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THE SUPPRESSION OF AFTERBURNING IN SOLID ROCKET PLUMES BY POTASSIUM SALTS

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Potassium added to the fuel-side of a H_2-CO-N_2-O_2 flat diffusion flame at near stoichiometry is more effective in inhibiting the flame reactions than KOH added to a H_2-N_2-O_2 flame at a stoichiometric ratio of 0.61. A ~~D~~escription is given of burner, optical and flow metering system used in experiments. Originator supplied keywords include:

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ABSTRACT

The exhaust plume of a minimum-smoke solid rocket contains significant concentrations of hydrogen and carbon monoxide which when mixed with ambient air react to water and carbon dioxide producing visible flash and increased infrared radiation. Both reactions produce undesirable signatures and interference with optical guidance systems. Potassium salts have been added to propellant charges to inhibit afterburning in both guns and rockets. They have not always been effective, the inhibiting effect of the salt being related to gas composition and temperature in a complex manner which is not completely understood. Further, there is disagreement as to whether it is KOH, KO_2, or K that is most important in the afterburning suppression. The results are presented here of the first year of the investigation sponsored by AFOSR on the efficacy of each of these on the combustion of diluted $H_2/CO-O_2-N_2$ mixtures. A flat diffusion flame produced by an opposed-jet burner, simulating the reaction conditions in the boundary layer of the plume, is being scanned incrementally in the infrared wavelengths to follow the inhibition reactions. It is planned also to use an alternative opposed-jet arrangement in which premixed combustible gases with additives are introduced in each opposed jet. Supporting equipment is in place, e.g. optical devices, spectrophotometer, flow metering system etc. A resistively heated burner permitting vaporization of potassium and potassium salts at temperatures up to 1000C has been built under the present grant, and has been placed in operation.

It has been found that potassium added to the fuel-side of a $H_2/CO/N_2/O_2$ flame at near stoichiometry is more effective in inhibiting the flame reactions than KOH added to the fuel-side of a $H_2/N_2/O_2$ flame at a stoichiometric ratio of 0.61 (lean). CO was not used in the latter experiments because of the formation of carbon in the heated burner at the higher temperatures (500-600C) required to vaporize a significant amount of the salt. It is likely that carbon also forms in the plume from a minimum smoke rocket contributing to the visibility of the flash.

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INTRODUCTION

The Services have increasingly emphasized the development and use of low signature tactical solid rocket motors in recent years. Visible primary and secondary smoke have been largely eliminated from rocket plumes by the removal of ammonium perchlorate oxidizer and most of the other energy and ballistic modifier additives from the propellant formulation, resulting in the formulation of the so-called "minimum smoke" (min-smoke) propellants. The exhaust gases from min-smoke propellants however contain significant concentrations of hydrogen and carbon monoxide which when mixed with ambient air in the plume react to water and carbon dioxide producing visible flash and increased infrared radiation. Also, some of the apparent secondary smoke advantage of min-smoke propellants over reduced smoke propellants (ammonium perchlorate oxidizer with low solids content) is lost since the hydrogen in the plume reacts to form additional water which is available for potential condensation to smoke. The research reported on below is directed toward preventing or at least inhibiting the signatures due to afterburning.

It is known that potassium salts inhibit the reactions of hydrogen and carbon monoxide to water and carbon dioxide respectively.[1,2] Potassium salts such as KNO_3 and K_2SO_4 have been added to propellant charges at a level of 1 - 3 wt pct to suppress gun muzzle flash [3] and rocket plume infrared signature [4]. The mechanism by which the potassium salts inhibit afterburning is controversial, but it probably involves K, KOH and possibly KO_2 reacting with H and OH radicals to break the chain reactions controlling the combustion of hydrogen and carbon monoxide.[5,6,7] Experimental evidence suggests that the reactions take place in the vapor phase. Since only small concentrations of the K, KOH and KO_2 are required, the amounts of salt that have been used in guns and rockets may have been excessive. Minimizing the amount used is important because the potassium salts increase radar signatures and the propensity for smoke formation. The effects of excess potassium salts on the latter have been demonstrated in recent studies reported by the US Army Missile Command.[8]

Under the present grant, the effects of K, KOH and KO_2 on the afterburning reactions are being evaluated by introducing them as a vapor into a flat diffusion flame of N_2-H_2-CO/O_2-N_2, scanning the flame incrementally and examining its infrared spectral emission. Initially methane was chosen as the fuel based on experimental convenience and because there was previous research reported in the literature on the effects of potassium and potassium salts on combustion of methane. Also, the formation of HCHO in the flame zone provided a convenient tag for following the progression of the reaction. However methane itself inhibits the H atom reactions being studied. Therefore it was decided to emphasize mixtures of N_2-H_2-CO as the fuel mixture since it is closer to the situation in the plume without introducing complicating side reactions. It is planned to define the conditions of gas composition and temperature for which the additives are effective.

An opposed-jet diffusion burner adapted from one described by Hahn, Wendt and Tyson [9] is being used. As discussed further below, the burner configuration permits study of the chemistry of inhibition of afterburning under conditions simulating those in the plume. In addition, the arrangement makes the injection of potassium and its salt vapors into the flame relatively convenient. A Beckman IR spectrophotometer has been modified for detection of emission spectra. The scanning technique is a modification of one previously developed by the principal investigator [10]. Eversole [11] is using a premixed $H_2/O_2/N_2$ premixed flat flame for his studies of afterburning suppression. This burner has advantages also of being well characterized, but the flame zone is relatively compressed compared with the opposed-jet burner requiring the use of reduced pressures to broaden the flame. Also, the range of composition and flow rates for flame stability is narrower and addition of additives is more difficult. Eversole is using LIF and Raman scattering for measuring concentration and temperature profiles in the flame. The techniques are much more sensitive but more restrictive than the emission spectrometer being used in the present research. The emission spectrometer more easily detects the range of flame intermediates than do the laser specroscopic techniques.

