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ANTRIM OIL SHALE—ELECTRIC HEATER AND PROPANE BURNER FIELD IGNITION EXPERIMENTS

Topical Report, August 1977–April 1978

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MASTER

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Dow Chemical U. S. A.
Midland, Michigan

U. S. DEPARTMENT OF ENERGY

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FIELD IGNITION EXPERIMENTS

Topical Report

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August, 1977 - April, 1978

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ABSTRACT

The Dow Chemical Company contract with the Department of Energy to investigate the feasibility of recovering energy from Antrim oil shale by an in situ process required that an extraction trial be conducted using wells previously drilled by Dow. During this trial, two systems were used to initiate in situ combustion. An electric heater ignited the shale after two similar heaters were unsuccessful. The failures were due to electrical short-circuits and corrosion. Of four attempts to initiate combustion with a propane burner, two were successful, although considerable difficulty was experienced in lighting the propane burner. Difficulty was also experienced because the thermocouples placed in the well to provide positive indication of burner ignition were not durable. These results indicate the need for additional developmental work to make the burner more dependable and more easily ignitable.

INTRODUCTION

In late 1976, The Dow Chemical Company entered into a four-year contract with the Department of Energy to investigate the feasibility of recovering energy from Michigan's Antrim shale by an in situ process. This process requires that combustion be initiated in situ to consume part of the organic content of the shale to produce heat. The heat from the combustion process serves to retort the kerogen, thereby driving off a portion of it as a combustible gas or oil. The contract required that an in situ extraction trial be made using an existing site in which Dow had previously drilled several wells. This trial, the details of which will be reported separately, was carried out using two different ignition systems. The purpose of this report is to describe the ignition systems and their use in shale ignition experiments.

EXPERIMENTAL DEVELOPMENT

Electric Heater

Three 52 kw, 480 VAC, 3 phase resistance heaters, which developed a maximum design temperature of 1600°F on the elements, were manufactured by Petrotherm Company for Dow. These heaters were 3 inches in diameter and 20 feet long. Two were made from S.S. 321 and the third was made of Inconel* 600. A drawing of the heaters is shown in Figure 1. The six hairpin elements were connected in a Y pattern, i.e., at the bottom of the heater the leads were all welded together, while at the other end (the head) two electrical leads each were welded together to form three pairs. Each pair was silver soldered to one leg of the three conductor MI-730/3 cables supplied with the heater. The MI-730/3 cables were manufactured by Emerson-Chromalox and the power supply was a 60 kw SCR furnished by Loyola Industries.

Propane Burner

The propane burner was a tubular burner, shown in Figure 2. Methane gas for the pilot, supplied through a 1/4" stainless steel tube, was ignited by one of two different igniters, a silicon carbide igniter or a Watlow igniter. A catalytic igniter was used (as a back up igniter) in several of the field experiments. A thermowell of 1/8" Sch. 40 stainless steel was used to house a thermocouple which gives an indication of the pilot flame temperature. The methane pilot flame was directed towards the bottom of a 1/4" Sch. 40 S.S. pipe. After a methane pilot flame had been established, propane flow was started through this pipe to ignite the burner. These igniters will be discussed in detail in the following section.

The propane burner was made up of two parts, the burner itself and a burner stop. The burner stop provided a channel for the air to be directed to the burner, a thermowell holder for flame and charcoal bed temperatures, a bluff body holder, a catalytic igniter support and most importantly a support that the actual burner fits into. The air was

*Trademark of Huntington Alloys, a Division of International Nickel Co.

SECTION A-A'

IGNITION ASSEMBLY 480 VOLT-3 ϕ -52 KILOWATT		
SCALE: NONE	APPROVED BY:	DRAWN BY <i>Clanahan</i>
DATE: 10-13-76		
		REVISED
PETROTHERM BAKERSFIELD, CALIF. 93309		
DOW CHEMICAL CO.		DRAWING NUMBER 300263

REV.	DESCRIPTION
A 1/11/77	ADDED THERMOWELL / SUB COUPLING

FIGURE 1

Figure 2
Propane Burner Assembly and Holder with Burner Section and Detail of Interior Piping

supplied two ways to the burner. The tubing air (burner air) was supplied down the annular space between the inside of the 2-1/2" EUE tubing which is connected to the burner stop at the bottom and the outside of a 1/2" propane supply pipe which has several other lines strapped to it to supply methane, electricity, and instrumentation (thermocouples) to the burner itself. When the burner, which was hung on the 1/2" propane line, is inserted into the burner stop it fits tightly. This tight fit forces the air through the four 1/2" holes near the top of the burner. The air is thereby directed over the internal parts of the burner to help keep them cool. The tubing air is the primary air supplied to the burner and is the only air needed to support the combustion of the propane.

