

A Flammability Evaluation of Materials that Pose a Hazard in Nuclear Power Plants

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Abstract

A detailed flammability evaluation of different materials used in the nuclear power industry is presented as an example of how important are physical observation and detailed analysis in the interpretation of test data. The objective is to provide different criteria that could allow the use of standard test data in fire modeling. So far most fire test data has only been used as an element for ranking and selection of materials.

The tests used are the open and closed cup burner tests (ASTM-D-56-87, ASTM-D-1310-86), the lateral ignition and flame spread test (ASTM-E-1321) and the oxygen consumption calorimeter (ASTM-E-1354). The materials evaluated are Lexan and two liquids, Tributyl Phosphate ($C_{12}H_{27}O_4H$) and Tetrapropylene hydrogen ($C_{12}H_{26}$). Lexan is a solid plastic sheet, generally considered as fire resistant. The solid plastic sheet is currently proposed as an alternate to Plexiglas (i.e. Poly-methyl-methacrylate) for use as windows in glove boxes for service and maintenance of radioactive components in nuclear power plants. The two liquids are used in a mixture as extraction agents for radioactive materials. The fraction of each liquid chosen has been selected to optimize the process of extraction of radioactive waste.

The tests indicated that the Lexan sheet will melt long before it can be ignited. When mounted in a vertical position, the plastic material starts to melt and move at much lower heat fluxes than necessary for ignition. As a result it is likely that, when exposed to a fire, the plastic will melt and create a pool under the original position of the sheet before it ignites. Due to the melting behaviour it is unlikely to get upward flame spread over the material, but consequently, downward flame spread is almost instantaneous due to dripping

The two liquids had very different fire properties. However, a mixture of the two liquids had the same fire properties as the most flammable liquid, even when the mixture only contained 30% of this liquid. A fire involving the mixed liquid should therefore be modeled with the properties of the most flammable liquid.

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College Park MD, June 2001

Olof Mångs

<p>While every effort has been made to ensure that the content within this report is true and correct, errors can never be guaranteed against, so the reader should show due care in using any of the content in the report.</p>
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1 INTRODUCTION

1.1 Introduction to the Study

This study is the result of a final year research project in the Bachelor of Science degree program in Fire Safety Engineering at Lund University, Sweden. The project was carried out at the Department of Fire Protection Engineering at the University of Maryland, USA. The supervisor for this project has been Dr. Jose L Torero at the University of Maryland and lecturer Berit Andersson at Lund University.

The project was sponsored by Institute Nationale de Protection et Surete Nucleaire (IPSN) which is the French Nuclear Safety Administration. IPSN provided the material and the economic foundation for the project.

1.2 Background for the Study

Material flammability assessment is an area of increasing complexity mainly due to the development of new technologies and of new-engineered materials. Traditional test methods give some indicators of the relative performance of materials but many times provide little or no information that could be used to predict the behavior of a specific material in the event of a fire. Currently, the information from test methods is used to define selection criteria for materials and cannot be directly incorporated in fire models and thus, cannot be used to estimate the impact that a particular choice of material can have in the potential growth of a fire. The evolution of fire regulations towards performance based engineering analysis and the increasing scientific basis that is being required for fire reconstruction makes necessary a more in depth definition of material flammability, one that relies more on physical properties and empirical observation and less on pre-defined selection criteria.

The nuclear power industry has been chosen, first due to the renewed interest in nuclear energy as an alternative to fossil fuels and second, and most importantly, because of the controlled nature of the environment. The tested materials will be present in areas where ignition sources can be considered very scarce; therefore there is a low probability of a fire. Even in case of a fire, the fuel load present will guarantee a controlled event with little potential to become a large fire. Thus, the rare nature of these fires do not justify the assumption of a given ignition event, which is common to less controlled environments, and which leads to hazard being mostly defined by the heat release rate. Furthermore, the catastrophic potential consequences of a fire in a nuclear power plant demand an accurate definition of the material properties that will lead to failure of the system. These characteristics of usage make this analysis particularly complex, since it requires detailed study of the impact of the material in all elements of the fire growth. This justifies the present approach as necessary for the decision making process involved in the selection of materials and development of the different processes that involve combustible materials.

1.3 Scope

This study serves as an example of a detailed flammability evaluation of different materials used in the nuclear power industry, and how important are physical observation and detailed analysis in the interpretation of test data. The objective is to provide different criteria that could allow the use of standard test data in fire modeling.

So far most fire test data has only been used as an element for ranking and selection of materials.

The flammability of three different materials is evaluated and the information obtained is presented as design criteria within the context of the proposed use. The materials correspond to three different categories: a solid plastic sheet, a liquid generally considered as fire resistant (NFPA-30 Class 3-B, Combustible Liquid), and a flammable liquid.

Different standard tests and better-adapted variants will be used to attempt a comprehensive description of the materials of interest. The test results will be complemented by empirical observation in an attempt to determine the parameters that can be considered critical to failure within the context of the materials use. The tests to be used are the open and closed cup burner tests (ASTM-D-56-87, ASTM-D-1310-86), the lateral ignition and flame spread test (ASTM-E-1321) and the oxygen consumption calorimeter (ASTM-E-1354) [6].

1.4 Background for the Materials

1.4.1 The Lexan Sheet

The solid material is a transparent thermoplastic sheet called Lexan. Lexan is currently proposed as an alternate to Plexiglas (i.e. Poly-methyl-methacrylate) for use as windows in glove boxes for service and maintenance of radioactive components in nuclear power plants.

Lexan and other thermoplastics are widely used in a variety of fields today. Components made of Lexan include: machine guards, vandal street furniture, balcony glazing, and skylights. Low part costs for large production runs and the ability to integrate and form the material has contributed to the popularity. In the IPSN application, Lexan is used as window glazing between control rooms and in glove boxes for service and maintenance of radioactive components.

Thermoplastics are usually inherently flammable. As a result, flame retardants are added to allow thermoplastics in building constructions [1]. Lexan, however, does not contain flame retardant additives. Instead, the thermoplastic is extruded from polycarbonate resin, which gives Lexan better impact resistance and fire performance than many other thermoplastic materials on the market [2].

The base raw material has a Limited Oxygen Index (LOI) of 25 % when tested according to ISO 4589. The LOI is the minimum concentration of oxygen that will support flaming combustion of a material in room temperature [2]. Lexan has also passed the Glow Wire test according to IEC695-2-1 for 850°C. For this test a glowing wire is applied near the lower edge of a vertical sample to see if the sample ignites and if so how fast the flame spreads [2]. Furthermore, according to the manufacturer, Lexan will not ignite when exposed to temperatures under 426°C [2]. A limitation of the two mentioned test methods is that they do not expose the material to any pre-heating before ignition. In a real fire situation, the response of the material to an external heat flux from an adjacent fire is a significant fire resistance parameter.

Since the material is a thermoplastic, heat (e.g. from a fire) will cause melting and, if the external heat flux is large enough, the material will also ignite and burn. Experiments with burning of thermoplastics has demonstrated that ignition of such materials is a very complex process [1]. The key factor, long recognized, is that most thermoplastics change shape as they are subjected to heat. Shape changes are typically accompanied by the movement of hot material, with lowered viscosity, to some new location under the influence of gravity. The melted material may burn both as it moves and at its new location. These time dependant changes in the material make the modeling of the burning process very difficult [3]. The complex behavior of these materials has resulted in a very small number of studies available in the literature [4]. Due to the melting behaviour of the thermoplastics, the repeatability is often consider poor, and no standard bench scale tests can be considered suitable for testing such materials [1].

1.4.2 The Liquids

The two liquids supplied by the IPSN are Tributyl Phosphate ($C_{12}H_{27}O_4H$) and Tetrapropylene hydrogen ($C_{12}H_{26}$). Tributyl Phosphate (TBP). The TBP liquid is not miscible with water and it can emit toxic fumes under fire conditions. Examples of its use are the extraction of rare earths, plutonium and uranium. TBP is also used as fire resistant hydraulic fluid. The liquid is produced from n-butanol and phosphorus oxychloride [5]. The precise composition of Tetrapropylene hydrogen (TPH) is proprietary and therefore unknown. The manufacturer claims that TPH is mostly n-dodecane mixed with different fractions of isomers. The use and application of this liquid compare with those of TBP.

IPSN uses the liquids as extraction agents for radioactive materials. For this purpose the liquids are currently in use in a mixture of 70% TBP and 30% TPH. The fraction of each liquid has been chosen to optimize the process of extraction of radioactive waste

1.5 Standardized Test Methods

Test methods that are commonly used for material flammability assessment can be crudely separated in two different categories. Those that provide a ranking based on a direct observation (i.e. floor radiant panel, upward flame spread test, etc. [6]) and those which rely on a physical principle to rank the materials (i.e. Limiting Oxygen Index, Flash Point, Heat Release Rate, etc. [6]). Within the later group, some test methods provide information that could be incorporated into a fire model.

In the case of liquids, the flash point could be used as the temperature the liquid needs to attain to generate enough fuel to lead to a flame in case there is a hot spot or pilot present. This value could be used as an ignition criterion as well as an indicator of flame spread [6].

In the test standard ASTM-E-1321 the lateral ignition and flame spread test (LIFT) is defined. This test method provides a similar temperature for solid fuels (also defined as a critical heat flux for ignition) as well as other thermal properties that can be linked to the material and could be used to model ignition and opposed flame spread [6]. These properties are mainly the thermal inertia ($k\rho C$) and the flame spread parameter (ϕ). The thermal inertia is the product of the thermal conductivity (k), density (ρ) and specific heat (C) and appears in both the model for ignition and that for flame spread presented in the standard [6]. The flame-spread parameter is a global representation of the energy contributed by the flame to the fire spread process. The heat release rate corresponds to

the total energy delivered by the combustion reaction [6]. Finally, the limiting oxygen index can be used as an extinction criterion under conditions where the oxygen concentration changes are expected [6]. Together and within an adequate model, these parameters provide the necessary elements to model the growth of a fire.

