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CHAPTER XX

1

ACID GAS PRODUCTION IN INHIBITED PROPANE-AIR DIFFUSION FLAMES

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The proposed replacements to halon 1301, mainly fluorinated and chlorinated hydrocarbons, are expected to be required in significantly higher concentrations than CF₂Br to extinguish fires. At these higher concentrations the by-products of the inhibited flames may include correspondingly higher portions of corrosive gases, including HF and To examine the chemical and transport-related mechanisms HCL. important in producing these acid gases, a series of inhibited flame tests are performed with several types of laboratory-scale burners, varying agent type and concentration. A wet-chemistry analysis of the final products of the flames using ion-selective electrodes for F and Cl provides an experimental basis for quantitative understanding of the HF and HCl production. Production rates are measured for co-flow laminar and jet diffusion flames. Systematic selection of the agent concentrations, burner type, and air flow rates allows an assessment of the relative importance of agent transport and chemical kinetics on the acid gas production rates. These experimental results are then compared to a model which estimates the maximum HF and HCl production rates based on stoichiometric reaction to the most stable products. The results demonstrate the relative significance of F, Cl, and H in the inhibitor and fuel, as well as the effect of different burner configurations.

Although the corrosiveness and toxicity of candidate fire suppressants have always been recognized as important, it has also been observed that since the most effective flame suppressants are not chemically inert the properties of their decomposition by-products are also important. Halogenated hydrocarbons are widely used and effective flame suppressants; however, the production of the most effective of these (for example halon 1301 CF₃Br and 1211 CF₂ClBr) has been discontinued. The proposed alternatives to these halons, primarily fluorinated and chlorinated hydrocarbons, are required in much higher concentrations. Consequently, they have the potential to have correspondingly higher amounts of decomposition by-products. Since most hydrocarbon-based compounds at flame temperatures typically undergo both thermal decom-

position and decomposition by radical attack, the formation of products other than the inhibitor itself is highly probable. The acid gases hydrofluoric (HF) and hydrochloric acid (HCl) are believed to be the most corrosive products. The objective of this research is to obtain an understanding of the chemical and physical process of acid gas formation in inhibited flames.

Background

The halogen acid or hydrogen halide HX (where X represents the halogen) is a thermodynamically stable product in mixtures containing hydrogen and halogen atoms. Formation of acid gases in inhibited hydrocarbon flames has been studied for many years. The research can be categorized as either global measurements of HF produced in suppressed fires, or detailed flame structure measurements. Burdon et al. (1955) ignited mixtures of fuel, air, and CH₃Br in flasks, analyzed the products and found copious amounts of HBr. Numerous premixed low pressure flame studies (e.g. Wilson, 1965; Biordi et al., 1973; Safieh et al., 1982; and Vandooren et al., 1988) used mass spectroscopy to measure the profiles of hydrogen halides and other products in hydrogen, carbon monoxide, and hydrocarbon flames inhibited by CH₃Br, CF₃Br, and CF₂H. These studies indicated conversion efficiencies of the halogens in the inhibitor into halogen acids on the order of unity. Acid gas formation in hydrocarbon-air pool fires suppressed by CF₃Br has been studied by Sheinson et al. (1981, 1982). These studies, in test volumes of 1.7 and 650 m³, stressed the difficulties in probe sampling for acid gases. The latter study described an in situ IR absorption method for measuring HBr and HF. To overcome these limitations and also provide time-resolved acid gas concentration data, Smith et al. (1993) developed a new HX sampling technique and obtained HX and inhibitor concentrations as functions of time for discharge of CF₂Br into a 56 m³ space. In a series of experiments with a variety of fuels and halogenated inhibitors, Yamashika (1973) showed that the extinction time for a compartment fire sprayed with inhibitor is dependent upon the discharge rate and room volume. He then showed (Yamashika, 1974) that the amounts of hydrogen halides and carbonyl halides are also dependent upon the discharge rate. Using a simple model of acid gas formation based on the steady-state rates, he developed a model of transient acid gas formation to explain his results.