THEORETICAL BACKGROUND

The most important reactions involved in afterburning are probably,

$$H + O_2 = OH + O \qquad (1)$$

$$H + OH + M = H_2O + M* \qquad (2)$$

and

$$CO + OH = CO_2 + H \qquad (3)$$

There is disagreement about the correct mechanism for the potassium suppression of these reactions. Jensen, Jones and

Mace [6] concluded from their experiments with premixed H_2-O_2-N_2 flames that atomic potassium was involved, i.e.

$$K + OH + M = KOH + M^* \qquad (4)$$

and

$$KOH + H = H_2O + K \qquad (5)$$

On the other hand, Friedman and Levy [3] found atomic potassium and sodium to be ineffective for a methane-air diffusion flame and stated that reaction (4) was unlikely to occur early enough in the reaction to be effective. In addition to reaction (5) they proposed,

$$KOH + OH = H_2O + KO \qquad (6)$$

They suggested that potassium salts first form molten K_2O which reacts with water to form gaseous KOH. Evidence [2] that sodium oxides are not stable at flame temperatures and are not effective inhibitors lends credence to their argument. Kaskan [7] proposed an alternative mechanism,

$$K + O_2 + M = KO_2 + M^* \qquad (7)$$

and

$$KO_2 + OH = KOH + O_2 \qquad (8)$$

based on observations of hydroxyl radicals in a lean flame. Jensen[12] in a more recent paper has been able to explain Kaskan's results without resorting to the postulation of the reactions of KO_2.

All of these interpretations were speculative in nature since no direct measurements had been made. It is expected that scanning the inhibited flames spectroscopically, as is being done in the present research, will help resolve the mechanism question, and contribute to a definition of how best to formulate rocket propellants with potassium salts.

EXPERIMENTAL APPROACH

A schematic sketch of the opposed-jet burner and optical system being used for research sponsored by Morton Thiokol is shown in Figure 1. It is working well but it is limited to relatively volatile additives. A new burner has been designed and built under this grant which utilizes resistive heating to vaporize the K, KOH, and KO_2. A sketch of the new burner is shown in Figure 2. It permits the introduction of the vapors into the gas stream. The burner is 2-in diameter by 15-in long and made of Inconel 900 for corrosion resistance at high temperatures. It is concentric at the heating end with a mullite tube bonded to it with sauereisen cement. Nichrome wire is wound around the periphery of the mullite tube and heated electrically. The other end of the burner is cooled by water in a copper-tube heat exchanger silver-soldered to the Inconel tube. Gasss entering the cool end are conducted through a plenum chamber into the heated section which is packed with 1/8-in mullite balls held in place at both ends with Inconel screening. The balls serve two purposes - to provide a flat velocity profile needed to obtain the flat flame, and to hold the potassium or potassium salts in place while they are being vaporized and mixed with the fuel gases. The exterior of the burner is insulated with a Nomex blanket.

The flow metering system for introducing N_2, O_2, H_2 and CO has been completely installed and calibrated. The optical system used to convert the Beckman 4240 IR spectrometer from absorption to emission measurements comprises front surface 2-in plane mirrors and a 6-in spherical mirror of f/1.8. The mirrors are held in adjustable mounts and positioned by means of rod carriers on an optical bench. These permit positioning of the burner and optics easily and reproducibly. The slit is variable between 0.05 and 4 mm, is 12 mm high and is mounted on a translating base in the sample compartment of a Beckman 4240 IR spectrophotometer. Magnification of the image on the slit is 0.8.

In reference 10, the flame itself was moved in order to scan the flat flame incrementally. We have improved the technique, focusing the flame image on the spectrometer slit and translating the slit instead. The flame spectra of some of the possible kinetic intermediates, e.g. K_2O, KO and KO_2, are not known. If possible these will be identified but their effects can be deduced from following the H, OH, H2O, CO and CO_2 spectra.

EXPERIMENTAL RESULTS

A. N_2-H_2-CO/O_2-N_2/MeBr Flames:

In order to correlate the operation of the experimental equipment with the initial tests which were conducted with methane fuel, a series of tests was run with MeBr added to H_2/CO flames on both the air- and fuel-sides of the burner at a 3 vol % and 0.6 vol % respectively for a lean flame of ϕ = 0.86. For the case of the 3 vol% MeBr added to the air-side particularly the IR width of the flame was reduced, and there was an increase in the total IR radiation in the 3800 - 2000 cm^{-1} wavenumber region, especially in the 3800 - 2900 cm^{-1} water-OH bands and less so in the 2400 - 2000 cm^{-1} CO_2-CO bands. Similar effects were found when 0.6 vol% MeBr was added to the fuel-side of the burner. Less MeBr was used for this test because of the formation of carbon at higher levels. The results with and without the MeBr additive are tabulated in Tables I through IV.

B. N_2-H_2-CO-K/O_2-N_2 Flames:

In this series of tests (and in that in which KOH was added) it was first necessary to establish the intensity of radiation of the flame as a function of temperature of the fuel gases exitting the heated burner. Subsequently, the spectra were measured with potassium in the fuel. Gas composition for the tests was as follows:

	Flow,	L per min
Fuel- Side	H_2,	4.3
	CO,	2.0
	N_2,	3.7
	K,	0.22,1.3,1440 ppm @ 125,163,338C respectively
Air- Side	O_2,	3.2
	N_2,	14.7

$\phi = 0.98$

Slit width was 0.4 mm. Integrated areas for the width of the flame at a wavenumber of 3440 cm^{-1}, the maximum radiation frequency, are shown in Figure 3 for flames with and without potassium vapor added to the fuel. At this frequency, the addition of potassium led to a decrease in emission at all temperatures, the effect increasing with increasing temperature or concentration of potassium vapor in the fuel.

C. N_2-H_2-KOH/O_2-N_2 Flames:

As noted above, CO was deleted from the fuel because of carbon was formed by reduction with the hydrogen at the higher temperatures required to vaporize the KOH. The gas composition for these flames was:

		Flow, L per min
Fuel- Side	H_2,	5.5
	N_2,	3.9
	KOH,	0.5,9.4,147 ppm @ 415,500,600C respectively
Air- side	O_2,	4.5
	N_2,	15.2

Ø = 0.61

Slit width was 0.2 mm. Total spectral areas for the scan across the width of the flame for the wavenumber range 3800 - 3000 cm^{-1} are summarized in Table V. Normalized areas versus temperature are shown in Figure 4. Both the Table and the Figure illustrate the increase in spectral emission produced when KOH is added to the flame. Details of the spectral scan as a function of the position in the flame are shown in Figures 5 and 6 for the flame without and with KOH additive respectively. These latter Figures indicate an initial inhibition of the delay of the flame reactions on the fuel-side but toward the air-side of the flame the reactions are completed. A plot of the normalized areas with and without KOH additive is given in Figure 7.

