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LASER RAMAN SPECTROSCOPY OF FLAMES;
TEMPERATURE AND CONCENTRATIONS
IN CH₄/N₂O FLAMES

J. A. Vanderhoff
R. A. Beyer
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January 1982



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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I. INTRODUCTION

Major stable decomposition products of HMX and RDX have been shown^{1,2} to be HCHO, N₂O, NO₂ and HCN. It is our plan to study premixed flames of these decomposition products in an effort to understand chemical mechanisms in the combustion of these propellants. Initially we have chosen to investigate CH₄/N₂O flames for several reasons. This flame is experimentally easier to produce so that the emphasis can be placed on developing the diagnostic technique. Secondly this flame system has some features analogous to the HCHO/N₂O flame system in that it reaches similar flame temperature, has one carbon atom, and exhibits nitrogen chemistry (i.e. NH and CN are formed). Finally the CH₄/N₂O flame is luminous which provides a test as to whether the Raman diagnostic technique can obtain sufficient signal to noise ratios from which adequate determination of temperatures and concentrations can be made on a reasonable time scale.

II. EXPERIMENTAL

The experimental apparatus used for generating and detecting Raman signals is shown schematically in Figure 1. A nominal 4W argon ion laser operating at 488 nm is used as the excitation source. The laser cavity has been extended; two highly reflective focusing mirrors with radius of curvature 0.3 and 1.0 m are used to make an intracavity beam waist of approximately 100 μm . The intracavity circulating power is about 50 W and only minor attenuation occurs when a steady CH₄/N₂O flame is inserted in the cavity at the beam waist. The scattered light is imaged onto the slits of a 0.25 m monochromator with two double convex quartz lenses. These are a f/1.33 collector lens with a 10 cm focal length and an f/1.5 focusing lens with 7.6 cm focal length. The detected light is from a sampled volume which approximates a cylinder of 100 μm diameter and 2 mm in length. A silicon intensified target vidicon tube (PAR Corp. Optical Multichannel Analyzer, OMA, Model 1205D) is used to detect the dispersed light. Using a grating of 1180 grooves/mm approximately 40 nm of radiation can be observed at one time with this system. This radiation is dispersed into 500 memory channels which, when coupled with 100 μm monochromator entrance slits, provides a resolution, FWHM, of 12 cm^{-1} . Two memories of the OMA allow for summation of scans and subsequent subtraction of background to obtain the signal due to Raman scattering or laser induced fluorescence. Accumulation times for the data reported here vary from 2 to 300 s per spectrum. For the temperature determination only the spectral data are required; however, for concentration measurements the laser power and a calibration factor are needed to determine absolute concentrations. The small fraction of laser light transmitted by one of the high - reflectivity mirrors is measured with a thermopile to determine the average laser flux during data acquisition. Calibration factors for N₂ and H₂O are obtained from ambient air. Pure H₂, CO, or CO₂ are flowed through the burner for these gas calibrations. The absence of a N₂ Raman signal for these pure gases insures that the flow is sufficient for removing ambient air.

¹C.U. Morgan and R.A. Beyer, "ESR and IR Spectroscopic Studies of HMX and RDX Thermal Decomposition", 15th JANNAF Combustion Meeting Proceedings, CPIA Publication No. 297 (1978).

²B.B. Goshgarian, "Thermal Decomposition of Cyclotrimethylenetrinitramine (RDX) and Cyclotetramethylenetetranitramine (HMX)," Report AFRPL-TR-78-76, Edwards AFB, CA (Oct. 1978).

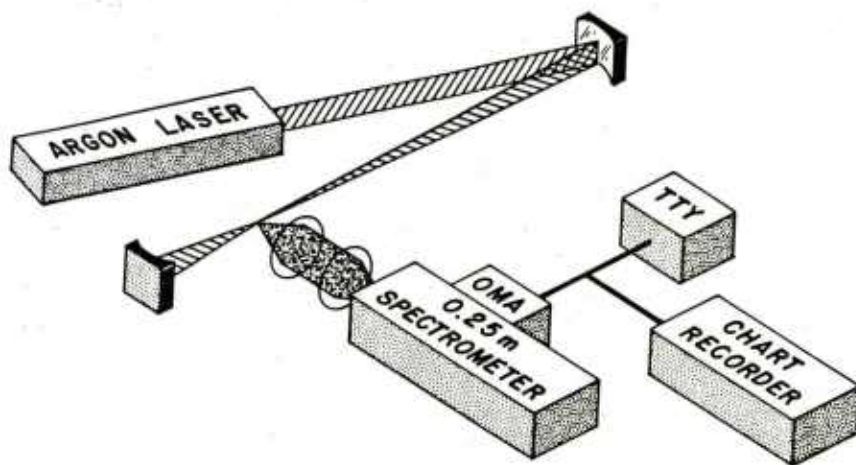


Figure 1. Pictorial diagram of laser Raman experiment.

