

The Different Effects of Flame Chemistry on the Formation  
of Ethylene and the Oxides of Nitrogen in Flames

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## I. Introduction

The presence of atoms and free radicals in a methane-air flame produces not only carbon dioxide and water but also a large number of trace constituents in part-per-million and part-per-billion concentration levels. These include the oxides of nitrogen, unsaturated hydrocarbons, and partially oxygenated species. This paper will discuss the effects of flame chemistry on ethylene and the oxides of nitrogen. It will be shown that similarities do exist in the formation of these species in flames at atmospheric pressure, but the dissimilarities are of even greater interest than the similarities. In particular, ethylene is found in much higher concentrations in the flame than NO and NO<sub>2</sub> despite the fact that it is thermodynamically unstable whereas the nitrogen oxides are not.

Studies of the reactions of nitrogen, nitric oxide, and oxygen at high temperature indicate that the formation and disappearance of nitric oxide, the primary oxide of nitrogen found in flames and flue products, occur at very low rates.<sup>1</sup> With the additional fact that, in

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<sup>1</sup> Kaufman, F. and Decker, L. J., "Seventh Symposium (International) on Combustion," Butterworths, London, 57 (1958).

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the methane-air flame and its hot reaction products, where there is usually little, if any, oxygen as such, it is apparent that the occurrence of the oxides of nitrogen are due to processes involving the reaction intermediates of the fuel-oxygen reaction. Details of such elementary processes are not known. The evidence obtained from studies on hydrogen-nitric oxide and hydrocarbon-nitric oxide flames indicates that if any nitric oxide exists in the reaction products, it is in equilibrium amounts<sup>2</sup> (that is, with nitrogen and oxygen).

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<sup>2</sup> Wolfhard, H. G. and Parker, W. G., "Fifth Symposium (International) on Combustion," Reinhold, 71<sup>a</sup> (1955).

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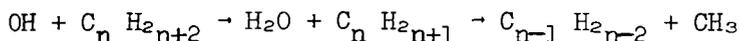
The general pattern of hydrocarbon oxidation in flames has been extensively studied although many of the detailed reactions remain to be identified and measured. It has been indicated<sup>3</sup> that in oxygen-rich

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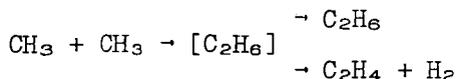
<sup>3</sup> Fristrom, R. M. and Westenberg, A. A., "Flame Structure," McGraw-Hill, New York, 750 (1955).

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saturated hydrocarbon flames, hydrocarbons lower than the initial fuel are formed through the reaction sequence



This is the case when the initial radical formed is higher than ethyl, the  $\text{CH}_3$  radical being split off to form the next lower olefin. In the methane-air flame, however, ethylene and other  $\text{C}_2$  species are expected to arise as a result of the recombination of methyl radicals



From this point of view, ethylene formation in flames might be expected to follow the pattern of the cracking reaction of methane.<sup>4</sup> However,

<sup>4</sup> Palmer, H. B., Lahaye, J., and Hou, K. C., J. Phys. Chem., 72, 348 (1968).

it must be noted, as will be emphasized in this paper, that the rate of formation and decay of ethylene (as well as the oxides of nitrogen) cannot be considered as merely a pure pyrolysis or direct thermal reaction, the chemistry of combustion grossly interfering and competing with these mechanisms.

## II. Experimental

This work was carried out with a large, single-port atmospheric pressure burner. The burner, schematically depicted in Figure 1, consists of a long vertical tube, approximately 3/4 inch in diameter, through which various methane-air mixtures are fed, surrounded by a larger annular region providing a secondary feed to the flame. By means of batch-sampling with a quartz microprobe<sup>5</sup> and analysis with a

<sup>5</sup> See Reference 3, Chapter 9.

Varian Aerograph series 1200 Chromatographic unit (employing a flame-ionization detector), concentration profiles for the various flat methane-air flames were obtained. A batch sampling time of about 15 seconds was used in most cases. The adjustable quartz probe was connected to the sampling and detection loop by means of Teflon-tubing. The diameter of the porous plate burner was 5.2 cm. Aeration of the primary mixture was varied from 84% to 100% to 120% of the stoichiometric air required for combustion. Primary mixture flow rates were varied from 15 to 30 SCF per hour, and secondary air flow rates were varied from 10 to 20 SCF per hour. NO and NO<sub>2</sub> were separated by means of molecular sieve/silica gel column. Ethylene concentration profiles were separated from other flame products by means of a column of Poropak-5 (50-80 mesh). The columns were operated at a temperature of 52°C to ensure good peak readout. Matheson Ultra-High Purity grade methane was employed in the study with no further purification.

