

## WALL INSULATION PRODUCTS: FULL-SCALE TESTS VERSUS EVALUATION FROM BENCH-SCALE TOXIC POTENCY DATA

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### ABSTRACT

Questions of the toxicity associated with unwanted fires are often raised in many countries. The latest ISO recommendations for toxicity assessment of fire products emphasise solely bench-scale testing for toxic potency (the 'per-gram toxicity'). Yet, hazards of products with regards to fire toxicity may be much more determined by their differences in burning rates than by any differences in toxic potency. Burning rates are not assessed in the pertinent standards (ISO 13344 and ISO TR 9122). For many product categories, techniques for predicting full-scale burning rates from bench-scale data are not yet in hand. Thus, today the best means of comparing actual, full-scale toxic fire hazards is the full-scale fire test, equipped with additional gas measuring instrumentation. Such an approach is not among the recommended methods of the international standards, yet it is the only one with innate validity. In the present work, a series of wall insulation products were tested using the international standard full-scale fire test ISO 9705. The full-scale test scenario examines the fire hazards of insulation products as they would be seen during building construction or renovation. Gas analysis followed the recommendations of Nordtest NT FIRE 047. Bench-scale comparison was made to the ISO 5660 Cone Calorimeter and the DIN 53436 tube furnace. The conclusions were striking: the products showing the highest per-gram toxicity in the full-scale tests were, in fact, the least hazardous products. The actual hazard was assessed on the basis of total toxic gas production, heat release rate, and smoke production.

### INTRODUCTION

Coroner examinations of fire victims invariably reveal that the most common cause of fire death is due to inhalation of toxic gases. A new International Standard, ISO 13344 [1], has recently been published which intends to address the fire gas toxicity problem. Its recommendations instruct the user: "The fire model, or laboratory combustion device...must be chosen...in ISO TR 9122 'Toxicity Testing of Fire Effluents, Part 4: The Fire Model'." The referenced ISO TR 9122 Part 4 document [2] discusses only *bench-scale* testing for toxic potency. Yet it has been known for some time that toxic effects from fires must be viewed as being caused by the product:

*(toxicity) x (mass loss rate).*

For many situations, the variations in toxic potency among products are modest, but the differences in mass loss rates can be large. For one category of products, upholstered furniture, it was recently shown that the variations in full-scale burning rate among products can be huge, on the order of a factor of 100 [3]. The corresponding variations in bench-scale  $LC_{50}$ , on the other hand are greatly smaller. We note here that toxic potency is, conventionally, measured in an inverse scale. According to the definition of ISO 13344,  $LC_{50}$  = the concentration of gas or smoke statistically calculated from concentration-exposure data to produce lethality in 50% of test animals within a specified exposure and postexposure time. The units of  $LC_{50}$  are  $g\ m^{-3}$ . Because of the definition, large numerical values of  $LC_{50}$  mean that the toxicity is low, and vice versa.

The actual hazard to humans from a toxic gas exposure can be defined by the concentration-time history of the various toxic gas components, as measured at the nose of the victim. In many cases, it is

sufficient to evaluate simply the concentration-time integral, above a certain concentration baseline [4]. The bench-scale test methods outlined by ISO [2] measure, instead, the concentrations at the outlet tube of the bench-scale test apparatus. Such a means of assessment has intrinsically no validity. It would have validity only if:

1. the burning rates of all products would be the same (or, at least, in the same relative proportion) within the bench-scale toxicity test as they are in the real fire, and
2. the fire test conditions in the bench-scale apparatus would be so similar to the real fire that the toxic gas generation rates would all be in the same proportion as in the real fire.

For the 'best' of the bench-scale test apparatuses, point #2 can hold true, within an approximate way, provided certain test conditions are correctly controlled and certain data corrections are made as necessary [5]. Point #1, however, cannot even remotely hold true for any bench-scale test apparatus, when applied to almost any category of products. The primary reason is due to **flame spread**. A major difference in the hazards of real products is due to their flame spread performance. No category of product, apart from liquid pools, ignites near-instantaneously over its entire surface. Instead, the contributions to hazard generally differ because some products will spread flame slowly, others will spread flame fast; some may never burn their entire surface, others may be consumed in totality. No known bench-scale test can directly capture this flame spread performance of real products. Instead, the best that can be done today is to utilise **fire modelling** to take data from bench-scale tests and to predict the full-scale fire behaviour. Successful fire-modelling based prediction schemes have been created for a few product categories (e.g., [6]). For the majority of commercial product categories, however, such bench-scale/full-scale predictive methods are still wanting.

## PRODUCTS TESTED

The wall insulation products tested were selected to represent the categories of wall insulation most commonly found on the European marketplace. They comprised the following:

Table 1 The products tested

Code	Description	Foil face	Thick [mm]	Density [kg m <sup>-3</sup> ]	Installation	Rating
W1	polystyrene foam	N	50	16	glued to gypsum bd. with waterglass	DIN 4102 - B1
W2	polyurethane foam	N	40	34	nailed to lt. wt. concrete walls	DIN 4102 - B2
W3	lt. dens. rock wool	N	50	29	nailed to lt. wt. concrete walls	DIN 4102 - A1 ISO 1182
W4	high dens. rock wool	N	47	145	nailed to lt. wt. concrete walls	DIN 4102 - A1 ISO 1182
W5	polyisocyanurate foam	Y	46	32	nailed to lt. wt. concrete walls	unlabelled
W6	polystyrene foam (walls only)	N	50	16	(same as W1, but only on walls)	(same as W1)

The dimensions and densities were determined by actually measuring the test products. Products W3 and W4 meet the highest German category of 'A1' according to their national test. These also qualify as noncombustible under the ISO 1182 test. Product W1 was labelled by its manufacturer to meet the German 'B1' category of fire behaviour, which is the highest obtainable by a product not made essentially of non-combustible materials. Product W2 carried the German 'B2' classification. Product W5 did not carry any labelling with respect to flammability ratings. Thus, one item of interest in the

present programme was to see if B1 and B2 categories reflect appropriate levels of fire performance in full-scale testing. Products W3 and W4, in addition to the main components, also contain a small amount of organic binder. Product W6 represents the same material as W1, except mounted on the walls only. Since there is not a great deal of information on the full-scale product performance in the ISO 9705 test comparing the walls + ceiling vs. the walls-only arrangement, this test was intended to explore these differences.

## EXPERIMENTAL: FULL-SCALE METHODS

In the present study, we examined the performance of wall/ceiling insulation products alone. In a finished building, insulation products are generally covered by wall finish-lining materials. Thus, for user occupancy of buildings where the wall has been covered up, the normal conclusion is that the fire behaviour of such insulation materials is of only limited import [7][8]. Yet, such a situation may not hold for buildings which are in the process of construction or renovation. Furthermore, the conditions in buildings where construction work is going on are invariably more hazardous: plumbers' torches, welding equipment, salamanders, and other sources of heat and flame are used which would not be used in normal occupancy. Thus, it is of interest to examine the fire conditions which would result in such an environment.

