ASSESSMENT OF THE THERMAL AND TOXIC EFFECTS OF CHEMICAL AND PESTICIDE POOL FIRES BASED ON EXPERIMENTAL DATA OBTAINED USING THE TEWARSON APPARATUS

Christian Costa, Guy Treand and Jean-Louis Gustin
Rhône-Poulenc Industrialisation, 24 avenue Jean-Jaurès – 69153 Décines - France

The Tewarson apparatus is a combustion calorimeter developed by Factory Mutual Research Co. USA, in the 1970s. A modified and computerised version of this calorimeter is used at the Rhône-Poulenc Decines Centre to study the combustion of plastics, fabrics, chemicals and pesticides on 30 grams samples in a 0.1 metre diameter glass dish.
The combustion of up to 100 products has been studied in this experimental set-up and the following thermal data obtained : mass of product burnt, experimental heat of combustion, combustion efficiency, burning mass flux, ratios of convection and radiant heat, flame height, flame temperature.
The on-line analysis of combustion gases provides the following chemical data : production of CO_2, CO, HCN, NO_2, NO, SO_2, HCl, HF, HBr, chemical yield for the combustion of carbon, nitrogen, sulphur, chlorine, fluorine, bromine.
The thermal data obtained is an input to the POOL 2.0 Computer code to estimate the thermal effect of chemical pool-fires.
The combustion chemical data obtained is an input to atmospheric dispersion codes to estimate the toxic effect of chemical pool fires.
The correlation of experimental data obtained using the Tewarson apparatus, based on the sample chemical formulae helps provide the missing combustion data. As an example, a correlation is given for the combustion characteristics of chlorinated organic chemicals.

Keywords : Pool fires, combustion data, Tewarson apparatus, combustion efficiency, chemical yield.

Introduction

The assessment of fire hazards in chemical and pesticide storages and warehouses is based on both the determination of the material combustion thermal data and the identification of the toxic emission from combustion gases.

The combustion thermal data is the input data required in fire simulation softwares to estimate the consequences of industrial fires. This combustion thermal data includes the determination of the mass of product burnt, the experimental heat of combustion, the combustion efficiency, the burning mass flux, the ratios of convection and radiant heat, the flame height and flame temperature. (1) (2).

The simulation of large industrial fires provides information on the thermal effect of the accidental fire on adjacent equipment and on the protection needed to prevent the fire from spreading.

Large industrial fires are also source-terms for modelling atmospheric dispersion of volatile toxic combustion products. The input data to atmospheric dispersion models are combustion chemical data including the production of combustion gases Cl_2, CO, HCN, NO_2, NO, SO_2, HCl, HBr, depending on the burning material composition. Also necessary is the determination of the chemical yield for the combustion of the chemical elements present in the burning material formula : Carbon, Nitrogen, Sulphur, Chlorine, Fluorine, Bromine if any (3).

The thermal and chemical data characterizing the combustion of chemicals and pesticides can only be obtained using a bench-scale apparatus, due to the great number of experiments to be performed on a wide range of products.

Such a combustion calorimeter was developed by A. TEWARSON at Factory Mutual Research Corporation (USA) in the 1970s (4). A modified and computerized version of the Tewarson apparatus was built at the Rhone-Poulenc Decines Centre to study the combustion of plastics, fabrics, chemicals and pesticides on 30 grams samples. This new experimental set-up is described in the following section with special attention to the improvement of the original design.

Description of the modified tewarson combustion calorimeter

The principle of the modified Tewarson combustion calorimeter built in Decines is shown on figure 1.

The experimental set-up may be divided in three sections :

- The lower part of the apparatus is the combustion chamber, section A on figure 1.
  The combustion chamber consists of a standing cylindrical quartz tube 0.160 metre in diameter and 0.490 metre high. In this combustion chamber, a 30 grams sample in a 0.1 metre diameter glass dish is placed on the plate of a balance to measure the sample weight during combustion experiments.
  An external heat-flux is applied to the combustion chamber by eight infra-red heaters in an air flushed jacket, allowing the sample to be heated to a temperature where its vapours or fumes can be ignited by an ignition source. The maximum external heat-flux applied to the combustion chamber is 30 kW/m^2. The ignition of the sample by an electric spark was preferred to the original pilot-flame ignition source, to avoid additional heat input and combustion gases to the experiments.

