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Spectroscopic Investigation of Atmospheric Pressure Counterflow Diffusion Flames Inhibited by Halons

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ARL-TR-857

September 1995

NOTICES

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
September 1995

3. REPORT TYPE AND DATES COVERED
Final, May 94–Apr 95

4. TITLE AND SUBTITLE
Spectroscopic Investigation of Atmospheric Pressure Counterflow Diffusion Flames Inhibited by Halons

5. FUNDING NUMBERS
PR: 1L161102AH43

6. AUTHOR(S)
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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

U.S. Army Research Laboratory
ATTN: AMSRL-WT-PC
Aberdeen Proving Ground, MD 21005-5066

8. PERFORMING ORGANIZATION REPORT NUMBER
ARL-TR-857

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSORING / MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Infrared spectra of atmospheric pressure counterflow diffusion flames inhibited by Halons and a few of their potential replacements are measured using Fourier transform spectroscopy. Results are compared to spectra of similar flame systems examined at low pressure. It is shown that for atmospheric pressure counterflow diffusion methane/air flames inhibited by CF_3Br, CF_2H_2, and CF_4, the two major fluorine-containing combustion products are HF and CF_2O. A correlation is shown between flame inhibition efficiency and CF_2O formation for atmospheric pressure counterflow diffusion flames inhibited by these Halons. For low-pressure premixed flames inhibited by CF_3Br, HF appears to be the only fluorine-containing combustion product, even at relative dopant levels 15 times higher than those capable of extinguishing atmospheric pressure counterflow diffusion flames. The results of these experiments illustrate the need for flame inhibitant testing over a wide spectrum of flame conditions, while providing further evidence that for atmospheric pressure inhibition of real fires by Halons, CF_2O may be a good indicator of inhibitor efficiency when that inhibition is at least partly accomplished by chemical scavenging of reactive combustion intermediates.

14. SUBJECT TERMS
flame inhibition, halon replacement, Fourier transform infrared (FT-IR) spectroscopy, diffusion flames

15. NUMBER OF PAGES
24

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	SAR

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
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ACKNOWLEDGMENT

The authors wish to acknowledge the support of the Strategic Environmental Research and Development Program (SERDP) of the U.S. Department of Defense (DOD). We would also like to thank Dr. Anthony Hamins of NIST for the loan of the counterflow diffusion burner.

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1. INTRODUCTION

Because of concern over the depletion of stratospheric ozone, production and sale of the widely used Halon 1301 (CF_3Br) and Halon 1211 (CF_2ClBr) have been banned (Copenhagen 1987) as of 1 January 1994. A search is presently underway for suitable replacements. To be an acceptable replacement, the new inhibitant must have high fire suppression efficiency, low toxicity, zero residue, compact storage capability, rapid dispersion upon release, and high materials and systems compatibility (Philipczak 1993). The need for environmentally friendly alternatives equal to, or surpassing, the flame inhibition efficiency of Halons 1301 and 1211 is especially important for critical applications encountered in the military. Scenarios range from extinguishment of electrical fires in computer facilities to suppression of a mist-fireball explosion in an armored vehicle following penetration of the fuel cell by a projectile (Finnerty and Polyanski 1993).

The overall goal of the Halon (a contraction of halogenated hydrocarbon) alternatives work being conducted at the U.S. Army Research Laboratory (ARL) is the experimental validation of halogen flame mechanisms developed at the National Institute of Standards and Technology (NIST) (Burgess et al. 1994). Once validated, these models will be used to predict inhibitor efficiency and toxic gas by-product formation. The experimental approach employed at ARL uses infrared tunable diode laser (TDL) (Hanson and Falcone 1978) and Fourier transform infrared (FT-IR) spectroscopies to measure in-situ flame temperatures and concentrations of species participating in the combustion occurring in low-pressure premixed and atmospheric pressure counterflow diffusion flames doped with small amounts of Halons and candidate Halon alternatives. Vibrational spectroscopy was chosen as the diagnostic technique because the measurement does not perturb the flame, and because nearly all of the combustion participants (with the exception of homonuclear diatomics) may be observed in simple infrared absorption spectra.

The two flame systems were selected for different reasons. Flat, low-pressure laminar flow premixed-gas flames are used because at low pressure, flame regions are expanded. This expansion provides better spatial resolution for probing preheating and combustion flame zones (Biordi, Lazzara, and Papp 1975). Atmospheric pressure counterflow diffusion flames are used because these flames may closely approximate real fire scenarios, where diffusion and nonpremixed combustion are important (Linteris, in press [a]). The experimental protocol involves qualitative flame species measurements using FT-IR spectroscopy, while spectral temperature (Ouyang, Varghese, and Cline 1989) and species concentration flame profiles are determined using tunable diode laser spectroscopy. Results from experiments in our lab using diode laser absorption spectroscopy have been reported elsewhere (Daniel et al. 1994).

