate to end use. This will require careful consideration of the available research data on the application. In general, two approaches are available:

- testing at a heat flux deemed to be that of the design fire;
- measuring the burning properties at a heat flux at which the material readily ignites.

Most measurements will be required for post ignition test conditions and the user should first decide if that is the case, as most properties measured will be very different depending on whether ignition has or has not occurred. When the intention is to assess post ignition properties of the product, it is important to test at a heat flux which causes ignition to occur within, at most, about 10 min because at lower heat fluxes specimens may show irreproducible ignition behaviour. In circumstances where materials show variable performance because they have been tested close to their critical ignition fluxes, it is suggestible to consider testing at least 5 kW/m^2 above this irradiance to obtain more reliable data concerning the intrinsic performance of the material.

It is suggestible to select levels from 25 kW/m^2, 35 kW/m^2, 50 kW/m^2 and 75 kW/m^2. These levels are not special but are useful levels within the operating range of the cone calorimeter, (10 kW/m^2 to 100 kW/m^2), and they are suggested in order that users produce data at a limited number of heat flux levels rather than at a wide number of arbitrary heat flux levels within the operating range.

Users will have the following objectives for results from the cone calorimeter:

a) to generate fundamental bulk material properties;

b) to generate input data into fire engineering models;

c) to predict performances in larger scale tests;

d) to develop specifications which require the use of the cone calorimeter.

Users with objectives a) are likely to need to map the properties of materials over a range of heat flux levels. For exploratory testing it is suggested that irradiances of 35 kW/m^2 be used. Further information can be found for materials that resist ignition at 35 kW/m^2 by retesting at 50 kW/m^2, and for those which readily ignite at 35 kW/m^2 by retesting at 25 kW/m^2. To obtain bulk material properties it is important to test thick specimens and to analyse data during the period when the material is behaving as a thermally thick specimen. In practice, this means testing specimens that are at least 6 mm thick but preferably 10 mm thick and analysing data obtained over a period of a few minutes starting about 1 min after ignition.

Users with objectives b) will generally be guided by the requirements of the models to be used.

Users with objectives c) will be making their decisions from recent research results, as this is a developing area. Predictions can be based on statistical correlations or the use of mathematical models. In both cases, it is important to match the level of heat received by the specimen in the cone calorimeter to that to be received by the majority of the specimen in the larger scale assessment.

Examples are as follows:

- If a material supplier is interested in assessing the possible application of a material for sheathing on an electric cable, indication of its performance in an IEC 60332-3-10[32] test could be gained from testing it at 50 kW/m^2. Testing at 75 kW/m^2 would be required in order to assess the likelihood of the same material surviving the UL 910[33] test required for US plenum cables.

- Many upholstered furniture composites will ignite and burn in a repeatable manner when tested at a heat flux of 25 kW/m^2 (defined in NFPA 264A[34]) but the more ignition-resistant composites may not, and the higher heat flux of 35 kW/m^2 (specified in ASTM E1474[31]) may be necessary.

Users with objectives d) have similar requirements to those with objectives c). This group should note that the simplest types of specification would require the selection of a “design” irradiance to which materials will be exposed. There will generally be materials that will ignite under the irradiance and those that will not. It need not necessarily be a requirement of the specification that the material showing “good” reaction (i.e. little reaction) to the radiation be tested at a higher irradiation to give good repeatability provided that the specification is written so as to foresee any possible variability with such materials. Generally comparisons between materials for ranking or pass/fail-based test specifications would need to be made on common irradiance tests but it is important to specify the relevant heat flux values since the relative rankings of materials can change at different heat flux values (see Reference [6]).

Table 2 gives some indication of the ranges of irradiances developed in some typical fires or fire test environments. Please refer to the actual reference to find details of the position of the heat flux measurement.

Table 2 — Ranges of irradiance developed in some typical fires or fire test environments

Irradiance kW/m^2	**Ref.**	**Examples of effects**
12 to 22	[7]	Critical ignition flux of many materials, e.g. polyethylene, polyacetal, PMMA, wood, hardboard
10 to 40	[8]	Output from waste paper basket fires
18 to 20	[9]	Match flames
20 to 25	[13]	Heat flux at floor level in flashover fire with ceiling temperature > 600 °C
20 to 40	[10]	Developing fire with 10 % to 15 % O_2 by volume
< 25	[10]	Oxidative pyrolysis fire 5 % to 21 % O_2 by volume
25	[12]	Flux below vertical spreading wall flame
25 to 45	[12]	Flux on wall from vertical wall burning
30 to 40	[9]	Small (up to 250 mm high) gas diffusion flames
40 to 70	[10]	Low ventilation fully developed fire 1 % to 5 % O_2 by volume
40 to 115	[12]	On facade 0,8 m to 3,3 m above window at lintel
60 to 120	[7]	Premixed gas burner
50 to 150	[10]	High ventilation fully developed room fire 5 % to 10 % O_2 by volume
70 to 150	[12]	Average ceiling values for post flashover room fire
90 to 200	[12]	Average wall values for post flashover room fire
105 to 175	[12]	Peak ceiling values for post flashover room fires
115 to 230	[12]	Peak wall values in post flashover room fires
120 to 145	[12]	Peak floor values in post flashover room fires
85 to 105	[7]	Kerosene blow lamp
140	[7]	Premixed blow torches oxyacetylene
200	[12]	Fully developed fire > 10 MW
200	[11]	Jet fire average
300	[11]	Jet fire peak

7 Ignition protocols

For all cone testing, it is important that a correct ignition time be recorded. In certain circumstances, for example furniture testing, the ignition time can be very short, i.e. less than 5 s. If this ignition point is missed, the concentration of pyrolysis products can become too high to allow the spark igniter system used in the cone to actually ignite the volatiles.

One method to ensure that the correct ignition time be recorded is to select a sufficiently low heat flux so as to give a longer ignition time. However, lowering the heat flux can cause other complications such as poor repeatability of test results and specimens extinguishing before complete combustion has occurred.

If the time to ignition cannot be lengthened then there are several alternative methods which can be used to ensure that the correct ignition time is recorded. In addition to ensuring the correct ignition time is recorded, it is critical to ensure that a steady reading for the initial mass of the specimen in the load cell be obtained.

It is also important that the load cell be correctly adjusted to give suitable damping characteristics. This will ensure a fast yet accurate response from the load cell.

For those load cell systems that can stabilize within 1 s to 2 s after positioning the specimen, either of the following ignition protocols will ensure that the correct data are collected at ignition.

The cone heater should be provided with a removable radiation shield to protect the specimen from the irradiance prior to the start of the test. The shield should be made of non-combustible material, with a total thickness not exceeding 12 mm. The shield shall be either of the following:

a) water-cooled and coated with a durable matt black finish having a surface emissivity of $\varepsilon = 0{,}95 \pm 0{,}05$; or

b) not water-cooled, which may be either metal with a reflective top surface or ceramic in order to minimize radiation transfer.

The shield should be equipped with a handle or other suitable means for quick insertion and removal. The cone heater base plate should be equipped with a mechanism for introducing the shield into position.

8 Guidance on the testing of non-standard products

8.1 General

ISO 5660-1 is, in general, applicable to essentially flat homogeneous products, which do not intumesce, swell, bend or collapse appreciably when exposed to radiant heat. This is because the space between the specimen surface and either the cone heater or the igniter is limited, and therefore physical as well as thermal damage can be caused to any expanding specimens. Any change in specimen surface position will take the exposed specimen surface into a different heat flux zone. The change in exposure condition during the test would be maximized when the specimen swells to the point of almost touching the heater coils. ISO 5660-1 also contains a method to test intumescent products.

8.2 Non-planar products

Products that are non planar, i.e. corrugated, or composites such as small electric cables, can physically be housed in the specimen holder. They do, however, show two non-standard characteristics that need to be addressed by the user. The first is that different parts of the specimen surface can experience different heat flux levels at any time. This can be calculated from specimen dimensions, the information given in the thermal maps given in Figure 2, and simple trigonometry. These variations are small for specimens with surface height variations less than 10 mm. The second is that planar specimens have surface areas that are the product of the exposed edge lengths, but the exposure area of non-planar products should be carefully estimated and used when calculating the test results.