Although the physical significance of these parameters links them directly to processes that can be described through a model, the conditions under which the tests are conducted have been standardized for reproducibility, therefore the quantitative data obtained from the tests corresponds only to these particular conditions. Therefore, this data can only be used for ranking and cannot be directly incorporated in models.

The ASTM standardized test methods for determining material ignition and flame spread properties incorporates numerous disclaimers to this effect [6]. Many studies have attempted to separate the particular experimental conditions of the tests from the relevant material properties [8, 9,12] but this work is far from being completed. A notable exception seems to be the heat release rate, where the presence of a cone shape heater in the standard test allows recreating conditions deemed representative of a real fire, thus the generalized use of data from this standard test in fire models [15,16] are limited.

The accuracy of this type of extrapolation has been questioned, leading to the development of larger scale calorimeters that provide direct estimation of the heat release rate [17]. It is clear that the results obtained from these tests represent an incomplete estimation of behavior of the material under fire conditions that can only be extrapolated by close physical observation of the phenomena occurring during the test.

2 WORK METHODOLOGY AND THEORY

2.1 Methodology

In this section a brief description of the test methods and theoretical approach towards calculating the fire properties described in ASTM-E-1321-standard [6] will be given. In the experimental approach a number of different experiments were conducted in order to find the relevant material parameters to describe the fire behavior of the materials. A preliminary test, similar to the glow wire test, was conducted for the Lexan sheet. Additional tests with external heat flux were then conducted with the cone calorimeter¹ and on the FIST²-apparatus. For the liquids, a variant of ASTM-E-1321-standard was used. Quintiere [7] and Wu et al. [8] provide details on the corresponding methodology. This test methodology includes, heat release tests from the cone calorimeter and flashpoint tests from the cup burner³.

The following material parameters, found in experiments or derived from the theory, are then used to describe the fire behavior of the material: The critical heat flux for ignition, $\dot{q}_{0,ig}''$, the flash point temperature, T_{fl} , the ignition delay time, t_{ig} , the ignition temperature at the surface, T_{ig} , and the thermal heating property, kpc . The fire properties are then compared with corresponding values for other materials with a more comprehensive documentation.

Some of the tests are conducted according to recognized standards [6] and others are developed at the University of Maryland, partly as modifications of existing test methods. The modifications were necessary due to the specific constraints imposed by the materials tested, but were always accompanied by an in-depth analysis of the physics of the process. As a result, it is important to take notice of the specific test method before interpreting the data.

Numerical criteria are established and can describe the hazard imposed by each material. Nevertheless, the implementation of these numerical criteria to fire models, has to be used together with the experimental observations presented.

¹ See section 2.4.2

² See section 2.3.3

³ See section 2.2.

2.2 The Open and Closed Cup Burner Test

The open cup-apparatus is used to determine the flash point¹ and fire point² of liquids having a flash point between -18°C and 165°C and a fire point up to 165°C. The closed cup apparatus is used to determine the flash point of liquids with a flashpoint below 93°C. Both setups consist of a cup (open or closed) containing the liquid. The cup is placed in a water bath that is heated up at a slow, constant rate. A small propane flame of specified size is directed over the cup at regular intervals.

The flash point is taken as the lowest temperature at which the application of the test flame causes the vapor over the liquid to ignite. To determine the fire point, the test is

continued until the application of the test flame causes the specimen to ignite and burn for at least five seconds. The temperature in the liquid is measured with a thermometer.

The open and closed cup burner used in this report is in accordance with the ASTM-D-56-87 and the ASTM-D-1310-86 standard [6].

- ¹ *The lowest temperature, corrected to a pressure of 101.3 kPa, at which application of an ignition source causes the vapor of the specimen to ignite under specified conditions of the test [6].*
² *The lowest temperature at which a specimen sustains burning for a minimum of 5 seconds by the procedure described in the standard [6].*

2.3 Ignition and Flame Spread (according to ASTM-E-1321)

2.3.1 Theory for Ignition

When a material is exposed to an external radiation, the surface temperature will rise and cause the material to pyrolyze (or vaporize if it is a liquid). This occurs at a minimum temperature that is labeled T_{ig} . The fuel begins to gasify leading to an increase of the fuel concentration in the gas. Once the fuel/air mixture reaches a minimum concentration that will sustain a flame, the pilot induces ignition of the mixture. This event is termed a flash. If the flash feeds back enough energy to sustain fuel evaporation a self-sustained flame will occur followed by flame spread over the surface. For solids the ignition temperature will be equivalent to the flash point.

Quintiere [7] first describes the theoretical developments that support the above-described phenomena. The theory relates to a vertical sample and is the basis of the ASTM-E-1321 standard [6]. Wu et al. [8] later modified the standard for use with liquids in a horizontal configuration. Only a brief summary of the most important steps in the analysis will be presented here, for details the reader should consult references [7, 8].

For this test method the ignition process is treated as the inert heating of a semi-infinite solid. Heating is induced by a constant external heat flux. Heat losses at the surface are included through a convective and radiative heat transfer coefficients that account for surface re-radiation.

$$\dot{q}_s''(0, t) = \dot{q}_i'' - h_c(T(0, t) - T_\infty) - h_r(T(0, t) - T_\infty) = a\dot{q}_i'' - h_T(T(0, t) - T_\infty) \quad (1)$$

Where \dot{q}_s'' is the net heat flux at the surface of the solid fuel sample, \dot{q}_i'' the imposed external heat flux, $T(0, t)$, is the surface temperature at time t . h_c is the convective heat transfer coefficient, h_r is the radiative heat transfer coefficient, and T_∞ is the ambient temperature. The total heat transfer coefficient, h_T is equal to the sum of the convective heat transfer, h_c , and the radiative heat transfer coefficient h_r . The absorptivity of the fuel is considered to be unity [8].

Solution to the one-dimensional energy equation leads to an expression for the time evolution of the surface temperature. This expression is simplified by means of a Taylor series expansion to eliminate all implicit terms leading to the following simple equation for the ignition delay time, t_{ig} .

$$\frac{1}{\sqrt{t_{ig}}} = \frac{2}{\sqrt{\pi}} \frac{1}{\sqrt{k\rho c}} \frac{\dot{q}_i''}{(T_{ig} - T_{\infty})} \quad (2)$$

Where “k is the thermal conductivity, ρ the density and C the specific heat of the solid.

Because of the limited range of the Taylor series expansion the above expression is only valid if the imposed heat flux, (\dot{q}_i''), is much larger than the surface heat losses, (\dot{q}_s'').

For weaker external heat-fluxes a different expansion is necessary leading to the following expression for the ignition delay time

$$\frac{1}{\sqrt{t_{ig}}} = \frac{\sqrt{\pi} h_T}{\sqrt{k\rho c}} \left[1 - \frac{h_T (T_{ig} - T_{\infty})}{\dot{q}_i''} \right] \quad (3)$$

The use of the appropriate simplified solution will allow the evaluation of the ignition delay time, t_{ig} , over the entire domain of imposed incident heat fluxes.

If the external heat flux is equal to the surface heat losses equation (3) shows that the ignition delay time tends to infinity. This means that the material will attain thermal equilibrium with the environment at a temperature that will not allow for gasification of the fuel. This imposed heat flux is termed the critical heat flux for ignition and is consequently described by equation (4)

$$\dot{q}_{0,ig}'' = h_T (T_{ig} - T_{\infty}) \quad (4)$$

To use this theoretical approach for material flammability evaluation, tests have to be conducted for different external heat-fluxes and the ignition delay times recorded. As the heat-flux is reduced the ignition delay time increases (equation (2)) until ignition is not achieved. The maximum heat flux that does not lead to ignition is the critical heat flux. The total heat transfer coefficient can be empirically determined using an inert material. Substituting h_T in equation (4), the ignition temperature, T_{ig} , can be extracted. With the ignition temperature, the ignition delay time data can be fitted to equation (2) and the “thermal inertia”, which corresponds to the products of $k\rho c$, can be calculated. Both ignition temperature (or critical heat flux for ignition) and thermal inertia are deemed as the material properties controlling ignition.

2.3.2 Theory for Flame Spread

Flame-spread takes place once the fuel is ignited and the flame spreads over the surface of the solid fuel under a decaying external heat flux. Under these conditions, the solid fuel is heated by the external radiant flux and the heat flux from the flame, and, when an element of the solid surface attains the temperature necessary for ignition, T_{ig} , the flame ignites the flammable mixture formed. The flame will then propagate forward through the mixture. Thus, the flame-spread process can be viewed as a continuous succession of piloted ignitions, where the flame acts both as a contributing heat source and a pilot. If the external radiant flux varies along the fuel surface, the flame-spread rate will be a function of this parameter. The theoretical analysis below was developed by Quintiere [7], and gives the following expression for the flame spread rate, V_f :

$$V_f = \frac{\Phi}{[\dot{q}_{0,ig}'' - \dot{q}_i''(x_f)]^2} \quad (5)$$

Where x_f is the location of the flame tip and Φ is defined as:

$$\Phi = \left[\frac{2}{\sqrt{\pi}} \dot{q}_f'' \sqrt{\frac{\delta_f}{t_c}} \right]^2 \quad (6)$$

Φ is considered a fire spread material property, but in reality is a combination of all the different parameters that are difficult to calculate independently, \dot{q}_f'' is the heat flux from the flame, δ_f the characteristic length scale through which it is delivered and t_c the characteristic heating time for the solid.

The test measures the flame-spread velocity for different heat-fluxes therefore a fit of the data to equation (5) permits the empirical determination of Φ .

For the liquids, the protocols need to be modified because thermal equilibrium cannot be attained since the fuel will be modified throughout the heating process. Equation (6) needs to be re-written since a direct relationship between the external heat flux and the surface temperature cannot be obtained. The flame-spread velocity is thus given by:

$$\frac{1}{\sqrt{V_f}} = \frac{\sqrt{k\rho c}}{a\sqrt{\Phi}} (T_{ig} - T_{S,i}) \quad (7)$$

Where $T_{S,i}$ is the surface temperature at the arrival of the flame front. $T_{S,i}$ can be determined experimentally.

