**Quenching and Stabilization of MIS Retorts:
Bench-Scale Experiments**

Topical Report

**F.A. Barbour
J.E. Boysen**

April 1991

Work Performed Under Cooperative Agreement No.: DE-FC21-86MC11076

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Western Research Institute
Laramie, Wyoming

MASTER

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SUMMARY

This research was conducted to evaluate in situ retort stabilization methods. The objective of the bench-scale simulations was to evaluate possible post-retorting operating procedures for the optimum cleaning of spent retorts. After simulating conditions of modified in situ (MIS) retorts at the time retorting had ended, procedures to accelerate retort cleanup without using large volumes of water were investigated. Samples from various levels of the retort were used to determine the amount of water-soluble constituents in the spent shale and the rehydration characteristics of the spent shale.

The organic material that remained after retorting was most effectively removed from the retort by the use of reverse combustion. The removal of the organic material in this manner cracked the oil on the unretorted shale and removed heat from the bottom of the retort. Both were then transported toward the top of the retort. Unretorted kerogen was coked as it emerged from the shale near the reverse-combustion front. The reverse-combustion technique had an additional benefit in that the carbon deposited on the spent shale in the combusted zone appeared to provide a barrier to rehydration of the shale on introduction of water into the retorts.

A hot quench immediately following retorting was also relatively effective in removing organic material from the retort. However, the quench did leave some organic material on the unretorted shale. This material was not readily removed by water leaching during laboratory testing.

A deluge of water on a cool retort did not efficiently remove the organic material from the unretorted shale nor did the addition of a biodegradable detergent.

INTRODUCTION

Occidental Oil Shale Inc. (OOSI) successfully demonstrated its MIS oil shale retorting technology by conducting large-scale field tests at its Logan Wash facility near De Beque, Colorado. OOSI operated the Logan Wash MIS retorting facility from 1972 until 1982 (Stevens and Zahradnik 1983). The MIS technology developed by OOSI was proven technically feasible, and commercial projections suggest that the technology can be profitable in the future.

One of the environmental concerns surrounding MIS retorting technology is the impact of the process on local groundwater. Chemical compounds that will solubilize in groundwater are created during MIS retorting. The hot spent retorts must be cooled and then cleaned of these water-soluble chemical compounds to mitigate the process's impact on local groundwater. Spent retorts were cooled and cleaned at Logan Wash by circulating water through the retorts to remove soluble materials. Water produced from the MIS retorts (process water) was used in the cooling and initial stages of the cleaning. Baseline quality groundwater collected in the mine sumps (mine water) was used in the later stages of the cleaning. The groundwater quality from most spent retorts at Logan Wash was restored to near baseline. These experiments demonstrated that impact on groundwater quality from MIS retorting can be mitigated. However, a commercial-scale operation requires careful water management to ensure that sufficient water is available to clean all spent retorts created.

OOSI is developing plans for demonstrating commercial-scale oil production from a federal prototype oil shale lease (Tract C-b) in northwestern Colorado. As a part of the initial investigations, Western Research Institute (WRI) is evaluating procedures for optimum water management by conducting bench-scale research.

WRI has constructed a laboratory-scale simulator for the MIS retort cleaning process. The objective of the simulations is to determine a post-retorting operating procedure that optimizes water management during the cleaning of the spent retorts. After conditions in spent retorts were simulated, procedures to accelerate retort cleanup without using large volumes of water were investigated.

EXPERIMENTAL

Simulation Retort

The tube reactor for one-dimensional simulations (Figure 1) of the MIS retorts is capable of simulating the thermal recovery process of retorting and reverse combustion. In addition, the reactor is equipped for water and steam introduction after retorting to simulate quench and deluge procedures.

Figure 1. Tube Reactor Schematic

Retort Design

The reactor tube (8.5-cm i.d. x 80-cm long or 3 5/16 x 32 inches) was uniformly packed and vertically oriented within a series of insulated shield heaters. Auxiliary equipment includes a steam generator, product separation equipment, a continuous oxygen analyzer, and a gas chromatograph. The entire system is instrumented and interfaced to a data acquisition computer that records temperature and flow rates every 5 minutes.

Six internal reactor thermocouples were spaced approximately every 15 cm within the center of the shale bed to monitor the thermal front movement and determine the thermal history of the shale. Four of these thermocouples were also electronically paired with additional internal-wall thermocouples. Each pair was connected to one of four individual temperature controllers and shield heaters. The heaters were operated adiabatically to reduce radial heat loss.

Product oil and water samples were collected from three knockout pots. The first was operated at ambient temperature, whereas the other two were chilled to 32°F (0°C) to condense water and oil vapors. The gas exiting the last knockout pot supplied the oxygen meter and gas chromatograph with sample, whereas the excess was vented to maintain a pressure of 2 psig in the reactor. This pressure ensured adequate flow to the chromatograph and oxygen meter.

Retort Loading

The retort was charged with approximately 5000 g of crushed shale sized -1/2 inch to +60 mesh. The sample was well mixed to ensure a uniform shale bed for each of the experimental tests and was loaded into the retort. During the loading procedure a composite shale sample was taken for analysis by Fischer assay.

The rubblized shale bed contained five sample pouches dispersed vertically throughout the reactor. These pouches were constructed of stainless steel screen and contained approximately 25 g of shale. During loading, the pouches were situated just above the thermocouples in the shale bed.

