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Formation of Polychlorinated Dibenzofurans and Dioxins during Combustion, Electrical Equipment Fires and PCB Incineration

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Polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) are likely formed by thermal synthesis of a variety of primary precursors. Highest levels of these compounds are expected, however, when the starting material requires only one or two reaction steps for their formation, as is the case with chlorophenols, chlorobenzenes and polychlorinated biphenyls (PCBs). Laboratory pyrolyses have indeed shown that PCBs give significant yields of PCDFs, and chlorobenzenes give both PCDFs and PCDDs. In addition, a variety of other chloroaromatic compounds are formed. From these experiments and from accidents involving PCB fires, it is known that PCDFs are the most important toxic compounds associated with PCBs.

Most commercial PCBs contain PCDFs in the low ppm range. PCDF concentration does not increase during normal operation in electrical equipment. Accidents (fires and explosions) involving PCBs can give PCDF levels in soot of up to 1000 ppm and higher. Effective thermal destruction of PCB is possible in modern incineration units, provided high temperatures, excess air and sufficient residence times are used. Exact figures for minimum temperature and residence time cannot be given, since feedstock and incinerator construction greatly influence destruction efficiency. Effluents from EPA-licensed incinerators used for PCB destruction contain only very low levels of PCDDs and PCDFs.

Introduction

The occurrence of polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in effluents of municipal incinerators (*1*) has stimulated interest in the thermal formation of these compounds from different sources and by a variety of processes (*7–11*).

The presence of PCDDs and PCDFs in combustion effluents can be attributed to several possibilities: (1) they could be trace components of the material to be burned and do not undergo effective thermal destruction; (2) they could be produced during the combustion and pyrolysis of chlorinated precursors like polychlorinated biphenyls (PCBs), chlorophenols, chlorinated phenoxyacids, polychlorinated diphenyl ethers (PCDPEs) and chlorinated phenoxy-2-phenols; (3) they could be formed as a consequence of a complex array of pyrolytic processes involving chemically unrelated organic compounds, including anthropogenic chlorinated organic chemicals such as poly (vinyl chloride), or chlorocarbons, anthropogenic nonchlorinated organic chemicals such as polystyrene and inorganic chlorine or naturally occurring organic chemicals like cellulose, lignin, coal, etc., and inorganic chlorine, i.e., Cl^- and HCl. Although data and theories in the literature support the theory of *de novo* formation of PCDDs and PCDFs (i.e., thermochemical synthesis from chemically unrelated precursors including naturally occurring substances), there is general agreement that higher levels of PCDDs and PCDFs are likely produced by more direct thermal conversion processes involving polychlorophenols, polychlorobiphenyl (PCBs) and polychlorobenzenes (PCBZs).

It has been reported that of about 1.4 billion pounds of PCBs produced in the U.S. between 1929 and 1977, about 750 million pounds are still in use in some form. Approximately 25% of this is estimated to be associated

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with the electrical utility industry where PCBs are used primarily in two types of equipment: transformers; containing mixtures (askarels) of PCBs and PCBZs, and capacitors; containing only PCBs (*12*).

It is likely that much of the remaining PCBs "in use" are being stored awaiting disposal. There are a number of different processes currently being used and/or tested for PCB destruction.

As discussed, various researchers have shown that PCDFs are formed when PCBs are heated and that PCDFs and PCDDs are formed when PCBZs are heated.

It is therefore possible that large-scale formation of PCDFs (and PCDDs) from PCBs and askarels could occur. Fires involving electrical equipment or inefficient PCB incineration may give rise to conditions ideal for the formation of PCDFs and PCDDs.

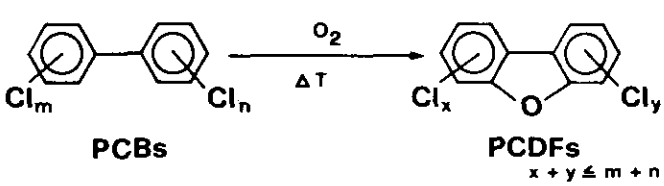
Formation of PCDDs and PCDFs from PCBs and PCBZs in Laboratory Experiments

Buser, Rappe and colleagues have investigated the potential for PCDD and PCDF formation from PCBs and PCBZs by heating the latter two types of compounds in sealed glass ampules in the presence of air and at different temperatures (*13–17*).

