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TOXFIRE - Fire Characteristics and Smoke Gas Analyses in Under-ventilated Large-scale Combustion Experiments

Storage Configuration Tests



SP Swedish National Testing and Research Institute Fire Technology and Chemical Analysis SP REPORT 1996:46



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Abstract

In the CEC project *Guidelines for Management of Fires in Chemical Warehouses, TOXFIRE* (EV5V-CT93-0275), carried out by a consortium of seven European partners, SP performed controlled large-scale combustions on amounts in the 100 kg range. The main part of the SP tests were carried out in the ISO 9705 room.

This report describes combustion experiments performed in a 256 m³ test enclosure, large enough to allow the samples to be burned in a real size storage configuration. The materials studied were, as in the ISO 9705 room test series, polypropene, Nylon, chlorobenzene, chloro-nitro-benzoic acid and tetramethylthiuram monosulfide. A special instrument, a phi-meter, was built and used to measure the equivalence ratio, a measure of the degree of ventilation. The objective of these tests was to explore the influence of the combustion configuration on the fire characteristics, in particular on the nature of the combustion products.

The solid samples were put in paper bags placed in cardboard boxes. Chlorobenzene, and polypropene in an additional comparison experiment, were burned in the ordinary open fuel pans. The door opening was kept the same in all of the storage configuration experiments, equal to the largest of the ISO room test openings.

Heat release rate was evaluated from the production rates of CO, CO_2 , soot and unburned hydrocarbons. FTIR was used for on-line measurements of the concentrations of most of the low molar mass species in the smoke leaving through the door opening, chemiluminescence for the nitrogen oxides. Adsorbents were used for the sampling and subsequent analysis of medium size organics. Under-ventilated conditions were formally not achieved in these combustions.

A model developed for calculation of the flow through the door opening allowed the evaluation of yields of the various compounds in the smoke from the concentrations measured. The resulting yields indicate that the combustions were, in fact, in part under-ventilated in spite of the measured ϕ values.

A more detailed comparison of the results from the storage configuration tests with those from the ISO 9705 room tests is presented in a separate report.

Key words: combustion of chemicals, oxygen depleted conditions, degree of ventilation, equivalence ratio, fire characteristics, fire chemistry, combustion products, FTIR on-line measurements.

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Foreword

The work described in this report has been sponsored by the CEC ENVIRONMENT programme (contract no. EV5V-CT93-0275), the Swedish National Board for Industrial and Technical Development (NUTEK; project nr P1226) and the Swedish Rescue Services (Räddningsverket; projects P 221-619/94, R 52-106/96).

We would like to thank our colleagues at the departments of Fire Technology and Chemical Analysis for valuable assistance throughout this work. The authors gratefully acknowledge the generous co-operation of Dr. Haukur Ingason in the planning and design of the storage configuration set-up. We would also like to acknowledge the generous co-operation of Dr. Vytenis Babrauskas in the over-all planning of the experiments.

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Nomenclature

Symbols

- A Area $[m^2]$
- *b* Width of the opening of the combustion compartment [m]
- C_{d} Discharge coefficient
- *g* Acceleration of gravity [m/s²]
- *h* Height of the opening of the combustion compartment [m]
- m Mass [kg]
- \dot{m} Mass loss rate [kg/s]
- Mass flow rate [kg/s]
- Y Yield [kg/kg] or [g/kg]
- χ Combustion efficiency
- ρ Density [kg/m³]
- $\rho_{\rm c}$ Density inside the combustion compartment [kg/m³]
- ϕ Equivalence ratio
- τ Time [s]

Subscripts

 a Ambient air
c Compartment Combustion
f Fuel Formation
u Upper layer in the compartment

Operator

Per unit time [s⁻¹]

Abbreviations

ads.	Adsorbent
b.	Ignition burner
CB	Monochlorobenzene
CNBA	4-chloro-3-nitrobenzoic acid
f.b.	Free burning under the Industry Calorimeter
FID	Flame ionisation detector
FTIR	Fourier transform infrared spectrometer/spectroscopy
GER	Global equivalence ratio
GSP	Gas sampling probe
HPLC	High pressure/performance liquid chromotography
HRR	Heat release rate
m.e.	Measurement error
ME	Manually extinguished
MLR	Mass loss rate
n.e.	Not evaluated

n.m.	Not measured
Ny	Nylon 66
PEHD	polyethene (high density)
PMP	Phi meter probe
PP	Polypropene
PTFE	Polytetrafluoroethene
SCTE	Storage configuration test enclosure
SPR	Smoke production rate
SSP	Soot sampling probe
THC	Total amount of unburned hydrocarbon
TMTM	Tetramethylthiuram monosulfide
t.t.c.	Two-tier configuration

1 Introduction

In many countries there are large numbers of chemical plants and storage facilities that handle and store substantial amounts of hazardous substances, *e.g.* pesticides. Chemical fires seem to be one of the most important hazards from these activities. Today only limited documentation is available concerning the assessment of the potential consequences from fires at chemical plants and chemical storage facilities.

The project *Guidelines for Management of Fires in Chemical Warehouses* (TOX-FIRE) was initiated in order to remedy some of these problems. The project has been financially supported by the CEC ENVIRONMENT programme (contract no. EV5V-CT93-0275). The project was carried out by an international consortium during a three years period (1993-1996) including the following partners:

- Risø National Laboratory, Denmark (coordinator)
- NERI Danish National Environmental Research Institute
- South Bank University, United Kingdom
- VTT Technical Research Centre of Finland
- Lund University, Sweden
- SP Swedish National Testing and Research Institute
- FOA Swedish National Defence Research Establishment

Based on a number of characteristics, the substances were classified comprising ignitability, heat release, burning rate, smoke production, combustion products and the influence of the packaging materials on the combustion products. The source characteristics were described by parameters obtained by carrying out combustion experiments at various scale and by studying the effects of scaling. In addition, the fire scenarios were characterised by the degree of ventilation, the packaging materials, the stacking of the materials and the response of the building. Also the suppression was an important parameter, *i.e.* active and passive suppression, and the fire brigade tactics.

The consequences to humans as well as the environment were assessed in the project. The existing modelling methods used for risk assessment were studied, along with the handling and prevention of the accidents. These investigations led to the development of the basis for two sets of guideline documents in relation to fires in chemical warehouses: guidelines for the safety engineers and guidelines for the fire brigades. In parallel, a quick decision system to be used by the fire chief in the case of a chemical fire was also developed.

The role of SP in the TOXFIRE project was to determine the source characteristics by carrying out controlled large-scale combustions with amount of material burned in the 100 kg range. The aim was to provide results as close to real scale as possible for comparison with results from the smaller scale measurements in the project. The real challenge was to achieve conditions yielding under-ventilated, *i.e.* oxygen-depleted, combustions. Most of the large-scale experiments were carried out in the ISO 9705 room with different degrees of restrictions of the size of the door opening. A few tests were performed in a larger scale test facility that was built to allow real storage configurations. The SP work was carried out in co-operation between the departments of Fire Technology and Chemical Analysis.

A phi meter was used to define the degree of ventilation. In addition to the common fire test parameters mentioned previously, extensive measurements were carried out to characterise the composition of the smoke gases. On-line measurements, *e.g.* Fourier Transform InfraRed spectroscopy (FTIR), and adsorbent techniques were utilised to measure quantitatively individual components in the smoke. For the ISO room tests, measurements of the mass loss rate allowed evaluation of yields in relation to amount of material burned for the various species in the smoke gases.

The storage configuration tests were performed to give an idea of the influence from the sample configuration on the fire characteristics, in particular on the nature of the products of combustion. This report gives a detailed account of the combustion experiments performed in the larger (256 m³) test enclosure, large enough to allow combustion of the materials in a real size storage configuration. A comprehensive record of the individual experiments and the results obtained is presented in three Appendices. The remainder of the SP work in the TOXFIRE project is accounted for in a set of five additional reports [1 through 5], where reference 1 summarises the SP work and the others give detailed accounts of the various parts.

2 Substances

The substances for all of the large-scale combustions were selected such that a reasonable complexity in smoke gas chemistry could be expected. At the same time, the materials themselves should not present any hazard for the people working with them on a hundred kilogram scale. Nor should they generate hazardous amounts of toxics that might be harmful to the people living in the vicinity of SP.

A carbon-hydrogen (CH), *polypropene*, a carbon-hydrogen-nitrogen (CHN), *Nylon 66*, a carbon-hydrogen-nitrogen-sulfur (CHNS), *tetramethylthiuram monosul-fide*, a carbon-hydrogen-chlorine (CHCl), *chlorobenzene*, and a carbon-hydrogen-nitrogen-chlorine (CHNCl) compound, *chloronitrobenzoic acid*, were chosen for the measurements; see Figure 2.1. Two of the materials are polymers, PP and Ny, two are crystalline organic compounds, TMTM and CNBA, and one, CB, is a fairly volatile liquid. All of them are substances that are normally used in bulk quantities.



Figure 2.1 Materials selected for the large-scale combustion experiments.

A more detailed account of the selection, origin and properties of the materials is given in the ISO 9705 room report [2].

For the storage configuration measurements the solid samples were put in paper bags that were then put in cardboard boxes, for details see Section 4.5.