  The ignition of the sample is obtained without external heat-flux applied if the sample is flammable under ambient temperature. If not, an external heat-flux is applied to raise the sample temperature until ignition is obtained.

  If the sample combustion is self-sustained, the sample is allowed to burn without external heat-flux applied. If not, an external heat-flux is applied to allow the sample combustion, in which case the radiant heat-flux cannot be measured. Preliminary experiments are necessary to choose the most suitable operating conditions.

  A permanent air flow of 5 m^3/h is blown to the combustion chamber bottom through a glass sphere bed to obtain homogeneous inlet gas composition and regular air stream.

  The inlet air is under flow control and its oxygen concentration may be varied by adding oxygen or nitrogen to the air flow. The combustion air composition is measured by a continuous oxygen analyzer during experiments.

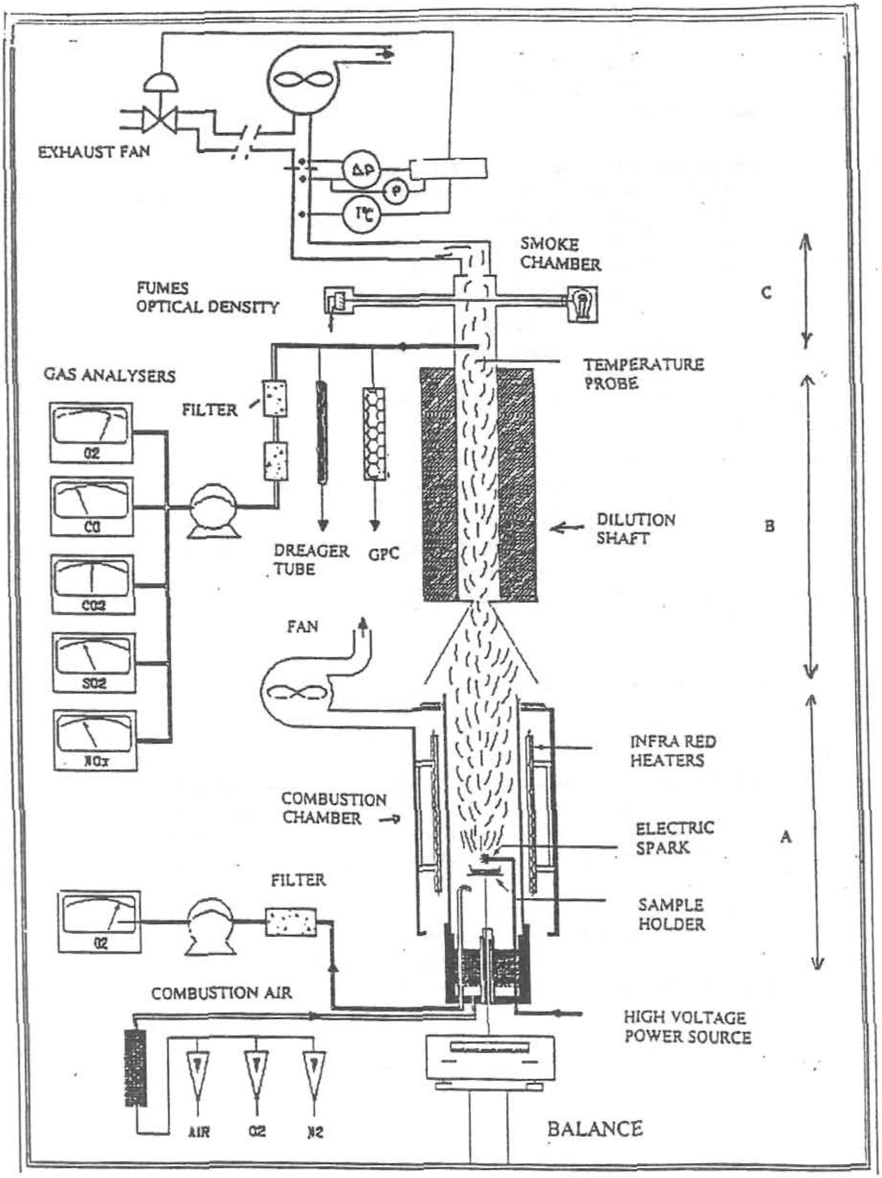

Figure 1 : Modified Tewarson apparatus

A calibration of the external heat input to the sample is achieved by replacing the sample holder by a heat-flux meter. The heat-flux received by the sample is measured as a function of the power input to the infra-red heaters. This calibration allows the compensation of the infra-red heater aging by an increased electric power supply to the external heating device.

