

## BURNING RATE OF PREMIXED METHANE-AIR FLAMES INHIBITED BY FLUORINATED HYDROCARBONS

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### Abstract

This paper presents the first measurements of the burning rate of premixed flames inhibited by three fluorinated hydrocarbons whose chemistry is similar to agents which may be used as replacements for  $\text{CF}_3\text{Br}$ . Measurements were made of the reduction in the burning rate of premixed methane-air flames stabilized on a Mache-Hebra nozzle burner. The burning rate was determined with the total area method from Schlieren images of the flame. The inhibitors were tested over a range of concentrations and fuel-air equivalence ratios. The measured burning rate reductions are compared with those predicted by numerical solution of the species and energy conservation equations employing a detailed chemical kinetic mechanism recently developed at the National Institute of Standards and Technology (NIST). This paper presents initial efforts at testing and validation of the mechanism using burning rate data. The mode of inhibition of these chemicals is inferred through interpretation of the numerical results.

### Introduction

Halogenated hydrocarbons are effective and widely used as fire suppressants. However, because of their suspected destruction of stratospheric ozone, the production of these agents, the most popular being Halon 1301 ( $\text{CF}_3\text{Br}$ ) will be discontinued in 1994. There exists a need to develop alternatives to the Halons, to establish the relative effectiveness of alternative inhibitors, and to understand the mechanism of inhibition of the new agents. The agents which are currently being considered are mostly fluorinated hydrocarbons and perfluorinated alkanes. This article describes measurements of the reduction in burning rate of premixed methane-air flames with the addition of three fluorinated inhibitors ( $\text{CF}_3\text{H}$ ,  $\text{CF}_2\text{H}_2$ , and  $\text{C}_2\text{F}_6$ ) which demonstrate some of the characteristics of the alternatives, while having structures simple enough so that their chemistry can be described by a recently developed kinetic mechanism. Methane, although its oxidation is somewhat different than larger alkanes, was also selected because its simple structure is more amenable to modeling. Important insights into the chemical and physical mechanisms of inhibition by fluorinated agents in hydrocarbon flames can be obtained by studying these fuels and agents. As the model is further developed and validated, experiments and calculations will be performed for larger fuels and agents.

### Background

Early studies of the inhibitory effects of halogenated hydrocarbons on flames were conducted in premixed systems. The premixed laminar burning rate is a fundamental parameter describing the overall reaction rate, heat release, and heat and mass transport in a flame. In addition, the reduction in the premixed flame burning rate is useful for understanding the mechanism of chemical inhibition of fires since diffusion flames often have a stabilization region which is premixed, and good correlation has been found between the reduction in burning rate and the concentration of inhibitors found to extinguish diffusion flames (Hastie, 1975). Premixed flame burners have flow fields which are relatively easily characterized, making interpretation of the inhibitor's effect on the overall reaction rate straightforward. Burgoyne and Williams-Lier (1948), Coleman (1951), Belles and O'Neal (1957), and Simmons and Wolfhard (1955)

determined the influence of halogenated methanes on the flammability limits of premixed hydrocarbon-air mixtures. The magnitude of the inhibitory effect and its dependence on halogen type and stoichiometry generally indicated a chemical rather than thermal mechanism. Simmons and Wolfhard (1955) studied the flammability limits of methane-air mixtures with bromine and methyl bromide inhibitors and showed that methyl bromide influenced the limits of flammability in the same manner as equivalent amounts of bromine and methane, indicating that the decomposition products of the halogenated methanes are responsible for the action. Gamer *et al.* (1956), and Rosser *et al.* (1959), and Lask and Wagner (1962) measured the reduction in burning rate of various burner-stabilized hydrocarbon-air flames with addition of several halogenated methanes, halogen acids and halogen dimers. In later work, Niioka *et al.* (1983) experimentally investigated the effect of  $\text{CF}_3\text{Br}$  on the extinction velocity of opposed premixed  $\text{C}_2\text{H}_4$  flames. The inhibitory effect was again found to be more effective in rich flames than in lean flames. Mitani (1983) used experimental results on the decrease of flame propagation speeds of premixed  $\text{H}_2\text{-O}_2\text{-N}_2$  and  $\text{C}_2\text{H}_4\text{-O}_2\text{-N}_2$  flames with  $\text{CF}_3\text{Br}$  addition to derive overall activation energies for the inhibition reactions, which correlated well with measured energies for key inhibition steps.

