AN INVESTIGATION OF BUTANE INJECTION INTO A REVERSE COMBUSTION PROCESS

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PREFACE

In order to find the best and most efficient method of recovering oil by in situ combustion, various techniques and methods of investigation must be employed. It was the purpose of this study to determine the effect of mixing butane with the injected air on a reverse in situ combustion process. The economics of such a process was not considered here, but it is realized that any process of recovering oil must meet certain economic requirements.

I wish to express my appreciation to Dr. J. H. Boggs and the faculty of the School of Mechanical Engineering for making my graduate study here at Oklahoma State University possible. I am especially grateful for the honor of receiving the Phillips Petroleum Company research assistantship and the Phillips Petroleum Company Graduate Scholarship. It is very difficult to express my appreciation to my adviser, Dr. David Cornell, for his help and guidance throughout the course of this study. His helpful advice and criticism have been an encouragement for which I am sincerely grateful. I wish to extend thanks to Professor B. S. Davenport, Mr. John McCandless, and Mr. George Cooper for their help and technical advice. I am indebted to Mrs. Mildred Avery for her excellent job in the typing of this thesis. To my wife, Sally, for her help and encouragement throughout my college career there cannot be said enough to show my thanks and appreciation.

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LIST OF SYMBOLS

Symbol										
U	olumetric velocity, standard cubic feet per hour per square foot									
Т	emperature, degrees Fahrenheit									
Ρ	pressure, psia									
X	distance from peak temperature, feet									
Subscrip	ts									
a	air									
g	gas, commercial butane									

ŗ.

p peak, T_p

CHAPTER I

INTRODUCTION

Dating back almost to the first commercial oil well, the petroleum industry has been trying to recover a maximum percentage of oil from underground formations. The accepted and well known methods of recovery still leave a high percentage of the oil remaining in such reservoirs. In recent years a process known as in situ combustion has been under investigation by many of the petroleum research laboratories.

There are various methods in applying the in situ combustion process to actual oil fields. One method is to initiate combustion at an injection well and cause the combustion zone to advance toward production wells. This method is known as forward combustion. Another method is to intentionally interrupt the advance of a forward combustion zone at a prescribed distance from the injection well thereby allowing radial transfer of heat stored in the burned sand sections in the direction of the producing wells. Still another method is to establish a combustion front at a production well and allow the front to advance toward the air injection well. The latter method is known as reverse combustion. In forward combustion the heavy components of the oil in place are utilized as fuel, while in reverse combustion a portion of the lighter components is burned.

Forward combustion has been extensively reported (5), (6), (7), (8), and several field tests have been conducted. Forward combustion is applicable to reservoirs containing 15 to 40 degrees API gravity crude oil. The lower limit is controlled by the maximum viscosity which will allow practical producing rates. The upper limit is controlled by the amount of coke deposited ahead of the combustion zone.

Some advantages that can be attributed to reverse combustion are: The recovered oil flows into the heated portion of the reservoir with a greatly reduced viscosity. This allows higher producing rates than during forward combustion where the produced oil flows into the colder regions of the reservoir.

All oil passes through the combustion zone where cracking and coke deposition take place to cause a significant upgrading of the oil. This is not true during forward combustion where the oil is swept ahead of the combustion zone to form an oil bank which never contacts a high temperature region and therefore is not upgraded.

Some disadvantages of reverse combustion are: In reverse combustion appreciable amounts of hydrocarbons are left behind as coke deposit; also the fuel consumed includes desirable hydrocarbons which would be recovered during forward combustion. In forward combustion the fuel consumed is the coke, and all other hydrocarbons are recoverable.

The heat required to vaporize the fluids during reverse combustion is lost since the fluids and the combustion zone move in opposite directions. In forward combustion the heat generated is partially recovered as it is used in heating up the injection air and the colder

regions of the reservoir beyond the combustion zone.

It was the purpose of this investigation to determine the effect of injecting butane into the air stream of a reverse in situ combustion process. The factors observed were temperature distribution, combustion front velocity, and inlet and flue gas composition. The system consisted of a 2-inch i. d. cylindrical vertical tube packed with an unconsolidated sand using a 23 degree API gravity crude as the in-place fluid.

