

Experimental Investigation of PIC Formation in CFC Incineration

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Abstract

Bench-scale tests were performed to characterize the combustion emissions from chlorofluorocarbon (CFC-11 and -12) incineration. The destruction efficiencies (DEs) of the CFCs and the major products of incomplete combustion (PICs) from each CFC were determined. DEs of at least 99.999% can be attained repeatedly for both CFCs. Major PICs identified were non-halogenated, with toluene and xylene being found most frequently. PIC concentrations were independent of the CFC concentration in the fuel. Sampling was performed one time to screen for polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDDs/PCDFs) and polyaromatic hydrocarbons (PAHs) while incinerating CFC-12. Even with a DE of greater than 99.999%, high levels of PCDDs/PCDFs were formed. The extensive PCDD/PCDF formation apparently occurred in the absence of entrained particulate matter.

Introduction

Chlorofluorocarbons (CFCs) are implicated in the depletion of stratospheric ozone, and are also contributors to global warming. As a result of the Montreal Protocol that will curtail the production of the traditional CFCs and halons, it may be necessary to destroy considerable quantities of these materials in order to reduce the current "bank" of the older (more stratospheric ozone depleting) CFCs and halons. A United Nations Environment Programme (UNEP) Ad-Hoc Technical Advisory Committee on CFC Destruction Technologies has recently been formed to evaluate the most appropriate destruction technologies for ozone depleting substances (ODS), including CFCs and halons. Incineration is currently the only widely used CFC destruction technology (1). A sound understanding of the combustion emission characteristics of CFC incineration is needed for CFC destruction technology evaluation.

CFCs are thermally stable because of strong carbon-halogen bonds, which are resistant to oxidation. Thus, CFCs are difficult to destroy by incineration. Furthermore, the halogen free radicals generated as intermediate species during the combustion of CFCs are well known flame retardants. Formation of products of incomplete combustion (PICs) becomes possible as the flame propagation is retarded. PIC formation, in general, is a poorly understood phenomenon associated with waste incineration, which is further complicated for CFC incineration processes since only limited data on PIC formation are available.

The release of halogenated aromatic compounds, which have been shown to be toxic in general, from CFC incineration may cause environmental concerns. It has been shown that the generation of halogenated aromatics from CFC decomposition is thermodynamically favorable and such compounds have been identified in a bench-scale CFC decomposition study (2). Formation of a wide variety of PICs from thermal destruction of chlorinated hydrocarbons (CHCs) has been shown in a theoretical analysis (3), and observed by experimental investigations (4, 5, 6). The extent of PIC formation in

CFC incineration is not known, and was investigated in this study. The combustion emission characterization data from a bench-scale CFC incineration study are reported in this paper.

Experimental

A turbulent flame reactor (TFR), which has been used in a previous hazardous waste incineration study (7), was employed to perform the incineration tests. The TFR (Figure 1) consists of a swirling air/gas injector burner firing into a 30 cm ID by 60 cm long water-cooled stainless steel enclosure with refractory quartz placed inside. Following the TFR is an afterburner (AB) section consisting of a 30 cm ID refractory-lined chamber with three fuel/air injectors near its base. The fuel/air injectors are spaced 120° radially apart and fired toward the axis of the cylindrical chamber. The TFR/AB system is shown in Figure 2. The flue gas exits through a 9.1 m long stainless steel duct to a venturi scrubber. The HCl and HF gases produced were neutralized by NaOH scrubbing liquor. The TFR was fired at 0.007 GJ/hr at a stoichiometric ratio of 1.1 (2% O₂) using propane gas fuel and preheated air at 190 °C. Propane and air were replaced by natural gas and compressed oxygen, respectively, in the afterburners in order to achieve the target AB conditions of 982 °C and 7% excess air. Incineration tests were performed by metering one of the CFCs (11 or 12) at a time into the primary flame. The CFCs were certified as 99.7% pure.