DISCUSSION

A. N_2-H_2-CO/O_2-N_2/MeBr Flames:

It had been previously determined that methyl bromide inhibits the combustion of methane-air mixtures. It was observed that there was an increased formation of HCHO when methyl bromide was added to lean flames, confirming the reaction scheme proposed by Wilson[13] based on his own research and that from reference 14 that the increased rate of reaction of methyl bromide with H over that of methane and H led to an increase in the formation of HCHO in the flame. We also observed an overall increase in IR radiation with methyl bromide added to the flame consistent with Wilson's observation of the the main reaction taking place at a higher temperature than in uninhibited flames. Similar effects were noted in the present experimental data obtained with the H_2-CO flames. HCHO was also observed in these flames. According to Fenimore & Jones[14], the inhibition is involves the reaction:

$$H + CH_3Br = CH_3 + HBr \qquad (9)$$

At 1000K and 1300K the rate of reaction of equation (9) was found to be significantly faster than the rate of reaction for H + O_2 given by equation (1) given in the THEORETICAL BACKGROUND section. In addition,

$$CH_3 + O = HCHO + H \tag{10}$$

and

$$HBr + H = H_2 + Br \tag{11}$$

further break the chain reaction for afterburning. Wilson proposed also that the following reactions were involved,

$$CH_3Br = CH_3 + Br \tag{12}$$

$$H + Br + M = HBr + M* \tag{13}$$

and

$$CH_3Br + OH = CH_2Br + H_2O \tag{10}$$

He proposed that the flame inhibition mechanism was due to the inhibitor prolonging the preignition zone of the flame and shifting the primary reaction to a higher temperature. The present experimental results with H_2-CO and the previous tests with methane confirm the conclusions about the narrowing of the flame zone and the higher temperature in that zone.

B. H_2/CO-K and H_2-KOH Flames:

Although there were differences in how the scans were made for these two different flames, it would appear that K is a more effective afterburning inhibitor than KOH. This is not consistent with the qualitative interpretations of the references cited. In addition, the forward rate of reaction of reaction equation (5) for the KOH is orders of magnitude greater than equation (4) for K and OH. However, the K experiments were conducted with a H_2/CO fuel and the KOH with only H_2 as the fuel. The fuel mixture for the potassium flame was also closer to stoichiometry but this would be expected to reduce the effectiveness of the potassium. It is likely that the CO in the K flame plays an important role explaining the difference in effectiveness. The forward rate of reaction of CO and OH, equation (3), the principal reaction controlling the combustion of CO to CO_2, is about one order of magnitude slower than the reaction of KOH and H. However the concentration of CO in the flame is several orders of magnitude higher than the concentration of KOH, generating H atoms and heat. The potassium reaction, by removing OH and inhibiting the CO reaction, effectively inhibits the H_2 oxidation as well. The effectiveness of these inhibitors is likely to be significantly altered as a function of stoichiometric ratio in the flame. We intend to investigate this factor further. Also, CO will be introduced into the KOH flames from the air side of the burner to confirm this mechanism.

FUTURE PLANS

(1) Complete the opposed-jet flame scans using N2-diluted H2/CO fuel and diluted air with K, KOH and KO2 vapor additives over a range of ϕ from rich to lean.

(2) Literature surveys and interpretation of the experimental results are a continuing process.

(3) Make comparison runs, again with opposed-jets but utilizing premixed gases of different ϕ's consistent with avoiding ignition of the premixed gases while vaporizing the additives in the burner tube.

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TABLE I

IR SPECTRAL EMISSION OF OPPOSED-JET H_2/CO DIFFUSION FLAMES

Ø = 0.86, 1:1 H_2/CO DILUTED WITH N_2 ON FUEL-SIDE, AIR DILUTED WITH N_2 & METHYL BROMIDE (3.0% WHEN ADDED) ON AIR-SIDE.
SLIT = 0.25 MM, 3800-2900 CM-1

	Slit Position, inch	Spectral Area sq.in.		
		w/o MeBr	w/ MeBr	% Increase
Fuel-Side	0.18	0.24	0.09	-62.5
		0.17	0.28	64.7
		0.35	0.31	-11.4
		0.58	0.72	24.1
		1.19	1.37	15.1
	0.23	1.70	2.04	20.0
		2.70	3.00	11.1
		3.77	4.37	15.9
		5.13	5.95	16.0
		6.75	7.32	8.4
	0.28	8.00	9.19	14.7
		9.36	10.97	17.2
		10.91	11.45	4.9
		10.87	12.48	14.8
		11.42	12.47	9.2
	0.33	11.39	11.54	1.3
		10.30	10.52	2.1
		9.07	8.82	- 2.8
		6.84	6.77	- 1.0
		5.23	4.86	- 7.1
	0.38	2.41	2.52	4.6
Air-Side		0.85	0.74	-12.9
	0.40	0.06	0.00	-100.0
	TOTAL	119.29	127.78	7.1

TABLE II

IR SPECTRAL EMISSION OF OPPOSED-JET H_2/CO DIFFUSION FLAMES
$\emptyset$ = 0.86, 1:1 H_2/CO DILUTED WITH N_2 ON FUEL-SIDE, AIR
DILUTED WITH N_2 & METHYL BROMIDE (3.0% WHEN ADDED) ON AIR-SIDE.
SLIT = 0.25 MM, 2400-2000 CM-1