If the lack of planarity does not involve some parts of the specimen because of air spaces and irregular thermal contact with the substrate, the main source of problems for these specimens will derive from radiative transfer aspects. The radiative problems are caused by different parts of the specimen receiving different radiative fluxes, which may arise from three main areas:

a) the variation of irradiance with distance from the cone due to the varying heights of the specimen surface;

b) the variation of irradiance with the angle presented by the various specimen surface orientation (configuration factor);

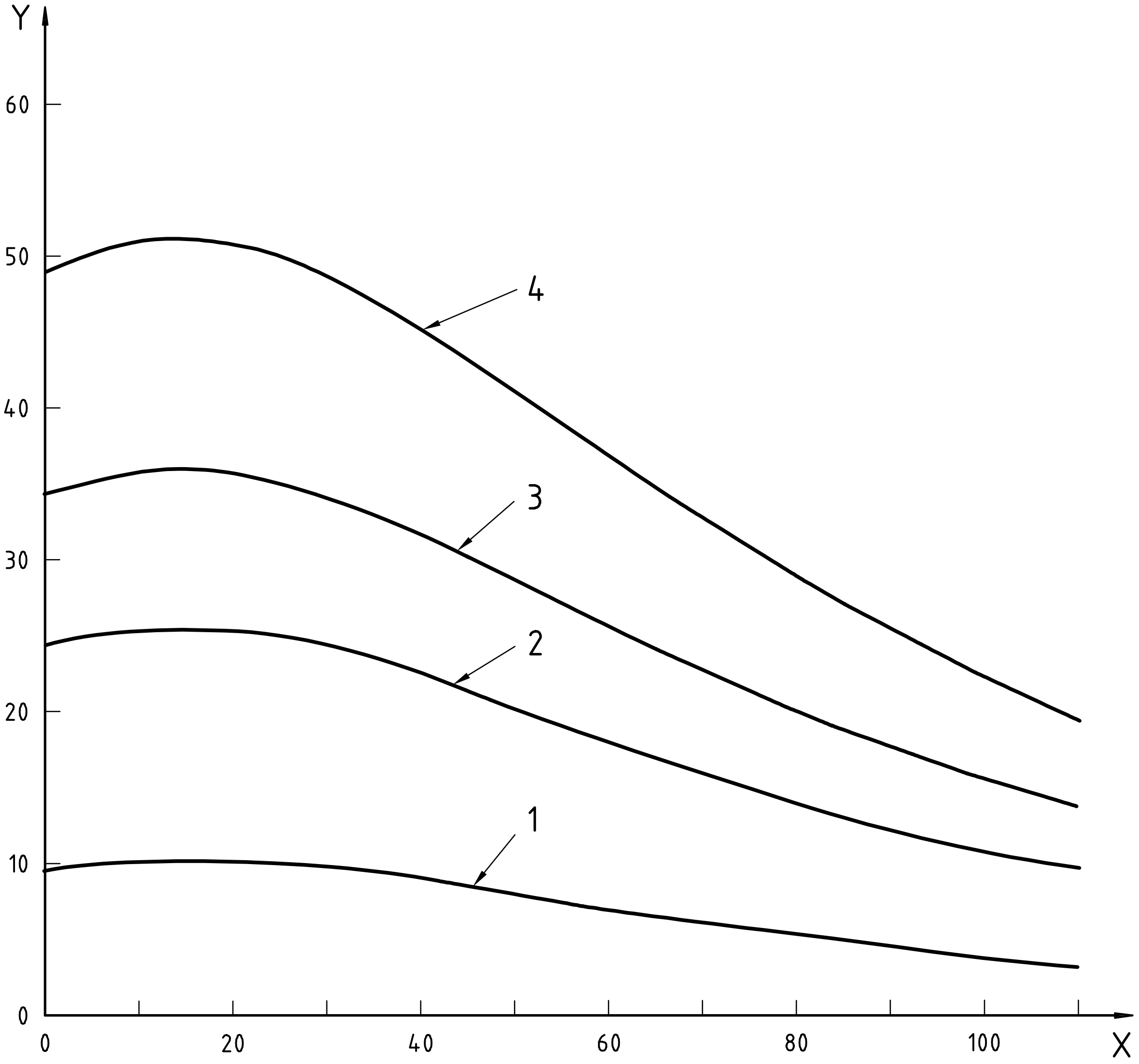
c) the post-ignition feedback of radiation (and in extreme cases, convective heat) to the burning surface as a result of the specimen's own combustion.

The separation of these contributions is generally very difficult and it is important to realize that data generated from such tests are comparative and not absolute.

Some experimental data are available in the literature [14][15], which show how the flux varies with the distance from the base of the cone heater, and also radially out from the centre.

In all cases the data are for a parallel configuration. Attempts to adapt these data for use with non-planar specimens will need to take into account the non-parallel nature of the surfaces. The information presented in 8.3 may assist in this.

The area of non-planar specimens is not the product of the dimensions of the plan of the specimen surface and hence allowances must be made for this in any calculations that are generally normalized to the surface area. Significantly different test data will be produced from flat sheets and profiled products made from the same material. This technique is therefore best used for comparing specimens of similar geometry, e.g. flat specimens with flat specimens, and circular cross-section specimens with others of a similar cross-sectional area.



Key

X spacing (mm)

Y irradiance (kW/m^2)

1 irradiance 10 kW/m^2

2 irradiance 25 kW/m^2

3 irradiance 35 kW/m^2

4 irradiance 50 kW/m^2

Figure 2 — Typical irradiance-spacing curves — offset 0 mm

8.3 Radiation transfer considerations

It is possible to use a simplified configuration factor, F_C, method, approximating the heater to disks, which allows the ready calculation of radiation arriving at a parallel receiver placed at varying distance from the base of the cone (i.e. "spacing") and at points off the centre axis (i.e. "offset"). The output from this simplified treatment[16] can then be compared with the experimental data in the literature [14][15].

For a parallel receiver with a spacing of "x" and an offset of "y" the configuration factor, $F_C(x,y)$ is given by:

$$F_C(x,y)=0{,}5\left[1-\frac{\left(x^2+y^2-80^2\right)}{\sqrt{80^4+2\times 80^2\left(x^2-y^2\right)+\left(x^2+y^2\right)^2}}\right]-0{,}5\left\{1-\frac{\left[(x+65)^2+y^2-40^2\right]}{\sqrt{40^4+2\times 40^2\left[(x+65)^2-y^2\right]+\left[(x+65)^2+y^2\right]^2}}\right\}$$

If the offset is zero, i.e. the configuration factor is relative to the axis of symmetry, then the expression simplifies to:

$$F_C(x,y)=\frac{80^2}{80^2+x^2}-\frac{40^2}{40^2+(x+65)^2}$$

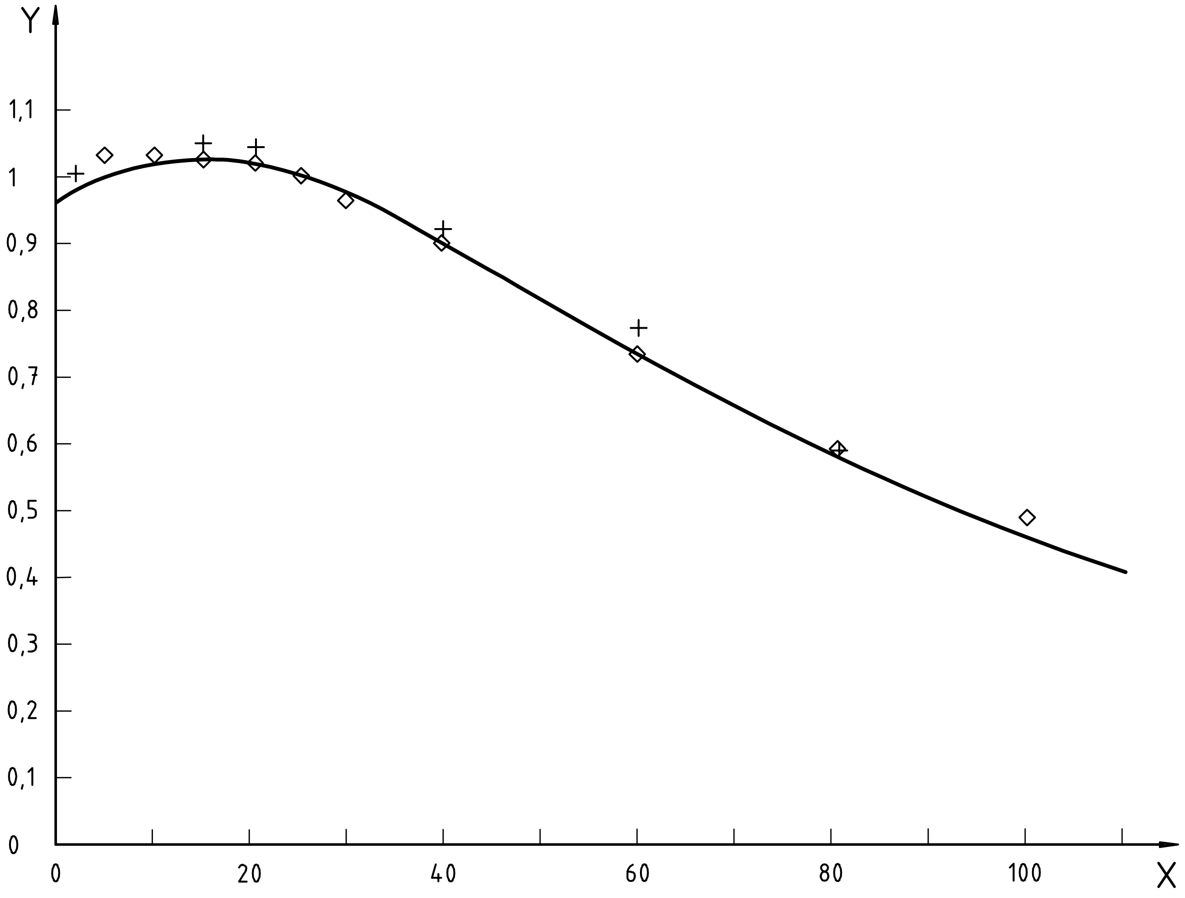
This latter expression is plotted, normalized to F_C(25,0), in Figure 3 and shows good agreement with the experimental data. It may therefore be used to determine the irradiance at any reasonable distance from the cone heater given the standard value, [F_C(25,0)].