- The intermediate part of the apparatus is the dilution shaft, section B on figure 1.
  The dilution shaft is a standing Teflon cylinder 0.1 metre in diameter and 0.6 metre high intended to dilute and mix the flow of combustion gases and smokes with air. Teflon was preferred to stainless steel, to avoid soot deposits which could absorb contaminants such as HCl, HCN, dioxines, etc . The dilution air inlet flow to the dilution shaft is controlled by the exhaust fan mass flow rate of 70 kg/h.

  The aim of the combustion products dilution is to avoid losses of volatile combustion products by condensation or leaks, while limiting the dilution ratio to keep the oxygen concentration analysis accurate and controlling heat losses from combustion products. Mixing of combustion products with dilution air is achieved by the convergent nozzle at the dilution shaft bottom.

  At the top of the dilution shaft, the smoke temperature is measured by a thermocouple and the gas flow is sampled for continuous on-line analysis.

  The on-line gas analysis of the diluted smoke includes the determination of O_2, CO, CO_2, SO_2 and NOx. This is achieved after passing the gas sample over a filter and a desiccant. Other analysis are performed after absorption of the gas on a Draeger tube followed by mass spectrometer analyses or after absorption on resins followed by gas chromatography.

- The upper part of the Tewarson apparatus is the smoke chamber, section C on figure 1.
  This device, intended to measure the optical density of smoke, is equipped with an external photoelectric system to measure the optical density in a standing stainless steel cylinder 0.1metre in diameter and 0.3 metre high, extending the combustion shaft. The smoke chamber exit is connected to the exhaust fan inlet. The fan flow control principle is shown on fig. 1. The fan volumetric flow rate is adjusted taking into account the smoke temperature to obtain a constant mass flow rate of exhaust gases.

- Data acquisition and processing system
  This section includes a CHESSEL recorded / converter and a graphic data treatment allowing display of the variation of the measured parameters as a function of time. A test result sheet is produced, giving the most important test characteristics and results.

Experimental results

To date, up to 100 products have been studied using the modified Tewarson apparatus. An overview of the results obtained on well known chemicals and pesticides is given in tables where the following data is listed under the product current name :

- Gross chemical formula	
- Molecular weight	(kg/kmole)
- External heat flux applied	(kW/m^2)
- Initial sample mass	(g)
- Mass fraction of product burnt	(%)
- Net calorific value	(kJ/kg)
- Heat of combustion per kg of product burnt	(kJ/kg)
- Heat of combustion per kg of sample	(kJ/kg)
- Combustion thermal efficiency i.e.The ratio of the combustion heat measured on 1 kg initial sample to the net calorific value of this 1 kg sample.	(%)
- Average combustion mass flux	(g/m^2.s)
- Maximum combustion mass flux	(g/m^2.s)
- Ratio of convection heat to combustion heat	(%)
- Ratio of radiant heat to combustion heat	(%)
- Production of CO_2, CO, HCN, NO_2, NO, SO_2, HCl	(g of gas / kg of sample)
- Chemical yield for the conversion of carbon into CO_2 , CO and HCN i.e.The ratio of the carbon contained in the CO_2 CO and HCN produced by 1 kg sample to the carbon present in that 1 kg sample.	(%)
- Chemical yield for the conversion of nitrogen into NO2, NO and HCN i.e.The ratio of the nitrogen contained in the NO2, NO and HCN produced by 1 kg initial sample to the nitrogen present in that 1 kg sample.	(%)
- Chemical yield for the conversion of chlorine into HCl i.e.The ratio of the chlorine contained in the HCl produced by 1 kg sample to the chlorine present in that 1 kg sample	(%)
- Maximum flame height	(cm)
- Maximum flame temperature	(°C)
- Specific extinction area i.e.The surface darkened by 1 kg of fuel, deduced from the measured optical density.	(m^2/kg)