CHAPTER II

PREVIOUS INVESTIGATIONS

Most of the literature on in situ combustion that is available today pertains to forward combustion. With the increasing interest in recovering low-gravity, high-viscosity crudes, most production research laboratories are investigating the possibilities of reverse combustion. The first patent was granted in 1923 on a process in which air and a combustible gas were pumped into an injection well and ignited. (1). However, it was not until 1947 that in situ combustion was tested satisfactorily by Sinclair Oil Company and Magnolia Petroleum Company, each working independently of the other. Since these first tests several papers written on thermal recovery have been published, although the number of papers found in the literature pertaining to laboratory results of reverse combustion were few.

The first publication on laboratory results of reverse in situ combustion was released by the Gulf Research and Development Company. (2). Reed, et al, did laboratory experiments on the reverse combustion of tar sands in a linear adiabatic system. They showed that a highly upgraded oil can be produced from an exceedingly viscous, immobile oil. They concluded that the average peak temperature attained during successful reverse combustion of most tar sands is a function of the air flux alone and is insensitive to heat loss from the system. They

also concluded that the average combustion zone velocity is approximately a linear function of the air flux. They found that oil recovery from reverse combustion passes through a maximum of about 50 per cent near an air flux of 40(scf/hr-ft²), corresponding to a peak temperature of 780 degrees Fahrenheit. Below this value, recovery drops rapidly, and above this value, recovery drops slowly. By increasing the air flux (and, hence, the peak temperature) the residual coke and oil decreased and fuel requirements increased. They stated that increasing the initial temperature decreases the peak temperature and increases the combustion zone velocity. Also, when air is the oxidizing agent, all oxygen supplied is always consumed in the process.

One of the first studies on the theoretical aspects of reverse combustion was carried out by Berry and Parrish (3). They state that the maximum temperature and velocity of movement are the two dependent variables defining the progress of the combustion zone. A differential equation describing the temperature distribution during a reverse combustion operation was derived by them assuming linear flow in a homogeneous system and accounting for conduction, convection, heat generation, and heat loss. Also, an oxygen-concentration equation was derived by an oxygen-mass balance on the element, accounting for convection and reaction. They conclude that the maximum temperature reached and the combustion zone velocity both increase with an increase in air-injection rate. Also, it was found that heat loss to surroundings reduces combustion zone velocity, but has little effect on maximum

combustion zone temperatures. They predicted that an increase in the oxygen-hydrocarbon reaction rate results in a reduction in the maximum temperature reached.

Another theoretical study of reverse combustion in tar sands was done by Warren, et al, (4). They studied the behavior of the reverse combustion process in a linear adiabatic system by means of an idealized physical model. A pair of non-linear equations involving heat and mass transfer which are coupled by a concentration-dependent reaction-rate function of the Arrhenius type describes the model. They show that the process can be mechanistically described by simple physical models whose behavior equations can be solved formally, and the derived equations for reverse combustion can be solved numerically within the error limits for the experimental data. They concluded that a practical evaluation of reverse combustion requires that the recovery efficiency, the rate of oil production, and the (injected air/produced oil) ratio be known.

Publications on forward combustion became prominent in the petroleum literature in 1953. Kuhn and Koch (5) described results of field and laboratory test with forward in situ combustion. They reported that under unfavorable high-heat-loss conditions crude oil could be ignited in the presence of sand and water and that it would continue to burn in the form of a slowly advancing high-temperature front of finite thickness as long as the necessary air flux was maintained to support combustion. They stated that less than 15 per cent by weight of the oil in place (after initial air drive and before

combustion) was consumed as fuel, and that substantial increases in recovery of the oil over that which could be obtained by hot-air drive alone were effected by the in situ combustion process. Also, they reported theoretical studies involving heat-transfer behavior during ignition of oil sand, temperature distributions in three-dimensional system, and fluid flow in a two-dimensional system.

Szasz and Grant (6) reported results of a field test of forward combustion using air and fuel gas as the injection gas. They state that propagation-rates are dependent on the oxygen-fuel ratio of the injected gases. It was observed by them that if the oxygen content was increased beyond 6 per cent, forward travel of the combustion front decreased. Also, if the oxygen content of the injected gas reached 12 per cent, the burning "flashed back" to the injection well bore.

