TECHNICAL
SPECIFICATION

ISO/TS 5660-3

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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

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Foreword

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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 other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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

This first edition of ISO/TS 5660-3 cancels and replaces ISO/TR 5660-3:2003.

ISO 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 Specification]

Introduction

The first edition of ISO 5660-1, which describes a test method for rate of heat release from building products by means of a cone calorimeter, was published in 1993, following approximately 10 years of development within ISO/TC 92, *Fire safety*, Subcommittee SC 1, *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 5660-1:2002 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 5660-1:2002 and ISO 5660-2:2002 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 5660-2:2002. Further work is under way to define a quality control tool for measuring burning rates of building products. ISO 17554 specifies a test apparatus similar to that of ISO 5660-1:2002 but measures only loss of mass when exposed to radiant heat. Mass loss may be a surrogate for measurement of heat release for some classes of building materials. A similar system which measures the temperature of combustion products generated by this apparatus has been standardized as ISO 13927 [23]. The cone calorimeter fire model is used to measure corrosivity of gases products of combustion in 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 5660-1:2002 and ISO 5660-2:2002.

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

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 measurement limitations and applications of the cone calorimeter data 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. This information is presented as 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.

2 Normative references

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

ISO 5660-1:2002, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 1: Heat release rate (cone calorimeter method)*

ISO 5660-2:2002, *Reaction-to-fire tests — Heat release, smoke production and mass loss rate — Part 2: Smoke production rate (dynamic measurement)*

ISO/TS 14934-4:2007 *Fire tests — Calibration of heat flux meters — Part 4: Guidance on the use of heat flux meters in fire tests*

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. These characteristics include 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.

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 parameters, rather than ad hoc parameters.

When functioning as a rate of heat release apparatus, the parameter which is measured in the exhaust 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 10.

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 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 5660-1:2002. 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)

Detailed information on heat flux meter calibration is provided in ISO/TS 14934-4:2007.

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.

4.3 Calibration frequency

The setting of the required heat flux is set out in the manuals of the various instruments. Once a steady-state value has been obtained (fluctuations 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 provides information on the frequency of calibration of the instrumentation for the operation of the Cone Calorimeter.

Table 1 — Frequency for calibration and maintenance procedures

Equipment item	Prior to run	Daily	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 burner	Heat release flow rates of 5 kW and methane burner 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, start to record the oxygen analyser output at the same time the calibration burner is placed underneath the exhaust hood and continue until 3 min after removal of the calibration burner.

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 5660-1:2002 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 8,37 l/min at 273 K (0°C) and 1 atm (101,3 Pa)). 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 12.2, 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_{O2} (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 ≥ 99,5 %.

4.6 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 change greater than 5 % is indicative of a malfunction. 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,
- faulty methane flow meter,
- cold-trap refrigeration system clogged,

— 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.)

4.7 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.

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. The light transition factors of the filters should be checked every three years as the factor may change in use over several years.

4.8 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 sulfate has been found to be the most reliable drying agent and is recommended when carbon dioxide is to be analysed.

4.9 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.

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. 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;

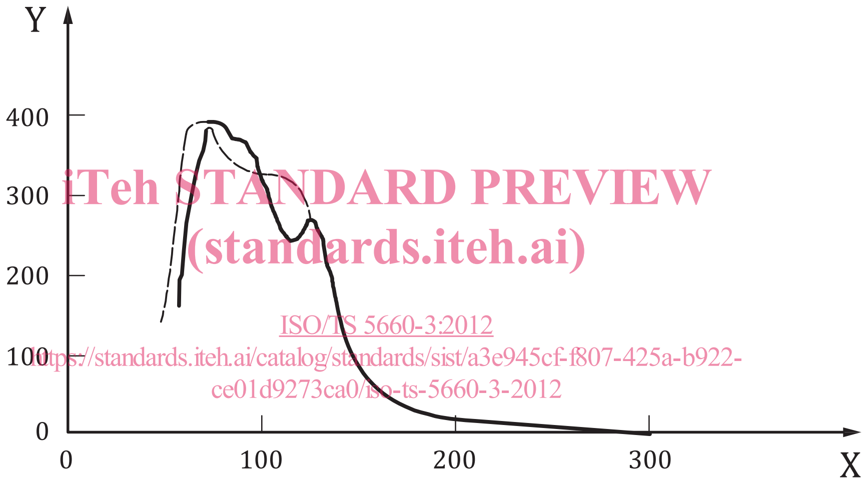
b) with an air gap that allows the specimen to burn on both sides;