The detailed calculation methods for the combustion heat, convection and radiant heat, opacity of smokes and other parameters are given in reference (5) and are not reproduced in the present paper. The total combustion heat was deduced from the oxygen depletion during combustion according to Thornton (6) (7). The convection heat is the heat carried out in the gas plume over the combustion chamber. The radiant heat is deduced from the total combustion heat, the convection heat and an estimation of the heat-losses obtained from calibration experiments.

The combustion data for some chemicals and solvent is given in table 1.

The combustion data for some pesticides is given in table 2.

Table 1 : Combustion characteristics of chemicals and solvents, obtained in the modified Tewarson apparatus

Parameters	units	ACETONE	ACETO NITRILE	ISOPROPYL ACETATE	CYCLOHEXANE
Gross chemical formula		C_3H_6O	C_2H_3N	$C_5H_{10}O_2$	C_6H_{12}
Molecular weight	kg/kmol	58	41	102	84
External heat-flux applied	kW/m^2	0	0	0	0
Initial sample mass	g	22	33	22	21
Mass fraction of product burnt	%	100	100	100	100
Net calorific value	kJ/kg	28 560	29 580	25 995	43 388
Heat of combustion /kg product burnt	kJ/kg	27 500	29 000	24 220	42 570
Combustion thermal efficiency	**%**	**96**	**97**	**93**	**98**
Mean combustion mass-flux	$g/m^2.s$	12	11	12	14
Maximum combustion mass-flux	$g/m^2.s$	14	12	14	18
Ratio of convection heat	%	56	55	53	47
Ratio of radiant heat	%	16	22	20	21
Production of CO_2 / kg sample	g/kg	2 188	2 040	2 004	2 834
Production of CO / kg sample	g/kg	17	25	22	29
Production of HCN / kg sample	g/kg	-	0.2	-	-
Chemical yield for carbon	**%**	**97**	**97**	**94**	**92**
Production of NO_2 / kg sample	g/kg	-	1	-	-
Production of NO / kg sample	g/kg	-	19	-	-
Production of HCN / kg sample	g/kg	-	0.2	-	-
Chemical yield for N_2	%	-	3	-	-
Production of SO_2 / kg sample	g/kg	-	-	-	-
Chemical yield for sulphur	%	-	-	-	-
Production of HCl / kg sample	g/kg	-	-	-	-
Chemical yield for Cl_2	%	-	-	-	-
Flame height	cm	30	30	30	40
Flame maximum temperature	°C	816	865	796	812
Specific extinction area	m^2/kg	0	35	0	0

Table 1 (continued) : Combustion characteristics of chemicals and solvents, obtained in the modified Tewarson apparatus

Parameters	*units*	ETHYL ALCOHOL	DIISOPROPYL ETHER	HEXANE	METHYL ALCOHOL	TETRA HYDROFURAN
Gross chemical formula		C_2H_6O	$C_6H_{14}O$	C_6H_{14}	CH_4O	C_4H_8O
Molecular weight	kg/kmol	46	102	86	32	72
External heat-flux applied	kW/m^2	0	0	0	0	0
Initial sample mass	g	23	23	20	20	22
Mass fraction of product burnt	%	100	100	100	100	99.8
Net calorific value	kJ/kg	26 810	36 194	44 740	19 892	32 200
Heat of combustion /kg product burnt	kJ/kg	26 450	35 010	42 210	18 800	30 320
Combustion thermal efficiency	**%**	**99**	**96**	**96**	**96**	**94**
Mean combustion mass-flux	$g/m^2.s$	9	17	15	8	14
Maximum combustion mass-flux	$g/m^2.s$	10	21	19	9	17
Ratio of convection heat	%	54	47	52	59	53
Ratio of radiant heat	%	20	25	22	14	18
Production of CO_2 / kg sample	g/kg	1 843	2 378	2 868	1 330	2 290
Production of CO / kg sample	g/kg	0	21	33	0	21
Production of HCN / kg sample	g/kg	-	-	-	-	-
Chemical yield for carbon	**%**	**96**	**93**	**95**	**97**	**95**
Production of NO_2 / kg sample	g/kg	-	-	-	-	-
Production of NO / kg sample	g/kg	-	-	-	-	-
Production of HCN / kg sample	g/kg	-	-	-	-	-
Chemical yield for N_2	%	-	-	-	-	-
Production of SO_2 / kg sample	g/kg	-	-	-	-	-
Chemical yield for sulphur	%	-	-	-	-	-
Production of HCl / kg sample	g/kg	-	-	-	-	-
Chemical yield for Cl_2	%	-	-	-	-	-
Flame height	cm	25	> 40	> 40	15	40
Flame maximum temperature	°C	843	768	782	780	811
Specific extinction area	m^2/kg	0	0	0	0	0