Pyrolysis of PCBs

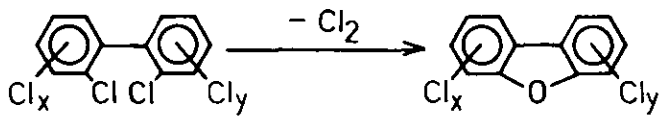
Heating PCBs (Aroclor 1248) in a sealed Pyrex glass tube for 1 week showed that PCDFs were formed above about 270°C and reached a maximum level at about 300°C (in oxygen). The product mixture contained di- and trichlorodibenzofurans as major components: the maximum yield of PCDFs in these experiments was 0.2%. Since major components of starting Aroclor 1248 were tetrachlorobiphenyls, it is apparent that one or two chlorine atoms were released in the reaction. The oxygen atom in PCDF skeleton was assumed to originate from gaseous oxygen in the sealed tube. It was concluded that temperatures above 270°C are necessary for the transformation, but at temperatures above 330°C PCDFs decompose. It is noteworthy that 2,3,7,8-tetrachlorodibenzofuran at a level of ca. 80 ppm (parts per million) was confirmed in the products.

Similarly, Buser and Rappe (*16*) have studied the pyrolysis of 18 individual PCB isomers in the presence of air and have demonstrated the formation of PCDFs via intramolecular cyclizations [Eq. (1)] where $m + n$ varies from 4 to 8. Individual starting PCBs



(1)

(100 μg each) were pyrolyzed at 600°C in separate quartz mini-ampoules. The yields of PCDFs were in the range of 0.1% up to several percent. The thermochemical generation of the PCDFs from PCBs was found to follow four general reaction routes. These routes include: loss of *ortho* Cl [Eq. (2)], loss of HCl involving a 2,3-chlorine shift at the benzene nucleus [Eq. (3)], loss of *ortho* HCl [Eq. (4)] and loss of *ortho* H [Eq. (5)]. The PCDFs obtained from the pyrolysis of 2,2′,4,5,5′-pentachlorobiphenyl show the four reaction pathways in a real sample [Eqs. (6)], The overall results obtained in this series of experiments are given in Table 1.



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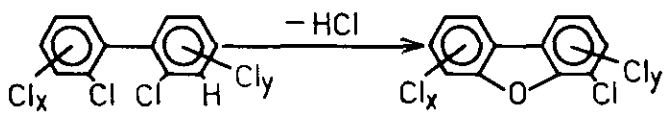

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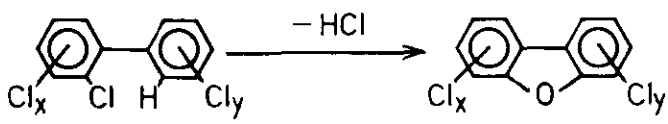

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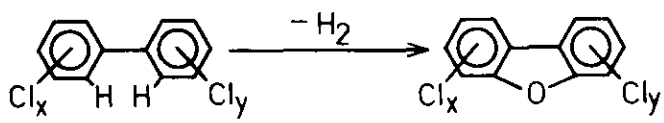

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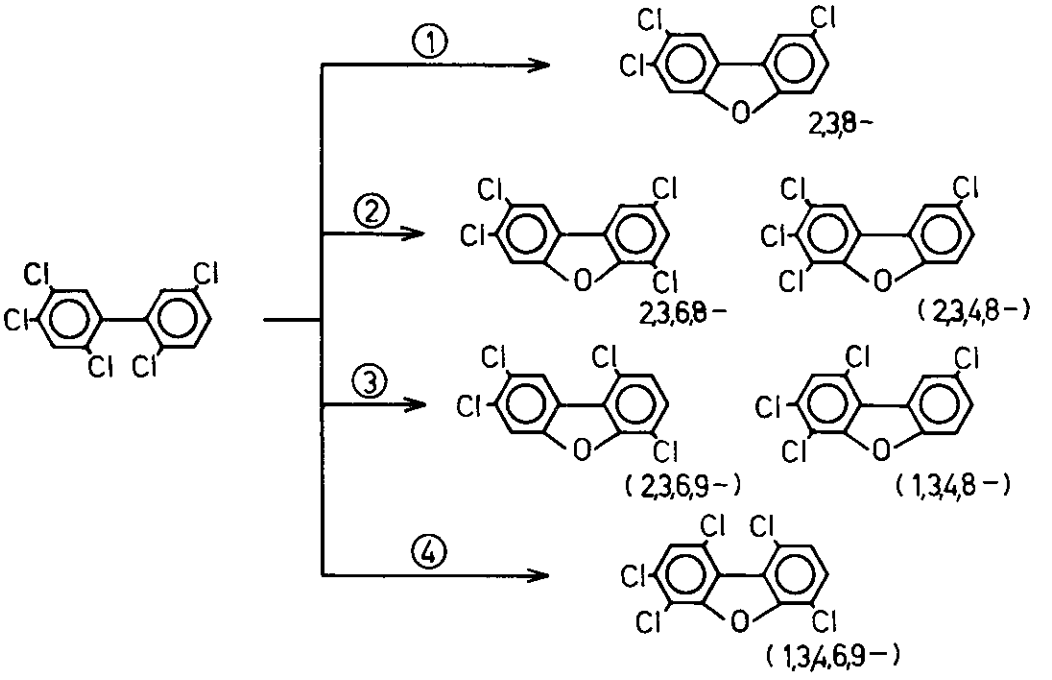
Pyrolysis of PCBZs

