CONE CALORIMETER BASED OXYGEN CONSUMPTION MEASUREMENTS ON "NON-COMBUSTIBLE" BUILDING MATERIALS

SAUERSTOFFVERBRAUCHSMESSUNGEN AN "NICHTBRENNBAREN" BAUSTOFFEN MIT DEM CONE - KALORIMETER

DETERMINATION DE LA CONSOMMATION D'OXYGENE DANS LES MATERIAUX DE CONSTRUCTION "INCOMBUSTIBLES" PAR LE CONE CALORIMETRE

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Abstract

It is reported here, that the oxygen consumption based calorimetry as realized in the ISO/DIS 5660 cone calorimeter apparatus, may be a helpful means for the assessment of non-combustibility of building materials being heterogeneous composites or assemblies as well.

Zusammenfassung

Es wird gezeigt, daß die Messung der Wärmeentwicklung im Cone Kalorimeter nach ISO/DIS 5660 nach dem Prinzip der Sauerstoffverbrauchsmessung hilfreich bei der Beurteilung der Nichtbrennbarkeit von mehrschichtigen, heterogenen Baustoffen sein kann.

Résumé

On montre que la determination du degagement de chaleur dans le cone calorimètre d'après le ISO/DIS 5660 et d'après le principe de la mesure de la consomation d'oxygene peut etre utile dans l'analyse des resultats de l'incombustibilité des materiaux de construction heterogènes et composés.

key words: oxygen consumption calorimetry, cone calorimeter, heat release, calorific potential, non-combustibility, inhomogeneous building materials, composites

1. INTRODUCTION

According to DIN 4102, part 1 [6], there are two german classes of noncombustibility of building materials: class A1 and A2. A1 and A2 classified materials are - within the single exception of chimney and furnace building purposes - identically used.
The difference between A1 and A2 classified materials is based on the requirements of heat release and flammability (see table 1).

Table 1
Tabulated requirements for classification of non-combustibility according to DIN 4102, part 1

requirement according to DIN 4102, part 1	classification A 1	A 2
I Non-combustibility test "Ofenversuch"		
duration of flaming	0 sec	≤ 20 sec
temperature rises	≤ 50 K	≤ 50 K
testing time	≤ 90 min	15 min
II determination of calorific potential "Bombenkalorimeter"		
calorific potential	not to be tested	≤ 4200 kJ/kg
heat release		≤ 16800 kWs/m²

Assessment of class A2 is performed by either using method I or II, of table 1, alternatively.

A future european classification system for building materials will certainly include only but one class of non-combustibility which will be defined on either passing a non-combustibility test according to ISO 1182 [7] or not exceeding an upper limit of calorific potential.

ISO 1182 test, however, is suitable only for homogeneous materials. Problems are obvious when testing non-combustibility of composites or assemblies.
The experiments of clause 5 may show that, on using a cone calorimeter according to ISO/DIS 5660 [9], results are obtained that are likely to serve as a helpful means for the assessment of non-combustibility [2] of these materials.

Determination of calorific potential according to ISO 1716 [8] by means of a bomb calorimeter is done with very small amounts of grinded material. Preparation of inhomogeneous materials will probably lead to separation of mixing proportions or, as far as composites or assemblies are concerned, yield not-to-the-real specimens of the grinded material.
The cone calorimeter [1] tests of clause 6 are to show if determination of calorific potential can be performed using ungrinded specimens of larger scale.

2. THE PRINCIPLE OF OXYGEN CONSUMPTION CALORIMETRY

It has long been known that there ist an empirically found relationship between the amount of heat produced in combustion systems and the oxygen consumed from the air stream. Hugget [3] made a detailed study of this relationship. He found out, that for most combustible materials such as gases, liquids, solids, included polymers, and natural materials, a value of 13.1 MJ heat released per kg of oxygen consumed was a close representation. This value may be used for practical applications and is accurate with few exceptions to within 5%. It must be emphasized here, that this constant must not be confused with the heat of combustion, which is defined as the heat released, per kg of fuel consumed.