A drawback to the use of FT-IR spectroscopy for the investigation of inhibited flames is the limited spatial resolution afforded by the polychromatic probe beam. Although the output beam waist may be apertured to less than 1 mm, this results in significant loss of throughput, which decreases the signal-to-noise ratio in the spectra. This combination makes obtaining spatially resolved information difficult. Still, it should be pointed out that some researchers have had success obtaining spatial resolution through combusting systems using FT-IR spectroscopy (Solomon et al. 1986; McNesby and Fifer 1993). Although no effort is made here to quantify spectra of inhibited flames measured using FT-IR spectroscopy, significant insight into the nature of flame inhibition may be gained from qualitative interpretation of FT-IR spectra. This insight into the nature of the inhibited flame is the subject of this report.

2. EXPERIMENTAL

2.1 Atmospheric Pressure Counterflow Diffusion Burner. The atmospheric pressure counterflow diffusion burner is shown schematically in Figure 1. The burner assembly was fabricated at NIST. A brief explanation of the operation of the apparatus is as follows. Fuel (methane) is flowed at atmospheric pressure into the flame region from below. Oxidizer (oxygen or air) and inhibitant is flowed into the flame region from above. The flame appears as a thin, flat luminous disc (with slight edge curvature pointing up toward the exhaust shroud) located between the fuel and oxidizer ports. Flame position in the volume between fuel and oxidizer ports is determined by gas flow rates and stoichiometry. For neat methane/oxygen flames using equal fuel and oxidizer flow rates, the flame disc is located nearer to the oxidizer port because of the stoichiometry of the methane/oxygen combustion reaction. All gases are exhausted from the flame region through an exhaust port that forms a shroud around the oxidizer port.

For the flames studied using the atmospheric pressure counterflow diffusion burner, typical flow rates were 600 ml/min oxygen and 500 ml/min methane. When air was used as the oxidizer, the air flow rate was 2.2 l/min and the methane flow rate was 1.1 l/min. Inhibitant flow varied up to a maximum of 1.3% of the total flow for each system investigated. These flow parameters were selected because they gave the most stable flame for that particular fuel/oxidizer combination.

Flow was controlled by an MKS Instruments Inc. type 147B gas flow controller. Although the burner exhaust shroud was connected to a high-volume vacuum pump, it was necessary to contain the atmospheric pressure counterflow diffusion burner within a large box equipped with optical ports and a chimney attached to a fume hood. This arrangement was to prevent noxious fumes (HF and CF_2O) from entering the main laboratory.

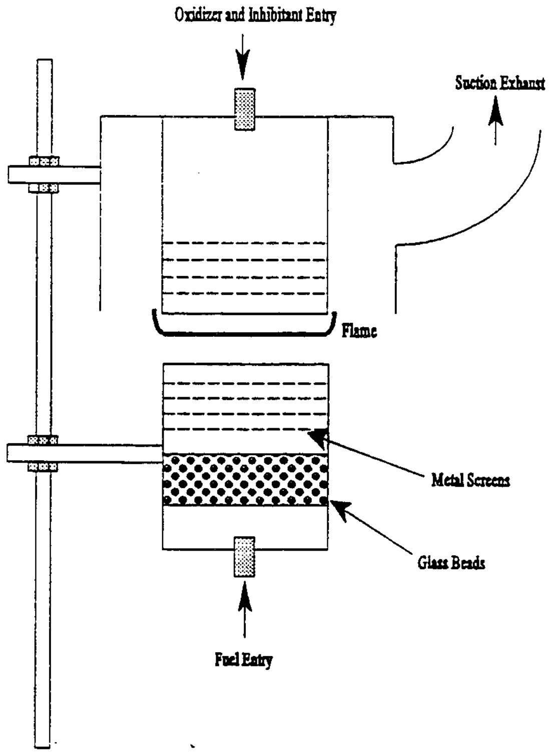

Figure 1. A schematic of the atmospheric pressure counterflow diffusion burner used in these experiments.

2.2 Low-Pressure Burner. Most of the low-pressure burner experimental apparatus has been described in a previous publication (Daniel, McNesby, and Miziolek 1993). The low-pressure premixed methane/oxygen flame was supported on a water-cooled, 6-cm-diameter stainless steel fritted, flat flame burner (McKenna Industries). Gases were mixed just prior to entering a final mixing chamber immediately below the fritted burner head. Gas flow was controlled by an MKS type 147B gas flow controller. The low-pressure burner was mounted on a translational stage, which was mounted to a linear motion feedthrough. This low-pressure burner assembly was contained in an evacuable chamber equipped with CaF_2 windows. Pressure was maintained within the chamber by a Heraeus-Leybold Model SV-100 rotary vacuum pump and controlled using an electrically actuated MKS type 253A butterfly valve. Pressure inside the chamber was monitored using MKS type 390 capacitance manometers. Typical flow rates were 200 ml/min oxygen and 100 ml/min methane. Halon flow rates varied up to 15% of the total flow. Typical pressure within the chamber during collection of flame spectra was 20 torr. The experimental apparatus is shown in Figure 2.