A continuous emission monitoring (CEM) system, which included O₂, CO₂, CO, NO_x as NO, and total unburned hydrocarbons (THCs), was used to monitor the flue gas downstream of the afterburner. Combustion gas samples were taken upstream and downstream of the afterburner simultaneously using EPA Method 18, "Measurement of Gaseous Organic Compound Emissions by Gas Chromatography." The sampling procedure involves drawing combustion gas into a tedlar bag by evacuating a sealed chamber containing the bag. Sampling points are indicated in Figure 2. Because of the high HCl and HF concentrations in the combustion gases, an impinger of 1 N NaOH solution followed by an impinger of deionized water were placed upstream of the tedlar bag, per Method 18. The tedlar bag gas samples were analyzed for the CFCs to determine the destruction efficiencies (DEs) of the CFCs. An attempt was made to quantify many of the major PICs in the samples. A known volume of the tedlar bag gas sample was transferred to an organic trapping sorbent, desorbed, and cryogenically concentrated on column for gas chromatographic/mass spectrometer (GC/MS) analysis. The analytical method used ensured adequate detection levels suitable for determination of CFC DEs.

A semivolatiles sampling train was run downstream of the afterburner one time at the baseline condition (8.3 vol.% CFC-12) with the objective of screening for PCDDs/PCDFs and PAHs. Sampling was performed according to California Air Resources Board (CARB) Method 428, "Determination of Polychlorinated Dibenzo-p-dioxin (PCDD), Polychlorinated Dibenzofuran (PCDF), and Polychlorinated Biphenyl Emissions from Stationary Sources." The combined components of the train were screened for PCDDs and PCDFs using a high resolution gas chromatography/low resolution mass spectrometry (HRGC/LRMS) method similar to EPA SW-846, "Test Methods for Evaluating Solid Waste," Method 8280. A sample fraction was also screened for the presence of PAHs. A gas chromatograph equipped with a flame ionization detector (FID) was configured for this analysis. The retention times of 16 chromatographically resolved PAHs were compared to the retention times of analytes present in the sample.

Results and Discussion

The flammability limits of the two CFCs were first determined in order to perform two incineration tests at concentrations close to the two CFCs' flammability limits. Flammability limits were determined by slowly increasing the flow of CFC into the steady stream of propane fuel primary flame until the flame extinguished. The flammability limits of both CFCs (39 vol.% for CFC-11 and 58 vol.% for CFC-12) are significantly higher than the concentrations of CFCs (1 to 5%) normally injected into a commercial incinerator. Because the flammability limits were higher than anticipated, such high CFC concentrations for test durations would have the unacceptable risks of high acid gas emissions and damage to the facility. The CFC-12 incineration tests were decided to be performed at concentrations of 1.3, 8.3, and 14.0 vol.% in fuel. The CFC-11 tests were performed at concentrations of 1.3 and 13.0 vol.%.

The test conditions for CFC incineration are summarized in Table 1. It was found that the THC concentrations were below detection limits under all test conditions. The NO concentrations were varied and showed no apparent dependence on any of the measured parameters. The concentrations of

HCl, Cl₂, HF, and F₂ were not measured in the present study. They were estimated by using the NASA (CET-85) thermodynamic equilibrium code. Results of the equilibrium analysis under CFC-12 test conditions are shown in Figure 3. The TFR conditions were 787 °C, 2% O₂ and the AB conditions were 982 °C, 7% O₂. The results indicate that fluorine favors combination with hydrogen more so than does chlorine, as the concentrations of species F₂ and F were consistently less than 10⁻¹¹ mole fraction. On the other hand, chlorine tends to form significant concentrations of both monoatomic and diatomic chlorine. Monoatomic chlorine is thermodynamically favored over diatomic chlorine at higher temperature.

Results of DEs are shown in Table 2. When no CFC was detected, the detection limit was used to calculate the DE and a ">" sign was placed in front of the DE. Note also that only 99.99% DE could be determined for the low CFC concentration tests. This is a mathematical, not physical, limitation. The generally high DEs in a primary flame observed in the present study were also reported for the thermally stable organics using the same TFR (7).