The basic understanding of the oxygen consumption principle is simple: For each Joule of combustion heat generated, there is a fixed number of oxygen molecules removed from the exhaust stream.

It is to be noted, that the material does not need to burn fully to CO_2 for the relation of 13.1 MJ/kg of oxygen to hold. Typical yields of reaction products from combustion as e.g. CO or soot do not affect the accuracity of the results. Figure 1 shows a conceptional view of the main features of the principle of oxygen consumption measurements.

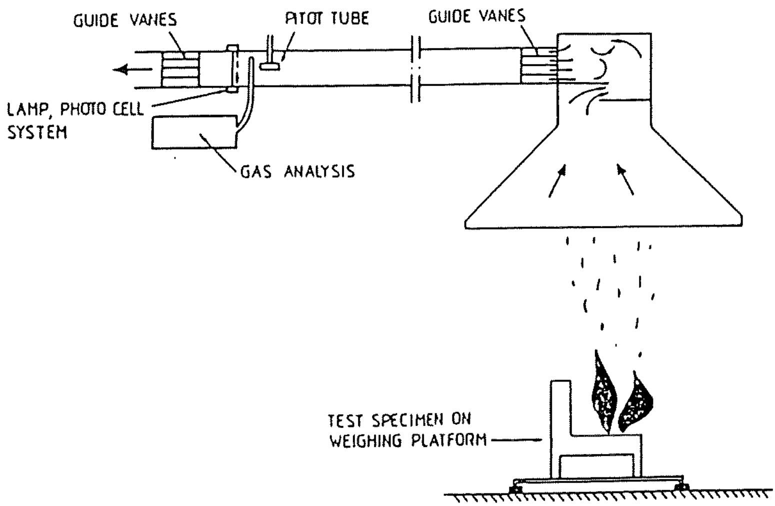

Fig. 1
Conceptional view of the main features of oxygen consumption calorimetry

3. TESTING APPARATUS : CONE CALORIMETER

The cone calorimeter apparatus as designed according to ISO/DIS 5660 [9] consists of the main components: a conical shaped heater, a load cell based sample holder, the exhaust duct, and the equipment for oxygen measurements.

The cone calorimeter run at the FMPA is a commercially available Stanton-Redcroft type (PL Thermal Sciences, Kiln Lane, Epsom, Surrey, UK). A general view is given in Fig. 2. The machine is equipped with optional CO/CO2-analyzer to render possible the measurement of combustion gases. A schematic diagramm is shown in Fig. 3. A compaq 286e personal computer is used to execute test runs an for data collection and storage.