All studies were in consensus that the magnitude of the inhibition was related to the number and type of halogen atoms present in the reactants, the concentration of the inhibitor, and the equivalence ratio  $\phi$ ; and that the effect was generally too large to be accounted for by thermal dilution effects. This conclusion was based on supporting calculations or measurements showing that the final temperatures in the inhibited flames were not low enough to account for the burning rate reductions observed. The two latter papers argued that the action of the inhibitors was to reduce H-atom concentration in the reaction zone.

Detailed information on the inhibition mechanism of brominated species has been provided by flame structure measurements. Mass spectrometric measurements of stable species concentrations in premixed flames of methane and air were conducted by several investigators: Levy *et al.* (1962) used HBr to inhibit a bunsen flame and concluded that HBr inhibited methane consumption but not CO oxidation; Wilson (1965) and Wilson *et al.* (1969) performed low-pressure inhibited premixed flame studies with methyl bromide, HCl, HBr,  $\text{Cl}_2$ , and concluded that both  $\text{CH}_3\text{Br}$  and HBr act to reduce radical concentrations in the preheat region of the flame by competing with the chain branching reaction  $\text{H} + \text{O}_2 \rightarrow \text{OH} + \text{O}$ , and that  $\text{CH}_3\text{Br}$  lowers the peak OH concentration to near equilibrium values, while uninhibited flames have the characteristic super-equilibrium OH concentrations. Biordi *et al.* (1973, 1975) used a molecular-beam mass spectrometer to provide stable and radical species concentrations as a function of position in low-pressure premixed flat flames of methane and air with and without addition of  $\text{CF}_3\text{Br}$ , and Safieh *et al.* (1982) performed similar experiments with a low-pressure  $\text{CO-H}_2\text{-O}_2\text{-Ar}$  flame. Their data and analyses provided detailed kinetic information on the mechanisms of Br and  $\text{CF}_3$  inhibition under low-pressure premixed, burner-stabilized conditions. Westbrook (1983) used the data of Biordi *et al.* to develop a detailed chemical kinetic model, albeit with many estimated reactions and rate constants, and performed numerical calculations of flame structure for the premixed low-pressure system.

In the research described above, the inhibition is dominated by the chlorinated and brominated species. Recently, studies have been performed in systems containing only fluorinated inhibitors. Vandooren *et al.* (1988) performed molecular-beam mass spectrometer measurements in a low-pressure premixed flame of  $\text{CO, H}_2, \text{O}_2$ , and Ar inhibited by  $\text{CF}_3\text{H}$ , determined the destruction route for  $\text{CF}_3\text{H}$ , and obtained rate expressions for key inhibition reactions. da Cruz *et al.* (1988) measured the burning rate of moist CO flames inhibited by  $\text{CF}_3\text{Br}$ ,  $\text{CFCl}_3$ ,  $\text{CF}_2\text{Cl}_2$ ,  $\text{CF}_3\text{Cl}$ ,  $\text{CF}_3\text{H}$ , and  $\text{CF}_2$ , and found that the effectiveness depended most strongly on number of Br and Cl atoms, and that the inhibitor effectiveness correlated with the rate of reaction of the inhibitor with H-atom. Using molecular beam mass spectrometer measurements in a low-pressure  $\text{H}_2\text{-O}_2$  flame, Richter *et al.* (1990) determined the destruction pathway for  $\text{CF}_3\text{H}$  and found the reaction paths important for CO, CO, and COF, formation and CO destruction.

None of these studies examine fluorinated inhibition in hydrocarbon flames. The present research extends the studies of burning rate reduction to fluorinated inhibitors in hydrocarbon flames and applies a newly-developed kinetic mechanism to model the experiments. Trifluoromethane was selected because it is the smallest molecule which is representative of the fluorinated alkanes and provides the simplest species with which to understand the chemistry which is believed to proceed through the  $\text{CF}_3$  radical. In addition, it is being considered as an agent suitable for total flooding applications because of its low toxicity. Difluoromethane is also relatively simple to model and shows the effect of higher H to F ratio in the fuel and the decomposition pathway which proceeds through the  $\text{CHF}_2$  radical. Fluoroethane is an example of a perfluorinated agent. These compounds have been argued to have either less chemical effect or to be inert (due to the absence of the hydrogen atom which is more easily abstracted than a fluorine).