### III. Results and Discussion

#### A. Oxides of Nitrogen

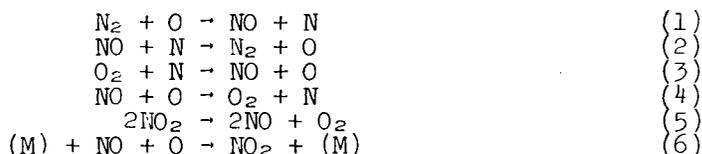
The formation of the oxides of nitrogen in methane-air flames is frequently considered as a side reaction of the combustion process; the oxides forming because air is heated to a high temperature. The data presented here indicates that this is not the case.

Figures 2 and 3 and Table 1 indicate that  $\text{NO}_2$  is found in the "preheat" or transport region between the surface of the flat flame burner and the flame. The concentration of  $\text{NO}_2$  increases from zero to as much as 10 ppm over this 1 mm distance. (In most cases, all reported concentrations are reproducible to within  $\pm 1$  ppm.)

The measured concentrations of the oxides of nitrogen before and after the flame show both an increase in the total,  $\text{NO} + \text{NO}_2$ , and a change in the ratio between these two species. For a stoichiometric methane-air flame with a secondary stream of air the concentrations are 7.5 ppm of  $\text{NO}_2$  and 0 ppm of  $\text{NO}$  at a height of 0.1 cm. At a height of 1.1 cm (about 1.0 cm above the flame), the concentrations along the centerline of the burner have become 2.0 ppm of  $\text{NO}_2$  and 11.0 ppm of  $\text{NO}$ . The decrease in the concentration of  $\text{NO}_2$  is 5.5 ppm while the increase in  $\text{NO}$  is 11.0 ppm. Both Figures 1 and 2 indicate that the bulk species (nitrogen oxides) concentrations are essentially constant in the region above the flat flame.

Beyond this "flame zone" the final ratio of  $\text{NO}$  to  $\text{NO}_2$  is strongly affected by the conditions in the "cooling" region above the flame. For example, in a stoichiometric flame the  $\text{NO}_2$  is observed in increase from about 2 ppm at a height of 1.1 cm to almost 5 ppm at 3.6 cm above the burner. The  $\text{NO}$  concentration decreases from about 10 ppm to 7 ppm over this same distance. The total concentration remains constant at 12 ppm.

A model of the detailed chemistry of  $\text{NO}$  and  $\text{NO}_2$  was developed to explain the rates of formation observed for the oxides of nitrogen. The reaction set includes the following reactions:



The importance of these reactions for  $\text{NO}_x$  formation in flames can be determined by comparing the calculated time required for each of these reactions to form the observed amount of  $\text{NO}$  and  $\text{NO}_2$  with the actual time available for reaction in the flame. In this way, the relative importance of the reactions can be determined. The results are expressed for three specific regions. The regions, of course, are not independent but are closely linked to each other (Table II). The process can be visualized as follows: The fuel-air mixture leaves the burner and flows through the preheat region. Here, the gases rapidly increase in temperature because of the heat flowing back to the burner. The preheated mixture then ignites, causing a rapid change

in composition and a further increase in temperature. These rapid changes in composition from a fuel-air mixture to combustion products together with the steep temperature gradients make the flame analysis complex - hence, the segregation of the flame into regions which are easier to handle by conventional calculations.

In the preheat region,  $\text{NO}_2$  is formed by rapid formation of  $\text{NO}$  and subsequent complete oxidation of the  $\text{NO}$  to  $\text{NO}_2$ . The calculated residence times of reactions 1, 2, 3, and 4 are in good agreement with the actual residence time in the system. Figure 4 shows that the total concentration of the oxides of nitrogen ( $\text{NO}_x$ ) increases while  $\text{NO}_2$  decreases. The kinetics of reaction 5 adequately describe the decomposition step, while reactions 1 and 3 accounted for the increase in ( $\text{NO} + \text{NO}_2$ ). Beyond the combustion region, the concentration of  $\text{NO}_x$  remains constant. The reaction of  $\text{NO}$  with atomic oxygen roughly predicts (within  $\pm 10$  ppm) the observed oxidation.

In an attempt to refine the calculations for the system, reactions 1 through 6 were combined with a mathematical model of a flat flame<sup>6,7</sup> so that a complete time-temperature composition history of

<sup>6</sup> Weil, S. A., Staats, W. R. and Rosenberg, R. B., Amer. Chem. Soc. (Div. Fuel Chem.) Preprints 10, No. 3, 84 (1966).

<sup>7</sup> Weil, S. A., Institute of Gas Technology Res. Bull. No. 35, Chicago (1964).