3 Equivalence ratio

3.1 Definition of the equivalence ratio

A parameter increasingly used to describe the ventilation conditions during a fire is the equivalence ratio, ϕ , defined as

$$\phi = \frac{\dot{m}_{fuel} / \dot{m}_{oxygen}}{\left(\dot{m}_{fuel} / \dot{m}_{oxygen} \right)_{stoich}}$$
(3.1)
$$\phi = 1 \qquad \text{stoichiometric combustion} \\ \phi < 1 \qquad \text{well ventilated combustion} \\ \phi > 1 \qquad \text{under-ventilated combustion}$$

i.e. the actual fuel/oxygen ratio is compared with the stoichiometric fuel/oxygen ratio. For the calculation of ϕ , molar ratios instead of mass ratios may be used in the formula. Since it is the over-all combustion process and not the spatial variations that is studied in this work, ϕ is defined in a wider sense as the equivalence ratio for the total combustion process. This is usually called the global equivalence ratio, GER. In this case the definition of the GER is the ratio between the mass loss rate, MLR, of the fuel and the mass flow of oxygen entering the combustion room normalized by the stoichiometric ratio.

3.2 The phi meter

To measure the GER in the room, a new apparatus, *the phi meter* [5, 6], was used. The essential parts of the phi meter are the combustor, into which the fire gases and additional pure oxygen are introduced, and the O_2 analyser (see Figure 3.1). In the combustor complete combustion of the fire gases is achieved by using a catalytic material (platinum) and additional oxygen. The readings on the O_2 analyser are compared with background measurements without fire gases through the phi meter. A simple computation gives the equivalence ratio. The phi meter was originally constructed and calibrated for hydrocarbons, but some of the substances studied within this project contain atoms of other elements (nitrogen, sulfur and chlorine). Some complementary investigation was therefore conducted, both experimentally and during the evaluation process, but no proof of specific influence by these elements has been found [6].



Figure 3.1 The phi meter, an instrument for measuring the equivalence ratio.

4 Equipment and instrumentation

4.1 The storage configuration test enclosure

To allow a real storage configuration, a storage configuration test enclosure, SCTE, (see Figure 4.1) was built beneath the industry calorimeter [7, 8] in SP's fire hall. The dimensions of the enclosure was 8.9 m \times 6.0 m \times 4.8 m with an opening on one of the long sides. The top of the opening was situated 1.5 m from the enclosure ceiling. The size of the opening (0.8 m \times 0.89 m) was chosen to be the same as the size of the largest opening used in the ISO 9705 room test series described in reference 2. The materials used for the walls and the ceiling were two kinds of non-combustible fibreboards. For further protection, the ceiling and the walls were covered by high-density mineral wool.



Figure 4.1 The test enclosure with the storage configuration, thermocouples and the measurement position in the opening. Dimensions in m.

In the tests, where the effects of the storage configuration were studied, the fuel was placed as shown in Figure 4.1 and described in the Sections 4.3 and 4.4. For safety reasons it was, after a screening test with CB in plastic bottles, decided not to use the storage configuration for chlorobenzene, but instead to put the fuel in a pan which was placed on a load cell. To prevent the fuel, especially the liquid, from being heated too much through the bottom of the pan a 0.05 m thick insulation board was

placed under the pan. This made it possible to compare the effects of the enclosure on the results. Therefore, the same pan sizes as were used in the ISO 9705 room tests [2] were also used here. No load cell was used in the configuration tests. A more detailed description of the tests performed can be found in Appendix 3. To make the set-up as similar as possible to the set-up described in reference 2, the fuel was positioned 2/3 of the depth into the room (see Figure 4.1). This was primarily done to keep the fire plume away from the opening and applies to both the configuration tests and the tests with a fuel pan. A difference between the SCTE set-up and the ISO room set-up was however that, due to the size of the fire hall and the position of the collector hood, the opening of the test enclosure, where the gas sampling was performed, had to be placed on one of the longer walls.



Figure 4.2 For the sampling in the opening, three different probes were used: the gas sampling probe (GSP), the phi meter probe (PMP) and the soot sampling probe (SSP). Dimensions in m.

Inside the test enclosure, the only instrumentation was thermocouples (0.5 mm type K). There were two thermocouple trees, one with four thermocouples above the centre flue of the storage configuration and one with six thermocouples in one corner, 1 m from the two nearest walls. In both thermocouple trees the distance between the thermocouples was 0.75 m, with the top thermocouple 0.10 m from the ceiling. In addition, there were five thermocouples 0.1 m beneath the ceiling, forming two rays from the thermocouple tree above the fuel, one connecting the two trees and one going towards one of the short walls (see Figure 4.1). The position of a thermocouple in the tree is described by the distance from the floor. This means that the top thermocouple has the position 4.70 m (see the graphs in Appendix 2). The purpose with the signals from the thermocouple tree, besides just recording the temperatures inside the room, was for them to be used for calculations of the mass flow of gases through the opening (see Section 4.6.4). A smaller vertical thermocouple tree with five thermocouples (0.5 mm type K) was placed in the opening, 0.1 m from the left hand side of it.

There were three different probes in the opening (see Figure 4.2): one for gas samples to the FTIR (Fourier Transform InfraRed spectrometer), the NO_x (nitrogen oxides), and the THC instruments, and the adsorption tubes, one for sampling of soot particles (see below) and one for samples to the phi meter. These three probes will be referred to as the gas sampling probe (GSP), the soot sampling probe (SSP) and the phi meter probe (PMP), respectively. The GSP and the PMP were placed diagonally

from the top right corner to the centre of the other side of the opening. The suction end of the probes was in the top right corner (see Figure 4.2). This arrangement was supposed to achieve a representative sampling over the opening because of a larger suction in the part of the opening with the lowest density of the smoke gases. The probes were made of stainless steel tubing with 6 mm inner diameter and had nine holes with a diameter of 3 mm, at distances of 0.10 m in between. The two probes were identical and parallel to each other, but with the holes in opposite directions. The PMP had the holes inwards in order to sample the soot as well, while the holes of the GSP were turned outwards to avoid sampling soot. For the soot sampling a special, heated (~ 180 °C) probe (SSP) was used with the 10 mm inlet pointing into the room facing the flow of combustion gases.

On the same wall as the opening there was a glass window. It was situated below the opening, a little to the left of the centre line with the lower side 1 m above the floor. The size of the window was $0.8 \text{ m} \times 0.8 \text{ m}$. Two video cameras, one facing the opening and one recording through the glass window, were used in all the tests to complement the direct visual observations.

4.2 Ignition sources

In the storage configuration tests with boxes on two tiers, a modified standard propane gas burner (NT FIRE 025) was used. The top surface, 170 mm x 170 mm, consisted of sand. The difference from the NT FIRE 025 standard was that in our case the gas was supplied near the bottom through one of the sides and not centrally through the bottom of the burner (see Figure 4.3). The heat release rate from the burner was 0.2 MW.



Figure 4.3 The propane gas burner (left) used as ignition source in the two-tier configuration (t.t.c.) tests and the rectangular ring burner used with propane for ignition of the pool fire test PP8. Dimensions in mm.

To ignite the polypropene in the test PP8 the rectangular ring burner used in the ISO 9705 room tests [2] was chosen. A pipe of stainless steel was used and 14 holes were made as shown in Figure 4.3. The fuel used for the burner was propane (\geq 95%). The HRR from the burner was about 0.1 MW.

Heptane was used for ignition in the tests CB2 and CB7 (see Appendix 3).

4.3 The SP Industry Calorimeter

The combustion gases from the room were collected by a hood, 6 m in diameter, connected to an exhaust duct. In the duct, 1 m in diameter, gas temperature and flow rate were measured, and gas was sampled and transported to the gas analysis equipment (see Figure 4.4). Concentrations of CO_2 , CO and O_2 were determined. A lamp/photo cell system was used to measure the light obscuration, making it possible to calculate the soot production. Since some problems in the O_2 measuring system arose, the method of calculating HRR from oxygen depletion could not be used. Instead HRR was determined using the production rates of CO_2 , CO, soot and THC (total amount of unburned hydrocarbons) [5]. The probe used for sampling of the THC is described in Section 4.6.1 and in reference 9. The mean flow rate in the duct was in the beginning of each test about 23 m³/s (~29 m/s). The SP Industry Calorimeter and the measurement station in the exhaust duct are further described in references 7 and 8.



Figure 4.4 The smoke from the fire is collected in a collector hood which guides the smoke to the exhaust duct, where gas analyses and measurements of temperature, flow rate and smoke density (light obscuration) are performed.

4.4 Storage configuration

In the storage configuration tests, the material was put in cardboard boxes, which were positioned on steel shelves in a two-level post pallet system [10]. On each tier (level) four shelves were placed, with a flue distance of 150 mm. The shelves were perforated allowing the melted or leaking fuel to spread freely downwards. The distance from the top of the cartons on the lower tier and the bottom of the cartons on

the upper tier was 300 mm. The substances were was placed symmetrically in the inner corner of each shelf. When deciding the actual positions of the commodities, experience from earlier work performed at SP has been used [11]. The storage configuration is shown in Figures 4.5 and 4.6.



Figure 4.5 Top view of the post pallet system, without and with commodities.