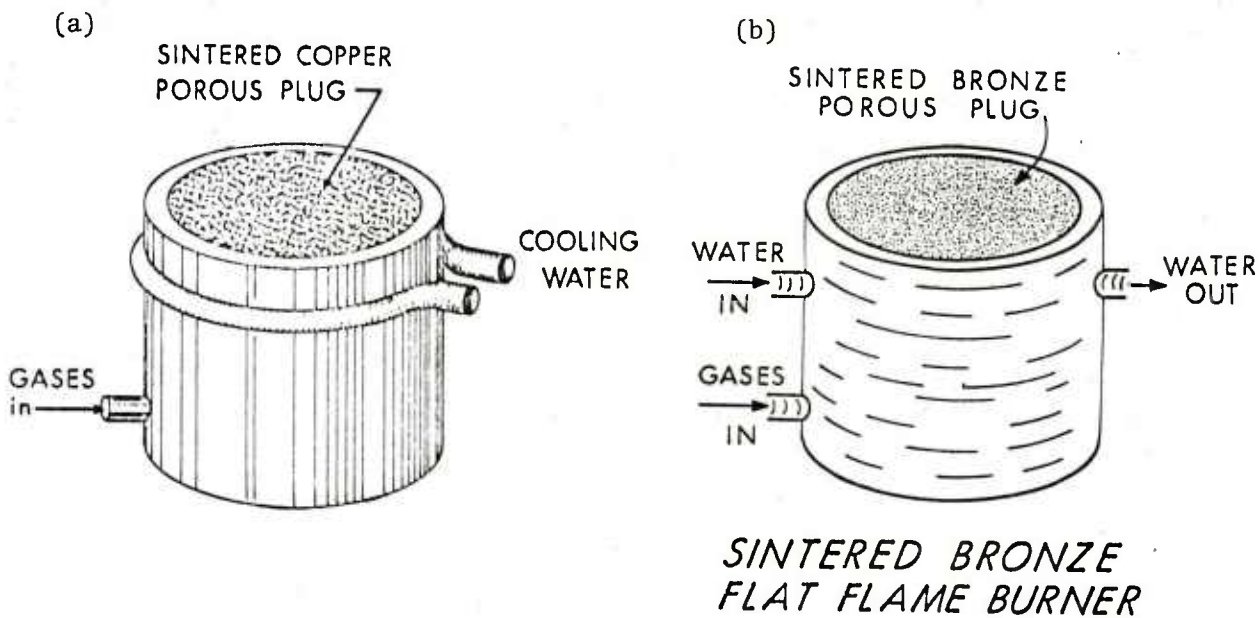


Figure 2. Sintered burners used in this study where (a) is 28 mm in diameter and (b) is 64 mm in diameter.

Two similar types of porous plug burners shown on Figure 2 have been used in this study. Burner (a) is a sintered copper burner with a 28 mm diameter porous plug which is edged cooled. This burner was fabricated entirely in house. Burner (b) is a sintered bronze burner with a 64 mm diameter porous plug and the water cooling coil embedded in the sintered material. This sintered plug was purchased commercially. These burners have the advantage of simple one dimensional geometry, that is the vertical distance above the burner is the only direction in which the flame properties are changing excluding edge effects, and can be operated over a wide range of fuel to oxidizer ratios. The disadvantages at atmospheric pressure are that heat extraction by the burner head cools the flame substantially and the thin primary reaction zone sits on the burner surface limiting its access.

Gas flows have been regulated with capillary flow meters which were calibrated with a wet test meter. The accuracy of the flows should be better than $\pm 5\%$. These burners are used without any gas shroud and some mixing occurs at the air/flame interface.

