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1983

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REPORT LUTVDG/(TVBB-3017)

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RHR OF BUILDING MATERIALS

Experiments with an OSU-apparatus using oxygen consumption

by

Jan Blomqvist

Research project financed by the Swedish Fire Research Board
(BRANDFORSK)

SUMMARY

The Ohio State University rate of heat release apparatus was calibrated with two measurement systems, the standard compensated thermopile and an instrumentation for oxygen consumption measurements. Based on the calibration results the oxygen consumption technique was chosen when testing 13 different materials including wood-based materials, wallcoverings and plastics. The same set of materials has also been tested in a full scale room-corner test and in a number of small scale reaction to fire tests at different Scandinavian laboratories. These results are reported separately.

Results for the 13 materials are presented as RHR and THR at three radiation exposure levels 2, 3 and 5 W/cm^2.

INTRODUCTION

Within ISO/TC92 development work on a set of new reaction to fire tests for building materials started in the late sixties. The most fundamental and also the most technically complicated of the proposed tests is a method for measuring the rate of heat release (RHR). A large number of equipments for RHR measurements in small scale have been developed mainly in the US. Some of these are at present only realistic as research tools /1/, /2/, but others are intended as possible standard methods /3/, /4/, /5/, /6/. The only equipment that is commercially available is the Ohio State University (OSU) apparatus. An ISO survey of heat release tests was recently published by Janssens /7/. Tsuchiya /8/ has also reviewed the existing RHR tests.

Since the use of oxygen consumption technique for measuring RHR was established /9/ most activity has concentrated on equipments using this technique. In this report parallel experiments in an OSU-apparatus with the standard measurement system and with an oxygen consumption measurement system are described. The oxygen consumption technique proved to be preferable. The same conclusion was drawn by Babrauskas /10/ from a similar calibration activity.

The main experimental test series here reported included 13 materials (wood-based, wallcoverings, plastics) tested at three radiation exposure levels 2, 3 and 5 W/cm^2. The same set of materials has also been tested in a full scale room-corner test /11/ and in a number of other small scale reaction to fire tests at different Scandinavian laboratories. One of the small scale tests was an open RHR test /12/, /13/. The results from the different experiments form a unique data base, that makes it possible to study e. g. the correlation between full scale behaviour and small scale tests.

OXYGEN CONSUMPTION

The determination of RHR from oxygen consumption is based on the fact that the energy release per unit oxygen consumed is nearly constant for complete combustion of the organic compounds of interest in fire studies.

The RHR is calculated as

$$\dot{q} = h(\dot{x}_o - \dot{x}) \qquad (1)$$

where

h is the heat release per mole of oxygen consumed and
$\dot{x}_o$ and $\dot{x}$ are the molar flow rate of oxygen in the incoming and outflowing gases, respectively.

Huggett /9/ has calculated the value 420 kJ/ (mole of O_2) to be used as an average h value in fire studies. If the heat of combustion and the exact composition of a sample is known, an exact value of h may be calculated.

When CO is produced the h value is reduced. When there is a large excess air flow, as in the OSU-apparatus, the production of CO is small, and no correction for CO is necessary.

The major practical difficulty in RHR measurements is the accuracy of the oxygen consumption $\dot{x}_o - \dot{x}$. In the OSU-apparatus the air flow is determined at the entrance, which is much easier than flow measurements in the hot outflowing gases. The combustion causes an increase in the number of moles in the flue gases. When calculating RHR from the measured oxygen concentration this expansion must be considered. If any gas component is trapped before the oxygen analyzer, the RHR calculation is also affected. Detailed information on RHR calculations for various experimental situations was given by Parker /14/. In the experiments here reported water was trapped in the gas sampling system. Especially when testing a material, that gives off a large amount of moisture, precision is improved by measuring on dried gas.

An equation for RHR based on the combustion of propane, oxygen measurements on dried gas and neglecting CO-production for this experimental situation is

$$q = h V \frac{(O_{2s} - O_2)(1 - X_{H_2O})}{100(1 - 0.004\, O_2)} \qquad (2)$$

where

V = flow rate of air into the apparatus (mole s^{-1}),
O_{2s} = oxygen reading prior to experiment (volume percent),
O_2 = oxygen reading (volume percent), and
X_{H2O} = mole fraction of water of the incoming air.

INTRODUCTORY STUDY FOR CHOICE OF MEASURING SYSTEM

During the first phase of the experimental work the compensated thermopile and oxygen consumption were used simultaneously. The equipment was calibrated at the standard air flow rate, 40 l/s, with

* methane/propane (normally 6 kW)
* radiant panel off/on ($3 W/cm^2$).

The thermopile consisting of three thermocouples in the stack and three at the air inlet was recorded separately from the compensation thermocouple. Results from four different calibration experiments are illustrated in Fig 1. The signals in the figure are the increase in mV output of the compensated thermopile calculated as

$$S = P - P_s - x(K - K_s) \quad (3)$$

where

P is thermopile signal (mV)

K is compensator signal (mV)

P_s, K_s are signals prior to the experiment

x is a constant chosen to get the best square wave response.

Fig 1 was plotted using x = 1.5, which gave the best square wave response. The value 1.5 is higher than the values possible according to the thermopile wiring diagram in the ASTM proposal /3/. From the figure the value 8.8 mV for 6 kW was chosen as calibration of the compensated thermopile.

A series of experiments with materials was carried out using the two different measuring systems. An impinging pilot flame at the lower end of the specimen was used and the radiation level was approximately 3 W/cm^2. No details about the oxygen consumption measurements in these introductory tests will be described here. The measurement system was however similar to the one used for the main test series. Calibration with gases showed a 10% maximum difference between measured RHR and heat content of the gas. An explanation for this relatively large error is the high standard air flow rate 40 l/s.

When testing a material that gives no rapid changes in heat release the two ways of measuring RHR show a good agreement. A particle board experiment, Fig 2, is one example of this behaviour. When testing a material which causes more dramatic changes in RHR, a large difference is obtained, see Fig 3 from a wallcovering test. The conclusion drawn from the introductory test series was that the compensated thermopile is unreliable and inferior to the oxygen consumption technique.

EXPERIMENTAL

In this section the equipment is described as it was used for the main test series when measuring oxygen consumption only.

OSU-apparatus

The experiments were carried out using an OSU-apparatus, Fig 4, modified to suit the oxygen consumption technique. The air supply into the exhaust system of the apparatus was sealed since this air flow only acts as a dilution when using oxygen consumption. A longer stack was used, where the gas sampled 430 mm from the bottom and an optical smoke measuring system was placed above the gas sampling point. Only the combustion chamber was insulated with a ceramic fiber blanket (Kaowool) and mineral wool. The insulation was used mainly for the comfort of the operator.

Electric supply

The radiation source, consisting of four Glowbar elements coupled in series, was supplied with electric power from a thyristor converter that can be adjusted in the range 0-12.5 kW. The heat flux generation capacity of this electric supply has not been chequed, but probably the radiation level 10 W/cm^2 could be reached.

Air supply

The distribution system for compressed air available in the building was used with two pressure regulators to obtain a steady air flow. This air supply has a stable dew-point at $2^{\circ}C$. The variation in air flow was calibrated with a vortex flow meter and the static pressure at the inlet of the apparatus was noted for various flow levels. During the test series the air flow was set at 11.0 ± 0.3 l/s (in some experiments 12.3 l/s) using the static pressure reading.

Test specimen

All materials were tested in the vertical direction. The test materials were conditioned at $20^{\circ}C$ and 65% relative humidity. The test specimen was covered with one layer of aluminum foil on all surfaces except the exposed area. As backing material a 10 mm thick Vermit-S board (density $830 kg/m^3$, thermal conductivity 0.1 W/m K) was used.

Pilot flame

The pilot flame was non-impinging positioned 10 mm above the centre of the specimen, 5 mm behind the surface and directed towards the radiation source. The pilot flame tube had an inner diameter of 4.8 mm. The pilot flame was fed with propane, 72 W, and enough air to get a blue flame.

Gas measuring system

The gas probe had five small holes spaced along the centre of the exhaust stack. The gas was pumped (20 l/min) through a filtering system consisting of a loosely packed glass-wool filter, a pleated capsule filter (3 μm Gelman) and a silica gel filter. The main stream of filtered gas was pumped through a MSA 803 P stack gas analyzer with a stabilized zirconium oxide electrolyte as sensor and with reference gas containing 21.0% O_2 from a gas bottle. A small fraction of the gas stream was pumped through an Infrared Industries IR 702 instrument analyzing for CO and CO_2. The T_{90} time of this measuring system was less than 10 seconds, excluding the transportation time to the instruments, which was 5 seconds for the O_2-meter and 8 seconds for the CO/CO_2-meter. In some experiments at the end of the test series these instruments were replaced by a Siemens Oxymat 2 (paramagnetic) and a Leybold-Heraeus Binos 1. 2. The experience from using the two different oxygen analyzers is, that the paramagnetic one is preferable, but the less expensive zirconium instrument is an acceptable alternative when measuring oxygen consumption in the OSU-apparatus. The zirconium instrument was also used in full scale room experiments, but in this case it was not possible to obtain acceptable measurements results with this instrument. The reason for the different experiences is yet unknown.

Smoke

The smoke measurement system consisted of a tungsten halogen lamp and a photocell with a human eye filter. The lamp was kept at a constant voltage to get a stable colour temperature. The same smoke measuring system has also been used in the full and model scale room-corner tests and in the open RHR test.

Data collection

The measured data (4 channels) were registered on a Solartron 3430 Compact logger. The sampling interval was 3-4 s. The logger stored the data on a cassette. The cassettes were then brought to a computer that made all calculations and plotted the data.

CALIBRATION

Heat flux

The heat flux was measured with a water-cooled Medtherm flux meter, that had been calibrated in a spherical black body calibration furnace. An attempt to measure the variation of heat flux over the specimen area was made with the air flow set at normal level and no specimen holder. The flux meter was introduced through a hole in a piece of noncombustible board, which replaced the steel sheet, that would normally close the combustion chamber when a specimen is inserted. Results from a calibration at 3 W/cm^2 are illustrated in Fig 5. The difference between the maximum value measured in the centre and the minimum measured in one of the lower corners was a little more than 10%.

During the test series heat flux was measured in one point slightly below centre position. For these measurements the heat flux meter was mounted in a simple stand, which when measuring heat flux replaced the lower radiation shield door. The use of a stand ensured that heat flux was always measured in the same position.

RHR

The calibration of the thermopile was discussed on page 5. In this section the calibration results for the oxygen consumption measurements will be presented.

A line burner with 7 holes was used for the calibrations.
Results from different calibrations with methane and propane are plotted in Fig 6. The calibrations illustrated includes results from the two different oxygen analyzers. The difference between heat release measured with oxygen consumption and heat input measured with a Porter F-150-A flow meter was within ± 5%. The two dominant sources of error were probably the gas flow meter and the air flow measurement.