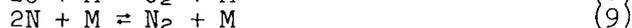
the feed mixture could be determined. This calculation, performed on an electronic computer, did not serve to improve the disparity between observed and calculated values. The computation used the data for rate constants presently available in the literature.<sup>8</sup> The combustion

<sup>8</sup> Schofield, K., Planet Space Sci. 15, 643 (1967).

reactions were described by an overall kinetic expression of the form

$$\frac{d[\text{CH}_4]}{dt} = k_0 e^{-E_a/RT} [\text{CH}_4]^a [\text{O}_2]^b \quad (7)$$

The sources of the atomic species were assumed to be the equilibrium set of reactions:



The numerical predictions from this model show that this mechanism can produce only about 1 ppm of  $\text{NO}$  and almost no  $\text{NO}_2$ . The most obvious source of error in the model is the assumption of equilibrium of  $\text{O}$  and  $\text{N}$ . It is well known that these species exist in concentrations greater than equilibrium (for  $\text{N}_2$  and  $\text{O}_2$  in a "non-reacting" system) but the exact concentrations at various points in an atmospheric pressure flame are not known.

## B. Ethylene

The concentration of ethylene in various regions of a flame is strongly dependent upon the fuel/air ratio in the primary mixture. For a fuel-rich mixture (84% of stoichiometric air in the primary mixture with a secondary air stream), the peak ethylene concentration is almost 800 ppm. This occurs at a radial position of about 0.7 cm from the burner centerline (the burner diameter is 2.6 cm). The ethylene concentration at the centerline is about 650 ppm. For a stoichiometric primary fuel-air mixture, the maximum ethylene concentration occurs at the same point but its concentration is only 400 ppm. The concentration at the centerline of this flame is almost zero. For a fuel-lean flame (120% of stoichiometric) the maximum concentration of about 300 ppm occurs at the burner centerline. This concentration decreases slowly with distance from the center out to 2 cm. At this point, dilution by the secondary air becomes large, and the concentration decreases from about 240 ppm to about 50 ppm between 2.0 and 2.5 cm. (See Figures 5 and 6.)

One striking difference between  $\text{NO}_x$  and  $\text{C}_2\text{H}_4$  is in the radial concentration profiles. As noted, both the fuel-rich and the stoichiometric flames show peak ethylene concentrations at 0.7 cm from the burner centerline. By contrast, the peak  $\text{NO}_x$  concentration occurs at the centerline in both of these flames, the concentration decreasing with increasing distance. The shape of the  $\text{NO}_x$  vs. distance curve changes as a function of the fuel/air ratio but the position of the maximum concentration is always at the burner centerline.

A second difference between  $\text{NO}_x$  and  $\text{C}_2\text{H}_4$  involves the postcombustion reactions. Ethylene forms in the visible flame region in concentrations as high as 800 ppm. Most of this ethylene reacts in the post-combustion region with 3 ppm or less persisting to a height of 3.5 cm above the burner. Consequently, the conditions in the post-combustion region are of primary importance in determining the final emissions of  $\text{C}_2\text{H}_4$ . By contrast, the total concentration of  $\text{NO}_x$  is determined by the flame and not by the post-combustion reactions. The total  $\text{NO}_x$  concentration remains constant in this region with only the ratio of NO to  $\text{NO}_2$  changing. For example, the concentration of  $\text{NO}_x$  produced by a stoichiometric flame is about 12 ppm. Leaving the flame front, the combustion products will contain about 2 ppm of  $\text{NO}_2$  and 10 ppm of NO. At a height of 3.5 cm, the concentrations are 5 ppm of  $\text{NO}_2$  and 7 ppm of NO with the total concentration remaining constant.

## C. Ethylene Formation in Methane-Air Flames

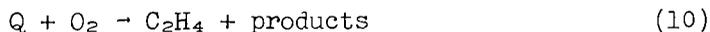
We have experimentally characterized the formation of ethylene ( $\text{C}_2\text{H}_4$ ) in a flat methane-air flame. Analysis of the data has yielded these important conclusions:

- Oxygen has two important effects on  $\text{C}_2\text{H}_4$  formation. 1) Oxygen or air in the primary methane-air mixture decreases the production of  $\text{C}_2\text{H}_4$ . 2) Oxygen from the secondary air has the opposite effect. The diffusion of secondary air into the flame increases the  $\text{C}_2\text{H}_4$  formation, with the increase being greatest for fuel-rich primary mixtures. We have developed a hypothesis for the effect of  $\text{O}_2$  to explain this anomaly which is consistent with studies of ethylene formation in other systems.

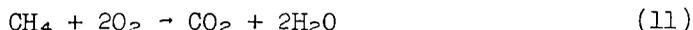
- A methane-air flame does produce significant amounts of  $C_2H_4$  in the combustion products. As much as 5 ppm have been observed. However, almost 800 ppm of  $C_2H_4$  is present in the visible reaction zone of the flame.
- All of the  $C_2H_4$  forms in the visible reaction zone. Most of this  $C_2H_4$  reacts (to form  $CO_2$  and  $H_2O$ ) downstream of the flame. Quenching the flame by impinging it on a cold surface could greatly increase the amount of  $C_2H_4$  in the combustion products by preventing the downstream reactions of  $C_2H_4$ .