In more recent studies, Ferreira *et al.* (1992a,b) injected CF_3Br , C_3HF_7 , and C_4F_{10} into an enclosure fire and measured the HF produced using ion-selective electrodes. Di Nenno *et al.* (1993) introduced halon alternatives into compartment fires and measured the HF, HCl, and COF_2 produced using Fourier transform infrared spectroscopy. These studies again confirmed the importance of injection rate and fuel consumption rate on the amount of acid gas produced. Filipczak (1993) introduced CF_2CIBr and CF_3Br into a methane flame and measured the O_2 , CO_2 , H_2O , HF, HCl, HBr, and unreacted inhibitor using a mass spectrometer. Hoke and Herud (1993) are currently developing a fast-response ion-selective electrode for measuring HF and HCl produced in extinguished fires in crew compartments of combat vehicles.

Previous research related to understanding acid gas formation in inhibited flames can be seen to include both detailed flame structure measurements and global measurements of HF produced in suppressed fires. The former provide the basis for obtaining a good understanding of the underlying chemical kinetics of the formation of acid gases. The global measurements provide important information on the magnitude of the acid gases produced and allow a comparison of the relative amount of acid gases formed by new halon alternatives. There remains a need to develop a fundamental basis for interpreting the data on acid gas formation in fires suppressed by halon alternatives and to understand the chemical kinetic rates of acid gas formation in diffusion flames inhibited by these alternative agents. In particular, there exists a need to understand the relationship between fuel and inhibitor type, flame characteristics, agent transport rates, and the concentrations of by-products formed.

Experimental Approach

The formation of toxic and corrosive by-products in flames suppressed by halogenated hydrocarbons may be determined by transport rates of the inhibitor into the flame, chemical kinetic rates, and equilibrium thermodynamics. These phenomena in turn will be affected by the fuel type, local stoichiometry, inhibitor type and concentration, and the characteristics of the flow field (mixing rates, strain, and stabilization mechanisms). The approach in this research is to study the influence of key parameters (inhibitor type and concentration and flame type) through systematic experiments on laboratory-scale flames. Inhibitor is added at steady-state to the fuel or air stream of co-flow diffusion flames. The diffusion flames are operated under both laminar and near-turbulent conditions to vary the mixing rates. The apparatus used to obtain these data, the results, and their interpretation are presented below.

The experiments are performed with a propane-air co-flow diffusion flame. Two burner types are used. The first is modelled after the cup burner described by Booth et al. (1973) and Bajpai (1974). The experimental arrangement is shown in Figure 1. The burner consists of a 28 mm diameter pyrex cup positioned concentrically in a 120 mm diameter 450 mm tall chimney 150 mm from the base. In these experiments with propane, the cup burner was modified for use with a gaseous fuel (liquid fuels will be tested in future experiments). The cup is filled with 1 mm diameter glass beads and covered with a stainless steel screen. The second burner consists of a 25 cm long pyrex tube with a 0.50 mm diameter opening positioned concentrically and at the same height as the cup burner, with the same chimney. The cold flow Reynolds number based on the exit velocity of the propane in the tube is 1050. This second burner, referred to here as the jet burner, is designed to provide better mixing of the inhibitor in the air stream with the fuel. Although a higher jet Reynolds number would have been desirable to achieve turbulent mixing, the flame is very close to blow-off at flows with a Reynolds number of 2000, and very little inhibitor can be added before blow-off occurs. Consequently, at these flows, it is difficult to study the effects of air stream inhibitor concentration on HF formation.

The air used is shop compressed air (filtered and dried) which is additionally passed through an 0.01 micron filter to remove aerosols and particulates, a carbon filter to remove organic vapors, and a desiccant bed to remove water vapor. The fuel gas is propane (Matheson¹, CP grade) at flow rate of 0.114 l/min at 21 °C. Gas flows are measured with rotameters (Matheson 1050 series) which are calibrated with bubble and dry (American Meter Co. DTM-200A and DTM-325) flow meters. Inhibitor gases are of different purities from various suppliers. The twelve agents tested are CHClF₂, CF₃Br, C₂HF₅, C₂H₅, C₂H₅F₄,

¹ Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for the intended use.

C₂HClF4, C₃F₈, C₃HF₇, C₃H₂F₆, C₄F₁₀, and CH₂F₂/C₂H₂F₄.