Table 3 summarizes the PIC identification results. PIC concentrations were generally low. Some PICs most likely reflect interaction of the CFC with the propane fuel, such as hexafluoropropene, trichlorofluoroethane, and chloroform. Transformation of CFCs to CHCs as PICs was evidenced. Toluene and xylene were the two most frequently occurring PICs. The non-halogenated aromatic structures of these PICs may be indicative of their formation from the pyrolysis of the propane fuel alone. Benzene and its derivatives, chlorinated species in particular, have been found to be the most dominant PICs produced from thermal destruction of chlorinated organics (4, 5). The high halogen input, chlorine in particular, with CFC incineration may promote aromatics formation. It is well known that chlorine addition in hydrocarbon flames enhances aromatics formation leading to sooting (8). Reaction pathways for the formation of aromatics from chlorinated alkanes have been proposed (9). The majority of the PICs identified in the present investigation were non-halogenated. The most interesting exception was the presence of CFC-11 as a PIC of CFC-12. The transformation of CFC and the formation of aromatics in CFC incineration observed in the present study are evidences that complex reaction pathways play an important role in the thermal incineration of CFCs.

Overall, PICs formed at least as frequently in the secondary flame as in the primary flame. For CFC-12 the greater number of PICs appeared when it was at its highest concentration in the fuel. However, CFC-11 tests yielded the seemingly odd result of many PICs at low CFC concentration and no detected PICs at high CFC concentration. The non-halogenated PICs may have formed as a result of flame anomalies unrelated to the presence of the CFCs. Comparison of PIC results to the data in Table 1 shows that such anomalies were not signaled by temperature or CEM readings.

Table 4 list the PCDD/PCDF emissions. Figure 4 presents the distribution of isomer groups. The PCDD/PCDF levels were surprisingly high. It has been suggested that PCDD/PCDF generation tends to be promoted by large (> 10 µm) particulate matter (10). Entrained particulate matter for the tests in this study using gaseous fuels should have been negligible. The filter on the sampling train used to collect the dioxin sample was observed immediately after the test to have turned a light beige color, with no large particulate discernable. A more thorough examination of the filter was not possible because, when the filter housing cooled down during the few minutes between testing and sample recovery, the condensate from the residual flue gas in the housing apparently contained enough HF to dissolve the glass filter. Only remnants of the partially dissolved filter remained at the time of sample recovery. The contents of the filter housing were combined with the rinse of the sampling glassware.

The presence of PCDDs/PCDFs from the field blank analysis was not detected, indicating no contamination from components of the sampling apparatus, recovery agents, or the ambient air. One could speculate that the HF in the flue gas could have etched the sampling glassware, particularly the last piece before the XAD module because condensation can occur there, releasing dioxin buildup from previous sampling. This argument assumes that the standard glassware preparation and sample recovery methodologies are inadequate. The field blank was taken before the actual sample was taken, and the same set of glassware was used for both the field blank and the sample. It is extremely unlikely that the microgram levels of PCDDs/PCDFs detected from the sampling run were caused by residues on glassware that had previously survived treatment using the same recovery method. To dispel the possibility of an acid etching artifact during sample analysis, a test was performed where HF was added to a method blank and analyzed. HF was added to a commonly used sample extraction apparatus and refluxed as an ordinary sample. Acid etching of the extraction glassware was similar to the etching observed during extraction of the original sample. However, no PCDD/PCDF material was present in the final analyzed extract.

The lack of contamination and particulate in the incineration system implies that a significant PCDD/PCDF formation mechanism exists that is not catalyzed by entrained particulate matter. The PCDDs/PCDFs probably formed either in the primary flame and survived the afterburner via cold pathways along the walls and near the burner jets or in the long section of the 10 cm stainless steel duct, which was about 6 m in length from the afterburner exit to the point of sampling. Flame-mode PCDD/PCDF formation may have been enhanced by the relatively low TFR flame temperature. Note from Figure 2 that the TFR wall at the location of the pyrometer port is no longer refractory, but is water-cooled stainless steel. Although the low flame temperature did not prevent 99.999% DE of the CFC, it may have had an influence on PCDD/PCDF formation.