Buser (*17*) reported the pyrolyses of tri-, tetra- and pentachlorobenzenes, which were carried out in sealed quartz mini-ampoules at 620°C in the presence of air. The pyrolyzates of chlorobenzenes were analyzed directly; no cleanup was required. In the pyrolyzed samples, chlorobenzenes were still the main component present. The decomposition was higher for the lower chlorinated species and was > 95% for the trichlorobenzenes, ca. 90% for the tetrachlorobenzenes and ca. 50% for pentachlorobenzene in the combined chlorobenzene pyrolyzate. Chlorobenzenes with a degree of chlorination higher than the compounds used for pyrolysis were observed in all pyrolyzates, e.g., tetra- and penta- from trichlorobenzenes, penta- and hexa- from tetrachlorobenzenes, and hexa- from pentachlorobenzene. These



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higher chlorinated benzenes must have been formed in a chlorination process from lower chlorinated congeners. The formation of chlorobenzenes with a degree of chlorination lower than the starting material utilized for thermolysis was not observed. In addition to chlorobenzenes, mass spectral analyses of the pyrolyzates revealed the presence of other chlorinated compounds which include PCDFs, PCDDs, chlorophenols and in some cases polychlorinated naphthalenes (mainly hepta- and octachloro), polychlorinated styrenes (hepta- and octachloro) and polychlorinated biphenyls (PCBs). Polychlorinated diphenyl ethers and polychlorinated biphenylenes were not observed.

According to Buser, the thermochemical formation of PCDFs and PCDDs from chlorobenzenes is bimolecular [Eqs. (7)]. The likelihood of this formation is highly dependent on the concentration of chlorobenzenes in the reaction system. The author uses high concentrations

Table 1. Results of pyrolysis of PCBs.

Chlorobiphenyl studied	Process	Product(s)	Yield	Remarks	Reference
18 different chlorobiphenyls containing 4,5,6,7 and 8 Cl atoms	Pyrolysis in air	PCDF	0.1–1%	PCDF isomers formed according to proposed reaction mechanism	(*16*)
2,2′,6,6′-Cl_4-PCB 2,2′,4,4′,5,5′-Cl_6-PCB 2,2′,4,4′,6,6′-Cl_6-PCB Aroclor 1245 and 1260	Pyrolysis in air	PCDF 2,3,7,8-TCDF[a]	ca. 2% ca. 2%	Formation according to proposed reaction mechanism Important formation of 2,3,7,8-TCDF during Aroclor pyrolysis	(*14*)
2,2′,6,6′-Cl_4-PCB 2,2′,4,4′,5,5′-Cl_6-PCB 2,2,4,4′,6,6′-Cl_6-PCB Aroclor 1254	Pyrolysis in air	PCDF		Pure PCB isomers did not contain any detectable PCDF	(*15*)
Aroclor 1248	Pyrolysis in air In O_2 In N_2	PCDF	300 ppm 1100 ppm 2 ppm	Presence of O_2 is necessary for the formation of PCDF	(*13*)

[a]2,3,7,8-TCDF indicates 2,3,7,8-tetrachlorodibenzofuran.