	Slit Position, inch		Spectral Area sq.in.	
		w/o MeBr	w/ MeBr	% Increase
Fuel-	0.17	0.65	0.67	3.1
Side		0.89	0.93	4.5
		1.30	1.27	- 2.3
		1.61	1.71	6.2
		1.97	2.14	8.6
		2.45	2.63	7.3
	0.23	2.96	3.24	9.5
		3.48	3.78	8.6
		4.32	4.46	3.2
		4.93	5.09	3.2
		5.15	5.54	7.6
	0.28	5.69	6.24	9.7
		6.20	6.40	3.2
		6.24	6.49	4.0
		6.15	6.66	8.3
		6.20	6.25	0.8
	0.33	6.10	6.14	0.7
		5.44	5.42	- 0.2
		4.82	5.07	5.2
		4.05	4.19	3.5
		3.02	3.02	0.0
	0.38	1.80	1.96	8.9
Air-		0.77	0.85	10.4
Side	0.40	0.09	0.05	- 44.4
	TOTAL	87.08	90.20	3.6

TABLE III

IR SPECTRAL EMISSION OF OPPOSED-JET H_2/CO DIFFUSION FLAMES Ø = 0.86, 1:1 H_2/CO DILUTED WITH N_2 ON & METHYL BROMIDE (0.6% WHEN ADDED) ON FUEL-SIDE, AIR DILUTED WITH N_2 ON AIR-SIDE.

SLIT = 0.25 MM, 3800-2900 CM-1

	Slit Position, inch	Spectral Area sq.in.		
		w/o MeBr	w/ MeBr	% Increase
Fuel-Side	0.18	0.00	0.00	0.0
		0.03	0.10	233.3
		0.14	0.15	7.1
		0.31	0.20	-35.5
		0.57	0.68	19.3
	0.23	1.47	1.38	- 6.1
		2.45	2.26	- 7.8
		3.27	3.12	- 4.6
		4.59	4.45	- 3.1
		5.60	5.92	5.7
	0.28	7.27	7.37	1.4
		8.42	8.58	1.9
		9.39	9.40	0.1
		9.65	9.70	0.5
		9.54	9.49	- 0.5
	0.33	8.64	8.78	1.6
		7.61	7.65	0.5
		6.19	6.14	- 0.8
		4.63	4.45	- 3.9
		2.81	2.66	- 5.3
	0.38	1.25	1.06	-15.2
Air-Side		0.07	0.08	-12.5
	0.40	0.00	0.00	0.0
	TOTAL	93.90	93.62	- 0.3

TABLE IV

IR SPECTRAL EMISSION OF OPPOSED-JET H_2/CO DIFFUSION FLAMES

Ø = 0.86, 1:1 H_2/CO DILUTED WITH N_2 ON & METHYL BROMIDE (0.6% WHEN ADDED) ON FUEL-SIDE, AIR DILUTED WITH N_2 ON AIR-SIDE.

SLIT = 0.25 MM, 2400-2000 CM-1

	Slit Position, inch	Spectral Area sq.in.		
		w/o MeBr	w/ MeBr	% Increase
	0.17	0.50	0.55	10.0
Fuel-Side		0.65	0.74	13.8
		1.01	0.89	-11.9
		1.23	1.21	- 1.6
		1.55	1.66	7.1
		1.87	1.93	3.2
	0.23	2.49	2.54	2.0
		2.88	3.08	6.9
		3.50	3.63	3.7
		3.89	4.06	4.4
		4.43	4.60	3.8
	0.28	4.80	5.00	4.2
		5.12	5.15	0.6
		5.35	5.38	0.6
		5.25	5.37	2.9
		4.98	5.33	7.0
	0.33	4.67	4.78	2.4
		4.06	4.34	6.9
		3.47	3.46	- 0.3
		2.70	2.94	8.9
		1.79	1.91	6.7
	0.38	1.12	1.10	- 1.8
Air-Side		0.38	0.40	5.3
	0.40	0.00	0.00	0.0
	TOTAL	67.69	70.05	3.5

TABLE V
IR SPECTRAL EMISSION OF OPPOSED-JET N_2-H_2/O_2-N_2 DIFFUSION FLAMES

Ø = 0.61, SLIT = 0.25 MM, 3800-3000 CM-1

Temperature, deg C	Spectral Area, Sq in	
	wo/KOH	w/KOH
400	8.71	
415		10.11
420	9.05	
425	9.19	
500	9.79	10.47
600	10.18	10.78