Martin, et al, (7) described the results of a laboratory investigation conducted to evaluate the forward-combustion process as a method of producing crude oil. The presented results showing air and fuel requirements, rates of advance, combustion temperatures, and coke and fluid distributions. Minimum air flux for self-sustained combustion was found to be $10(scf/hr-ft^2)$ for crude oils ranging in gravity from 10.9 to 32.2 degrees API in a semi-adiabatic, unconsolidated sand pack. They concluded that air injection must be supplied at a rate required to maintain a 600 degree Fahrenheit temperature level in order for stabilized, self-sustained in situ combustion to take place. They further stated that the rate of advance of a combustion zone is nearly proportional to the rate at which air is supplied to the zone and

inversely proportional to the amount of residual material deposited. For conditions where high combustion efficiencies exist and the kinetics of combustion are not controlling, they indicated that the effect of pressure on in situ combustion is small.

Moss, et al, (8) performed field tests to evaluate problems associated with initiation of combustion in a natural, oil-sand reservoir and to establish a relationship between field observations and information derived from laboratory experimentation and computations. They reported the confirmation of laboratory-derived data for a particular reservoir by actual field experimentation. Cooperman (9) presented a steady state theory relating hydrodynamic, chemical, and thermal aspects with each other for forward in situ combustion of oil in an underground reservoir. He showed for successful combustion the chemical criterion gives a minimum carbon-hydrogen mole ratio of 3.05.

Benham and Poettmann (10) compared results obtained from theoretical considerations of forward in situ combustion to results obtained by Martin, et al, (7) from experimentations. They derived an equation relating the point velocity of the combustion front as a function of the air flux, fuel content, efficiency of oxygen utilization, hydrogencarbon ratio, and the ratio of carbon dioxide to carbon monoxide produced. They concluded that this equation is useful in analyzing experimental data for apparent inconsistencies.

CHAPTER III

THE TEST APPARATUS

Some of the equipment used in a previous investigation of the combustion of butane-air mixtures in a porous media by Woods (11) was utilized in making this study.

The control board as shown in Plate I consisted of water manometers for measuring differential pressures across the orifice meters, two Bourdon tube pressure gauges which provided upstream pressure readings, control valves, and five 0-140 volt variable transformers which controlled the wall heaters of the combustion tube.

The temperature recorder shown in Plate I was a Weston Model 6702. It was a multipoint potentiometric type with a three second print cycle. The recorder was used to record temperatures from 17 type J iron-constantan thermocouples on the combustion tube. The thermocouples were made from 24 gauge iron-constantan thermocouple wire enclosed in woven glass insulation by fusing together the ends of the wires.

The combustion tube assembly is shown in Plate II and in detail in Figures 1, 2, and 3. The combustion tube was made of SAE 321 stainless steel tubing 27 inches in length with a 2.0-inch outside diameter and a 0.032-inch wall thickness. A $\frac{1}{4}$ -inch stainless steel plate with flow connections was attached to the bottom of the tube by

PIATE I PANEL BOARD AND TEMPERATURE RECORDER







PLATE II TUBE ASSEMBLY





Figure 2. Ignitor

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brazing. The top of the tube was equipped with a flange in order that the ignitor-cap with flow connections could be bolted in place. Thermocouples were placed on the outside surface of the tube at intervals of 1 inch starting at a point $2\frac{1}{2}$ inches below the top of the tube. The 16 wall thermocouples were fixed to the wall of the tube by Sauereisen No. 6 cement.

The four electrical resistance heaters used to control the wall temperature of the combustion tube had a power output of 600 watts each. The heaters were made from 18 gauge Nichrome Alloy V electrical resistance wire rated at 0.40 ohms per foot by coiling it around a 1/8-inch rod at 8 turns per inch. The heaters were coiled on a $2\frac{1}{2}$ -inch i. d. Transite pipe and coated with a thin layer of Sauereisen No. 6 cement to hold them in place. Each of the heaters covered an axial length of 6 inches and was controlled by separate variable voltage transformers. Diatomaceous earth was packed into the annulus between the combustion tube and the Transite pipe to aid in keeping a uniform temperature distribution along the tube wall. In some of the tests the entire tube assembly as shown in Plate II was placed in an 8-inch pipe with diatomaceous earth packed in the annulus between the heaters and pipe wall. The latter was done to minimize the heat loss.