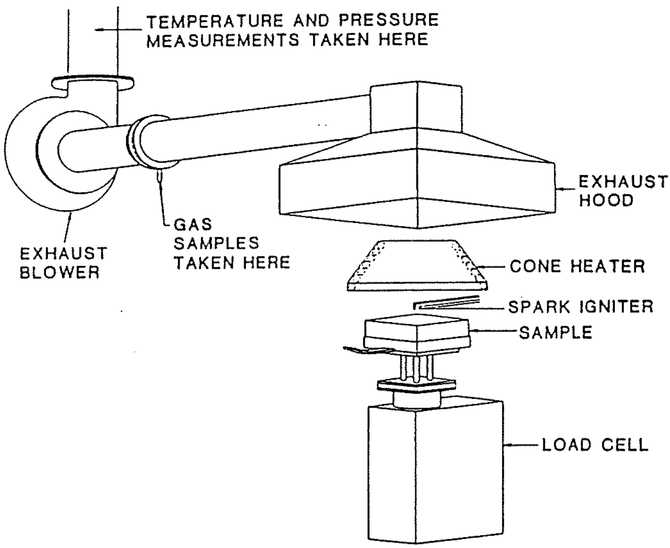

Fig. 2
General view of the main features of a cone calorimeter

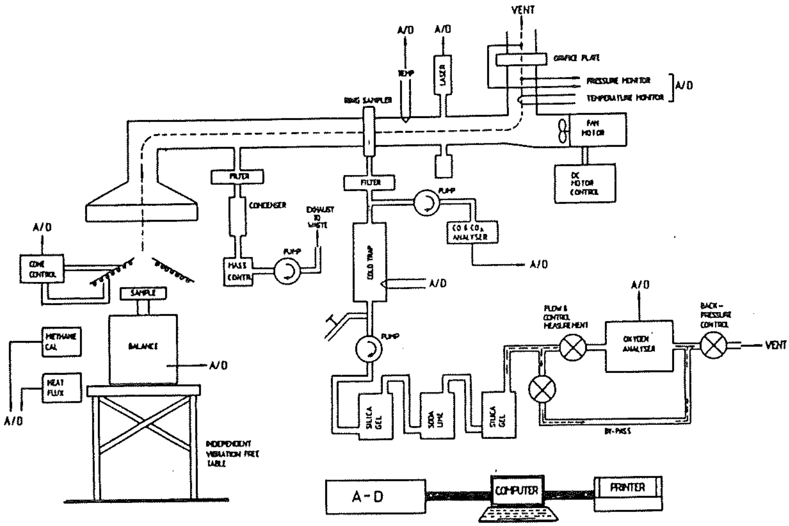

Fig. 3
Schematic diagramm of cone calorimeter

4. CONDITIONS FOR TESTING NON-COMBUSTIBILE MATERIALS

Radiation level of 75 kW/m² was decided to be a suitable value for testing non-combustible material [2], [5].
Specimen preparation was according to ISO/DIS 5660 [9], a retrainer frame was used, with the exception of F-series tests. All tests were performed in horizontal specimen orientation.
Data collection started at first positive rate of heat release.
Data reduction also started with first positive rate of heat release so to check the entire behaviour of tested material under the force of radiation, regardeless if there was ignition or not.
Calculations have been made by help of Parker's equations [4].

5. TEST ON PARTICLEBOARD COMPOSITE MATERIAL - DETERMINATION OF FLAMMABILITY AND HEAT RELEASE

5.1. Materials

Acoustic-board A2; particle board, 19 mm thick, visible side covered with coated glass fibre fleece, specific weight total about 540 kg/m³.

5.2. Testing conditions

- radiation level 75 kW/m²
- data collection started at first positive rate of heat release
- data collection ended at 320 sec
- retrainer frame was used

5.3. Results and discussion

Cone calorimeter results of tested acousticboard are shown in table 2. The results are from four series of tests on material from production series of different months.

ISO 1182 non-combustibility test [7] may rise problems when testing inhomogeneous composite or multi-layered materials. This is effect of sample preparation that causes specimens to be tested which are likeley different to the material's probable built-in or end-use form, respectively.

Both, DIN 4102, part 1 [6] and ISO 1182 [7] non-combustibility test requirements (see table 1) are:

- duration of flaming
- temperature rises, i.e. heat release

Table 2

Tabulated summary of cone results according to ISO/DIS 5660

Material: Acousticboard A2

Data collection: Start at first positive rate of heat release
End after 320 sec

Radiation: 75 kW/m²

1	test No			282i	282k	748	786/2
2	number of specimen tested			3	3	3	3
3	specific weight		kg/m³	531	560	499	540
4	specimen initial mass		g	101	106	100	103
5	total mass loss		g	14.5	16.7	12.0	13.5
6	time to ignition		s	15	12	13	17
7	duration of flaming		s	7	8	5	7
8	total heat evolved		kJ	26.2	29.1	18.3	24.4
9	effective heat of combustion		MJ/kg	1.8	1.7	1.5	1.8
10	rhr	average	kW/m²	8.6	9.3	5.7	7.6
11	60s	average	kW/m²	9.3	22.3	8.0	9.7
12		maximum	kW/m²	27.7	68.2	19.3	29.7
13	time to	maximum	s	18	17	15	18

The cone calorimeter test results in an identical set of data (see table 2, lines 7 & 8) as to:

- duration of flaming
- total heat evolved

It is obvious, that cone calorimeter testing opens access to estimating the non-combustibility of inhomogeneous or multi-layered building materials. Cone Calorimeter test of table 2 from building material of this kind points to the fact that results, concerning duration of flaming and total heat released, are rather close to each other and thus, seem to be reproduceable although showing some differences from various production series (see table 2, test No 282k).