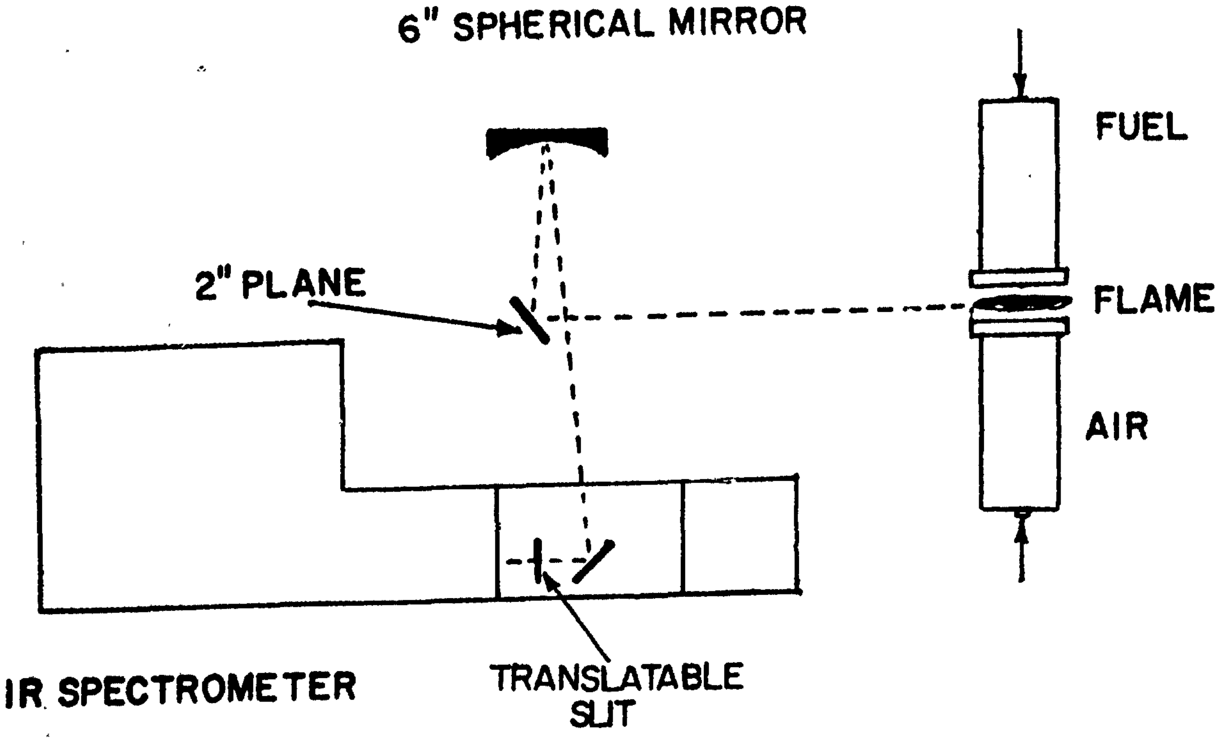

6" SPHERICAL MIRROR
FUEL
2" PLANE
FLAME
AIR
IR SPECTROMETER
TRANSLATABLE SLIT

FIGURE 1

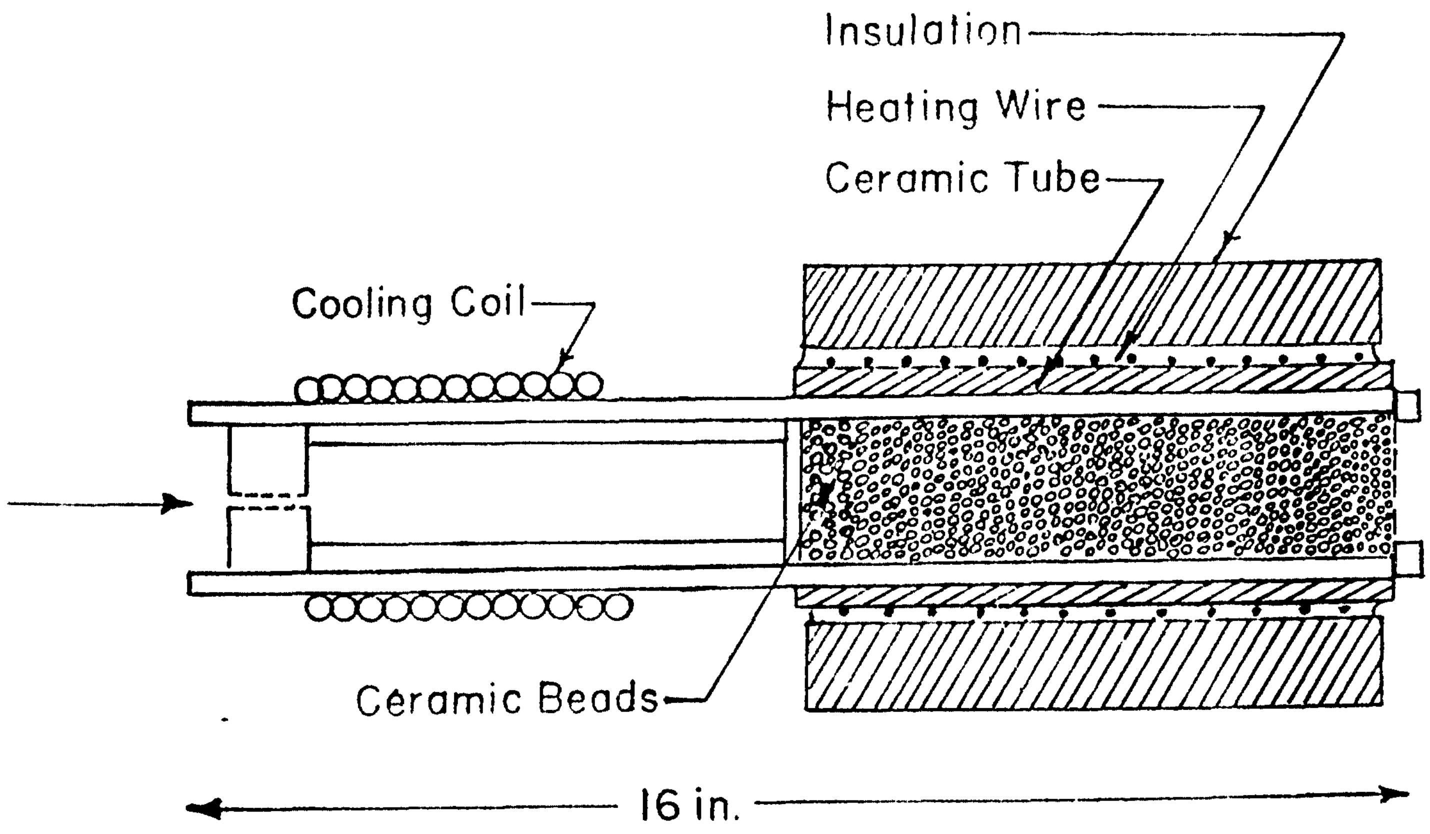

Insulation
Heating Wire
Ceramic Tube
Cooling Coil
Ceramic Beads
16 in.