The former expression reproduces the form and values of the experimental data fairly well and can also, therefore, be used to estimate irradiances for any spacing and offset given the standard irradiance.

Various plots are given in Figure 4 and show significant variation for the corners, (nominal 70 mm offset) and also for the edges, (nominal 50 mm offset) for small separations. Of particular note is the fact that the corners of the specimen will show significantly enhanced flux levels, relative to the centre, as the spacing becomes small.

The simplifying assumptions mean that the equations given should not be used beyond a 75 mm offset, although there is no limitation on the spacing value provided it is positive.

The expressions may find application in testing, for example, intumescent materials where it may be desirable to use a greater spacing and to allow the material to intumesce unrestrained. If required, from observations of the height changes, it would be possible to indicate the entire thermal history of the surface.

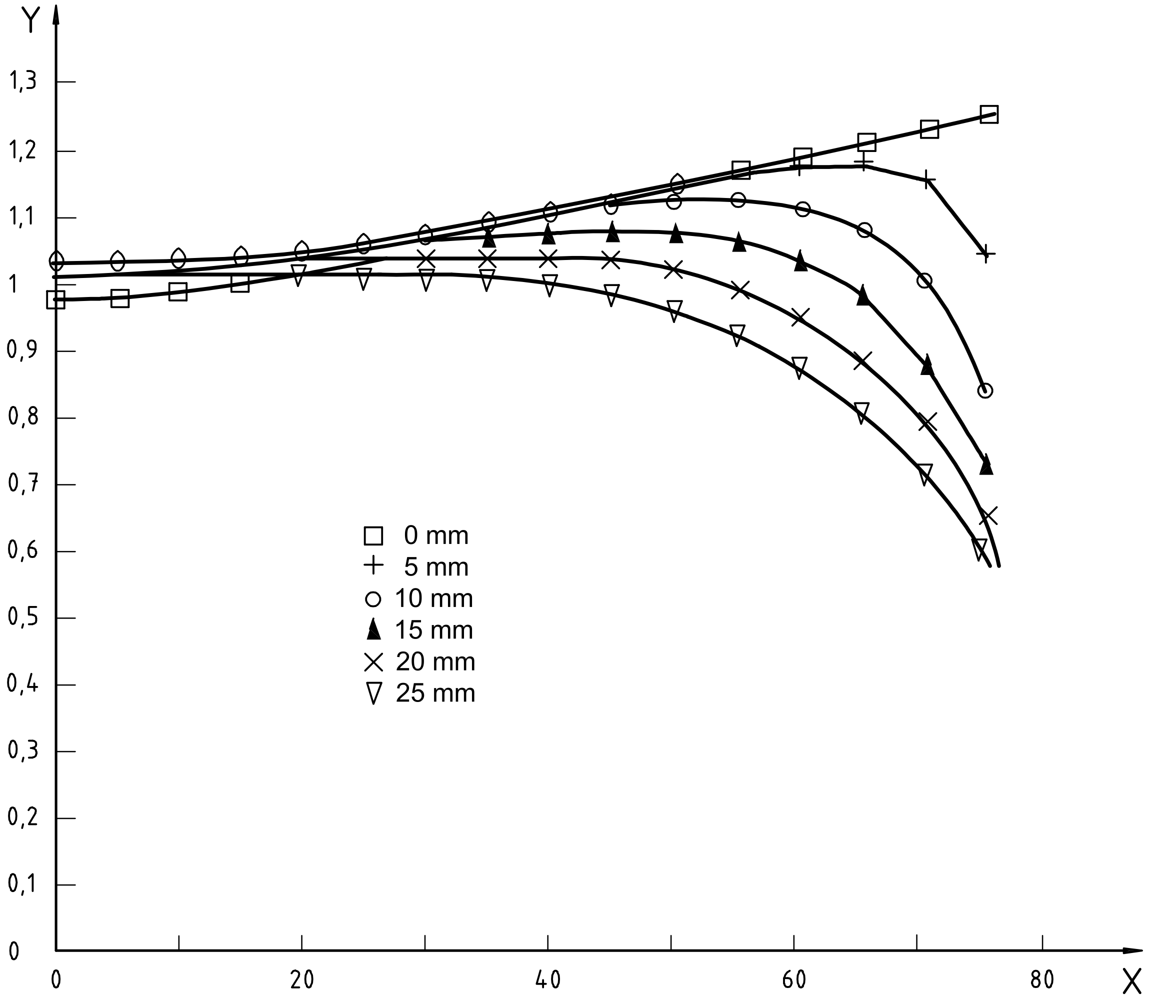


Key

X spacing (mm)

Y F_C normalized to F_C (25, 0)

Figure 3 — F_C versus spacing curve — offset 0 mm

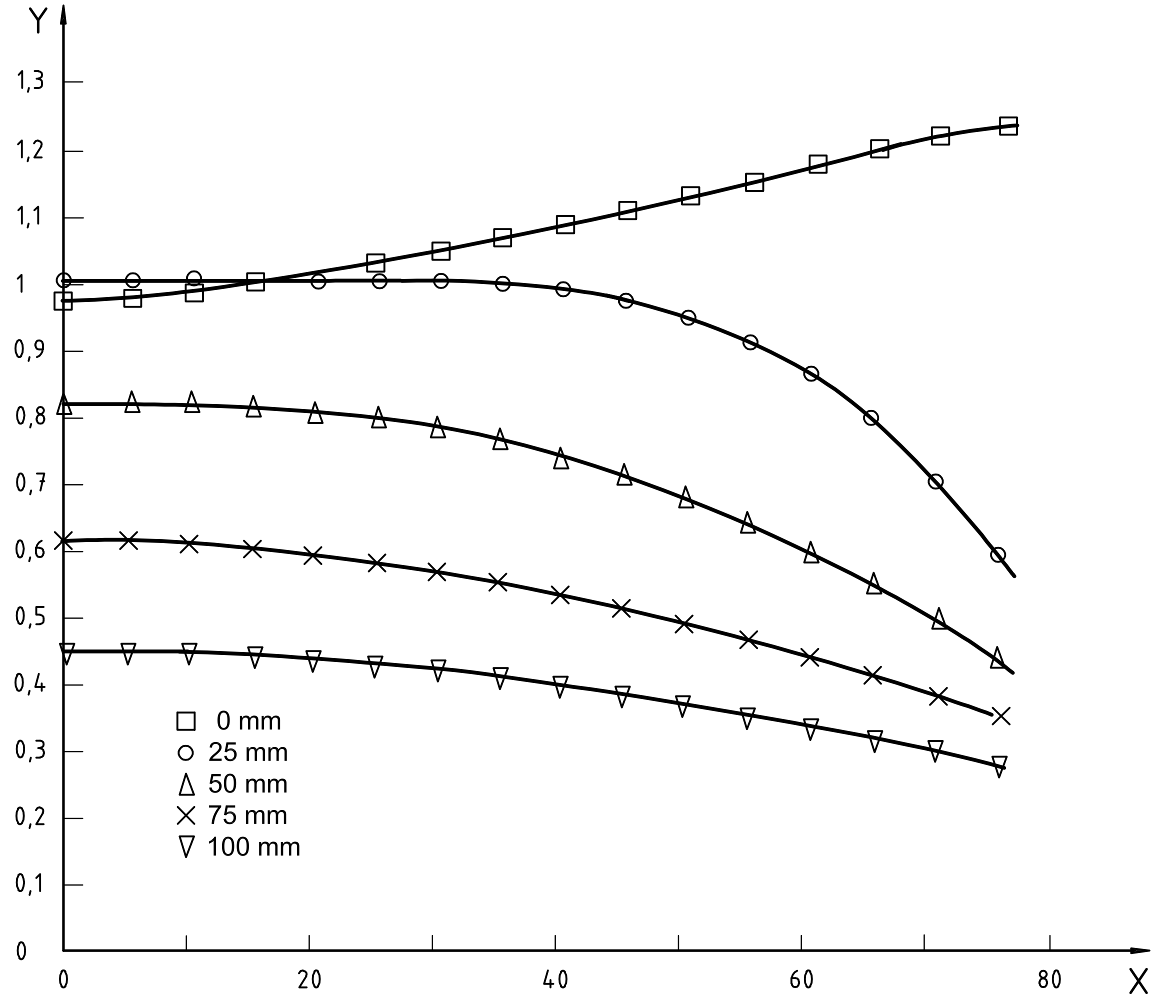


Key

X offset (mm)