Note : n.m. = not measured

Table 1 *(continued)* : *Combustion characteristics of chemicals and solvents, obtained in the modified Tewarson apparatus*

Parameters	**units**	**XYLENE**	**TOLUENE**	**PROPYLENE GLYCOL**	**PHENOL**	**DIETHYL OXALATE**
Gross chemical formula		C_8H_{10}	C_7H_8	$C_3H_8O_2$	C_6H_6O	$C_6H_{10}O_4$
Molecular weight	kg/kmol	106	92	76	94	146
External heat-flux applied	kW/m^2	0	0	0	0	0
Initial sample mass	g	30	30	28	30	30
Mass fraction of product burnt	%	100	100	100	100	100
Net calorific value	kJ/kg	*40 900*	*40 550*	*21 630*	*31 000*	*18 600*
Heat of combustion /kg product burnt	kJ/kg	27 100	29 055	21 310	27 625	17 700
Combustion thermal efficiency	**%**	**66**	**71**	**99**	**89**	**95**
Mean combustion mass-flux	g/m^2.s	18	35	6	12	9
Maximum combustion mass-flux	g/m^2.s	25.8	46	7	15	11
Ratio of convection heat	%	37	35	62	43	57
Ratio of radiant heat	%	29	27	28	33	25
Production of CO_2 / kg sample	g/kg	2 170	2 184	1 664	2 625	1 819
Production of CO / kg sample	g/kg	73	76	21	57	18
Production of HCN / kg sample	g/kg	-	-	-	-	-
Chemical yield for carbon	**%**	**69**	**69**	**98**	**96**	**100**
Production of NO_2 / kg sample	g/kg	-	-	-	-	-
Production of NO / kg sample	g/kg	-	-	-	-	-
Production of HCN / kg sample	g/kg	-	-	-	-	-
Chemical yield for N_2	%	-	-	-	-	-
Production of SO_2 / kg sample	g/kg	-	-	-	-	-
Chemical yield for sulphur	%	-	-	-	-	-
Production of HCl / kg sample	g/kg	-	-	-	-	-
Chemical yield for Cl_2	%	-	-	-	-	-
Flame height	*cm*	*35*	*40*	*16*	*32*	*24*
Flame maximum temperature	°C	630	715	826	758	850
Specific extinction area	m^2/kg	1 738	1 600	0	1 000	0

Table 2 : Combustion characteristics of various pesticides, obtained in the modified Tewarson apparatus