III. RESULTS AND ANALYSIS

The Raman effect is a light scattering phenomena whereby a small fraction of photons of light scattering from molecules have their energy changed. If the molecule gains energy it is termed Stokes scattering and anti-Stokes scattering if the molecule loses energy. Raman scattering is well suited to probing atmospheric pressure flames since the scattering event is essentially instantaneous ($<10^{-12}$ s). This insures that the process is unaltered by collisions. Raman signals arise from both allowed rotational and vibrational energy levels. The selection rules are $\Delta v=0, \pm 1$ and $\Delta J=0, \pm 2$ where v and J are the vibrational and rotational quantum numbers, respectively. In this report we are only concerned with the transitions where $\Delta v=+1$ and $\Delta J=0$. These transitions are called Raman Stokes Q-branch rotational-vibrational (ro-vib) transitions.

Figure 3 is a plot of 500 channels of OMA data obtained in a $\text{CH}_4/\text{N}_2\text{O}$ flame. Raman Stokes Q-branch ro-vib signals for N_2 , CO , and CO_2 are clearly observable; moreover the v_{0-1} , v_{1-2} , v_{2-3} , and v_{3-4} transitions are distinct for N_2 . The total accumulation time for this data was 157s. Raman Stokes Q-branch signals for H_2 and H_2O are shown on Figure 4. Again the accumulation time was 157s. Due to the large rotational constant for H_2 distinct rotational transitions Q(1) through Q(7) are observed within the v_{0-1} transition for H_2 . Figures 3 and 4 comprise typical examples of data from which temperature and concentration determinations are made.

On an expanded wavelength scale digital data for H_2 , N_2 and CO are shown on Figures 5 and 6 where the data accumulation times are 157, 6, and 320 s, respectively. Any of these signals can be used for a determination of flame temperature; however the signals for N_2 have, by far, the best signal to noise ratio and therefore were used for the temperature determination.

Two methods of extracting temperature from the recorded Stokes Q-branch ro-vib spectra of N_2 were used here. Peak height ratios of different vibrational bands provide a simple method discussed earlier³, to obtain temperatures. Since this method uses a limited portion of the recorded

³J.A. Vanderhoff and R.A. Beyer, "Laser Raman Spectroscopy of Flames: Theory and Preliminary Results", ARBRL-TR-02279, January 1981. AD A096195

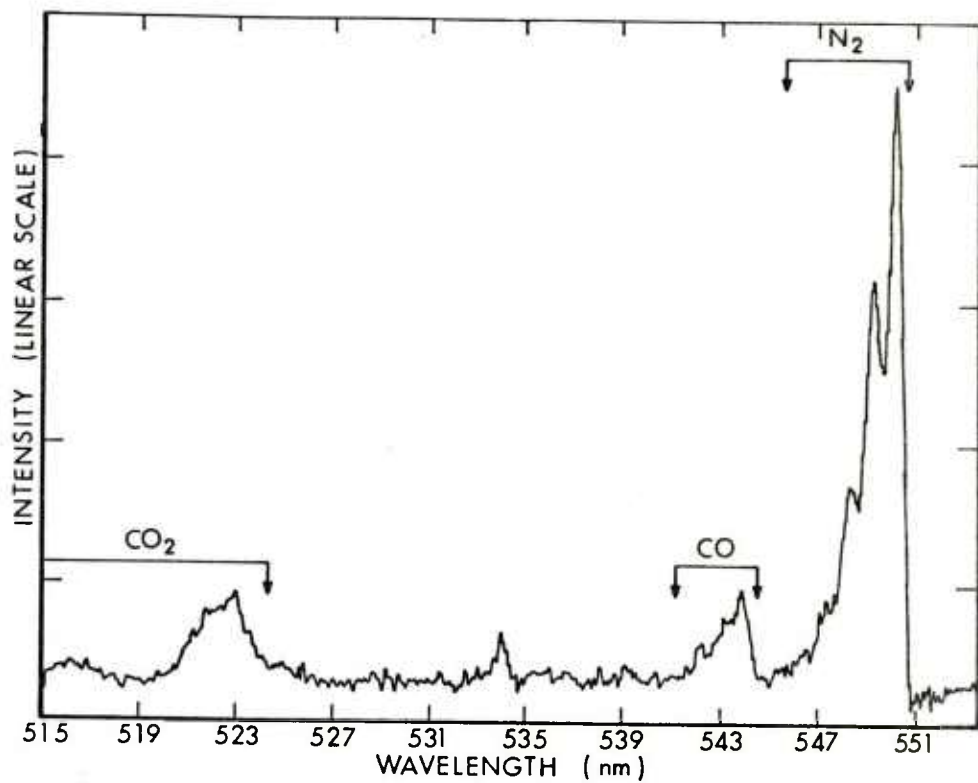


Figure 3. Raman Stokes Q-branch spectrum for N₂, CO, and CO₂.