A full-scale fire test method, ISO 9705 [9], has recently been established which is intended to examine the products used for the inside surfaces of rooms. This method has been studied and validated extensively [10]. The basic test configuration is shown in Fig. 1. The ignition source is a propane gas burner, 0.17 m by 0.17 m face dimensions. The burner is located in a far corner of the room, away from the door opening, and is placed directly on the floor. The burner power is 100 kW for 10 min, after which it is raised to 300 kW for 10 additional min.

The first half of the test allows the performance of general products to be differentiated. After the burner output is raised to 300 kW, the test is intended to discriminate between products which are of a higher fire performance. The test is continued for 20 min, unless strong flashover conditions demand extinguishing the fire at an earlier time. In the present tests, data gathering was continued for several additional minutes after the end of the test proper until gas analysis indicated that concentrations had returned to near-ambient.

## EXPERIMENTAL: BENCH-SCALE

The bench-scale test methods were selected from the recommendations made in ISO TR 9122-4. This document offers 8 different 'fire models,' which is its nomenclature for 'bench-scale tests.' Three of these are Japanese test methods which are not available outside of that country. The University of Pittsburgh test method has received some strong criticism for its design principles [11][12]. The NBS Cup Furnace method has been superseded by the NIST/SwRI radiant furnace test [5][13]. The latter is considered to be the optimum test method for *animal-based* testing, but is not commonly available in European laboratories and does not offer special advantages for *gas-analysis based* testing. Thus, the two remaining methods were selected: the DIN 53436 and the ISO 5660 tests.

### THE DIN TUBE FURNACE

The DIN 53436 tube furnace [14] is a method which can be used either for animal-based or for gas-analysis based toxic potency testing (Fig. 2). The test method uses a quartz tube which is surmounted by a moving annular furnace. Inside the tube, a trough holds the specimen. The preferred specimen size is 400 mm x 15 mm x 2 mm, but other specimen sizes may be used; for specimens of density < 400 kg

m<sup>3</sup>, additional thickness is mandated. A forced air flow rate of 100 to 300 L h<sup>-1</sup> is to be used, but more detailed guidance on air supply is not given. The standard test prescription envisions four test temperatures: 300, 400, 500, and 600°C. The three lowest temperature settings, however, in practice normally correspond to flameless pyrolysis, rather than actual burning. The 600°C temperature often gives transitional flameless/flaming results. For the work here, it was decided to include testing at one low and one high temperature. Noting that the developer of the DIN method, Prof. Einbrodt [15], has stated that "at 650°C selfignition took place partly only" and adopted 700°C for his own studies, we also adopted 700°C as the temperature for representing flaming combustion conditions. Following the recommendations of Kallonen *et al.* [16], we adopted 500°C as the low temperature test condition. In our view, the relevance of testing at temperatures lower than ones corresponding to robust flaming is tenuous, since the amount of gases liberated at such conditions tends to be greatly less than under higher temperature testing. Nonetheless, we included both test temperatures in the experimental work in order to conform to conventional practice for this test method.

In the present work, since animal tests were not made, an animal exposure device was not set up. Instead, following conventional practice, a simple mixing chamber was used at the outlet end of the tube furnace. The chamber was about 600 mL in volume and was insulated and heated. It provided a suitable location wherein sampling ports for gas analysis could be located. Secondary air was introduced into the mixing chamber at a ratio of 3:1 with respect to the primary air delivery rate.

#### THE CONE CALORIMETER

Cone Calorimeter testing followed the instructions of ISO 5660 Part 1 [17]. The smoke measurements were carried out as prescribed in ASTM E 1354 [18]. Following current recommendations [19], two test irradiances were utilised: 35 and 50 kW m<sup>-2</sup>. In all cases, ignition was with the use of the spark plug and an edge frame was used. A wire grid was not used. Specimens which are covered in aluminium foil were tested in two alternative ways: with and without Al foil.

#### EXPERIMENTAL: GAS ANALYSIS

The gas analysis was generally similar for both the full-scale and the bench-scale components of the study. The CO/CO<sub>2</sub> analyser was a Siemens Ultramat 22P, operating on the non-dispersive infrared absorption principle. It was only used for recording the CO<sub>2</sub> component, since better results for CO were achieved with FTIR measurements. The oxygen analyser was a paramagnetic-type, Siemens Oxymat 5E. An NO<sub>x</sub> analyser and a total unburned hydrocarbon (TUHC) analyser were also fitted. NO, NO<sub>2</sub>, and NO<sub>x</sub> were analysed using a Eco Physics CLD 700-EL-ht chemiluminescent analyser. The TUHC analyser was JUM Engineering model VE5. The analyser was heated internally as was also the sampling system feeding it. A temperature of 150°C was maintained.

For the remaining gases of concern, analysis was by FTIR (Fourier Transform InfraRed) procedures, as standardised in the recently-published standard Nordtest NT FIRE 047 [20]. The FTIR spectrometer was Bomem model MB 100, fitted with a DTGS detector. Its gas cell (supplied by Infrared Analysis Inc.) had a volume of 0.7 L and an optical path length of about 4 m, fitted with gold plated mirrors. The temperature of the cell was kept at 148°C. The temperature as well as the pressure inside the cell were continuously monitored. The pressure was used in the calculation algorithms for the gas concentrations. The instrument was used in the 4 cm<sup>-1</sup> resolution mode, and during the test three scans were co-added for each measuring record.

Table 2 Mass loss calculations

	W1	W2	W3	W4	W5 <sup>a</sup>	W6
<b>ISO 5660-1</b>						
C from CO <sub>2</sub> [g]	5.07	7.25	0.17	0.86	3.95	5.07
C from CO [g]	0.29	1.00	0.08	0.18	1.42	0.29
C from SPR [g]	1.1	0.68	0	0.02	0.18	1.1
ΣC [g]	6.46	8.93	0.25	1.06	5.55	6.46
Mass loss [g]	7.3	12.9	0.6	2.1	10.7	7.3
Ratio: mass loss/ΣC	1.13	1.44	2.39	1.98	1.93	1.13
<b>ISO 9705</b>						
C from CO <sub>2</sub> [kg]	4.99	6.89	0.24	0.65	11.99	7.42
C from CO [kg]	0.43	0.44	0.04	0.14	1.20	0.83
C from SPR [kg]	0.33	0.49	0.03	0.02	1.17	0.78
ΣC [kg]	5.75	7.82	0.30	0.81	14.36	9.03
Est. mass loss [kg]	6.50	11.30	0.72	1.60	27.69	10.20
Tot. test mass [kg]	25	42	45	209	45	18

<sup>a</sup> ISO 5660-1 data are from tests without the Al-foil as surface covering.