#### Hypothesis for Ethylene Formation

We are postulating that ethylene forms via a reaction of some species, as yet undetermined with oxygen. This can be illustrated as follows:



The oxygen has an alternate reaction path available to it:



Thus, there is a competition for the available oxygen between species Q (which forms ethylene) and  $CH_4$  (which does not form ethylene). It is probable that this is not the only path by which ethylene forms, another being -



The effect of oxygen in the primary methane-air mixture is to consume  $CH_4$ . By so doing, it decreases the formation of ethylene via Reaction 12. The effect of oxygen from the secondary air is to enhance Reaction 1 and thereby increase ethylene formation.

For the fuel-rich and stoichiometric flames (Figures 5 and 6), there is a considerable difference in the  $C_2H_4$  concentrations between the burner center line and 0.7 cm from the center line; there is more ethylene at 0.7 cm than at the center line. This must be the result of some physical or chemical difference between the two points that is caused by some outside influence on the system because temperatures and species concentrations across a flat flame burner would otherwise be uniform.

The possibility of temperature differences causing  $C_2H_4$  concentration differences was ruled out; previously obtained profiles show that the temperature difference between these two points is only a few degrees Kelvin - not enough to account for a 150-350 ppm concentration difference.

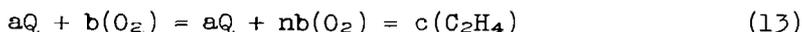
It appears that  $O_2$  enhances the formation of  $C_2H_4$  and that the difference in  $C_2H_4$  concentrations between the two sampling points is the result of differing  $O_2$  concentrations. Essentially,  $O_2$  diffuses into the primary reaction zone from the secondary air stream. A concentration gradient results where there is approximately 0.5-3% more  $O_2$  at a radial position of 0.7 cm than at the center line of the burner (Figures 7 and 8). The additional  $O_2$  at 0.7 cm then causes  $C_2H_4$  to form in excess of what is formed at the center line.

The  $O_2$  profiles show that no oxygen from the secondary is present at the burner center line. Therefore, the value of the  $C_2H_4$  concentration at this point is the value that would appear uniformly across the burner should no secondary air exist.

Beyond a radial position of 0.7 cm (Figures 5 and 6), the lower concentrations of ethylene are probably caused by lower temperatures overshadowing  $O_2$  enhancement. More extensive dilution by secondary air will also account for part of the concentration decrease. In this region the formation reaction or reactions would have the potential of being speeded up by the additional  $O_2$  found nearer the secondary. However, the lower temperatures would have the opposite effect of slowing the formation reaction. Apparently, temperature is the stronger of the two effects. This results in less  $C_2H_4$  being produced at the edges of the burner.

The data indicate that there is also some upper limit of  $O_2$  concentration in the primary, above which  $C_2H_4$  formation is no longer detectably enhanced by secondary  $O_2$ . For a 120% of stoichiometric flame composition, where considerable excess  $O_2$  already exists in the primary reaction zone, the highest  $C_2H_4$  concentration is at the center line and steadily decreases away from the center line (Figure 9).

We believe the excess  $O_2$  from the primary stream overshadows any effect of  $O_2$  from the secondary stream. For example, if some species Q reacts with  $O_2$  to form  $C_2H_4$  and the reaction is fast enough so that all of Q reacted prior to sampling, then the addition of even more  $O_2$  (from the secondary) would not result in detecting more  $C_2H_4$ :



where  $n > 1$ .

Literature lends support to the postulate of  $O_2$  enhancing ethylene formation. Some related experiments by Robertson and Matson (reported by Minkoff and Tipper<sup>9</sup>) indicate that the addition of small quantities

<sup>9</sup> Minkoff, G. J. and Tipper, C.F.H., "Chemistry of Combustion Reactions," London, Butterworths (1962).

of  $O_2$  - up to 2% - to a fuel-rich acetylene mixture can increase ethylene production. More recently, Fenimore and Jones,<sup>2</sup> investigating ethylene flames, found the same indications of  $O_2$  enhancement. The work of Haskell<sup>10</sup> on the pyrolysis of propane between 350° and 700°C

<sup>10</sup> Haskell, W. W., Dissert. Abst. 18, 80 (1958).

shows that an increase in ethylene formation is caused by the addition of small amounts of  $O_2$ . The applicability of Haskell's work to the combustion of  $CH_4$  is, however, in doubt.

### Ethylene in Combustion Products

Our subsequent studies of ethylene formation will center around the primary reaction region because little or no formation occurs in other regions of this specific flame. The  $C_2H_4$  concentration drops drastically just beyond the reaction zone and then remains essentially constant. The drop in concentration is approximately 500 ppm (Figure 1) and is probably due to the reaction of  $CH_4$  with  $O_2$  to form  $CO_2$  and  $H_2O$ . The result is a final concentration of 0.5 to 3 ppm just beyond the reaction zone. The flue gas exiting from the experimental chamber also contains about 3.0 ppm of  $C_2H_4$ . This concentration can presumably be enhanced if the  $C_2H_4$  reactions are prevented from occurring, such as by quenching.

