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The decomposition of many solid reactants during combustion leads to the formation of gaseous hydrocarbons and oxides of nitrogen which can react to support a flame above the surface of the solid. These flames can provide heat which is fed back to the propellant surface and thereby influence the burning rate of the solid. In the case of nitramine based solid rocket propellants, the gas phase decomposition products include significant amounts of CH_2O, HCN, NO_2, NO, N_2O and O_2. This study is intended to provide experimental data on the structure of hydrocarbon flames supported by oxides of nitrogen in order to establish the reaction mechanism for such flames. Laminar, premixed, flat flames of $CH_4/NO_2/O_2$ and $CH_2O/NO_2/O_2$ have been investigated and a reaction mechanism is suggested which accounts for all of the major observations in the data.

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CHEMICAL KINETICS OF NITRAMINE PROPELLANT COMBUSTION

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RESEARCH OBJECTIVES

Many solid rocket propellants and other energetic materials consist of complex chemical compounds of carbon, hydrogen, oxygen and nitrogen. The decomposition of these solid reactants leads to the formation of gaseous hydrocarbons and oxides of nitrogen which can react to support a flame above the surface of the solid. These flames can provide heat which is fed back to the propellant surface and thereby influence the burning rate of the solid. In the case of nitramine based solid rocket propellants, the gas phase decomposition products include significant amounts of CH_2O, HCN, NO_2, NO, and N_2O (Kubota, 1982, Kuo and Summerfield, 1984 and Schroeder, 1985).

Several distinct luminous flame zones are observed in the combustion of nitramine propellants which have also been seen in hydrocarbon flames with NO_2 as an oxidizer. A very rapid, luminous reaction zone is found adjacent to the surface of the propellant followed by a dark, nonluminous zone at greater distances from the surface. Finally, an additional visible flame zone appears after the dark zone. The detailed chemistry of this flame structure is not known at the present time.

In early studies of fuel/NO_2 flames, Parker and Wolfhard (1953) qualitatively characterized flames supported by NO and NO_2. They observed multiple luminous zones in flames with a number of different hydrocarbons as fuels and they also were able to establish a pure NO decomposition flame. More quantitative data have been reported on methyl nitrite, methyl nitrate and ethyl nitrate decomposition flames (Hall and Wolfhard, 1957) which also show multiple luminous zones. Arden et al. (1957) and Hicks (1962) measured stable species composition profiles in a variety of these flames and provide some insight into the structure of the multiple luminous zones. The fuel evaporates from the liquid pool and rapidly decomposes into a variety of products which then react in the gas phase. In the case of the nitrites the products are hydrocarbons and NO. In the case of the nitrates the products are hydrocarbons and NO_2. When NO_2 is formed as a decomposition product, rapid reaction between the hydrocarbon intermediates and NO_2 is observed leading to the formation of NO. In all of the flames where NO is formed, there is the possibility of the exothermic decomposition of NO if the temperature is sufficiently high.

This study is intended to provide experimental data on the structure of hydrocarbon flames supported by oxides of nitrogen in order to establish the reaction mechanism for such flames. Laminar, premixed, flat flames of $Ch_4/NO_2/O_2$ and $CH_2O/NO_2/O_2$ mixtures have been investigated and a reaction mechanism is suggestesd which accounts for all of the major observations in the flame data. These measurements represent the first data on intermediates in the reaction of hydrocarbons with NO_2 in flames.

STATUS OF RESEARCH

Burner Assembly

Flame reactions are followed experimentally by precise, spatially resolved measurements of species concentration and temperature profiles above a one-dimensional, laminar flat flame burner. Reactant gases are metered by Tylan electronic, linear mass flow controllers accurate to 1% and reproducible to 0.3%. The flame resulting from the reactant mixture is stabilized above the burner which is housed in a reaction chamber at a pressure of 50 torr. The chamber allows low pressure operation to distribute the flame reaction spatially and thus enhance spatial resolution of the measurements.