2.3.3 The Forced Ignition and Spread Test (FIST)

The FIST apparatus was originally designed to utilize the theory of the LIFT (Lateral Ignition and Flame Spread Test) apparatus as defined in the standard ASTM-E-1321 standard [6]. The LIFT, developed by Quintiere, is capable of providing important material properties, including the critical heat flux for ignition, ignition delay time, ignition temperature, and flame-spread rates. The LIFT subjects a material to an external heat flux and then either a piloted ignition test or a flame-spread test can be conducted.

The FIST was developed at the University of Maryland and was originally designed for NASA's Spacecraft Fire Safety Facility, and can be used to implement a forced flow and thus can be used for Micro gravity testing. The attempt to provide a complimentary test method for micro-gravity has been a research project continuing for several years. The first step in the research was provided by Long [11]. The LIFT apparatus was scaled down ensuring that the LIFT protocol could be followed using different length scales.

The scaling down of the LIFT apparatus was done by using different size samples in the LIFT apparatus, to characterize the effect of length on piloted ignition as well as flame spread. After addressing the effect of length, the FIST, was developed to address material flammability at a reduced scale that could utilize a forced slide velocity to

simulate space conditions. Tests were also conducted with the FIST only for piloted ignition to compare the results with those of the LIFT.

Since there is no standard developed for the FIST yet, the apparatus is introduced below. Figure 1 depicts a frontal view of the apparatus, showing the heating panels in the middle. Figure 2 is a schematic of the FIST apparatus. The FIST apparatus consists of a sample holder, a radiant heater, an igniter, and a data acquisition system.

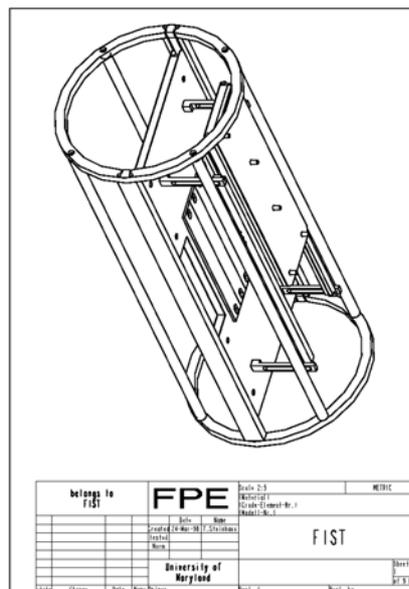


Figure 1: Frontal picture of the FIST.

Figure 2: Schematic figure of the FIST.

The radiation source consists of chrome steel sheet electric strip heaters, wired in pairs in series. Each heater is 203 mm by 25 mm and is powered by a 120 VAC source. The heat release output is fully adjustable for each panel, allowing the heat flux to decay in the vertical direction. The heating panels are capable of heating to about 800°C and can produce a maximum heat flux of approximately 40 kW/m².

The sample holder is a ceramic frame with a 40 mm by 143 mm hole to fit the sample. The sample holder is attached to an aluminum frame with a centimeter scale on it to help record the position of the flame.

The igniter consists of an Iron-Chromium-Aluminum high temperature heating alloy wire. The wire is 0.32 mm in diameter and is heated by a transformer that enables variation of the heat output of the wire. The igniter is placed 10 mm above the top edge of the sample.

The data acquisition system uses LABTech Software and an IOTech TempBook 66 data acquisition board. The software controls the tests and records the data. The heater output temperature is adjusted through the LABTech software.

2.3.4 Horizontal Ignition Test

To study the time to ignition and to find the critical heat flux (necessary for ignition) of the liquids, an apparatus described by Wu et al. [9] was used. This apparatus was developed as an attempt to mimic the LIFT, but had to be oriented horizontally because of the liquid nature of the fuel. The theory for ignition will then compare to the theory described for the LIFT in section 2.3.1. The setup is shown in Figure 3 and consists of a sample holder in form of a steel tray, a cone heater, a pilot flame, and an electric fan.

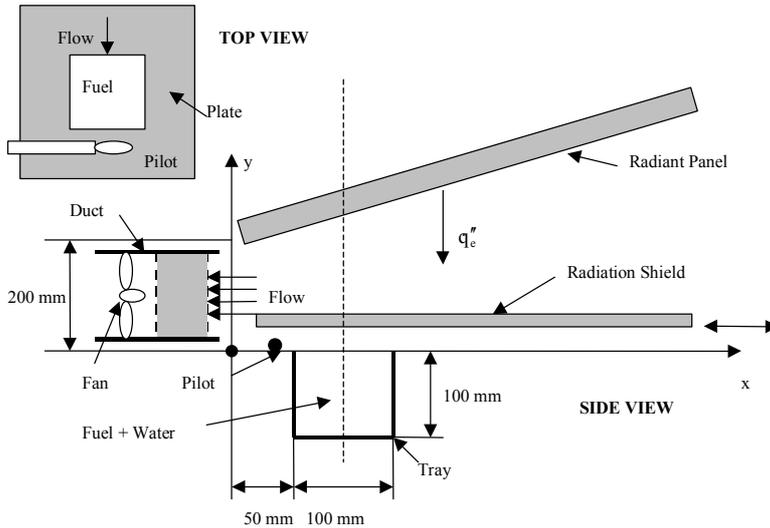


Figure 3: Schematic of the modified LIFT apparatus in the horizontal configuration.

The cone heater is placed over the sample tray so that the radiation cone is parallel to the surface of the specimen. The heat flux level at the surface can be adjusted by changing the distance to the cone.

An electric fan is used to create a flow parallel to the surface. The fan is attached to a duct which contains an 80 mm thick bed of packed steel wool. The steel wool homogenizes the flow creating a flow of approximately 0.1 m/s over the tray. By inducing the flow over the surface, a well-defined boundary layer is established. The robust flow structure over the fuel surface can then be considered independent of the environment.

The sample holder and the tray are created from 1.2 mm thick stainless steel. The tray is 100 mm square with a dept of 100 mm. Additionally a 250 x 250 mm aluminum plate is placed around the tray to simulate a floor around the liquid pool.

A propane diffusion flame is used to get a piloted ignition of the sample. The flame is established on a 3.5 mm steel nozzle. The flame is adjusted to a height of 20 mm and is positioned 10 mm above the fuel surface and 10 mm from the rim of the ignition tray. The position of the flame is according to recommendations by Wu et al. [9]. By positioning the flame outside the rim of the tray, the heat feedback to the fuel surface is minimised. This issue has been addressed previously by Glassman et. al. [12], who showed that heat feedback from the pilot flame can significantly alter ignition and spread.

2.4 Heat release Rate

2.4.1 Theory for the Heat release Rate

The rate of heat release is one of the most important variables when determining the hazard from a fire. The heat release calculated in the cone calorimeter is the total heat release, as a function of time per unit area. The calculations are based on the concept of oxygen consumption calorimetry and the well recognised assumption that approximately 13.1 MJ/kg of heat is released per 1 kg oxygen consumed [6]. The heat release rate can be calculated from the following equation:

$$\dot{Q}(t) = \left(\frac{\Delta H_c}{r_o} \right) (1.10) C \sqrt{\frac{\Delta P}{T_e}} \frac{(X_{O_2}^0 - X_{O_2}(t))}{1.105 - 1.5X_{O_2}(t)} \quad (8)$$

The value for $\frac{\Delta H_c}{r_o}$, where r_o is the stoichiometric oxygen fuel mass ratio, is set to be

equal to 13.1 MJ/kg. The mass flow, \dot{m} , from the fire is $\sqrt{\frac{\Delta P}{T_e}}$. ΔP is the pressure differential at the orifice meter and T_e is the absolute temperature of the gas at the orifice meter. X_{O_2} is the oxygen analyzer reading and $X_{O_2}^0$ is the initial value of the oxygen analyzer reading. The heat release rate per area unit can then be found by solving:

$$\dot{q}''(t) = \frac{\dot{Q}(t)}{A_s} \quad (9)$$

where A_s is the area of the sample.

The total heat release during the combustion can then be summarized as follows:

$$q'' = \sum \dot{q}''(t) \Delta t \quad (10)$$

2.4.2 The Cone Calorimeter

The cone calorimeter was mainly developed to measure bench scale heat release rate for different materials. The test method can also be used to determine ignitability, mass loss rates, and effective heat of combustion. The rate of heat release is determined by the oxygen concentration and the flow rate of the exhaust. The heat release rate is then calculated using the equations above.

Specimen with a size of 100mm by 100 mm are burned in ambient conditions, while being subjected to predetermined external heat flux between 0 kW/m² and 75 kW/m². Ignition may occur either with or without a spark ignition.

The cone calorimeter used in this report is in conformance with ASTM-E 1354 [6] and the test procedure also follows the procedure outlined in the standard. The test specimen were first weighed and then placed in the sample holder. The holder was then placed on the load cell under the cone heater. A number of tests were performed with different

heat flux levels. This was done to find the critical heat flux, and if ignition occurred, the heat release rate of the burning sample. All tests were performed with a spark ignition.

3 EXPERIMENTAL APPROACH

3.1 Test results for the Lexan Sheet

3.1.1 Simple Ignition and Upward Spread Test

The first test performed was a simple ignition and flame spread test similar to the glow wire test according to ICE 695-2-1, a standard the Lexan sheet fulfills.

A 40 by 143 mm slab of the Lexan was arranged according to Figure 4. A glowing wire was placed under the slab and held there for 5 minutes. No ignition was observed during that test.