### IV. Conclusion

The data of this work have confirmed that the formation of the oxides of nitrogen in atmospheric methane-air flames is primarily determined by the combustion chemistry with the chemistry of heated air playing a secondary role. Two different processes describe the  $NO_x$ -methane flame system: a) the decomposition of  $NO_2$  to  $NO$ , and b) formation of additional  $NO$  from  $N_2$  and  $O_2$ -derived flame species. The disparity between calculated and observed values is attributed to the lack of a quantitative inclusion of the effects of "superequilibrium" concentrations of  $N$  and  $O$  atoms.

Ethylene is shown to form to a very large extent within the combustion zone of the flame (visible region). Although the final flue products contain little ethylene, concentrations approaching one part per thousand or observed in this flame region. Ethylene formation in flames is seen to be dependent upon post-combustion reactions whereas  $NO$  and  $NO_2$  formation depend only upon flame zone reactions.

### V. Acknowledgement

The authors wish to express their grateful appreciation to the American Gas Association whose support made this work possible.

Table I. CONCENTRATION CHANGES OF NO AND NO<sub>2</sub> WITH INCREASING HEIGHT OF THE BURNER FOR A STOICHIOMETRIC METHANE-AIR FLAME WITH AN AIR SECONDARY

	<u>0.0</u>	<u>-0.7</u>	<u>1.4</u>
	<u>ppm</u>		
0.1 cm Above Burner			
NO	0.0	0.0	0.0
NO <sub>2</sub>	7.5	7.1	5.7
NO + NO <sub>2</sub>	7.5	7.1	5.7
1.1 cm Above Burner			
NO	11.0	2.0	0.0
NO <sub>2</sub>	2.0	5.6	3.5
NO + NO <sub>2</sub>	13.0	7.6	3.5
Conc. Increases			
NO	+11.0	+2.0	0.0
NO <sub>2</sub>	-5.5	-1.5	-2.2
NO + NO <sub>2</sub>	+5.5	+0.5	-2.2

Table II. CALCULATED AND ACTUAL RESIDENCE TIMES\*

	<u>Calculated</u>	<u>Actual</u>	<u>Difference</u> <sup>†</sup>
	Seconds		
<u>In the Preheat Region</u>			
$N_2 + O \rightarrow NO + N$	$0.587 \times 10^{-2}$	$1.22 \times 10^{-2}$	$-0.634 \times 10^{-2}$
$O_2 + N \rightarrow NO + O$			
$N_2 + O_2 \rightarrow 2NO$	$2.0 \times 10^1$	$1.22 \times 10^{-2}$	$\approx 2.0 \times 10^1$
$NO + O \rightarrow NO_2$	$2.12 \times 10^{-2}$	$1.22 \times 10^{-2}$	$+0.90 \times 10^{-2}$
$2NO + O_2 \rightarrow 2NO_2$	$4.25 \times 10^{15}$	$1.22 \times 10^{-2}$	$\approx 4.25 \times 10^{15}$
<u>In the Combustion Region</u>			
$N_2 + O \rightarrow NO + N$	$0.35 \times 10^{-2}$	$1.04 \times 10^{-1}$	$-0.965 \times 10^{-1}$
$O_2 + N \rightarrow NO + O$			
$2NO_2 \rightarrow 2NO + O_2$	$0.78 \times 10^{-2}$	$1.04 \times 10^{-1}$	$-0.922 \times 10^{-1}$
$N_2 + O_2 \rightarrow 2NO$	$1.1 \times 10^1$	$1.04 \times 10^{-1}$	$\approx 1.1 \times 10^1$
<u>In the Cooling Region</u>			
$NO + O \rightarrow NO_2$	$5.0 \times 10^{-2}$	$6.25 \times 10^{-2}$	$-1.25 \times 10^{-2}$
$2NO + O_2 \rightarrow 2NO_2$	$4.8 \times 10^{15}$	$6.25 \times 10^{-2}$	$\approx 4.8 \times 10^{15}$

\* The comparison of these reactions has been made on the basis of the residence time of reaction necessary to form the experimental observed quantity of  $NO_x$ .

† Seconds calculated minus seconds actual.