2.3 FT-IR Spectrometer. The FT-IR spectrometer was manufactured by Mattson Instruments. All counterflow diffusion flame spectra were measured at 4 cm^{-1} resolution employing triangular apodization

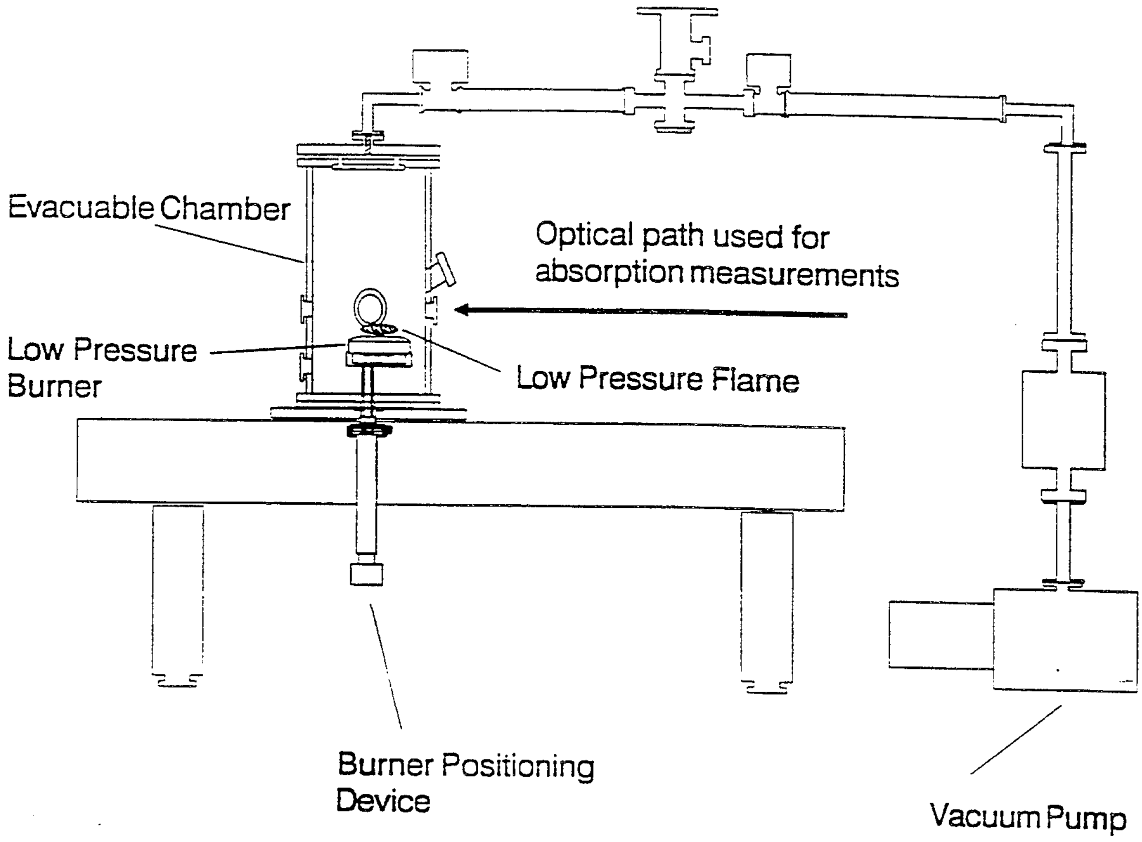

Figure 2. A schematic of the low-pressure burner apparatus used in these experiments, showing the burner inside the evacuable chamber.

using coaddition of 128 scans. Total scan time was approximately 4 min. The infrared beam path was not purged for these experiments. Each single beam flame spectrum was ratioed to a spectrum measured over the same path, but with no flame or fuel/oxidizer/inhibitor mixture present. Since the minimum beam waist diameter of the spectrometer was approximately equal to the distance between fuel and oxidizer ports in the counterflow diffusion burner (see Figure 1), no spatial resolution within the flame was possible, and the resultant spectra show both precursors (fuel and inhibitant) and combustion products.

For the low-pressure experiments, spectra were measured at 1 cm^{-1} resolution, employing triangular apodization using coaddition of 500 scans. Total scan time was approximately 20 min. The infrared probe beam path was purged with dry nitrogen, and each single beam flame spectrum ratioed to a spectrum through the purged beam path with no flame present and the low-pressure chamber evacuated. The probe beam aperture was set to a diameter of 4 mm when passing through the low-pressure chamber. Since the beam position of the infrared probe was fixed, spectra of different lines-of-sight through the low-pressure flame were measured by moving the burner assembly within the evacuable chamber.