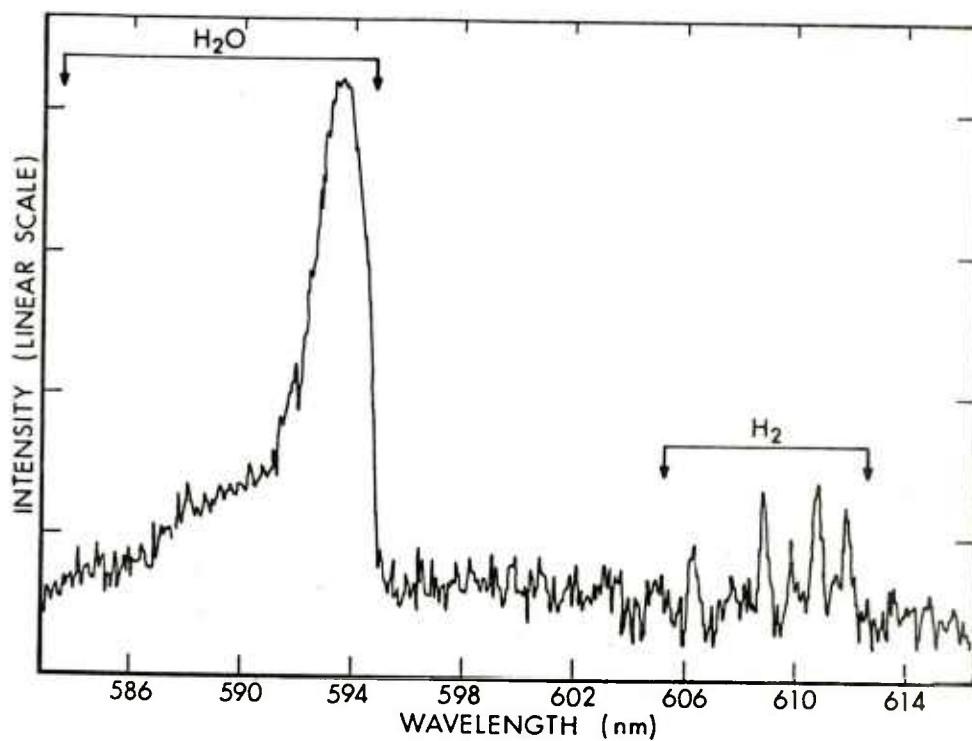


Figure 4. Raman Stokes Q-branch spectrum for H₂ and H₂O.