FIGURE 2

INTENSITY vs BURNER TEMPERATURE

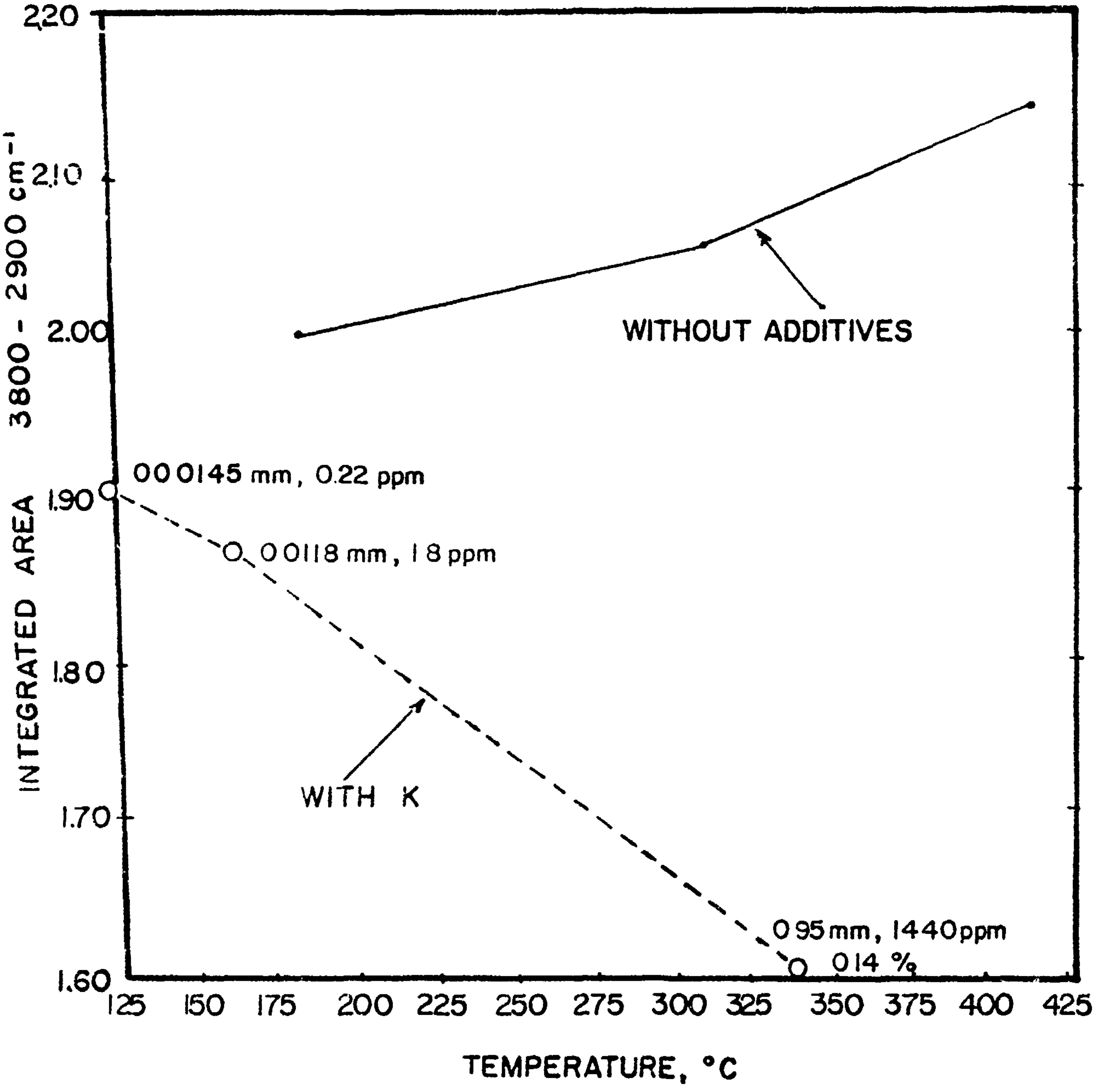