Calibration experiments with different levels of radiation from the panel showed as expected, that the radiation level does not influence the measured heat release.

During the main test series a one point calibration at 6 kW was performed daily with ±5% deviation as acceptance criteria.

TEST SERIES

RHR measurements have been performed for 13 materials, Table 1, at three levels of radiation exposure 2, 3 and 5 W/cm^2. The materials were tested three times at each exposure level to obtain some indication of repeatability. Normally, two of the three identical experiments were run immediately after one another and the third experiment was not run the same day. The test series was carried out by two operators each running approximately one half of the experiments.

Table 1

Material	Thickness mm	Density kg/m³	Basis weight kg/m²	Results in Fig
Particle board	10	670	6.7	7
Insulating fibreboard	13	250	3.2	8
Medium density fibreboard	12	655	7.9	9
Solid wood (spruce)	11	450	5.0	10
Melamine faced particle board	13	870	11.3	11
Gypsum plaster board	13	725	0.25*	12
Paper wallcovering on gypsum plaster board	13+0.5		0.45*	13
PVC wallcovering on gypsum plaster board	13+0.7		0.49*	14
Textile wallcovering on gypsum plaster board	13+0.5		0.63*	15
Textile wallcovering on mineral wool	42+0.5	150	6.5	16
Paper wallcovering on particle board	10+0.5		0.20 (paper)	17
Rigid polyurethane foam	30	32	1.0	18
Expanded polystyrene	49	18	0.9	19

*weight of combustibles on the exposed surface

TEST PROCEDURE

The daily starting procedure was that, after the air flow had been set at the correct level, the radiation panel was turned on. The time to stabilize the radiation at the decided level was 30-60 min. The time needed between experiments to allow the apparatus to return to the correct radiation level was less than the time needed for preparing the equipment for a new run.

The starting procedure of an experiment was that, after the gas pumps had been started, the data collection began. Thirty seconds later the specimen holder was placed in the hold chamber with the radiation shield doors closed. The specimen was retained in the hold chamber for 60 seconds before injection into the combustion chamber. The exact time of the injection was noted by the operator, which made it possible to compensate for differences when presenting the data. The experiments were normally terminated approximately 10 min after injection or when the specimen seemed to be totally consumed.

The glass-wool and silica gel in the filtering system was changed after 3-6 experiments. The gas analyzers were calibrated before and after their use in the test series. No significant changes in calibration constants were observed except in the CO_2 channel of the Infrared IR 702 instrument.

RESULTS AND DISCUSSION

Rate of heat release and total heat release

The results for the thirteen materials are presented in Fig 7-19 (Figure numbers for the different materials are included in Table 1). Each figure consists of a RHR-curve and an integration of the RHR-curve here named total heat release (THR) for the three exposure levels. Only one representative example of the triplicate experiments is illustrated. In the figures, time zero is the time of injection into the combustion chamber. For the calculations Eq. 3 was used with h=420 kJ/mole and $(1-X_{H2O})/(1-0.004\ O_2)=1.05$.

Reproducibility

The test results indicate that the reproducibility is good for most of the materials. The largest variation was noted for polystyrene as could be expected for a thermoplastic material tested in vertical orientation. Fig 20 (melamine faced particle board, 5 W/cm^2) and Fig 21 (particle board, 2 W/cm^2) are two examples of the reproducibility. The figures were chosen to be illustrative of results with a large variation in RHR. The melamine faced particle board shows an oscillating RHR during the first minute. The particle board shows some variation in time to ignition, which is not surprising considering the variation in ignition time observed in true ignition tests at low exposure levels /15/. In both examples the variation in THR appears to be much smaller. Probably the reproducibility of a RHR test could best be expressed in terms of THR. To obtain quantitative information on the reproducibility of the OSU equipment a larger number of repeated experiments is required.

Smoke and CO

Because of the relatively high air flow and the short light pathway, the decrease in transmittance observed during experiments was often small. A few examples of smoke production are illustrated in Fig 22, where it is expressed as

$$TSR = 10\frac{\dot{V}}{\ell}\int_0^t \log\frac{I_o}{I(\tau)}d\tau \quad (dB\cdot m^2) \qquad (4)$$

where

$\dot{V}$ = air flow (0.011 m^3/s)

I = transmittance

I_o = start value of transmittance

ℓ = light pathway (0.133 m).

An analysis of all smoke measurements (full scale, model scale, OSU and open RHR, NBS smoke density chamber, Swedish box test) on the 13 materials is scheduled to be finished this year. The usefulness of smoke measurements in connection with the OSU method is of course dependent on the correlation with the full scale results.

The CO concentrations were normally low, < 500 ppm, but when testing polyurethane up to 7000 ppm was recorded. Even at the highest CO level measured a correction for CO production can only be motivated, when the energy release per unit oxygen value used is based on known chemical composition of the burning material.

Comparisons

Of course the most interesting kind of comparison is an examination of the correlation between this and other small scale tests with the full scale results. Such work was started recently and will hopefully be reported this year. Examples of heat release curves from the OSU-apparatus and from room experiments for a few materials were presented in /16/.

In this paper only some comments on the different THR curves at 3 W/cm^2, Fig 23, will be given. The difference between the different wood-based materials in the THR is small. In the room experiment all these materials except the insulating fibreboard behaved in a similar way. The insulating fibreboard experiment developed much faster. For the textile wallcovering, that was tested on two different backings, again the effect of the density difference was much stronger in the room experiments than in the OSU tests.

Experiments with low THR

An indication of the possibility to use the OSU-apparatus for measuring the heat release for materials, that contain relatively small amounts of combustibles, are the results from the testing of gypsum plaster board with and without a paper wall-covering (Fig 12-13). Assuming that the heat of combustion of the paper is 16 MJ/kg, the expected THR of the gypsum plaster board at a high exposure level is 4 MJ/m^2 and with the paper 7 MJ/m^2. The measured THR at 5 W/cm^2 reached in both cases somewhat higher levels, 5 and 9 MJ/m^2. Taking into account that the zirconium type oxygen analyzer was used for these experiments, the measurement results do indicate, that the equipment can with some precision handle materials releasing small quantities of heat.

Cost of testing

It is possible to make RHR tests at a high speed using the OSU-apparatus. In the test series here reported a maximum of 20 tests were performed during one day of work (8 hours). In this test series the measured data were stored on cassettes, which were then brought to a computer. This procedure is time consuming and the operator has no possibility to see if the experiment has worked out correctly.

If the apparatus is used as a standard it should be connected to a local computer system, which without almost any action from the test operator does the calculations and presents the data in a suitable graph.

Testing in horizontal direction

For this activity the specimens were always in the vertical direction. When testing with a horizontal specimen a number of new problems arise, e. g. the reflector and the pilot ignition source. An experimental study of the horizontal direction is now being planned.

Babrauskas /10/ has made experiments with PMMA in horizontal direction observing a continuous increase in RHR, because the apparatus is heating up. Whether this is a serious problem or not, is a question that will be answered when a way of using heat release data to predict fire development becomes available. If only the initial period of heat release is interesting, the heating of the apparatus will be of minor importance. For testing in vertical direction the increasing temperature of the upper part of the apparatus cannot be of great importance, since the specimen receives little radiation from the upper part because of the small view angle.

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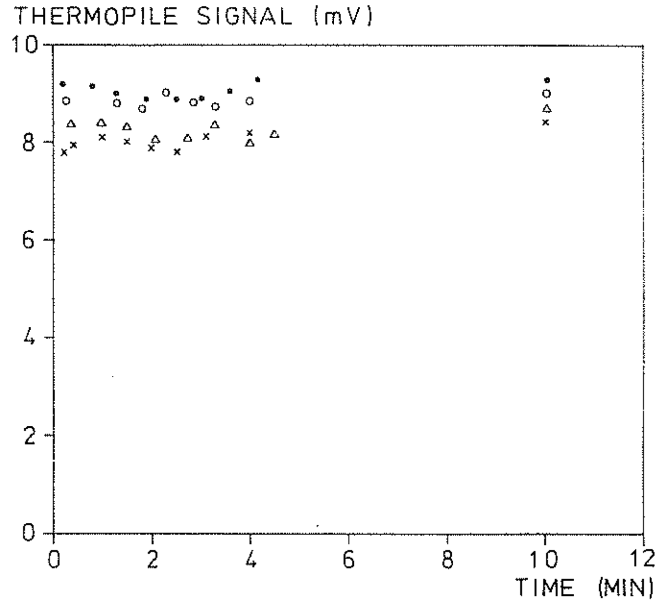


Fig 1 Calculated response of the compensated thermopile for a 6 kW step increase of the gas flow

• Propane no radiation
o Methane no radiation
Δ Propane 3 W/cm²
x Methane 3 W/cm²

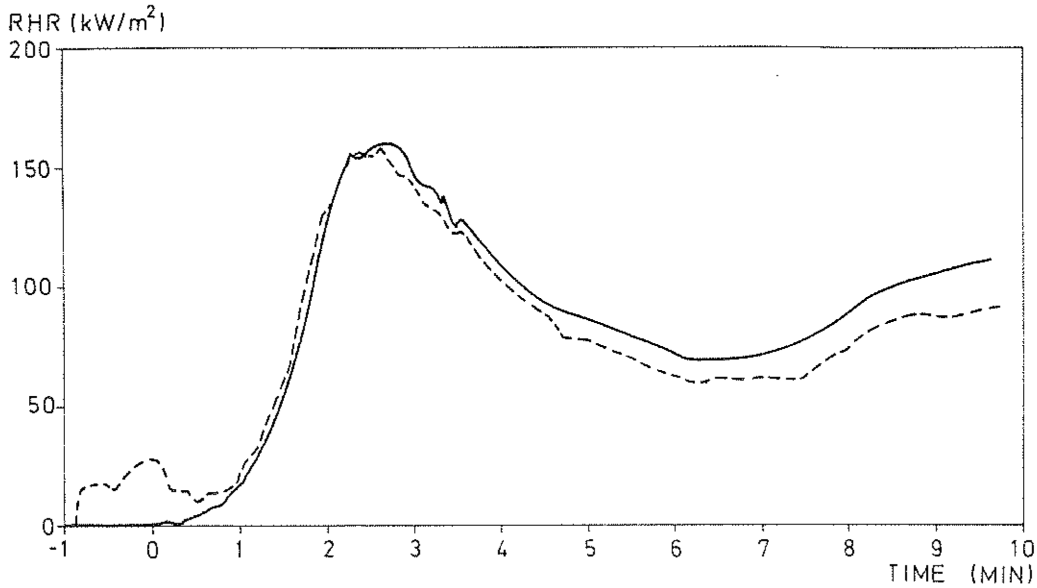


Fig 2 RHR measured with the compensated thermopile and oxygen consumption for particle board with an impinging pilot flame. Air flow 40 l/s