Retort Operation

Ignition of the shale was accomplished by preheating the top 15 cm of the reactor with the first shield heater. This heater was set to 1200°F (649°C), and air was introduced into the top of the reactor at a rate of 0.09 scfm. The oxygen content of the gas exiting the retort was monitored and ignition noted by a drop in oxygen content. The remaining temperature controllers were operated adiabatically for the duration of the experiment. The retorting was halted when the conditions in the simulation retort were similar to those in MIS retorts (Boysen et al. 1990). This provided three zones of interest: combusted, retorted, and unretorted.

Retort Unloading

The retort was unloaded in three sections, which correspond to the following zones of interest.

- Combusted >1000°F (538°C)
- Retorted 500 to 1000°F (260 to 538°C)
- Unretorted <500°F (260°C)

The cutpoints between these zones were estimated by the temperature profiles observed during retort operation. The samples were removed from the retort by a vacuum and collected by placing a tared clean plastic bag in a vacuum canister. The sample pouches were removed as the retort was unloaded. Each pouch was weighed and labeled appropriately.

Spent Retort Remediation

Once the retorting had been halted, several post-retorting operating procedures were tested. The quenching and deluge cleaning techniques used by OOSI (Hester and Jacobson 1983) and EMR_X (EMR_X Corporation 1989) were evaluated with respect to usefulness and affect on individual zones of the retort. Reverse combustion was examined as a method of organic source removal after completing the retorting operation. These methods of remediation were compared with retort simulations in which nothing was done after the retorting.

Deluge Procedure

Simulation of the deluge procedure was performed on cooled retorts. The oil collection system was isolated from the retort, and the bottom of the retort was opened to a clean collection pot. When the deluge simulation was started immediately following the quench procedure, the retort temperature was very near that of steam. When the deluge procedure was preformed without quenching, the retort was allow to cool to ambient temperature prior to starting the deluge.

Water was introduced into the top of the retort. The amount of water injected into the retort was approximately 40 cm³. Based on the cross-sectional area of the MIS retorts, the flow rate should have been about 200 mL/min for the deluge simulations. However, because the simulation retort has a higher void volume than the MIS retorts, the rate of water injection was reduced to achieve a solid-to-water ratio that more closely reproduced the injection rate that used during the field tests. The deluge simulation was followed by analyzing the water for total organic carbon (TOC) and was terminated when the TOC reached an asymptote.

A composite sample of the deluge water was preserved for further analysis. The sample was composited based on the volume of each sample collected during the deluge simulation.

Quenching Procedure

Immediately after the retorting was completed, the quenching procedure was started. The retort was still very hot with most temperatures above 1200°F (649°C). The oil collection system was isolated from the retort, and the bottom of the retort was opened to a clean collection pot. The top of the retort was vented through the condensers for collection of the steam generated during the quench procedure.

Well water was introduced into the top of the retort. The amount of water injected into the retort was approximately 4 mL/min. Based on the cross-sectional area of the MIS retorts, the flow rate should have been about 0.4 mL/min for the quench simulations. The 4-mL/min rate, although higher than the calculated flow based on field data, was easier to maintain and permitted cooldown of the retort in a reasonable amount of time. Using this flow rate, the simulation retort was cooled in about 16 hours.

A composite sample of the quench water that migrated through the retort was preserved for further analysis. The sample was composited based on the volume of each sample collected during the quench simulation.

Reverse Combustion

Reverse combustion was started after the retorting reached a point at which about 85 percent of the shale had been retorted. At the end of retorting, the air to the top of the retort was shut off, and the top of the retort was vented through the condensers for oil and water collection. The oil collection pot at the bottom of the retort was isolated from the retort production and air lines. Air flow was introduced into the bottom of the retort very slowly. The air flow was increased to the operating flow of 0.15 scfm over 5 minutes. The reverse burn was terminated when the thermocouple at the bottom of the retort indicated the combustion front had been drawn to that point.

Analytical Procedures

Shale Leaching

Shale samples from the sample pouches and the three retort zones were leached with deionized water to determine the amount of water-soluble constituents in the spent shale and rehydration characteristics of the spent shale.

The shale sample pouches were added to water in a ratio of one part shale to 20 parts water. The mixture was stirred for 24 hours at ambient temperature and then separated by filtration. The amount of water that was readsorbed as a result of leaching the sample pouches was determined gravimetrically. A weighed sample isolated from the leaching mixtures was dried for 24 hours at 104°C (219°F) in a drying oven. The sample was then weighed, and the weight loss was recorded as water adsorbed on the shale during leaching.

Samples of each of the retorted zones were also leached for 24 hours at the same water-to-solid ratio to obtain enough liquid for other analysis. The liquids were separated from the solids by filtration, and the liquids were preserved for further analysis. The analyses performed were from the limited or full analyses suite for liquid samples.

Analytical Methods

TOC measurements were made during the bench-scale simulations at the time the samples became available. The analysis consisted of combustion of the sample to CO₂ at 1472°F (800°C); the CO₂ concentration was determined by coulometric titration.

Gas generated during the retort and reverse combustion operations was analyzed by gas chromatography (GC). The gaseous components were separated by a 2-mm-i.d. x 6-m stainless steel column packed with Chromosorb 102 and detected using a thermoconductivity detector. The gas flowed through a gas sample valve at the GC; gas was sampled as the GC started.

The analyses of water and solid samples were performed by WRI's analytical laboratory using U.S. EPA, ASTM, standard (American Public Health Association 1985) and WRI-developed methods. The analytical methods are listed in Table 1. The WRI methods are modifications of standard methods, and these have been devised to obtain more reliable data when applying standard techniques to synthetic fossil fuel products.