To provide a larger and more intense heat source, a propane burner was used to try to ignite the sample. The burner flame was applied for about five seconds to the lower part

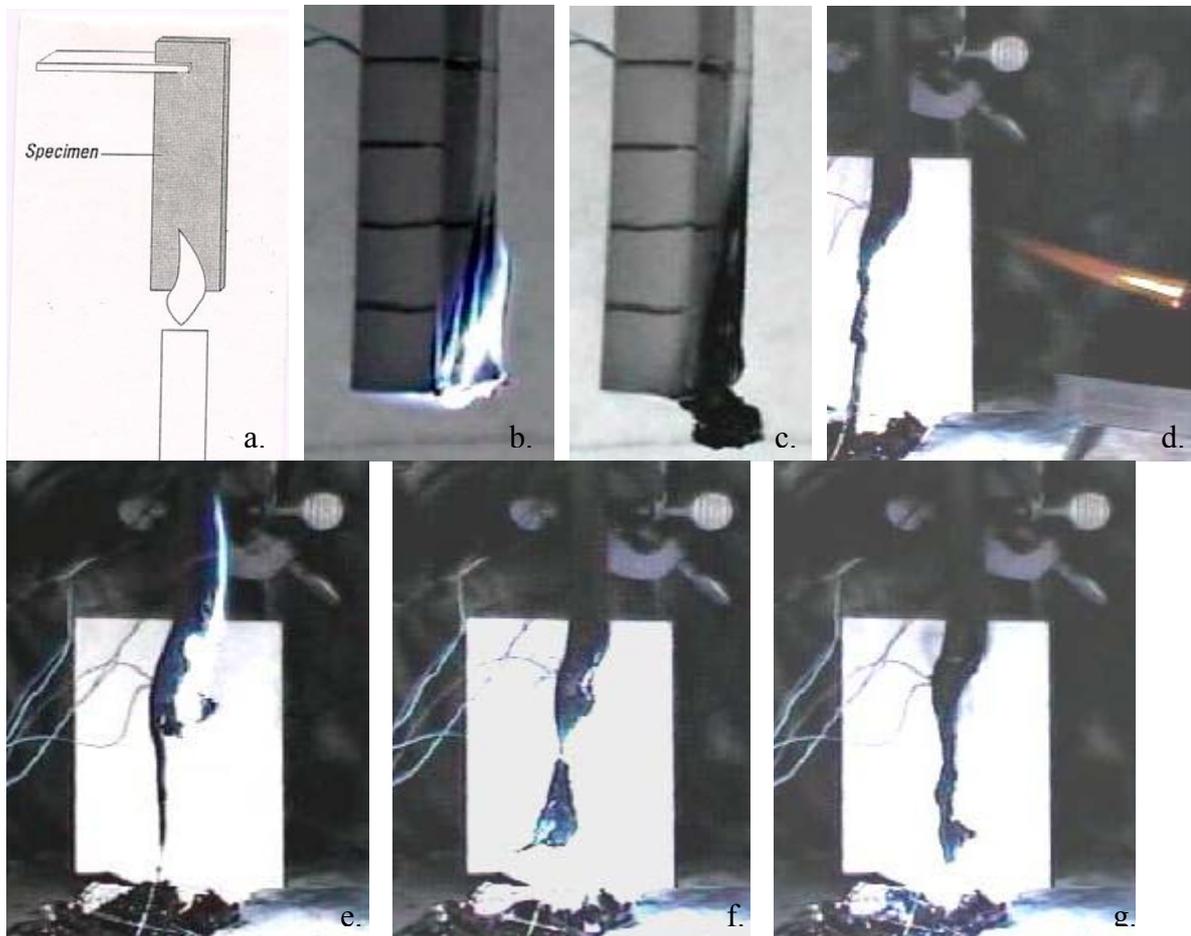


Figure 4: a) Schematic of the test, b) The flame from the propane burner is applied to the sample, c) The propane flame is removed, d) The propane flame is applied once again under a longer period of time and the plastic starts to melt, e-g) When the propane flame is removed the melted plastic burns with a self-sustained flame. The viscosity decreases and the material starts to drip, causing a small fire under the sample.

of the sample. This caused the sample to ignite, but as soon as the burner was removed, the flame died. This procedure was then repeated a few times until the sample started to melt. The flame did not spread upwards but continued to drip downwards, forming a flaming pool below the original sample.

It was evident from this tests that the plastic slab is difficult to ignite. It seemed like the material had to melt before it was possible to ignite, but the melting did not result in any upward flame spread under those conditions.

Although the sample did not have any upward flame spread, it was clear that this was not due to the non-flammable nature of the material, but mainly to its mechanical properties. The material escapes before the flame can spread. The combination of the mechanical properties and fire properties limited these results to the specific experimental conditions, and made it impossible to predict the performance of the material in a real fire scenario. A more detailed analysis was necessary.

3.1.2 Heat Release Tests

The cone calorimeter was used to study how Lexan behaved when it was exposed to radiation. The ignition time, the critical heat flux for ignition, and the heat release rate was measured. A number of tests were performed at different heat flux levels. All tests were performed with a spark ignition.

When the plastic board was exposed to external radiation in the cone calorimeter, it started to melt. After 30-60 seconds (depending on the heat flux), the bubbles started to form on the surface. As the bubbles started to grow, the material expanded, causing the surface to rise. The state of the material was changing from a solid to a highly viscous liquid embedded in a char matrix. When the bubbles burst, the vapor inside ignited, leading to a sustained flame. Figure 5a shows a sample with sustained flaming.

The ignition of the samples did not follow a well-defined pattern. Generally, ignition occurs when the igniter touched and cracked the bubbles. Therefore, under the configuration of the cone calorimeter, the location of the pilot has a significant effect on the ignition delay time. The ignition delay times obtained following this methodology have to be looked upon with caution. For Lexan the ignition protocol of the LIFT (FIST) was deemed more appropriate since the pilot is placed outside the fuel surface area in this test. The importance of bubble formation on ignition delay time was greatest close to the critical heat flux for ignition, i.e. below 30 kW/m^2 . For the higher heat fluxes, the material ignited before the surface had risen to the level of the igniter. Therefore these data will not be included in the results. The ignition times and corresponding heat fluxes are displayed in Figure 10 and will be discussed in the context of the FIST tests.

When the sample was ignited, the flame spread rapidly over the entire melted surface. Char was formed at the center of the specimen. The char intumesces¹ from the surface to form a cone shaped char matrix with its center in the middle of the sample. After just a few minutes of burning, the intumesced char had risen to touch the cone heating element.

¹ *To swell or expand; enlarge. To bubble up, especially from the effect of heating [13].*

This phenomenon is a common behaviour for engineered polymers, and has also been observed and described by Kashiwagi et al. and by Zhang et al. [3,4].



Figure 5: a) Piloted ignition of the melted and expanded plastic, b) Full fire (~30 seconds after ignition), c) The intumeced char matrix after the test.

No char was observed at the edge of the sample. Because of heat losses to the metal frame, the material near the frame melted.

The melted material created a seal that prevented gaseous degradation products located beneath the sample surface to escape. This caused the gaseous degradation products to accumulate below the sample surface. As a result, the central part of the charred sample was pushed upwards, creating the char matrix.

Figure 6 shows how the heat release rate from the samples vary when the samples are exposed to different external heat fluxes. The results show comparable heat releases for the five different external heat fluxes. For the ignition times though, a decrease is observed when the external heat flux is increasing. In the 30 kW/m²-test, a significant increase in ignition delay time is observed. This indicates that 30 kW/m² is close to the critical heat flux for ignition. This issue will be discussed more thoroughly in the context of the FIST tests.

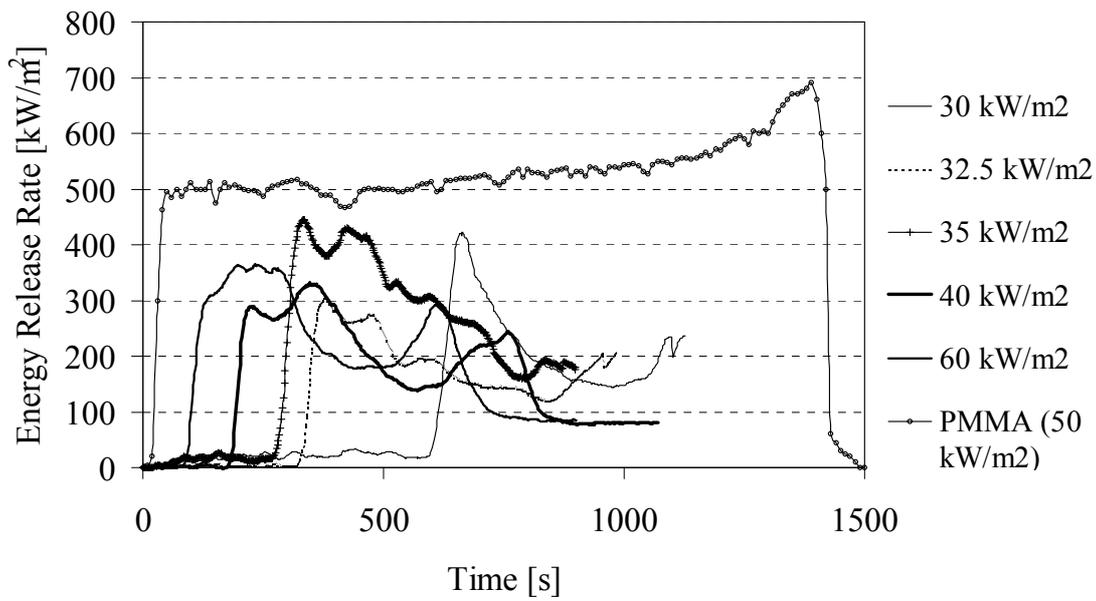


Figure 6: Heat release rate as a function of time for different external heat fluxes. For comparison a similar test conducted for PMMA is presented, the data for PMMA was obtained with (50 kW/m²) and has been translated in time to make ignition, $t=0$ sec [7, 15].

The heat release rate, \dot{Q} , seems to stabilize at a value of approximately 200-400 kW/m². The peak in the beginning of the tests typically occurred before the intumesced char matrix had reached its full height, and thus blocked some of the heat flux from the cone. This effect could also explain why the heat flux does not seem to have any particular influence on the heat release rate. Kashiwagi et al. also observed this peak in the beginning of the test, when testing two different samples of polycarbonates [4]. It is important to note that although the external heat flux as a negligible effect on the peak heat release rate it does have a significant effect on the decay period. For 30 kW/m² burning lasted less than 300 seconds while for 60 kW/m² the decay period continued for more than 800 seconds. The external heat flux served to support the evaporation of the fuel even in the presence of a thick char layer.