The analysis for the standard gases was done exactly as specified in the relevant test standards. For the supplementary gases measured, in the case of the full-scale tests in the room calorimeter, gas samples were taken from the exhaust duct (Fig. 3). A stainless steel probe was used having 31 holes evenly distributed over its length, which was spanning the diameter of the 400 mm diameter exhaust duct. To compensate for the pressure drop in the tube the holes furthest away from the suction end of the tube were made of a larger diameter. A heated filter was directly fitted to the outlet of the sampling probe. For the full-scale tests, the filter was a stainless steel cylindrical tube containing a ceramic filter element with 2 µm porosity and was heated to 180°C. For the bench-scale tests, a ceramic membrane filter was used, heated to approx. 100°C. From the filter, the gas sample was led through a heated sampling line comprising a 6 mm diameter PTFE inner tube and a heating sleeve on the outside. The sampling line from the filter was 5 meter long. The heating sleeve kept a temperature of 150°C measured at a location between the inner tube and the heating sleeve. The heated sample gas was

Table 3 Heat release and smoke production results

Code	W1	W2	W3	W4	W5	W6
Peak HRR <sup>a</sup> [kW]	> 6042 <sup>bc</sup>	> 8842 <sup>bc</sup>	29	78	2304	3383
Time to peak HRR [s]	120	24	840	1156	640	160
Time to extinguish [s]	140	70	—	—	—	—
Avg. HRR <sup>a</sup> [kW]	1591	4856	7	38	356	266
THR <sup>a</sup> [MJ]	> 223	> 340	9	46	428	319
Peak SPR [m <sup>2</sup> s <sup>-1</sup> ]	> 98	> 123	0.5	0.5	91	139
Total smoke prod. [m <sup>2</sup> ]	> 3285	> 4859	268	227	11678	7792

<sup>a</sup> The 100/300 kW output of the burner has been subtracted out.  
<sup>b</sup> Fire was extinguished.  
<sup>c</sup> Values over 5000 kW are considered only approximate.

transported to an intersection just in front of the FTIR gas cell, where it branched to the FTIR and to separate NO<sub>x</sub> and TUHC analysers. At the outlet of the FTIR instrument, a sampling pump was located, maintaining a constant flow rate of 4 L min<sup>-1</sup>.

The detection limits for the gases measured were determined to be the following: CO<sub>2</sub> < 5 ppm, CO = 5 ppm, HCl < 5 ppm, HBr = 10 ppm, HCN = 2 ppm, NO = 1 ppm, NO<sub>2</sub> = 1 ppm, TUHC = 1 ppm (when set to its lowest, 0-100 ppm range).

The general arrangements for the supplementary gas analysis used in the ISO 5660 and DIN 53436 bench-scale tests were very similar as for the full-scale testing, with the exception plumbing details. For the DIN 53436 tests, only FTIR, NO<sub>x</sub>, and TUHC analysers were used.

**Table 4** Peak concentrations and peak production rates for full-scale tests

Parameter	W1	W2	W3	W4	W5	W6
Peak conc. HCN [ppm]	*	414	9	22	733	*
Peak conc. HCl [ppm]	5	1069	16	12	992	36
Peak conc. HBr [ppm]	*	19	*	*	15	*
Peak conc. CO [ppm]	8320	11320	87	301	4367	4294
Peak conc. NO [ppm]	78	102	5	10	74	28
Peak conc. NO <sub>2</sub> [ppm]	3	7	2	7	5	*
Peak conc. TUHC [ppm]	7850	7373	21	36	2190	8197
Peak prod. rate HCN [mg s <sup>-1</sup> ]	*	851	21	49	2049	*
Peak prod. rate HCl [mg s <sup>-1</sup> ]	19	3092	56	42	3853	188
Peak prod. rate HBr [mg s <sup>-1</sup> ]	*	116	*	*	127	*
Peak prod. rate CO [g s <sup>-1</sup> ]	21.30	24.20	0.219	0.702	12.92	17.23
Peak prod. rate CO <sub>2</sub> [g s <sup>-1</sup> ]	436.2	491.6	22.80	25.80	267.9	290.3
Peak cons. rate O <sub>2</sub> [g s <sup>-1</sup> ]	269.6	534.3	26.02	30.13	235.7	283.8
Peak prod. rate NO [mg s <sup>-1</sup> ]	210	258	13.9	25.8	224	91.5
Peak prod. rate NO <sub>2</sub> [mg s <sup>-1</sup> ]	11.9	30.8	9.9	26.8	25.5	*
Peak prod. rate TUHC [mg s <sup>-1</sup> ]	17170	10440	31.4	48.0	2727	17010

\* measured concentrations were at or below the detection limit of the instrument.

## FULL-SCALE RESULTS

The ISO 9705 test does not provide a direct measurement of mass loss, since instrumentation to measure mass loss of large surfaces within a room is very difficult to implement and can be subject to significant errors due to buoyancy effects. By the use of the carbon-balance method, however, it is possible to estimate the mass loss. The method is based on the assumption that: (1) all the carbon released goes to CO, CO<sub>2</sub> and soot. (2) The carbon mass/fuel mass ratio is the same for the full-scale test as for the Cone Calorimeter test, where mass can be measured. (3) All soot is carbon. (3) Optical smoke measurements can be used to compute the soot production, according to the relation that 10 m<sup>2</sup> of smoke extinction are equals approximately 1 g of soot. The pertinent ISO 5660-1 tests were taken at an irradiance of 50 kW m<sup>-2</sup>. The CO<sub>2</sub> values were corrected by subtracting out the burner contribution. The results are given in Table 2. Table 3 provides summary data for heat release and smoke. Average values are calculated from ignition until 20 min after ignition.

Results from gas analysis in the full-scale tests are summarised in Table 4. Values of peak gas concentrations and peak production rates are given in Table 4; corresponding values of species production and yield are cited in Table 5. Data for NO<sub>x</sub> and TUHC are reported in Table 5. For yields,

the mass loss values estimated in Table 2 have been used. Note that the units used for CO<sub>2</sub> are 10<sup>3</sup> higher than for the other gas species.