The analyses performed on the water samples taken from the quench and/or deluge remediation tests were selected from one of two suites of analyses. Table 2 lists the constituents in each suite as well as special analysis performed on some samples. The smaller limited suite was used for most of the work done on the retort quench and stabilization studies. This suite was based on constituents that showed the largest concentration or variation in previous quench tests performed on MIS retorts (Hester and Jacobson 1983). The full-suite analysis provides a more complete picture of the dissolved metals that were present in the water. This suite was randomly selected for use on the quench and deluge waters. The special analyses listed in Table 2 were run on selected samples to determine organic species that were present in these waters.

RESULTS AND DISCUSSION

Shale Retorting

The operation of the simulation retort was duplicated as close as possible for all of the tests. This was done to simulate post-retorting conditions that resembled the conditions in the MIS retorts. In addition, the duplication also gave approximately the same starting point for remediation tests that followed retorting. Gas analyses and Fischer assay data were used to evaluate the retorting portion of the experiments and provide material balance data.

Table 1. Analytical Methods for Water and Solid Characterization

Parameter	Analytical Technique	Method
Total organic carbon	combustion to CO ₂ followed by coulometric titration	SM 505 ^a
Total dissolved solids	gravimetric	EPA 160.1
Alkalinity	titrimetric	EPA 310.1
Conductivity	electrometric	EPA 120.1
pH	electrometric	EPA 150.1
Ammonia	automated colorimetric	EPA 351.2
Phenols	automated 4-AAP with distillation	EPA 420.2
Sulfate	turbidimetric	EPA 375.4
Dissolved Metals	inductively coupled plasma	EPA 200.7
Fluoride	ion chromatography	EPA 300.0
Volatile organics	GC-MS	EPA 624
Semivolatile organics	GC-MS	EPA 625
Fischer Assay		

^a SM refers to Standard Methods for the Examination of Water and Wastewater (1985).

Table 2. Components of Analysis Suites

Limited Suite	Full Suite	Special Analyses
Total organic carbon	Total organic carbon	Volatile organics
Total dissolved solids	Total dissolved solids	Semivolatile
Alkalinity	Alkalinity	organics
pH	pH	
Ammonia	Ammonia	
Phenols	Phenols	
Sulfate	Sulfate	
Fluoride	Fluoride	
	Conductivity	
Dissolved Metals	Dissolved Metals	
Arsenic	Arsenic	Aluminum
Boron	Boron	Cadmium
Molybdenum	Molybdenum	Magnesium
	Lead	Manganese
	Calcium	Zinc
	Selenium	Thallium
	Sodium	Silver
	Barium	Beryllium

The experimental tests and conditions are listed in Table 3. The amounts of oil removed by retorting conditions are also given. The first four tests were stopped when approximately 60 percent of the shale had been retorted. The remaining tests were allowed to continue until about 85 percent of the oil present in the shale had been removed. The amount of oil removed from the shale was based on Fischer assay data before and after retorting. In tests that included reverse combustion as a remediation procedure, the oil amount of recovered during the retorting portion of the experiment was included in the calculation.

The average gas analysis data for the thirteen retorting experiments are listed in Table 4. Although these data show differences between tests, there does not seem to be a large variation. The tests were probably duplicated well enough for the purpose of conducting the remediation tests. The amount of gas produced as determined by nitrogen balance was calculated from the average gas analysis listed in Table 4. The results of individual gas analyses performed on an hourly basis during each test are listed in Appendix A.

Fischer assay data on composite samples from each retort test show that the shale was about the same for each of the tests. The oil yield data for these samples are depicted in Figure 2. The oil present in each of the composite samples is about 9.5 wt %; with the exception of test 3, all of the shale samples are fairly close to 9.5 wt %. All of the Fischer assay data obtained on the composite and retort zone samples are in Appendix B.

The oil yield data determined by Fischer assay for each of the retort zones show how well the cuts between the zones were defined. These data are shown in Figure 2. When the spent shale was removed from the retort, the zones were determined by changes in the spent shale. This determination was easy for the retort tests that were not exposed to water. The color of the combusted shale was light tan when the retort was dry. The retorted shale was black because of carbon layer from the retorting. The shale in the unretorted portion of the reactor was generally similar to the original shale, but it had a coat of oil derived from the retort operation. The oil yield data for the dry test (tests 1 and 2) show that the cuts between the retorted and unretorted zones were good.

When the spent shale was wet, as a result of a quenching or deluge procedure, it was more difficult to visibly distinguish between the individual zones. The shale from the combusted zone was somewhat darker but could still be readily distinguished from the shale in the retorted zone. The retorted and the unretorted zones were more difficult to identify after exposure to water, and the temperature profiles were relied on for separation of these two zones. The oil yield Fischer assay data suggest that the cuts between the retorted and unretorted zones were not quite as clean as we expected. Tests 4, 9, and 10 have some unretorted material in the retorted fraction. The opposite occurred for tests 6 and 13 with a portion of the retorted material mixed in with the unretorted fraction. These variations amount to approximately 20 wt % and must be taken into account when comparing the oil yield data.