Numerous techniques exist for measuring burning rates of flames and there are many good reviews in the literature (for example: Linnett, 1953; Andrews and Bradley, 1972). All of the flame and burner geometries employed, however, cause deviations from the desired one-dimensional, planar, adiabatic flame. In the present research, a premixed conical Bunsen-type nozzle burner was selected. This method was selected for the flame speed measurements because its simplicity allows rapid assessment of the behavior of a number of halon alternatives. The low rate of heat loss to the burner, the low strain rate, and the low curvature facilitate comparisons of the experimental burning rate with the predictions of a one-dimensional numerical calculation of the flame structure. The burning rate in Bunsen-type flames is known to vary at the tip and base of the flame, and curvature and stretch can influence the burning rate (compared to the planar burning rate); however, these effects are most important over small regions of the flame. Although measurement of a true one-dimensional, planar, adiabatic burning rate is difficult, the relative change in the burning rate is more robust. Consequently, the burning rate reduction in the present work is normalized by the uninhibited burning rate. For comparison with the results of other researchers, the absolute burning rates of the uninhibited flames are also presented.

## Experiment

In the present research, the flame speed measurements are performed using a nozzle burner (Mache-Hebra, 1941). The burner consists of a quartz tube  $27 \pm 0.1$  cm long with an area contraction ratio of  $4.7 \pm 0.1$  and a final nozzle diameter of  $1.02 \pm 0.005$  cm. The nozzle contour was designed to produce straight-sided Schlieren and visible images which are very closely parallel. The burner is placed in a square acrylic chimney 10 cm wide and 86 cm tall with provision for co-flowing air or nitrogen gas. Gas flows are measured with digitally-controlled mass flow controllers (Sierra Model 860\*) with a claimed repeatability of 0.2 % and accuracy of 1 %, which have been calibrated with bubble and dry (American Meter Co. DTM-200A) flow meters so that their accuracy is  $\pm 1\%$ . A frame-grabber board in an Intel 486-based computer digitizes the image from a 512 by 512 pixel CID array for subsequent analysis, and the mass burning rate is determined using the total area method.

The product gas temperature was measured with Pt/Pt 6%Rh - Pt/Pt 30% Rh thermocouples which were coated with Yttrium oxide to reduce catalytic reaction on the thermocouple surface. Measurements with two bead diameters (344 and 139  $\mu\text{m}$ ) allowed correction for radiation losses. The gas temperatures are estimated to be accurate to within  $\pm 70$  K. Some deterioration of the coating was observed.

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\*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.

The experimental technique is similar to that used extensively by Van Wonerghem and Van Tiggelen (1954). The present burner, however, is larger, providing less curvature and strain; the flame holder is not water-cooled and the material (quartz) has a much lower thermal conductivity, reducing heat losses to the burner; the flame is shorter so that there is less curvature, strain, and buoyancy-driven flow; and the burner is enclosed in a chimney with provision for any co-flow gas. The mass flow controllers provide additional flexibility in the operation of the burner.

The fuel gas is methane (Matheson UHP) and the inhibitors are trifluoromethane (Matheson), hexafluoroethane (Dupont), and difluoromethane (Allied Signal). House compressed air (filtered and dried) is used after it has been further cleaned by passing it through an 0.01 micron filter, a carbon filter, and a desiccant bed to remove oil droplets, organic vapors, and water vapor. The Row controllers are operated using custom-written software which monitors all of their operating parameters in real time and allows independent specification of the equivalence ratio, inhibitor concentration, and total flow. By varying the total flow while maintaining constant values of the equivalence ratio and inhibitor concentration, the visible flame height is held constant at 1.3 cm to provide a nearly constant heat-loss rate to the nozzle for each value of the inhibitor concentration.