The secondary air, called annular air, was supplied as excess air to help support the combustion of the shale or charcoal when it had reached its auto-ignition temperature. This air was supplied between the outside of the 2-1/2" EUE tubing and the casing (or borehole). The casing sizes ranged from 5-1/2" to 9-5/8" casing. The open borehole size ranged from 7-1/2" to 4 feet.

The thermocouples on the burner stop indicated flame temperature of the propane burner, charcoal temperature (locally), and a temperature profile up the well.

Propane Burner Igniters

Three different types of igniters were used. They were silicon carbide, Watlow, and a catalytic igniter, shown in Figure 3.

The silicon carbide igniter, which was manufactured by Carborundum, has been used in gas ovens for the past several years. It operated in the range of 95-120 volts DC at 2.3 to 3.3 amps, which can provide a temperature range of 1800-2800°F. This temperature was high enough to ignite the methane pilot (methane autoignition temperature is 1170°F).

The igniter was very fragile and easily broken if special care were not taken to protect it. Accordingly, it was protected by a stainless steel shroud which had wax melted into it to act as a shock absorber. The igniter shroud and wax are shown in Figure 4. The development of the igniter shroud and wax was discussed in an earlier Dow report, No. FR-327,⁽¹⁾ which under the terms of the Department of Energy contract, was previously submitted to DOE.

Watlow Igniter

The Watlow igniter, manufactured by Watlow Industries in Hannibal, Missouri, was a solid rod heater element as shown in Figure 5. The outside sheath of the igniter was made of Incoloy* 800. Electrical power was supplied to the inside of the element and the outside sheath

(1) Cole, J. L. "Antrim Shale - Atmospheric Pressure Testing of Downhole Burners."

*Trademark of Huntington Alloys, A Division of International Nickel Co.

THREE IGNITERS USED
IN PROPANE BURNER

CARBORUNDUM
IGNITER

WATLOW
IGNITER

CATALYTIC
IGNITER

FIGURE 3

Installation of Shroud Around Stainless Igniter Bushing

FIGURE 4

WATLOW IGNITER

FIGURE 5

acted as the electrical return. The unit was more durable compared to the Carborundum igniter, but had a low operating temperature range. The igniter operated at approximately 1.9 amps and 110 volts DC which delivered a temperature range of 1600-2000°F. The development of the igniter was done completely by Watlow Industries. The only addition to the igniter was a stainless steel shroud similar to that used with the silicon carbide igniter. The shroud helped to contain the heat in close proximity to the igniter, which is important because of the lower operating temperature.

Catalytic Igniter

Unlike the previous igniters which were used to ignite a methane pilot, the catalytic igniter was used to ignite propane directly. With this igniter, hydrogen and air were made to react at room temperature by passing them over a precious metal catalyst (eg., Pt, Pd). This highly exothermic reaction caused the temperature of the catalyst bed to exceed the propane autoignition temperature.

The igniter consisted essentially of a 1/4" diameter and 1" long bed of palladium sponge catalyst connected to the end of a 1/4" S.S. tubing line. The catalyst bed was formed by making a 1/4" diameter tube from stainless steel screen. Another tubular screen made from 80 mesh platinum gauze was inserted inside the S.S. screen. Although platinum also catalyzes the reaction, this inner screen was mainly used to retain the smaller catalyst particles.

The igniter was positioned alongside the bluff body, but not in contact with it. With air flowing around the outside of the catalyst bed, hydrogen flow was started at approximately 0.16 scfm through the 1/4" S.S. tube and into the catalyst bed. The catalyst initiated a hydrogen flame which stabilized at temperatures between 1200°F and 1400°F. Once the igniter had reached its operating temperature, propane flow was started and the propane/air mixture flowing past the bluff body was ignited (Figure 6).