Table 3. Retort Quenching and Stabilization Simulations

Exp. #	Experimental Conditions	Oil Removed, % (retorting)
1	No quench, no deluge	58
2	Reverse combustion, no quench, no deluge	58
3	Reverse combustion, no quench, deluge	59
4	No quench, deluge	65
5	Reverse combustion, no quench, deluge	89
6	Quench, deluge	90
7	Reverse combustion, quench, deluge	89
8	Quench, deluge with recycle	85
9	No quench, deluge with surfactant	77
10	Quench at combustion front, deluge	79
11	No quench, steam injection at combustion front, deluge	73
12	Simultaneous reverse combustion and quench, deluge	84
13	Quench, deluge with fly ash slurry	91

Table 4. Average Gas Analyses for Retorting Experiments, mole %

	Experiment Number												
	1	2	3	4	5	6	7	8	9	10	11	12	13
Hydrogen	0.97	1.30	2.11	1.17	1.41	0.91	1.20	0.59	1.15	1.23	1.39	0.91	1.11
Nitrogen	64.46	68.12	56.85	60.58	60.66	63.13	62.61	64.92	63.00	62.80	62.69	65.52	63.95
Oxygen	0.24	0.04	0.00	0.08	0.10	0.41	0.06	0.28	0.39	0.05	0.06	0.27	0.13
Argon	0.73	0.77	0.66	0.69	0.68	0.74	0.71	0.75	0.69	0.70	0.70	0.75	0.73
Carbon Monoxide	2.08	1.76	3.36	2.73	2.75	2.17	2.45	1.91	2.46	2.16	2.22	2.14	2.15
Carbon Dioxide	29.36	25.62	35.17	32.89	32.37	30.73	31.02	29.40	30.00	31.07	30.82	28.03	29.08
Methane	0.96	1.00	1.06	0.93	0.91	0.93	0.93	0.94	1.08	1.00	1.03	1.03	1.02
Ethane	0.28	0.28	0.29	0.23	0.25	0.25	0.27	0.26	0.32	0.28	0.29	0.28	0.26
Ethylene	0.09	0.06	0.06	0.09	0.07	0.04	0.06	0.07	0.08	0.07	0.08	0.08	0.10
Propane	0.28	0.48	0.12	0.24	0.32	0.21	0.22	0.39	0.35	0.18	0.23	0.39	0.83
Propylene	0.27	0.35	0.08	0.24	0.19	0.20	0.14	0.19	0.15	0.11	0.11	0.28	0.29
Butanes	0.13	0.16	0.03	0.12	0.11	0.11	0.14	0.14	0.18	0.14	0.14	0.17	0.20
Hydrogen Sulfide	0.16	0.05	0.20	0.02	0.19	0.16	0.20	0.13	0.15	0.20	0.23	0.15	0.17

Figure 2. Fischer Assay Oil Yield Data for Experimental Tests 1-13

Material balances were performed for each simulation retort test. These data are listed in Table 5. The balances were calculated from the feed shale, products, and water used for retort quench and deluge studies. The amount of gas produced was determined by nitrogen balance and calculated from the average gas analysis for each test. The mass closures ranged between 96 to 104% with most of the tests in 98 to 102% range.

Deluge Simulation

The deluge procedure was simulated on cooled retorts. The retorts were allowed to cool overnight to ambient temperature prior to the introduction of water to the top of the retort. Tests dealing only with the deluge procedure (4 and 9) are listed in Table 3. There was no quench or reverse combustion applied during these tests. Test 4 was approximately 60% retorted, whereas test 9 was about 85%.

The water mixtures used for the deluge tests were different for each of these tests. Well water with nothing added was used during test 4. The water used for the first deluge during test 9 contained a biodegradable detergent (Igepal CA-720). This was followed by a well-water deluge.

The deluge simulation was monitored by analyzing the water that exited the retort for total organic carbon; the simulation was terminated when the TOC reach an asymptote. These data for all deluge simulation tests are presented in Appendix C. The organic concentration in the deluge water was plotted against pore volume, which is defined as that volume of water that equals one void volume of the reactor. This provides an easy nondimensional method for comparing one test to another.

Tube reactor test 4 simulates an in situ retort that has been allowed to cool and is cleaned by a deluge of water. The TOC concentrations in the water exiting the retort are depicted in Figure 3. The organic content is relatively high at the start of the deluge with a value near 550 mg/L. Within one pore volume, the value has decreased to about one-third of the original value. The TOC concentrations between the first and third pore volume show quite a bit of variation. Here, the TOC values tend to stabilize near a value of 50 mg/L. Because the TOC values reached an asymptote after four pore volumes of water had been run through the retort, a composite of the water samples accumulated to this point was made for further analysis. The analyses of the deluge water composite from test 4 provide a baseline for comparison with other remediation tests. These analyses are listed in Table 6.

The TOC concentration of the deluge composite sample is lower than we expected based on the grab sample analysis taken during the deluge procedure. The composite sample was an accumulation of all the water through the retort and not isolated grab samples. Further evaluation of Figure 3 and study of the data traced by the lower TOC values in the figure show that there is a rather smooth curve. This curve will approach the asymptote after the passage of about one pore volume of

Figure 3. Total Organic Carbon Concentrations from Tube Reactor Test 4

Figure 4. Total Organic Carbon Concentrations from Tube Reactor Test 9

Table 5. OXY Tube Test Material Balance

	Test Number												
	1	2	3	4	5	6	7	8	9	10	11	12	13
Loaded Retort													
Shale, g	4,917	4,894	4,910	5,005	4,978	4,915	4,945	4,968	4,980	4,990	5,081	5,120	5,031
Retorted, g													
Combusted Zone	1,957	1,163	2,087	2,651	3,350	3,513	3,511	3,717	3,428	3,510	3,584	3,613	3,433
Retorted Zone	416	843	728	859	420	420	473	608	688	668	481	522	696
Unretorted Zone	1,537	1,734	1,130	1,239	672	746	730	572	626	601	711	600	679
Sample Pouches	160	129	149	165	168	178	145	124	153	151	162	159	228
Total Shale	4,070	3,870	4,095	4,915	4,609	4,857	4,859	5,020	4,896	4,930	4,938	4,894	5,036
Oil and Water Produced, g													
Retorting	231	292	316	303	415	378	412	424	417	521	472	407	428
Reverse Burn		73	79		60		76					a	
Gas Produced, g													
Retorting	583	323	833	797	850	831	816	618	751	768	767	629	690
Reverse Burn		404	365		123		218					40	
Water In, g													
Quench				7,190	9,100	8,300				4,700	7,800	5,800	6,320
Deluge			18,620	18,752	13,000	9,400	12,400	15,300	23,160	9,930	9,690	9,200	5,460
Water Out, g													
Quench						5,560	7,610	7,590		4,690	6,480	5,490	5,980
Deluge			18,687	16,687	12,142	8,920	12,063	15,600	22,359	8,490	10,100	8,350	4,120
Mass Balance, %	99	101	104	96	101	96	99	102	101	99	101	98	97

a Reverse combustion and quench run at the same time. Oil was lost because the heat exchangers were unable to handle load.