Before measuring HF in the product gases, the concentration of inhibitor in the air stream necessary to extinguish the flame is determined. The inhibitor is then added to the co-flowing air stream at a concentration of either 50 or 90% of the extinguishing concentration, and the product gases are sampled for acid gas. In one series of experiments with the cup burner, the inhibitor is added to the gaseous propane stream at 70% of the concentration which would extinguish the flame.

A wet chemistry technique is used to measure the HF and HCl concentrations in the exhaust gases from the co-flow diffusion flames. A glass funnel is placed over the chimney and the exhaust gases pass through the 4.0 cm diameter neck. A quartz probe, centered in the neck, extracts a measured fraction of the product gases (approximately 0.5%), and directs the gases through polyethylene sample lines to polyethylene impingers filled with water which trap the acid gases. The sample flow is continued for a total collection time of sixty seconds. The quartz probe and sample lines are washed with water which is returned to the impinger. The sample is tested for F and Cl using ion-selective electrodes (Orion models 96-09 and 96-17B). To reduce the effects of sampling losses as have been reported by other investigators, a quartz probe and polyethylene sample lines are used, the distance from the chimney top to the bubbler is kept small (~ 10 cm) and the sample lines are washed with the bubbler fluid immediately after the sample is collected. It should be noted that since COF_2 is know to hydrolyze rapidly in the presence of water, this technique for acid gas measurement includes F from both HF and COF_2 . When HF formation is described in this paper, it actually refers to an equivalent amount of fluoride ion collected in the bubbler.

Model for Acid Gas Formation.

A model for the amount of acid gas formed in an inhibited diffusion flame can be developed in a manner analogous to the determination of the local equivalence ratio and structure for diffusion flames. In the classic Burke-Schumann analysis (Burke and Schumann, 1928), the chemical reaction is assumed to occur at a sheet. This location serves as a sink for the fuel and oxidizer which are assumed to diffuse there in stoichiometric proportions based on complete reaction to the most stable products (i.e., HF formation is assumed to be controlled by equilibrium thermodynamics rather than chemical kinetics). This model will be referred to as the stoichiometric model.

In the case of a flame inhibited by halogenated hydrocarbons, a thermodynamic analysis shows that in equilibrium, the inhibitor readily breaks down to CO_2 , HX, and H_2O . Consequently, the inhibitor is assumed to be consumed like a fuel and form the most stable products. The assumptions used in the stoichiometric model are:

- 1. there is complete reaction of the inhibitor molecule with fuel and air to form the most stable products;
- the inhibitor in the air stream which by-passes the flame sheet does not decompose through interaction with the post-combustion product gases;
- 3. there is no loss of acid gases to the chimney walls; and
- the product gases are perfectly mixed.

An equation for the reaction of an arbitrary hydrocarbon with air and arbitrary halogenated hydrocarbon inhibitor is:

$$\alpha C_{d}H_{b} + \beta C_{c}H_{d}F_{e}Cl_{f} + \gamma'(O_{2} + 3.76N_{2}) -$$

$$\delta CO_{2} + \lambda H_{2}O + \eta HF + \phi HCl + \epsilon COF_{2} + \pi COCl_{2} + \xi O_{2} + 3.76\gamma' N_{2}$$
(1)

In this equation α is specified as is β when inhibitor is present only in the fuel stream. For inhibitor present in the air stream, β is determined by the concentration of inhibitor in the air stream and the ratio of the diffusion rates for oxygen and inhibitor.

$$\beta = \frac{\alpha (a+b/4)}{1/(\rho r) - [c+1/4(d-e-f)]}$$
(2)

Where ρ is the ratio of the binary diffusion coefficient of the inhibitor in nitrogen to that of oxygen with nitrogen and r is the ratio of the concentration of inhibitor to oxygen in the air stream. An atom balance for all species provides all of the unknown coefficients, and an estimate of amount of acid gas formed per mole of fuel, $(\eta + \phi)/\alpha$, is then readily found.

The assumption that the ratio of the diffusion rates for oxygen and inhibitor controls the amount of HF that forms basically implies that the characteristic flame height over which oxygen and inhibitor react is the same (as related to assumption 2 above).

Results and Discussion.