For each set of experiments, reflection of infrared emission through the spectrometer and back onto the detector was checked by collecting spectra with the tungsten carbide filament turned off. In all cases, no interferometer-modulated emission was observed. All gases used in these experiments were of spectral purity and were obtained from Matheson, Inc., except for the CF_3Br, which was obtained from PCR Inc. All gases were used as supplied without further purification.

3. RESULTS AND DISCUSSION

Figure 3 shows the absorbance spectrum through an atmospheric pressure counterflow diffusion methane/air flame with no agent added. The methane fuel is the most prominent feature in the spectrum. Figure 4 shows the same flame doped through the oxidizer port side with 1.0% CF_3Br. Addition of CF_3Br beyond 1% of the total flow causes flame flicker and eventual extinguishment, in agreement with previous studies of diffusion flame extinguishment by CF_3Br (Bajpai 1974). Interesting features of this spectrum are the presence of carbonyl difluoride (CF_2O), near 1,950 cm^{-1}, hydrogen fluoride (HF), near 3,700 cm^{-1}, and Halon 1301 (CF_3Br), near 1,200 cm^{-1}. Both HF and CF_2O are highly toxic and corrosive gases (Sheinson, Musick, and Carhart 1981); therefore, care must be exercised when doping any flame with Halon 1301 when extinguishment is either not a goal or does not occur immediately. Figure 5 shows spectra of the atmospheric pressure methane/air counterflow diffusion flame with successive additions of Halon 1301. It is evident from these spectra that CF_2O formation in these flames is dependent upon the Halon 1301 dopant level.

Figure 6 shows spectra of atmospheric pressure counterflow diffusion methane/air flames doped at the 1% level with Halon 1301 (CF_3Br), 1.3% CF_4, and 1.3% CF_2H_2. For the CF_2H_2 doped flame, there is a considerable amount of HF formation near 3,700 cm^{-1}. For the CF_4 doped flame, there is no indication of HF or CF_2O formation, presumably because the CF bond in CF_4 is not broken in the flame (Linteris, in press [b]). The degree to which CF_2O is formed in the atmospheric pressure methane/air counterflow diffusion flames doped with CF_3Br, CF_2H_2, and CF_4 parallels their efficiency as flame inhibitors determined using cup burner experiments ($CF_3Br > CF_3H > CF_2H_2 > CF_4$) (Linteris, in press [b]).

Figure 7 shows spectra of atmospheric pressure methane/air and methane/oxygen counterflow diffusion flames doped with CF_4. The hotter methane/oxygen flame shows significant HF formation, indicating cleavage of the C-F bond in CF_4. It should be noted that there is no spectral evidence of CF_2O formation in this flame, even though HF is formed. For this reason, we believe that it is questionable to correlate HF formation with inhibitor efficiency.

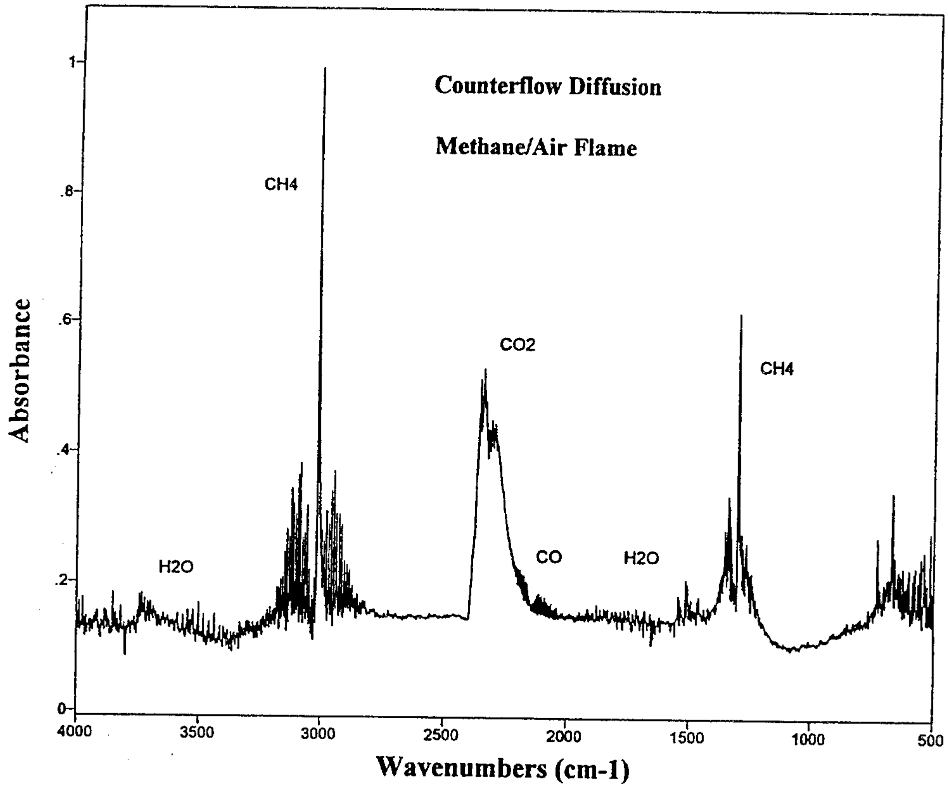

Figure 3. The FT-IR absorbance spectrum measured through an atmospheric pressure counterflow diffusion methane/air flame.