Table 6. Deluge Water Analysis after Deluge Only

Test Number	T4
TOC, mg/L	50
TDS, mg/L	2410
Total Alkali, mg/L	830
Ammonia, mg/L	13
Phenols, μ g/L	910
Fluoride, mg/L	
Sulfate, mg/L	760
pH	
Conductivity, mS/cm	4.5
Aluminum, mg/L	36.2
Boron, mg/L	0.11
Cadmium, mg/L	<0.01
Magnesium, mg/L	0.278
Lead, mg/L	<0.004
Manganese, mg/L	<0.003
Calcium, mg/L	17.4
Molybdenum, mg/L	1.69
Zinc, mg/L	0.015
Arsenic, mg/L	0.033
Selenium, mg/L	0.03
Thallium, mg/L	<0.005
Sodium, mg/L	651
Silver, mg/L	<0.007
Barium, mg/L	0.125
Beryllium, mg/L	<0.002

water through the retort. The TOC values on this curve agree better with the TOC value from the composite water sample. The higher values measured during the deluge procedure were probably a result of excursions of oil and grease and are not representative of the bulk of the water exiting the retort.

The phenol concentration (910 μ g/L) accounts for only a small portion of the organic carbon present in the composite water. It is very likely that low molecular weight carboxylic acids were also present in this water; however, no analysis was done to determine if these acids were present. If present, these acids would be salts at the alkaline pH of the water (9.7) and reasonably soluble.

Other analyses performed on the composite water show elevated concentrations of sodium, aluminum, and calcium. The major anion present is sulfate. The carbonate and bicarbonate concentrations are represented by the total alkalinity of 830 mg/L. The total dissolved solids concentration of 2410 mg/L is a result of metals and carbonate, bicarbonate, and sulfate ions.

Tube reactor test 9 simulates an in situ retort that has been allowed to cool and is cleaned by a deluge of water that contains approximately 0.1 percent biodegradable detergent. The TOC concentrations of the water exiting the retort are depicted in Figure 4. The dashed horizontal line in Figure 4 represents the TOC contribution due to the detergent added to the well water.

The initial TOC value is greater than 1000 mg/L. This is about 200 mg/L greater than the detergent solution and appears to be slightly less than twice the TOC value for test 4 that used only well water. However, after the passage of one pore volume, the TOC concentration dropped below that of the detergent solution alone, suggesting that a portion of the detergent was being adsorbed by the spent shale. If detergent is being adsorbed, the 550-mg/L TOC detected after 2.5 pore volumes of detergent solution indicates the amount of detergent exiting the column. The initial values may have a retort-induced organic contribution very near the 550 mg/L range detected for test 4.

After the introduction of well water without detergent, the TOC concentration in the deluge water dropped fairly rapidly. The vertical line in Figure 4 shows the point at which the detergent solution was halted, and clean water was introduced into the retort. Within three pore volumes, the TOC level dropped to about 30 mg/L.

Quenching Simulation

Water quenching is a possible method of accelerating the cooling of the spent MIS retorts. The recovery of residual heat from spent retorts as well as gas generated through steam-char reaction was also investigated (Hester and Jacobson 1983).

The quenching procedure was started immediately after retorting stopped, except for test 11. The retort was still very hot with most temperatures greater than 1200°F (649°C). Tests dealing with the quenching procedure (6, 8, 10, 11, and 13) are listed in Table 3. There was no reverse combustion applied during these tests. However, the retorts were deluged with well water after the quenching procedure. All of these tests were retorted until about 85% of the oil had been removed.

The quenching and deluging procedures change from one test to another. The same quenching procedure of injecting water into the top of the retort was used for tests 6, 8, and 13. Part of the deluge water was recycled during test 8. In test 13, a fly ash sample was added to the well water prior to the deluge procedure. Tests 10 and 11 were quenched by injecting water into the combustion-front zone. In test 10, this was done by injecting water immediately after the retorting was completed. For test 11, the retort was allowed to cool to room temperature, and then steam was injected into the combustion-front zone.

The combination of quenching followed by deluging the retort was followed by TOC analysis of the water exiting the bottom of the retort. In tests 6 and 8, where the quench water was introduced to the top of the retort, most of the water was produced as steam that vented out the

top of the retort. Approximately one pore volume of water migrated through the retort before the conclusion of the quench procedure for these tests. The water quench was performed with the adiabatic shields of the tube reactor remaining on. After the retort temperatures dropped below that of steam, the deluge procedure was started.

Figures 5, 6, and 7 show the TOC concentrations versus time for tests 6, 8, and 13 during the quench and deluge procedure. These figures include all of the water that flowed through the retort either from the quench or deluge portion of the remediation. The changes from one procedure to another are designated by the vertical lines on the figures. All of the tests involving a hot quench demonstrate that most of the organic material was removed by the hot water during the quench procedure. By the time the retort temperature was low enough to start the deluge, most of the organic material had been removed from the retort. During the quenching, several grams of thick shale oil-water emulsion were observed in the water collection vessel.