Parameters	**units**	**DIURON**	**ISO PROTURON**	**ACID 2,4 D**	**MANCOZEBE**
Gross chemical formula		$C_9H_{10}ON_2Cl_2$	$C_{12}H_{18}ON_2$	$C_8H_6O_3Cl_2$	$(C_4H_6N_2S_4Mn)_1 Zn_{0.1}$
Molecular weight	kg/kmol	233	206	221	269
External heat-flux applied	*kW/m²*	*25*	*25*	*25*	*25*
Initial sample mass	g	30	30	15	30
Mass fraction of product burnt	*%*	*99.7*	*100*	*100*	*60.9*
Net calorific value	kJ/kg	20 300	32 800	(11 540)	14 000
Heat of combustion /kg product burnt	kJ/kg	10 240	23 870	4 500	15 590
Heat of combustion / kg sample		10 210	23 870	4 500	9 500
Combustion thermal efficiency	**%**	**51**	**72**	**(39)**	**69**
Mean combustion mass-flux	*g/m².s*	*26*	*28*	*24*	*5*
Maximum combustion mass-flux	g/m².s	45	50	n.m.	16
Ratio of convection heat	*%*	*38*	*30*	*63*	*44*
Ratio of radiant heat	%	n.m.	n.m.	n.m.	n.m.
Production of CO_2 / kg sample	g/kg	760	1 704	504	498
Production of CO / kg sample	g/kg	80	56	74	0
Production of HCN / kg sample	g/kg	12	7	-	0
Chemical yield for carbon	**%**	**53**	**70**	**39**	**76**
Production of NO_2 / kg sample	g/kg	2	1	-	1
Production of NO / kg sample	g/kg	3.5	8	-	7
Production of HCN / kg sample	g/kg	12	7	-	1
Chemical yield for N_2	%	7	7	-	4
Production of SO_2 / kg sample	g/kg	-	-	-	535
Chemical yield for sulphur	%	-	-	-	56
Production of HCl / kg sample	g/kg	144	-	121	-
Chemical yield for Cl_2	%	46	-	37	-
Flame height	cm	n.m.	n.m.	n.m.	n.m.
Flame maximum temperature	°C	*n.m.*	*n.m.*	*n.m.*	*n.m.*
Specific extinction area	m²/kg	1 600	1 160	1 640	0

Note : n.m. = not measured

The net calorific value of 2,4 D acid was not available and was deduced from the combustion chemical yield for the conversion of carbon.

Combustion properties as a function of time

In addition to integral combustion data deduced from experiments, dynamic and time dependent properties are also obtained from combustion experiments performed in the modified Tewarson apparatus.

As an example, the combustion heat-flux during the combustion of a 30 g sample of cyclohexane is given as a function of time on figure 2. In this figure, the combustion heat per unit time or combustion thermal power is estimated using the oxygen consumption deduced from the oxygen depletion measured in the fumes. The reference liquid sample area is that of the glass dish.

The convection heat-flux measured in the same experiment is given as a function of time on figure 3. The convection heat was deduced from the diluted fumes mass-flow and temperature. The reference area for the estimation of the convection heat-flux is that of the glass dish.

The radiant heat-flux is deduced from heat-flux measurements and calibration experiments using the infra-red external heating device. The radiant heat-flux during the combustion of a cyclohexane sample is given as a function of time on figure 4. The reference area for the estimation of the radiant heat-flux is that of the glass dish.

Figures 2-3-4 show that the combustion heat-flux is not the sum of the convection heat-flux and radiant heat-flux. The missing heat is the experimental set-up heat-losses or conduction heat, mainly due to the combustion chamber quartz tube heat capacity.

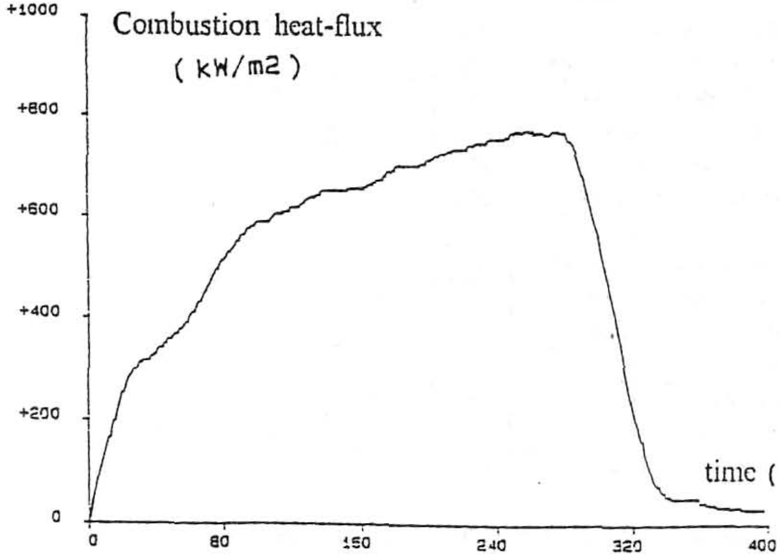

Figure 2 : Combustion heat-flux as a function of time measured during the combustion of a 30 g sample of cyclohexane in the modified Tewarson apparatus.