Y F_C normalized to F_C (25, 0)

a) Six spacings — F_C normalized to F_C spacing 25 mm, offset 0 mm

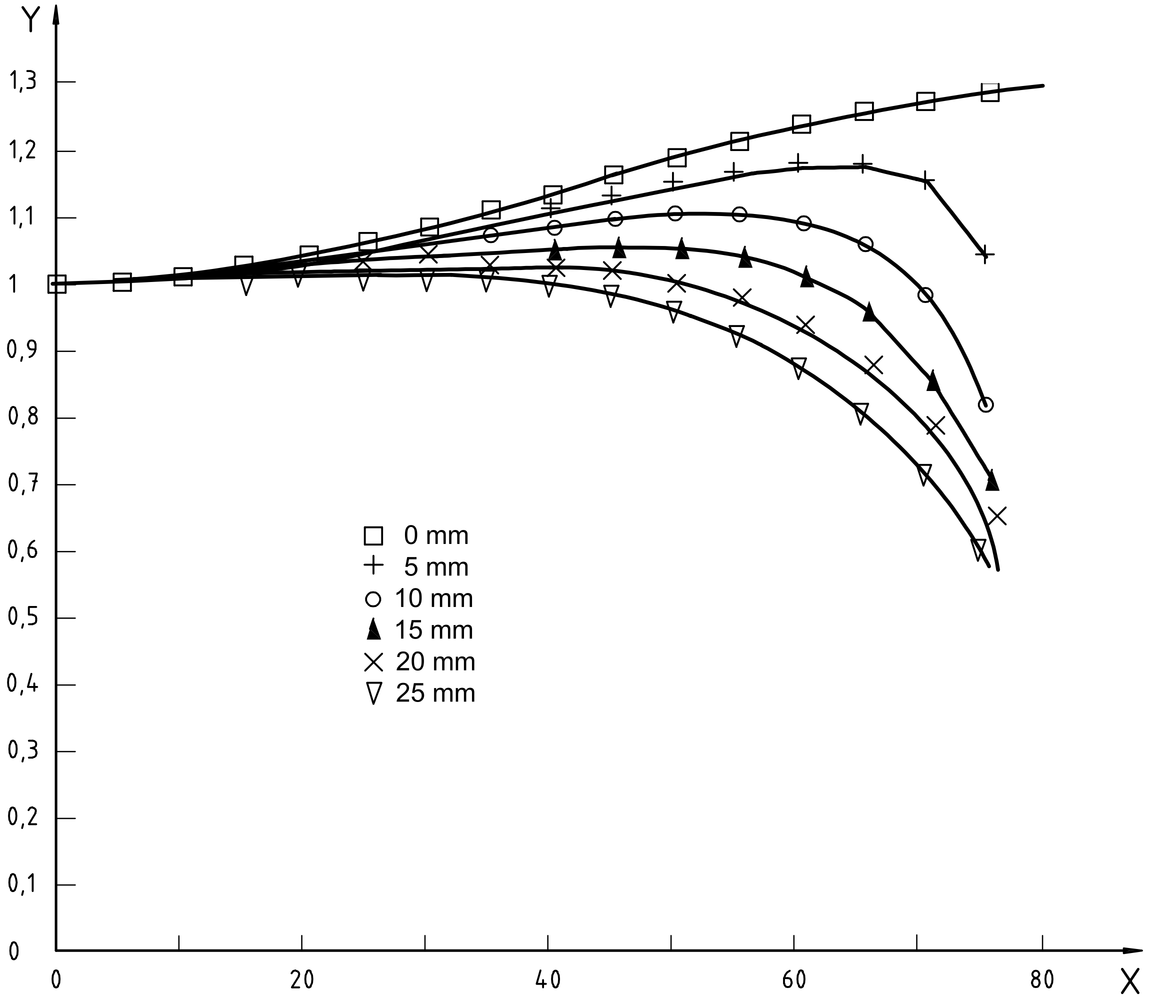


Key

X offset (mm)

Y F_C normalized to F_C (25, 0)

b) Five spacings — F_C normalized to F_C spacing 25 mm, offset 0 mm

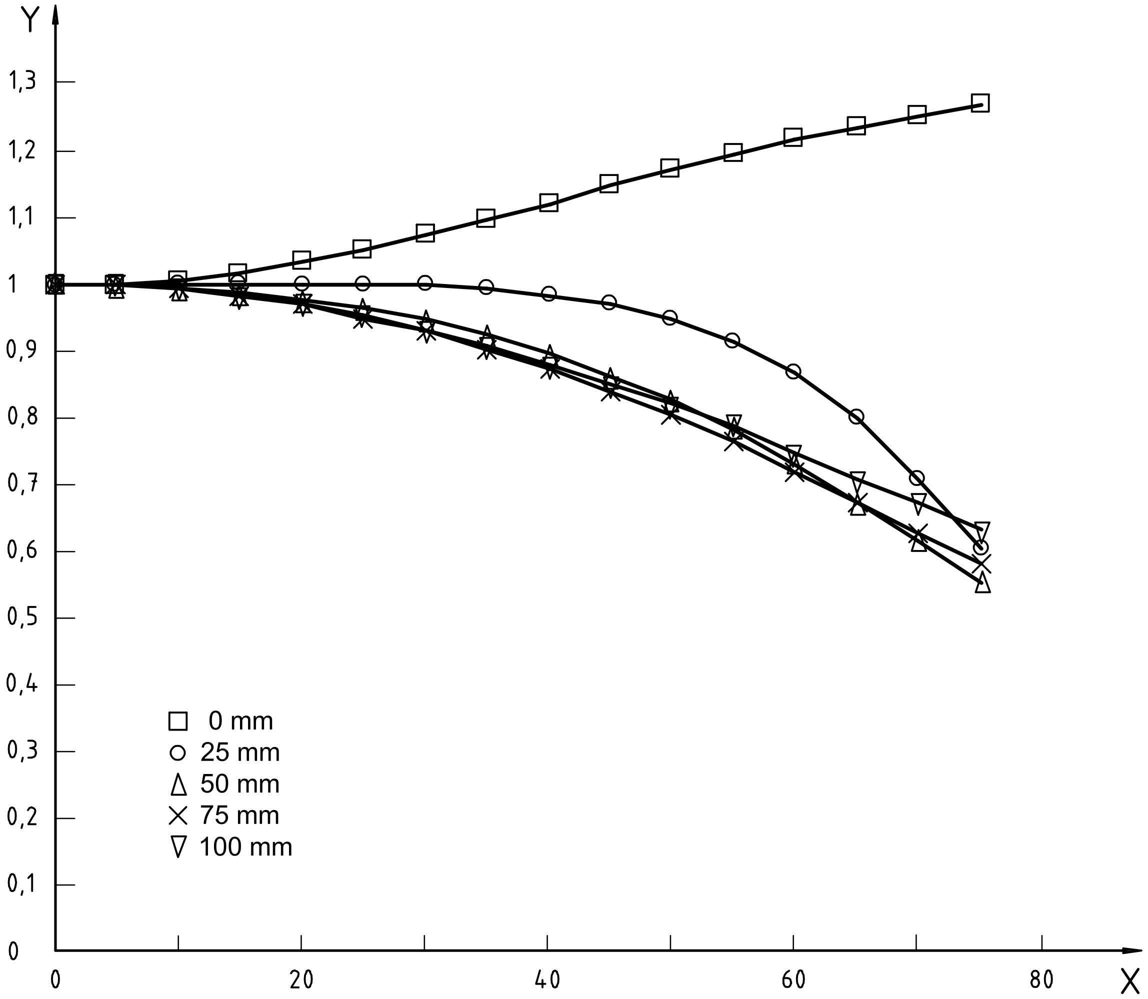


Key

X offset (mm)

Y F_C normalized to F_C (each, 0)

c) Six spacings — F_C normalized to each F_C spacing, offset 0 mm



Key

X offset (mm)

Y F_C normalized to F_C (each, 0)

d) Five spacings — F_C normalized to each F_C spacing, offset 0 mm

Figure 4 — F_C versus offset

8.4 Thermally mobile specimens

8.4.1 Thermoplastic materials

Specimens, which melt to become mobile liquids, require special attention. The aluminium foil tray should be tightly constructed so that it does not leak during the test and other aspects of testing liquids (see Clause 10) may be usefully applied. One variant is to use high profile edges on the aluminium tray wrap that efficiently holds any liquids generated in the test.