The TOC analysis of the quench and deluge water during tests 10 and 11 are shown in Figures 8 and 9, respectively. The quench for both of these tests was performed by injection of water (test 10) and steam (test 11) at the combustion front. The data for test 10 are very similar to data for the other quenching experiments (tests 6, 8, and 13) with most of the organic material being removed after about one pore volume of water had passed through the retort.

The TOC data for test 11 is depicted in Figure 9. These data suggest that the temperature is important for removing organic components from the retort. The maximum TOC value was not reached until approximately one pore volume of water as steam had been injected into the retort. This was at the same time when most of the organic had been removed by the hot quench. The bottom portion of the retort had just reached steam temperature. The steam quench required two more pore volumes of water to approach the same point in organic removal as the hot quench did in one pore volume.

The analyses of the deluge water composite from tests 6, 8, 10, and 11 provide comparison between the cool deluge and the various quenching techniques. These analyses are given in Appendix D. For ease in comparing the remediation procedures, these data have been reduced to the total amount of material removed from each retort. This was done by taking into account the amount of water produced from the bottom of the retort during the quench and deluge procedures. These data are listed in Table 7. The deluge analysis data from test 4 (cool deluge) have also been converted to total amount removed and are listed in Table 7 for comparison.

The organic loading in the deluge water after quenching the retorts is significantly lower compared with cool deluge only (test 4). The TOC concentrations for the deluge after quench are one-fifth or less than TOC values during the cool deluge. A similar reduction can also be noted in the phenol concentration with the quenched retorts showing one-tenth or less than the cool-deluged retort. A corresponding increase in both the TOC and phenol concentrations can be noted in the quench water

Figure 5. Total Organic Carbon Concentrations from Tube Reactor Test 6

Figure 6. Total Organic Carbon Concentrations from Tube Reactor Test 8

Figure 7. Total Organic Carbon Concentrations from Tube Reactor Test 13

Figure 8. Total Organic Carbon Concentrations from Tube Reactor Test 10

Figure 9. Total Organic Carbon Concentrations from Tube Reactor Test 11

Table 7. Total Amount of Components Removed by Quench and Deluge

Test Number:	T4		T6		T8		T10		T11	
	Deluge		Deluge		Quench	Deluge	Quench	Deluge	Quench	Deluge
TOC, mg	403		70		1,276	24	698	48	544	40
TDS, mg	19,365		7,658		416	6,476	1,938	30,719	4,211	36,071
Total Alkali, mg	6,669		5,573		422	3,744	1,378	11,369	2,527	17,990
Ammonia, mg	104		8		130	6	134	4	21	35
Phenols, µg	7,312		675		6,394	64	2,584	188	972	511
Fluoride, mg	0		22		42	24	17	120	41	105
Sulfate, mg	6,107		5,990		48	1,413	233	8,809	635	10,045
pH			12.3		5.9	12.3	6.7	11	9	12.3
Conductivity, mS/cm	4.5		0.39		0		0.71			
Aluminum, mg	291				0		0			
Boron, mg	1		1		1	1	1	3	3	3
Cadmium, mg	0		0		0		0			
Magnesium, mg	2				1		64			
Lead, mg			0			0		0		
Manganese, mg	0				0		0			
Calcium, mg	140				5		32			
Molybdenum, mg	14		2		0	1.08	0	12	1	13
Zinc, mg	0				0		0			
Arsenic, mg	0		0		0	<0.005	0	0	0	0
Selenium, mg	0				0		0			
Thallium, mg	0				0		0			
Sodium, mg	5,231				40		469			
Silver, mg	0				0		0			
Barium, mg	1				0		0			
Beryllium, mg	0				0		0			

for tests 8, 10, and 11. The use of steam during the quench in test 11 was not as effective on reducing the organic carbon and phenol as the addition of water to the hot retorts (tests 8 and 10)

The amount of inorganic material present in the deluge waters after quenching varies considerably. The inorganic material, represented by total alkalinity and TDS of the deluge water collected after the hot quench from the top of the retort (test 6 and 8), is substantially lower compared with cool deluge (test 4) and the quenches at the combustion front (tests 10 and 11). The retorts quenched at the combustion front show much higher inorganic loading than the retort that received the cool deluge (test 4).

Reverse Combustion

Reverse combustion was examined as a method of organic source removal from the simulation retorts. After completing the retorting portion of the test, the air flow direction was reversed, and the combustion front was drawn to the bottom of the retort countercurrent to the air flow.

Reverse combustion was used in five of the simulation retorting tests (2, 3, 5, 7, and 12). Different combinations of quench and deluge procedures were performed in conjunction with the reverse-combustion tests. In test 2, only the reverse combustion was tested, with no water added for quench or deluge afterward. A deluge of the retort followed the reverse burn in tests 3 and 5. Both tests 2 and 3 were retorted until about 60% of the oil had been removed. Test 5 differed from test 3 in that about 85% of the shale had been retorted before reverse combustion was started. Test 7 was quenched after the reverse burn was completed, whereas the quench procedure was attempted simultaneously with the reverse combustion in test 12.

The combination of quenching and/or deluging following the reverse combustion of the retort was monitored by TOC analysis of the water exiting the bottom of the retort. In tests 7 and 12, the quench water was introduced into the top of the retort; most of the water vented out the top of the retort as steam. Approximately one pore volume of water migrated through the retort before the quench procedure ended in these tests. The water quench was performed with the adiabatic shields of the tube reactor remaining on. After the retort temperatures were below that of steam, the deluge procedure was started. Figures 10, 11, 12, and 13 show the TOC concentrations versus time for tests 3, 5, 7 and 12, respectively, during the quench and/or deluge procedures. These figures take into account all of the water that flowed through the retort either from the quench or deluge portion of the remediation. The changes from one procedure to another are designated by the vertical lines on the figures.