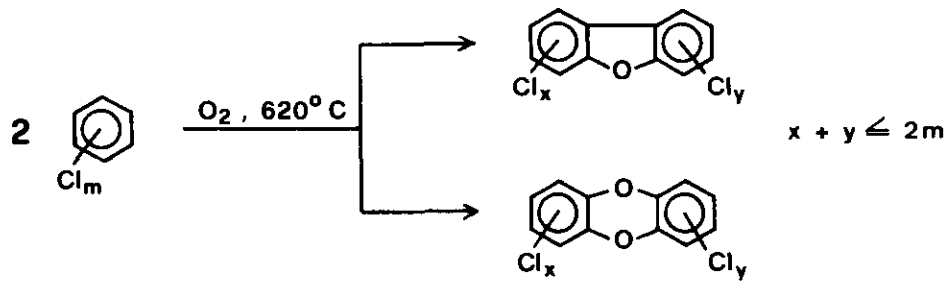

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Table 2. Formation of polychlorinated dibenzofurans (PCDFs) and polychlorinated dibenzo-*p*-dioxins (PCDDs) from the pyrolysis of chlorobenzenes at 620°C.[a,b]

	PCDFs formed, ng/sample					PCDDs formed, ng/sample				
Compounds	Tetra-Cl	Penta-Cl	Hexa-Cl	Hepta-Cl	Octa-Cl	Tetra-Cl	Penta-Cl	Hexa-Cl	Hepta-Cl	Octa-Cl
Trichlorobenzenes[c]	400	1100	550	50	<5	30	20	<5	<5	<5
Tetrachlorobenzenes[d]	<2	5	160	450	200	<2	5	140	160	30
Pentachlorobenzene[e]	<2	<5	<5	5	30	<2	<2	<5	<5	5
Combined chlorobenzenes[f]	80	600	1100	600	60	50	220	220	70	5

[a]Data of Buser (*17*).
[b]The reaction was performed in the sealed quartz mini-ampoules in the presence of air.
[c]200 μg total, with equal amounts of 1,2,3-, 1,2,4- and 1,3,5-trichlorobenzene.
[d]200 μg total, with equal amounts of 1,2,3,4-, 1,2,3,5- and 1,2,4,5-tetrachlorobenzene.
[e]200 μg pentachlorobenzene.
[f]500 μg total with equal amounts of all tri-, tetra- and pentachlorobenzenes (7 compounds).

in these experiments; the yields are expected to be substantially smaller if lower concentrations were used. The yields of PCDFs and PCDDs obtained as a result of pyrolytic decomposition of chlorobenzenes are recorded in Table 2.

A postulated mechanism for the thermochemical formation of PCDFs and PCDDs from chlorobenzenes is given in Eq. (8). As stated earlier, all pyrolyzed samples contained chlorophenols. The degree of chlorination of these phenols was the same and higher than the degree of chlorination of the chlorobenzene used: tri-, tetra- and pentachlorophenol were observed from trichlorobenzenes, tetra- and pentachlorophenol from tetrachlorobenzenes, and pentachlorophenol also from pentachlorobenzene. These chlorophenols could possibly serve as reaction intermediates in the formation of PCDFs and PCDDs from chlorobenzenes. A reaction of chlorophenol with unreacted chlorobenzene could lead to polychlorinated diphenyl ethers, which are known to form PCDFs (and to a lesser extent PCDDs) upon pyrolysis. However, PCDEs were not actually observed in the samples analyzed here. Dimerization of chlorophenols is a further route to PCDDs. According to Buser, the former condensation (path A) via PCDPEs may be preferred in these pyrolyses due to the initially much higher concentration of chlorobenzene present, but for a substantiation of these assumptions and to obtain a more detailed picture of the reactions involved, further work will be required.

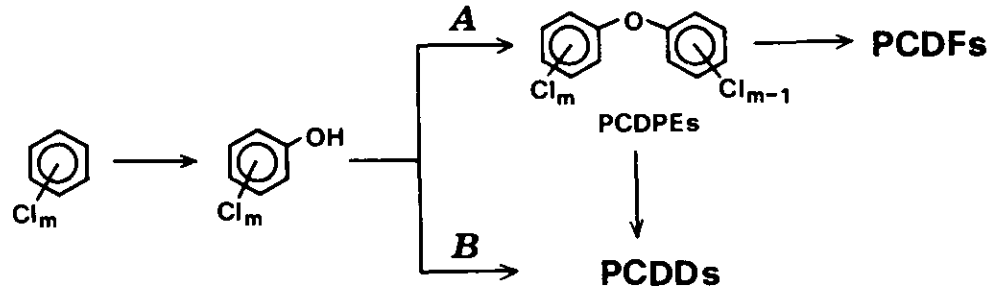

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Table 3. Fires involving PCB askarel-filled electrical equipment.