8.4.2 Materials that intumesce

Swelling of a specimen during testing can lead to physical contact with the spark igniter or the heater coils, and exposure of the specimen surface to fluxes that differ from the set heat flux at the specimen surface. Such action has been physically restrained by the use of a flat grid that several researchers have criticized as this can delay the times to ignition and influence the performance of surface coatings specially designed to enhance fire performance. These failures had been partially overcome by use of a raised grid as described by Mikkola[17], but it is considered now that they are better overcome by testing at ranges greater than 25 mm (e.g. 60 mm). In the latter case, the swelling specimen would then avoid physical contact with the spark igniter or the heater coils and experience a less severe thermal overexposure after swelling. The calculations described in 8.3 can be used to calculate the thermal regimes the specimen surface would pass through.

ISO/TC 61 is now recommending that a spacing of 60 mm between the specimen surface and the cone calorimeter baseplate be used for testing intumescent specimens.

It should be noted that although the heat flux can be adjusted to be the same as it was at 25 mm, the change in geometry might affect the burning behaviour of the sample.

8.4.3 Specimens that retreat from the heat source

Some specimens collapse on initial exposure to the radiant heat source. In the case of some thermoplastic cellular polymers, this can be a reduction of almost the full specimen thickness, so there has been considerable difference in opinion on the level of reduction in potential heat flux received at the surface after collapse of the specimen. The variation of heat flux with distance is different from those discussed earlier because the specimen is housed in a tray with edge frames and reflective aluminium foil trays which reflect more heat into the collapsed specimen than a free-standing surface would experience. Lukas[18] has made a study of this situation and found that the heat flux at the bottom of a 50 mm aluminium specimen tray is only 1 % lower than the 25 kW/m^2 measured at what would be the specimen surface. When the edge frame was used in similar experiments, the heat flux reduction was 8 %. This work implies that reduction in heat transfer to the specimen is not a significant problem. These empirical results[18] show smaller differences than would be expected from 8.3.

9 Composites and layered products

9.1 General

Laminated products may require some form of edge sealing to ensure that the burning process used in the cone calorimeter reflects that found in practice. This may be done by the use of edge frames, which reduce edge flaming and specimen bowing, or by sealing the edges of laminates or composites using impermeable cements. Care should be taken when using results from products where:

a) the end use orientation differs from the test orientation;

b) one of the layers acts as a fire-resisting barrier under the conditions of test;

c) the end use application has different boundary conditions to that used in the test;

d) the pyrolysis products are vented in a non-representative way.

9.2 Non-homogeneous products

Products that are non-homogeneous set other limitations. Problems of specimen mounting for laminated specimens with components of different levels of combustibility can lead to significant edge effects. Specimen mounting is particularly difficult when the specimen is thermally thin and is used in a composite with air. In some cases, it is impossible to assess such composites. In other cases, for example where the specimen is used in hollow square sections but at thicknesses greater than the thermal thickness of the material, valuable data can be produced from testing plaques of the material.

9.3 Specimens with short test duration

Some specimens ignite and complete burning quickly. Problems associated with load cell stability and other aspects of initiating tests can be overcome by the use of the heat shields described in Clause 7. It is also necessary to decrease the scan time of the measurements in order to collect as much test data as possible. Scan times of 2 s should be considered.

10 Liquids

10.1 General

Liquids can be tested in the cone calorimeter but they introduce a number of problems that are not apparent with solid specimens, for example effects of convection currents in the liquid, surface tension effects and sometimes vigorous boiling of the liquid.

10.2 Testing without the radiant heater

Flammable solvent materials and materials with low flash points may be carefully tested in the cone by this mode. A satisfactory protocol for testing in this mode is as follows:

- the cone calorimeter should be in the normal testing condition with the heater power off and the heater cold;
- pour 20 g to 30 g of the flammable liquid for test into a quartz dish, which should be weighed externally prior to inserting into the specimen holder of the cone;
- place the dish into the normal cone specimen holder, lined with the fibre blanket with a minimum thickness of 13 mm;
- commence data acquisition;
- ignite the flammable liquid with a pilot flame, rather than with a spark igniter;
- allow the data acquisition to continue for at least 1 min after all the liquid has been burnt.

If, for example, propan-2-ol were used, the total heat released would be 30,2 kJ per gram of propane-2-ol. This can then be compared with the result from the run to check whether the orifice flow constant, C, was correct or not. Other alcohols may be used for calibration purposes. The heat release rate from ethanol or propan-2-ol in the normal specimen tray gives a heat release rate of between 3,5 kW and 4 kW.

The liquids are generally burnt in a square or circular container. These liquids burn reasonably constantly after an initial stabilizing period, although the rate of heat release versus time curve is relatively noisy due to the flames pulsing and flickering at the liquid surface. The manner in which these liquids burn varies considerably; for example, petroleum ether (60 °C to 80 °C) burns smoothly at the upper rim of the container, after the initial ignition. Other liquids, such as *n*-hexane, burn with relatively large flames. In both these cases, the smoke production has been shown to be affected by the position of the cone heater and can increase up to 35 %, increasing in the following order:

a) 65 mm above the specimen in the horizontal position;

b) in the normal testing position;

c) in the vertical position;

d) with the cone heater removed altogether, although no heat flux is imposed.

However, liquids burning with smaller flames show no such variation.

Since some liquids are immiscible with water, they can be burnt floating on water which ensures that they burn at steady state conditions without showing a rapid increase at the end of their burning, i.e. the conditions ensure that the material burns as a thermally thick specimen.

A liquid depth of about 6 mm to 12 mm in a container 15 mm deep ensures free access of air to the liquid burning surface whereas a deep tray might not behave in an identical way due to less air being available. However, it may be necessary to have a reasonable difference between container depth and liquid depth because of surface tension effects and also to prevent vigorous boiling which would cause the liquid to spill over the edge of the container.

In all cases, it is essential to allow the specimen holder to return to approximately room temperature before using it for a subsequent test to avoid any preheating of the specimen prior to testing.

10.3 Testing with the radiant heater

WARNING — Many liquids have low flash points and it is very important to ensure that volatile liquids are NOT tested under radiant heat. The volatility of liquids increases with increasing temperature and care is necessary to avoid hazardous test conditions. The use of a shield between the radiant heater (if used) and the test specimen is recommended.

This applies to dielectric materials such as transformer liquids, including both silicones and mineral oils. It also applies to hydraulic liquids. A study was carried out by IEC/TC 10/WG 8 on materials with a range of ignition points, including a mineral oil and a silicone fluid with a viscosity of 50 cSt. The conclusion of this study, which involved other heat release equipment, was that there was no necessity for the development of a new method intended specially for electrical insulating fluids, but that ISO 5660-1 was suitable for this work.

IEC/TC 10/WG 8 used the following test conditions for these liquids:

- heat flux: 25 kW/m^2;
- maximum test duration: 20 min;
- test vessel: quartz crucible;
- inner diameter: 50 mm;
- height: 13 mm;
- thickness of wall: 2,2 mm;
- volume of the specimen: 15 cm^3.

The test vessel was placed on a cylindrical or square, asbestos-free, mineral fibre pad.

11 The theory of oxygen consumption calorimetry

11.1 General

Thornton[19], in 1917, showed that, in the case of complete combustion, a large number of organic fuels release an approximately constant amount of heat for every unit of oxygen, which is consumed. In 1980 Hugget[20] obtained an average value of 13,1 kJ of heat released for each gram of oxygen consumed and he also showed that the value was not significantly affected by incomplete combustion. He concluded that this value may therefore be used for practical applications and is accurate, with only a few exceptions, to within ± 5 %.