The first tests were made using a Chromalox 240 volt, 200 watt electric heating element. Due to the corrosive power of the flue gases, the electric heater burned out after completing only two tests. A gas fired heater was then designed as shown in Figure 2. The combustion chamber of the latter was located beneath a baffle inside a

1 3/8-inch stainless steel tube so that the flames from the burner would be projected down on to a stainless steel heating plate. When the ignitor had been bolted on to the combustion tube the heating plate was in contact with the oil sand face. The heater was fired by an airpropane mixture entering through a 3/16-inch tube to the burner. Two valves controlled the gas mixture, thereby controlling the heat output of the ignitor.

The flue gas analysis was performed by the use of an orsat type apparatus and chromatography unit shown in Plate III. The orsat was a Burrell Gas Analysis Apparatus no. 39-540. It was used in determining carbon dioxide, oxygen, and carbon monoxide composition of the flue gas. The chromatography unit was a Cenco no. 70130 Vapor Phase Analyzer equipped with a Sargent Recorder Model SR and was used to determine the n-butane, iso-butane, propane, carbon dioxide, and nitrogen content of the injected and flue gases. The column used with the analyzer was 10 feet long and packed with di-2-ethylhexyl phthalate (30 weight per cent) on 20-60 mesh firebrick.

The flow diagram is shown in Figure 4. The air and gas was supplied to the combustion tube through 0.374-inch i. d. stainless steel tubing. The air and gas on their way to the combustion tube passed through Hoke control valves which controlled the flow rate through orifice meters made from Catawissa orifice unions. The flow lines were arranged so that air and gas injection could be accomplished through the top or bottom of the combustion tube. The flue gas after leaving the combustion tube was allowed to flow through a copper cooling coil submerged in ice water, then through a liquid trap to the flue gas analysis equipment.

PIATE III ORSAT AND CHROMATOGRAPHY UNITS







CHAPTER IV

PROCEDURE

The preparation and execution of each test were varied to meet different conditions. The saturation, initial temperature profile on the tube surface, type of sand, and the position (vertical or horizontal) of the combustion tube were changed in some tests. Although these conditions varied slightly, for most runs the procedure was carried out in the following manner.

The thermocouples were fixed on the 2-inch tube by Sauereisen No. 6 cement. After allowing the cement to set for twenty-four hours, the tube was placed inside a $2\frac{1}{2}$ -inch i. d. Transite pipe, and the annulus was packed with diatomaceous earth. The thermocouples were then connected to the temperature recorder.

A predetermined amount of sand saturated with a 23 degree API crude oil was packed inside the combustion tube. The sand was packed inside of the tube by filling it with 2 inches of sand then tamping the sand into place. This procedure was repeated until the sand was within $2\frac{1}{4}$ inches from the top of the tube.

With a center-insulated copper gasket placed between the tube flange and the ignitor cap, the ignitor cap was bolted in place. The Nichrome Alloy V heaters which were coiled on the Transite pipe and cemented in place with Sauereisen No. 6 cement were connected to the

variable transformers. The transformers were turned to a predetermined position to allow the skin temperature of the combustion tube to reach 200 degrees Fahrenheit. The inlet and outlet gas lines were then connected to the combustion tube.

When the skin temperature reached 200 degrees Fahrenheit as indicated on the temperature recorder, the ignitor was lighted. The sand face temperature was observed until it reached approximately 900 degrees Fahrenheit. At this time a differential pressure which was determined from flow rate curves was set across the orifice meters. Throughout the test the flow rate was held constant by regulating the differential pressure across the orifices.

After the air was allowed to flow in the forward direction for five minutes, a flue gas sample was taken. If the flue gas analysis indicated combustion was taking place by a high percentage CO₂ composition, the valves were switched to reverse air injection.