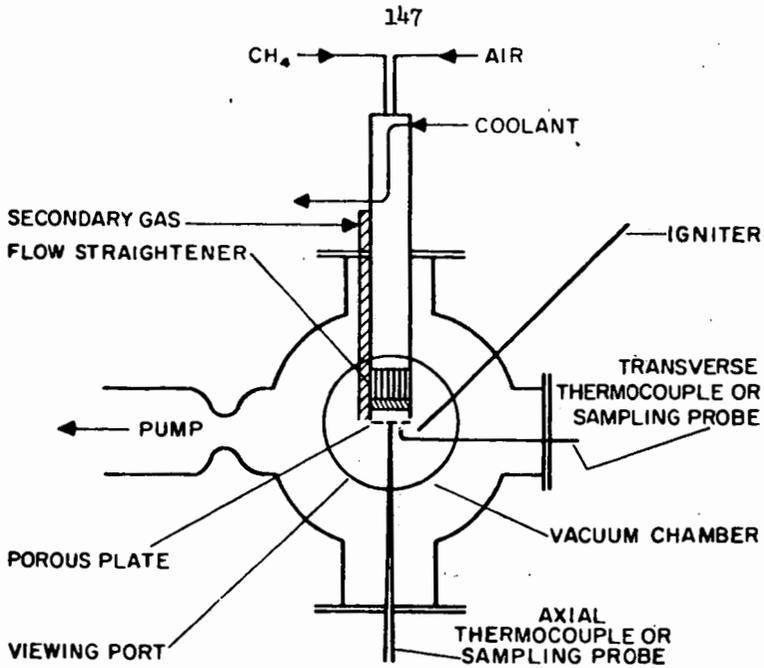


Fig. 1. - BURNER DESIGNED TO DETERMINE CHEMISTRY OF NITROGEN OXIDES PRODUCED IN METHANE-AIR FLAMES

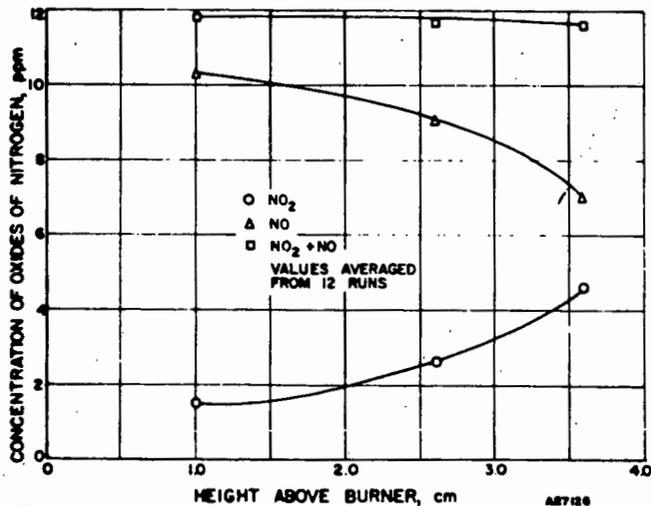


Figure 2. TOTAL CONCENTRATION OF OXIDES OF NITROGEN IN A METHANE-AIR STOICHIOMETRIC FLAME AT THE BURNER CENTER LINE

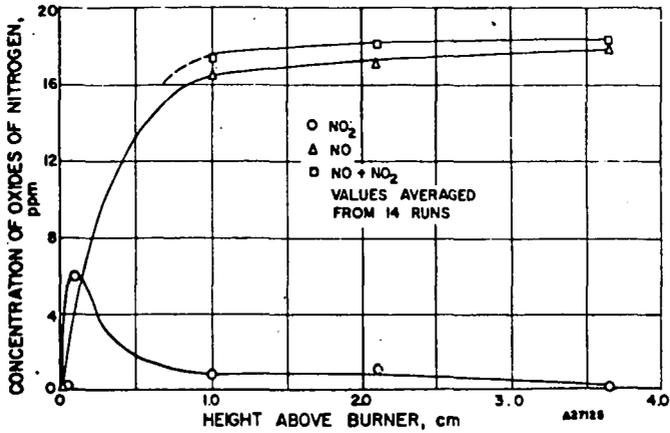


Figure 3. TOTAL CONCENTRATION OF OXIDES OF NITROGEN IN A FUEL-RICH (84% of Stoichiometric) FLAME AT BURNER CENTER LINE