Gas temperature measurements are made using silica coated 0.0076 cm diameter Pt/Pt-13% Rh thermocouples. The coating recommended by Fristrom and Westenberg (1965) is followed in order to minimize the catalytic effects associated with bare platinum wires. Gas samples are withdrawn by quenching quartz microprobes and analyzed by a Varian 3700 gas chromatograph. Possible errors and limitations associated with flame probing have been described by Fristrom and Westenberg (1965) and procedures for minimizing these errors are in common use. The burner is mounted on a micrometer positioning mechanism accurate to 0.01 cm. A reliable gas chromatographic concentration measurement technique for the nitrogenous species N_2, NO NH_3, HCN and N_2O and the combustion products CO, CO_2, CH_4 and H_2 has been developed (Banna and Branch, 1978).

Formaldehyde Generator

Formaldehyde is an intermediate in the combustion of most hydrocarbon fuels and a major reactant in the combustion of energetic materials. It has not been studied as extensively as other species, however, because it is so difficult to generate in the gas phase. We have developed a new technique for the generation of a steady flow of gaseous, monomeric formaldehyde for use in combustion studies (Sadeqi and Branch, 1987).

The major difficulty in the generation of gaseous formaldehyde is its extreme reactivity, even with itself, at ordinary temperatures. Therefore, formaldehyde is usually supplied commercially either in a liquid solution with methanol or as a solid polymer such as paraformaldehyde. A second difficulty in the production of gaseous formaldehyde for use in combustion experiments is in obtaining a steady flow rate for long periods of time. The system that we have developed was designed to overcome these difficulties and provide a relatively pure, continuous supply of gaseous formaldehyde.

A mixture of 70 grams of paraformaldehyde powder per liter of caster oil is supplied to a 2.5 liter stainless steel formaldehyde generator housed in a conventional oven. The generator is approximately half filled with the liquid suspension and half filled with gas. When the oven is heated to 423 K, the suspension evolves gaseous formaldehyde which is bled out a heated outlet line, through a variable area flow metering valve and to the flame burner system. The formaldehyde generator can be maintained at a pressure of 2.0 atm with little interference while continuously extracting several standard liters per minute from the vessel. Considerable care had to be taken in the design of the generator to avoid polymerization of the gaseous formaldehyde and clogging of lines leading from the generator.

The system has demonstrated long term stability with little operational interference. Repeated measurements over time of flames established with the formaldehyde generator have shown that the system can be operated stably for over eight hours. The generator makes possible a variety of studies of continuous combustion systems with formaldehyde as the primary reactant or as an independently variable intermediate.

Laser Induced Fluorescence Spectroscopy

The reactive intermediates which are of interest in this study are primarily OH, H, NH, NH_2, CH, and CN. Identification and quantitative measurements of most of these species and of NO is possible by laser spectroscopy. In addition, gas temperature can be measured by spectroscopy for comparison to thermocouple readings.

The optical arrangement for the laser absorption and laser induced fluorescence measurements is given in Branch et al., 1987. A Lambda Physik EMG 53 MSG Excimer Gas Laser is used to pump a Lambda Physik FL2001 Dye Laser. The excimer laser pulses at 0.1 to 100 Hz and has a pulse energy of 50 mJ at 308 nm. The Lambda Physik FL2001 Dye laser has a wavelength range

of 320 to 970 nm and a pulsewidth of 5 to 20 nsec with a background of less than 1. The combination of the excimer laser and the dye laser with the dyes available, provides high pulse energy, narrow bandwidth and extremely low background.

The laser light output from the dye laser is filtered and a beam splitter used to divert part of the beam to a power meter monitor. Light then passes through the burner pressure vessel and is focused through the flame. Separate focusing arrangements and mirrors are used for the laser absorption and laser fluorescence measurements. The collected light from fluorescence or the attenuated light from absorption is then focused onto the entrance slit of the SPEX 1401, 0.75 m double monochrometer. The signal from the photomultiplier tube is processed by a Stanford Research Systems signal averager and recorder. The Hewlett-Packard 9816S computer is available for controlling the spectrometer scan and other experimental variables and analysis of data from the experiments.

The flame which is being studied is a premixed, laminar, flat flame. The flame is therefore nonsooting to minimize soot extinction and limit background luminosity and laser induced particulate fluorescence. Since the path length through the flame is 8 cm, defocusing is minimal. Laminar flow and the use of electronic mass flow controllers accurate to 1% also eliminate temporal variations in the flowfield. Beam trapping helps avoid spurious scattering of laser light.