H₂/RAMAN

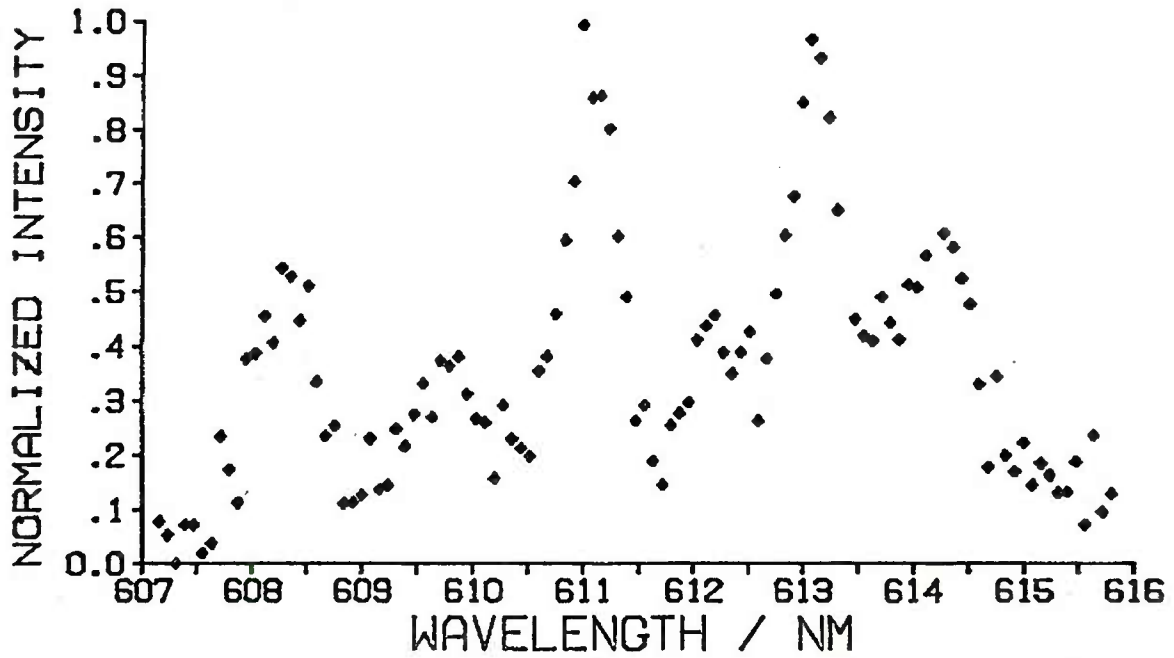


Figure 5. An expanded scale plot of digital Raman data for H₂ obtained in a CH₄/N₂O premixed flame.

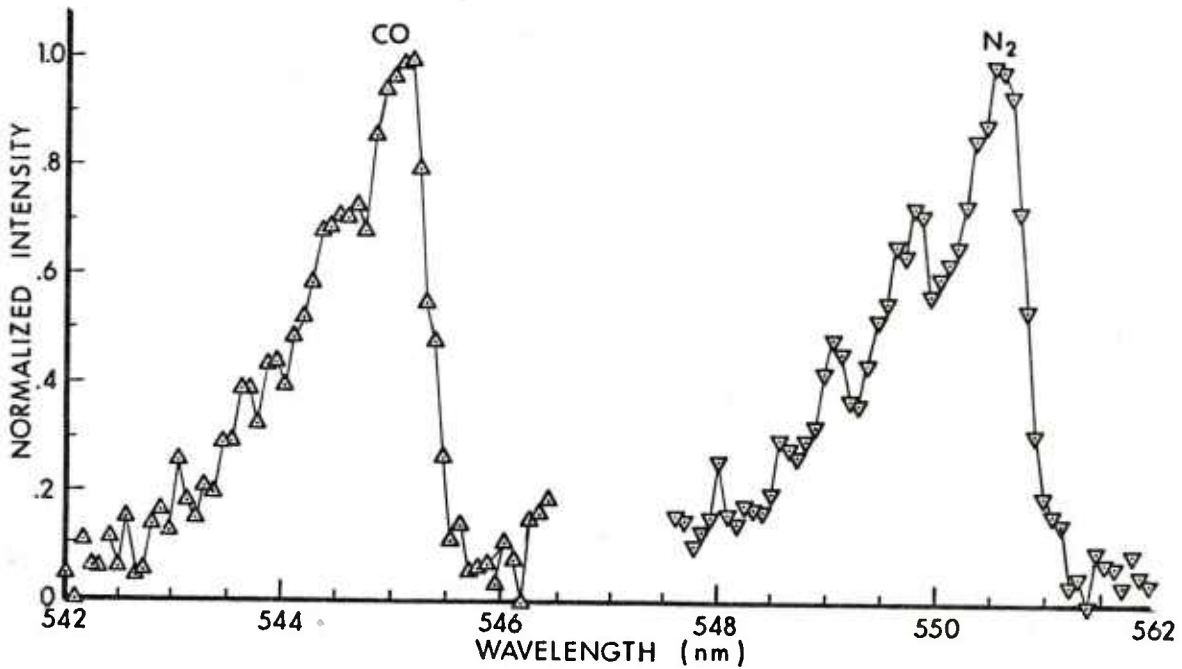


Figure 6. An expanded scale plot of digital Raman data for N₂ and CO. The solid line is used to connect the data points for clarity.

spectrum, a multiparameter least squares Raman fitting program has been developed⁴ to make full use of the entire Raman spectrum of nitrogen. This program also calculates the standard deviation in the temperatures. When comparing the results of the fitting program with the peak height ratio method, one finds that the ratio method is accurate to $\pm 50\text{K}$ in most cases.

For temperature comparison purposes we have measured a vertical temperature profile in the burnt gas region of a stoichiometric $\text{CH}_4/\text{N}_2\text{O}$ flame produced on the 64 mm sintered bronze burner. Eight different positions on a centerline above the burner surface were sampled. The data together with the computer fits of the data are shown on Figure 7a-i. One position 13.5 mm, was sampled twice to check consistency. Three other techniques⁵ have been used to measure the stoichiometric $\text{CH}_4/\text{N}_2\text{O}$ flame temperatures in the burnt gas region using this burner system. These are laser excited fluorescence, laser absorption, and band reversal. Rotationally resolved fluorescence of OH was analyzed to give a temperature of $2199\pm 53\text{K}$. The fitting of laser absorption profiles of OH resulted in a temperature of $2179\pm 21\text{K}$. The band reversal of OH method gave a temperature of $2194\pm 45\text{K}$. These results agree well with the temperatures shown on Figure 7. At distances of greater than 8 mm above the burner surface it appears that the flame starts to cool slightly. The adiabatic flame temperature for an atmospheric stoichiometric $\text{CH}_4/\text{N}_2\text{O}$ flame is 2922K calculated from the NASA-Lewis thermochemical equilibrium code⁶. The significantly lower temperature that we measure is due to heat extraction by the burner head. Crude estimates of the heat being carried away by the cooling water can account for all of the temperature decrease from the adiabatic value.⁵