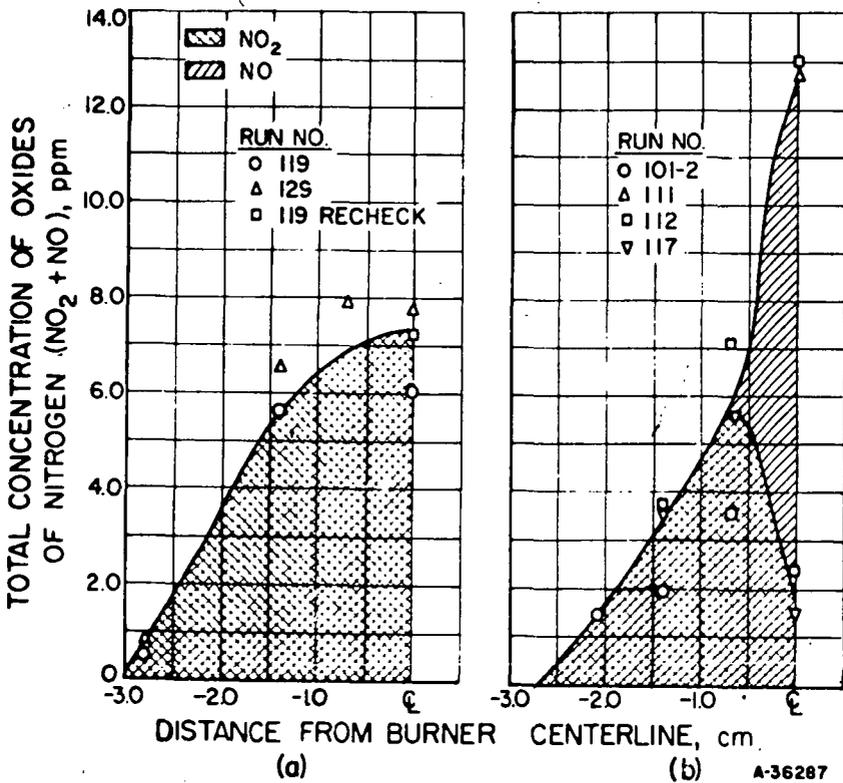
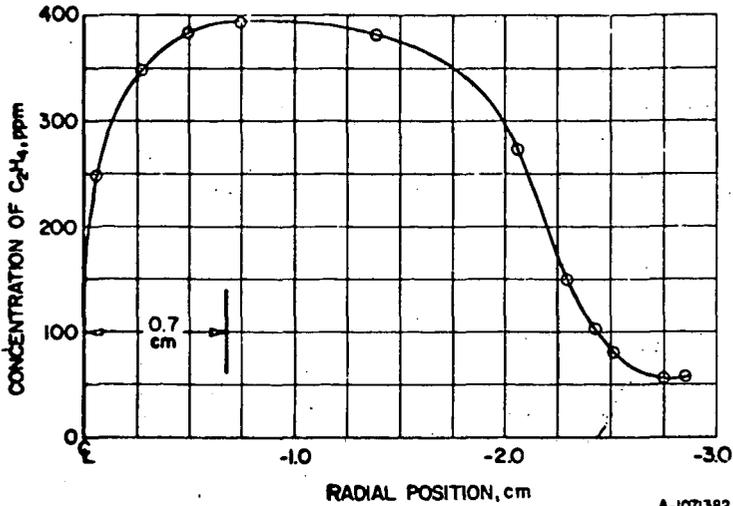
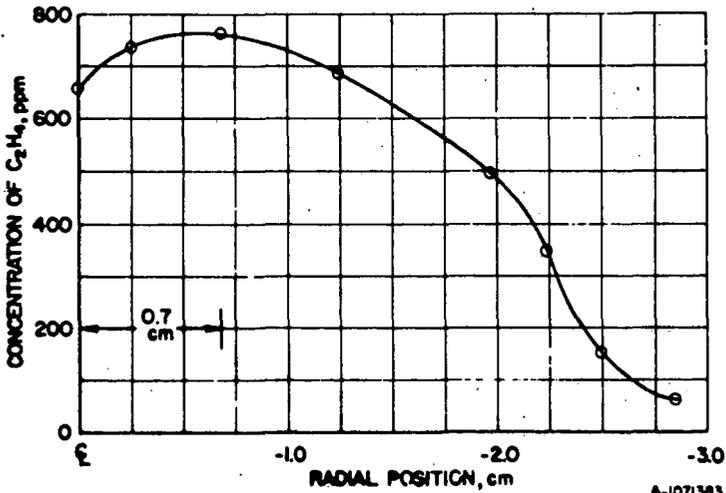


Figure 4. TOTAL CONCENTRATION OF NITROGEN FOR A METHANE-AIR STOICHIOMETRIC FLAME AT a) 0.1 cm AND b) 1.1 cm ABOVE BURNER



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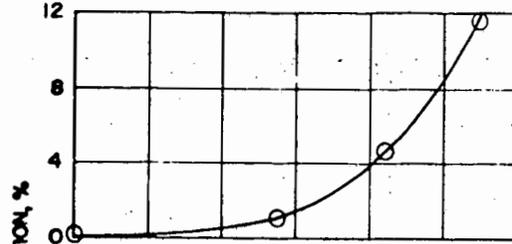
Figure 5. ETHYLENE CONCENTRATION AS A FUNCTION OF RADIAL POSITION AT 0.1 cm ABOVE THE BURNER FOR STOICHIOMETRIC  $CH_4$ -AIR FLAME