Solid line = oxygen consumption
Dashed line = thermopile

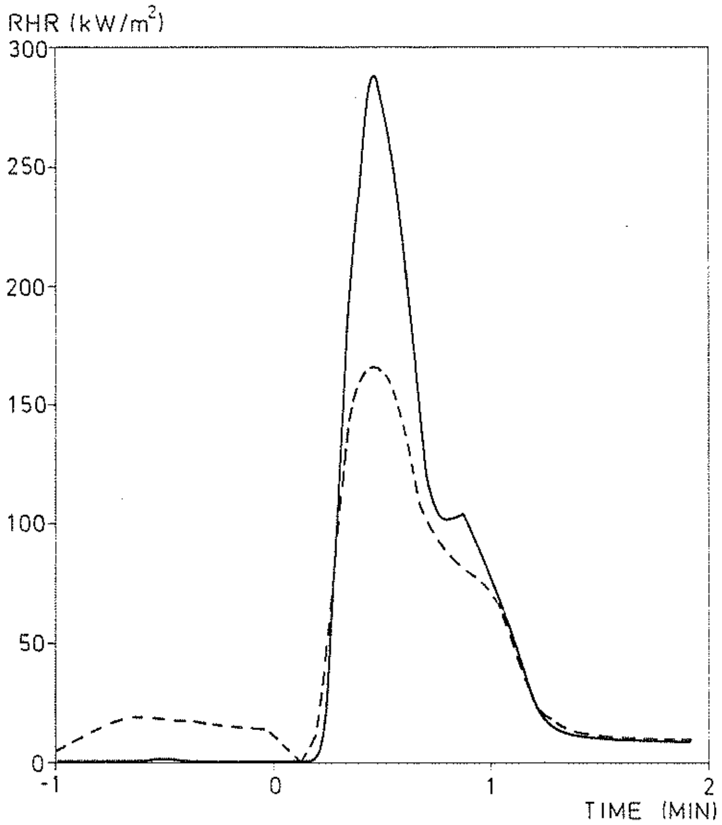


Fig 3 RHR measured with the compensated thermopile and oxygen consumption for a PVC wallcovering on noncombustible backing material with an impinging pilot flame. Air flow 40 l/s
Solid line = oxygen consumption
Dashed line = thermopile
Notice the different time scales between Fig 2 and 3

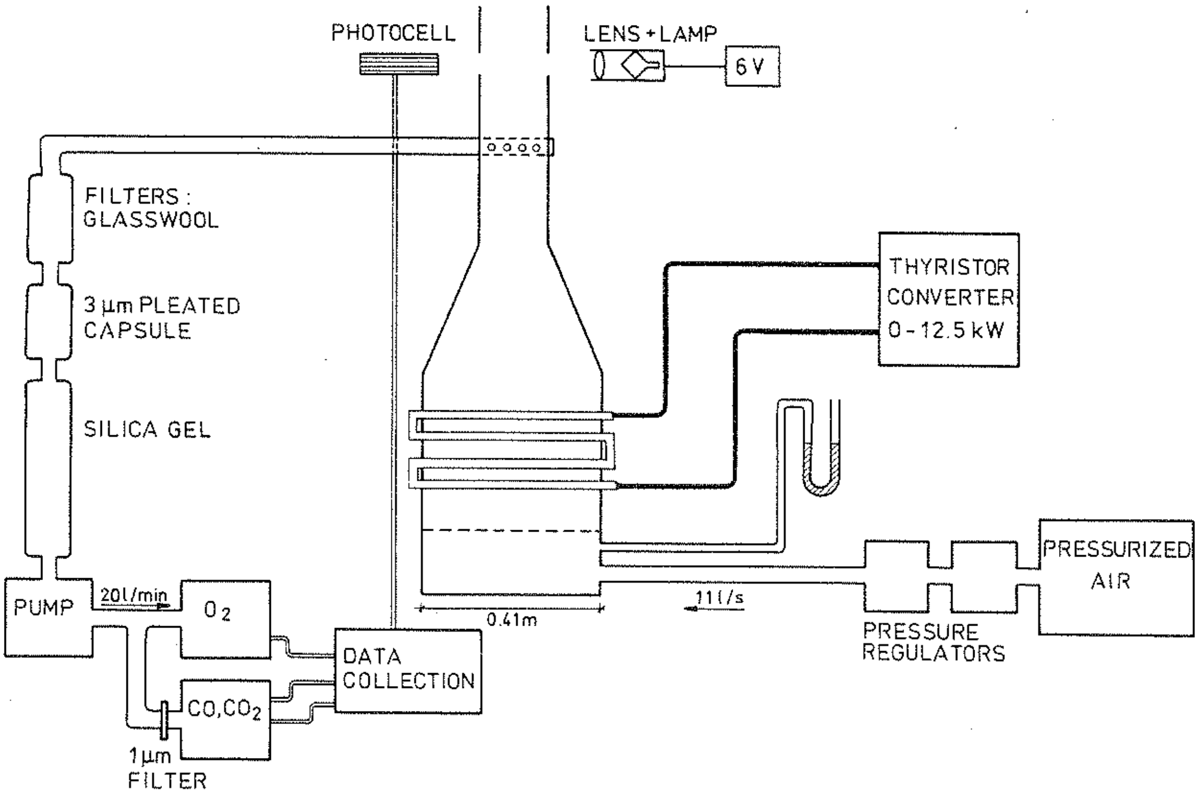


Fig 4 The OSU-apparatus with instrumentation

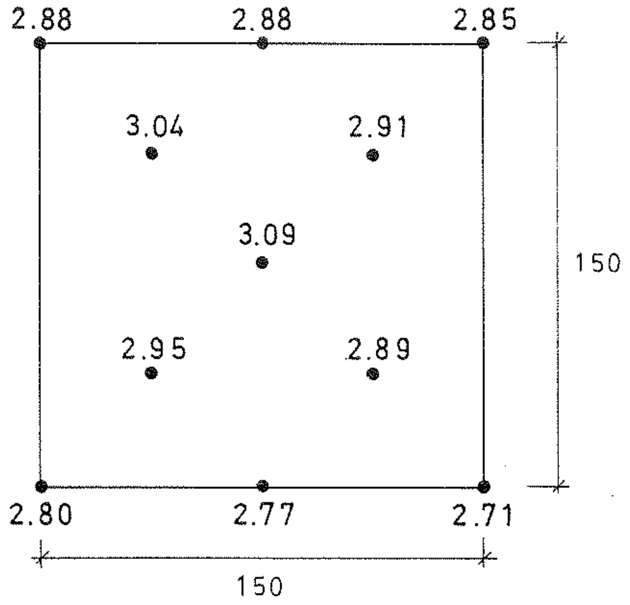


Fig 5 The distribution of radiation over the test specimen area at 3 W/cm^2

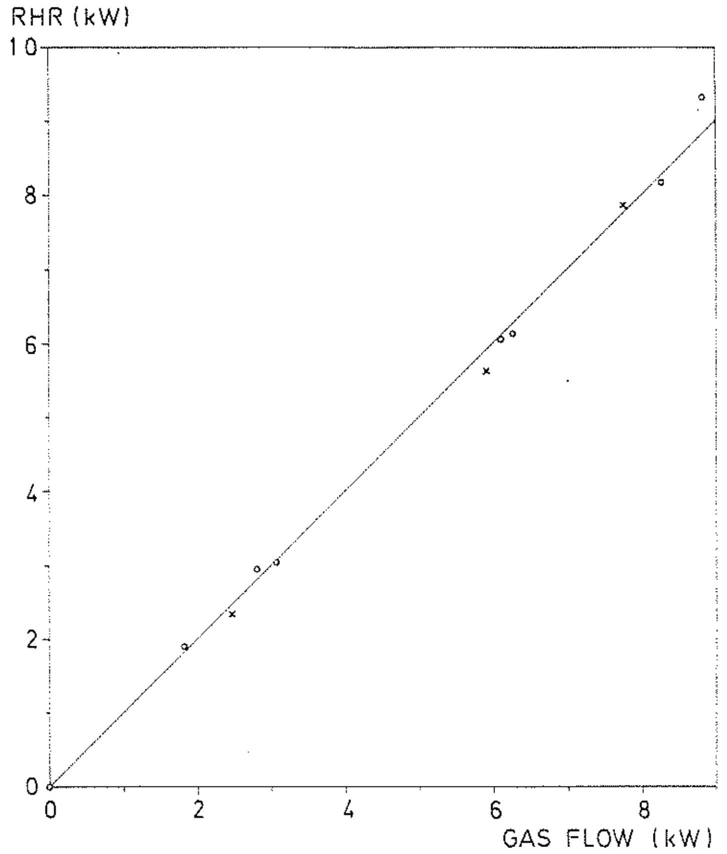


Fig 6 Results from some calibrations at the oxygen consumption measurement system. The time in the figure corresponds to measured RHR being equal to the heat content of the gas
o = propane, x = methane