The species of interest in the flame studies we are conducting generally have known absorption and emission spectra and are all accessible by the turnable dye laser system. They also have previously determined lifetimes for radiative decay. The spectral lines used were selected so the correction for the effect of temperature on the line intensity was a minimum. Data from the previous observations of these species in other chemical systems aid in the interpretation of the fluorescence and absorption spectra. In addition, the measurement of stable species concentration and of temperature in the flames by gas sampling and gas analysis also aids in interpretation of the fluorescence data. These and other requirements of signal interpretation are discussed in detail in Eckbreth (1981), Bonczyk et al. (1979) and Crosley (1979). Although all necessary corrections are not completely characterized, saturated fluorescence and fluorescence calibrated by absorption or some other technique have been successful in giving reliable species concentration data for laboratory flames.

The accuracy of the stable species composition measurements is approximately 8%, the accuracy of the unstable species measurements is estimated to be 20% and the accuracy of the temperature measurements is about 3%. The flame sampling position is determined by moving the burner relative to the probes or the laser beam with a positioning micrometer. The use of slits in the laser fluorescence collection system gives a position accuracy of 200 microns and is comparable to the dimension of the probe flow disturbance.

Flame Modeling

The data generated by the experimental measurements consist of one-dimensional profiles of species composition and temperature throughout the flame. The data can then be compared to a numerical model of the multicomponent reacting flow including an elementary reaction mechanism for the detailed chemistry of major species and intermediates. The comparison between the experimental profiles and those generated by the numerical model provide insight into the appropriateness of the reaction mechanism and fluid mechanics of the process.

RESULTS AND DISCUSSION

The flames of $CH_4/NO_2/O_2$ mixtures were characterized by two distinct luminous zones separated by dark zones. At the face of the burner where the gas temperature was generally low, there was a dark or nonluminous zone usually a few mm in thickness. Next there was a yellow/orange region which was typically 3 or 4 mm thick followed by the second dark zone. Finally there was a violet luminous zone about 5 mm in thickness. The thickness of the zones could be changed by changing the relative proportion of the reactants. For example, if NO_2 is increased and O_2 decreased the yellow region becomes wider and brighter while the violet region becomes thinner and less bright. A complete listing of the flames studied thus far is given in Table I.

Measured flame profiles for a lean $CH_4/NO_2/O_2$ flame are given in Figure 1 and for a lean $CH_2O/NO_2/O_2$ flame in Figure 2. The flames both demonstrate that NO_2 is much less effective as an oxidizer than O_2. The O_2 in both flames is completely reacted and much of the No_2 remains unreacted in the burnt gas mixture. With pure CH_4/NO_2 flames rich mixtures could not be stabilized on the burner. Measurements of CH_4/O_2 and CH_2O/O_2 flames show that the reaction rate of either fuel with O_2 alone is much more rapid than with NO_2.

There are significant differences in the final products in the methane and the formaldehyde flames. With methane there is some reduction of the NO which is formed from NO_2 into molecular nitrogen whereas with formaldehyde little molecular nitrogen was detected. The limited reduction of NO to N_2 contributes to the flame temperature being much lower than the adiabatic flame temperature since NO decomposition is exothermic. Another difference between the two flames is that H_2 remains in the burnt gas with formaldehyde even though the flame is lean whereas H_2 is only an intermediate at lower concentration in the methane flames.

The intermediate species measured in the methane flame are CH, NH, CN, NH_2, and OH. All but the OH rise to a peak and then decrease quickly in the reaction zone. The CH radical is observed earliest in the flame and the profile has two peaks.