The ignitor remained lighted until the flue gas analysis and temperature recorder indicated reverse combustion was taking place. Flue gas samples were taken throughout the test and analyzed on both the orsat and chromatography units. The slow process of the orsat limited gas samples to one every fifteen to twenty minutes.

After the ignitor had been turned off and a definite maximum combustion zone temperature established, butane was injected into the inlet air stream. By taking a sample of the inlet gas, a definite composition of the inlet gas was determined.

After the combustion zone reached the bottom of the tube or

combustion ceased, the system was disassembled. A $\frac{1}{4}$ -inch drill welded on an extension rod was used to remove the sand from the tube. The sand was visually inspected to determine, if possible, the point of advance of the combustion front. This visual inspection was compared with peak temperatures indicated on the temperature recorder.

CHAPTER V

RESULTS AND DISCUSSION

The results of the reverse combustion tests at atmospheric pressure are shown in Tables I, II, and III. The test results of Table I were obtained to determine the minimum per cent butane that could be injected with air to cause a combustion front to move opposite to gas flow. Table II shows the data obtained in the reverse combustion test before injection of butane into the inlet gas stream. In Table III inlet and flue gas analysis data are shown for four different flux rates.

<u>High Flux Rate Tests</u>. Due to previous tests of the combustion of air-butane mixtures (11) at high flux rates, the first tests were performed at rates which are much higher than any reported in the literature. These results are reported here to show that reverse combustion can take place at very high flux rates.

In the tests to obtain the data in Table I Ottawa sand saturated with an oil of 27.4 degree API gravity to approximately 40 per cent saturation was used. The oil was originally a 37 degree API gravity crude, but it was heated to boil off the lighter hydrocarbons. The flux rates were varied from 2,808 to 3,225 (scf/hr-ft²), and the airgas mixture was varied from 0.73 to 3.9 per cent commercial butane by volume.

TABLE I

HIGH FLUX RATE DATA

2:12 2,940 120 3.9 2,000 2:25 2,863 117 3.87 2,020 2:30 2,990 110 3.56 2,030 2:35 2,988 102 3.27 2,030 2:47 3,157 23 0.73 2:52 2,808 32 1.14	ure Movement*
2:12 $2,940$ 120 3.9 $2,000$ $2:25$ $2,863$ 117 3.87 $2,020$ $2:30$ $2,990$ 110 3.56 $2,030$ $2:35$ $2,988$ 102 3.27 $2,030$ $2:47$ $3,157$ 23 0.73 $2:52$ $2,808$ 32 1.14	
2:252,8631173.872,0202:302,9901103.562,0302:352,9881023.272,0302:473,157230.732:522,808321.14	OGF
2:302,9901103.562,0302:352,9881023.272,0302:473,157230.732:522,808321.14	OGF
2:352,9881023.272,0302:473,157230.732:522,808321.14	OGF
2:47 3,157 23 0.73 2:52 2,808 32 1.14	OGF
2:52 2,808 32 1.14	0
	0
2:58 2,834 46 1.6	0
3:04 2,811 69 2.4 2,100	OGF
4:03 2,767 53 1.88 1,970	OGF
4:09 2,754 46 1.66 1,800	0
4:23 2,909 51 1.72 1,800	OGF
4:30 3,180 60 1.85 1,830	OGF
4:33 3,169 51 1.57 1,800	0
4:35 3,175 55 1.71 1,810	OGF
4:40 3,225 55 1.68 1,830	· · ·

*OGF - Opposite Gas Flow, 0-Out, S-Stabilized.