Table 3 lists some of the values reported by Hugget[20]. With the exception of three materials, ethene, ethyne and polyoxymethylene, all the calculated heats of combustion per gram of oxygen consumed lie between 12,32 kJ and 13,61 kJ.

The values were calculated assuming complete combustion. However, as stated above, Hugget does discuss the effects of possible incomplete combustion and calculates values of ΔH_c for several such cases. For example, in the case of cellulose burning to give a 9:1 ratio of CO_2 to CO;

$$(C_6H_{10}O_5)_n + 5{,}7\ O_2 \rightarrow 5{,}4\ CO_2 + 0{,}6\ CO + 5\ H_2O + (C_6H_{10}O_5)_{n-1} \qquad H_c = 13{,}37\ \text{kJ/g of } O_2$$

or burning to give an appreciable amount of carbonaceous char;

$$(C_6H_{10}O_5)_n + 3\ O_2 \rightarrow 3\ CO_2 + 3\ C + 5\ H_2O + (C_6H_{10}O_5)_{n-1} \qquad H_c = 13{,}91\ \text{kJ/g of } O_2$$

compared with:

$$(C_6H_{10}O_5)_n + 6\ O_2 \rightarrow 6\ CO_2 + 5\ H_2O + (C_6H_{10}O_5)_{n-1} \qquad H_c = 13{,}59\ \text{kJ/g of } O_2$$

He discusses several other examples and concludes that the assumption of a constant heat release per unit of oxygen consumed will be sufficiently accurate for most applications.

If the correct value of H_c is known for a particular material, then this should be used instead of the approximate value.

11.2 Silicones

It might be expected that silicone oils and rubbers would deviate from the 13,1 kJ/g of O_2 “rule” because, unlike conventional organic materials, the chemistry of combustion involves the conversion of the SiO backbone into amorphous silicon dioxide.

R. Buch[21] reported that the gross heat of combustion of poly(dimethylsiloxane) is 26,8 kJ/g. Therefore the net heat of combustion can be calculated using the following equation:

$$[Si(CH_3)_2O]\ (s) + 4\ O_2 \rightarrow SiO_2\ (\text{solid amorphous}) + 2\ CO_2\ (g) + 3\ H_2O\ (g)$$

NOTE Water is considered to be a gaseous product in the calculations.

The heat of gasification of liquid water is 43,2 kJ/mole. Therefore,

$$H = -(26{,}8 \times 74{,}16)\ \text{kJ} + (3 \times 43{,}3)\ \text{kJ} = -1\ 858\ \text{kJ}$$

and 128 g of O_2 are consumed, thus $H_c = -14{,}5$ kJ/g of O_2, which is significantly different from the general rule.

Table 3 — Heats of combustion for a variety of fuels

Fuel	Heat of combustion [a] kJ/g	Heat of combustion [a] kJ/g of O_2
Methane	50,0	12,5
Ethane	47,5	12,7
Butane	45,7	12,8
Octane	44,4	12,7
Ethene	47,1	13,8
Ethyne	48,2	15,7
Benzene	40,1	13,1
Butan-1-ol	33,1	12,8
Butanal	31,9	13,1
Butanoic acid	22,8	12,5
1-Aminobutane	38,0	12,8
Butane-1-thiol	32,8	12,3
Chloroethane	19,0	12,8
Bromoethane	11,9	12,5
Polyethylene	43,3	12,6
Polypropylene	43,3	12,7
Polyisobutylene	43,7	12,8
Polybutadiene	42,7	13,1
Polystyrene	39,8	13,0
Poly vinyl chloride	16	12
PMMA	24,9	12,9
Polyacrylonitrile	30,8	13,6
Polyoxymethylene	15,4	14,5
Polyethylene	22,0	13,2
Polycarbonate	29,7	13,1
Cellulose triacetate	17,6	13,2
Nylon 66	29,5	12,6
Cellulose	16	13
Cotton	15,5	13,6
Paper (newsprint)	18,4	13,4
Wood (maple)	17,7	12,5
Coal (lignite)	24,8	13,1
Coal (bituminous)	35,2	13,5

[a] Reactants and products at 25 °C, all products gaseous.

11.3 Effect of additives and fillers

It is important to realize that the heat release measured by oxygen consumption calorimetry is the heat released during burning. It does not define whether this heat is released from the system, whether it is used in some other chemical or physical change, or if released, what form the release of the energy takes. The data from the cone test does not inform the researcher where the heat may be in the system at any stage in the combustion process.

Heat release is normally quoted normalized to surface area of the sample. It is also commonly quoted per gram of sample lost. This is called the effective heat of combustion. Because some fillers can absorb heat and can also generate mass loss by dehydration as well as by combustion some concern has been shown that the oxygen depletion work erroneously reports the heat release measurements. A mathematical analysis of the thermodynamics associated with the use of high (e.g. 60 %) loadings of highly endothermic and highly hydrated fillers shows that maximum error would be 7 % overestimation of the heat release (i.e. at these 60 % loadings, the system is actually releasing 7 % less heat than the cone calorimeter indicates). The mathematical analysis is described fully in Annex A. Thus the flame retardant is probably suppressing heat release even better than perceived.

12 Start and end of test

12.1 Start of test

The user should decide if it is essential to use pre-ignition data in any test or average results that are to be used in the assessment. ISO 5660-1 begins measurement of heat-associated properties from the point of ignition because most materials, when present in sufficient bulk, will generate significantly more heat and smoke after ignition than they would before. There are some examples where this is not true and some of these involve systems where the bulk of the material is pyrolyzed prior to ignition. Considerable pre-ignition pyrolysis may however lead to significant generation of smoke and toxic gases that would not be accounted for if the only data used are collected after ignition occurs. In practice all commercially available cone calorimeters collect data from the point of exposure and allow the operator to define the test start point and many actually tell the operator the recorded ignition time and ask if that is to be appointed as the start of the test. Thus no software or hardware changes are needed for pre-ignition data to be included in test reporting and test averaging.

12.2 End of test

ISO 5660-1 states that the test ceases if the sample has not generated any data or if the specimen did not ignite after the spark igniter had been sparking in position for a period of 30 min or after the mole fraction of oxygen remains within ± 0,005 of that of the pre-test value for 10 min.

ISO 5660-1 specifies that if ignition occurs and the spark arm igniter is removed but the specimen then extinguishes, the spark arm igniter should be re-inserted and remain throughout the test. This can be somewhat problematic with PVC, which burns for long periods and also generates electrically conductive cobweb-like structures on the spark arm. These can become substantially short to the edge frame and the loss of mass loss data can result. In these cases, the spark arm igniter should be cycled in and out of the burn zone to disturb any such deposits (e.g. once every 120 s).

End of test situations are defined as one of the following, whichever occurs first:

a) 32 min after the time of sustained flaming;

b) 30 min have elapsed and the specimen has not ignited;

c) after the mole fraction of oxygen remains within ± 0,005 of that of the pre-test value for 10 min.

Observe and record physical changes to the sample such as melting, swelling and cracking.

The above timings are adequate for standard testing but researchers interested in properties of materials in smouldering phases of fires may require that these be extended until after the required parameter peaks are seen. For example, those interested in carbon monoxide measurements may wish to continue the test for 5 min longer once peak values have been measured in the duct.

For very accurate work on gas species the variation in atmospheric pressure should be used in assessing the gas concentrations.

ISO 5660-1 requires that if the flame extinguishes after sustained flaming occurs and the spark igniter is removed, the spark arm igniter should be reinserted and the spark turned on within 5 s. The spark arm igniter should then not be removed until the entire test has been completed.

13 Recommendations for presentation of data

13.1 Current situation

ISO 5660-1 gives the format for presenting data derived from heat and mass related aspects of the calorimeter. These data (all with time) include the heat release rate, in kW/m^2, as well as the average heat release rate for specified intervals timed from ignition.

Many test houses supply, in addition to the above data, information with respect to smoke emission and toxic fume emission (commonly CO, CO_2 and HCl, but others are also possible). Many of these data are given in the form of rates of production but it should be noted that these rates are calculated on a “per unit rate of mass loss of specimen” basis such as toxic fumes on a kg/kg (i.e. $kg{\cdot}s^{-1}/kg{\cdot}s^{-1}$) basis.

In ISO 5660-2[30], the smoke production rate expressed in m^2/s (smoke extinction area per second).

In addition to the time to ignition, the following five parameters are commonly reported as a function of time:

— specimen mass loss rate	(g/s);
— heat release rate	(kW/m^2);
— smoke production rate	(m^2/s);
— carbon monoxide yield	(kg/kg);
— carbon dioxide yield	(kg/kg).