The acid gases produced are measured at inhibitor concentrations of 50 and 90% of the concentration of inhibitor found to extinguish the flame when the inhibitor is added to the co-flowing air stream in the cup burner and jet burner, and at 70% of the extinction concentration for inhibitor added to the propane fuel stream for the cup burner. Table 1 lists the extinction concentrations for each agent for inhibitor added to the air stream of both burners, and for inhibitor added to the fuel stream of the cup burner. As the table indicates, the jet burner flame typically requires about 50% less inhibitor in the air stream to extinguish the flame than the cup burner, even for identical fuel and air flows, although there are notable exceptions: CF_3Br , which requires about one fifth as much inhibitor in the jet burner than in the cup burner, and $C_2H_2F_4$ and the $CH_2F_2/C_2H_2F_4$ mixture which had nearly the same extinction concentrations. In addition to providing the necessary extinction conditions for specification of inhibitor flows at 50 and 90% of extinction, these results also demonstrate the sensitivity of the extinction conditions to the burner geometry.

The measured HF for these diffusion flames is shown in Figures 2 and 3. These figures present the HF produced (moles/min) for each inhibitor for the five burner/inhibitor combinations. The total flow is about 1 mole/min. For each inhibitor, the measured HF is plotted for the cup burner and for the turbulent jet burner with inhibitor present in the air stream at 50 and 90% the extinction value, and for inhibitor added to the fuel stream in the cup burner at 70% of the value necessary to cause extinction. The figure indicates that the amount of HF varies, for a given agent, by a factor of about five for these two flames. For a given flame and fraction of extinction concentration of agent, the amount of HF formed varies by about ten for these twelve agents. Note that the fuel and air flow rates are held constant in these data. Chloride ion is also measured in these experiments and the results are qualitatively the same as for fluoride. For clarity of presentation, however, only the HF results are presented.

The results of the agent addition to the fuel stream are also presented in Table 1. The halogen to hydrogen ratio of the inlet reactants in the fuel stream at the agent loading of the tests (70% of extinction) is listed, as is the fraction of the fluorine converted by the flame to a species which hydrolyses in the bubbler. While conversion is only 30 to 55%, there is no clear dependence on halogen to hydrogen ratio in either the parent inhibitor molecule or the reactant stream.

In order to provide insight into the controlling parameters in inhibited diffusion flames, the data of Figures 2 and 3 are presented in an alternative form in Figures 4 to 15. In these figures, the amount of HF produced is plotted as a function of the inhibitor concentration in air. The symbols represent the experimental data, while the lines marked F and H represent estimates of the fluxes of fluorine and hydrogen into the reaction zone based on the stoichiometric model described above.

Figure 4 shows the measured and estimated HF production rates in a propane-air diffusion flame for C_2F_6 in the cup and jet burners. The curve labeled F in Figure 4 is the maximum fluoride atom molar flux into the reaction sheet of the diffusion flame calculated using the stoichiometric model described above. The curve labeled F' in Figure 4 is the fluoride molar flux when the diffusion rate of the inhibitor relative to oxygen is modified to account for preferential diffusion of oxygen relative to the inhibitor using the molecular weight correction factor

$$\sqrt{W_i + W_{N_2} / W_{N_2} W_i} / \sqrt{W_{O_2} + W_{N_2} / W_{O_2} W_{N_2}}$$

In this equation, W_{N_2} , W_{O_2} , and W_i are the molecular weights of nitrogen, oxygen, and inhibitor. The predicted fluorine and hydrogen fluxes are based on actual experimental flows which vary slightly from run to run. The slight variations in flows cause the slight discontinuities in the F and H curves as in Figure 5.

Qualitatively, the curves F and F' are seen to increase with increasing inhibitor concentration in air, and the molar flux of inhibitor into the reaction zone is lower when a lower rate of diffusion is used for the inhibitor. The curves labeled H and H' (coincident for C_2F_6) show the estimated hydrogen atom flux into the reaction zone as a function of inhibitor concentration in the air stream. Since this inhibitor does not contain hydrogen, all of the hydrogen is from the propane, and increasing inhib.tor in the air stream does not increase the hydrogen flux into the flame. One would expect that the HF production rate would not be greater than the estimated flux of F or H into the reaction zone. For this inhibitor, the flame appears to be hydrogen limited above about 5% C_2F_6 in the air stream; however, when there is not enough hydrogen, the most stable product is COF_2 , which is known to rapidly hydrolyze in the presence of water, and would also appear as F in the bubbler. Consequently, the hydrogen limit may or may not exist (depending upon whether the kinetics are fast enough to form COF_2 in the hydrogen-limited case).