These reactant, intermediate and product species measurements and other recent studies of the kinetics of similar systems (Le and Vanpee, 1985; Vandooren et al., 1986; Thorne et al., 1987), make it possible to suggest a reaction mechanism for both of the flames. Methane conversion proceeds by the sequence

$$CH_4 + R = CH_3 + RH \quad (1)$$

$$CH_3 + R = CH_2O + RH \quad (2)$$

which leads to the formation of formaldehyde. Once formed, formaldehyde reacts according to (Thorne et al., 1987)

$$CH_2 + R = CHO + RH \quad (3)$$

to form HCO which is consumed by the reactions

$$CHO + R = CHO + RH \quad (4)$$

$$CHO + M = CO + H \quad (5)$$

$$CHO + O_2 = CO + HO_2 \quad (6)$$

The formation of CO_2 may result from either of the reactions

Table 1

Reactant Mole Fractions of Methane Flames

Flame	Mole Fractions			
	CH_4	NO_2	O_2	Ar
1	0.11	--	0.24	0.65
2	0.24	0.56	0.20	--
3	0.16	0.73	0.11	--
4	0.12	0.85	0.03	--

Reactant Mole Fractions of Formaldehyde Flames

Flame	Mole Fraction			
	CH_2O	CH_4	NO_2	O_2
5	0.247	--	0.628	0.126
6	0.297	--	0.703	--
7	0.61	--	--	0.39
8	0.346	0.215	--	0.439