Figure 4.6 Side views of the post pallet system showing the two tier configuration (t.t.c.) with commodities. Dimensions in mm.

This experimental set-up was used in the whole series of tests but for three tests. In the screening CB test (CB2) only one box with CB in PEHD bottles, polyethene (high density), was used (see Figure 4.8) and this box was placed on a perforated

steel shelf 150 mm over the floor. As mentioned above, a fuel pan was in two test (PP8 and CB7) used instead of the storage configuration.

4.5 **Packaging of the substance**

The substances were packed in 0.05 mm thick paper bags. Each bag, which weighed 0.012 kg by itself, contained 1.1 kg substance (granules, pellets or powder). The height of a packed and closed bag was 0.11 m. Eighteen filled bags were put in two levels in a cardboard carton (0.6 m \times 0.4 m \times 0.5 m) as shown in Figure 4.7. Each cardboard carton, without substance, weighed 2.0 kg. The cartons were compartmented which means that the bags were separated by 4 mm corrugated paper walls. Each such compartment measured 200 mm \times 133 mm \times 195 mm (including the separating walls). In the pre-test CB2, 24 one litre PEHD bottles filled with CB were placed on one level in a compartmented cardboard box as shown in Figure 4.8. Each bottle weighed 50 g.



Figure 4.7 In each cardboard carton 18 paper bags with substance were placed on two levels.



Figure 4.8 In CB2, 24 one litre PEHD bottles filled with chlorobenzene were placed in a compartmented cardboard box.

4.6 Measurements performed

The majority of the measurement apparatus for gas analysis were placed in a "measurement cabin". Heated sampling lines from the probes in the duct and in the opening were guided through the roof of the cabin to the respective analysis equipment. Fresh air was blown into the cabin to cool equipment and create an overpressure, minimizing the toxic risks for the personnel. A cooling unit was also used to prevent the instruments from overheating. The cabin was placed close to the front side of the storage configuration test enclosure.

Parameters measured in the duct were CO, CO_2 , smoke obscuration, THC, NO_x temperature and pressure (see Section 4.6.1, Table 4.1 and reference 8). In the opening of the room, a more thorough investigation of species produced was made. The parameters measured in the opening were the equivalence ratio, THC, NO_x , organic compounds, compounds measured with FTIR, soot and temperature (see Sections 4.1 and 4.6.2 and Table 4.1).

Instrument	Measurement principle	Measured species	Sampling position
THC analyser	Flame ionisation	Unburned hydrocarbons	Duct
NO _x analyser	Chemiluminescence	NO _x	Duct
CO/CO ₂ analyser	IR-absorption, selected bands	CO and CO ₂	Duct
THC analyser	Flame ionisation	Unburned hydrocarbons	Opening
NO _x analyser	Chemiluminescence	NO, NO ₂ and NO _x	Opening
FTIR	IR-absorption, full spectra	$H_2O, CO_2, CO, HCI, SO_2, HCN and NH_3$	Opening
O ₂ analyser (Phi meter)	Paramagnetic	O ₂	Opening

Table 4.1On-line instruments used for measurements in the duct and in the
opening.

4.6.1 Measurements in the duct

In addition to the measurement station described above (see Section 4.3), an additional probe was used for gas sampling in the exhaust duct. The sampling probe was equipped with a sampling tube/filter holder unit and a flow divider. The probe *METLAB GSV-1500*, consisted of a heated jacket made from polished stainless steel with a removable glass insert comprising a sampling tube and a filter holder. The temperature of the probe was kept at 200 °C in all experiments. The sampling tip of the probe was positioned in the centre of the duct and was facing the flow. The smoke was considered well mixed in this point. The smoke gas sampling flow was divided using short heated (200 °C) PTFE tubing prior to entering a NO_x analyser and a THC analyser. The NO_x analyser, *A.A.L. 440*, was a heated chemiluminescence instrument with two measuring modes, NO concentration or NO_x concentration. The NO_x mode was used during all the tests. The THC analyser, *Siemens Fidamat K*, was a heated flame ionisation (FID) instrument measuring the total amount of unburned hydrocarbons.

4.6.2 Measurements in the opening

The gas sampling probe, GSP, in the door opening (see Section 4.1) was connected to a heated filtering unit. The filtering unit, model *PSP 4000 H* from *M & C Products Analysentechnik GmbH*, is made of stainless steel having a ceramic filter element with 2 μ m filter porosity. The filter was heated to 180 °C.

Smoke gas samples were drawn from the gas probe through a 7 m heated flexible tube (200 °C), having an inside removable Teflon tubing with an inner diameter of 6 mm and a 1 mm wall thickness.

The sampled smoke gases were divided to the various individual analysis instruments and adsorbents at the end of the 7 m heated sampling line (see Figure 4.10). Heated flexible tubes (200 °C), having an inside removable Teflon tubing with an inner diameter of 4 mm and a 1 mm wall thickness, were used for the sub flows after partitioning. In all tests, the THC analyser and the NO_x analyser were drawing their sample flows from the outlet of the FTIR cell. Thus, omitting a separate pump to the FTIR reduced the total flow through the heated filter. This was a way to prevent the filter from getting blocked by soot which was a problem in the tests made in the ISO 9705 room (see reference 2).



Figure 4.9 Schematic figure showing equipment set-up for measurements in the opening.

A heated chemiluminescence instrument, *ECO PHYSICS CLD 700 EL HT*, was used for measuring the concentrations of the nitrogen oxides, NO and NO_2 , in the smoke gases. This instrument has two reaction chambers, with a shared detector, allowing NO and NO_x to be measured simultaneously. NO_2 is determined as the calculated difference. The total amount of unburned hydrocarbons, THC, was measured using a heated flame ionisation detector (FID) instrument, *J.U.M. VE 5*. (see reference 9 for a detailed description of these two instruments).

The smoke gases were also continuously drawn through a heated (150 °C) IR absorption cell (see Figure 4.10) of an FTIR spectrometer, model *BOMEM MB 100*. FTIR is based on infrared absorption. Polyatomic and heteronuclear diatomic compounds have absorption in the infrared region and can be identified and quantified on the basis of absorbance. Concentrations of selected compounds were measured by taking 4 scans giving a new spectrum every 18 s. The resolution used was 4 cm⁻¹. Table 4.2 shows the compounds measured and evaluated during the tests. The FTIR

spectra contain more information than what is presented in this report, and may be used for further evaluation (see also reference 3).

Compound	Lowest calibrated concentration	Highest calibrated concentration
H ₂ O	0.02 %	16 %
CO2	0.20 %	20 %
СО	100 ppm	80 000 ppm
HCl	50 ppm	5100 ppm
SO ₂	10 ppm	26 000 ppm
HCN	10 ppm	2000 ppm
NH ₃	10 ppm	1600 ppm

Table 4.2	Compounds measured and evaluated with FTIR technique and infor-
	mation on calibrated concentration spans (volume concentrations).

At some instances during the tests, concentrations of compounds measured with FTIR exceeded their calibrated concentration spans. In these cases a linear approximation was used for extrapolation. This does not give the exact concentration, but gives a reasonable estimate of the true concentration [3].

Organic components were sampled on selected adsorbents. Integrated sampling was conducted, not over the whole test, but during a specified time period (typically 10 minutes). One set of adsorbents consisted of one tube containing XAD-2 (a polyaromatic adsorbent) and one tube with activated charcoal (for high-volatility hydrocarbons), to be desorbed with a solvent. The second set consisted of a Tenax tube and a Carbotrap/Carbosieve tube (for the high-volatility hydrocarbons), adsorbents allowing thermal desorption, *i.e.* without a solvent. Identification and quantification of individual components were made using mainly gas and in some cases liquid chromatography techniques (see references 9 and 4 for detailed information on adsorbent sampling and analysis).

Soot was sampled in the opening using the probe (SSP) described in Section 4.1. The soot was captured on a stocking type filter made of quartz glass. With one exception (CB7), integrated sampling was done during the same time periods as for the adsorbents. The sampling flow used was 5 L/min to 8 L/min, giving a velocity in the probe tip of 1.1 m/s to 1.7 m/s. The mass of the soot sampled was determined by weighing.

Sampling of smoke gases for the phi meter was done in the opening (see Sections 2.2 and 3.1.3).

4.6.3 Data handling

All data, but for the FTIR signal, was collected using a *Schlumberger 3530 ORION data logging system*. The configuration of the data logger, the collecting of the data and the saving to file were controlled by a *LabVIEW*® routine on a *Macintosh Quadra 700*. Data sampling was performed every 3 s. Since only the temperatures (°C) and the load cell signal (kg) were saved in their correct form, all the other values had to be converted from a voltage signal according to the calibration results. A new FTIR spectrum was collected every 18 s to a *Compac Deskpro 560*. An evaluation routine made in *GRAM/386 (Galactic industries corporation)* presented evaluated concentration data on the computer screen during the tests and saved the concentration data in a text file. In many cases a second evaluation of the spectra had to be done manually afterwards because of difficult evaluation conditions. Even if the data acquisition started at the same time for the data logger and the FTIR, the sampling frequencies were not the same. Consequently, the two time scales were matched afterwards.