Formation of PCDDs/PCDFs through gas-phase homogeneous reactions remains controversial. Based on the results of numerical calculations on the kinetics of a model PCDD formation mechanism, it has been suggested that the probability of gas-phase formation of PCDDs is likely to be very low (11). However, a recent study of dioxin formation reaction mechanism based on fundamental thermochemical principles demonstrated that the gas-phase formation is possible (12). Elementary reaction pathways leading to the formation of PCDDs at both high and low temperature regimes in incineration are identified by the study. It has been predicted that if PCDDs are already present in the gas phase, the formation of PCDFs from PCDDs in the gas phase is more likely (11). The much higher levels of PCDFs compared to those of PCDDs measured in this study are apparently consistent with the gas-phase formation mechanisms.

Considering the possibility of heterogeneous formation mechanisms, the temperature of the flue duct ranged from about 620 °C at its entrance to 110 °C at the sampling point. PCDD/PCDF formation has been shown to be enhanced in the temperature range of about 200-370 °C and in the presence of some metals, especially copper. The flue duct in the experimental system had previously been exposed to parts per million levels of copper, which may have condensed inside the duct. The long, narrow, metal duct might provide the conditions, including sufficient surface area without entrained particulate, to catalyze PCDD/PCDF formation. No PAH was detected in the PCDD/PCDF sample.

Conclusions

Based on bench-scale test results, destruction efficiencies of at least 99.999% are attainable for both CFC-11 and CFC-12 even from a relatively low temperature flame. The use of an afterburner was not necessary to attain high DE of CFC-11 and CFC-12. The results were repeatable. Most PICs identified were non-halogenated species. PIC concentrations were independent of the concentration of CFCs in the fuel. The wide variety of PICs, ranging from aliphatic to aromatic species, observed in the present study is a strong indication that CFC destruction during the thermal incineration occurs through complex reaction pathways. DEs of greater than 99.999% still allow for high generation of PCDDs/PCDFs. Extensive PCDD/PCDF formation when incinerating CFC-12 was apparently independent of entrained particulate matter. Further studies are needed to investigate the levels of PCDDs/PCDFs generated in pilot and commercial scale CFC incinerators.

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Table 1. Test Conditions

CFC	Vol.% in Fuel	Dry Flue Gas Composition					Primary Flame	Secondary Flame			
		O ₂ %	CO ₂ %	CO ppm	NO ppm	THC* ppm	Pyrometer °C	Pyrometer °C	TC-1 °C	TC-2 °C	TC-3 °C
Blank	0	6.5	22.6	28	84	<5	813	983	1001	954	621
12	8.3	7.25	22.4	33	68	<5	784	957	1009	978	NA
12	8.3	7.5	22.3	33	118	<5	777	951	1002	979	643
12	8.3	8	22.2	35	120	<5	798	986	1016	NA	NA
12	8.3	6.8	22.5	27	70	<5	NA	NA	996	944	626
12	14	7.25	22.4	32	68	<5	792	977	988	957	629
12	1.3	7.25	22.4	30	115	<5	798	986	978	949	617
11	13	7.4	7.4	30	85	<5	787	971	989	954	621
11	1.3	6.8	6.8	30	73	<5	792	977	1012	966	639

NA = not available

* THC measurements taken during flammability tests. Increasing CFC content in the fuel (within the flammability limit) had no effect on THC levels.