For comparison, Figure 6 presents typical data for a PMMA sample of identical dimensions. It can be observed that both the peak heat release rate and the burning period are much smaller in the case of Lexan. The data for PMMA was obtained with an external heat flux of 50 kW/m² and a shift in time was introduced that brought ignition to $t=0$ sec. This was done to avoid confusion of the data since the objective of showing the PMMA data is to point that Lexan represents a better choice of material from both the heat release rate and total heat released perspectives.

By integrating the area under the curve for the different heat fluxes, a value for the total heat release is obtained. Figure 7 shows the total heat for the different external heat fluxes undergoes a transition between 32.5 kW/m² and 35 kW/m². This transition shows that after the initial period, burning has to be supported by an external heat-flux. If the external heat-flux is less than approximately 35 kW/m², Lexan will cease to burn rapidly after attaining the peak heat release rate and stop contributing to the total heat release rate of the system. This information is of extreme importance for modeling, since it can provide a quantitative extinction criterion.

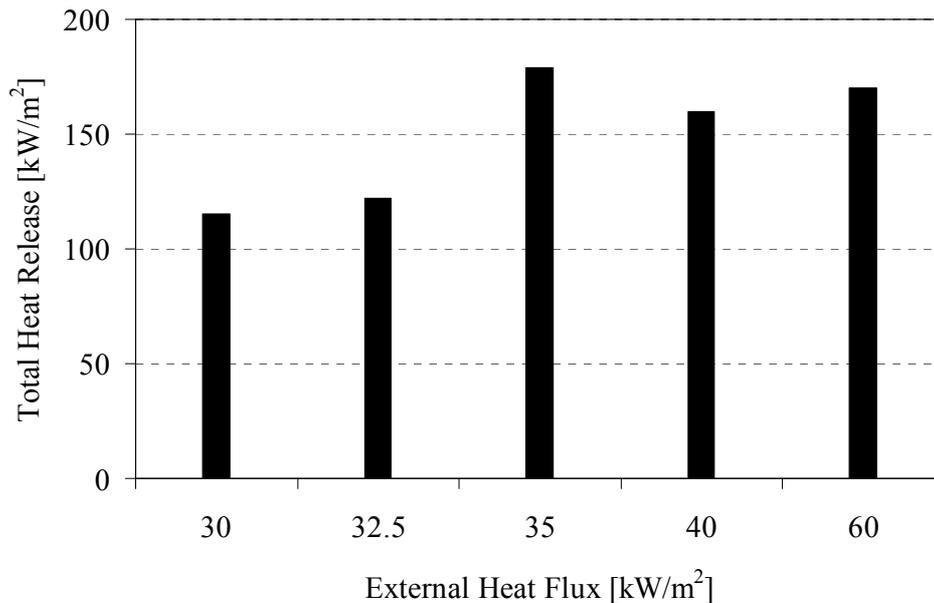


Figure 7: Total heat release for different external heat fluxes.

3.1.3 Ignition Time and Critical Heat Flux for Ignition

A slab of Lexan was also tested in the FIST-apparatus to further establish ignition properties and to determine the flame spread characteristics of the material. The specific events observed during these tests are the existence of a critical heat flux for ignition, the ignition delay time, and flame spread rate characteristics. The most common application for this material is a vertical sample, therefore, vertical flame spread tests were given priority.

The Lexan sheet was cut up in pieces of 40 mm by 143 mm to fit in the sample holder. A piece of aluminium foil was placed between the sample holder and the actual sample to prevent the plastic from getting stuck in the sample holder. After the sample holder had been placed in front of the heating panel, the timing started. Only the two upper radiation panels were used. These panels generated a distribution of radiation that had its maximum on the upper part of the sample and decreased towards the lower end. This setting was in accordance with the protocols established for the FIST by Olenick [10].

In the vertical test, the samples were tested at eight different heat flux levels, ranging from 10 kW/m² to 32 kW/m² to find the critical heat flux and the time to ignition.

Initially, the samples performed similarly to the samples tested in the cone calorimeter. Bubbles started to form after 30-60 seconds. As the bubbles grew in size, the plastic expanded, and started to melt. As a result, the upper part of the sample (where the heat flux was applied) began to slide down and out of the frame. After about 120-180 seconds, the samples started to pyrolyze. At the higher heat fluxes (31 kW/m^2 and 32 kW/m^2), the sample ignited after 290 seconds and 420 seconds, respectively. During the 32 kW/m^2 tests, flashes were observed already at 240 seconds. For the tests at lower heat fluxes (27 kW/m^2 - 30 kW/m^2), the tests had to be interrupted since all the plastic had slide out of the sample holder. No ignition or flash occurred during these tests.

At 17 kW/m^2 , the plastic started to melt and partly deformed. However, it seemed like the material kept its integrity throughout the duration of the test (15 min). When tested at a heat flux of 10 kW/m^2 , the surface of the sample started to melt, but no flowing movements were observed in the plastic. Based on these observations, the critical heat flux for melting is considered below 10 kW/m^2 , and the critical heat flux for melting followed by movements in the material is 17 kW/m^2 . The result from the different vertical rests are displayed in table 1.

When ignition occurred, the plastic become less viscous, started to drip, and the flame spread almost instantaneously over the entire sample. Figure 8 shows sequences of the flame spread for a heat flux of 30 kW/m^2 . The first picture depicts the expanded and melted plastic before ignition. The second picture is taken just after ignition and the remaining pictures in the sequence are taken with a three seconds interval.



Figure 8: a) The plastic is expanding and starts to slide out of the frame, b) The sample has just been ignited, c-e) The flame is spreading rapidly throughout the melted material and as the viscosity is decreasing, the material starts to drip (picture e).

It is clear from these observations that the flame spread rate cannot be defined in the traditional manner for this material. If the material is to be placed vertically the flame will not spread. Instead, within the modeling of the fire, if ignition is accomplished it should be followed by a translation of the fuel to the floor allowing for burning following the characteristics described in the previous section.

Because of the problems with the plastic sliding out of the sample, an attempt was made to determine the horizontal flame spread. For this purpose the apparatus was rotated 90° so that the heating panel was above and parallel to the sample. This setup was

comparable to the cone calorimeter. To obtain good reproducibility of the results a fan is used to induce a forced flow parallel to the surface. The fan was placed 190 mm from the edge of the sample and produces an airflow of approximately 0.1 m/s. Details on the implications of this modification and the protocol followed can be found in references [8] and [10].

The samples were tested at four different heat flux levels, 24 kW/m², 25 kW/m², 26 kW/m² and 27 kW/m². At 26 kW/m² and 27 kW/m², the samples ignited after 690 and 353 seconds respectively. At 25 kW/m² flashing was observed after 420 seconds and 600 seconds. However, the flashing did not lead to ignition and sustained flaming. No ignition or flames were observed at 24 kW/m².



Figure 9: a) A test sample placed in the sample holder, b) Four samples tested in the Vertical FIST at different heat fluxes. From the bottom: 10, 17, 20 and 30 kW/m². None of those samples ignited.

The scenarios for the horizontal tests were the same as described for the vertical tests, but instead of sliding or flowing out of the frame, the samples continue to expand and formed large bubbles similar to the tests in the cone calorimeter. When the bubbles burst, flaming was observed. For the samples that did not ignite, the upper layer started to char, and it seemed like the char layer prevented the melted plastic from igniting. When the sample ignited, the melted plastic became less viscous and flowed over the sample and out past the edges to the sample holder. As a result a flame was rapidly spread all over the sample. Again, the deformation of the material and the low viscosity of the molten plastic preclude the determination of a flame-spread rate. Nevertheless, it is important to note that once ignition occurs the flames cover the entire sample area almost instantaneously. Although this might not be the case for larger samples, this information is important since it indicates that flame spread rates, for this material, are controlled by the motion of the molten fuel and not by the transport of energy. Therefore, geometrical considerations are of extreme importance.

The results from the tests in the FIST, and the initial upward tests with the propane burner clearly show that the flame spread was strongly controlled by melting. No samples ignited without melting first. As a result, the flame spread tests can not be considered accurate enough to be analyzed quantitatively.

3.2 Summary of the results

In table 1 the tests result from the cone calorimeter and from the FIST are displayed.

Test	Orientation	External heat flux [kW/m^2]	Time to ignition [s]
Cone	Horizontal	30	370
Cone	Horizontal	32,5	280
Cone	Horizontal	35	240
Cone	Horizontal	40	150
Cone	Horizontal	60	60
LIFT	Vertical	10	-
LIFT	Vertical	17	-
LIFT	Vertical	20	-
LIFT	Vertical	27	-
LIFT	Vertical	29	-
LIFT	Vertical	30	-
LIFT	Vertical	31	310
LIFT	Vertical	32	440
LIFT	Horizontal	24	-
LIFT	Horizontal	25	-
LIFT	Horizontal	26	690
LIFT	Horizontal	27	350

Table 1: Summary of the result from the different tests on the Lexan sheet.

In Figure 10 the ignition delay times, from the cone calorimeter and from the FIST are plotted as functions of the external heat flux.

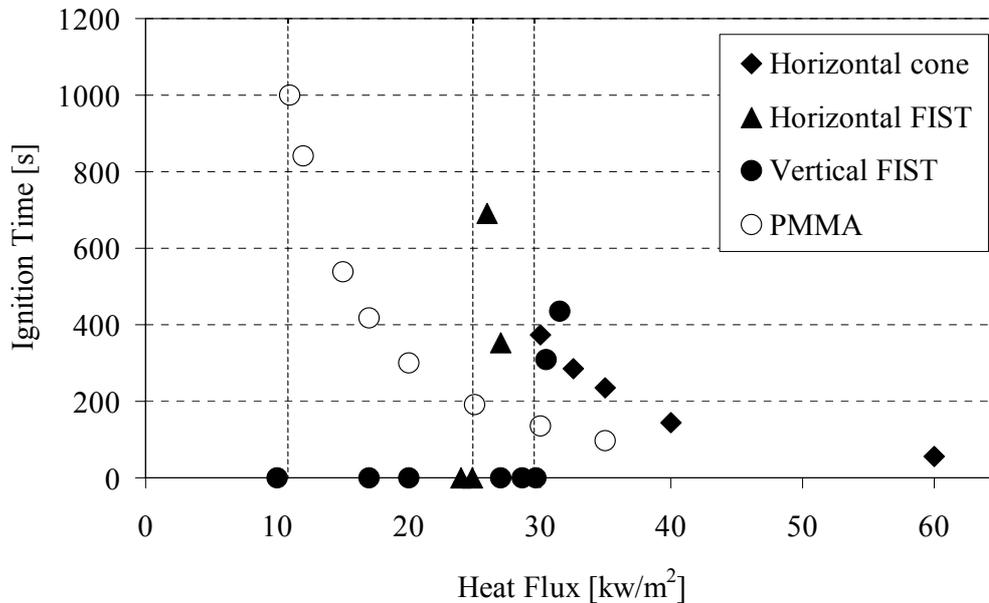


Figure 10: Ignition delay time as a function of the external heat flux for different test methods and geometrical orientation. Data for PMMA from the literature [7,15] is included for comparison.