**Table 5** Values of species production and yields for full-scale tests

Parameter	W1	W2	W3	W4	W5	W6
Mass prod. HCN [g]	*	32.1	4.1	22.0	287.3	*
Mass prod. HCl [g]	0.72	137.6	35.3	25.9	1132	59.7
Mass prod. HBr [g]	*	2.4	*	*	29.8	*
Mass prod. CO [g]	1000	1035	84.8	322.4	2809	1943
Mass prod. CO <sub>2</sub> [kg]	18.30	25.27	0.87	2.37	43.97	27.20
Mass cons. O <sub>2</sub> [kg]	12.57	28.13	19.25	22.49	57.93	45.64
Mass prod. NO [g]	4.97	7.62	6.96	16.14	30.92	9.24
Mass prod. NO <sub>2</sub> [g]	0.21	0.38	4.29	13.28	5.50	*
Mass prod. TUHC [g]	555	124	14.8	31.2	328	1235
Yield HCN [g kg <sup>-1</sup> ]	*	2.84	5.73	13.7	10.4	*
Yield HCl [g kg <sup>-1</sup> ]	0.11	12.2	49.3	16.2	40.9	5.85
Yield HBr [g kg <sup>-1</sup> ]	*	0.21	*	*	1.08	*
Yield CO [g kg <sup>-1</sup> ]	155	91.6	119	2 <sup>o</sup> 1	101	190
Yield CO <sub>2</sub> [kg kg <sup>-1</sup> ]	2.83	2.24	1.21	1.48	1.59	2.67
Yield NO [g kg <sup>-1</sup> ]	0.76	0.67	9.7	10.1	1.12	0.91
Yield NO <sub>2</sub> [g kg <sup>-1</sup> ]	0.03	0.03	6.0	8.3	0.20	*
Yield TUHC [g kg <sup>-1</sup> ]	86.0	10.9	20.6	19.5	11.9	121.0

\* measured concentrations were at or below the detection limit of the instrument.

## BENCH-SCALE TEST RESULTS

### CONE CALORIMETER RESULTS

Cone Calorimeter tests were typically performed at the fluxes of 35 and 50 kW m<sup>-2</sup>, first with two replicates where only standard test data were collected, followed by a third test during which FTIR analysis was also used. Table 6 presents the summary data, averaged for the replicate tests. Gas data are given in Table 7. The yields for TUHC have been computed on the basis of CH<sub>4</sub>-equivalent mass.

### DIN TUBE FURNACE RESULTS

All specimens were tested without any Al-foil (for products having an Al-foil face), since the method was considered not intended for composite constructions. The values given at each temperature condition represent the average of two replicates. The results are given in Table 8.

## ANALYSIS

### HEAT RELEASE AND SMOKE PERFORMANCE

By now it is very well-known that heat release rate (HRR) is the single most important variable determining hazard from fire [21]. This is because it is the *driving force* for fire, and when it is increased, all other aspects of hazard generally also tend to increase. The two primary HRR characteristics listed in Table 3 are peak HRR and average HRR, along with information as to whether the test needed to be extinguished. Two specimens, W1 (polystyrene foam) and W2 (polyurethane foam) caused fires in excess of 5 MW and needed to be extinguished. Specimen W6 was the same as

W1, except that it was installed on walls-only, instead of walls+ceiling. In the ISO 9705 room, the walls+ceiling arrangement comprises 31.68 m<sup>2</sup> of specimen area, while the walls-only arrangement has 23.04 m<sup>2</sup> of specimen area. Thus, on the basis of test W6, one might anticipate that W1 results would have shown a peak HRR value of (31.68/23.04)×3383 = 4652 kW. Instead, the measured value was recorded as > 6042. Such measurements of HRR values over 5 MW in the standard ISO 9705 are uncertain, but in any case the comparison shows that ceiling contributions are somewhat more dangerous than wall contributions to HRR. A detailed presentation of HRR results is given in Fig. 4.

Table 6 Main results from Cone Calorimeter tests

Flux	W1		W2		W3		W4		W5 <sup>a</sup>	
	35	50	35	50	35	50	35	50	35	50
<i>t<sub>ig</sub></i>	64	32	4	2	NI	NI	NI	NI	3	2
<i>t<sub>f</sub></i>	236	176	377	797					277	756
<i>q''<sub>pk</sub></i>	305	291	124	170	6	8	8	12	79	106
<i>q''<sub>60</sub></i>	284	274	93	123	3	5	2	9	35	51
<i>q''<sub>180</sub></i>	141	149	70	91	3	4	5	9	20	34
<i>q''<sub>300</sub></i>	89	89	42	74	2	3	5	8	16	31
<i>q''<sub>tot</sub></i>	25.5	26.9	21.1	34.2	0.93	1.1	2.0	4.0	4.7	21.6
Pk. SPR	0.145	0.139	0.073	0.124	<0.002	<0.001	<0.001	0.001	0.056	0.098
Avg. SPR	0.065	0.077	0.01	0.0087	*	*	*	*	0.003	0.0023
<i>m<sub>o</sub></i>	8.0	8.2	13.6	13.6	16.3	14.7	75.0	81.1	14.2	14.2
$\Delta m$	7.1	7.3	8.7	12.9	0.5	0.6	1.6	2.1	3.4	10.7
MLR	4.50	5.23	2.38	1.63	*	*	*	*	1.31	1.44
$\Delta h_c$	36.1	36.7	24.2	26.5	17.1	16.9	12.7	19.4	14.0	20.2
TSP	10.21	10.95	3.73	6.78	*	*	*	*	0.74	1.78
SEA	1450	1500	430	530	*	*	*	*	220	170

Values in this table represent average for three test runs.

\* Measured values were at or below the detection limit of the instrument.

<sup>a</sup> Tested without the Al-foil.

The following nomenclature applies to the Cone Calorimeter test results in the above table. *t<sub>ig</sub>* = ignition time (s). *t<sub>f</sub>* = flameout time (s). *q''<sub>pk</sub>* = peak HRR (kW m<sup>-2</sup>). *q''<sub>60</sub>* = 1-min avg. HRR (kW m<sup>-2</sup>). *q''<sub>180</sub>* = 3-min avg. HRR (kW m<sup>-2</sup>). *q''<sub>300</sub>* = 5-min avg. HRR (kW m<sup>-2</sup>). *q''<sub>tot</sub>* = total heat released (MJ m<sup>-2</sup>). *m<sub>o</sub>* = initial sample mass (g).  $\Delta m$  = mass lost (g). fract. MLR = average mass loss rate (g m<sup>-2</sup> s<sup>-1</sup>).  $\Delta h_c$  = effective heat of combustion (MJ kg<sup>-1</sup>). SPR = smoke production rate (m<sup>3</sup> s<sup>-1</sup>). TSP = total smoke produced (m<sup>3</sup>). SEA = test average specific extinction area = (total smoke produced, m<sup>3</sup>)/(total mass lost, kg).