All of the tests involving reverse combustion demonstrate that most of the organic material was removed by the combustion prior to the quenching procedure. The organic loading of the quench and deluge water exiting the retort were considerably lower than the exit water in tests without reverse combustion. The very first TOC values measured during

Figure 10. Total Organic Carbon Concentrations from Tube Reactor Test 3

Figure 11. Total Organic Carbon Concentrations from Tube Reactor Test 5

Figure 12. Total Organic Carbon Concentrations from Tube Reactor Test 7

Figure 13. Total Organic Carbon Concentrations from Tube Reactor Test 12

the quench and/or deluge for these tests were the same or less than TOC values measured at the end of the deluge procedure for tests using only the quench and/or deluge procedures as method of retort remediation. Only test 12 showed a high TOC value during the first part of the quench suggesting that the reverse combustion was hampered by the simultaneous quench.

The analyses of the deluge water composite from tests 3, 5, 7, and 12 provide a means of comparison between reverse combustion and other methods of remediation previously mentioned. These analyses are listed in Appendix D. The analyses performed on composite quench samples from test 7 and 12 are listed in Appendix D. For ease in comparing the remediation procedures, these data have been reduced to the total amount of material removed from each retort as was done earlier for the quench and deluge comparisons. These data are listed in Table 8.

The analyses of the deluge water after quenching (tests 7 and 12) and the cool deluge (test 3 and 5) do not differ much in organic loading. The TOC values for all of these deluge waters are very similar, with the total amount of organic carbon produced during the deluge procedure about 40 mg. This value is quite similar to the TOC value of the deluge waters after quenching the retorts with water (test 6, 8, and 10).

Comparison of the analyses of the quench waters shows that very little organic material was left in the retort after reverse combustion. The TOC analyses of the quench waters (tests 8, 10, and 11) listed in Table 7 suggest that the amount of water-soluble organic material removed from the retorts was approximately 1000 ppm. The total organic material in the quench water after reverse combustion (tests 7 and 12) was an order of magnitude smaller than that without reverse combustion and was very near the detection limit of the analytical method. In addition, no oil was removed from the retort during the quench of the reverse combusted test as was the case for the other quench experiments.

A significant difference between the hot quenching and the reverse combustion is apparent. The reverse combustion converts most of the residual oil from retorting into carbon and lighter organic compounds. The carbon will remain in place and act as adsorbent, whereas the lighter organic compounds can be easily removed by a deluge with water. The hot quench removes most of the organic material by thermally reducing its viscosity and allows it to drain from the retort. This permits a certain portion of the heavier oil to adhere to the unretorted shale as an organic source. These organics can undergo slow chemical changes, for example, low-temperature oxidation, and be slowly leached from the retort by groundwater.

The amount of inorganic material present in the deluge waters after reverse combustion varies considerably, similar to the quenched retorts. The inorganic material, represented by total alkalinity and TDS of the deluge water collected after the hot quench (tests 7 and 12), is substantially greater than the amount in the cool deluge analyses (tests 3 and 5). The inorganic loading for tests 7 and 12 is closer to the loading in the deluge water after hot quenching (tests 6, 8 and 10).

Table 8. Total Amount of Components Removed by Quench and Deluge after Reverse Combustion

Test Number:	T3	T5	T7		T12	
	Deluge	Deluge	Quench	Deluge	Quench	Deluge
TOC, mg	61	13	41	42	40	57
TDS, mg	4,828	13,187	163	25,463	405	20,220
Total Alkalinity, mg	2,431	11,790	229	13,580	446	12,088
Ammonia, mg	0	3	106	5	140	8
Phenols, μ g	0	480	1,136	250	1,155	469
Fluoride, mg	0	0	17	100	1	81
Sulfate, mg	1,174	699	75	4,493	57	256
pH			5.3	12	5.9	11.5
Conductivity, mS/cm	0.96	3.2	0.42			
Aluminum, mg	62	1,214	0			
Boron, mg	1	2	0	4	0	1
Cadmium, mg	0	0	0			
Magnesium, mg	7	1	1			
Lead, mg	0	0	0			
Manganese, mg	0	0	0			
Calcium, mg	30	90	7			
Molybdenum, mg	9	35	0	0	0	0
Zinc, mg	0	0	0			
Arsenic, mg	0	1	0	0	0	0
Selenium, mg	0	0	0			
Thallium, mg	0	0	0			
Sodium, mg	1,660	4,113	5			
Silver, mg	0	0	0			
Barium, mg	0	2	0			
Beryllium, mg	0	0	0			

The water quench while the retorts are at high temperatures is probably removing part, if not all, of the carbon layer deposited on the spent shale during the reverse combustion. The hot quench promotes steam-char reactions that produce hydrogen and carbon oxides. This carbon layer can prevent the deluge water from contacting the spent shale and dissolving inorganic components. Evidence for the carbon layer isolating the spent shale from the deluge water can be noted by comparing tests 3 and 5. Neither test was subjected to the water quench, but test 3 shows much less inorganic material present in the deluge water. More unretorted shale (approximately 30 wt % of the retort) was involved in the reverse burn during test 3, depositing more carbon on the spent shale above. Heavier carbon deposits were seen when retort tests 2 and 3 were unloaded.