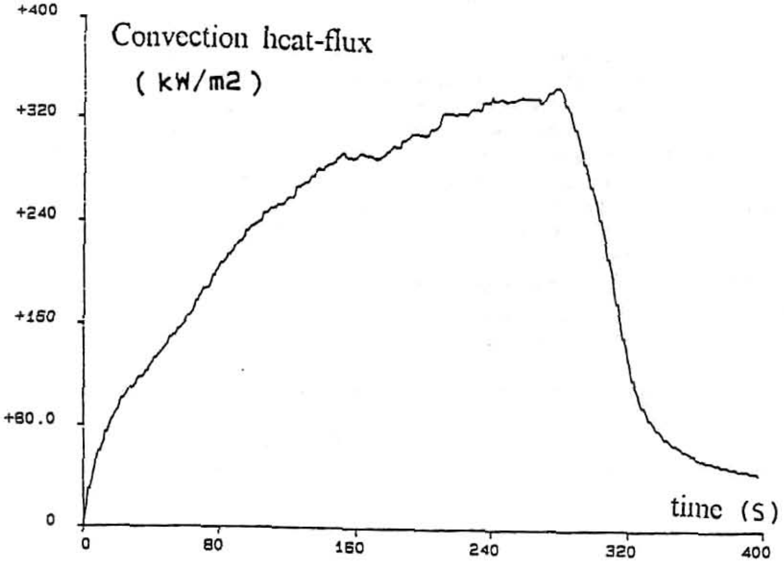

Figure 3 : Convection heat-flux as a function of time measured during the combustion of a 30 g sample of cyclohexane in the modified Tewarson apparatus.

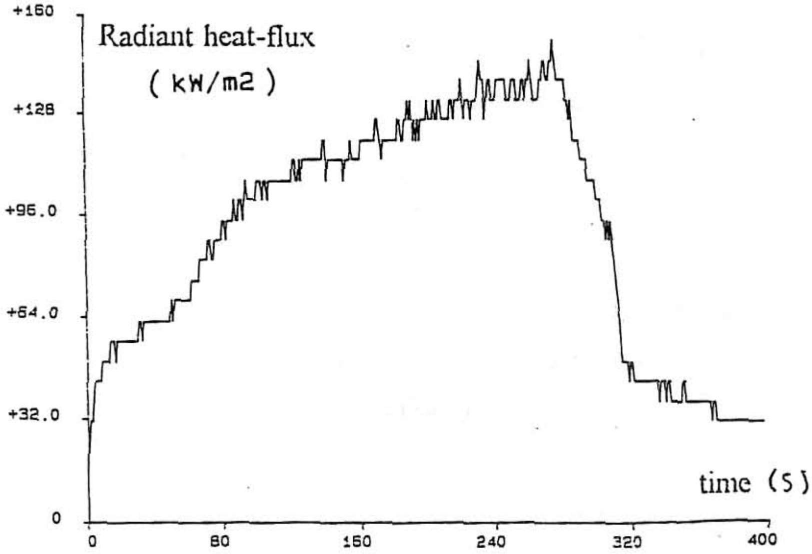

Figure 4 : Radiant heat-flux as a function of time measured during the combustion of a 30 g sample of cyclohexane in the modified Tewarson apparatus.

Influence of the chemical formula on the combustion thermal data

The experimental results obtained on the chemicals and pesticides studied in the modified Tewarson apparatus, show that the combustion thermal data is influenced by the chemical formula. For the most useful combustion characteristics used as input data for the simulation of large industrial fires i.e. the combustion thermal efficiency, the chemical *yield for the conversion of carbon, the ratio of radiant heat, which were defined in the* previous section, recommended specific values can be deduced from the experimental results obtained on the different types of chemicals. A summary of the thermal data suggested is given in table 3 for aliphatic derivatives, in table 4 for C, H, O, N aromatic and unsaturated cyclic compounds and in table 5 for miscellaneous organic compounds including chlorinated and fluorinated organic compounds.

The combustion thermal data in table 3, 4 and 5 may be used as a first estimate in fire simulation softwares such as the POOL 2 program in the absence of specific experimental data.