## Model

The structure of the inhibited premixed methane-air flame was calculated using currently available techniques (Smooke, 1982; Kee *et al.*, 1980; Kee *et al.* 1983). The equations of species and energy conservation were solved numerically for the initial gas compositions of the experiments. The solution assumes isobaric, adiabatic, steady, planar, one-dimensional, laminar flow and neglects radiation and the Dufour effect but includes thermal diffusion. The adopted boundary conditions, corresponding to a solution for a freely-propagating flame, are a fixed inlet temperature of 298 K with specified mass flux fractions at the inlet and vanishing gradients downstream from the flame. The calculations employed a chemical kinetic mechanism recently developed at NIST (Burgess *et al.*, 1993; Westmoreland *et al.*, 1994) for fluorine inhibition of hydrocarbon flames. The 85-species mechanism uses the Miller and Bowman (1989) hydrocarbon sub-mechanism (140 reactions) and adds C, (200 reactions) and C, (400 reactions) fluorochemistry. Fluorinated-species reaction rates and thermochemical data are from the literature when available (for example Westley *et al.*, 1991) and are otherwise estimated. Although all of the reactions are not necessary to adequately describe the present flames, the comprehensive full mechanism was used for these initial calculations. Reduction of the mechanism will be performed later after comparison with experiments and examination of the dominant reaction pathways.

## Results and Discussion

The burning rate of the methane-air flame was measured as a function of equivalence ratio. Figure 1 presents the data together with the results of Rosser *et al.* (1954) and Law (1993). As shown, the present measurements are within 5% of those of Law, and somewhat higher (up to 20%) than those of Rosser *et al.*, particularly at  $\phi > 1.0$ . The burning rate of the uninhibited flames together with the model-calculated burning rate is presented in Table 1. The model is predicting burning rates about 20% higher than the measurements. By selection of a slightly different hydrocarbon sub-mechanism, it will be possible (Law, 1993) in continuing research to obtain burning rates within 5% of the measurements; for the discussion in this article, the NIST mechanism described above is used in its present form.

The measured burning rates and final gas temperatures are presented (boxes) for  $\text{CF}_2\text{H}_2$ ,  $\text{CF}_3\text{H}$ , and  $\text{C}_2\text{F}_6$  as a function of the inhibitor mole fraction in Figures 2 to 10 for  $\phi$  from 0.95 to 1.05. Also shown in the figures are the final temperatures and burning rates predicted by the numerical solution of the premixed

$\phi$	Flame Speed cm/s		Inhibition Index $\Phi$		
	Experiment	Model	CF <sub>2</sub> H <sub>2</sub>	CF <sub>3</sub> H	C <sub>2</sub> F <sub>6</sub>
0.95	37.0	44.5	0.82	1.11	2.39
1.00	38.9	41.5	1.03	1.22	2.45
1.05	39.5	49.5	1.22	1.29	2.24

$$\Phi = \frac{[O_2] (V_o - V_i)}{[I] V_o}$$

where  $V$  is the flame speed, the subscripts  $i$  and  $o$  refer to the inhibited and uninhibited cases, and  $[O_2]$  and  $[I]$  denote the concentration of oxygen and inhibitor. The parameter  $\Phi$  is based on an analysis which compares the rates of H atom reaction with oxygen to the reaction of H atom with the inhibitor and inhibitor fragments. Table 1 lists the values of  $\Phi$  calculated for the three inhibitors and three stoichiometries.

These values of  $\Phi$  compare to about 16 for  $CF_3Br$  (Fristrom and Van 'tiggelen, 1979) and 0.86 for CO, (Hastie, 1975). The low values of  $\Phi$  for these inhibitors imply that they perform slightly better than inert agents such as  $CO_2$  (which have values of  $\Phi$  near 0.8), but much less effectively than  $CF_3Br$ . The inhibitor  $CF_2H_2$  shows a mild increase in  $\Phi$  with  $\phi$ . The values of  $\Phi$  in Table 1 are in accord with the numerical results in Figures 2 to 10; the inhibitors perform slightly better than if they were inert. Of course, once the mechanism is validated through a comparison with a variety of experimental data it can be used to obtain detailed information on the actual mechanism of inhibition of the alternative agents.

## Conclusions

The reduction in burning rate has been determined for the fluorinated inhibitors  $CF_3H$ ,  $CF_2H_2$ , and  $C_2F_6$  in near-stoichiometric premixed methane-air flames at inhibitor concentrations up to about 4%. Even at this early stage of development, the NIST fluorine-inhibition mechanism predicts the burning rate reduction quite well for these flames. The experiments and the modeling results indicate that the inhibitors  $CF_3H$  and  $CF_2H_2$  reduce the burning rate slightly more than if they were inert. Contrary to expectations,  $C_2F_6$  reduced the burning rate more than a factor of two greater than if it were inert. Future research will continue mechanism refinement and validation and examine the chemical kinetic mechanisms of inhibition of hydrocarbon flames by fluorinated species.