Bluff Bodies

A bluff body is any object that tends to recycle hot gases from a flame back near the opening where cooler gases enter, preheating them to autoignition temperature. This process helps to make the flame more stable. A drawing of the propane burner with a bluff body attached is shown in Figure 7.

During initial burner testing, it was found that the flame was stable and would stay attached to the burner without the pilot being left on. However, during the test at the field site, it was found that the flame wouldn't stay attached to the burner when the pilot was turned off. The reason for the change was that during initial testing the burner was confined in a test chamber. As the chamber reached autoignition temperature (874°F), the flame wouldn't go out. In above ground tests, cooling prevented the burner from remaining at the propane autoignition

CATALYTIC IGNITER DESIGN

SCALE: 1" = 1/2"

FIGURE 6

*Trademark of Crawford Fitting Co.

PROPANE BURNER
AIR FLOW PATTERN

FIGURE 7

temperature, therefore, when the pilot (methane) was turned off the flame became unstable and went out. At this point, it became apparent that something was needed to stabilize the flame. A bluff body was the easiest available means to solve the problem.

For the initial tests, it was decided to use a series of round shapes: 1/4" tube, 3/8" tube, 1/4" pipe, 1/2" pipe and 1" pipe, all made of stainless steel. Each bluff body was positioned at the end of the burner, centered across the opening as shown in Figure 7. As the size of the bluff body was increased, it was seen that a higher air flow rate could be used with better stability. The 1/2" pipe and the 1" pipe gave very similar results, therefore, it was decided to use the 1/2" pipe which would restrict the opening as little as possible without changing the flame stability.

The 1/2" bluff body was also tested in a different manner. An adjustable support was made for the 1/2" pipe which could move the bluff body away from the end of the burner stop. The purpose was to see if the flame would stay attached to the bluff body at varying distances. It was thought that this would make the burner run cooler. The tests showed very little change in the burner operating temperature. Therefore, the bluff body was attached directly to the end of the burner stop.

FIELD TESTS

Electric Heater

The Petrotherm electric heater was used in three different combustion tests. The first two tests used an electric heater made of S.S. 321 and the third used one made of Inconel 600.

Experiment No. 1

The electric heater was first tested above ground by heating the unit up to 392-572°F. All indications were that the heater was functioning correctly.

The heater was placed in Well #4 on 8/15/77 and after the heater had been in operation for 2 hours, the kilowatt meter went to full scale, indicating a short had developed. The well was vented and the heater was pulled. The short, found to be in the heater head, was attributed to the proximity of the connections between various elements and the MI cable, which had touched.

Experiment No. 2

The second (back-up) heater was checked in an above ground test for the similar shorting possibility and indeed the heater did short out in a similar manner. The heater was repaired at the site by wrapping and isolating the bare leads with glass tape. This heater was installed in

Well #4 and the combustion test was started again on 8/25/77. The heater was run at 44 kw for 6 hours at which time it also developed a short.

Inspection of the heater after removal revealed cracks on 5 out of the 6 elements approximately 3 inches down from the top of the head. The heater elements were analyzed by Dow Materials and Mechanical R&D. They determined that the cracks were produced by stress corrosion cracking. Stainless steel is known to be susceptible to this effect in the presence of chloride ion at temperatures over 800°F. Since the well contains dilute brine, it is probable that enough contact occurred with the hot metal to produce the stress corrosion cracking problem.

Experiment No. 3

A new electric heater was installed in Well #4 and set to a depth of 1306 feet. This heater was made of Inconel 600 which had been selected because of its resistance to chloride induced stress corrosion cracking. The heater was started on 11/13/77. The heater worked well until the second day when it developed a short in one power leg. At this point, the temperature near the heater began to drop off to a steady temperature of about 932°F from 1292°F. Then it was decided to lower the air flow rate from 60 scfm to 40 scfm to get the temperature back up to 1292°F. These conditions were maintained until 11/24/77 when the other two legs of the heater shorted out. The air flow was continued for an additional 1-1/2 days but was finally terminated due to declining Btu content in the production gases.