The first of these, mass loss rate, is calculated using the set of five-point difference equations, i.e. the mass loss of the specimen as a function of time.

The second parameter, the heat release rate, is normalized to the surface area of the specimen and since all standard test specimens have the same surface area, heat release rate values from different materials can be directly compared.

The last two parameters are customarily normalized to the rate of mass loss, i.e. CO or CO_2 produced per unit time is divided by the mass of specimen lost in that unit of time. Since different materials lose mass at different rates, direct comparison of these parameters can be misleading.

The dimensionless units for CO and CO_2 arise because what is being reported is the mass of gas produced per unit mass of specimen volatilized. The quantity is therefore a dimensionless ratio.

This approach of normalizing several of the measured parameters to mass loss rate has some important consequences. Whenever the mass loss rate falls to low levels or tends towards zero (e.g. the later stages of testing a flame retarded product, or during pyrolysis prior to ignition) then these normalized parameters will become large or become indeterminate. An additional effect is to incorporate into the parameter of interest an amplification of the noise in the mass loss data.

It also means that it is impossible to study the pre-ignition pyrolysis behaviour of materials, because before ignition the mass loss rate will be close to zero, or in some cases it may even be positive when the increase in mass due to oxidation is greater than the loss of mass due to the evolution of volatiles.

The cone calorimeter does record data between the start of the test and the ignition time, but these data are not normally analysed.

Users wishing to convert the mass loss rate normalized data to “as measured” data or to surface area normalized data face considerable problems in that the averaging procedures used by the various software programs cause the 1:1 correspondence between the “as measured” data and the rate of mass loss from the specimen to be lost. This means that for accuracy and convenience it is necessary to return to the raw data and to recommence the entire calculation procedure.

It is proposed to rationalize the basis on which data are presented to overcome this problem.

13.2 Additional useful data

It is proposed that an additional data set and graphs in which all the parameters are given on an “as measured” basis would be valuable. These would be calculated directly from the raw cone data.

With this as the basis, it is possible to report data from $t = 0$ which, because of the small rates of mass loss, is not practicable on the rate of mass loss basis. The use of the $t = 0$ origin is considered to be particularly important for smoke and toxic fume aspects.

It is recommended that two basic data sets be used as follows:

a) Set 1:

    1) graphs of the cumulative value as a function of time of the following parameters from $t = 0$ to t(end);

    2) heat release, expressed in kilojoules (kJ);

    3) smoke production, expressed in square metres (m^2);

    4) CO and CO_2 production, expressed in grams (g);

    5) specimen mass, expressed in grams (g);

a) Set 2:

    1) graphs of rate data as a function of time for the following parameters from $t = 0$ to t(end);

    2) heat release rate, expressed in kilowatts (kW) (kJ/s);

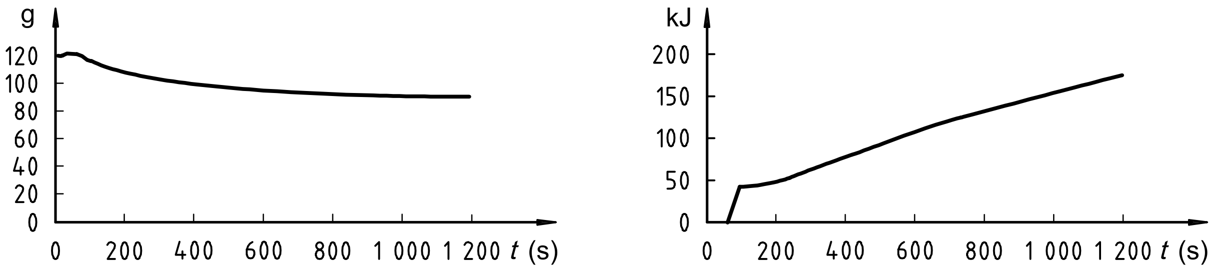
    3) rate of smoke production, expressed in square metres per second (m^2/s);

    4) rate of CO and CO_2 production, expressed in grams per second (g/s);

    5) mass loss rate, expressed in grams per second (g/s).

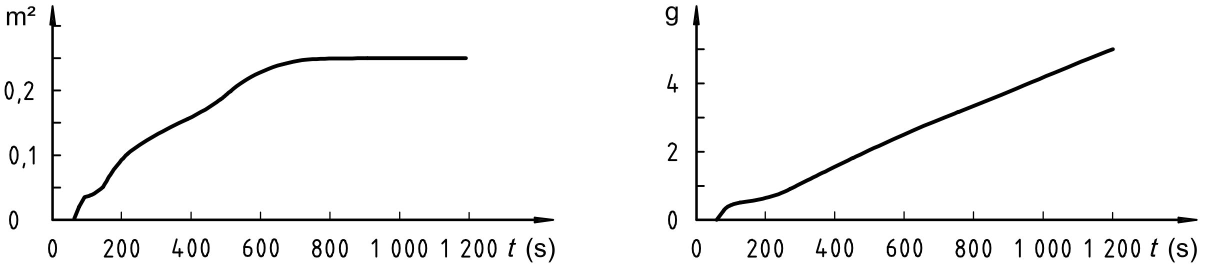
Such a scheme would mean that all the basic data measured by the cone calorimeter would be reported and it would allow users to calculate derived data sets, e.g. on a rate of mass loss basis, or a surface area basis, easily if they so desired.

An example of a typical graphical report is shown in Figure 5.



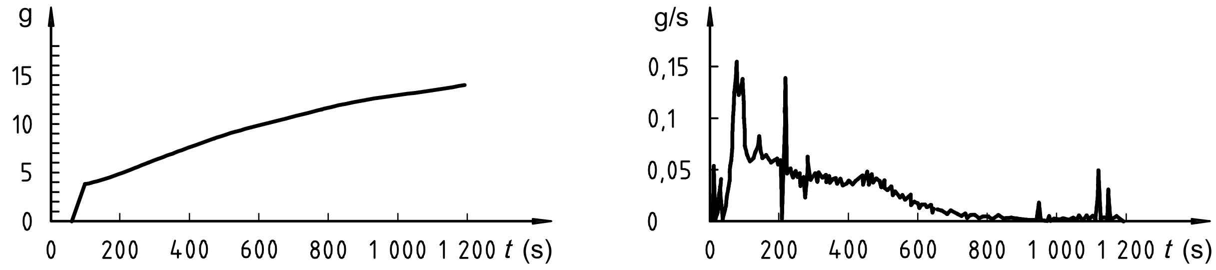
a) Specimen mass

b) Total heat release



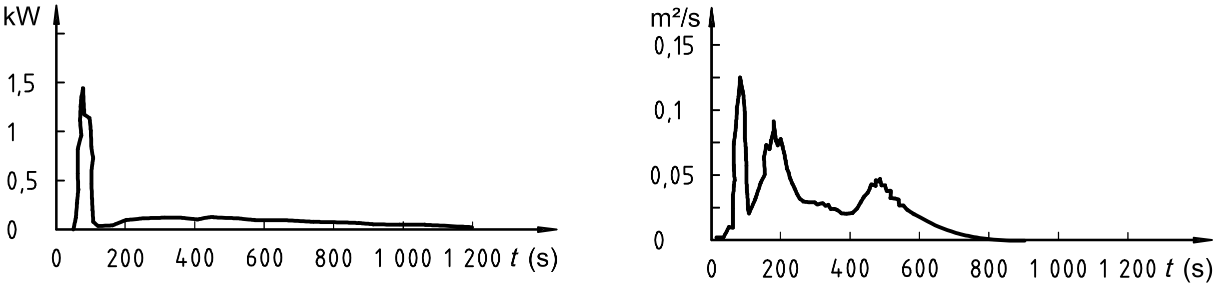
c) Total smoke production

d) Total carbon monoxide production



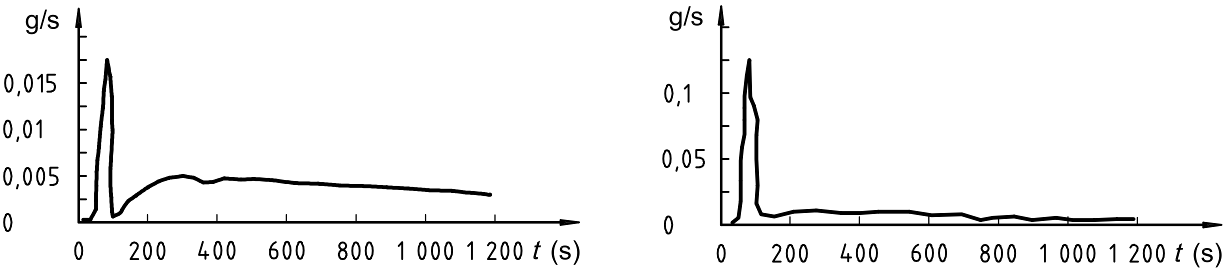
e) Total carbon dioxide production

f) Mass loss rate



g) Heat release rate

h) Smoke production rate



i) Rate of carbon monoxide production

j) Rate of carbon dioxide production

Figure 5 — Example of a typical graphical report

13.3 Recommendations

13.3.1 Provision should be made in the software for mass, heat release, smoke, carbon monoxide and carbon dioxide data to be recorded as a function of time on an “as measured” basis, i.e. not normalized with respect to either mass loss rate or specimen surface area.