4.6.4 Calculations of the flow through the opening

To calculate yields etc. for the reaction products, the mass flow is required for the smoke leaving through the opening, where the sampling was performed. It is a difficult task to use velocity and temperature sensors in the opening plane in order to establish the mass flow rates. With a high degree of attention to detail, such techniques can be used for steady state fires [12, 13]. However, for transient fires, when similar techniques are applied, the results are often nonsensical [14]. This depends on the following three different facts.

(1) The outflows in the doorway opening have a velocity component in directions other than just the normal to the exit plane. With the commonly available velocity probes, this component is partially assigned – erroneously – to the normal direction.

(2) For some fires the velocity distribution is not uniform across the width of the opening. Not only is the velocity different near the edges of the opening, but some fires show [14] a stagnant region near the centre and higher velocity flows away from the centre.

(3) The velocity measurements obtained are usually highly noisy, since the pressure difference is only a few pascals due to the low flow.

The above considerations suggest that the mass flows in the opening should be computed by some means, rather than using direct measurement techniques. There are different models available for calculating the flow. The model chosen has been described by Babrauskas [15]. It assumes a well-stirred uniform-temperature compartment which means that the density inside the compartment should be uniform. The calculations are based only on temperature measurements and assumes that the outflow and the inflow are equal. This model was developed somewhat further to include also the MLR [5].

$$\dot{m}_{out} = \frac{\frac{2}{3} \cdot C_{d} \cdot b \cdot h^{\frac{3}{2}} \cdot \sqrt{2 \cdot g \cdot (\rho_{a}^{2} - \rho_{a} \cdot \rho_{c})}}{\left[1 + \left[\frac{\rho_{a}}{\rho_{c}} \left(\frac{\dot{m}_{out}}{\dot{m}_{out} - \dot{m}_{f}}\right)^{2}\right]^{\frac{1}{3}}\right]^{\frac{3}{2}}} + \dot{m}_{f}$$
(4.2)

This equation has to be solved numerically by *e.g.* iteration.

4.6.5 Calculation of yields

To be able to better compare results from different tests, the measured concentrations of a number of species were converted into yields, *i.e.* the mass of a certain species produced (m_i) is divided by the consumed mass of the fuel (m_{fuel}) :

$$Y_i = \frac{m_i}{m_{fuel}} \tag{4.3}$$

where *i* represents the different species.

Since load cell measurements were available only in two tests (PP8 and CB7), m_{fuel} had to be estimated for the other tests. By assuming a combustion efficiency of about 60 %, the calculated HRR could be used to calculate the amount of fuel burned.

In the tests PP8 and CB7, the load cell signal was affected by the heat inside the test enclosure. The pool fires where, however, burning very slowly and it was therefore assumed that the MLR curve and the HRR curve had the same shape. This meant that the MLR could be calculated from the HRR since the total mass loss was known.

The results from these calculations can be found in Appendix 1. Some results are also presented and discussed in Chapter 7.3.

5 Measurement procedure

Before the experimental test period started, the over-all calorimeter system was calibrated. As calibrating reference, methanol was used. Calibrations of the separate gas analysing instruments were performed on a daily basis.

The typical measurement procedure before and during each test was as follows:

- Ambient conditions were recorded (for most of the tests the ambient temperature was about 20 °C and the relative humidity was about 50 %)
- The on-line instruments were calibrated against reference gases.
- A new background spectrum was recorded on the FTIR with nitrogen flowing through the cell.
- The duct flow was established (about $23 \text{ m}^3/\text{s}$).
- Background sampling was performed for the phi meter and for the adsorbent measurements.
- Safety back-up was ensured.
- All time devices for the experiment time function were synchronized.
- The data acquisition was started to collect background data for three minutes before ignition.
- The video cameras were turned on.
- Ignition (see Section 4.2 for information about the ignition sources used).
- When the fire had reached a certain level the flows through the adsorption tubes and for the soot sampling were started.
- When the fire had to be extinguished, most of the gas analysing instruments and the flow through the adsorption tubes were switched off to avoid clogging problems and water contamination.
- Generally one test was performed each day. In Appendix 3, where observations during the tests are presented, the actual experimental schedule and ambient conditions can be found.

6 Measurements performed

The experimental conditions for each test in the series of tests are put together in Table 6.1; PP7 and CB2 were pre-tests. Also some changes in the experimental setup and in the sampling strategy are described. For more details about each test, see Appendix 3.

Test	Fuel mass /kg	Pool size /m ²	Opening height /m	Total ignition duration /min:s	Comments
PP7	130	t.t.c	f.b.	1:00	Polypropene in paper bags placed in compartmented cartons. The total mass of the cardboard was 16 kg. ME 8:56.
CB2	20	see Comments	f.b.	1)	CB in PEHD bottles in one cardboard carton. The total mass of the cardboard was 2 kg.
PP8	42.5	1.2	0.89	1:46	Burner on 0:00-1:00, 1:15-1:26, 1:38-2:06 and 2:38-2:45. The material was burned in a pool.
PP9	160	t.t.c.	0.89	1:10	Polypropene in paper bags placed in compartmented cartons. The total mass of the cardboard was 16 kg. ME 19:25.
Ny6	160	t.t.c.	0.89	1:12	Nylon 66 in paper bags placed in compartmented cartons. The total mass of the cardboard was 16 kg. ME 21:08.
TMTM6	160	t.t.c.	0.89	1:00	TMTM in paper bags placed in compartmented cartons. The total mass of the cardboard was 16 kg. ME 16:02.
CNBA5	160	t.t.c.	0.89	1:00	CNBA in paper bags placed in compartmented cartons. The total mass of the cardboard was 16 kg. ME 11:50.
CB7	39.5	0.8	0.89	0:38	The material was burned in a pool.

Table 6.1Experimental conditions for the tests given in order of performance.

1) The ignition source consisted of fibre board soaked in heptane (see Section 4.2)

7 **Results**

The aim with the tests in the SCTE (storage configuration test enclosure) was to see how the increased scale affected the results. The two-tier configuration (t.t.c.) tests were performed to also study the influence of the storage configuration on the results. To study the influence of the larger combustion room itself, the tests with the fuel pan/load cell configuration were performed in such a way that the pan sizes and the amounts of fuel were the same as for the ISO 9705 room tests with the same opening height (0.89 m) [2]. In this chapter mainly results from the tests in the SCTE are presented. Some comparisons with the ISO 9705 room tests are done, but most of the comparisons of the results are presented in reference 1.

7.1 Fire related parameters

The influence of the larger volume of the combustion room is most obvious when it comes to the temperatures inside the room. The fire load in the larger-scale fuel-pan tests is not enough to reach high temperatures. The temperatures in the storage configuration t.t.c. tests are higher, but also in this case the thermocouple tree in the corner recorded rather moderate temperatures, about 600 °C.

A comparison between the heat release rates shows, of course, a difference in maximum values, where the t.t.c. tests with their larger amounts of fuel and larger burning surface areas reach higher HRR. However, there were also differences' in the development of the fires. The fire spread was quicker in the t.t.c. tests due the cardboard boxes and the configuration. The higher heat release rate from the ignition source in these tests may also have affected the development of the fire.

As can be seen in Appendix 2, the HRR in the tests PP9, Ny6 and TMTM6 finally reached a steady state value of about 800 kW. However, the development periods to reach this value differ significantly. In PP9, the HRR increased rather constantly up to about 900 kW. After some minutes of quite constant HRR it decreased to a level somewhat higher than 800 kW and stayed there for about ten minutes until the fire was extinguished. The situation was very different in Ny6, where the HRR after the first increase up to about 200 kW in fact decreased a little before it slowly increased to the steady state value of about 800 kW. The fastest increase of the HRR can be found in the test TMTM6 where also the highest value of the HRR was recorded. After the initial period of a few minutes the HRR decreased to a value of approximately 800 kW. In CNBA5, the HRR was lower, but that was also expected since it was found in the tests in the ISO room that the CNBA burned very poorly by itself [2]. However, in CNBA5, the cardboard boxes helped raising the temperature inside the room, which made it possible to maintain the combustion.

The CNBA5 test was exceptional in that unstable conditions were reached inside the room and twice two enormous outbursts of smoke and flames came out of the opening (after which the test was terminated). A consequence was visible cracks in the walls of the test enclosure. The outbursts are not very well recorded in the HRR measurement since much of the smoke filled the fire hall instead of entering the exhaust duct where the measurements were performed. However, in the temperature graph in Appendix 2 two high peaks can be seen.

7.2 Equivalence ratio

Due to the large volume of the SCTE, under-ventilated conditions, as determined by the phi meter, were not obtained. In the tests with fuel pans, the very modest burning rates were not able to force the equivalence ratio higher than about 0.5, *i.e.* the conditions were well ventilated. PP9, Ny6 and TMTM6 followed about the same development trends as described above for the HRR. They all reached a value of the equivalence ratio of about 1, *i.e.* they reached stoichiometric conditions, before the equivalence ratio decreased. In CNBA5, the equivalence ratio was not as high because of the lower burning rate. The values recorded during the two outbursts may be incorrect due to the totally different flow pattern in the opening.