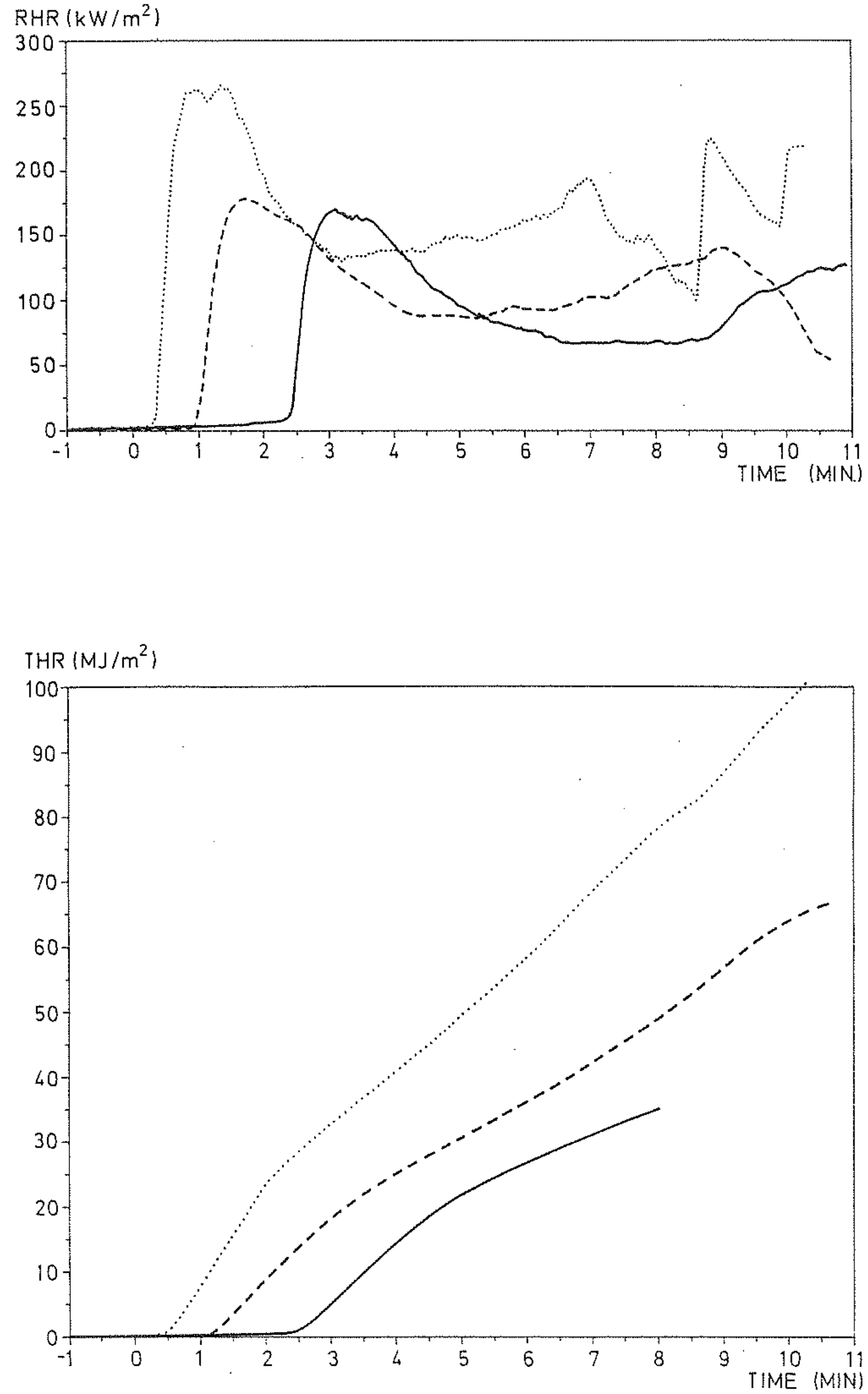
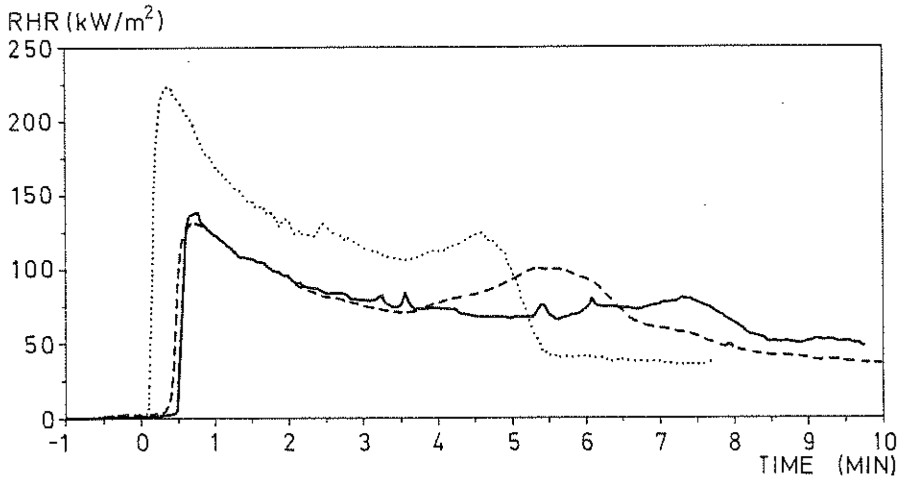


Fig 7 Particle board
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



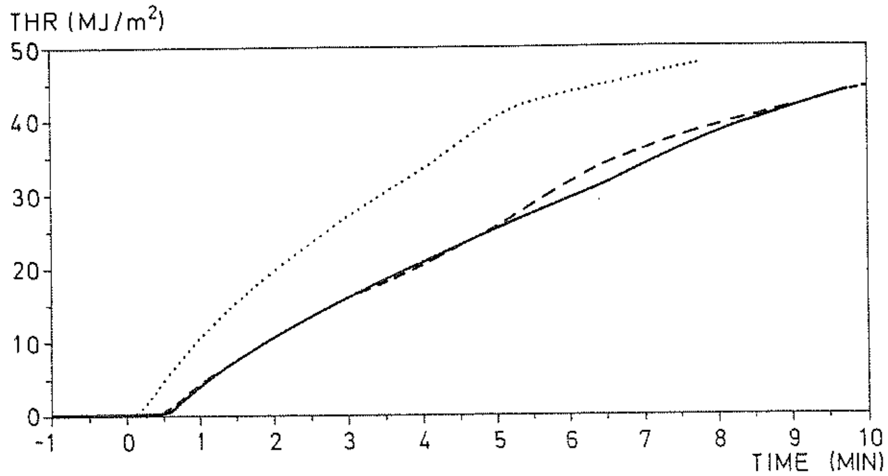
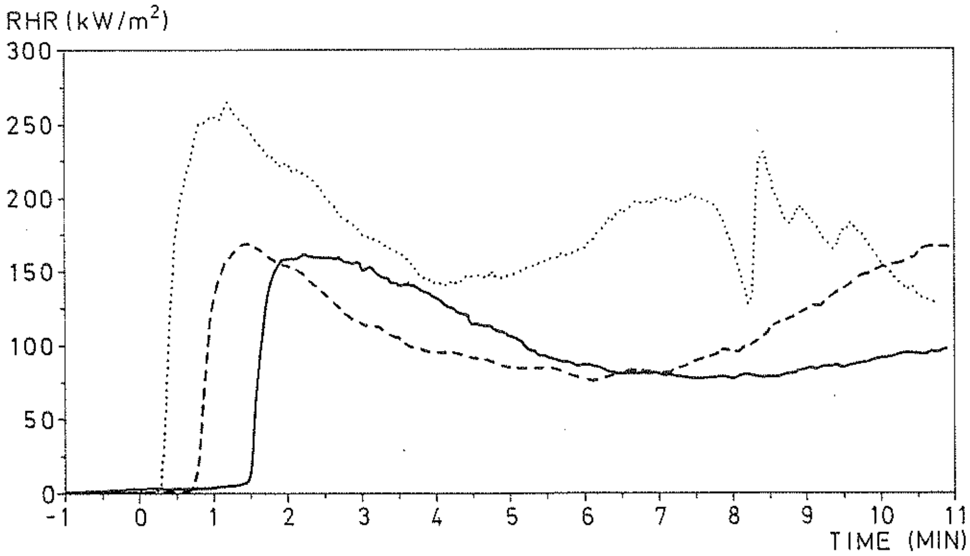


Fig 8 Insulating fibreboard
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



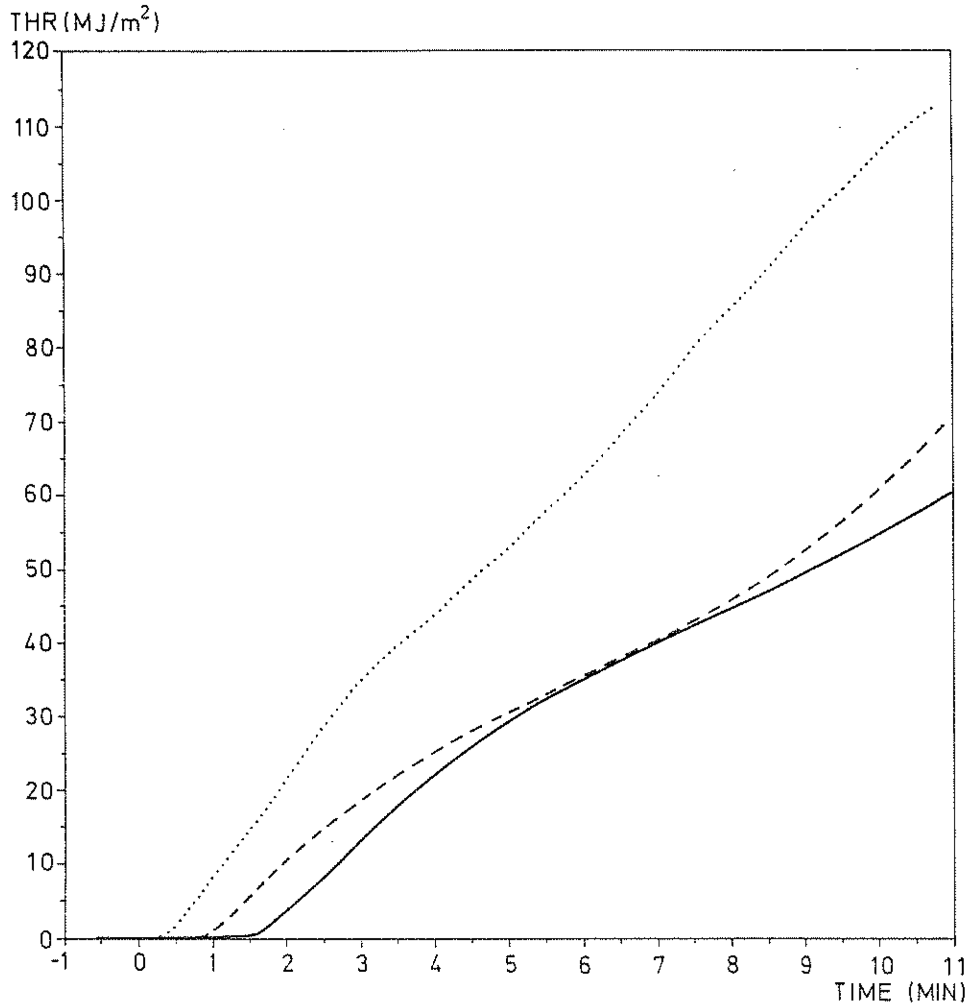
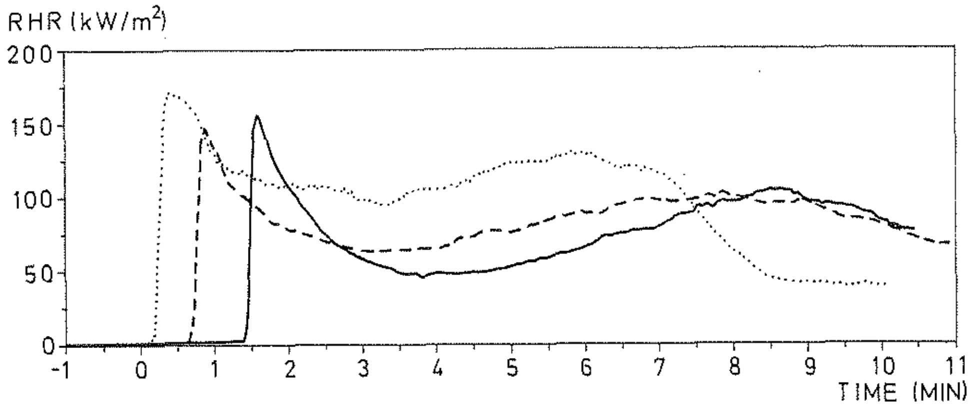


Fig 9 Medium density fibreboard
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