13.3.2 Data should be collected, following extinction of the specimen, for a sufficient period to ensure that carbon monoxide and smoke production and any other relevant information during smouldering is not lost. Test durations of 30 min may be required.

13.3.3 Two sets of graphical data should be presented, as detailed in 13.2 above, the first set being cumulative values as a function of time and the second set being rates of production as a function of time.

13.3.4 If required an effective heat of combustion plot should be from t(ignition) to t(end). All graphs mentioned in 13.2 should be from $t = 0$ to t(end).

13.3.5 Numerical data for all the graphs should be readily accessible. Also numerical data should be supplied which states all of the following:

- the value of all cumulative parameters at t(end);
- the maximum rates of production;
- the times at which these maxima occurred.

13.3.6 The effective surface area of the specimen should be stated and software should be available to allow users to normalize the data with respect to the effective surface area and to the rate of mass loss if required.

13.3.7 Software should be available to allow average values to be calculated between selected times. For example, between $t = 0$ and t(end), $t = 0$ and t(ignition), t(ignition) and t(end), t(ignition) and t(ignition + 2 min), t(ignition + 1 min) and t(ignition + 3 min), etc.

Annex A
(normative)

Effect of additives and fillers

Heat release measured by oxygen consumption calorimetry is the heat released in burning. It does not define whether this heat is released from the system, whether it is used in some other chemical or physical change, or, if released, what form the release of the energy takes.

Because some fillers can absorb heat and can also generate mass loss by dehydration as well as by combustion, some concern has been shown that the oxygen depletion work erroneously reports the heat release measurements. This mathematical analysis of the thermodynamics associated with the use of high loadings (e.g. 60 % by mass) of highly endothermic and highly hydrated fillers shows that maximum errors would be a 7 % overestimation of the heat release (i.e. at these 60 % loadings the system is actually releasing 7 % less heat than the cone calorimeter indicates).

Heat release data can be expressed in three mass-related ways:

a) per gram of specimen;

b) per gram of organic material lost;

c) per gram of specimen lost.

The effective heat of combustion as used in cone calorimetry is expressed as per gram of specimen lost.

Discrimination between the three bases will be illustrated by an example where 3 similar compounds of PMMA are studied. Each compound is filled with 60 % loadings. The different fillers used have the following listed properties:

- Filler 1: inert;
- Filler 2: will undergo an endothermic reaction of 1,17 kJ/g (without mass loss);
- Filler 3: will undergo an endothermic reaction of 1,17 kJ/g and loses 34,6 % mass as water.

For the purpose of this illustrative calculation, as given below, it is assumed that Fillers 2 and 3 had no direct effect upon the combustion process other than via endothermic activity and the dilution of combustion products.

a) PMMA plus Filler 1:

Any changes in the temperature of the filler brought about from the heat generated by the combustion process rather than from the external heat source is not reflected in the heat release data from the cone as it is assumed in the calculations that the products are in their standard states (e.g. water is gaseous). This simple example illustrates an important principle, which is that any attempt to partition the heat output as measured by the cone is entirely a matter for post-processing of data.

The effect of including, 60 % by mass of Filler 1 in PMMA, giving a 60:40 mass ratio, on the output from the cone is given in Table A.1

The cone supplies effective heat of combustion data on a dynamic basis. The average value of this parameter is the same as that derived by dividing the total heat output by the total mass loss. The effective heat of combustion from the cone for both of the specimens shown in Table A.1 should be 24,89 kJ/g. In practice the value will not achieve this theoretical level constantly throughout the test due to the dynamic nature of the combustion conditions, although it may be approached at certain points.

Table A.1 — Heat release data for PMMA with insert Filler 1

Filler	Total heat release per gram of specimen kJ/g	Total heat release per gram of organic material lost kJ/g	Total heat release per gram of specimen lost kJ/g
No filler	24,89	24,89	24,89
60 % filler data 1 uncorrected	9,96	24,89	24,89
60 % filler data 1 corrected[a]	9,96	24,89	24,89

[a] The term “corrected” refers to corrections due to phase changes.

b) PMMA plus Filler 2:

Consider the situation where the filler actively absorbs or emits heat, in an irreversible manner during combustion. It is necessary that the phase change is irreversible or the filler would be considered to be inert under standard state conditions.

For total heat output, the cone will report the same values it did for the compound containing Filler 1 and hence will overestimate the standard state heat output value.

On a specimen mass basis this will be overestimated by 1,17 × 0,6 = 0,70 kJ/g (i.e. 9,96 kJ/g and not 9,25 kJ/g of specimen).

The effective heat of combustion calculated on a mass loss basis by uncorrected oxygen depletion calorimetry and the organic mass loss based heat release rates will also be overestimated by:

$$24{,}89 - [(0{,}4 \times 24{,}89 - 0{,}6 \times 1{,}17)/0{,}4] = 1{,}76\ \text{kJ/g}$$

Thus the uncorrected values reported from calorimetry data will be 24,89 kJ/g whereas the actual value will be 23,13 kJ/g as shown in Table A.2.

Table A.2 — Heat release data for PMMA with insert Filler 2 that undergoes phase change

Filler	Total heat release per gram of specimen kJ/g	Total heat release per gram of organic material lost kJ/g	Total heat release per gram of specimen lost kJ/g
No filler	24,89	24,89	24,89
60 % filler data 2 uncorrected	9,96	24,89	24,89
60 % filler data 2 corrected[a]	9,96	23,13	23,13

[a] The term “corrected” refers to corrections due to phase changes.

c) PMMA plus Filler 3:

This is the most complex situation whereby the filler undergoes an irreversible process that involves a change in heat content and also has an associated mass change. In this case, a mass loss from the filler of 34,6 % has been specified which is 0,207 6 g/g for a 60 % loading. This mass loss will be measured by the cone load cell as part of the specimen loss and will influence the effective heat of combustion. The actual heat release on a per gram mass loss basis is therefore given by:

$$[(0{,}4 \times 24{,}89) - (0{,}6 \times 1{,}17)]/(0{,}4 + 0{,}207\ 6) = 15{,}23\ \text{kJ/g}$$

The effective heat of combustion as measured directly from oxygen depletion data would be given by:

$(0{,}4 \times 24{,}89)/(0{,}4 + 0{,}207\,6) = 16{,}39$ kJ/g

The values on the specimen mass and organic mass basis will not change from those quoted for Filler 2 above.

Table A.3 — Heat release data for PMMA with insert Filler 3 which undergoes phase change and mass change

Filler	Total heat release per gram of specimen kJ/g	Total heat release per gram of organic material lost kJ/g	Total heat release per gram of specimen lost kJ/g
No filler	24,89	24,89	24,89
60 % filler data 3 uncorrected	9,96	24,89	16,39
60 % filler data 3 corrected[a]	9,25	23,13	15,23

[a] The term “corrected” refers to corrections due to phase changes.

Filler 1 is typically of quartz whereas Filler 3 is of aluminium hydroxide.

Endotherm/water release fillers such as aluminium hydroxide and magnesium hydroxide, are commonly used as flame retardant additives. For aluminium hydroxide, the change is:

$Al(OH)_3\ (s) \rightarrow 0{,}5\ Al_2O_3\ (s) + 1{,}5\ H_2O\ (g) \quad + 1{,}17$ kJ/g

With an understanding of the above, it can be considered unnecessary to be overly concerned about errors due to even very high loadings of highly active fillers as this is shown to lead to relatively small errors in heat release rate measurements.

The worth of these fillers is seen in dynamic parameters such as rate of heat release but is also seen in effective heat of combustion where the diluent effect of the water release has a significant effect. Benefits related to this dilution are not seen with inert fillers.

The value of 15,23 kJ/g (or the less accurate value of 16,39 kJ/g) for the effective heat of combustion found for the aluminium hydroxide filled system should be contrasted with the value of 24,89 kJ/g found for the inert fillers.

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