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 $(x_{i})_{i \in I}$

TABLE II

Run Number	Type Sand and Mesh Size	Oil Saturation % of Pore Volume	Air Flux U scf/hr-ft ²	Average Peak Temperature ^o F ^T p	CO	Produced CO2 mole %	Gas O
11:13-01	Ottawa (20-40)	43. 0	97	950	9.0	8.0	0
11:13-02	Ottawa (20-40)	43.0	106	990	2.2	10.0	0
11:13-03	Ottawa (20-40)	43.0	94	930	5.2	7.0	0.2
11:16-01	Ottawa (20-40)	41.0	100	970	2.3	11.2	0.4
12:09-01	River (25-45)	60.0	101	980	6.0	3.8	0
12:09-02	River (25-45)	60.0	70	860	8.6	8.4	0.6
12:13-01	50% Ottawa(20-	40) 40.0	106	1000	2.8	11.8	0.8
12:13-05	50% River (25-4	45) " 40.0	58	810	8.3	7.8	Ō
12:13-07	ñ ñ ĥ	ũ 40.0	83	900	8.4	10.5	0.5
12 :13- 09	Î Î Î	û 40.0	106	990	6.2	9.0	0.8
12:13-11	ũ ũ ì	îi 40.0	124	1030	1.2	9.9	0

REVERSE COMBUSTION DATA

TABLE III

GAS ANALYSIS DATA

Component	Run 12:13- Ug=94 scf/hr T_= 930°F	01 -ft ²	Run 12:13- Ug=100 scf/hr Tn= 9900F	05 -ft ²	Run 12:13 Ug=106 scf/h Tn= 1010 ⁰ F	-07 r-ft ²	Run 12:13-09 U _{g=} 124 scf/hr-ft ² T _g - 1030°F	
	Inlet Mole Rati	Flue	Inlet Mole Rat	Flue	Inlet Mole Rati	Flue o	Inlet Mole Rat	Flue io
02:N2	0.231	0	0.232	0	0.234	0	0.232	0
$CO \& CO_2: N_2$	0	0.132	0	0.118	0	0.120	0	0.122
C _{3H8} :N2	0.116	0.061	0.159	0.081	0.239	0.073	0.274	0.074
$i-C_4H_{10}:\mathbb{N}_2$	0.255	0.142	0.448	0.165	0.454	0.168	0.521	0.170
n-C4H10:N2	0.281	0.181	0.467	0.216	0,655	0,208	0.748	0.230

These tests were performed by holding the air flow rate constant and varying the gas flow rate, and also by holding the gas flow rate constant and varying the air flow rate. The center line temperatures were determined by equally spaced chromel-alumel thermocouples and recorded on a multipoint recorder. The results of these tests show that with a minimum of 1.68 per cent butane by volume a combustion zone can be caused to move opposite to the flow of the gas at flux rates from 2,808 to 3,225 (scf/hr-ft²).

<u>Temperature Distribution</u>. A typical temperature profile of the sand pack is shown in Figure 5. The high temperature is that of the combustion zone and the lower temperature $(200^{\circ}F)$ is that of the tube surface (skin temperature). It was found that ignition took place more quickly if the temperature profile was impressed on the end of the tube before air injection was started. Reed, et al. (2) in their experiments impressed a steady state temperature profile on the end of their tube after air injection had started. In this way they believed a steady state could be achieved more rapidly at low air rates.

In Figure 6 the average peak temperature is plotted against air rate. These temperatures are taken from the data prior to injecting butane into the inlet air stream. The reason no plots of temperatures were shown for the data after butane injection had started was due to the scatter of the data. It should be noted that, if the peak temperature of the combustion zone was below the self ignition temperature of the butane, a significant drop in peak temperature was observed upon the injection of butane. However, if the peak temperature exceeded the self-



FIGURE 5. SURFACE TEMPERATURE PROFILE ALONG TUBE LENGTH



FIGURE 6. PEAK TEMPERATURE

ignition temperature of butane, a rise in peak temperature was noted at the injection of butane into the inlet air stream. Since the addition of butane to the air stream decreased the per cent oxygen content of the inlet gas, the reaction rate per volumetric flow rate decreased at the combustion zone, therefore allowing the peak temperature to decrease. The heat absorbed by the gas in expanding and raising of its temperature as it moved through the combustion tube aided in lowering the entire temperature profile of the combustion tube. The self-ignition temperature of the commercial butane, which ranged from 961 to 995 degrees Fahrenheit (12), allowed some of the butane to go through the combustion zone without burning. With the factors stated above tending to decrease the peak temperature the heat of reaction and combustion must be increased with the injection of butane for an increase in peak temperature to take place. To increase the heat of reaction and combustion, the initial peak temperature must be greater than the ignition temperature of the gas to be added. Combustion Zone Velocity. The combustion zone velocity varied from experiment to experiment so much that an average combustion zone velocity could not be plotted. In some tests the zone was stationary at the outlet end of the tube, while in other tests the zone moved from the outlet to the inlet end.