A-1071383

Figure 6. ETHYLENE CONCENTRATION AS A FUNCTION OF RADIAL POSITION AT 0.1 cm ABOVE THE BURNER FOR 84% OF STOICHIOMETRIC  $CH_4$ -AIR FLAME

(a)



(b)

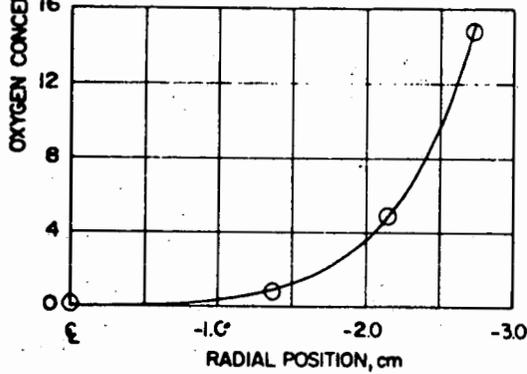
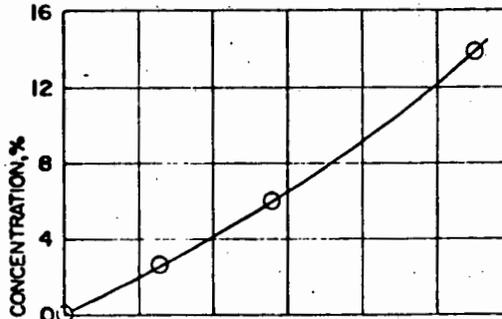


Figure 7. OXYGEN CONCENTRATION IN 84% OF STOICHIOMETRIC CH<sub>4</sub>-AIR FLAME (From Secondary Air Only) AT a) 1.1 cm AND b) 0.5 cm ABOVE BURNER

(a)



(b)

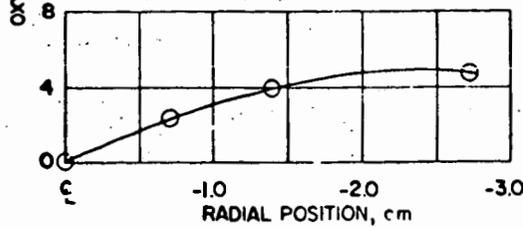


Figure 8. OXYGEN CONCENTRATION IN 100% OF STOICHIOMETRIC CH<sub>4</sub>-AIR FLAME (From Secondary Air Only) AT a) 1.1 cm AND b) 0.5 cm ABOVE BURNER

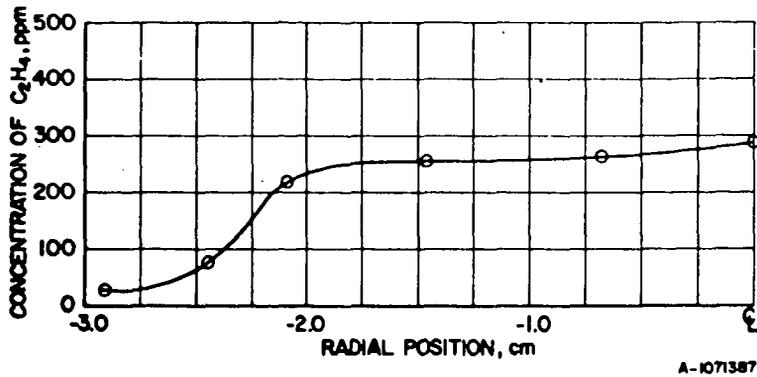


Figure 9. ETHYLENE CONCENTRATION AS A FUNCTION OF RADIAL POSITION AT 0.1 cm ABOVE BURNER FOR 120% OF STOICHIOMETRIC CH<sub>4</sub>-AIR FLAME

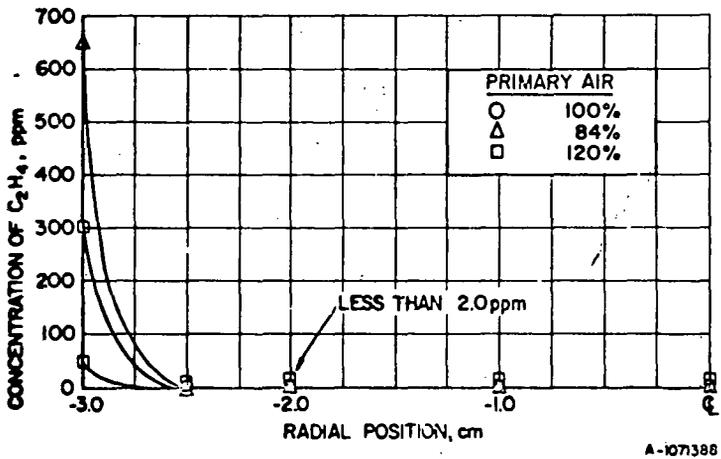


Figure 10. CONCENTRATION OF ETHYLENE AT CENTER LINE OF CH<sub>4</sub>-AIR FLAMES