Some of the results from the chemical analysis, especially when compared with the results from the ISO 9705 room tests, indicate that the combustion conditions were, in fact, not well ventilated. The phi meter registered an overall equivalence ratio for the gases leaving the room, but there may have been large variations in the equivalence ratio inside the room. Therefore, it is possible that locally under-ventilated conditions have existed in the combustion zone. This is discussed further below.

The SCTE with its large thermal ballast has also a completely different temperature distribution than the smaller ISO 9705 room. This also changes the combustion conditions.

7.3 Concentrations in the opening

The concentrated smoke gases in the opening represent a worst case for humans as regards the toxicity of species produced in the storage configuration fire.

To get a comprehensive picture of measured concentration levels in the opening for some harmful species, one minute mean peak values are presented in Table 7.1 for all tests made in the storage configuration enclosure.

Test	СО	NO	NO ₂	HCN	NH ₃	SO ₂	HCI
PP8	7600	240	5	420	< 5	-	-
PP9	28000	72	6	510	310	-	1.010
Ny6	5800	300	2	1300	100	+	÷ .
TMTM6	21000	270	13	7700	220	43000	-
CNBA5	39000	6900	220	2700	< 5	-	16000
CB7	3900	5	1	< 5	< 5	-	7000

Table 7.1One minute mean peak values (ppm (V/V)) in the opening.

Note that this relates to maximum concentrations. These should not be compared to those reported from the tests in the ISO 9705 room, where mean concentrations in under-ventilated and well ventilated conditions are compared [2].

To get an idea of the potential risks for humans inhaling the more or less undiluted smoke gases from the opening, the measured concentrations in Table 7.1 can for example be compared with the limit values in Table 7.2. The values in Table 7.2 are hygienic limit values for short time (15 min) exposure in work place environments (ceiling values).

A general observation is that the maximum concentration of CO for all substances vastly exceeds the limit value.

The concentration of NO exceeds the limit values for the nitrogen containing substances, and also for PP. Also NO₂ exceeds the limit value, especially for the test on CNBA. All compounds except CB produced HCN in concentrations exceeding the limit value, and in much higher concentrations for the nitrogen containing substances. The concentration of NH₃ exceeds the limit value for Ny, TMTM and in PP9. The unexpected high production of nitrogen containing species from PP appears to originate from reactions with ambient nitrogen.

The concentrations of SO_2 and HCl are drastically higher than their limit values in the test on TMTM respective the tests on CNBA and CB.

Species	Maximum concentration /ppm (V/V)
Carbon monoxide, CO	100
Nitrogen monoxide, NO	50
Nitrogen dioxide, NO ₂	5
Hydrogen cyanide, HCN	5
Ammonia, NH ₃	50
Sulfur dioxide, SO ₂	13
Hydrochloric acid, HCl	5
	di.

Table 7.2Short time maximum allowable concentrations (ceiling value) in work
place environments.

7.4 Yields and recoveries

One way to express the production rate of a certain species during combustion of a substance is in the form of yields (see Section 4.6.5 for definition of yield). As opposed to concentrations, yields are independent of the degree of dilution and can for example be used for comparisons between different test conditions.

As in the ISO 9705 room tests, certain time periods were selected for which yields were calculated. For most tests, one single period was sufficient to describe the nature of the test. The period was chosen to reflect stable conditions, so the first phase of each test was omitted. In Ny6, two time periods were chosen as the production pattern changed markedly during the test.

When checking the measurement/analysis system, one way is to calculate the carbon recovery, *i.e.* the total amount of carbon in the different compounds measured is compared with the total amount of carbon burned. It is generally expressed as percentage by mass.

The differences in composition between the cardboard boxes and the substances tested have not been taken into account when the recoveries have been calculated. The cardboard boxes were, however, to a large extent burning in the initial phase of each configuration test and therefore this simplification does not affect the recoveries very much.

An uncertainty when discussing yields and recoveries for the tests conducted is that the load cell was used only in the tests with a pool fire. The mass loss in the storage configuration tests had to be estimated. It was calculated from the heat release rate using the approximation that the combustion efficiency was 0.60 in all four cases. These estimated mass losses gave **carbon recoveries** ranging from 62 % (Ny6) to 99 % (CNBA5) in the opening and from 62 % (Ny6) to 90 % (CNBA5) in the duct. These values should be compared with the two pool fire tests where the carbon recoveries where between 50 % and 70 %.

Since all experiments were well ventilated (see Section 7.2 for discussion on the degree of ventilation) the major combustion product of the carbon should be carbon dioxide. As can be seen in Table 7.3 the **recoveries of CO₂**, measured in the opening, are between 60 % and 80 % of the individual nominal (maximum) yield for each substance. The exception is CB with a recovery of 34 %. These results can be compared to what was seen in the experiments in the smaller ISO-room (see reference 2). When looking at the well-ventilated parts of these experiments, recoveries of CO₂ in the range of 50 to 80 % was seen. CB gave a low recovery also in this case, but this experiment was not a well-ventilated one and may not be suitable for comparison. After the test CB7, there was a lot of soot on the walls inside the SCTE. This part of the soot was of course not accounted for in the carbon recovery.

It is of course possible to investigate the fate of elements other than carbon by calculations of their respective recoveries. The recovery of chlorine as HCl and the recovery of sulfur as SO_2 have been investigated. One important point to consider, when investigating these recoveries, is the size of the test enclosure and the inherent temperature differences and large surface area. A rather large degree of condensation, especially of aqueous HCl, is to be expected on cold surfaces.

Test	Y _{CO2} ,measured / (kg/kg)	Y _{CO2} ,nominal / (kg/kg)	Recovery / %
PP8	1.70	3.14	54
PP9	2.02	3.14	64
Ny6	1.44, 1.61	2.33	62, 69
TMTM6	0.83	1.27	65
CNBA5		-1.53	77
CB7	0.81	2.35	34

Table 7.3Yields of carbon dioxide compared to nominal yields.

For the chlorine containing compounds one would expect almost all chlorine to form hydrochloric acid in a well ventilated combustion. As can be seen in Table 7.4 less than 50 % of the chlorine can be found as hydrogen chloride. This implies that a large part of the chlorine can be found, as discussed before, condensed on the walls or, in the case of the volatile CB, evaporated as a 'survival fraction'. The same low recoveries of chlorine as HCl was also seen in the tests in the ISO-room (see reference 2). The mean recovery of the tests on CNBA, which were all well ventilated, was 23 %. The mean recovery of the experiment CB6 was 49 %. This experiment was, as opposed to the experiment in the SCTE, under-ventilated.

Table 7.4	Yields of hydrogen	chloride compared	l to nominal yields.
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Test	Y _{HCl,measured} / (g/kg)	Y _{HCl,nominal} / (g/kg)	Recovery / %
CNBA5	78	181	43
CB7	141	324	44

For the sulfur containing substance TMTM one would expect to find the major part of the sulfur as SO₂ in a well ventilated combustion. Only 69 % of the nominal yield was measured in the test. This is in contrast with the results from the tests in the ISOroom (see reference 2). There a mean recovery of sulfur as SO₂ of 91 % was found for the well ventilated parts of the tests ($\phi < 1$). In the SCTE test it was also found that the maximum production rate of SO₂ was at stoichiometric concentrations of oxygen ($\phi = 1$).

A possible explanation, in addition to the losses to the walls of the test enclosure, is that there has been local under-ventilated conditions in the combustion zone, whereas the overall degree of ventilation was measured. An indication of this is that substantial absorption of COS can be found in the FTIR spectra. The results from the tests in the ISO room show that COS is produced mainly during under-ventilated conditions.

Test	Y _{SO2} , measured	Y _{SO2} ,nominal	Recovery
	/ (g/kg)	/ (g/kg)	/ (%)
ТМТМ6	630	920	69

Table 7.5Yields of sulfur dioxide compared to nominal yields.

7.5 Principal organic compounds

Adsorbent sampling of the smoke was carried out for one basically predetermined period in each of the storage configuration tests, an exception being PP8 where no adsorbent sampling took place. The length of the sampling periods was 10 min for PP9, CNBA5 and CB7, for Ny6 18 min, and for TMTM6 12 min. All results from the adsorbent measurements represent mean values over the entire sampling period and refer to the integrated mean values of the degree of ventilation, ϕ , as measured by the phi meter. The values of ϕ listed in Table 7.6, imply that all the results formally pertain to well ventilated conditions.

Generally, for all experiments the two types of adsorbents for thermal desorption (4.6.2), Tenax and Carbotrap/Carbosieve, respectively, were analysed by GC techniques. The XAD-2 adsorbents were also analysed by GC but in that case only total amount adsorbed and the principal component were quantified. All quantitations were made from GC-FID measurements, whereas the identifications were based on the parallel mass detector (MSD) measurements. More detailed information on the adsorbent measurements is given in reference 4.

The combined **yields** from the two types of adsorbents are, for the individual components listed in Table 7.6, on an absolute scale approximate ones, since mass loss rate could not be directly measured in the storage configuration tests. But the relative amounts for components in the individual experiments are as valid as for the ISO 9705 room tests [2].

In spite of particular efforts to retain the most volatile components on the CC adsorbent, no trace of for example methane was found. Ethane was identified in smaller amounts in some tests. Whether these compounds were not adsorbed properly during sampling or whether they were lost in the cooling trap of the gas chromatograph in the analysis step, or both, has not been clarified.