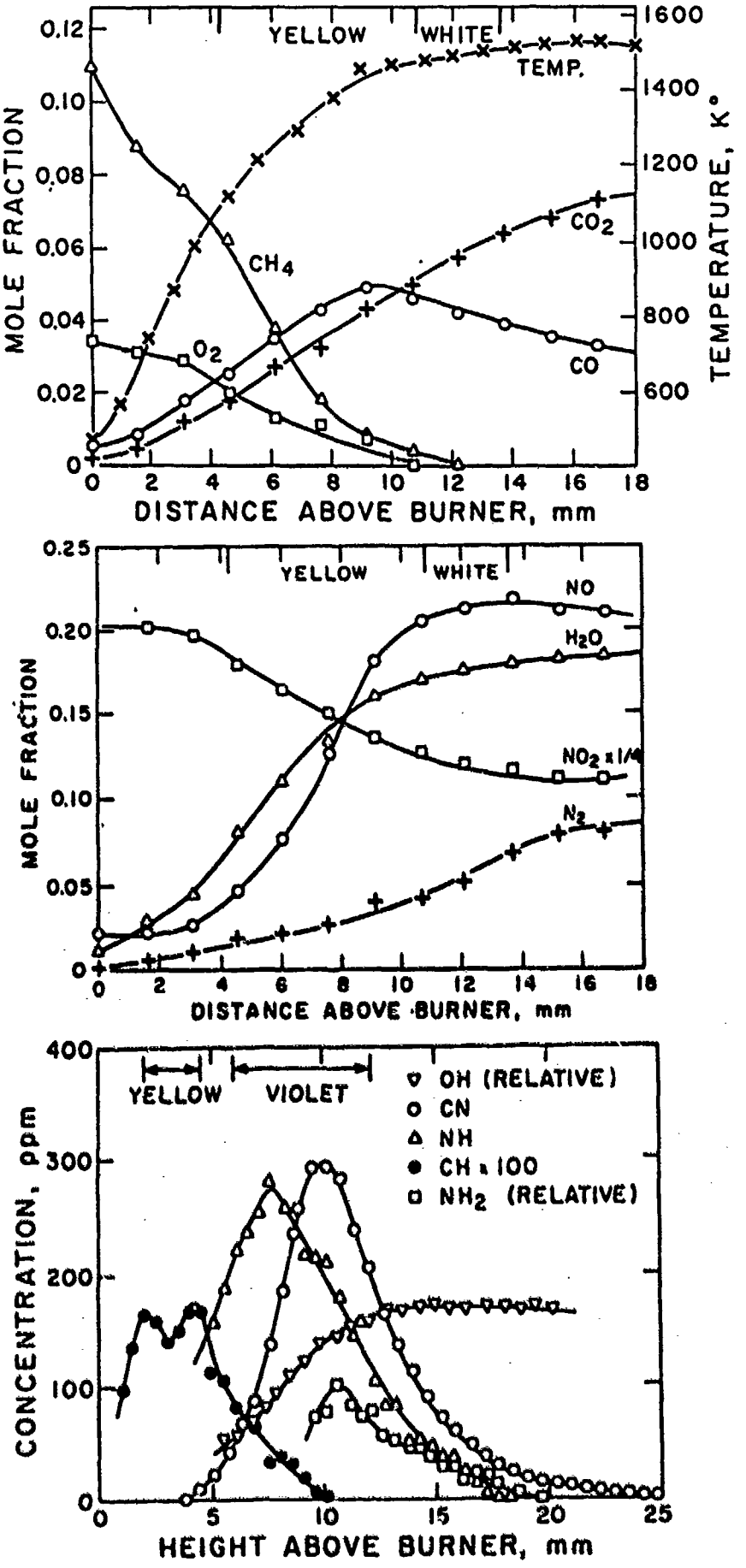

Figure 1. Composition of stable and unstable species and temperature measured in a lean, $CH_4/NO_2/O_2$, laminar, premixed flame at 50 torr. The reactant mole fractions are 0.12 CH_4, 0.85 NO_2 and 0.03 O_2.

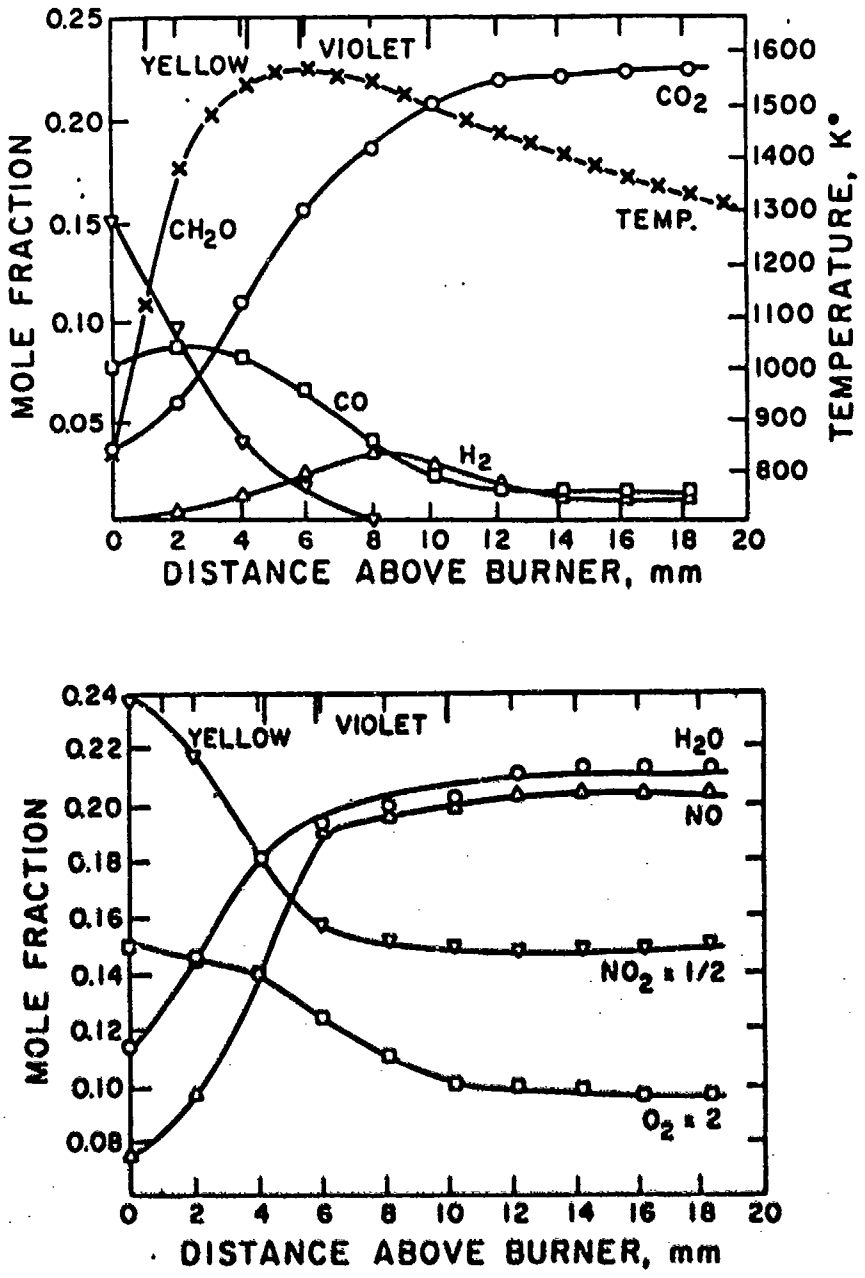

Figure 2. Composition of stable species and temperature measured in a lean, $CH_2O/NO_2/O_2$, laminar, premixed flame at 50 torr. The reactant mole fractions are 0.247 CH_2O, 0.628 NO_2 and 0.126 O_2.