A general classification and product rating scheme based on ISO 9705 results has not been proposed, but, qualitatively, it is generally agreed that:

- flashover in the first 10 min (during 100 kW burner exposure) is more dangerous than flashover during the subsequent time period (of 300 kW burner exposure)
- no flashover at all during the test indicates the highest level of product performance
- products which flash over sooner are more hazardous than those which take a longer time to flash over
- products which show high amounts of smoke evolution are more hazardous than those which show limited smoke production.

Based on these considerations, we may organise the product performance according to Table 9.



Table 7 Cone Calorimeter gas analysis results

Flux	W1		W2		W3		W4		W5 <sup>a</sup>	
	35	50	35	50	35	50	35	50	35	50
Pk. conc. HCN	*	*	29	16	3	*	*	*	4	5
Pk. conc. HCl	7	11	77	153	9	8	*	7	195	223
Pk. conc. HBr	*	*	*	12	13	13	*	13	*	*
Pk. conc. CO	324	347	188	219	39	64	*	95	133	232
Prod. CO	619	677	421	2330	*	189	*	428	573	3300
Prod. CO <sub>2</sub>	18.5	18.8	16.8	25.6	0.27	0.64	1.66	3.15	3.50	15.7
Prod. HCl	16	37	414	518	*	41	*	15	692	1310
Prod. HCN	*	*	110	56	*	*	*	*	6	60
Prod. HBr	*	*	*	43	*	158	*	117	*	*
Prod. NO	5	5	68	94	*	27	*	28	11	52
Prod. NO <sub>2</sub>	2	2	3	26	*	7	*	17	6	25
Prod. TUHC	563	510	144	122	*	6	*	31	110	357
Cons. O <sub>2</sub>	16.56	18.48	14.10	20.90	*	0.64	*	1.84	3.16	14.92
Yield HCl	85	77	48	180	*	300	*	204	170	310
Yield CO <sub>2</sub>	2.47	2.52	1.94	1.97	0.50	1.01	1.04	1.53	1.07	1.52
Yield HCl	2.2	4.3	47	40	*	66	*	7.0	206	123
Yield HCN	*	*	13	4.3	*	*	*	*	1.7	5.6
Yield HBr	*	*	*	3.3	*	251	*	56	*	*
Yield NO	0.6	0.6	7.8	7.2	*	42	*	13	3.2	4.9
Yield NO <sub>2</sub>	0.2	0.3	0.3	2.0	*	11	*	8.0	1.9	2.3
Yield TUHC	77	58	16.5	9.4	*	10	*	15	33	33

\* Measured values were at or below the detection limit of the instrument.  
<sup>a</sup> Tested without the Al-foil.

The following nomenclature applies to the Cone Calorimeter test results in the above table. Pk. conc. x = Peak concentration of species x (ppm). Prod. of species x = (total mg of species x); except, Prod. CO<sub>2</sub> = (g CO<sub>2</sub> produced). Cons. O<sub>2</sub> = consumption of O<sub>2</sub> (g). Yield of species x = (total production of species x, g)/(total mass lost, kg); except, Yield CO<sub>2</sub> = (kg CO<sub>2</sub>)/(total mass lost, kg).

Three distinct groups of performance can be seen in the results. Products W3 and W4 (lt. dens. rock wool; high dens. rock wool) never resulted in room flashover and emitted only very small quantities of smoke. Product W5 (polyisocyanurate foam) showed flashover after the initial 0-10 min period; it also produced a very large amount of smoke. Products W1 (polystyrene foam) and W2 (polyurethane foam) resulted in very fast flashover, 70 s and 12 s, respectively. These fires were so large—over 5 MW—that extinguishment was required. As a result, heat and smoke production could only be characterised by “greater than” values.

#### TOXIC FIRE HAZARD ANALYSIS

To perform an evaluation of the toxic fire hazard, under ISO 13344 the basic analysis is done by use of the *Fractional Effective Dose* (FED) principle. This principle, arithmetically, simply represents that the toxic effects of various gases are linearly additive. Thus, if we have three gas species, a, b, and c, then,

Table 8 Results of DIN tube furnace tests

	W1		W2		W3		W4		W5	
	500°C	700°C	500°C	700°C	500°C	700°C	500°C	700°C	500°C	700°C
Sampled vol. (20°C)	380	388	384	389	395	379	387	393	380	384
Mass lost [g]	2.6	2.65	3.97	5.04	0.2	0.19	0.18	0.2	4.00	4.56
Conc. HCN	*	*	566	46	3.5	4.5	11	11	297	38
Conc. HCl	*	*	*	216	*	*	*	*	*	353
Conc. HBr	*	*	*	*	*	*	*	*	*	*
Conc. CO	260	770	9070	234	299	101	302	263	7280	542
Conc. CO <sub>2</sub>	0.008	0.99	0.62	1.55	0.071	0.095	0.074	0.084	0.58	1.34
Conc. NO	0.3	2.2	2.2	18.4	3.8	10.3	3.5	16.3	2.0	18.8
Conc. NO <sub>2</sub>	<0.2	<0.2	5.3	3.6	5.5	4.3	5.4	9.0	3.9	3.0
Conc. TUHC	7960	162	1230	23	39	3.2	31	11	890	90
Prod HCN	*	*	244	20	1.5	1.9	4.7	5.0	127	16.3
Prod HCl	*	*	*	128	*	*	*	*	*	207
Prod HBr	*	*	*	*	*	*	*	*	*	*
Prod CO	115	348	4050	106	137	45	136	120	3220	241
Prod CO <sub>2</sub>	0.056	7.04	4.32	11.1	0.52	0.66	0.53	0.61	4.09	9.47
Prod NO	0.16	1.06	1.07	8.9	1.89	4.87	1.70	7.98	0.96	9.00
Prod NO <sub>2</sub>	<0.1	0.13	3.9	2.74	4.1	3.1	4.0	6.8	2.8	2.2
Prod TUHC	2020	42	316	5.9	10.3	0.81	8.1	2.8	226	23
Yield HCN	*	*	61	3.9	7.7	10.0	34	25	32	3.6
Yield HCl	*	*	*	25	*	*	*	*	*	45
Yield HBr	*	*	*	*	*	*	*	*	*	*
Yield CO	44	131	1020	21	686	237	948	608	805	53
Yield CO <sub>2</sub>	0.022	2.66	1.09	2.20	2.59	3.50	3.68	3.09	1.02	2.08
Yield NO	0.060	0.40	0.27	1.8	9.6	25	11.3	40	0.24	2.0
Yield NO <sub>2</sub>	*	*	*	*	*	*	*	*	*	*
Yield TUHC	777	16	80	1.17	52	4.3	54	14	57	5.0

\* Measured values were at or below the detection limit of the instrument.