In a more complete study we have measured temperature and major species concentration in a $\text{CH}_4/\text{N}_2\text{O}$ flame stabilized on the 28 mm sintered copper burner. Two different fuel to oxidizer ratios were studied where the ratios are given in terms of the equivalence ratio ϕ defined as the fuel/oxidizer ratio divided by the stoichiometric fuel/oxidizer ratio. Here the temperatures as a function of height above burner surface are obtained by spectral fitting of N_2 for the cases when $\phi=1.13$. Where $\phi=1.22$ peak height ratios of N_2 vibrational transitions were used. Note that the flame temperature is higher for this burner. This is due to the fact that the burner head operates at a higher temperature because it is edge cooled, which is less efficient.

The number density for N_2 in the scattering volume is obtained from the fitting program with the inclusion of a room air reference (80% N_2) and the measurement of relative laser power for each spectrum. For H_2 , CO , H_2O , and CO_2 areas under recorded spectra have been used to obtain the densities. In these

⁴A.J. Kotlar, *Ballistic Research Laboratory Report, in preparation.*

⁵W.R. Anderson, L.J. Decker, and A.J. Kotlar, "Temperature Profile of a Stoichiometric $\text{CH}_4/\text{N}_2\text{O}$ Flame from Laser Excited Fluorescence Measurements on OH," submitted to *Combustion and Flame*.

⁶R.A. Svehla and B.J. McBride, "Fortran IV Computer Program for Calculation of Thermodynamic and Transport Properties of Complex Chemical Systems," NASA TND-7056, 1973.