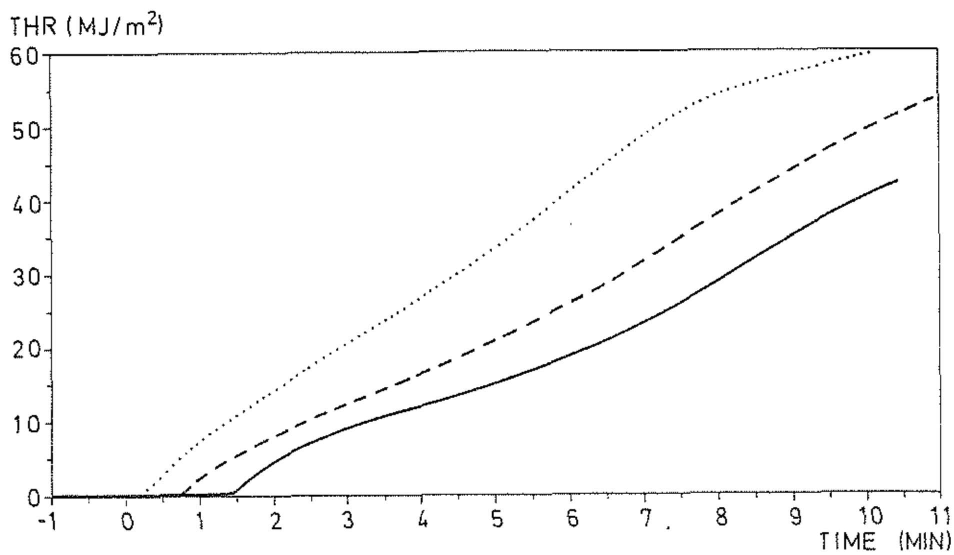
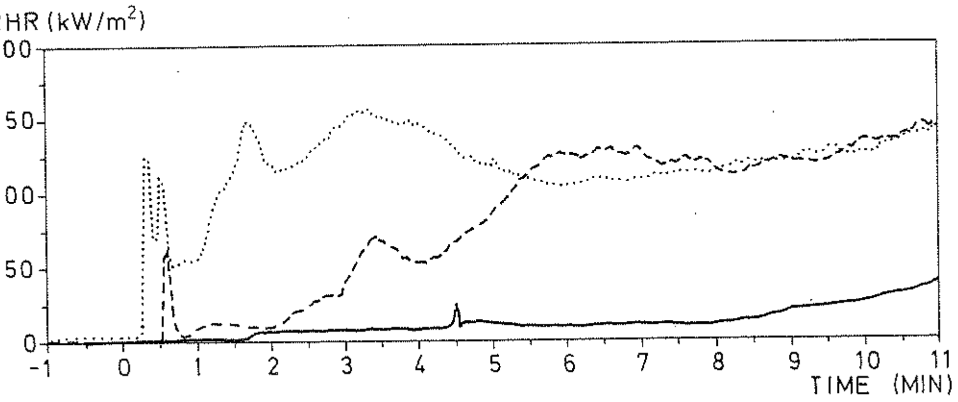


Fig 10 Solid wood (spruce)
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



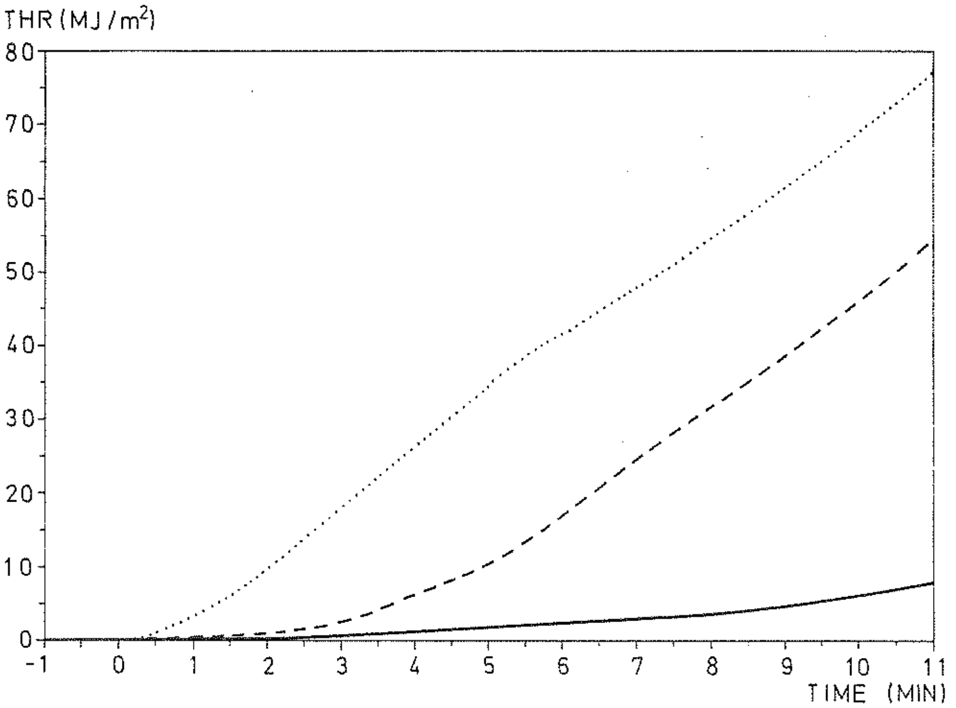
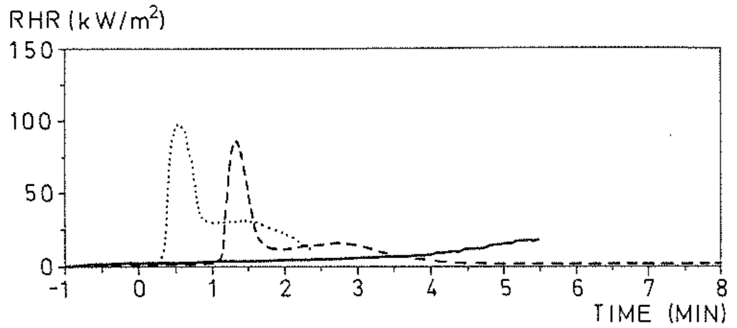


Fig 11 Melamine faced particle board
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



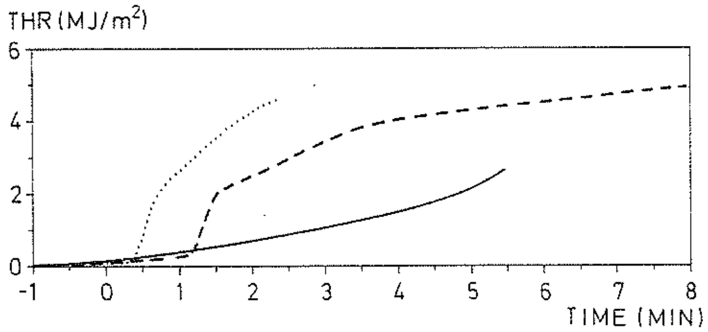
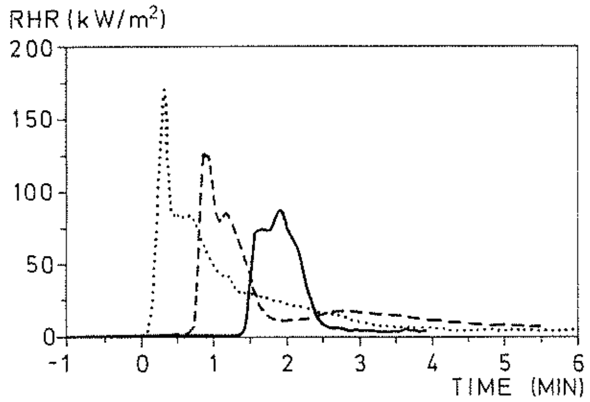


Fig 12 Gypsum plaster board,
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



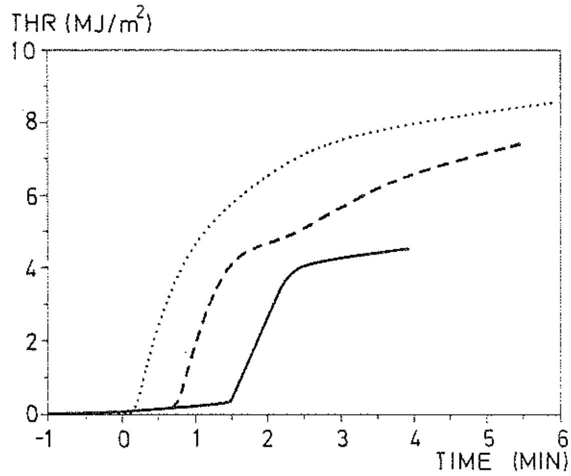
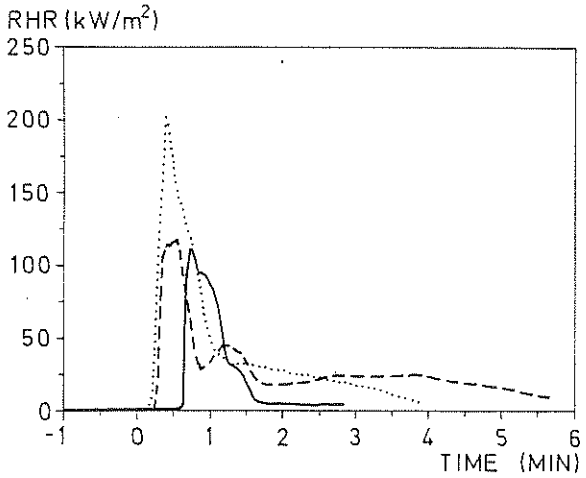


Fig 13 Paper wallcovering on gypsum plaster board
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



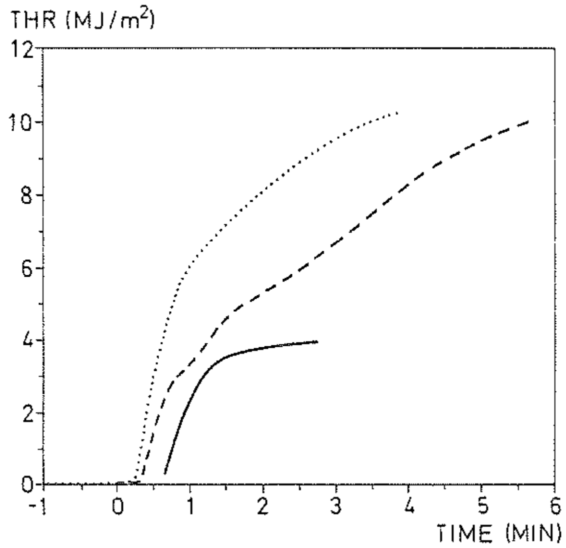
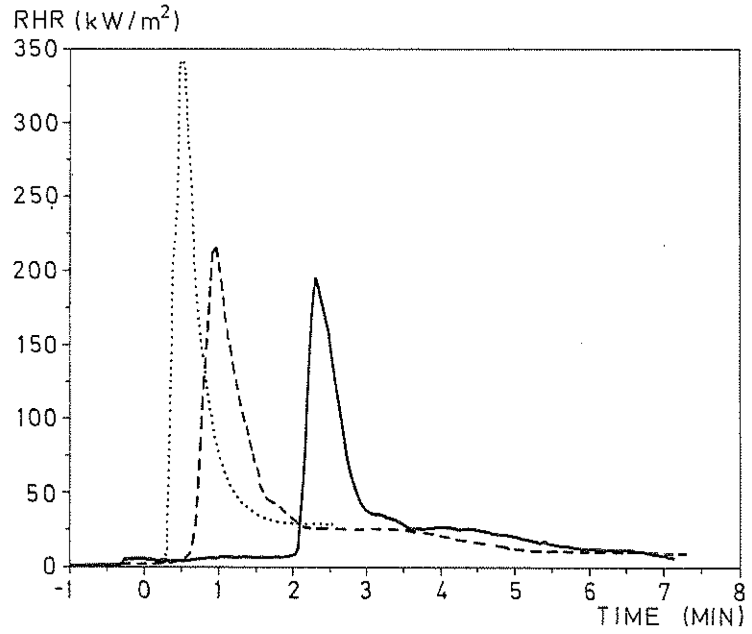