6. TEST ON ALUMINIUM ASSEMBLY - DETERMINATION OF CALORIFIC POTENTIAL

6.1. Material

Facade board A2; aluminium - resin - composite, total thickness 4 mm, consisting of two (top and bottom) layers of aluminium, each o.5 mm thick and a 3 mm resin bonded core with a specific weight of 1600 kg/m³. The layers are joined together with a thin foil of glue.

6.2. Testing conditions

First tests (see table 3, P-series) with the complete specimen's top aluminium layer faced to the cone heater showed no signs of ignition nor combustion and no heat being evolved.
To solve for this problem, another set of tests was performed using specimens without aluminium layers (i.e. the resin bonded core only). Aluminium layers can be removed at a temperature of about 110°C.
Two testing series (see table 3, E- and F-series) at an irradiance level of 75 kW/m² were performed with specimens prepared this way. Tests of E-series were run with the use of a retrainer frame at a testing time of 1000 sec.

Owing to the fact that a testing-time schedule of 1000 sec may not have been sufficient for complete combustion, and the use of the retrainer frame may have affected the results due to heat conduction and/or edge-protection effects [5], the specimens of F-series were provided, unlike the other tests, without use of a retrainer fame, and not wrapped up in aluminium foil, with an expanded time-scale of 1800 sec.

6.3. Results

The results of this tests are given in table 3, F-series. It can be seen, in comparing the E- and F-series results, that the values of "duration of flaming" and "maximum rhr" are the most different ones and this is due to the fact of combustion being more violent with burning specimen's edges contributing to the flaming at once.

Figure 5 shows compared results of E- and F-series, given as graphs of the most typical cone calorimeter data:

- rate of heat release (heat evolved, related to time and specimen area),
- mass loss,
- effective heat of combustion (heat evolved, related to mass loss).

It is evident, that a short and violent burning comes out in an increased heat release rate maximum, together with shortened duration of flaming. It can also be seen, that with an expanded testing time of 1800 sec, there was obviously no more heat evolved at the end of time scale. This is confirming the evidence of specimen's complete combustion with no black residues of char to be detectable.

Table 3

Tabulated summary of cone results according to ISO/DIS 5660

Material: **Facadeboard**

Data collection: Start at first positive rate of heat release,
End after 600 sec (P-series),
1000 sec (E-series), and
1800 sec (F-series), respectively

Radiation: 75 kW/m²

test No			P1P2P3	E1E2E3	F1F2
number of specimen tested			3	3	2
specific weight		kg/m³	1863	1617	1617
specimen initial mass		g	74.5	48.5	48.5
total mass loss		g	3.7	23.3	25.7
time to ignition		s	no ign	180	162
duration of flaming		s	-	232	67
total heat evolved		kJ	0.06	127.1	155.9
effective heat of combustion		MJ/kg	0.01	5.5	6.07
rhr	average	kW/m²	0.0	12.7	8.66
60s	average	kW/m²	0.08	0.31	1.96
	maximum	kW/m²	0.97	28.7	147.9
time to	maximum	s	20	198	190