Table 3 : Suggested values of thermal data for aliphatic derivatives

Chemical compound	**Combustion thermal efficiency (%)**	**Chemical yield for carbon (%)**	**Ratio of radiant heat (%)**
Alkanes	97	93	22
Alcohols	98	97	22
Esters, Ethers, Ketones	96	95	23
Nitriles	*98*	*98*	*26*
Amines	91	82	12
C, H, O, N compounds	95	90	22
C, H, O, S compounds	95	91	/

Table 4 : Suggested thermal data for aromatic and unsaturated cyclic compounds

Chemical compound	**Combustion thermal efficiency (%)**	**Chemical yield for carbon (%)**	**Ratio of radiant heat (%)**
C, H, O, N compounds	70	70	30
C, H, O, N aniline derivatives	75	75	40
C, H, O, N, nitro derivatives	*40*	*45*	*40*

Table 5 : Suggested thermal data for miscellaneous organic compounds

Chemical compound	**Combustion thermal efficiency (%)**	**Chemical yield for carbon (%)**	**Ratio of radiant heat (%)**
Monochlorinated compounds with C, H, N, Cl atoms	55	51	35
Polychlorinated compounds with C, H, O, Cl atoms	50	50	40
C, H, N cyclic compounds	90	95	40
Fluorinated aromatic compounds	55	*55*	*40*

Influence of the fuel chemical formula on the toxic emission of fires

The combustion chemical data obtained in the modified Tewarson apparatus, on a great number of chemical compounds can be used to estimate the chemical yield for the conversion of Nitrogen, Sulphur, Chlorine, Fluorine, during combustion. This allows the prediction of missing data for chemical compounds which have not yet been studied in combustion experiments. As an example the chemical yield for the conversion of Chlorine into HCl during the combustion of chlorinated organic compounds was studied on a selection of 25 chlorinated organic derivatives. A summary of the results obtained is given in table 6 for chlorinated and poly-chlorinated derivatives. The data in table 6 can be used as a first estimate of the chemical yield for the conversion of chlorine into HCl, to replace missing data on specific chemical compounds for which no experimental data is available. The chemical yield for the conversion of hetero atoms is an input data to atmospheric dispersion models.

Table 6 : Suggested values of chemical yield for the conversion of chlorine into HCl during the combustion of chlorinated organic compounds

Type of chlorinated compound. Number and position of chlorine atoms	Chemical yield for the conversion of Chlorine into HCl during combustion
One Cl atom per chain or ring	80
Two Cl atoms per chain or ring	40
3 Cl atoms per chain or ring	20
4 Cl atoms per chain or ring	0
5 or 6 Cl atoms per chain or ring	0
3 Cl atoms on the same carbon (- C Cl_3)	100 %
Amine hydrochloride (RNH_2 . HCl)	95 %

The definition of the chemical yield for the conversion of chlorine in table 6 is given in chapter 3 above.

Conclusion

The modified Tewarson apparatus described in this paper, is a useful experimental set-up to study the combustion of chemicals and pesticides. To date, this apparatus has been used to investigate more than 100 different chemicals and pesticides. The thermal and chemical data obtained was used as input data for computer simulation of large industrial fires and for atmospheric dispersion calculations to evaluate the toxic impact of industrial fires.

The modified Tewarson apparatus is also a valuable tool to compare the combustion behaviour of plastics, resins and fabrics while determining the nature of the combustion products.

The modified Tewarson apparatus was used as a bench scale apparatus in the STEP European Program to study fires and their consequences, in the **MISTRAL** 1 program. The partners associated with this European program are :

CEA / IPSN	(France)
CISI	(France)
CNRS / LCRS	(France)
ENEA / AEAS	(Italy)
ISSEP	(Belgium)
Rhône-Poulenc Industrialisation	*(France)*
University of Aveiro	(Portugal)
University of Poitiers	(France)

other partners who joined this project in the course of the program are :

EDF / CLI	(France)
INERIS	(France)

It was shown in the course of this European project that the modified Tewarson apparatus was a key item to investigate the consequences of large chemical fires on a wide range of chemicals and pesticides.

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