FIGURE 3

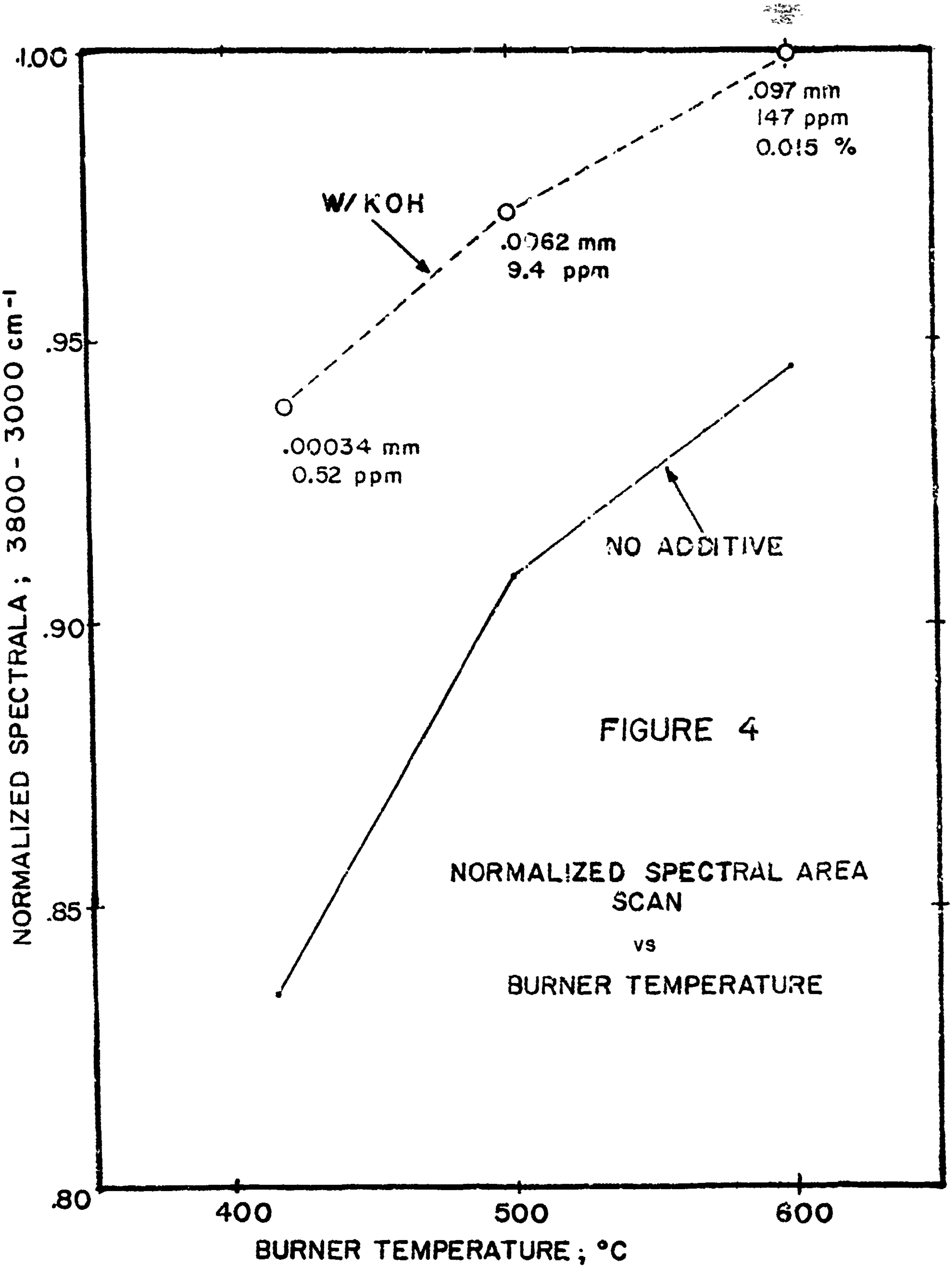
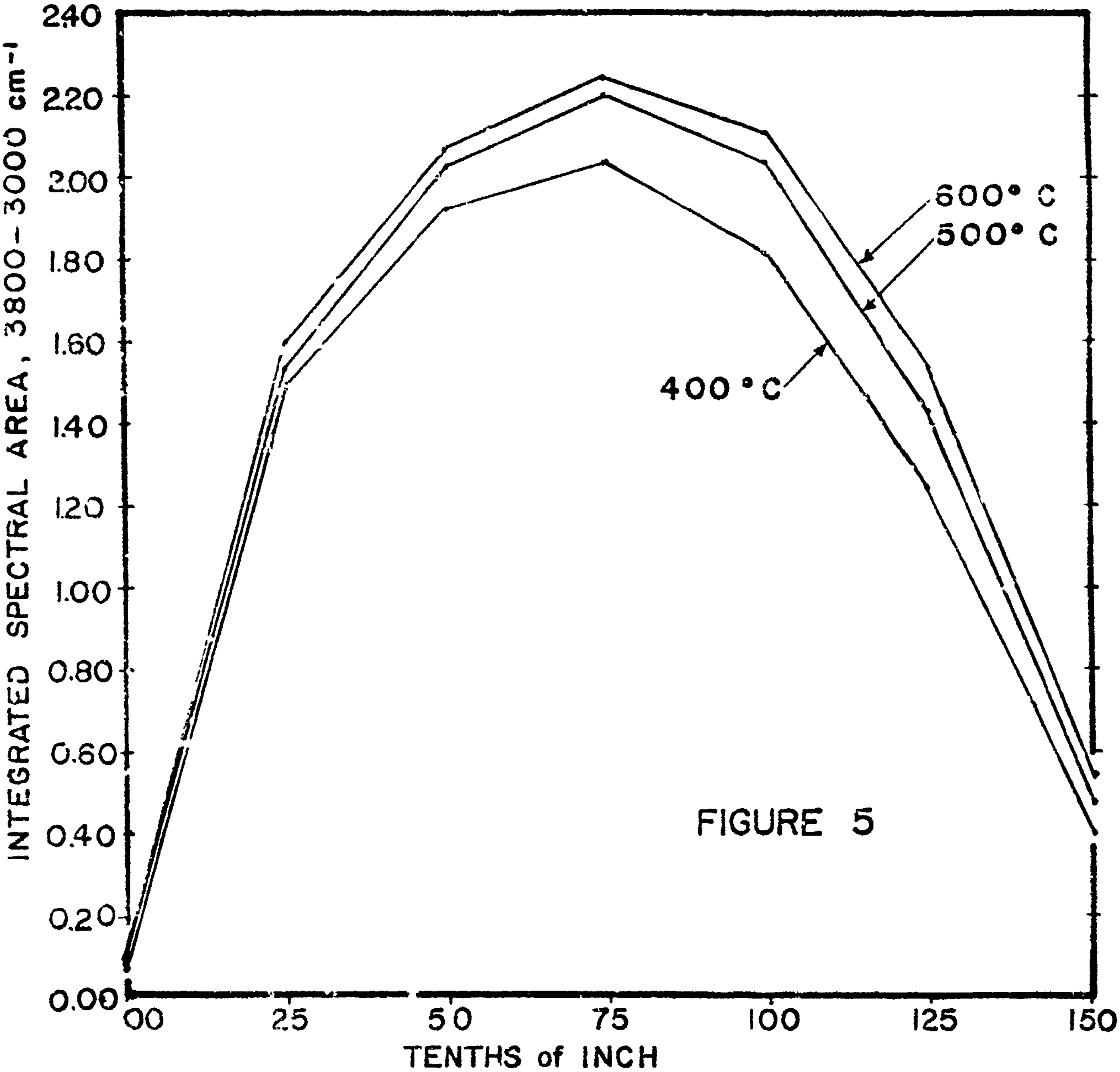