Fig 14 PVC wallcovering on gypsum plaster board
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



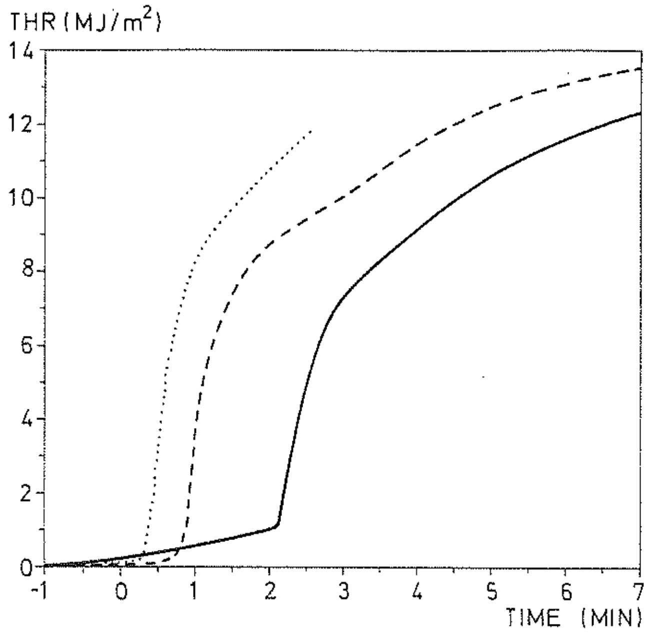
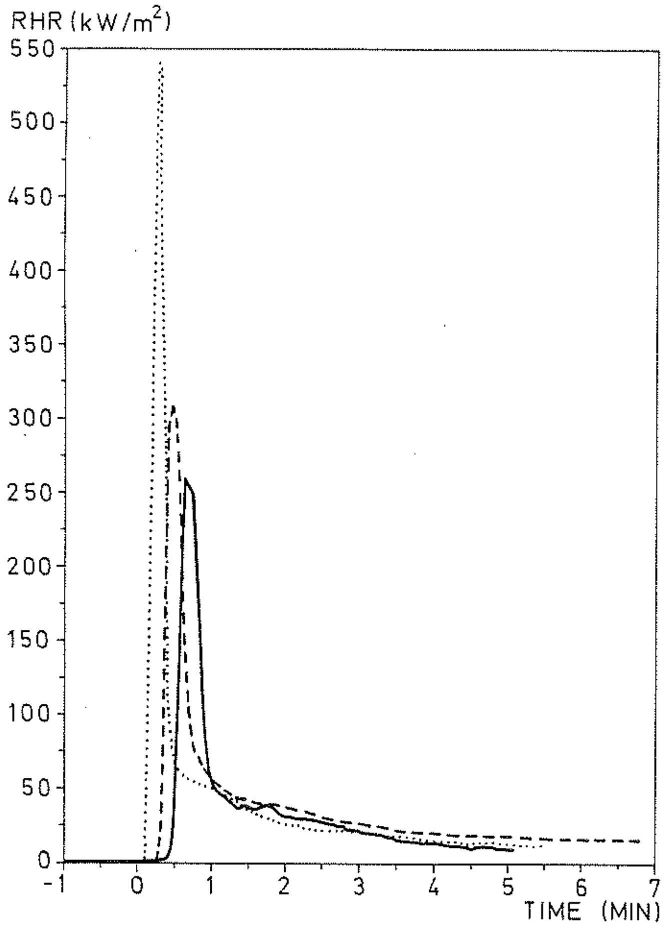


Fig 15 Textile wallcovering on gypsum plaster board
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



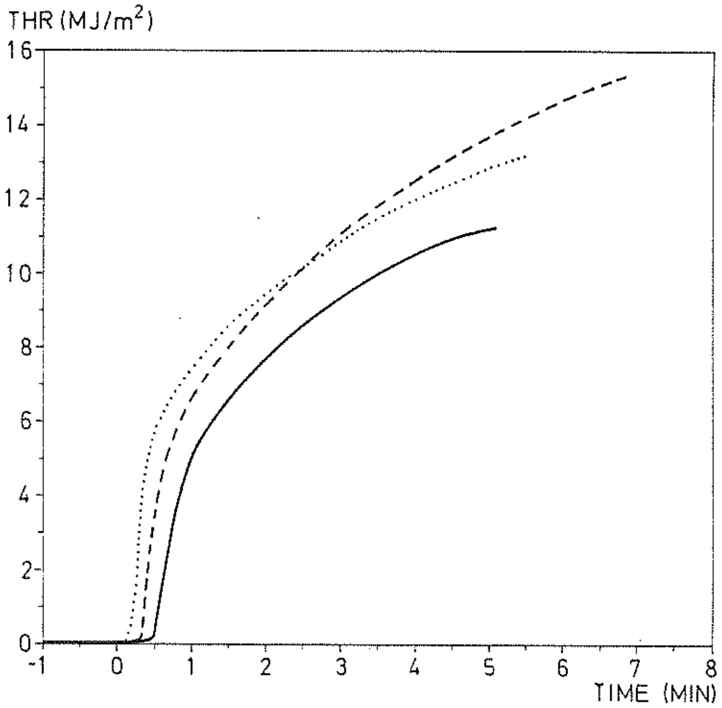
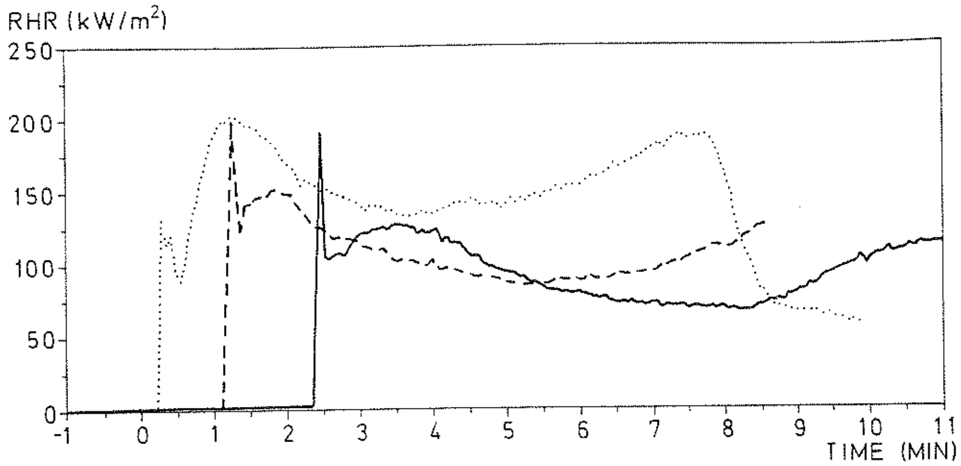


Fig 16 Textile wallcovering on mineral wool
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



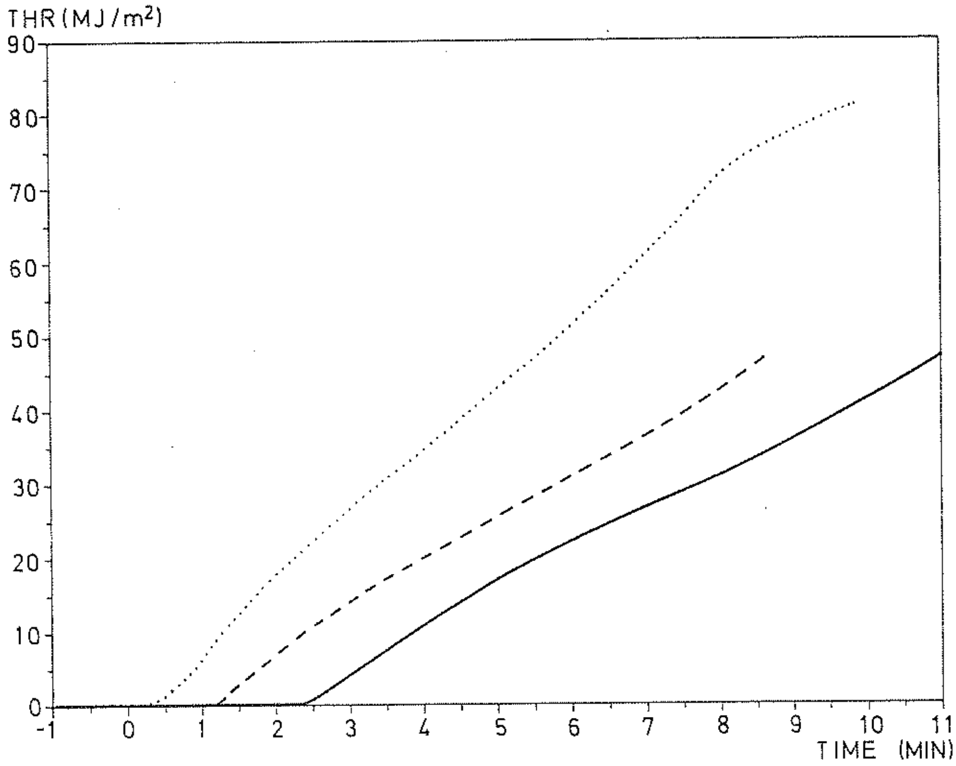
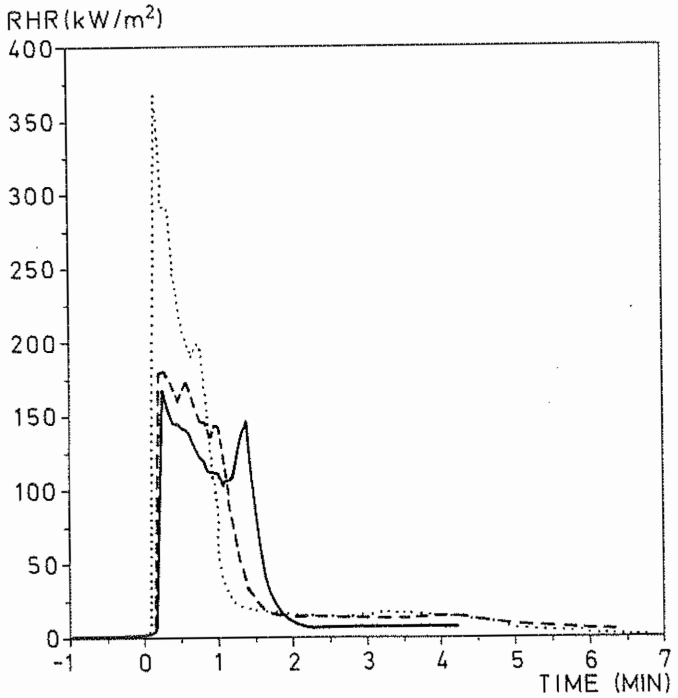