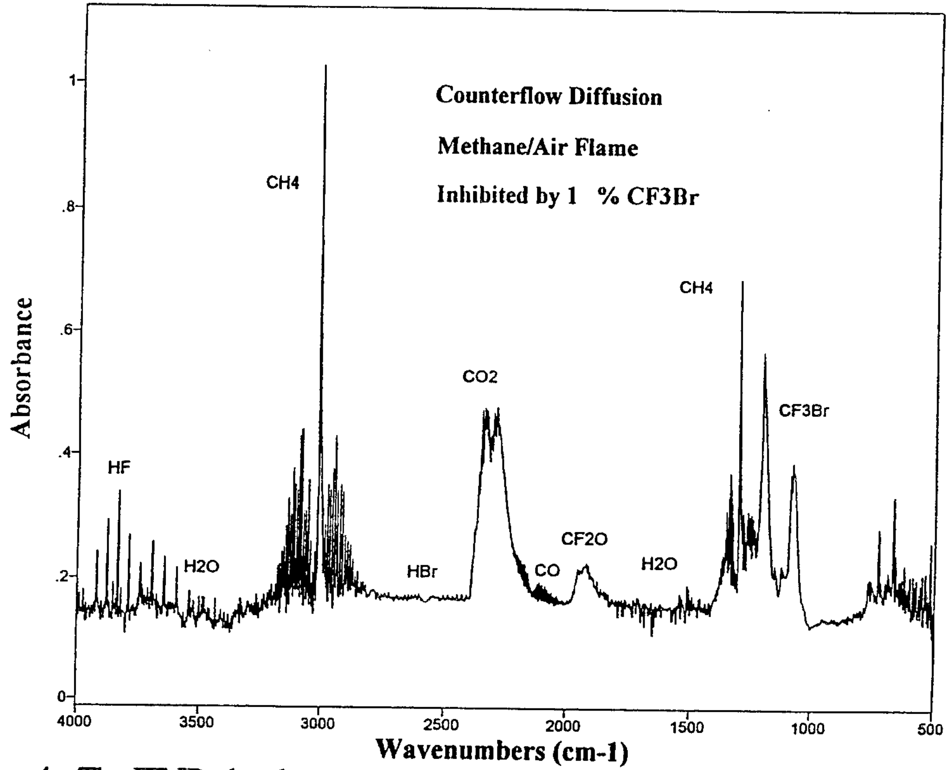

Figure 4. The FT-IR absorbance spectrum measured through an atmospheric pressure counterflow diffusion methane/air flame doped with 1.0% CF_3Br.

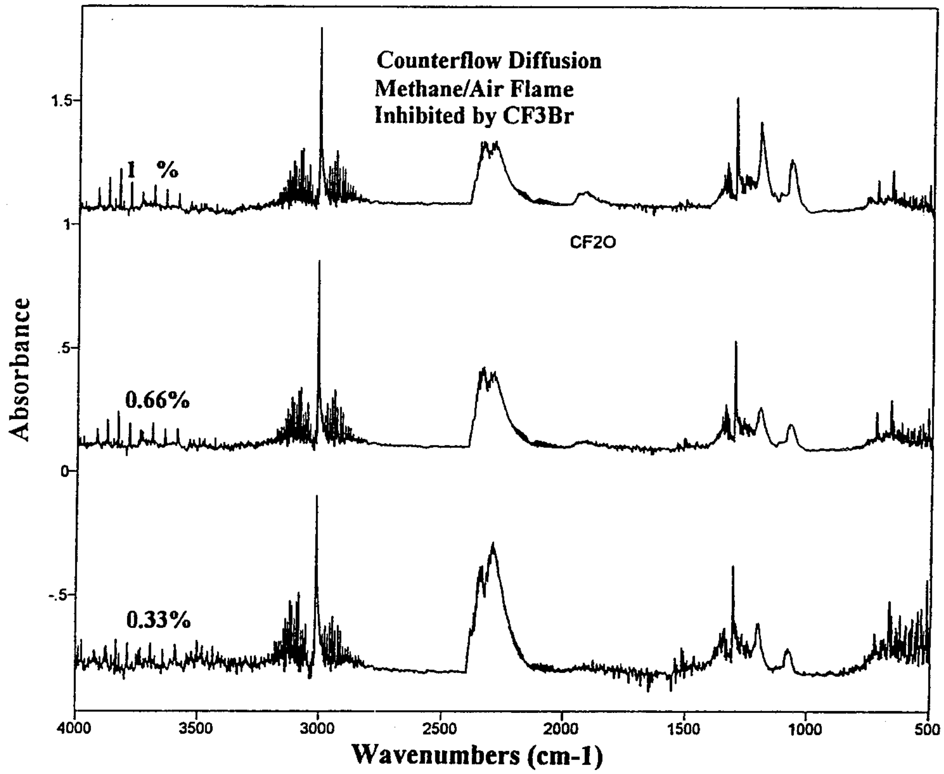

Figure 5. The FT-IR absorbance spectra measured through an atmospheric pressure counterflow diffusion methane/air flame doped with successively increasing amounts of CF_3Br. Note the increase in the CF_2O feature near 1950 cm^{-1}.