FIGURE 4

NORMALIZED SPECTRAL AREA SCAN
vs
BURNER TEMPERATURE



SPECTRAL AREA SCAN vs BURNER TEMPERATURE
NO ADDITIVE

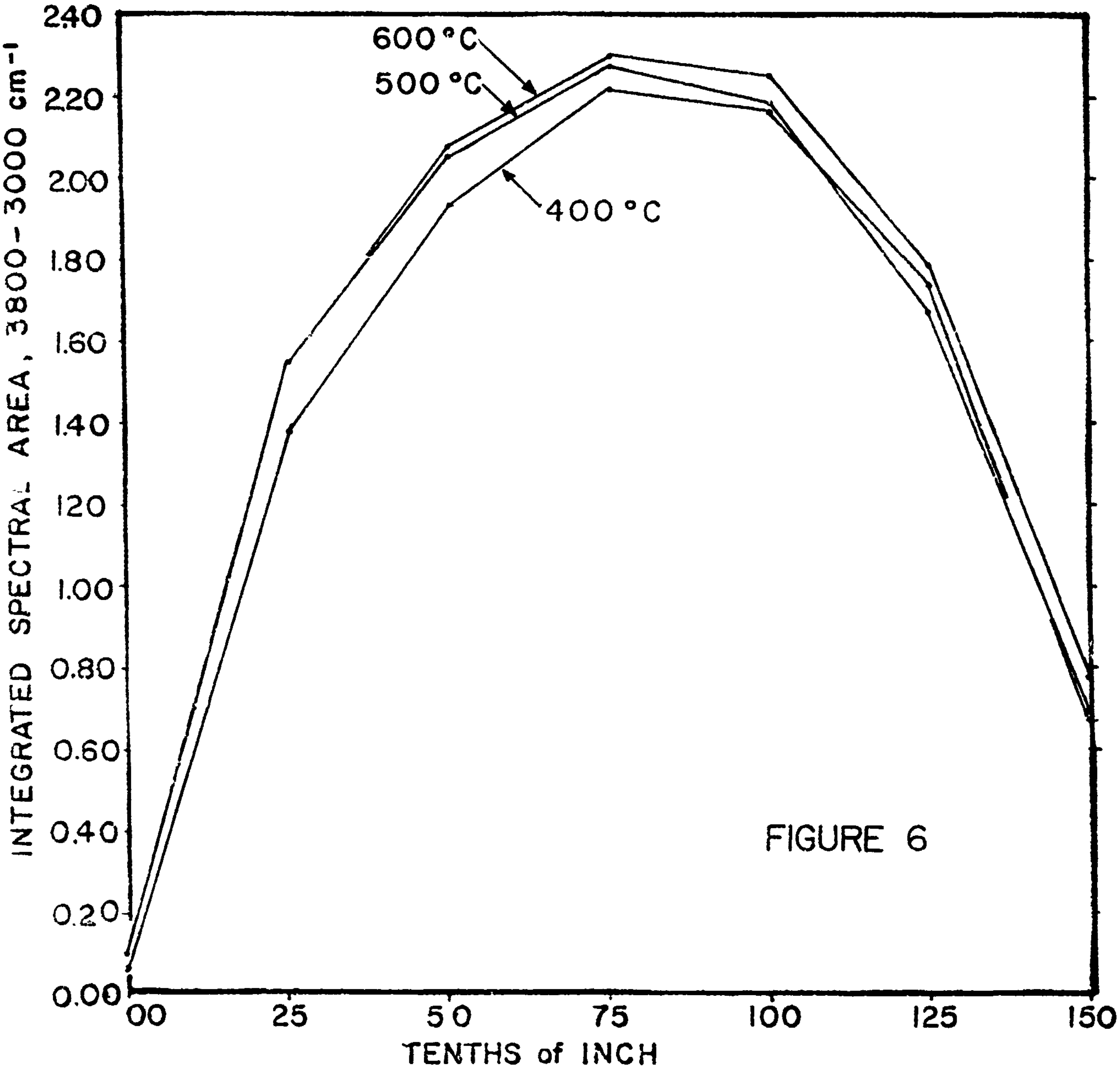


FIGURE 6

SPECTRAL AREA SCAN vs BURNER TEMPERATURE

WITH KOH ADDED

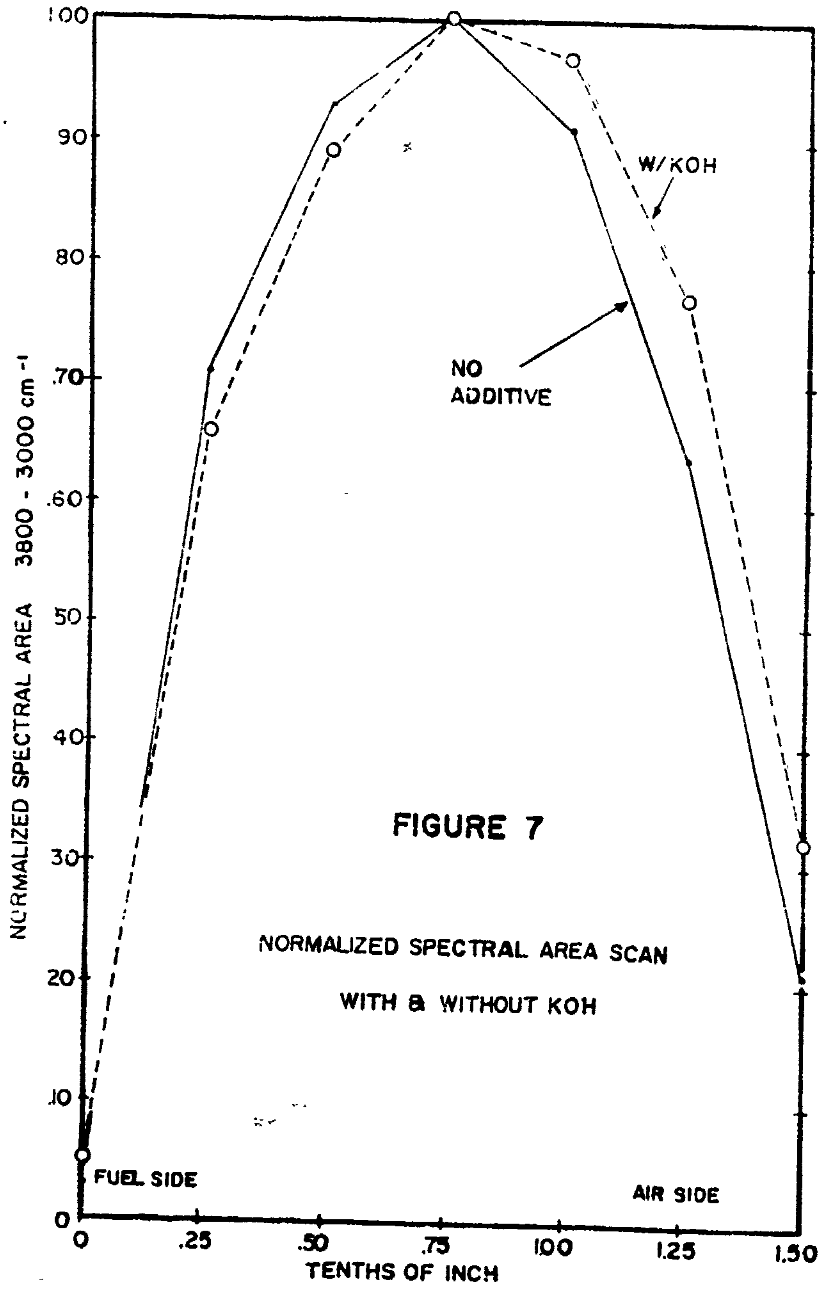


FIGURE 7

NORMALIZED SPECTRAL AREA SCAN

WITH & WITHOUT KOH