In experiments where the sand pack was placed in a horizontal position during reverse combustion, the high temperature zone moved about erratically but never moved from the outlet end of the pack. This behavior was caused by the oil-in-place being moved toward the

combustion zone by air or gas drive. The heat conducted upstream from the combustion zone was used to raise the temperature of the flowing oil, and an insufficient amount of heat was left to raise the upstream sand temperature and cause the combustion zone to move in the upstream direction.

With the sand pack in the vertical position, the high temperature zone moved from the downstream to the upstream end. In these tests the zone would move a short distance, then remain stationary for a period of time, then move again, etc. Again this was contributed to an air or gas drive of the oil toward the combustion zone. The tighter the pack the less erratic movement of the combustion zone was observed.

<u>Butane Injection</u>. The commercial butane used in these experiments consisted of 48 mole per cent n-butane, 17 mole per cent i-butane, 33 mole per cent propane, and less than 2 mole per cent of ethane and methane. The butane was injected into the air stream after the steady state temperature profile was established by reverse combustion. <u>Gas Analysis</u>. Figure 7 shows plots of carbon monoxide and carbon dioxide from the data obtained in experiment numbered 12:13 in Table II. By taking the data for these plots from only one experiment, it eliminated the changes in data due to different conditions for each test. The plot of carbon dioxide is typical of all experiments that were conducted. The per cent of carbon dioxide reached a maximum for each experiment at approximately 106 (scf/hr-ft²) flux rate. The carbon monoxide was less predictable, but it was always at a maximum below



FIGURE 7. COMBUSTION PRODUCTS

80 (scf/hr-ft²) flux rate. In some of the experiments no traces of carbon monoxide could be found in the flue gas at flux rates from 94 to 120 (scf/hr-ft²). The oxygen content of the flue gas was at a minimum (and for most experiments zero) at flux rates in the vicinity of 100 (scf/hr-ft²).

The results in Table III were calculated from the data taken by the vapor phase analyzer. This gives a comparison of the inlet gas composition to the flue gas composition. A comparison of mole ratios of the inlet and flue gases was determined by assuming that the nitrogen was unaltered in passing through the heated combustion tube. The single column vapor phase analyzer limited the analysis to the lighter hydrocarbons.

Nitrogen and oxygen, as did carbon monoxide and carbon dioxide, had the same elution time on the Cenco Analyzer; therefore the orsat was used to determine the oxygen, carbon monoxide, and carbon dioxide content of every sample taken with the analyzer. Since the selfignition temperature of propane was 995 degrees Fahrenheit and that of butane 961 degrees Fahrenheit, the propane and butane was expected to react with the oxygen and cause an increase in temperature at the combustion zone. As the results show some reaction of the propane and butane did take place. As mentioned before, this did not raise the temperature of the combustion zone, and in some experiments the injection of butane caused a decrease in temperature as much as 70 degrees Fahrenheit.

In Figure 8 a plot of mole ratio differences (inlet gas minus





flue gas) versus air flux was made using the data from Table III. The mole ratio of the component to nitrogen was used because the nitrogen did not undergo a reaction in passing through the combustion The difference in mole ratio (inlet minus flue) of the propane zone. increases rapidly until a flux rate of 106 (scf/hr-ft²) is reached. This flux rate corresponds to a peak temperature of 1010 degrees Fahrenheit, which is sufficiently above the ignition temperature of propane (995 degrees Fahrenheit) to allow a portion of the propane to burn at the combustion zone, depending upon the availability of oxygen at the high temperature front. The flux rate, which corresponds to the inflection point on the isobutane curve in Figure 8, has a peak temperature shown in Table III as 990 degrees Fahrenheit. This temperature was 30 degrees higher than the ignition temperature of butane. The normal butane curve in Figure 8 shows an inflection point at 106 (scf/hr-ft²). Therefore, the amount of gas that passed through the combustion zone at rates above 106 (scf/hr-ft²) must be dependent only upon the availability of oxygen at the combustion zone. This last statement accounts for the decrease in slope of the curves in Figure 8 beyond the inflection points.