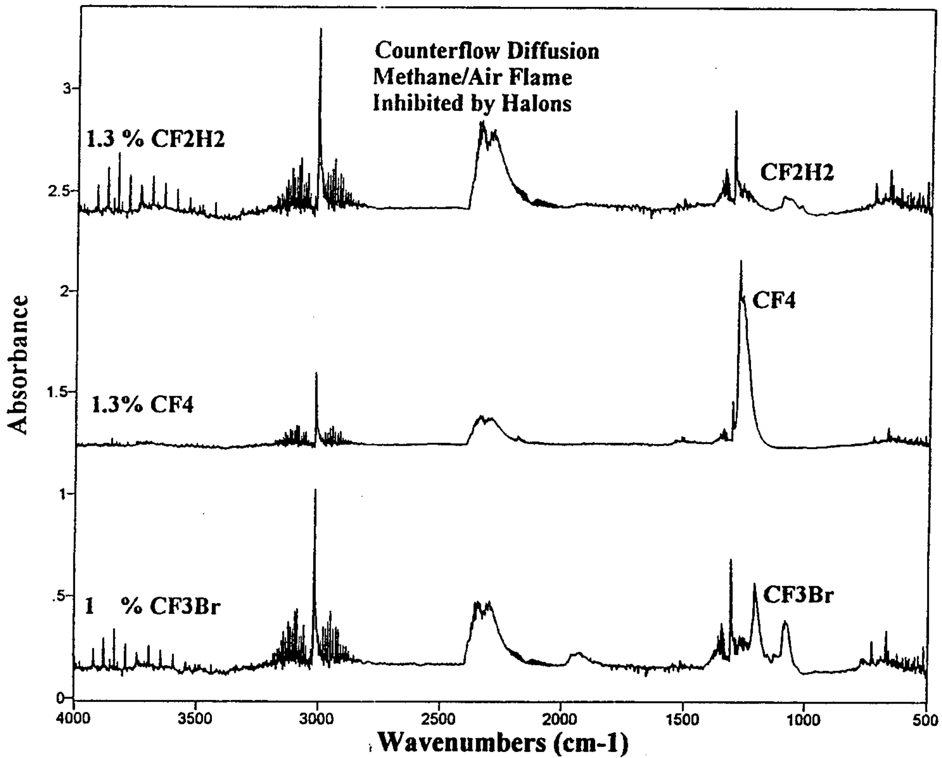

Figure 6. The FT-IR absorbance spectra measured through an atmospheric pressure counterflow diffusion methane/air flame doped with CF_3Br, CF_2H_2, and CF_4.