Fig 17 Paper wallcovering on particle board
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



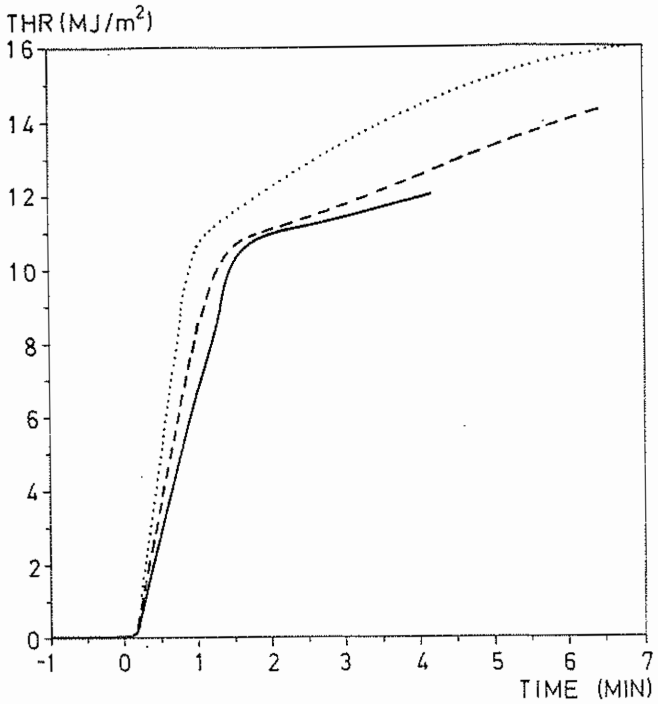
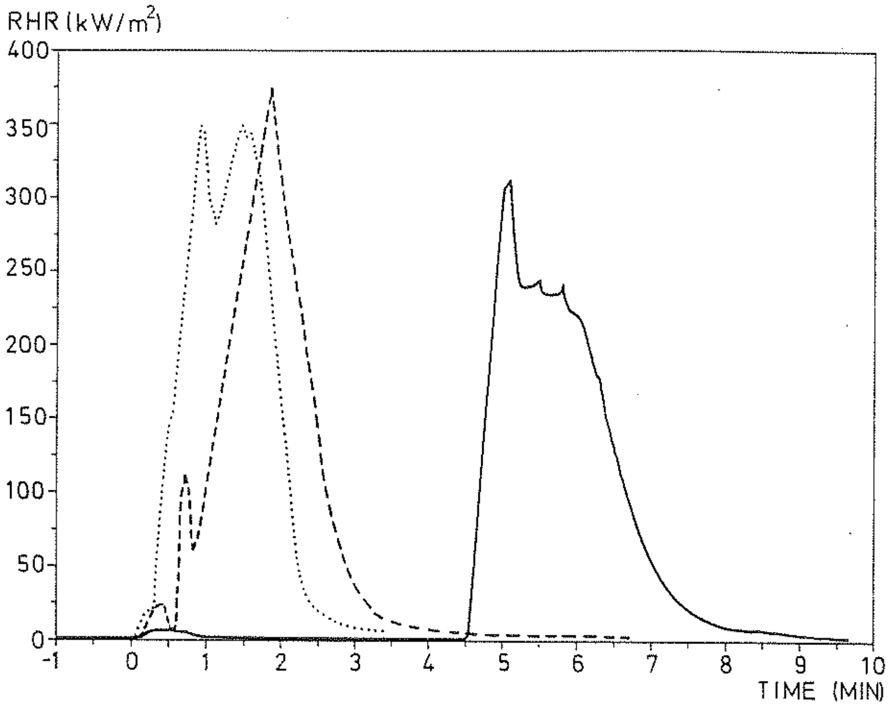


Fig 18 Rigid polyurethane foam
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



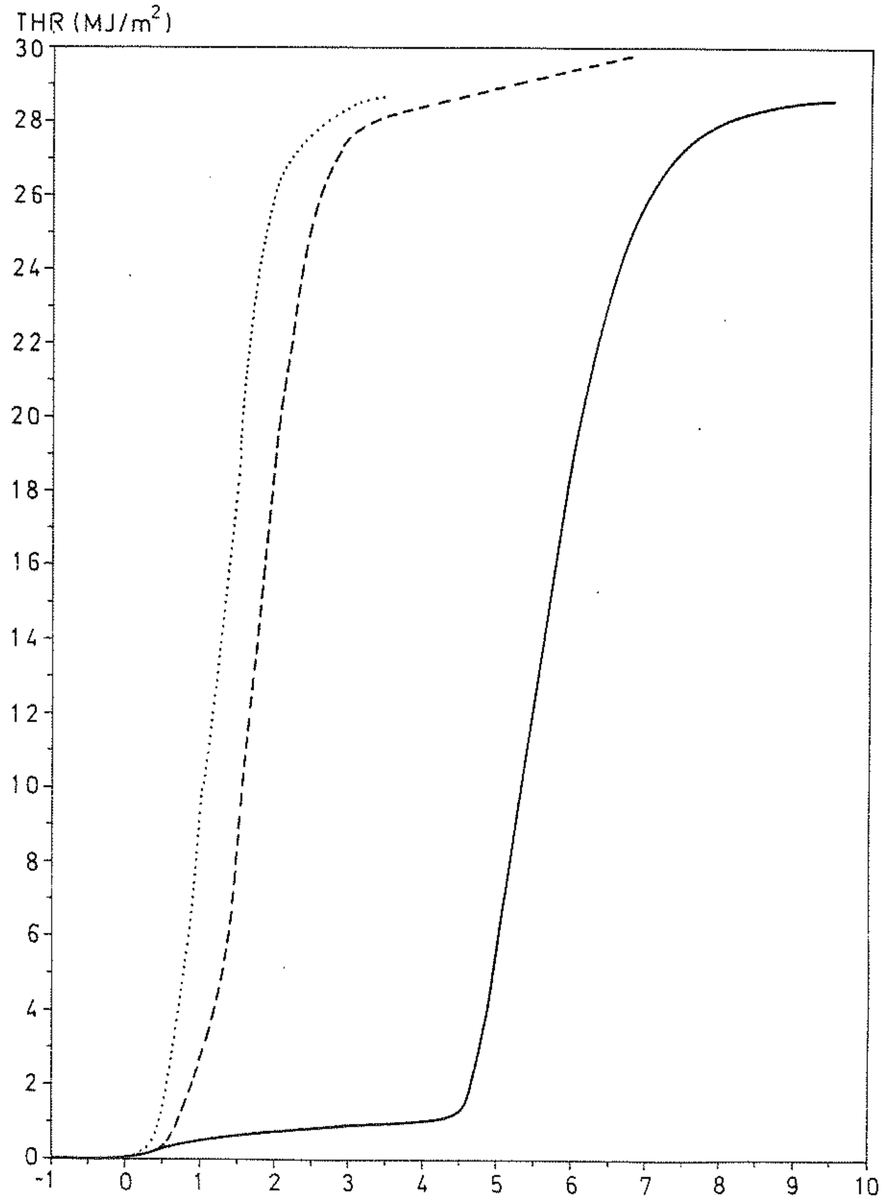
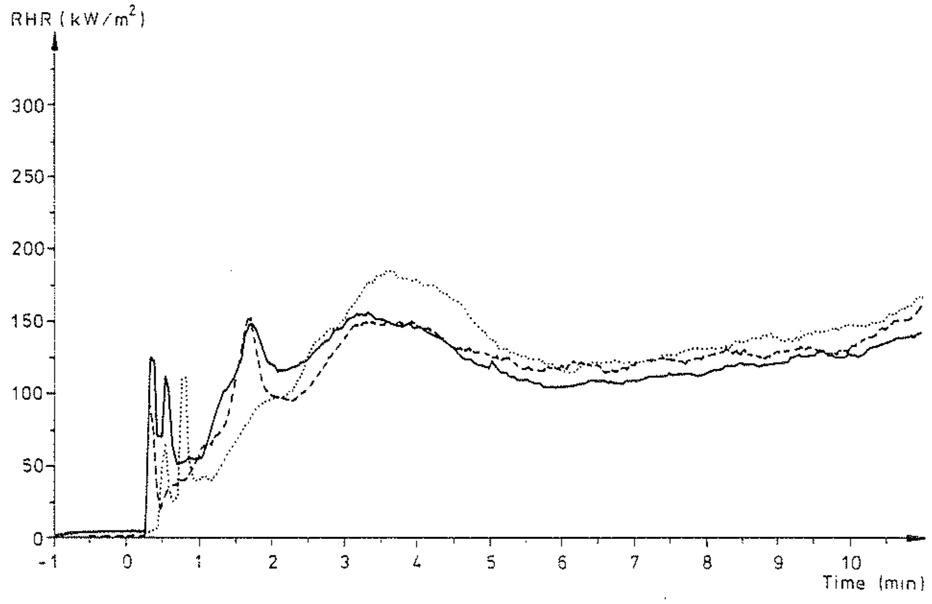


Fig 19 Expanded polystyrene
Solid line = 2 W/cm^2, dashed line = 3 W/cm^2, dotted line = 5 W/cm^2