Figure 9 shows a plot of inlet and flue gas composition as recorded from the Cenco Vapor Phase Analyzer. The oxygen content of the air shown in the plot was determined by the orsat apparatus. Curves of this type were used in calculating the data shown in Table III and Figure 8.



FIGURE 9. CURVES FROM CHROMATOGRAPHY UNIT

ω S <u>Sand Pack Material</u>. The porous matrix used in these experiments as shown in Table II consisted of 20-40 mesh Ottawa fracturing sand and sand taken from the Cimarron River bed with a 25-45 mesh size. The average porosity of the river sand was 33 per cent and the effective air permeability was 19 darcys for an oil saturation of 40 per cent. The Ottawa sand has an average porosity of 35 per cent and 22 darcys effective air permeability with an oil saturation of 40 per cent.

The sand pack after the completion of an experiment starting at the outlet end usually had from 0-6 inches of loose unconsolidated light brown colored sand with 15-20 inches of hard consolidated grey sand beneath it. No chemical analysis was made on the sand.

CHAPTER VI

SUMMARY AND CONCLUSIONS

Reverse in situ combustion has been investigated by observing the results when commercial butane was injected with the air into an oil saturated sand pack. The study was carried out at flux rates from 2,808 to 3,225 (scf/hr-ft²) and from 58 to 124 (scf/hr-ft²). Data have been reported for most experiments performed at these flux rates. The effect to inlet and flue gas analyses, temperature distribution, and combustion zone velocity when butane was added to the inlet air stream of a reverse combustion process has been reported and discussed.

With a minimum of 1.68 per cent butane by volume a combustion zone can be caused to move opposite to the gas flow at flux rates from 2,808 to 3,225 (scf/hr-ft²). These rates are not the upper or lower limits for reverse combustion. The combustion zone can be stabilized (no up or downstream movement) by controlling the per cent of fuel gas supplied with the air, for the given conditions. It should be noted here that oil saturation should be an important factor in the stabilization of a combustion zone.

At flux rates from 58 to 124 $(scf/hr-ft^2)$ the peak temperatures can be represented as a function of air flux. With the injection of commercial butane into the air stream, it was found that the

combustion zone temperature decreased as much as 70 degrees Fahrenheit when the combustion zone temperature was below the ignition temperature of the butane. It was found that ignition occurred more readily if a temperature profile as shown in Figure 5 was impressed on the sand pack before air injection was started.

Commercial butane can be used to raise the temperature of a reverse combustion process, if the peak temperature is above the ignition temperature of the butane before injection is commenced, and if the injected air has a sufficient amount of oxygen content to allow reaction with the butane at the combustion zone.

Combustion zone velocity could not be represented as a function of air flux in these experiments. The permeability was such in these tests that oil movement by air or gas drive caused erratic combustion zone movement.

For air flux from 58 to 124 $(scf/hr-ft^2)$ carbon dioxide can be represented as a function of injection rate. Carbon dioxide reaches a maximum per cent of the flue gas at approximately 106 $(scf/hr-ft^2)$. Carbon monoxide is at a maximum below 80 $(scf/hr-ft^2)$.

CHAPTER VII

RECOMMENDATIONS FOR FUTURE STUDY

Since many people believe in situ combustion is a good candidate for tertiary recovery, it seems that the maximum and minimum limits of water and oil saturation should be investigated. The maximum water saturation and minimum oil saturation should be of considerable importance when considering application of the in situ combustion process to a water flooded reservoir.

Another study that should be of importance is a study of the air/oil ratio. The air/oil ratio is a major factor in any combustion process, and the literature available today gives very little usable information toward arriving at a favorable air/oil ratio.

It is recommended that for any future study in in situ combustion additional columns capable of determining the entire composition of the flue gas be obtained for the vapor phase analyzer. Also, a larger diameter combustion tube with immersion type thermocouples should be considered in any future study.

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