Table 2. Summary of Destruction Efficiencies

CFC	Vol.% In Fuel	Location	CFC Flow NL/h	Mass CFC Spiked grams	Combustion Gas Flow NL/h	CFC Detected ng/L	Mass CFC Out grams	Destruction Efficiency %
12	8.3	TFR exit	76	383.3	24569	<63	<0.0015	>99.9996
		AB exit	76	383.3	33826	<63	<0.0021	>99.9994
12	8.3	TFR exit	76	383.3	24569	<63	<0.0015	>99.9996
		AB exit	76	383.3	33917	<63	<0.0021	>99.9994
12	8.3	TFR exit	76	383.3	24569	<63	<0.0015	>99.9996
		AB exit	76	383.3	34099	<63	<0.0021	>99.9994
12	14	TFR exit	133	667.2	24682	93	0.0023	99.9997
		AB exit	133	667.2	33948	<63	<0.0021	>99.9997
12	1.3	TFR exit	11	56.79	24439	<63	<0.0015	>99.9997
		AB exit	11	56.79	33686	<63	<0.0021	>99.9996
11	13	TFR exit	127	725.8	24671	<63	<0.0016	>99.9998
		AB exit	127	725.8	33990	<63	<0.0021	>99.9997
11	1.3	TFR exit	11	64.52	24439	181	0.0044	99.993
		AB exit	11	64.52	33524	<63	<0.0021	>99.997

Table 3. Summary of Products of Incomplete Combustion

	CFC-12				
Vol.% CFC in Fuel Primary Flame	8.3	8.3	8.3	14	1.3
CFC, ng/L DE, % PICs, ng/L	<63 >99.9996 ND	<63 >99.9996 CFC-11, 81	<63 >99.9996 ND	93 99.9997 CFC-11, 81 unknown hydrocarbons, 44, 256, 25, 225 toluene, 488 bicyclo[4,2,0]- octa-1,2,5-triene, 644	<63 >99.997 chloroform, 213 pentane, 225 toluene, 50 xylene, 50
Secondary Flame					
CFC, ng/L Overall DE, % PICs, ng/L	<63 >99.9994 unknown hydrocarbon, 69 toluene, 31 xylene, 19	<63 >99.9994 hexafluoro- propene, 781 unknown hydrocarbon, 1000	<63 >99.9994 hexafluoropropene , 2400 unknown hydrocarbon, 494 carbon disulfide/ trichloro- trifluoroethane, 438	<63 >99.9997 CFC-11, 250 methyl propene, 550 pentane, 581 trichlorotrifluoro- ethane, 325 toluene, 219 ethyl benzene, 113 xylene, 369	<63 >99.996 chloroform, 213

ND = not detected

(continued)

Table 3. Summary of Products of Incomplete Combustion (continued)

	System Blank	CFC-11	
Vol. % CFC in Fuel Primary Flame	0	1.3	13
CFC, ng/L DE, % PICs, ng/L	<63 NA ND	181 999.993 unknown hydrocarbons, 550,806,5600 methyl propene, 713 trichlorotrifluoroethane, 413 cyclohexane, 556 ethylbenzene, 144 xylene, 450	<63 >99.9998 ND
Secondary Flame			
CFC, ng/L Overall DE, % PICs, ng/L	<63 NA ND	<63 >99.997 unknown hydrocarbons, 44, 81 toluene, 19 xylene, 31	<63 >99.9997 ND

NA = not applicable

ND = not detected

Table 4. PCDD/PCDF Results for 8.3 Vol.% CFC-12 in Propane

	Flue Gas Concentration µg/dscm	Generation Rate µg/g of CFC
Total PCDD	3.13	0.28
Total PCDF	20.70	1.82
PCDD/PCDF	23.80	2.09

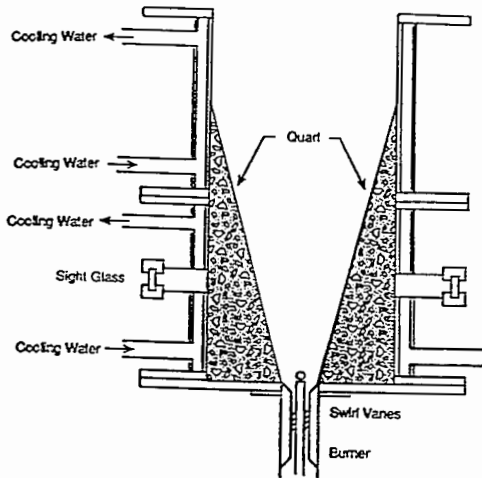


Figure 1. Turbulent Flame Reactor (TFR)