Above ground examination of the heater revealed that the MI cable 12 feet above the heater was burned through. This may have been the cause of the first short. One element had been burned through one inch below the heater head which let water vapor into the head of the heater. The inside of the heater head was corroded and moist. The heater head and components were analyzed by Dow's Materials Engineering Group and also by Huntington Alloys and their report concluded that the problem was due to stress corrosion cracking caused by the use of silver solder.

A section of the tube was metallographically mounted and examined on the electron microprobe analyzers. These results are shown in the photographs of Figure 8. Very high levels of silver, copper, zinc, and cadmium in the cracks indicated that this failure was caused by molten silver stress-corrosion cracking. This occurs frequently when brazing nickel alloys with silver brazes. If sufficient stress is present when the brazing alloy is molten, cracking may occur. This stress can be from cold working, age hardening, or joint fitup stress. The remedy is to either remove the stresses or change to a copper or nickel base brazing material.

Propane Burner

The 250,000 Btu/hr propane burner was used in four different shale combustion attempts, two of which were successful. The other two failed due to equipment problems.

No. 379X-2 Sample Current 1000X

No. 379X-2 Silver Scan 1000X

No. 379X-2 Zinc Scan 1000X

No. 379X-2 Copper Scan 1000X

No. 379X-2 Cadmium Scan 1000X

Experiment No. 1

The burner was installed in Well #4 at 1294 ft. After pressure testing of the well, the resistance of the igniter was found to be 10-20 megohms. The igniter resistance should have been approximately 250 ohms. It is conjectured that when the well was pressurized, the pressure was transmitted through the wax unevenly to the igniter, causing it to break. To resolve this problem for future tests, it was decided to melt the wax from around the igniter before pressurizing the well to distribute the pressure equally over the entire igniter.

Experiment No. 2

The igniter was replaced in the propane burner and the burner was reinstalled in Well #4. This time the wax was melted from the igniter before the well was pressure checked. The well was pressurized to 850 psig and the igniter was found to be in good working order. The igniter was brought up to temperature and the methane flow was started. At two different intervals, ignition of the methane was thought to have occurred, since the temperature on the thermocouples had risen rapidly. Each time when the methane was lit, the propane flow was started to the burner, but there was no indication of propane ignition. Further attempts to establish the methane pilot were unsuccessful so the igniter temperature was increased. The igniter burned out after approximately 10 minutes, which made it impossible to continue attempts to ignite the methane pilot, so the burner was pulled from the well.

Experiment No. 3

After the third electric heater trial, Well #4 was filled with coal to 1306 ft, charcoal to 1208 ft, and the burner stop was set at 1204 ft. The coal and the charcoal were used to create a larger fire for initiating the shale combustion. This propane burner was equipped with two igniters, a Watlow igniter made of Incoloy 800 and a catalytic igniter.

To ignite the burner, the catalytic igniter was tried first for approximately 45 minutes with no success. Then the Watlow igniter was tried for approximately 45 minutes with no success either. Finally, the two igniters were tried at the same time, and the burner was successfully ignited. The burner was operated for 8 hours before it went out. Attempts to reignite it failed but combustion in the charcoal bed had been initiated.

After completion of the extraction trial, the burner was removed from Well #4 with little problem. The 2-1/2" EUE tubing holding the burner stop was also pulled from the well with no problem.

The outside bundle of thermocouples were burned off approximately 5 feet from the burner stop. The pilot flame thermowell was burned off at the

wall on the burner stop. The thermowell extending into the charcoal was found to be intact.

The intense heat had burned several holes through the wall of the burner stop, as well as on the 1" x 1/4" fins. The holes in the pipe were 2-1/2" to 3-1/2" long and 1/2" to 1" wide. The fins were burned off 4" to 6" up the burner stop in three locations.

The burner was found to be plugged 10" up from the bottom. The burner shroud had sagged 10" from the bottom. This buckle was due to the high heat and weight on the burner.

The 1/4" S.S. tubing line used for hydrogen was burned off for 5 feet up from the bottom of the stop. The bluff body (Sch. 40, carbon steel 1/2" pipe) on the end of the stop was also burned off. The Watlow igniter had burned out; the inside coil was burned through.

The burner was examined by Dow Materials and Mechanical R&D. Their conclusions are summarized in comments included in Figure 9, which shows a photograph of parts of the burner after the inner pipe had been cut into three pieces.