Location (Date)	Cause	Conditions	PCDFs/PCDDs formed	Comments	References
Toronto, Ontario (December 1977) underground vault	Fault in circuit breaker caused arcing and small fire	Arcing caused crack in transformer bushings; ca. 100 gal. of askarel spilled; some vaporized fire	Soot samples contained ca. 5 ppm PCDFs	Soot contained ca. 10,000 ppm PCBs (500 ppm PCDF in PCB in soot)	(*18,19*)
Binghamton, New York (February 1983) office building	Electrical failure in switching equipment	Extremely hot fire; transformer ruptured releasing ca. 180 gal. of askarel	Soot samples analyzed; 2,3,7,8-TCDF 12-270 ppm; total PCDF 765-2160 ppm; 2,3,7,8-TCDD 0.6–2.8 ppm; total PCDDs 20 ppm	Chlorinated biphenylene (PCBPs) also detected total PCBPs 54 ppm	(*18,20,21*)
Stockholm, Sweden (August 1981) transformer station	Malfunction in electrical system	Fire developed; 18 PCB capacitors were involved; some exploded	Wipe tests analyzed; 2,3,7,8-TCDF 150 ng/m^2, total PCDFs 1.4 μg/m^2 No PCDDs Total PCDF in PCB in exploded capacitor 75 ppm vs. 1 ppm in intact capacitor	PCBPs and chlorinated pyrenes (PCPYs) detected PCBP levels greater than PCDF levels due to temperature of fire (see Skovde fire)	(*18,22,23*)
Skovde, Sweden (March 1982), capacitor battery in metal treatment factory	Electrical malfunction caused fire in mineral oil filled capacitor	Fire spread to other capacitors; 21 contained PCBs; 12 PCB capacitors were broken open by fire	Wipe test samples from capacitor building analyzed High PCDF levels were only found in immediate vicinity of fire Total PCDFs 800 ng/m^2 2,3,7,8-TCDF, 100 ng/m^2 No PCDDs detected	PCPYs detected No PCBPs were detected due to "low" temperature of fire	(*18,22*)
Surahammar, Sweden (September 1982) steel mill	Explosion in steel kiln; molten steel flowed into capacitor room	Fire developed; 500 capacitors involved; 200 PCB/300 mineral oil	Wipe test samples in the capacitor room showed levels of 4 ng/m^2 TCDF No PCDDs detected	PCPYs also detected	(*18,24*)

Formation of PCDDs and PCDFs in Electrical Equipment

PCB Transformer and Capacitor Fires

There have been few documented incidents of fires involving PCB-filled electrical equipment (*18–24*). This is likely to be due to the fact that PCBs are fire resistant, although it is possible that many such fires go unreported.

In North America, to our knowledge, only two such incidents have been reported and are reasonably well documented (see Table 3) (*18*). Both were transformer fires caused by electrical malfunction.

In Scandinavia, since 1977, there have been seven such fires (five in Sweden, two in Finland). All of these involved capacitors and all but one was caused by an electrical malfunction.

These fires were characterized by the release of large amounts of oily black soot with very little fire. Where the fire involved a transformer containing PCBs and PCBZs, PCDFs and PCDDs were detected; where capacitors were involved, PCDFs and no PCDDs were found. In addition to PCDFs and PCDDs, chlorinated biphenylenes (PCBPs) and pyrenes (PCPYs) have also been found to be formed in some of these fires.

PCDF and PCDD contamination of the surrounding environment was almost entirely due to movement of the soot formed during the fire.

Five of these fires, their cause and the resultant contamination, are summarized briefly in Table 3.

Other Factors Leading to PCDF and PCDD Formation in Electrical Equipment

PCDFs have been found as contaminants in virtually all commercial PCB mixtures, both North American and European, as shown in Table 4 (*25–27*). However, there has been very little effort to determine if these levels increase during the operation of PCB-filled electrical equipment and, if they do, what factors are responsible.

Table 4. PCDFs in Aroclor, Clophen and Phenoclor.[a]

PCB	PCDFs, ppm			
	Cl_4	Cl_5	Cl_6	Total
Aroclor 1248 (1969)	0.5	1.2	0.3	2.0
Aroclor 1254 (1969)	0.1	0.2	1.4	1.7
Aroclor 1254 (1970)	0.2	0.4	0.9	1.5
Aroclor 1260 (1959)	0.1	0.4	0.5	1.0
Aroclor 1260 (lot AK3)	0.2	0.3	0.3	0.8
Aroclor 1016 (1972)	ND	ND	ND	–
Clophen A-60	1.4	5.0	2.2	8.4
Phenoclor DP-6	0.7	10.0	2.9	13.6

[a]Data of Vos et al. (*25*), Roach (*26*) and Bowes et al. (*27*).