$$CO + OH = CO_2 + H \quad (7)$$

$$CO + HO_2 = CO_2 + OH \quad (8)$$

Some of the CH_3 formed above can react by another path to form CH, for example

$$CH_3 = CH_2 = CH$$

The major reaction consuming NO_2 is

$$NO_2 + H = NO + OH \quad (9)$$

Once CH and NO are formed the other intermediates observed in the flames with methane can result from

$$CH + NO = NH + CO \quad (10)$$

$$CH + NO = HCN + O = CN + OH \quad (11)$$

The molecular nitrogen results from

$$NH + NO = N_2 + OH \quad (12)$$

$$CN + NO = N_2 + CO \quad (13)$$

This mechanism has the virtue of being able to explain nearly all of the observations on the flames we have studied. The limited effectiveness of NO_2 as an oxidizer results from the importance of the chain propagating Reaction 9 when No_2 is oxidizer versus the chain branching reaction

$$O_2 + H = O + OH \quad (14)$$

which dominates when O_2 is oxidizer. This also explains why NO_2 remains in excess in these flames in comparison to the more complete consumption of O_2.

The fact that formaldehyde reacts more readily with NO_2 than methane reacts with No_2 results from the fact that methane must first form formaldehyde in order to complete its oxidation. The initial attack on the methane is well known to be relatively slow.

The observation that N_2 is not found in significant quantities in the formaldehyde flames arises because CHO cannot be converted to CH. Therefore there is no simple path to the formation of CH in the flames with formaldehyde and the path to molecular nitrogen cannot proceed.

CONCLUSIONS

Flames of methane and formaldehyde have been stabilized with mixtures of NO_2 and O_2 as oxidizer, the flame structure measured and the kinetics discussed. Nitrogen dioxide is a poor oxidizer in relation to O_2 due to the chain propagating reaction of No_2 with H atoms in contrast with the chain branching reaction of O_2 with H atoms. In the methane flames some reduction of NO to N_2 is possible because of the formation of CH. In the formaldehyde system little CH is formed and the formation of molecular nitrogen is much more difficult.

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PERSONNEL

Abdulghani Al-Farayedhi, "Laser-Induced Fluorescence Measurements of CH, CN, NH, OH and NH_2 in $CH_4/NO_2/O_2$ and $CH_2O/NO_2/O_2$ Flames, *Ph.D. Thesis*, University of Colorado, Boulder, August 1987.

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Fuad Alasfour, "Flame Structure Modeling of the Gas Phase Decomposition Products of Nitramine Propellants," *Ph.D. Thesis*, University of Colorado, Boulder, in progress.

Mohammed Habeebullah, "Laser-Induced Fluorescence Spectroscopy of $CH_2O/NO_2/NH_3$ Flames," *Ph.D. Thesis*, University of Colorado, Boulder, in progress.

INTERACTIONS

The research has benefited from close interaction with related investigations on H-C-N flame reactions at Sandia Laboratories, Livermore. Professor Branch is a member of the advisory group for the Combustion of Energetic Materials Group at Sandia and participated in a workshop on "Combustion Probes for Solid Nitramines" there in June 1986.

Formal presentations of results obtained in this study have included an oral progress report and abstract entitled "Chemical Kinetics of Nitramine Propellants" presented at the AFOSR/AFRPL Rocket Propulsion Research Meeting, June 1987 in College Park, Pennsylvania. Other presentations and publications from this AFOSR support are summarized in the section, RECENT PUBLICATIONS, from this and previous AFOSR support.

Interaction with Edwards Air Force Base, California has been maintained. The studies at the Air Force Aerospace Laboratory at Edwards Air Force Base are providing data on the distribution of gas product species above the surface of solid rocket propellants, including nitramines. These are among the only studies of this type being conducted. Those studies and the studies we are conducting provide a unique combination of studies on the real propellant and on flames chemically representative of the propellant.