The following nomenclature applies to the DIN tube furnace test results in the above table. Conc. x = average concentration of species x (ppm); except, Conc. CO<sub>2</sub> = average concentration of CO<sub>2</sub> (%). Prod. of species x = (total mg of species x); except, Prod. CO<sub>2</sub> = (g CO<sub>2</sub> produced). Yield of species x = (total production of species x, g)/(total mass lost, kg); except, Yield CO<sub>2</sub> = (kg CO<sub>2</sub>)/(total mass lost, kg).

Table 9 Performance ranking based on full-scale test results

Code	Product	Time to f/o [s]	F/o period	Tot. smoke produced [m <sup>3</sup> ]
W3	lt. dens. rock wool	—	never	268
W4	high dens. rock wool	—	never	227
W5	polyisocyanurate foam	630	10 - 20 min	11700
W6	polystyrene foam (walls only)	73	0 - 10 min	7792
W1	polystyrene foam	70	0 - 10 min	> 3285
W2	polyurethane foam	12	0 - 10 min	> 4859

$$FED = \frac{C_a}{LC_a} + \frac{C_b}{LC_b} + \frac{C_c}{LC_c}$$

where  $C_a$  etc. are the concentrations of the pertinent species ( $\text{g m}^{-3}$ , or alternatively, ppmv), and  $LC_a$ , etc. are the values of the  $LC_{50}$  for species  $a$ , etc. A value of  $FED=1$  means that, at the 50% probability level, the lethal level has been reached,  $FED < 1$  denote gas mixtures not expected to cause lethal conditions, while  $FED > 1$  means 'more than enough' for lethality. It can readily be seen that if there is only one toxic component present, and if its concentration is equal to its  $LC_{50}$  value, then  $FED = 1$ , as expected from the basic definition of  $LC_{50}$ . In actual fact, some non-linearities can be expected when more than one toxic species is present. A very extensive research programme at the National Institute of Standards and Technology (NIST), led to recommendations [13] which include the non-linear interaction of CO and  $\text{CO}_2$ . This interaction relationship, however, will be dependent on the overall dilution within the building fire scenario, which is outside the scope of the present comparison. Thus, here we will simply utilise a linear expression:

$$FED = \frac{[CO]}{5000} + \frac{[HCN]}{150} + \frac{[HCl]}{3800} + \frac{[HBr]}{3000} + \frac{[NO]}{1000} + \frac{[NO_2]}{200}$$

We omit a term for  $\text{CO}_2$  here. The innate toxicity of  $\text{CO}_2$  is very low (ca. 400 000 ppmv). Its main effect is to change the breathing rate and, therefore, indirectly to change the uptake of other gases. Because of the abovementioned concern with dilution, it is best to omit this term when performing a comparative product analysis, in the absence of knowledge of actual building air flow rates. In the same vein, an additive term in the FED due to oxygen depletion (ca. 5.4%) is not included. The actual values of  $LC_{50}$  will depend on the exposure time. In the present situation, the length of time that occupants might be exposed to toxic fire gases is not pre-determined. For the sake of convenience, the reference values used are for 30 min exposure times, as determined from experiments on rats, since data for other exposure times are more scant. The values in the equation above are literature values quoted in ppmv, and these then need to be individually converted to  $\text{g m}^{-3}$ , when the experimental measurements are on a gram basis. Total unburned hydrocarbons (TUHC), if they occur as paraffinic compounds, are nearly non-toxic. In the cases of some materials, however, they can represent highly irritant components, such as aldehydes, which are significantly more toxic. In general, however, ISO 13344 does not include TUHC amongst the toxicants to be analysed; thus we do not assign a toxic effect to the TUHCs measured.

Without a specific building fire in mind, not only is the exposure time arbitrary, but also the volume into which the gases are to be distributed in order to compute specific concentrations. Again, relative comparisons will not suffer if an arbitrary volume of  $10 \text{ m}^3$  is assigned. The results are given in Table 10. Also shown are comparative values from the two bench-scale tests. For the bench-scale tests, an arbitrary volume of  $0.01 \text{ m}^3$  was taken.

**Table 10** Relative toxicity, expressed as FED, from various tests

Code	Product	Full-scale	DIN 500°C	DIN 700°C	Cone 35 kW m <sup>-2</sup>	Cone 50 kW m <sup>-2</sup>
W3	lt. dens. rock wool	7	5.1	3.5	≈ 0	9.9
W4	high dens. rock wool	26	7.0	8.3	≈ 0	17.4
W6	as W1, walls-only	36	2.0	6.2	12.2	13.6
W1	polystyrene foam	> 18	2.0	6.2	12.2	13.6
W2	polyurethane foam	> 41	221	18	86	100
W5	polyisocyanurate foam	248	135	19	29	131

In the above table, the relative toxicity performance of the products which did not need to be extinguished can be evaluated quantitatively. For the products which were extinguished (W1 and W2), an exact ranking is not possible; we have suggested a possible ranking for those, however. In Table 10 we can observe that, for most of the products, the ranking from full-scale FED is identical to the ranking obtained from the HRR+smoke analysis performed in Table 9. The only exception is product W5. This product, while not among the fastest burning products, obviously produces a somewhat more toxic gas mixture than average.

How well bench-scale results do represent full-scale fires can be gleaned from Table 10. None of the bench-scale test methods/conditions lead to the same rank ordering as in the full-scale tests. The Cone Calorimeter results at an irradiance of  $50 \text{ kW m}^{-2}$  are much more consistent with the full-scale results than tests conducted at  $35 \text{ kW m}^{-2}$ . The DIN results at  $700^\circ\text{C}$  are quantitatively very far away from what would be expected, since they show only a slight difference between W4 (which, in full-scale, hardly burned and produced very little of toxic gases) and W5 (which caused flashover and produced copious products of combustion). The DIN results at  $500^\circ\text{C}$  are also unsatisfactory, since they would suggest that W6 (which rapidly caused flashover in the full-scale test) is a 'better' product than W3 (which, in the full-scale test, hardly burned or emitted gases at all). We can conclude that, of the bench-scale test conditions, the Cone Calorimeter results at a flux of  $50 \text{ kW m}^{-2}$  produced results most nearly consistent with what was actually measured in full-scale fires. Nonetheless, even these bench-scale results are 'compressed.' In the full-scale tests, W5 was 35 times worse than W3, yet the bench-scale  $50 \text{ kW m}^{-2}$  results only show a ratio of 13 : 1.

Since it is known that the role of one toxicant, CO, is dominant in determining fire fatalities [22], it is appropriate to look at some comparisons of CO yields in more detail. Table 11 presents this comparison.