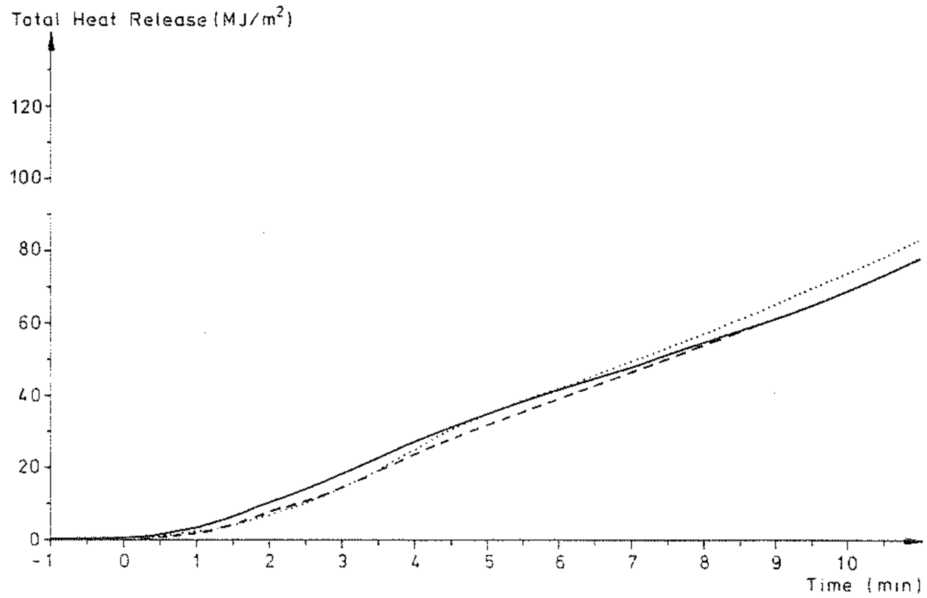
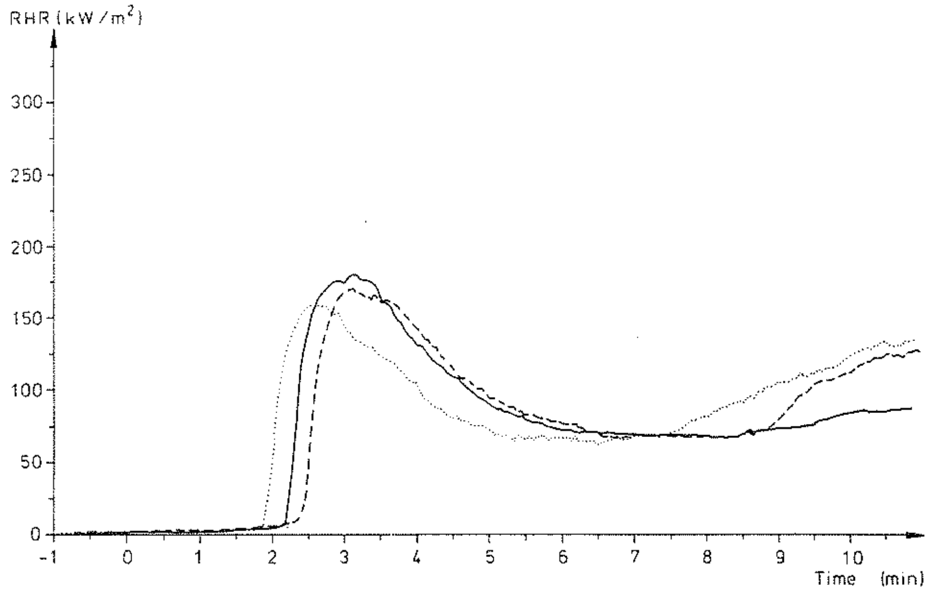


Fig 20 Melamine faced particle board, 5 W/cm^2. Results of triplicate experiments



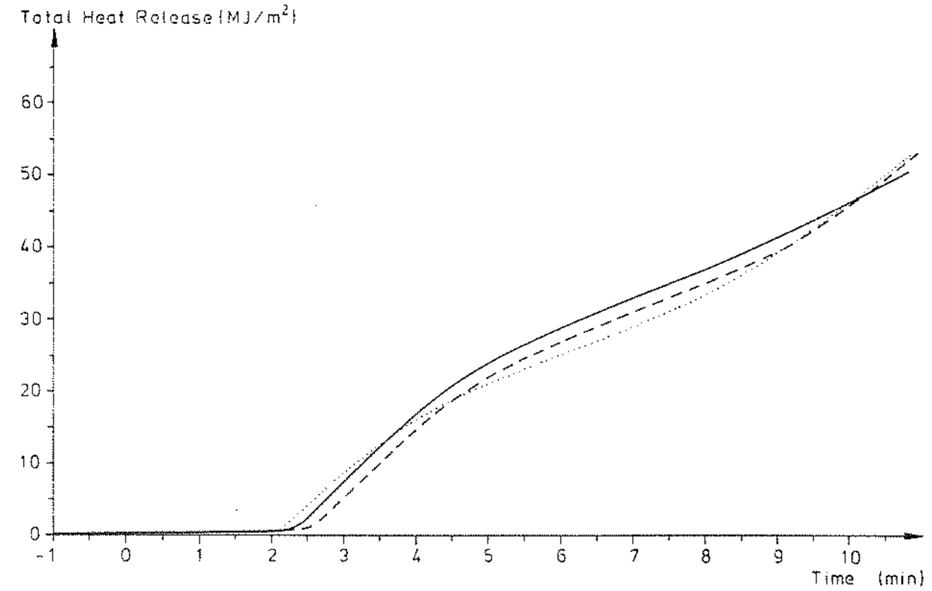


Fig 21 Particle board, 2 W/cm^2. Results of triplicate experiments

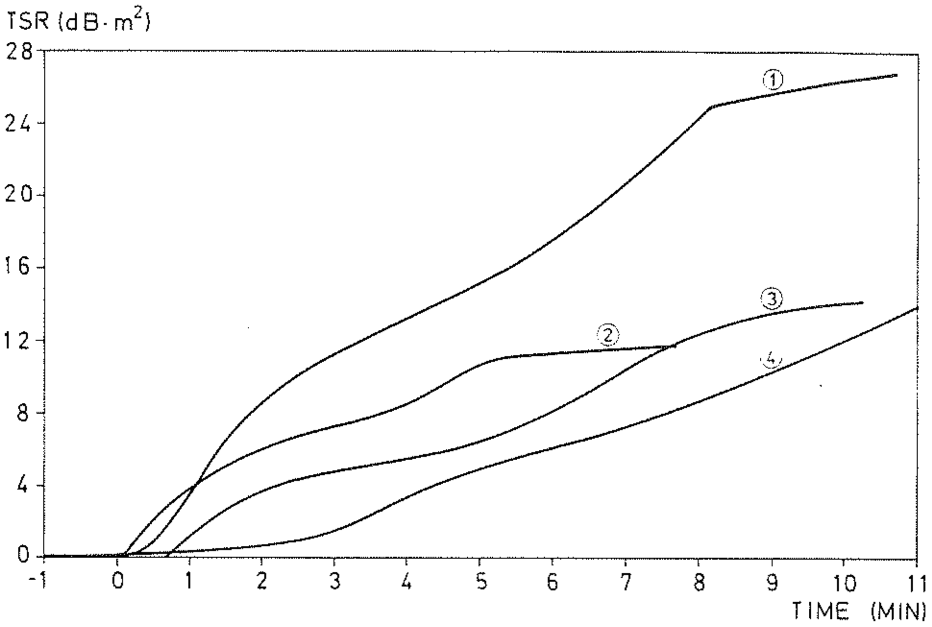


Fig 22 TSR at the exposure level 5 W/cm^2
1. Medium density fibreboard
2. Insulating fibreboard
3. Particle board
4. Melamine faced particle board

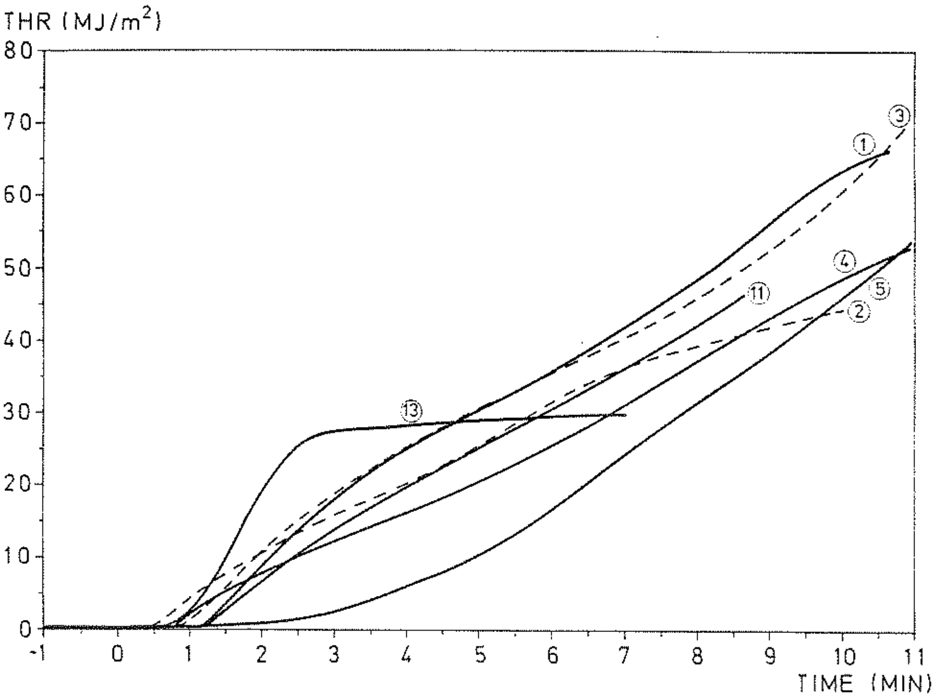


Fig 23 THR of all materials at 3 W/cm^2
1. Particle board
2. Insulating fibreboard
3. Medium density fibreboard
4. Solid wood (spruce)
5. Melamine faced particle board
11. Paper wallcovering on particle board
13. Expanded polystyrene

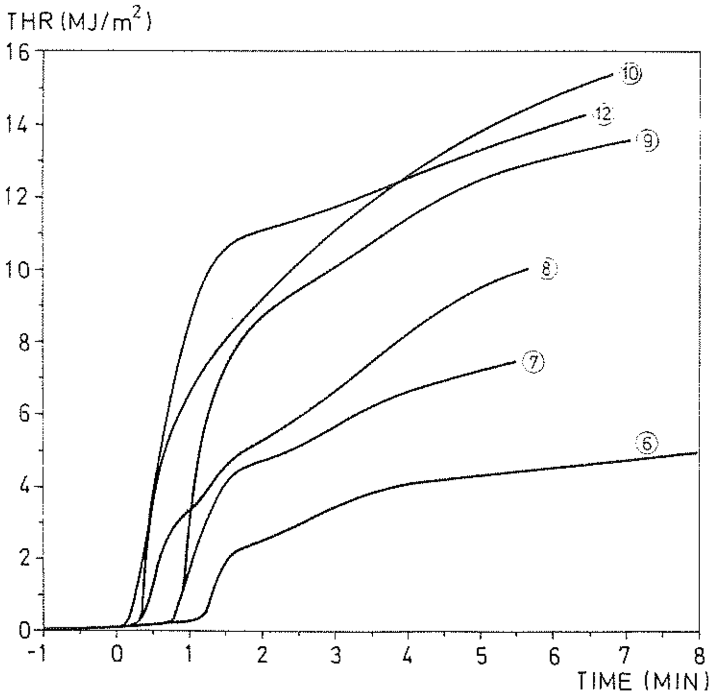


Fig 23 Cont.
6. Gypsum plaster board
7. Paper wallcovering on gypsum plaster board
8. PVC wallcovering on gypsum plaster board
9. Textile wallcovering on gypsum plaster board
10. Textile wallcovering on mineral wool
12. Rigid polyurethane foam