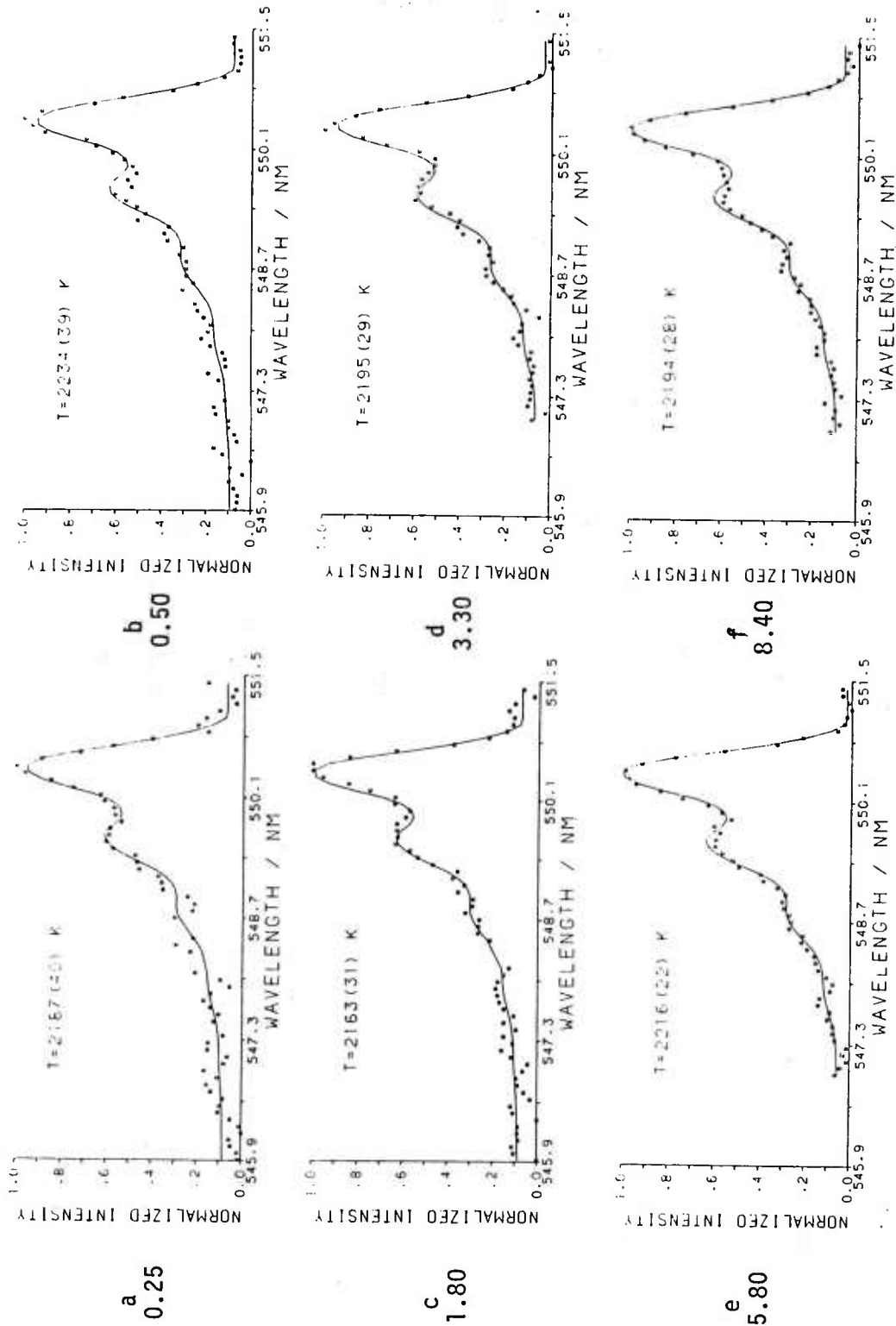


Figure 7a-f. Raman Stokes Q-branch data for N_2 with computer fits (solid lines) used to obtain flame temperatures for eight different vertical positions above the burner surface for a CH_4/N_2O premixed flame. The distances above the burner are given in mm to the left of each graph.

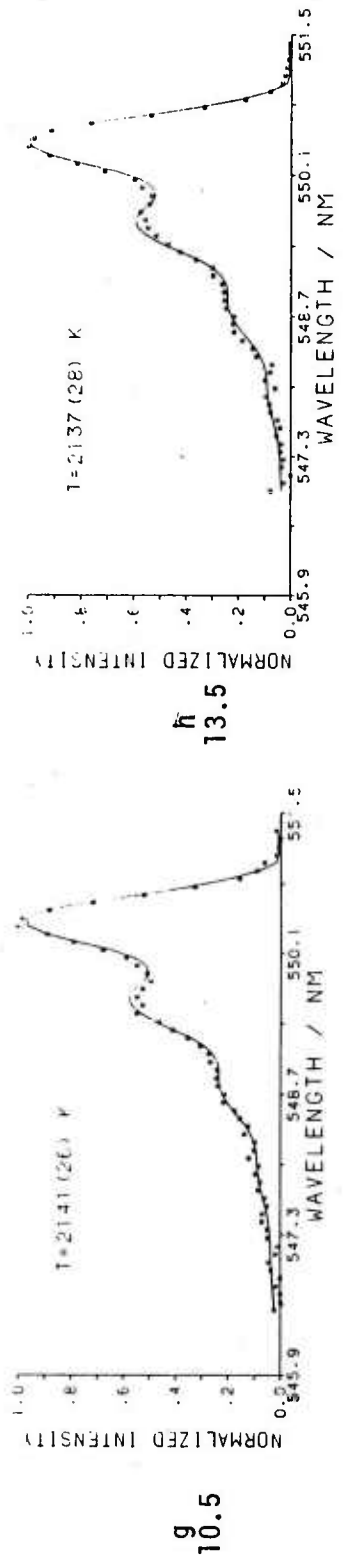


Figure 7g-i. Raman Stokes Q-branch data for N₂ with computer fits (solid lines) used to obtain flame temperatures for eight different vertical positions above the burner surface for a CH₄/N₂O premixed flame. The distances above the burner are given in mm to the left of each graph.