Experiment	periment <i>\phi</i> Compound		Concentration in the smoke /(g/m ³)	Yield* /(g/kg)
PP9	0.74	Benzene	0.77	7.1
	8	Propene	0.12	1.09
	5	Naphthalene	0.098	0.91
		Toluene	0.057	0.53
		Propyne	0.020	0.19
		Styrene	0.020	0.18
Ny6	0.48	Propane	0.04	0.4
		Benzene	0.021	0.23
		Toluene	0.010	0.11
		Cyclopentanone	0.005	0.05
		Benzonitrile	0.004	0.04
		2-Propene nitrile	0.004	0.04
TMTM6	0.83	Propene	0.030	0.12
		Acetonitrile	0.020	0.10
		Benzonitrile	0.008	0.04
		Benzene	0.007	0.04
		2 Propene nitrile	0.005	0.03
		Thiophene	0.005	0.03
CNBA5	0.63	Chlorobenzene	1.34	6.3
		Dichlorobenzenes	0.75	3.6
		Propene	0.42	2.0
		Chlorobenzonitriles	0.100	0.48
		Benzene	0.050	0.23
		Benzonitrile	0.049	0.22
CB7	0.17	Chlorobenzene	4.9	106
		Benzene	0.14	2.9
		Naphthalene	0.039	0.84
		Chloroethynylbenzene	0.021	0.45
		Ethynylbenzene	0.017	0.37

Table 7.6Concentrations and approximate yields for the main individual or-
ganic components in the smoke.

* Calculated using estimates of the amount combusted during the sampling period; see Section 4.6.5.

One consequence of the loss of the, in some cases probably dominant, low molar mass hydrocarbons is that it becomes meaningless to compare total yields from the adsorbent measurements with those determined by the on-line FID measurements of THC. The presence of methane in the smoke gases during the adsorbent sampling periods has, in fact, been confirmed in many of the experiments by semiquantitative analysis utilising the FTIR spectra. Occasionally, the adsorbent measurements have served as a source of information on the presence of particular low-molar-mass species that could then be quantified from the FTIR measurements [2].

In comparisons with the results from the ISO 9705 room measurements one needs to keep in mind that, with the exception of the CB combustion, combustible packaging materials were also involved in the combustions. On the whole, however, the product pattern appears to be very much the same from the two types of measurements. A more direct comparison of adsorbent results obtained from measurements in the two SP 'scales' is found in reference 1.

7.5.1 Survival fractions

A particular hazard in the event of fire in chemicals, such as for example pesticides, is the so-called survival fraction, *i.e.* unchanged original substance that may be carried with the smoke over long distances. In order to survive in the fire the compound itself must, of course, be thermally stable.

Polymers generally depolymerise or decompose upon heating and may yield toxic components in the process. These 'monomers' are hardly what is meant by survival fractions. A volatile compound such as CB rather easily escapes a fire unchanged, in particular when large amounts and high surrounding temperatures are involved. In fact, blanks taken prior to ignition of the CB sample generally contain significant amounts of evaporated CB. The rapid evaporation of the CB may, of course, be designated 'survival fraction'. But it is hardly one giving rise to other than short term effects in the environment.

Among the compounds involved in the SP studies TMTM and CNBA are the only ones that might be of potential interest in this context. CNBA is, however, not very stable thermally. For TMTM particular efforts were made to identify unchanged TMTM but without much success. An HPLC study of XAD fractions showed what might have been traces of TMTM. Not enough for proper identification though.

8 Conclusions

The storage configuration measurements have largely turned out as expected and provided valid, interesting results. In contrast to the ISO room tests the storage configuration experiments appear to have yielded prolonged periods of rather stable ventilation conditions.

As measured, conditions with values of ϕ in excess of 1.0 (under-ventilated) were not achieved in any of the storage configuration tests. The results in terms of the yields and types of species found in the smoke do, however, indicate that the actual fire was in itself under-ventilated and that the determinations of the degree of ventilation may somehow have been obscured by the large excess of air oxygen in the large-volume test enclosure. Or, that for comparable ϕ values differences in combustion temperature caused by more efficient cooling in the storage configuration enclosure has had a decisive influence on the product pattern. A detailed, more quantitative, comparison with the results from the ISO room tests is made in reference 1.

For various reasons, mostly economic, it was necessary to perform **all** of the experiments during just one-limited period in time, with the storage configuration tests directly following the ISO 9705 room tests without time for at least a brief evaluation of the ISO room results. This meant that no other than the most obvious corrective actions could be taken in the design and performance of the storage configuration experiments. In retrospect, somewhat larger amounts of the materials per experiment would have been a preferred choice. In spite of this, the storage configuration combustion experiments have yielded results that are overall quite satisfactory.

More detailed accounts of specific parts of the TOXFIRE work at SP are given in references 2 through 5. A summary of the total work and a more detailed discussion of the overall findings and results are given in reference 1.

9 **References**

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Appendix 1 Detailed information on the results

The test conditions were presented in Table 6.1 and in this Appendix 1, the main part of the evaluated results is presented in several tables. For each test a certain time period has been selected of rather steady state conditions was chosen. The mass losses (and MLR) were estimated according to the methods described in Section 4.6.5. All the data tabulated are mean values over the corresponding time period. Much of the results in Table A1.2-A1.4 are presented in graphical form in Appendix 3. The symbols and the abbreviations used in Appendix 1 are explained in Nomenclature.

Test id	Time period /min	φ	HRR /kW	MLR /(g/s)	-Δ h _c /(MJ/kg)	X	T _u /K	SPR /(m ² /s)
PP8	37-47	0.50	513	24.0	21.3	0.49	439	7.5
PP9	10-16	1.01	856	32.9	26.0	0.60	604	6.7
Ny6	6-14	0.43	444	25.3	17.5	0.60	386	0.75
Ny6	15-19	0.91	779	44.4	17.5	0.60	577	3.2
TMTM6	5-13	0.96	830	50.4	15.4	0.60	544	0.0
CNBA5	5-11	0.73	508	62.0	8.2	0.60	523	28.9
CB7	23-33	0.30	150	21.3	7.0	0.27	291	16.2

Table A1.1 Fire-related parameters

Table A1.2Yields of carbon oxides, THC and nitrogen oxides measured in the
opening.

Test id	Time period /min	φ	Y _{CO2} /(kg/kg)	Y _{co} /(g/kg)	Y _{тнс} /(g/kg)	Y _{NO} /(g/kg)	Y _{NO2} /(g/kg)	Y _{NOx} /(g/kg)
PP8	37-47	0.50	1.71	90	2.9	3.31	0.074	3.39
PP9	10-16	1.01	2.02	293	66.5	0.14	0.003	0.14
Ny6	6-14	0.43	1.44	8.7	1.3	2.72	0.029	2.75
Ny6	15-19	0.91	1.61	33	3.8	0.28	0.022	0.30
TMTM6	5-13	0.96	0.83	123	7.0	0.17	0.098	0.27
CNBA5	5-11	0.73	1.18	188	16.1	35.2	1.13	36.3
CB7	23-33	0.30	0.81	61	112	0.07	0.003	0.07

Table A1.3Yields of carbon oxides, soot, THC and nitrogen oxides measured in
the duct. The contribution from the cardboard boxes has not been
corrected for, but the contribution from the propane burner has been
subtracted (assuming that all the carbon gives CO_2 as product).

Test id	Time period /min	φ	Y _{co2} /(kg/kg)	Y _{co} /(g/kg)	Y _c /(g/kg)	Y _{тнс} /(g/kg)	Y _{NOx} /(g/kg)
PP8	37-47	0.50	1.48	82	31	17	5.91
PP9	10-16	1.01	1.68	175	20	160	0.04
Ny6	6-14	0.43	1.41	6.9	3	5.7	5.72
Ny6	15-19	0.91	1.38	24	7	16	1.27
TMTM6	5-13	0.96	0.67	74	0	23	3.21
CNBA5	5-11	0.73	0.92	106	47	40	66.9
CB7	23-33	0.30	0.65	48	76	172	0.21

Table A1.4Yields of HCN, NH_3 , HCl and SO_2 . measured in the opening (* indicates that the calibrated concentration span was exceeded.)

Test id	Time period /min	φ	Y _{HCN} /(g/kg)	Y _{NH3} /(g/kg)	Y _{нсі} /(g/kg)	Y _{so2} /(g/kg)
PP8	37-47	0.50	4.6	<0.05	-	
PP9	10-16	1.01	0.8	1.09	-	-
Ny6	- 6-14	0.43	1.6	0.39	-	-
Ny6	15-19	0.91	8.0	0.16	-	-
TMTM6	5-13	0.96	44.7*	0.68	-	634*
CNBA5	5-11	0.73	12.5*	<0.02	77.8*	-
CB7	23-33	0.30	<1.0	<0.05	141*	-

Appendix 2 Time dependent parameters

For each test there are two pages with a number of graphs. To make the comparisons easier the graph for a certain parameter always occupy the same position on the page. For some substances a certain product is not expected to be present (e.g. SO₂) and therefore blank graph positions can be found.