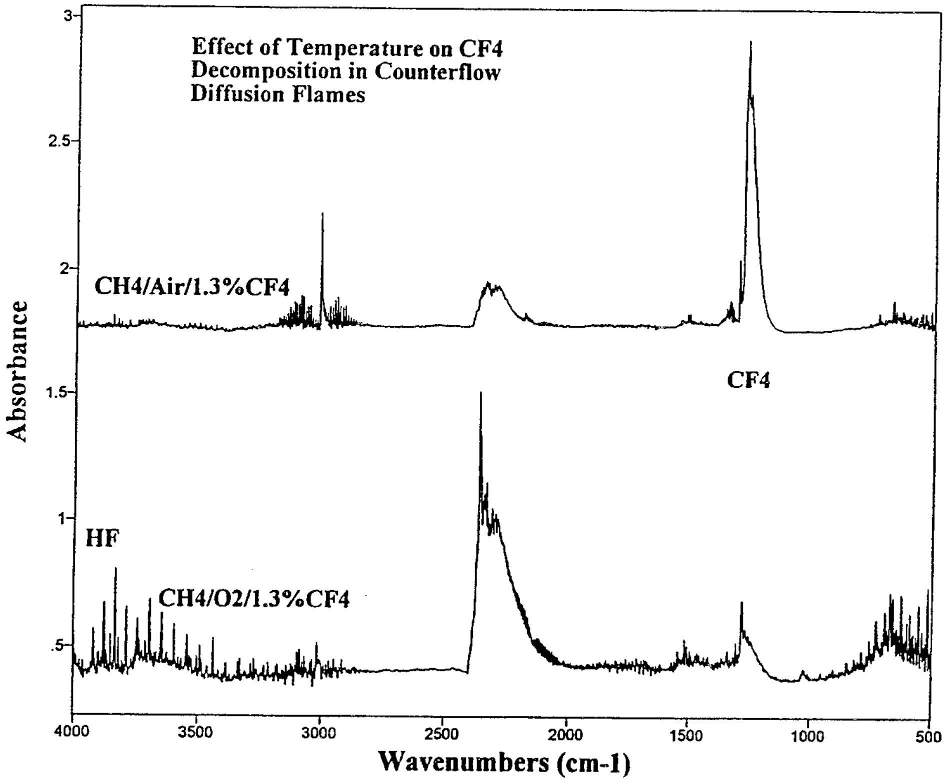

Figure 7. The FT-IR absorbance spectra measured through an atmospheric pressure counterflow diffusion methane/air flame doped with 1.0% CF_4 and an atmospheric pressure counterflow diffusion methane/oxygen flame doped with 1.0% CF_4.

Figure 8 shows the spectra of a low-pressure premixed-gas stoichiometric methane/oxygen flame with and without addition of 2.6% CF_3Br. In contrast to the counterflow diffusion atmospheric pressure methane/air flame doped with CF_3Br, the low-pressure flame shows no evidence of CF_2O formation near 1,950 cm^{-1}. Figure 9 shows the spectrum of the low-pressure premixed-gas methane/oxygen flame doped with 15% CF_3Br. Evident from this spectrum is copious HF formation near 3,700 cm^{-1} and significant HBr formation near 2,600 cm^{-1}. However, even at this high CF_3Br dopant level, there is no indication of CF_2Br formation at any probe beam position within the flame.

Tunable diode laser infrared absorption measurements of low-pressure flames inhibited by CF_3Br have shown that CF_2O is formed and then rapidly consumed. Modeling studies of these results indicate that at high temperatures, CF_2O is decomposed before leaving the luminous zone of the flame (Miziolek et al., in press). We believe that with line-of-sight spatial resolution of less than 1 mm and spectral resolution better than 1 cm^{-1}, CF_2O would have been detected in these flames using FT-IR spectroscopy.

It is noteworthy that Halon 1301 was unable to extinguish a low-pressure premixed gas stoichiometric methane/oxygen flame even at percent total flow levels 15 times higher than those required to extinguish

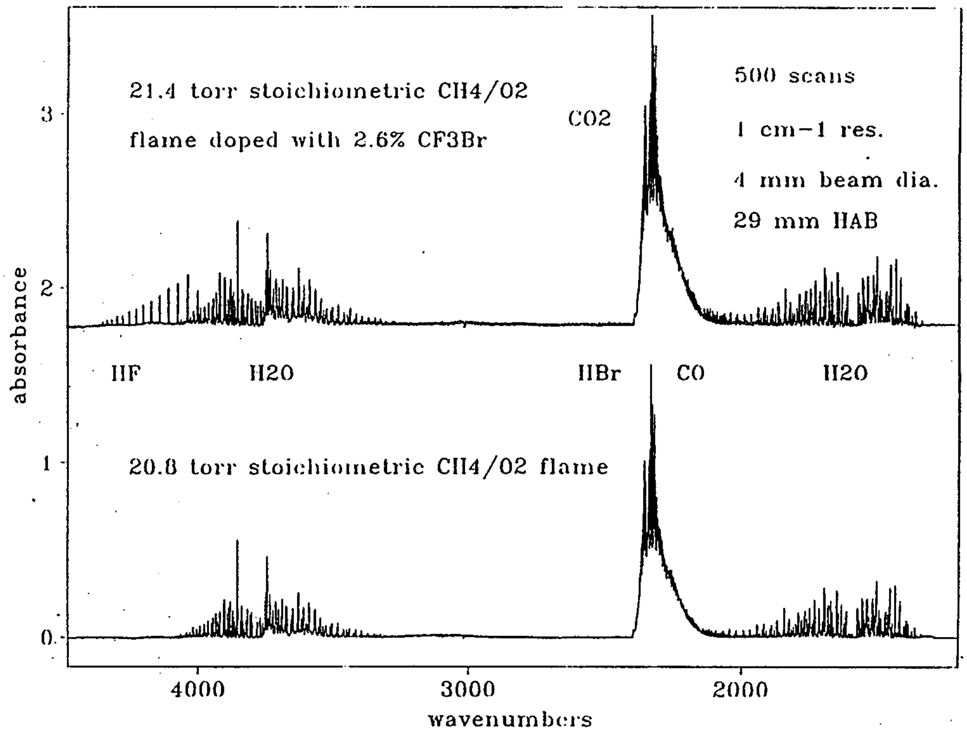

Figure 8. The FT-IR absorbance spectra measured through a low-pressure methane/oxygen flame with and without 2.6% CF_3Br added to the premixture.