**Table 11** Comparison of CO yields ( $\text{g kg}^{-1}$ ) for the different tests

Code	Flashover	Full-scale CO	Cone CO		DIN CO	
			35	50	500°C	700°C
W1	yes	155	85	77	44	131
W2	yes	92	48	180	1020	21
W3	no	119	a	300	686	237
W4	no	201	a	204	948	608
W5	yes	101	170	310	805	53
W6	yes	190	85	77	44	131

<sup>a</sup> Could not be determined, since mass consumed during test was very small.

From the above table, we can first observe how closely the full-scale CO values agree with the general recommendation of assuming a CO yield =  $0.2 \text{ kg kg}^{-1}$  for all flashed-over fires [23]. The four values measured in this study were 0.155, 0.092, 0.101 and 0.190; such factor-of-2 agreement is consistent with the general level of predictability for toxicity data [5]. Specifically, we can note that concerns expressed in some quarters [24] about a possibility of much higher CO yields are not sustained by the present findings.

For non-flashed over tests, Cone Calorimeter results at a  $50 \text{ kW m}^{-2}$  irradiance showed CO yields within a factor of 2-3 of the full-scale results. The Cone Calorimeter results at  $35 \text{ kW m}^{-2}$  irradiance are similar in some cases, while being lower in others. Because of the inability to pyrolyse significant amounts of gases from the low combustibility products, however, the  $35 \text{ kW m}^{-2}$  irradiance would not

be recommended for this product class. For the full-scale tests which did lead to flashover the Cone Calorimeter agreement was about at the same level as for the non-flashed over tests.

For the DIN test, however, the discrepancies between measured CO yields and full-scale values are greater. At the 500°C condition, for all the products except W1/W6, the values recorded are enormous, ranging from 0.686 to 1.020 kg kg<sup>-1</sup>. These bench-scale values are 4.7 to 11.0 times the actual full-scale values. At the 700°C test condition, the DIN test yields were slightly less aberrant, but still of poor agreement. Especially troublesome is the extremely wide discrepancy between the results at the 500°C and 700°C conditions. For product W2, the results at the 500°C condition were 49× those at 700°C; for product W5, the disparity was 15×. The DIN method does not provide any means of selecting the 'right' test temperature and, in fact, there is some implication that worst-case results should be sought. The present findings do not lend confidence in the realism of such procedures.

The above comparisons of the predictive capabilities of bench-scale toxicity tests suggest that there are physical differences among the bench-scale test methods, and that some methods are more suited to predictive tasks than others. The use of the DIN furnace test at a 'low' temperature, such as 500°C, is clearly indicated to be unproductive in representing real-scale fires of the kind examined in this study. More important, however, such comparisons merely emphasise the fact that a flame spread/burning rate *model* does not exist for insulation products. Such a model (which would presumably involve more than just linear scaling) would be necessary to use bench-scale data productively to predict full-scale fires.

#### THE ROLE OF TOXICITY VERSUS BURNING RATE

The procedures of ISO TR 9122 and ISO 13344 imply that it is sufficient to examine the toxic potency of the test products and it is not necessary to consider relative burning rates. Such an approach would be viable if the products tended to show large differences in their toxic potencies, but small differences in their burning rates. We can examine this thesis using the ratio of the full-scale FED to two measures of burning rate (total heat released and total mass lost), as given in Table 11. The ratio FED/ $\Delta m$  is especially instructive, since this is the actual measured determination of *toxicity per gram* of the tested products. Thus, comparing the results of Table 12 against those of Table 10, we can make a striking observation: **the products showing the highest per-gram toxicity in the full-scale tests were the least hazardous products.** The fact that W3 and W4 were the least hazardous products is consistently evident by all measures: they produced the lowest amount of heat, the lowest amount of smoke, and the lowest total toxicity of the gas stream (as evidenced by their full-scale FED values). Yet, if one were to judge product solely by their per-gram toxicity, W3 and W4 (rock wool specimens) would be judged to be the worst, while W1 (polystyrene foam)—which led to room flashover in less than 2 min—would be judged the best. The reason why the better-performing products performed well is clear: they showed very little mass loss and HRR. Consequently, the actual amount of toxic gases released was very small.

An additional conclusion emerges from this: **if ranking products by their per-gram toxicity in a full-scale test leads to nonsensical results, ranking products solely by their per-gram toxicity from bench-scale test results is even less appropriate**, since a bench-scale test will hardly represent the full-scale reality with 100% accuracy.

Exactly how well bench-scale data even come close to full-scale data can be considered separately. Since the Cone Calorimeter test results at an irradiance of 50 kW m<sup>-2</sup> were seen to be the best bench-scale descriptor of the full-scale results, we will focus on this test condition. The Cone Calorimeter LC<sub>50</sub> values were obtained using Eq. 5 of ISO 13344:

$$LC_{50} = \frac{\Delta m}{FED \times Tot. air vol.}$$

The comparison is given in Table 13.

**Table 12** Ratio of FED to several measures of burning rate in the full-scale tests

Code	Product	Ratio FED/THR	Ratio FED/ $\Delta m$	Full-scale $LC_{50}$ [g m <sup>-3</sup> ]
W3	lt. dens. rock wool	0.778	9.72	10.3
W4	high dens. rock wool	0.565	16.25	6.2
W5	polyisocyanurate foam	0.579	8.95	11.1
W6	as W1, walls-only	0.113	3.53	28.3
W1	polystyrene foam	0.081	2.77	36.1
W2	polyurethane foam	0.121	3.63	27.6
$\Delta m$ = total mass loss (kg)				

**Table 13** Comparison of toxicity in full-scale and in bench-scale tests

Code	Cone Calor. FED	Cone Calor. $\Delta m$	Cone Calor. $LC_{50}$ [g m <sup>-3</sup> ]	Full-scale $LC_{50}$ [g m <sup>-3</sup> ]
W3	9.9	0.6	6.1	10.3
W4	17.4	2.1	12.1	6.2
W5	131.	10.7	8.2	11.1
W6	13.6	7.3	53.7	28.3
W1	13.6	7.3	53.7	36.1
W2	100.	12.9	12.9	27.6

The above table is arranged in the same order as Table 10, that is, in increasing order of estimated toxic hazard. The last two columns of Table 13 show that the Cone Calorimeter  $LC_{50}$  values are within a factor of two, or closer, to the corresponding full-scale values. However, as demonstrated above, even a perfect bench-scale prediction of  $LC_{50}$  would not correct rank-order the actual fire performance of the products seen in full-scale fire testing.

## CONCLUSIONS

A series of tests on five different wall/ceiling insulation products, representing the main general types of products found on the European marketplace, has shown that:

1. Until more predictive fire models are evolved, a full-scale fire test is the best means of evaluating the toxic fire hazards from products.
2. Reliable, time-resolved toxic gas data can be obtained for multiple gas species by use of the FTIR technique.