Experiment No. 4

Well #7 was loaded to 1216 ft with charcoal and the burner stop was then set at 1213 ft. The stop was set within 3 ft of the charcoal by using a micro switch which was attached to the 10 foot long thermowell, 5 feet below the burner stop. When the switch hit the charcoal the resistance went to zero in the line and the burner stop position was known. The stop was then lowered 2 more feet. After approximately 16 hours of varying air/fuel ratios and changing the pilot fuel from methane to hydrogen, the burner lit and was used to ignite the charcoal. The burner was turned off less than a day later because most of the oxygen was being burned up in the combustion of propane, and thus not enough oxygen was available for the burning of the charcoal.

After completion of the extraction trial, the propane burner was pulled from Well #7 with no problems. Some of the MI igniter power cable was worn through in two locations. The MI cable had been used two times previously in Well #4. Abrasion from the tubing walls and MI cable rubbing together for 1200 feet one way probably caused the cable to wear through. Even though the cables are banded with S.S. straps to the propane pipe, there is little distance between the cable and the inside wall of the 2-1/2" EUE burner stop tubing.

The Watlow igniter had burned out again on the inside coil, meaning the inside coil was run at 2000°F or higher for an extended period of time. The S.S. shroud around the igniter was burned through in one location, indicating a localized hot spot.

The burner had a slight ripple in it near the inlet air hole. This showed some fatigue due to the high temperature and weight. The burner thermocouple string was in good shape with only minor pitting shown. The propane line was plugged with carbon and shale which could have been

1. These arrows point to holes that were burned through the outer pipe wall. This would require a minimum temperature of 2850°F.
2. This arrow points to a small copper rod which had melted, thus indicating a temperature of at least 2000°F.
3. This arrow points to an area of severe oxidation and sagging which would require a temperature of about 2500°F.

FIGURE 9

caused by repeated venting of the well. The methane pilot line was also burned through for 2-1/2". This burning through is attributed to a localized hot spot. As for the methane line burning off, a pin hole may have developed and the methane and/or hydrogen could have been burning in this area.

The 2-1/2" EUE tubing was pulled from Well #7 with no problems. The thermocouples along the outside were burned off 5 feet back up from the bottom of the stop. The thermocouples were badly pitted for about 5 more feet. The catalytic igniter was burned off, and 4-1/2 feet up from the bottom of the stop the 1/4" hydrogen tubing line was burned through. The end of the catalytic igniter was burned off due to the high temperature of the propane flame.

The propane burner flame thermowell was burned off at the weld. This was caused by the high temperature of the flame. The charcoal thermowell was found to have a cork screw look. This was caused when the thermowell was lowered into the charcoal. The 1/2" thermowell wasn't heavy enough to push into the charcoal without bending.

ENVIRONMENTAL

The impact of this project has been minimized by the rather small size and limited operating time. The environmental aspects of our field activities are monitored by the Environmental Research Institute of Michigan. A summary of their findings are included in the Quarterly Technical Progress Reports of this project.

SUMMARY

The Antrim shale of Michigan underlies most of the Lower Peninsula. This shale, which contains an appreciable concentration of organics (kerogen), is a vast and potentially very valuable energy resource. An in situ combustion process is considered to be the most economical process for extracting energy at this time. In this process some of the organic material in the shale is burned to supply the temperature necessary to break down kerogen into oil and gas products. Two systems to ignite the shale were designed and tested in the field. The two systems tested were a 52 kw electric heater and a 250,000 Btu/hr propane burner.

The electric heater was tried in three different attempts to ignite the shale. The first two were unsuccessful and the third test met with limited success. The electrical heater has one major limitation, materials of construction having better corrosion resistance are needed for a longer life in the well.

The propane burner has been tried in four different attempts to ignite the shale. The initial two attempts were unsuccessful while the third and fourth attempts were successful. In the third and fourth trials the

burner was used in conjunction with charcoal. Several problems were apparent; additional work is needed before the burner could be considered to be dependable.

- 1) Better flame indication is needed. The thermocouples don't last long enough.
- 2) The burner needs to be easier to ignite under actual field conditions.
- 3) The burner needs to be reignitable easily at pressure and after being operated for some period of time.