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

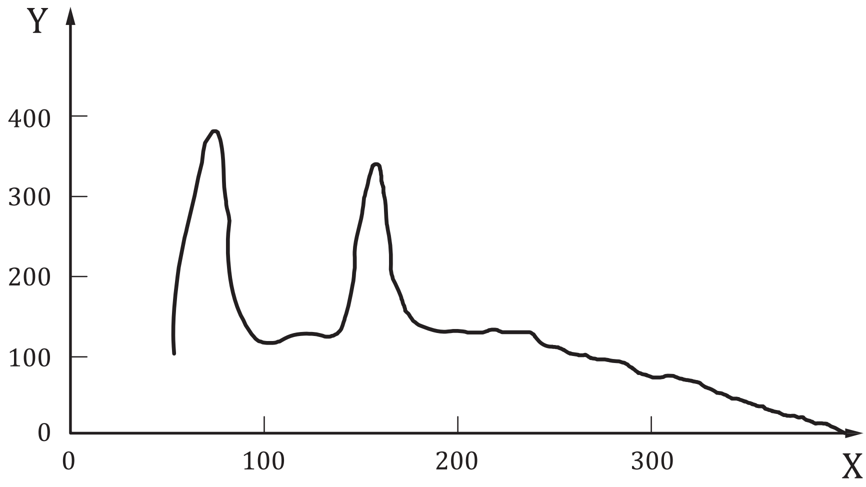
In all cases, it is important that specimen construction enable pyrolysis products that are generated behind surface layers to be vented from composite specimens in a similar way to that which they would be released in the burning end product. Combustion products can either be vented from the top of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

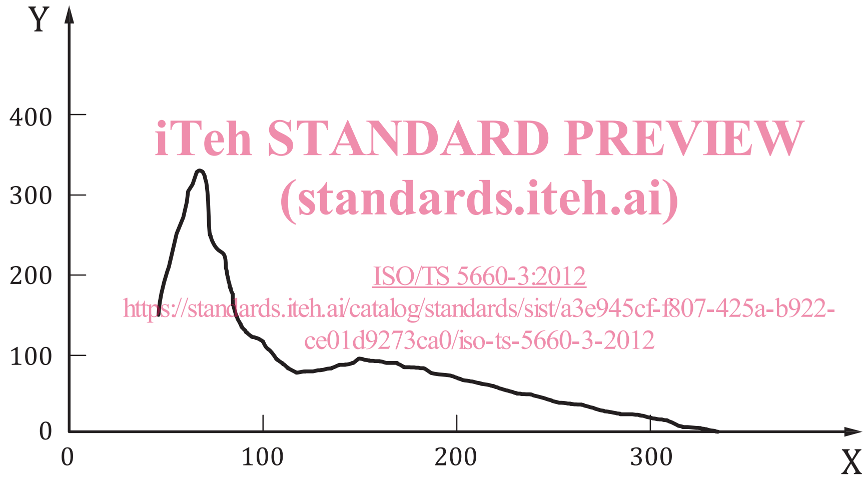
In other circumstances, pyrolysis can take place so rapidly that the combustion concentration limit is exceeded before the pyrolysis fumes ignite. The use of the specimen shield (6.2 of ISO 5660-1:2002) or positioning the igniter before the test may be of assistance. It is important that specifiers and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

ISO 5660-1:2002 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 [30].

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 5660-1:2002 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 selected are 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. This is 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 recommended 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 recommended 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.