It can be observed that the ignition delay times are fairly consistent between the different tests for heat fluxes above 30 kW/m^2 . For the horizontal configuration ignition could not be achieved below 27 kW/m^2 , therefore this value was deemed as the critical heat flux for ignition. Data points presented in the horizontal axis show tests where ignition did not occur. When the tests were conducted vertically, ignition was only accomplished for external heat fluxes above 30 kW/m^2 . As mentioned before, ignition could not be achieved because the fuel will drip away. This shows that conducting the test vertically can be misleading since the critical heat flux for ignition can be assumed as being higher than its real value.

For comparison, Figure 10, shows the ignition delay times for PMMA obtained using the LIFT [7] and FIST [15]. It is clear that PMMA has a much lower critical heat flux (11 kW/m^2) and leads to much shorter ignition delay times. Nevertheless, it is important to note that melting of the Lexan was observed to start at 10 kW/m^2 while any different types of PMMA will keep its integrity long after ignition. Therefore, Lexan will ignite and contribute to the total heat release rate of a fire much later than PMMA, its qualities as a barrier however, will be lost much earlier because even after ignited, PMMA will not slide and burning can continue for extensive periods of time before the surface regression can reach the back side of the material [15].

3.2.1 Calculating T_{ig} and k_{pc}

To calculate the surface temperature, T_{ig} , a reasonable value for the total heat transfer coefficient, h_T , has to be found. h_T consists of the radiative, h_r , and the convective, h_c , heat transfer coefficient. These values have been shown to vary with orientation and environmental effects. Olenick [10] suggested a value of the heat transfer coefficient based on a numerous experiments using the LIFT-apparatus with PMMA as the reference material. The values suggested are: $h_c = 20 \text{ W/m}^2\text{K}$ and, $h_r = 18 \text{ W/m}^2\text{K}$.

Figure 11 shows the inverse of the square root of the ignition delay time as a function of the external heat flux. By presenting the data this way, a linear correlation is obtained. Non-ignition data are presented on the \dot{q}'' -axis since the time for ignition is assumed to approach infinity.

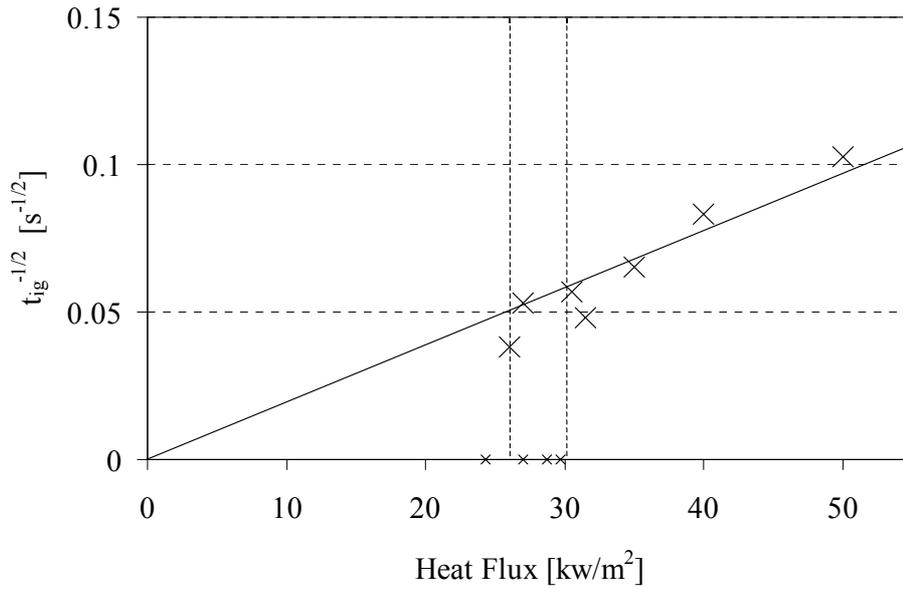


Figure 11: The inverse of the square root of the ignition delay time as a function of the external heat flux.

Three different tests methods (horizontal cone, horizontal FIST, and vertical FIST) were used, indicating three different values for the critical heat flux. The vertical FIST test provides the highest values. The vertical FIST test is probably the most relevant of the three tests, since the Lexan sheet is supposed to be used in vertical constructions. However, since the future use of the material is not known here, an average value, between the worst-case scenario, i.e. horizontal FIST, and the best-case scenario, i.e. vertical FIST, will be provided. The surface temperature, T_{ig} , is calculated using equation (4). The value for the thermal heating property, $k\rho c$, can be calculated using equation (2). The value for the ignition delay time, t_{ig} , is obtained after solving the linear equation $t_{ig}^{-1/2}$ in Figure 11 for $\dot{q}'' = \dot{q}_{0,ig}''$. Table 2 shows the calculated and experimentally obtained properties for Lexan.

Material	$\dot{q}_{0,ig}'' [kW/m^2]$	$h_T [W/m^2K]$	$T_{ig} [^\circ C]$	$t_{ig}^{-1/2} [s^{-1/2}]$	$k\rho c/a [kW^2s] m^4K^2$
Lexan	27.5 kW/m ²	38 W/m ² K	744 °C	0.052	0.68

Table 2: Fire properties for Lexan

In Table 3 the values for $k\rho c$ and T_{ig} for the Lexan sheet are compared to other materials with the similar thermal heating property. T_{ig} for Lexan is high compared to other materials studied in the literature [14]. This makes it difficult to find materials with the similar fire properties as the Lexan. The material closest when considering the parameters T_{ig} and $k\rho c$ is in this case the fire resistant plywood. Nevertheless, the Glow Wire-test had no ignition for 850°C, which is higher than the temperature of 744°C calculated here. The current configuration provides more precise answers, and the ignition temperature should be estimated as being closer to 744°C than to 850°C.

<i>Material</i>	$T_{ig} [^{\circ}C]$	$k\rho C [kW^2s/m^4K^2]$
Lexan	744	0.68
Fire Resistant plywood [14]	620	0.76
Carpet (Nylon/wool) [14]	412	0.68
PMMA Polycast (1.59 mm) [14]	278	0.73

Table 3: Ignition temperatures and thermal heating properties for Lexan and other materials.

Due to problems with melting and dripping, as described in section 3.1.3, and shown in Figure 4 and 8, the most likely scenario will be that the Lexan sheet, when exposed to fire, will melt and drip, or slide out of its frame, before ignition occurs. The temperatures when melting and melting followed by sliding movements occur can be calculated in the same way as described above for the ignition temperature. Equation (4) gives a melting temperature of 293°C and a melting/sliding temperature of 467°C. Because of this behaviour the theory for flame spread as described in section 2.3.2 cannot be applied to Lexan.

3.3 Test Results for the Liquids

3.3.1 Flash and Fire Point Tests

The closed cup burner test was used to determine the flash point and the fire point. Each liquid was tested three times. When testing the TBP liquid, the flash point was not reached before the water in the bath was close to the boiling point and the test had to be interrupted. Instead engine oil with a boiling point of approximately 250°C was used for heating the liquids. The thermometer (scaled to 150°C) was also replaced with a thermocouple and a voltmeter.

The results are displayed in Table 4. The results indicate that, when mixing the two liquids, the flashpoint will still be similar to the flash point of TPH.

<i>Liquid</i>	<i>Flash point closed cup [°C]</i>	<i>Flash point open cup [°C]</i>	<i>Fire point closed cup [°C]</i>
TBP	155	160	181
TPH	59	64	71
Mixture	67	69	73

Table 4: Fire properties for the liquids.

The flash point and the fire point temperatures provide an indication of the evaporation temperature of the fuel, but not of the thermal properties that will lead to the attainment of this temperature. Therefore, those temperatures are of importance, but not sufficient to describe the ignition process [8]. Also of importance are the rates of flame spread and the energy that can be released by the fuel in the case of a fire. The following sections will describe other material properties that serve as a complement to the flash and fire points

3.3.2 Ignition Time and Critical Heat Flux for Ignition

To find the ignition time and critical heat flux for ignition, the liquids were tested in the apparatus for horizontal ignition test as described in section 2.3.4. The heat flux level at

the top of the ignition tray was measured with a heat flux meter for each test. Adjusting the distance to the cone heater could vary the heat flux.

The 100 mm deep tray was filled with stones and glass beads to take up volume from the liquid leaving only a 10 mm depth liquid on the top. This approach is consistent with the observations of Wu et al. [9], who showed that for fuel depths larger than 5 mm no difference in the ignition delay time was observed. The stones and beads act as a good insulation and preclude fluid motion deeper than the fuel layer of interest, which result in a much better boundary condition. Furthermore, limited consumption was necessary due to the low abundance of the test liquids.

A radiation shield was placed under the cone heater to block the radiation during setup of the sample. After the ignition tray had been positioned in the sample holder and the pilot flame was lit, the radiation shield was removed, and the time recording starts.

In Figure 12 the ignition time is plotted as a function of external heat flux. Trend lines are added to further visualize the results. If the sample did not ignite, the tests were interrupted after 900 seconds. These non-ignition data are presented on the $\dot{q}_{0,ig}''$ -axis since the time for ignition is assumed to approach infinity. For TPH and the Mixture, the first flash observed also caused the ignition of the whole sample tray. For the TBP, the ignition was preceded by several minutes of flashing behaviour. The flashes existed for a maximum of two seconds and did not cause ignition. When the samples finally ignited, the flame spread instantaneously over the entire surface. The time when the first flash occurred is displayed in the Figure 12.