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## Figure Captions

Figure 1 - Measured burning rate of premixed methane-air flame as a function of fuel - air equivalence ratio  $\phi$  together with the results of Rosser et al. (1959) and Law (1993).

Figures 2 to 10 - Measured burning rate of premixed methane-air flames as a function of the inhibitor mole fraction for  $CF_2H_2$ ,  $CF_3H$ , and  $C_2F_6$  at fuel-air equivalence ratios of 0.95, 1.00, and 1.05. The boxes present the experimental data; the solid lines the results of the numerical calculation allowing inhibitor reaction, and the dotted lines the numerical calculation with the inhibitor present but non-reacting.

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Figure 1

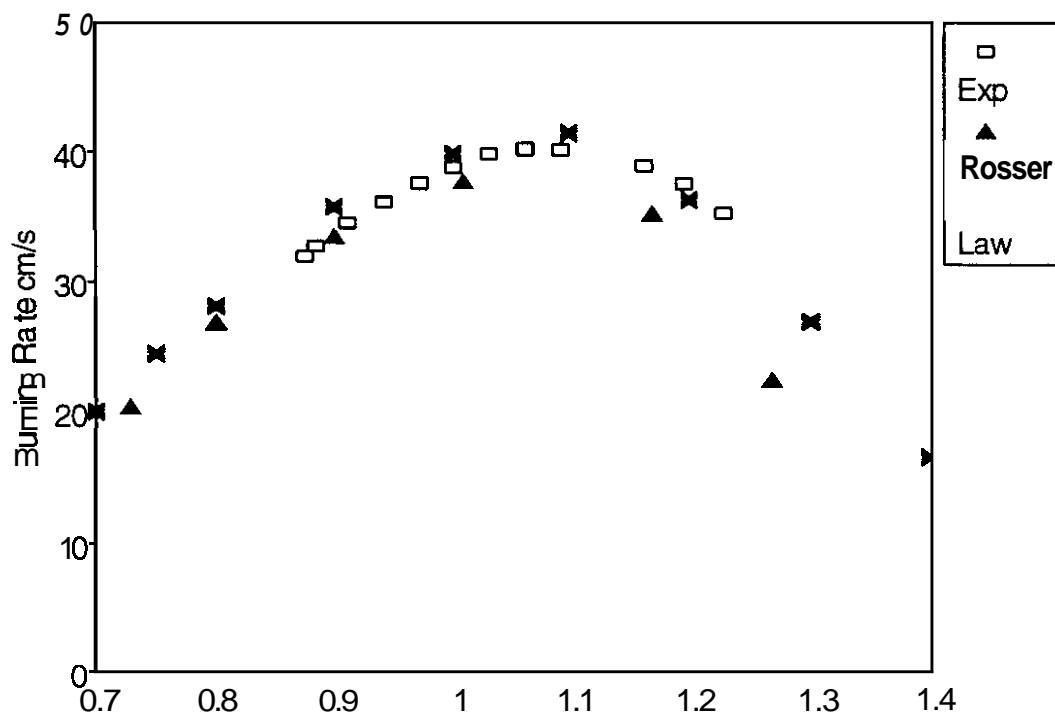


Figure 2

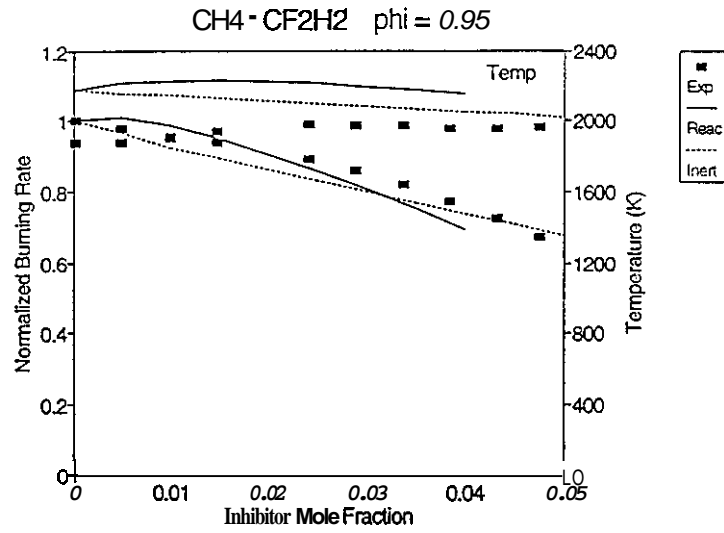
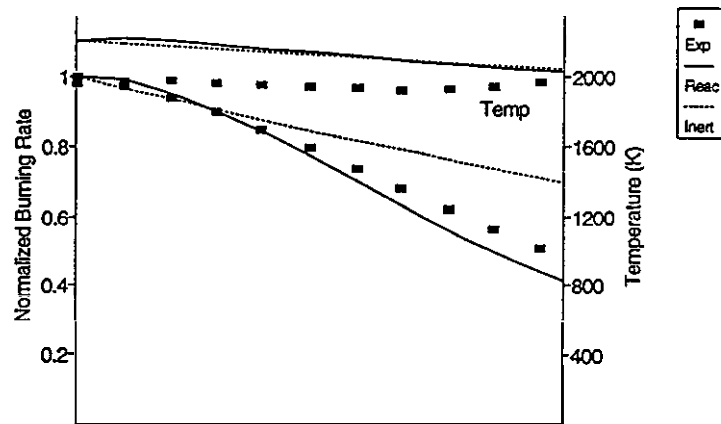
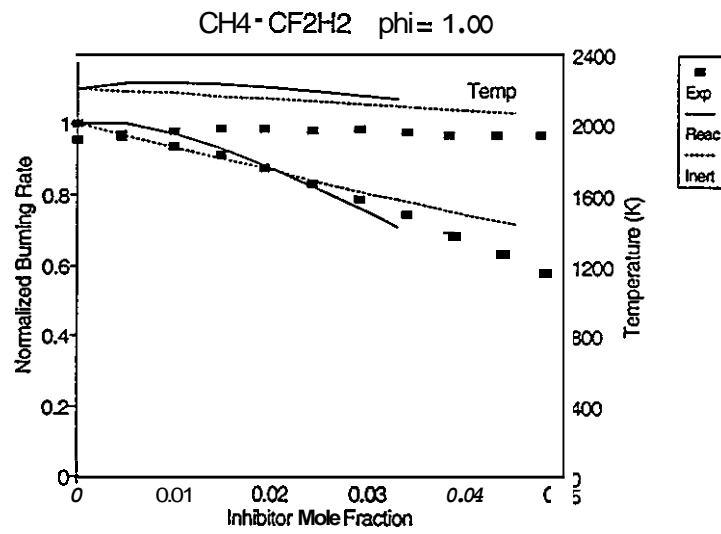


Figure 3



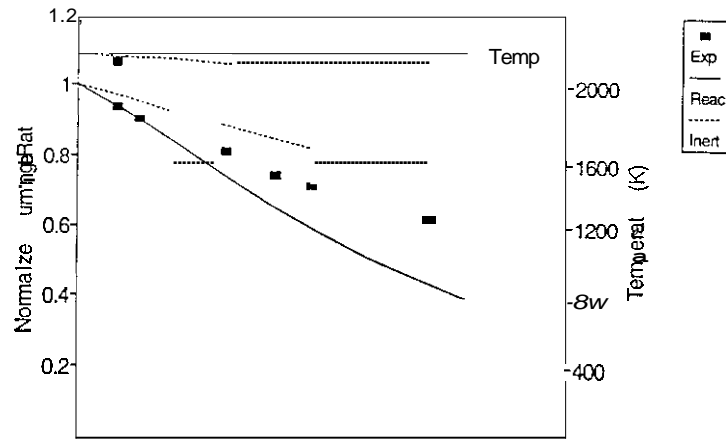


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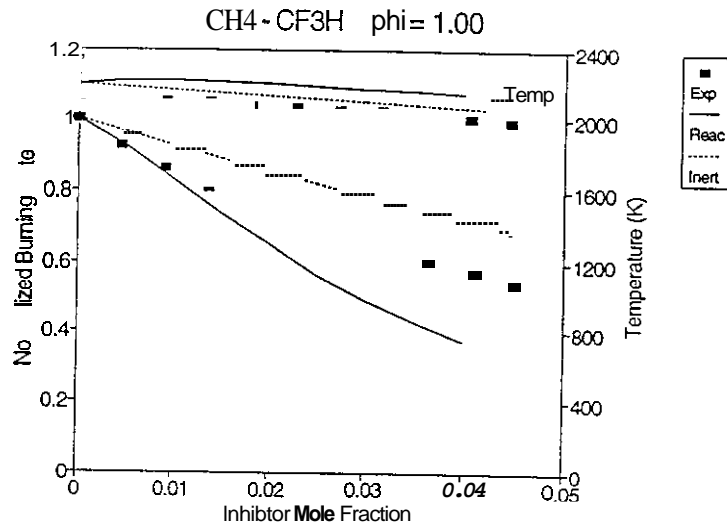