FMPA

Material identification: Al-sandwich

Heat flux: 75.0 kW/m2

Orientation: Horizontal

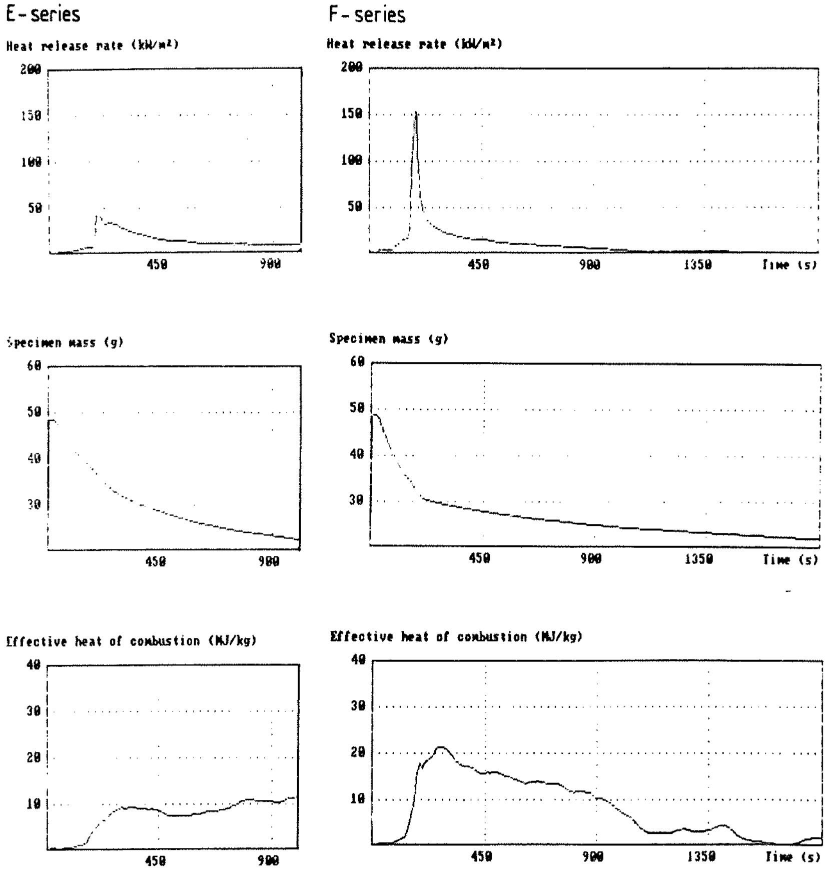

Fig. 4
Graphs of Heat release rate, specimen mass, and Effective heat of combustion; test performed with (E-series), and without (F-series) use of retrainer frame

6.4. Comparison of heat release data on basis of ISO 1716 and ISO/DIS 5660

This comparison is done based on results of test series F and the determination of calorific potential with bomb calorimeter.
The calorific potential of the glue-foil was known to be

$$Q = 41650 \text{ kJ/kg}$$

Thus, by means of a weighing and glueing procedure, the contribute of the glue-foil, removed together with the aluminium-layer from the specimen before test, is calculated.

6.4.1. Heat release from calorific potential Q according to ISO 1716

Q of core with attached glue foil	2931 kJ/kg
Heat release (calculated) of core with attached glue foil	14295 kWs/m²
Heat release (calculated) of glue foil attached to Aluminium-layers	2232 kWs/m²
Heat release (calculated) of entire specimen	16527 kWs/m²

6.4.2. Heat release from cone calorimeter tests according to ISO/DIS 5660

Total heat evolved	155.9 kJ
Heat release (calculated) of core with attached glue foil	15590 kWs/m²
Heat release (calculated) of glue foil attached to removed Aluminium-layer	1072 kWs/m²
Heat release (calculated) of entire specimen	16662 kWs/m²

7. CONCLUSION

As could be seen from the above investigations,

- cone calorimeter based determination of duration of flaming and total heat released in well defined time periods, may serve as a tool for the assessment of requirements for classification of non-combustibility of building material, being either inhomogeneous or composite assemblies or multi-layered products.

- heat release from inhomogeneous materials tested in cone calorimeter with suitable specimen preparation and testing conditions (as to radiation level and duration of test), is very close to calorific potential Q, gained by means of bomb calorimeter according to ISO 1716. Moreover, this is confirming the relationship of 13.1 MJ heat released per kg of oxygen consumed, and this is accurate for the complexity of composites or assemblies as well.

8. REFERENCES

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