TABLE 1. VERTICAL PROFILES OF TEMPERATURE AND CONCENTRATION, ABOVE THE SINTERED COPPER BURNER SURFACE, IN A CH₄/N₂O FLAME TOGETHER WITH CALCULATED EQUILIBRIUM VALUES

Height (mm)	φ	Temperature (K)	Percent				
			N ₂	Molar CO	H ₂	H ₂ O	CO ₂
2.5	1.13	2510 ± 16	45.6	7.77	4.79	25.1	24.5
5.0	"	2533 ± 19	44.7	6.56	2.81	23.8	21.1
7.5	"	2498 ± 22	45.8	6.44	3.05	25.0	21.3
10.0	"	2490 ± 23	44.7	5.79	3.02	24.3	22.4
NASA LEWIS	"	2500	53.2	5.75	2.71	26.9	9.39
2.0	1.22	2400 ± 50	45.5	10.0	5.1	15.3	15.4
5.0	"	2500	50.8	9.7	4.9	26.7	17.3
10.0	"	2450	60.4	10.3	4.9	30.4	26.7
10.0	"	2300	48.6	8.0	---	---	22.7
3.5	"	2450	58.4	8.6	10.8	30.8	30.8
7.5	"	2350	56.9	7.1	8.4	28.8	21.7
NASA LEWIS	"	2400	51.9	7.5	4.2	27.1	8.3

cases a scaling of $\frac{v+1}{Q_v}$ needs to be applied⁷ where v is the initial vibrational quantum number and Q_v is the vibrational partition function. For N₂ or CO at 2500K, this correction changes the number density values by about 30 per cent. At flame temperatures the signal to noise ratio for H₂ was such that only the rotational transitions through Q(7) within the v_{0-1} transition were recorded; the temperature dependent correction for the fraction of H₂ in other states has been calculated and used here. At 2500K this correction is about 20 percent. Analogous to the work of Stephenson⁸, we have used the v_1 band

⁷J.A. Koningstein, *Introduction to the Theory of the Raman Effect*, D. Reidel Publishing Co., Dordrecht, Holland. 1972.

⁸D.A. Stephenson, "Non-Intrusive Profiles of Atmospheric Premixed Hydrocarbon-Air Flames," 17th Sym. (Int.) on Combustion, p. 993, Leeds England (1978).

area as a measure of H₂O species concentration at room temperature. This approach has been pointed out as a good approximation under conditions where $h\nu_1 > KT$, applicable for H₂O but not CO₂. Although an analysis of the CO₂ Raman spectrum has been reported⁹, we have not yet implemented this approach. The reported values for CO₂ come from the areas of the ν_1 band without correction. However, this approach is adequate for relative concentrations since the flame temperature is practically constant.

As a comparison to the experimental measurements for species concentrations the equilibrium flame code⁶ was used to calculate species concentrations for atmospheric pressure flames of CH₄/N₂O of $\phi=1.13$ and $\phi=1.22$ where the temperature was fixed at 2500 and 2400K, respectively. Here the adiabatic flame temperature for $\phi=1.13$ is 2915K and 2910K for $\phi=1.22$. The horizontal lines in Table 1 separate data sets taken on different days and there appears to be a systematic trend observed in the variation of the concentration for the $\phi=1.22$ case. For each data set, calibration runs were also made to relate flame concentrations to known room temperature concentrations. It is most likely that the variations result from possible changes in the calibration process. Nevertheless, there is a general good agreement between the calculated equilibrium concentrations and the measured values.

Experimental collection efficiencies were improved prior to obtaining the results for the $\phi=1.13$ case and a noticeable improvement in consistency is observed. The experimental results indicate N₂, H₂O, and CO₂ are practically constant as a function of burner height. Temperature is also constant indicating that we are above the reaction zone in a region where the concentrations are near the equilibrium values. CO decreases some with burner height and H₂ exhibits scatter which may be due to low signal levels or slight changes in the flow rate. In this equivalence ratio regime if one changes the fuel flow by 5 percent the calculated H₂ concentration changes by $\sim 50\%$ and the CO concentration by $\sim 25\%$. Hence minute changes in flow can alter these concentrations significantly. As can be seen in Table 1 very good agreement is found between the equilibrium calculations and experimental measurements with the exception of CO₂ discussed previously. Using just the ν_1 band area for H₂O results in obtaining excellent agreement with the equilibrium calculation.

IV. CONCLUSION

We have demonstrated the ability to obtain complete Raman spectra for N₂, H₂, CO, CO₂, and H₂O within several minutes using a CW laser in a luminous flame. From these spectra we are able to extract accurate temperatures with good spatial resolution. Measurements of the major species concentrations are also obtained and agree well with the results of equilibrium calculations.

⁹R.J. Blint, J.H. Bechtel, and D.A. Stephenson, "Carbon Dioxide Concentration and Temperature in Flames by Raman Spectroscopy," *J. Quant. Spectrosc. Radiat. Transfer* 23, 89 (1980).

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