Figure 7

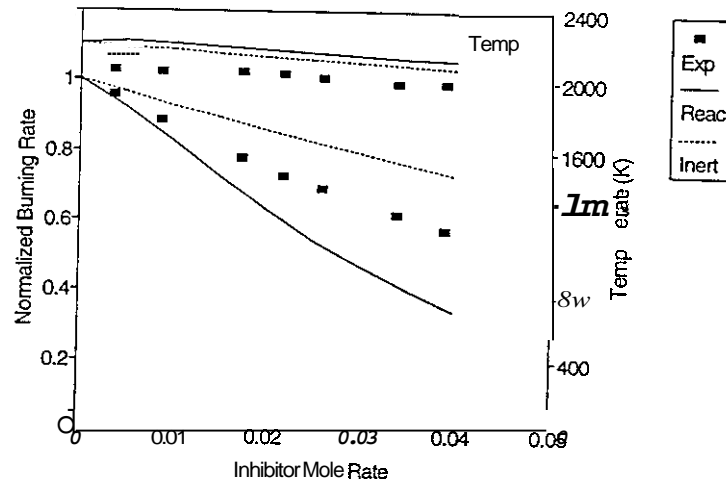


Figure 8

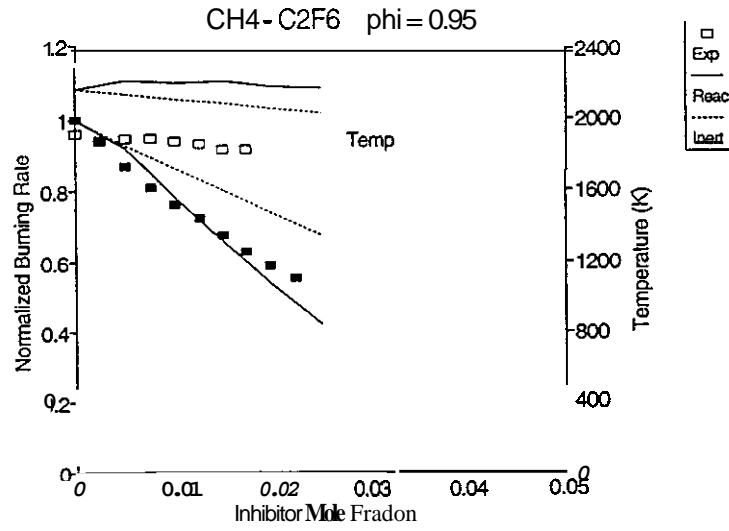


Figure 9

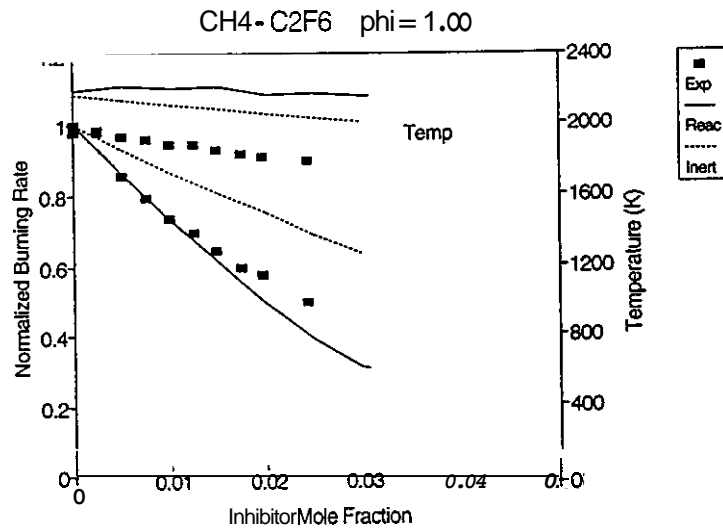


Figure 10