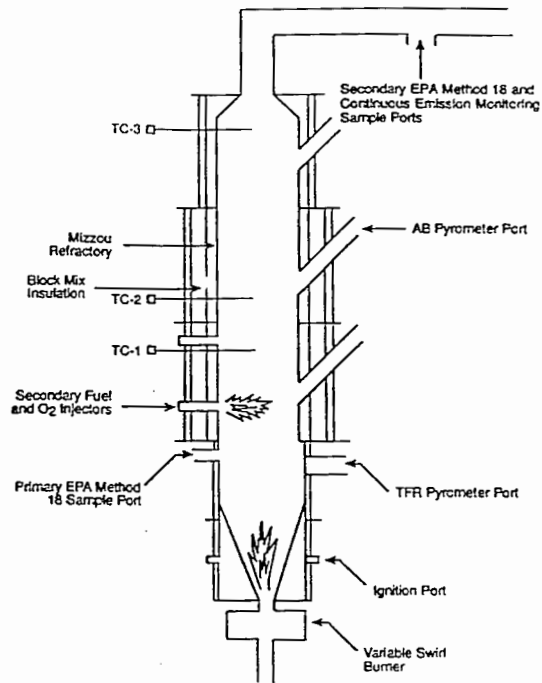


Figure 2. TFR/AB System Diagram

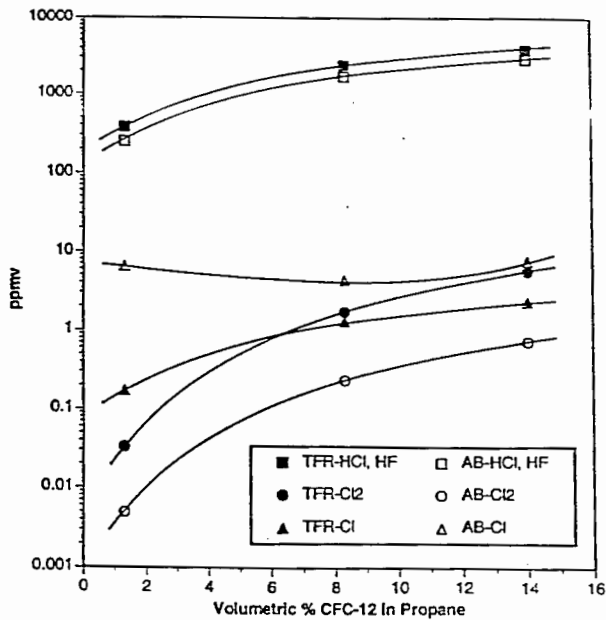


Figure 3. Equilibrium Concentrations of the Major Halogenated Products of Combustion of CFC-12

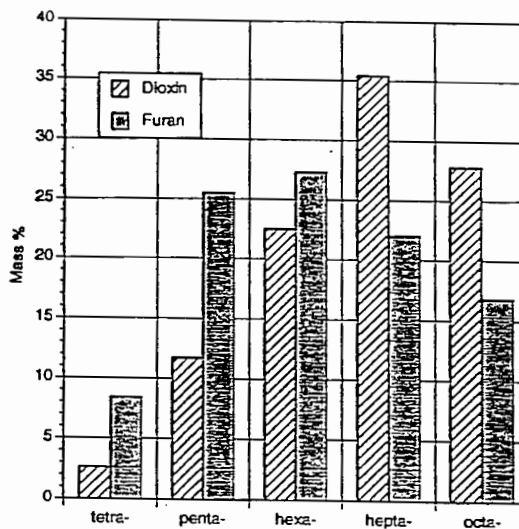


Figure 4. Distribution of Isomer Groups

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Incinerators	Stationary Sources		
Halohydrocarbons	Chlorofluorocarbons	07C	
Combustion	Destruction Efficiency	21B	
Emission	Products of Incomplete	14G	
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