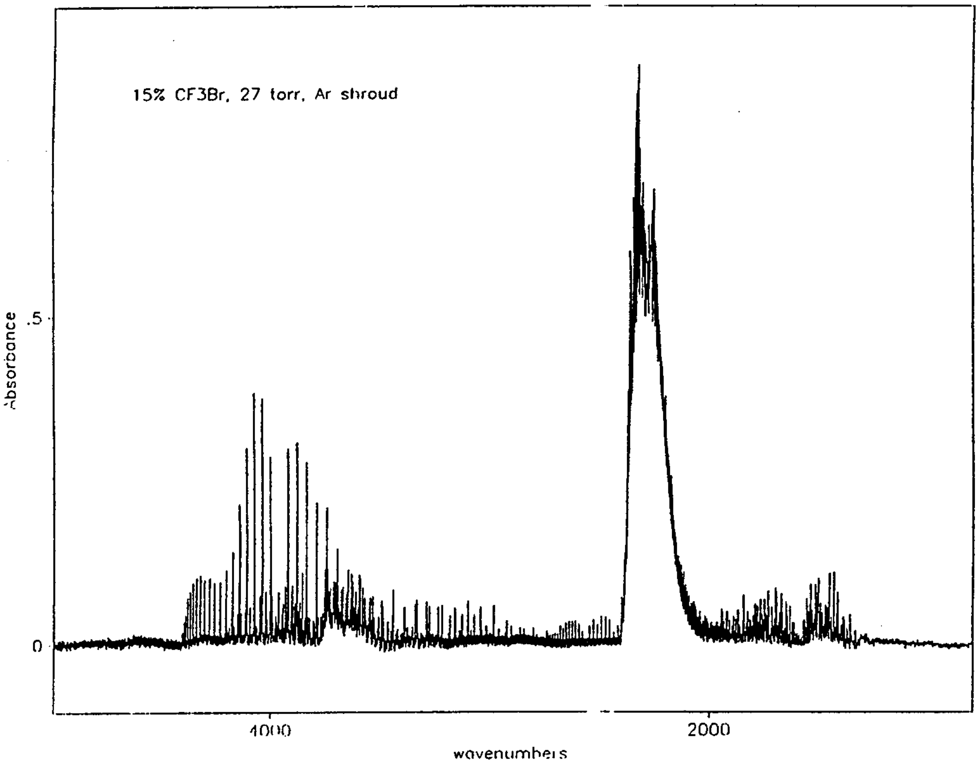

Figure 9. The FT-IR absorbance spectrum measured through a low-pressure methane/oxygen flame with 15% CF_3Br added to the premixture.

atmospheric pressure counterflow diffusion flames. This suggests that the inhibiting effect of Halon 1301 may be most important in flame regions where combustion is nonstoichiometric and diffusion fed. These conditions describe most "real" fires.

Finally, from Figures 4, 8, and 9, it should be noted (assuming similar infrared absorption cross sections for HF and HBr) that there is much less HBr formation than would be expected based on the amounts of HF formation and the molecular formula of Halon 1301 (CF_3Br). This supports calculations that show the rapid removal of HBr from the flame system by reaction (Safieh, Vandooren, and Tiggelin 1982) with H and OH, and the formation of molecular bromine, which is not observable using infrared absorption spectroscopy.

4. CONCLUSION

Although the data are somewhat limited, we believe several conclusions may be reached. Most importantly, we believe the data show that inhibitor effect is highly dependent on the type of combustion system being investigated. Specifically, for atmospheric pressure counterflow diffusion methane/air flames inhibited by CF_3Br, large amounts of CF_2O are observed.

Conversely, for low-pressure premixed-gas stoichiometric laminar methane/oxygen flames inhibited by CF_3Br, no measurable (by FT-IR) CF_2O is observed. This suggests that inhibition of real flames by Halon 1301 may be most important in regions of nonstoichiometric combustion.

Additionally, CF_2O may be a good indicator of the degree of chemical flame inhibition by Halons. Also, it appears that although CF_4 may decompose in hot flames to yield HF, the absence of CF_2O formation indicates that CF_4 does not participate in flame inhibition by the radical scavenging mechanism assumed to be responsible for chemical flame inhibition by Halon 1301. Finally, both low-pressure and atmospheric pressure flames inhibited by Halon 1301 show less HBr formation than would be expected based solely upon the amount of HF observed and the molecular formula of the inhibitant (CF_3Br). This supports the mechanism of rapid HBr removal from the flame via reactions with H and OH, and the conversion of Br atoms to molecular bromine.

We believe that this rather straightforward experimental setup is a valuable tool and may be used to see differences in flame inhibition of real fires by different agents. The technique may be a useful screening test for candidate replacement agents prior to full-scale testing. We are presently engaged in the construction of a new counterflow diffusion burner that will provide larger flame surface area and be capable of low-pressure operation.

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