3. The full-scale test method used—ISO 9705—does not yet have an approved classification system. Nonetheless, by evaluating the critical performance aspects, one can derive the following rankings.

*No flashover:* Light density rock wool; high density rock wool.

*Flashover reached, but only during the later (10-20 min) period:* Polyisocyanurate foam.

*Flashover reached early in the test:* Polystyrene foam, polyurethane foam.

Both of the latter two products (polystyrene foam, polyurethane foam) led to severe enough fire conditions that these tests needed to be quickly terminated by manual extinguishment.

4. The recommendations of ISO 13344 and ISO TR 9122 suggest to the user that bench-scale data alone are sufficient to evaluate the toxic fire hazards from products. For many product categories, however, the differences between 'good' and 'poor' performance (from a fire toxicity point of view) does not lie in the per-gram toxicity as determined with the  $LC_{50}$  variable reported from those tests. Instead, many products show toxic fire hazard reduction due to *reduced rates of burning* (which is not examined in ISO 13344 testing), rather than *improved  $LC_{50}$  values* (which is the sole topic of ISO 13344).

5. In the full-scale tests reported here, the two products showing the best actual performance (by all measures: HRR, smoke, and toxic gas production) showed the worst ranking when assessed solely by 'per-gram toxicity' in those full-scale tests. This graphically demonstrates the inadequacy of ranking products according to their per-gram toxicity (or  $LC_{50}$ ) **even in full-scale tests**. Ranking products by their  $LC_{50}$ , as measured in bench-scale tests, will lead to even more misleading conclusions, since no bench-scale test is a perfect predictor.

6. Bench-scale test results differ significantly, depending on the test conditions—temperature or heat flux settings. Correct values cannot be established *a priori*, without the benefit of full-scale testing.

7. For bench-scale testing, the Cone Calorimeter gives more realistic CO yields than does the DIN test. The CO levels from DIN tests at 500°C are especially discordant with the full-scale measurements.

8. Full-scale testing can be highly costly. Thus, the cost-effective strategy is to seek to develop, for various product categories, predictive models which will allow flame spread, HRR, and mass loss rates to be predicted on the basis of bench-scale test results.

9. Once such models are available, valid predictions will be possible from bench-scale data. It will be more appropriate to obtain the toxic component of the fire hazard using the *same* bench-scale test which is used to obtain parameters for predicting other aspects of the full-scale fire. The Cone Calorimeter is the only widely-used test method today which has this capability.

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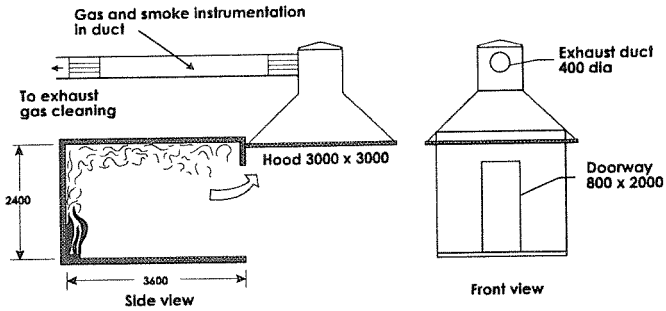


Figure 1 Basic arrangements of the test room according to the ISO 9705 test.

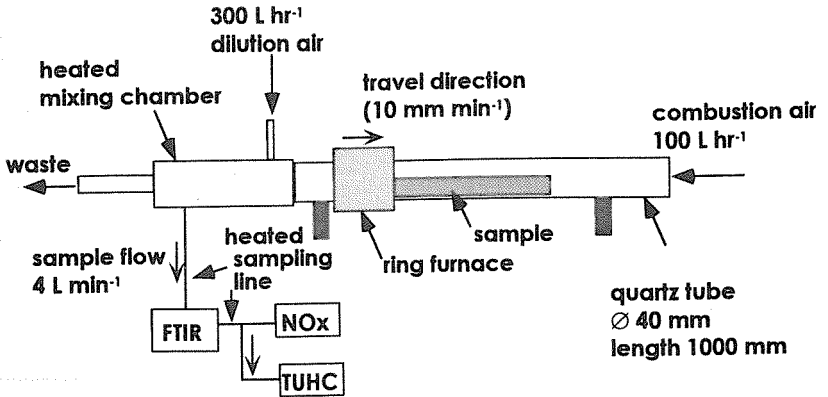


Figure 2 General view of the DIN 53436 tube furnace test.

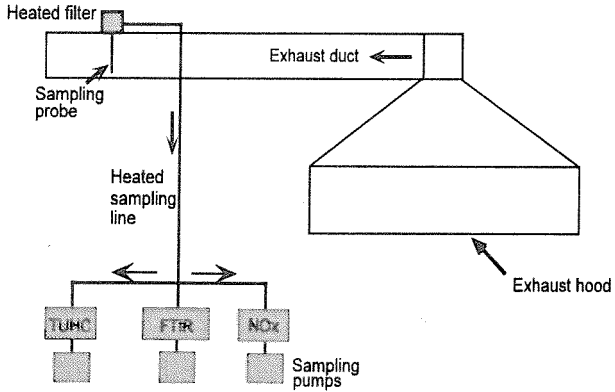


Figure 3 Arrangement of the supplementary gas analysis equipment in the full-scale tests.

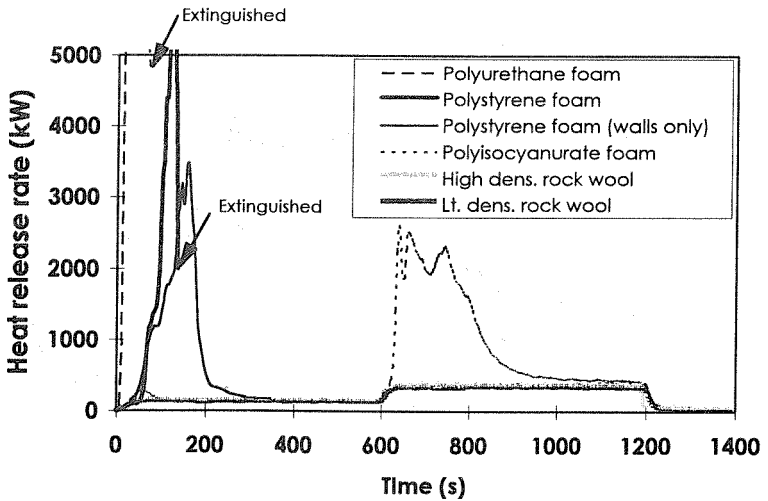


Figure 4. Comparative HRR results for the tested insulation products. Note that the burner output is not subtracted out in these curves.

# **INTERFLAM '96**

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