In the HRR graphs some important events have been indicated. That is

- b. The time period when the ignition burner was used. In PP8, the burner was turned on again after having been off for a while. However, this is still indicated with a solid line for the entire ignition period (see Appendix 3).
- ads. The time period when adsorbent measurements and soot sampling were performed in the opening. If the time periods did not coincide, two separate lines are drawn (CB7) where the soot sampling period simply is named "soot".
- soot see "ads."
- ME The moment when water application to extinguish the fire was started

 Δh_c is calculated from the experimental values of HRR and MLR. $\Delta h_{c,th}$ is either measured (PP) or calculated from literature data (CB).

Although the load cell with its electric cables was thermally insulated, the signal was after a while affected by the heat since the load cell was placed inside the SCTE and not outside the combustion room as it was during the ISO room tests [2]. For that reason, the MLR was calculated from the HRR when the total mass loss was known.

The temperature values from the thermocouple tree in one of the corners inside the SCTE (see Figure 4.1) have been used to represent the temperatures inside the test enclosure and are presented in the graphs. Tempertures at different heights above the floor are presented. The temperatures named $t_{opening}$ in the graphs were measured with a thermocouple situated in the upper half of the opening. These temperatures represent the temperatures of the outflowing gases.

All concentrations of the combustion products presented in the graphs are given in volume concentrations, [%] or [ppm].

The smoke production rate, SPR, is the same as "the instantaneous rate of light-obscuring smoke", R_{inst} [5, 16].

Polypropene, PP8







THC duct [v/v-ppm propane]

Polypropene, PP9













Time (min)



Time [min]

Total hydrocarbon, THC

THC duct [v/v-ppm propane]



Tetramethylthiuram monosulfide, TMTM6



THC duct [v/v-ppm propane]



4-chloro-3-nitrobenzoic acid, CNBA5





Chlorobenzene, CB7





NH3 below determination limit.



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Appendix 3 Detailed experimental observations

In Appendix 3 the experimental conditions for each test are given and the observations during the tests are presented. Information about changes in the experimental set-up or sampling strategy during a test or from one test to another, can also be found. The tests are given in order of performance.

If nothing else is stated, the soot sampling periods are the same as the time periods of sampling of organic components on adsorbents.

For the on-line measurement instruments, start and stop times are only mentioned when an instrument was not used during the whole test. When start and/or stop times for FTIR, FID and NO_x analyser are mentioned, these times refer to the measurements performed in the opening. NO_x was measured in the duct in almost all tests (when not mentioned in the comments for each test).

The times are given as minutes: seconds.

Date: 950908 Experiment id: PP7 - cartons in configuration (free burning)

Air temperature: 21.5 °C Relative humidity: 46 % Air pressure: 990 mbar

Test object: Polypropene (granules) in paper bags placed in compartmented cartons Amount of fuel: 130 kg - three cartons filled with only nine paper bags, the rest with 18 paper bags Fuel area: Fuel pan, 2 m x 1 m, on the floor Opening height: Free burning under the SP Industry Calorimeter

Comments:

Ignition source consisted of a 170 mm x 170 mm propane sand burner located centrally on the bottom of the pan. The surface sand bed was just below the corners of the four cartons. Flamc height of burner adjusted to height of storage. After half a minute the burner was shut off. PP7 was a screening test; no gas samples, other than those for the HRR calculations, were taken.

Observations during testing:

Time

-2:00	Measurements start
0:00	Ignition - the flames are adjusted to the height of storage
0:30	Ignition source off
0:43	Granules start to fall to the floor
2:00	A large amount of granules fall to the floor (over the burner)
2:56	Most of the cartons burn; 60-80 % of the granules have fallen down, the rest is
	burning on the shelves; the whole upper tier is burning; very little smoke
3:47	The whole storage is burning like one flame; most of the cartons are consumed

- 4:37 The flames reach a height of 5 m.
- 5:56 Black smoke; the radiation is unbearable 10 m from the fire
- 6:23 A fully developed pool fire
- 6:57 Flame height is about 6 m
- 8:56 Manually extinguished; a small amount of fuel is left on the shelves

Date: 950908 Experiment id: CB2 - one free burning carton

Air temperature: 21 °C Relative humidity: 48 % Air pressure: 990 mbar

Test object: Chlorobenzene in PEHD bottles in one carton Amount of fuel: 20 kg CB+PEHD + 2 kg cardboard; each PEHD bottle weighs 50 g Fuel area: Fuel pan 2 m x 1 m on the floor Opening height: Free burning under the SP Industry Calorimeter

Comments:

One carton on a shelf. Under the shelf a 2 m x 1 m fuel pan was placed to collect leaking CB. The ignition source consisted of a standardized ignition source positioned centrally 300 mm below the carton. 24 one litre PEHD bottles filled with CB were put on one level in a compartmented carton. CB2 was a screening test; no gas samples, other than those for the HRR calculations, were taken.

Observations during testing:

-2:00	Measurements start
0:00	Ignition with an ignition source; a pool fire is very soon developed
0:46	The flames reach over the top of the carton; the fire develops rapidly
1:15	Much grey/black smoke
1:50	The smoke gets thicker and more black. The carton is now fully involved in the
	flames and a large pool fire, only limited by the sides of the pan, has been
	created under the shelf
3:15	The smoke rises in large puffs. The flames do not reach very high, but the
	column of smoke is wide all the way up to the collector hood
4:44	The intensity is decreasing; the pool fire does not cover the whole pan
6:15	The smoke production has decreased considerably; the column of smoke is
	thinner and behave more calmly
12:30	Almost self extinguished, only very small fire on the position of the carton and a
	small pool fire exist

Date: 950922 Experiment id: PP8 - pool fire in the SCTE

Air temperature: 19 °C Relative humidity: 43 % Air pressure: 999 mbar

Test object: Polypropylene (granules) Amount of fuel: 42.5 kg (41 kg from PP6 and 1.5 kg new polypropene) Fuel area: 1.2 m² (square) Opening height: 0.89 m

Comments:

Ignition source consisted of a rectangular ring propane burner. The load-cell could not be placed outside the house as was done in the ISO 9705 tests [2], but had to be inside the test enclosure. Even if the cables etc. were insulated with ceramic insulation the mass signal was effected by the heat. An approximate correction was performed on the load cell signal before the differentiation. No adsorption sampling were performed, but all the on-line instruments described in Chapter 4 were used. The filter unit on the gas sampling line from the opening was not heated during this test.

Observations during testing:

-3:11	Measurements start
-0:11	Ignition burner on
0:00	Burner reaches the fuel
1:00	Burner turned away from the fuel
1:15	Burner reaches the fuel
1:26	Burner turned away from the fuel
1:38	Burner reaches the fuel
2:06	Burner turned away from the fuel
2:38	Burner reaches the fuel
2:45	Burner off; it is still burning very poorly, the flames barely reach the rim of the
	pan
12:00	Still almost no flames over the rim of the pan
15:00	The fire very slowly starts to grow; thin grey/black smoke is coming out through
	the opening
16:00	The amount of smoke out through the opening is increasing even if the flow still is rather low
18.00	The flames reach half a metre over the rim: it is only burning along the sides of
10.00	the pan
18:30	The smoke out through the opening is rather thick and occupy about half the
	opening
20:00	The flames reach 1.5 m over the rim; it is still burning mostly along the sides of
	the pan
22:00	The smoke layer inside the SCTE is getting thicker an descends over the fire.
	The upper parts of the flames are hard to see
22:45	Due to the smoke, flames can be seen only about 1 m above the pan

- 24:00 One can only perceive flames very close to the pan
- 25:00 Almost completely dark inside the SCTE
- 29:12 The smoke out through the opening is thick and occupy more than half the opening
- 59:00 It is discovered that a hole is burnt in the back wall; smoke was also leaking out through a hole in the ceiling
- 61:00 The amount of smoke out through the opening is decreasing
- 62:00 Cooling of the back wall
- 66:38 FTIR off
- 69:00 Much thinner smoke out through the opening and it does not occupy as large part of the opening as earlier
- 71:00 Almost no smoke at all out through the opening

Date: 950925 Experiment id: PP9 - storage configuration inside the SCTE

Air temperature: 19.5 °C Relative humidity: 39 % Air pressure: 991 mbar

Test object: Polypropene (granules) in paper bags placed in compartmented cartons Amount of fuel: 160 kg - eight cartons with 18 paper bags each Fuel area: Concrete fuel pan 4 m x 3 m on the floor Opening height: 0.89 m

Comments:

Ignition source consisted of a 170 mm x 170 mm propane sand burner located centrally on the bottom of the pan. The surface sand bed was just below the corners of the four cartons.