Also shown in the figure are the experimentally measured HF production rates for the jet and cup burners (labeled c and j respectively) at 50 and 90% of the extinction concentration of C_2F_6 . As indicated, the measured quantities of HF are lower than both the fluorine and hydrogen limits, and the measured values are closer to the estimated limits when the effects of preferential diffusion (H' and F') are included as described above. The results for the inhibitor addition to the fuel stream in the cup burner (labeled f) are plotted at an inhibitor concentration of zero so that they can be included in the figure.

Although the cup and jet burner results are plotted together, the phenomenological behavior of jet burner is distinctly different from that of the cup burner. Because the flame of the jet burner first stabilizes as a co-flow diffusion flame anchored at the outlet of the jet, the heated gases have a much lower Reynolds number, keeping the flow laminar. As inhibitor is added to the air stream, the flame grows in length (as it would in increasing the fuel flow rate). Eventually, the flame lifts off the burner surface by about 5 cm to form a lifted jet diffusion flame. With further inhibitor addition, the flame eventually blows off. These blow-

off concentrations are referred to as the extinction concentrations (see Table 1) and are found to be much lower (about half) of the values determined for the cup burner. In the tests at 50% extinction, the flow is laminar, whereas at 90% of extinction, the flow is nearly turbulent and the flame is lifted. Transport of the agent into the flame is estimated in the stoichiometric model assuming molecular diffusion. The goal of these experiments is to compare the model's prediction of HF formation for a diffusion flame where more vigorous mixing occurs, and identify if the enhanced mixing increases the HF production. Figures 4 to 15 show that HF production in the turbulent burner at 90% of extinction is higher relative to the model prediction than the cup burner results at 90% (except for C_2F_6 and C_4F_8), but that the jet burner HF production rates are still not above the estimate of the fluorine flux based on equal transport for O_2 and the inhibitor (the curve labeled F).

When viewed as in Figures 4-15, the behavior of the alternative inhibitors falls into three categories. In the first category are the inhibitors C₂F₆, C₃F₈, C₄F₁₀, C₃F₈, C₂HF₅, and C₃HF₇ (Figures 4 to 9). For these inhibitors, at the highest inhibitor concentration tested (cup burner at 90% of extinction) the estimated hydrogen flux into the reaction zone is lower than the fluorine flux. The ratio of hydrogen to total halogen flux ranges from 0.31 to 0.68, and the H flux is not a strong function of the inhibitor concentration. For these inhibitors, the HF produced does not increase significantly when the inhibitor concentration in the air stream increases above that necessary for a hydrogen/fluorine ratio in the reaction zone of about unity (the region of where the lines marked F and H or F' and H' cross in Figures 4 to 15). A second category includes those inhibitors (C2H2F4, C2HClF4, C3H2F6, and CH2F2/C2H2F4; Figures 10 to 13) for which the estimated H and X fluxes are closer, with H/X ratios of 0.68 to 0.85. For these inhibitors, the amount of HF produced increases with increasing inhibitor concentration in the air, but the highest concentration tested corresponds F/H ratio of about unity in the reaction zone. The last category consists of CF₃Br and CHClF₂ (Figures 14 and 15) for which the estimated hydrogen flux is much higher than halogen flux (in a ratio of 2.3 and 1.1, respectively), and there is estimated always to be more hydrogen than halogen in the reaction zone. For these agents, the HF produced is always increasing with higher agent concentration in the air stream.

Although the stoichiometric model is very simple and is only expected to provide an upper limit on the amount of HF formed, it is instructive to investigate the possible reasons that the measured HF production rates might be lower than the estimates. Lower HF may be measured in the experiments due to experimental difficulties, for example: loss of HF to the chimney walls, loss in the sampling system, HF undetected by the ion-selective electrodes, or imperfect mixing in the product gases. Based on exploratory parametric tests, these loss mechanisms are considered to be of secondary importance. The predicted values of the HF production do not include chemical kinetic limitations and the estimates of transport rates into the reaction zone are only approximate. Additional experiments will be performed to allow examination of these important parameters.