It has been found that transformer askarels are contaminated with PCDFs and that their levels increased with the time in service of the transformer (*28*). Levels of TCDF in these fluids reached a maximum of approximately 5 ppm. This study, however, was carried out in 1978 and used analytical techniques that have since been outdated. In addition, the authors found that it was very difficult to obtain samples from various transformers so that one variable (e.g., size, age, volume) could be unequivocally isolated. Since that time it has been reported (*29*), although using a limited number of samples, that askarels that have been in use in electrical equipment do not contain higher levels of PCDFs relative to the unused fluids. In retrospect, it was considered that the normal operating temperatures in such equipment were not high enough for PCDF formation.

There is also a possibility that "abnormal" operation of a transformer or capacitor may create conditions (e.g., arcing, overheating) that may lead to the formation of PCDFs (and PCDDs). Again this is an area where much more research is required.

The Electric Power Research Institute has recently (February 1983) initiated a project to study the formation of PCDFs and PCDDs in electrical equipment under normal and abnormal conditions (*30*). This project has not as yet been completed.

Destruction/Incineration of PCBs: PCDD/PCDF Formation

Technologies for PCB Destruction

A number of processes are currently being evaluated or, in some cases, are already in use for the destruction of PCBs or PCB containing wastes (*31*). These include: incineration, including conventional incineration (high temperature oxidation) and "novel" incineration (i.e., molten salt, oxygen incinerators, diesel engine incinerators); chemical processes such as dechlorination, photolysis and wet-chemical oxidation; and, biological processes (e.g., bacterial degradation).

By far, one of the most promising, on the basis of both efficiency and cost, is high-temperature incineration. In 1970 the possibilities of using high temperature for the disposal of PCBs was put forward by the major manufacturer of these compounds in North America, Monsanto (*32*). Since that time, several groups have investigated and/or are using high temperature processes. In 1970, Monsanto reported that liquid PCBs are destroyed at 800°C with a transit time of 10 sec or at 1000°C with a transit time of 2 sec. PCBs were reportedly destroyed in a multiple hearth sludge incinerator equipped with an after burner at 6000°C with a contact time of 0.1 sec (99.9% efficiency) (*33*). Waste chlorinated hydrocarbons, including PCBs, were burned in a rotary cement kiln (99.98% destruction efficiency) (*34*). PCBs were destroyed in a laboratory scale two-stage, quartz furnace at 1000°C (99.995% efficient) (*35*). PCBs were, again, incinerated in a rotary kiln at a final temperature of approximately 1400°C (99.99998% efficiency) (*36*). Organochlorine chemical wastes were incinerated in the Gulf of Mexico using the incineration ship Vulcanas (OCS, Rotterdam). A minimum average flame temperature of 1200°C was maintained with a residence time of 1 sec (*37*). PCBs were ignited and burned by atomizing with oxygen at the tip of an oxygen-PCB burner giving a flame temperature over 2000°C (99.99999% destruction reported) (*38*).

The Energy Systems Group of Rockwell International recently (1981) developed a PCB destruction unit (incinerator) which features a fluid-bed combustion chamber followed by a catalytic afterburner. Temperatures in both units do not exceed 695°C (99.9999% destruction efficiency reported) (*39*). Two conventional incineration units, one in Arkansas (ENSCO Energy Systems) and one in Texas (Rollins Environmental Services) have been licensed by the U.S. Environmental Protection Agency (EPA) for destruction (*31,39*). Both of these systems use combustion temperatures greater than 1200°C and report PCB destruction efficiencies in excess of 99.9999%. Several other similar incineration units have been tested and are awaiting approval by EPA (*31*).

PCDFs and PCDDs Formed During Large-Scale PCB Incineration

There is very little data on the levels of PCDFs and PCDDs emitted from large-scale PCB incineration.

The Rollins and ENSCO incinerators have been tested for such emission, and "small amounts" were reported in the incineration products. The levels were such that the "additional cancer risk" was considered to be extremely low (*31*).

PCBs were test burned in a rotary cement kiln in Norway and the products and emissions analyzed for PCDFs and PCDDs (*22*). None could be found in any of the samples.

It is quite probable that the temperatures used in these incinerators (i.e., > 1200°C) completely destroy the PCBs and any PCDFs or PCDDs that are formed.

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