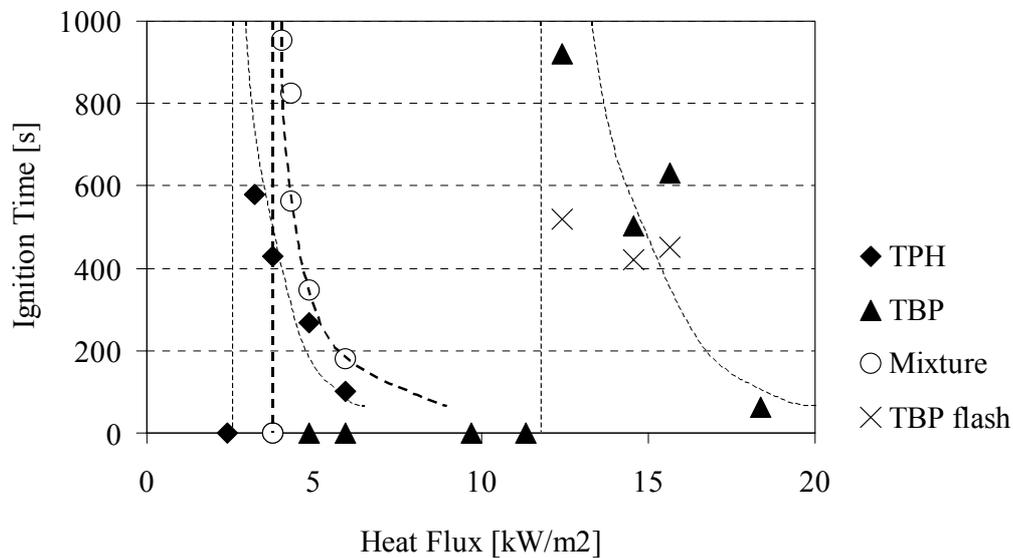


Figure 12: The ignition delay time as a function of the external heat flux for the liquids.

The results show a strong dependence between the ignition time and the external heat flux. A small change in heat flux causes a considerable earlier ignition. The result also show, as expected from the results of the flash point tests, a significant difference between the TPH and the TBP liquids. Furthermore, the ignition delay times for the TPH resemble those of the Mixture. Even though the Mixture contains 70% TBP, the

ignition time is almost similar to the TPH (30% by volume). This result was somehow expected, since the most volatile components will be the first to vaporize, and thus form a flammable mixture with air over the surface. Nevertheless, the thermal properties of the TBP should have an effect on the evolution of the surface temperature. This will be addressed later when evaluating the global thermal inertia of the fuel.

3.3.3 Heat Release Tests

The cone calorimeter was used to measure the heat release from the liquids. A sample tray with the dimensions 100 mm by 100 mm and with a depth of 40 mm was used. The sample tray was filled with glass beads to approximately half the volume. Then the liquid was filled up so all the beads were covered. After weighted the sample the sample holder was placed under the heating cone and the test was started. The spark ignition was used to ignite the sample. Each liquid were tested at two different external heat fluxes, 7 kW/m² and 8 kW/m² for the TPH and the Mixture, and 17 kW/m² and 20 kW/m² for the TBP.

The ignition protocol of the cone calorimeter is very different to that of the tests described in the previous section. For liquid fuels this has been shown to result in significantly different ignition temperatures. For the present study the ignition delay times obtained in the cone calorimeter will not be used. Figure 13 displays the heat release rate as a function of time for some sample tests, repeats of the tests presented showed very similar trends. In this plot the time to ignition was set to zero for all liquids to make the comparison easier between the different curves.

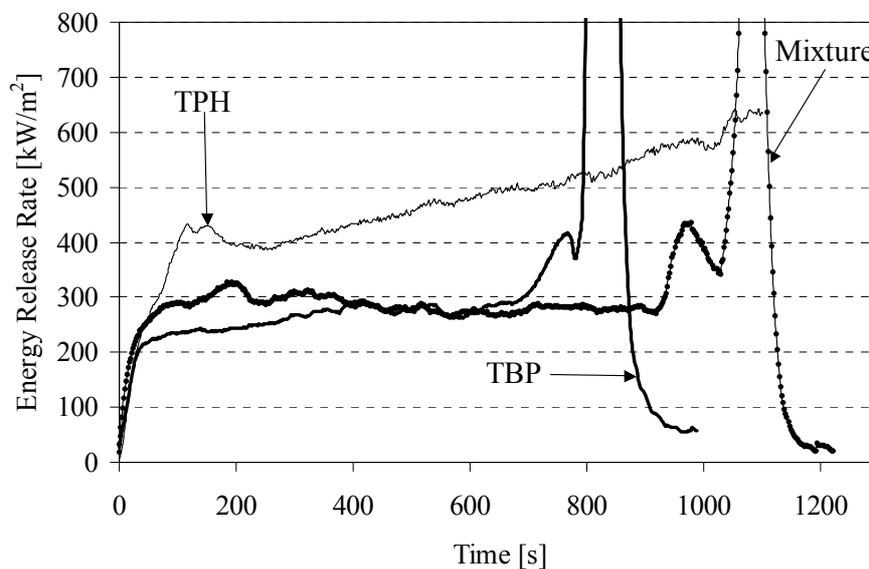


Figure 13: Heat release rate as a function of time for the three liquids.

Throughout the tests the mixture behaved in a similar manner to the most abundant component (TBP). Although the time scales varied from test to test, TBP and the mixture showed an heat release rate of approximately 300 kW/m² that remained almost constant throughout the entire test. The TPH within the mixture seems to burn showing only a slight increase in the heat release rate in the first 200 seconds. It seems that beyond this point the TPH is almost entirely gone and the mixture behaves like the

TBP. Towards the end of the test, a sudden increase in the heat release rate for both the TBP and the mixture can be observed. This sudden increase has been associated to surface tension phenomena occurring towards the end of the burning process [18]. This sudden increase is not common to all materials but it has also been observed in PMMA. This is a distinctive feature of the TBP that has been carried to the mixture but will not be necessarily present in a real-scale fire [18], although, depending on the nature of the spill or its containment might be reproduced under more realistic conditions. The data of the mass loss rate also shows a similar peak towards the end, indicating on a very fast evaporation of the last remains of the liquid.

The TPH shows an increasing magnitude of the heat release rate that starts at about 400 kW/m² and rises beyond 600 kW/m². The monotonic increase of the heat release rate is common to low flash point fuels and is an edge effect. The sample holder heats up in time and, since the fuel remains at a low temperature, heat transfer from the sample holder to the fuel increases with time. This results in an increase in the mass burning rate and thus, of the heat release rate. For this fuel, the initial heat release rate should be considered as the characteristic value. The final stages of the test with the TPH are not presented in the plot but correspond to a classical abrupt decay when the fuel is finally consumed.

3.3.4 Calculating T_{ig} and kpc

The methodology to be used here to extract the ignition temperature and thermal inertia for the liquid fuels was developed by Wu et al. [8]. Incorporating the critical heat flux for ignition and the total heat transfer coefficient in equation (4) the ignition temperature can be evaluated. The total heat transfer coefficient, h_T , used in this study is that calculated by Wu et al. [8], $h_T = 125 \text{ W/m}^2\text{K}$. This value is high but the large magnitude of the heat transfer coefficient can be associated to the high reflectivity of the liquid fuels, since the absorption coefficient is incorporated within the total heat transfer coefficient. Table 5 summarizes the results.

Material	$\dot{q}_{0,ig}'' [kW/m^2]$	$h_T [W/m^2K]$	$T_{ig} [^\circ C]$	$t_{ig}^{-1/2} [s^{-1/2}]$	$kpc/a [kW^2s] m^4K^2$
TPH liquid	2.4	125	39	0.032	19.2
TBP liquid	11.3	125	110	0.042	11.4
Mixture liquid	3.8	125	50	0.032	20.0

Table 5: Fire properties for the liquids.

The results indicate again comparable fire properties for TPH and for the Mixture, especially ignition delay time, kpc , ignition temperature and critical heat flux. For the heat release rate, on the other hand, the Mixture behaves more like the TBP liquid. It is important to note that the ignition temperature obtained when using this methodology is lower than the flash point, this has been observed before [12] and this temperature has been deemed as more adequate when attempting modelling of the spread of a flame across the liquid.

A comparison between the ignition temperature and the critical heat flux for ignition is shown in Figure 12. As expected from equation (4) the critical heat flux for ignition has a linear dependency with the ignition temperature. This shows that the total heat transfer

coefficient is not affected by the changes in ignition temperature. This plot can be used as validation of both methodologies.

A different way of interpreting Figure 15 is by calculating the slope of the line fits. Both line fits converge at ambient temperature (as expected if the critical heat flux for ignition is zero) and they grow with different slopes, the slope being the total heat transfer coefficient. If the upper curve is used (T_{ig} obtained from the critical heat flux for ignition) then $h_T = 125 \text{ W/m}^2\text{K}$ as expected. If the flash point temperature is used as ignition temperature then $h_T = 91 \text{ W/m}^2\text{K}$. Although the discrepancies can be attributed to the different testing methodologies, this information provides an estimate of the errors that can be incurred when using data from different test methods. As can be seen, the error for this particular information can be considered of approximately 20-25%.