Conclusions

The formation rate of HF in diffusion flames is strongly influenced by the mass flux of inhibitor into the flame sheet. For diffusion flames with the inhibitor added to the air stream, there appear to be kinetic limitations to the rate of HF formation for most but not all of the agents tested which increase as the inhibitor concentration in the air stream increases. Many of the agents (for example $C_2H_2F_4$, C_2HClF4 , $C_3H_2F_6$, CH_2F_2/C_2HF_5 , CF_3Br and $CHClF_2$) produced HF at rates within about 25% of that given by equilibrium thermodynamics in the diffusion flames tested. Most of the perfluorinated agents tested (C_2F_6 , C_3F_8 , and C_4F_{10}) and the agents C_4F_8 , C_2HF_5 and C_3HF_7 produced 0 to 35% less than the equilibrium values except when the estimated fluorine to hydrogen flux into the flame goes above unity when they show

no further increase with increasing inhibitor concentration in the air stream.

Co-flow diffusion flames with inhibitor added to the fuel stream show HF production rates 30 to 55% of the values given by equilibrium thermodynamics, clearly implying kinetic limitations. Further research is needed to understand these kinetic limitations, as well as kinetic limitations present at high fluorine loading when the agent is added to the air stream.

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Tables

Table 1 - Extinction conditions for halon alternatives added to the air or fuel of co-flow propane-air cup burner and jet burner flames, and HF produced and H/X ratio with agent addition to the fuel stream.

	Agent Addition to Air		Agent Addition to Fuel		
Inhibitor	Extinction Concentration in Air (mole percent)		Inhibitor/Fuel ratio in Fuel Stream at Ext.	H/X Ratio in Flame (70% Ext.)	Fluorine Mole Fraction Collected
	jet	cup	cup	cup	cup
C ₂ F ₆	3.8	9.4	2.5	0.76	0.43
C ₃ F ₈	3.8	7.5	2.0	0.70	0.55
C ₄ F ₁₀	3.2	5.0	1.6	0.70	0.38
C ₄ F ₈	5.1	7.6	2.2	0.65	0.38
C_2HF_5	6.2	10.2	3.1	0.94	0.30
C3HF7	4.2	7.6	2.2	0.87	0.45
$C_2H_2F_4$	9.5	11.1	5.6	1.01	0.44
C ₂ HClF ₄	4.2	8.6	2.6	1.33	0.42
$C_3H_2F_6$	4.0	7.2	3.5	0.88	0.30
CH_2F_2/C_2HF_5	15.5	15.2	11.8	1.03	0.37
CF ₃ Br	0.8	4.3	0.88	4.4	0.35
CHCIF ₂	6.7	13.8	4.3	1.82	0.55

Figure Captions

Figure 1 - Experimental apparatus for co-flow diffusion flame studies of acid gas formation in inhibited propane-air flames.

Figures 2 and 3 - Measured HF production rates in co-flow propane-air diffusion flames. Data are shown for cup and jet burners at 50 and 90% of the extinction concentration for agent added to the air stream, and at 70% in the fuel stream in the cup burner. Note that in Figure 3 the curves for CF_2H_2/C_2HF_5 and $C_2H_3F_4$ are reduced by a factor of 4.

Figures 4 to 15 - Moles of HF produced as a function of the inhibitor concentration in the air stream for the cup (c) and jet (j) burners at 50 and 90% of extinction, and with inhibitor addition to the fuel stream (f) of the cup burner at 70% of extinction. The squares are the experimental data. The lines show the estimated fluorine (F) and hydrogen (H) flux into the reaction zone using the stoichiometric model, based on equal rates of diffusion for O₂ and inhibitor (un-primed) and with binary diffusion coefficients corrected for molecular weight variations (primed). The estimated error bars on the HF measurements are \pm 10%. Figures 4 to 15 provide the results for C₂F₆, C₃F₈, C₄F₁₀, C₄F₈, C₂HF₅, C₃HF₇, C₂H₂F₄, C₂HClF₄, C₃H₂F₆, CF₂H₂/C₂HF₅, CHF₂Cl, and CF₃Br respectively.