Observations during testing:

-3:03	Measurements start
0:00	Ignition burner on
0:28	Polypropene from the upper tier is falling down to the lower tier as the bags burn
1:00	The flames reach out to the sides on the upper tier
1:10	Burner off; the whole upper surface and the central parts of the lower tier are
	burning; the flames barely reach to the height of the lower side of the opening; almost no smoke out through the opening
1:50	Almost the whole upper surface of the lower tier is burning
1:57	Adsorption sampling on
2:00	The whole upper tier is involved in the flames
2:45	It is burning severely; the boxes on the upper tier can hardly be seen through the
	flames; the amount of smoke is increasing all the time
3:45	The smoke layer inside the SCTE is descending over the fire and it is hard to see
	the flames, no flames can be seen through the opening

4:17	Flames can be seen through the opening and the smoke out through the opening
	is not as thick as before
6:50	No flames can be seen through the opening
7:30	The upper tier of the post pallet system can not be seen
8:00	It is completely dark inside the SCTE and one can only just perceive some
	flames now and then
9:00	A distinct smoke layer can be seen in the opening; the smoke occupy about 3/4
	of the opening
11:57	Adsorption sampling off
15:00	The smoke production seems to decrease a little
17:00	FTIR off
19:25	Manually extinguished

Date: 950926 Experiment id: Ny6 - storage configuration inside the SCTE

Air temperature: 21 °C Relative humidity: 40 % Air pressure: 984 mbar

Test object: Nylon 66 (pellets) in paper bags placed in compartmented cartons Amount of fuel: 160 kg - eight cartons with 18 paper bags each Fuel area: Concrete fuel pan 4 m x 3 m on the floor Opening height: 0.89 m

Comments:

Ignition source consisted of a 170 mm x 170 mm propane sand burner located centrally on the bottom of the pan. The surface sand bed was just below the corners of the four cartons. The positions of the GSP and the PMP were by mistake exchange, which means that in the present test the GSP had the holes turning inwards while the PMP had the holes in the opposite direction.

Observations during testing:

-3:09	Measurements start
0:00	Ignition burner on; the flames just barely reach the lower side of the opening
0:51	Adsorption sampling on
1:00	The flames reach out to the middle of each side of the upper tier
1:12	Burner off; Nylon from the upper tier is falling down to the lower tier as the
	bags burn
1:30	The whole upper surface of the upper tier is burning
1:40	The flames reach out to the middle of each side of the lower tier
2:15	The whole upper surface of the lower tier is burning; the fire on the upper tier
	has started to decrease; nylon continues to fall down from the upper tier;
2:25	The flames do not even reach the lower side of the opening; no smoke out
	through the opening
3:00	Very thin smoke out through the opening

- 4:00 The upper tier hardly burns at all and it is only burning a little on the lower tier 5:00 On the upper tier the only thing remaining are black piles of burned cardboard; it is burning very little; most of the nylon originally on the upper tier has now fallen down to the lower tier 5:30 The intensity of the fire on the lower tier is increasing a little 8:00 The fire on the lower tier has increased further. It is also burning a little on the upper tier but it seems to be an effect of the flames from the lower tier 8:30 The flames reach now and then to the height of the lower side of the opening 9:00 The whole post pallet system is now involved in the flames 10:30 The flames reach the ceiling 11:00 The intensity continues to increase 14:45 The amount of smoke out through the opening has increased a little The black smoke starts to conceal the highest flames 16:00 17:00 Black smoke out through the opening 17:30 It is hard to see the upper tier due to the smoke 18:51 Adsorption sampling off; FTIR off The smoke is concealing everything inside the SCTE 20:00 21:00 FID and NO, analyser off
- 21:08 Manually extinguished

Date: 950926	Experiment id:	TMTM6 - storage	configuration	inside the	SCTE
	Daperment	Initiatio broinge	comparation		

Air temperature: 20 °C Relative humidity: 36 % Air pressure: 984 mbar

Test object: Tetramethylthiurammonosulfide (TMTM) (granules) in paper bags placed in compartmented cartons Amount of fuel: 160 kg - eight cartons with 18 paper bags each Fuel area: Concrete fuel pan 4 m x 3 m on the floor with a steel pan 2 m x 2 m under the post pallet system. Opening height: 0.89 m

Comments:

Ignition source consisted of a 170 mm x 170 mm propane sand burner located centrally on the bottom of the pan. The whole post pallet system was elevated 0.20 m. Under the post pallet system a smaller pan (steel) was put to make the pool fire smaller and the cleaning up easier. The distance from the pan to the bottom side of the boxes on the lower tier was now 0.35 m.

Observations during testing:

- -3:00 Measurements start
- 0:00 Ignition burner on
- 0:30 The flames reach out to the middle of each side of the upper tier; TMTM from the upper tier is falling down to the lower tier as the bags burn

1:00	Burner off; adsorption sampling on
1:05	The flames reach out to the middle of each side of the lower tier
1:10	The whole upper surface of the upper tier is burning
1:30	The whole upper surface of the lower tier is burning; It is burning severely
1:50	Thin grey/blue smoke out through the opening ; the amount of smoke is increasing
2:30	The whole upper tier is involved in the flames
3:20	No smoke at all out through the opening; the intensity of the fire has decreased
3:45	The fire has changed; some purple element in the flames can be seen and the flames are fine and thin; this probably means that more of the TMTM is burning (see reference 2); the intensity seems to stabilize on about the same ventilation controlled level as in the tests PP9 and Ny6, but the development periods up to this point looked different in the three tests
13:00	FTIR and adsorption sampling off
13:50	The grey/blue smoke is coming back, but it is not as much smoke as in the beginning of the test
15:00	FID and NO_x analyser off
16:02	Manually extinguished (16:08)

Date: 950927	Experiment id:	CNBA5 - storage	configuration	inside the	SCTE

Air temperature: 18 °C Relative humidity: 38 % Air pressure: 983 mbar

Test object: Chloronitrobenzoic acid (CNBA) (powder) in paper bags placed in compartmented cartons Amount of fuel: 160 kg - eight cartons with 18 paper bags each Fuel area: Concrete fuel pan 4 m x 3 m on the floor with a steel pan 2 m x 2 m under the post pallet system Opening height: 0.89 m

Comments:

Ignition source consisted of a 170 mm x 170 mm propane sand burner located centrally on the bottom of the pan. The same set-up as in TMTM6 with a second smaller pan under the post pallet system was used. The thermocouples over the post pallet system and in the ceiling was not working due to a melted connecting cable leading to a change in the measuring positions of the thermocouples. The piles after the test on the positions of the boxes were about as big as in the other storage configuration tests, but it looked as if the piles on the floor under the post pallet system were larger.

Observations during testing:

- -3:02 Measurements start
- 0:00 Ignition burner on
- 0:25 CNBA from the upper tier is falling down to the lower tier as the bags burn

0:35	The flames reach out to the middle of each side of the upper tier
0:50	Material falls down in the middle of the post pallet system and this leads to a
	sudden increase in the intensity; a lot of smoke suddenly comes out and fills the
	opening
0:58	Adsorption sampling on
1:00	Burner off; the whole upper surface of the upper tier is burning; the flames reach
	out to the middle of each side of the lower tier
1:05	Several bags on the upper tier fall down to the lower tier increasing the fire
	intensity
1:20	A whole box on the upper tier breaks and much substance falls down to the
	lower tier and to the floor; The whole upper surface of the lower tier is burning;
	the whole upper tier is involved in the flames; a thick layer of smoke is
	descending over the fire
1:33	The smoke layer reaches the highest point of the posts of the post pallet system
1:40	The smoke layer hides the whole upper tier
2:05	It is completely dark inside the SCTE
2:20	Much smoke comes out through the opening; the smoke is almost rolling like
	large balls
4:10	Much less smoke comes out through the opening
4:16	Flames can be seen through the opening, but only for a short while until the
	amount of smoke increase to the same level as earlier
7:10	A large puff of smoke comes out through the opening and goes up into the
	collector hood
7:25	A new puff of smoke, but it is this time immediately followed by a larger puff of
	smoke and flames straight out from the opening
7:32	The smoke from the puff has disappeared and no smoke at all can be seen
	through the opening only fine, thin flames inside the SCTE
8:00	Smoke is coming out through the opening again
9:40	The flow of smoke starts to pulsate
10:50	The flow of smoke is violent for a short while
10:58	Adsorption sampling off
11:22	A new large puff of smoke and flames is coming straight out from the opening
11:32	No smoke can be seen through the opening
11:32	A minor puff of smoke comes straight out from the opening; flames can be seen
	inside the SCTE, but it gets dark very quickly
11:50	Manually extinguished

Date: 950928 Experiment id: CB7 - pool fire inside the SCTE

Air temperature: 16 °C Relative humidity: 42 % Air pressure: 975 mbar

Test object: Chlorobenzene (liquid) Amount of fuel: 40 kg Fuel area: 0.8 m² (square) Opening height: 0.89 m

Comments:

Ignition source consisted of a with heptane soaked cloth on a long pole. The thermocouples over the post pallet system and in the ceiling was not working due to a melted connecting cable leading to a change in the measuring positions of the thermocouples. After the test there was very much soot inside the SCTE, a several millimetre thick layer on the walls and large piles on the floor. There was however a 0.8 m wide band around the inside of the SCTE with no soot at all. This band started 1.25 m above the floor.

Observations during testing:

-3:00	Measurements start
0:00	Ignition with an ignited cloth soaked with heptane on a pole
0:38	The igniting cloth is removed
1:00	Grey smoke starts to come out through the opening
1:30	Rather much black smoke out through the opening
1:45	The smoke layer is very quickly descending over the fire
2:00	Adsorption sampling on ; completely dark inside the SCTE
12:00	Adsorption sampling off
19:00	FTIR off
22:00	FTIR on
44:00	The amount of smoke has decreased
50:00	The amount of smoke out through the opening is now little
56:00	Almost no smoke out through the opening at all (extinguished)