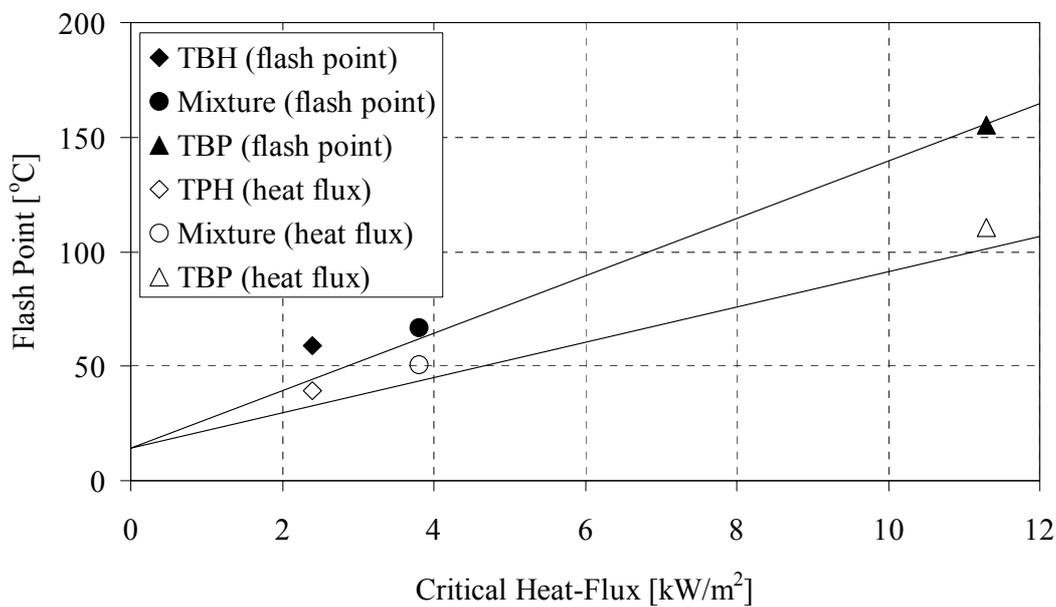


Figure 15: Ignition temperature obtained from two different test methods as a function of critical heat flux.

The value of k_{pc} is usually not calculated for liquids, since the theory presented by Quintiere [7] is based on ignition of solids. Therefore, the only data found are for two different crude oils tested by Wu et al. [8]. Those tests were performed with the same type of test equipment and under the same conditions and are therefore considered comparable. The values obtain for the crude oils are: $k_{pc} = 4.4 - 7.8 \text{ kW}^2/\text{m}^4\text{K}^2$. The difference can be explained by the fact that TBP, and especially TPH, are more viscous and the convective heat transfer within them are therefore slower. The value for k_{pc} for the Mixture was expected to be closer to that of TBP, since the mass percentage for TBP is greater than for the TPH. However, the test data show that this is not the case. The reasons for this anomaly can be many among which separation of the mixture could provide a possible explanation. As the evaporation temperature of the TPH is approached this compound separates and ignition is achieved following the properties of TPH. It is difficult to conclusively determine the reasons for this observation so this issue will not be addressed further.

4 DISCUSSION AND CONCLUSION

A simple ignition and spread test, such as the glow wire test, indicated that the Lexan sheet is difficult to ignite. It will probably also perform well in a test that does not expose the material to pre-heating before ignition. The glow wire temperature reported from the test does not provide further insight that is essential to the performance of the material. A more detailed observation of the test shows that the behavior of the material in the case of fire will be governed by melting.

The results from the tests in the FIST apparatus show that Lexan does not ignite before the material has melted. The material starts to melt at a heat flux of approximately 10 kW/m². When mounted in a vertical position, the plastic starts to slide or move at approximately 17 kW/m². To ignite the material, it has to be subjected to a heat flux of approximately 25 kW/m² when mounted horizontal, and 30 kW/m² when mounted vertically.

Flame spread modeling for Lexan is not necessary if placed vertically. The material has to melt before a flame can spread over its surface. In the vertical position the downward flame spread is very fast because of dripping of molten material. In this case it takes just a few seconds for the flame to spread over the test sample. For all practical considerations the fuel can be transferred to the floor below and allowed to burn as a pool fire. It is very difficult to achieve upward flame spread, since the material melts and escapes before it ignites. The critical heat flux for dripping (17 kW/m²) could be used as a criterion to transfer mass from the vertical sample to the floor. Nevertheless, it has to be noted that flames have a tendency to fall with the fuel, therefore the heat flux necessary to continue the transfer of fuel downwards needs to be modeled carefully.

PMMA has a critical heat flux for ignition of approximately 11 kW/m² and does not melt or drip. This material is less resistant to ignition but will serve better as a barrier. This is an important point since ignition sources in the environment of the present application are very scarce. Therefore, when selecting a material it is necessary to well define the critical heat flux for ignition of all other materials susceptible to ignition. If any of these materials has a lower critical heat flux for ignition than PMMA (most liquid fuels), then the qualities of PMMA as a barrier need to be strongly considered in the material selection process.

Due to the high ignition temperature and melting behaviour, Lexan will probably not be the first burning item in a fire. For fire modelling purposes it should therefore be included into the fuel load, but it can be discarded as the “first burning item. If the fire can grow enough and Lexan reaches the melting/sliding temperature the fuel should be transferred to a pool underneath the material, and an opening should be included where the Lexan sheet was. The material should be allowed to burn under a heat release rate curve corresponding to that of the tests presented in this report. Spread can be considered as fast enough to not be included in the model.

The two liquids showed to have different fire properties. The TPH-liquid had a flashpoint of 60°C, while the TBP-liquid had to reach a temperature of 155°C before it could be ignited. When exposed to an external heat flux, the ignition temperatures

decreases with about 20-25%. The maximum heat source a spill of the liquid could be exposed to without igniting (i.e. the critical heat flux for ignition) was also different for the both liquids. The TPH-liquid ignites at approximately 2.4 kW/m^2 while the TBP-liquid has to be exposed to more than 11 kW/m^2 before it ignites. When ignited the flame spreads instantaneously over the entire surface.

In a mixture consisting of 70% TBP and 30% TPH, the Mixture had similar fire properties as the most volatile liquid, in this case TPH. This was expected since the most volatile liquid will be the first to evaporate and ignite.

The test foundations for this report has resulted in identifications of fire properties and recommendations for modelling a fire growth scenario. These recommendations are based on physical principles but mostly on the observation of the different processes. This type of information is necessary when attempting modeling of a fire within the context of a compartment. The next step will be to use the results together with the geometry of the actual fire room to model a fire scenario. This modelling will again provide suggestions for how to make improvements of the fire safety in the actual nuclear power plant.

LIST OF REFERENCES

1. Ohlemiller, T.J., Shields, J., Butler, K., Collins, B., and Seck, M., *Exploring the Role of Polymer Melt Viscosity in Melt Slide and Flammability Behavior*, National Institute of Standard and Technology, MD, USA, 2000.
2. <http://www.structuredproducts.ge.com/> (May 2001)
3. Zhang, J., Shields, T., and Silcock, G., *Fire and Materials* 21, 1996.
4. Kashiwagi, T., Cleary, T., *Effects of Sample mounting on Flammability Properties of Intumescent Polymers*, National Institute of Standard and Technology, MD, USA, 1993.
5. *Ashford Dictionary of Industrial Chemicals*, London, 1994.
6. *ASTM Fire Test Standard*, Third Edition, chapter: D 1310-86, D56-67, E1321-97, E1354-90, PA, USA, 1990.
7. Quintiere, J., *A Simplified Theory for Generalizing Results from Radiant Panel rate of Flame Spread Apparatus*, *Fire and Materials* 5, 2, 1981.
8. Wu, N., Kolb, G., Torero, J.L., *The Effect of Weathering on the Flammability of a Slick of Crude Oil on a Water Bed*, University of Maryland, MD, USA, 2000.
9. Wu, N., Kolb, G., and Torero, J.L., *Piloted Ignition of a Slick of Oil on a Water Sub layer: The Effect of Weathering*, 27th International Symposium on Combustion/The Combustion Institute, 1998.
10. Olenick, S., *Validation of the Forced Slide Ignition and Flame Spread Test (FIST), A Reduced Scale Test Apparatus, to Assess Material Flammability for Micro-Gravity Environments*, Master's Thesis, University of Maryland, USA, 1998.
11. Long, R.T., *An Evaluation of the Lateral Ignition and Flame Spread Test for Material Flammability Assessment for Micro-Gravity Environments*, Master's Thesis, University of Maryland, USA, 1998.
12. Glassman, I., and Dryer, F., *Flame Spread Across Liquid Fuels*, *Fire Safety Journal* 3, 1980.
13. <http://www.dictionary.com> (May 2001)
14. *The SFPE Handbook of Fire Protection Engineering*, Second edition, section 2-14, Quincy, MA, USA, 1994.

15. Cordova, J.L., Walther, D.C., Torero, J.L. and Fernandez-Pello, A.C., *Oxidizer Flow Effects on the Flammability of Solid Combustibles*, Combustion Science and Technology, 2001.
16. Karlsson, B., *A Mathematical Model for Calculating Heat Release Rate in the Room Corner Test Fire*, Fire Safety Journal, Vol. 20, No. 2, 1993.
17. Karlsson, B. and Quintiere, J.G., *Enclosure Fire Dynamics*, CRC Press, 1999.
18. Babruskas, V., Twilley, W.H. and Parker, W.J., *Effect of Specimen Edge Conditions on Heat Release Rate*, Fire and Materials, Vol. 17, No. 2, 1993.

NOMENCLATURE

$k\rho c$	Thermal Inertia (kWm^2)
ϕ	Flame Spread Parameter (kWm^3s)
k	Thermal conductivity (W/mK)
ρ	Density (kg/m^3)
c	Specific Heat (J/kgK)
$q''_{i,0}$	Critical Heat Flux for Ignition (kW,m^2)
q''_i	Imposed External Heat Flux (kW,m^2)
q''_s	Net Surface Heat Flux (kW,m^2)
q''	Total Heat Release (kW/m^2)
$\dot{Q}(t)$	Heat Release Rate as a Function of Time (MJ/m^2)
T	Temperature (K)
t	Time (s)
h_T	Total Heat Transfer Coefficient ($\text{W/m}^2\text{K}$)
h_r	Radiative Heat Transfer Coefficient ($\text{W/m}^2\text{K}$)
V	Speed (m/s)
ΔH_c	Heat of Combustion (MJ/kg)
m	Mass Flow ($\text{kg/m}^2\text{s}$)
P	Pressure (Pa)
A	Area (m^2)