Retort Zone Leachate Analysis

Analyses of the quench and deluge water samples show approximately how much organic and inorganic material is removed from the spent retort during the remediation procedures. The leaching of the various retort

zones provides information that can be used to determine what material is left behind and what may later be removed from the retort by groundwater.

The first simulation test was to determine baseline data for post-retorting operation not involving quenching or deluge. The objective of this experiment was intended to provide basic physical data regarding the properties of the spent oil shale and the behavior of the spent retorts. In addition, this test permitted the evaluation of the analytical methods for comparing remediation procedures. Test 1 was retorted until approximately 60% of the shale had been retorted and then stopped. The retort was closed in and allowed to cool to ambient temperature. No remediation procedures were attempted on this test.

Samples of the retort zones were leached with deionized water at a 1:20 shale-to-water ratio to decrease the time necessary to reach solubility equilibrium. Working in areas of low solute concentration helps drive components with marginal water solubilities into solution.

The analytical data for the leachates from the three retort zones in test 1 are listed in Table 9. Several differences can be noted between the three retort zones. The organic compounds are concentrated at the bottom of the retort on the shale in the unretorted zone; this is suggested by the higher TOC. The phenol concentration was higher for test 1 than any of the leached retort zone samples from other tests (320 $\mu\text{g/L}$). There was very little evidence of organic components present in either the combusted or the retorted zones.

Inorganic components are in higher concentration in the thermally affected zones (combusted and retorted) than in the unretorted section of the retort. The sulfate, TDS, and total alkalinity are all elevated in the combusted zone compared with the other zones. The TDS and total alkalinity values from test 1 are low compared with combusted zones from other retort tests. There is no obvious reason for these low values.

Boron and molybdenum also appear to be affected in the thermally altered zones. Boron concentration is lower in the combusted zone and higher in the retorted zone compared with the concentration in the unretorted shale. Molybdenum concentrations are higher in both of the thermally affected zones compared with the unretorted shale. In all zones, arsenic is very close to the detection limit, and concentration changes from one zone to another are difficult to assess.

The analytical data (Table 10) for the leachate samples derived from the deluge-only retort tests (4 and 9) suggest that a good share of the organic material remains in the unretorted zone. The cool deluge did remove some of the organic material from the retort, as suggested by the TOC analysis of the deluge solution exiting the retort. However, compared with TOC values from test 1 (no remediation), the TOC values for the leachates suggest that a substantial portion of the organic components remains on the unretorted zones of test 4 and 9. The phenol concentrations for the unretorted zones are lower in test 1 than concentrations for both tests 4 and 9. The detergent deluge (test 9) removed substantially more phenol than the water deluge alone.

Table 9. Leachate Water Analysis after No Remediation

Test Number:	T1		
Retort Zone:	Combusted	Retort	Unretort
TOC, mg/L	2	0	28
TDS, mg/L	202	70	75
Total Alkalinity, mg/L	540	99	94
Ammonia, mg/L	0.49	0.26	1.9
Phenols, µg/l			320
Fluoride, mg/L	7.7	13	
Sulfate, mg/L	160	35	12
pH			
Conductivity, mS/cm			
Aluminum, mg/L			0.689
Boron, mg/L	<0.02	1.19	0.391
Cadmium, mg/L			<0.01
Magnesium, mg/L			3.22
Lead, mg/L			<0.004
Manganese, mg/L			<0.003
Calcium, mg/L			8.38
Molybdenum, mg/L	0.672	0.329	0.054
Zinc, mg/L			<0.003
Arsenic, mg/L	<0.005	0.008	0.009
Selenium, mg/L			<0.005
Thallium, mg/L			<0.005
Sodium, mg/L			41.8
Silver, mg/L			<0.007
Barium, mg/L			0.05
Beryllium, mg/L			<0.002

Further evaluation of the TOC data for test 9 leachates provides additional evidence that the detergent was adsorbed by the spent shale during the deluge procedure. The TOC value for the combusted zone of test 9 is much higher than TOC for any of the leached zones. This is probably a result of detergent adsorption. It is also interesting to note that only shale in the combusted zone appears to have adsorbed the detergent. The retorted and unretorted zones are quite similar to those of the water deluged retort (test 4) and do not seem to have adsorbed a great deal of detergent.

The total alkalinity and TDS values are an order of magnitude greater than those measured in the leachate from the test with no remediation (test 1). Although the absolute values are higher for both of these zones, there is still an order of magnitude difference between the two zones. One would expect less soluble material in the retorted zone because of the lower temperatures and the carbon deposit.

Table 10. Leachate Water Analysis after Deluge Only

Test Number:	T4			T9		
Retort Zone:	Combusted	Retort	Unretort	Combusted	Retort	Unretort
TOC, mg/L	1.1	5.2	26.1	68.2	4.7	21.4
TDS, mg/L	1250	310	85	1820	448	142
Total Alkalinity, mg/L		1450	200	49	2370	320 89
Ammonia, mg/L	0.4	0.5	1.7	<0.2	<0.2	<0.2
Phenols, µg/l			230			110
Fluoride, mg/L	4.4	3.8	1.3	5.3	13	0.7
Sulfate, mg/L	60	43	11	32	71	8.3
pH	12.3	10	5.9	11.5	11	
Conductivity, mS/cm						
Aluminum, mg/L						
Boron, mg/L	0.049	0.096	0.593	0.035	0.493	0.191
Cadmium, mg/L						
Magnesium, mg/L						
Lead, mg/L						
Manganese, mg/L						
Calcium, mg/L						
Molybdenum, mg/L	0.098	0.122	0.048	0.109	0.145	0.034
Zinc, mg/L						
Arsenic, mg/L	<0.005	<0.005	0.01	<0.005	<0.005	0.008
Selenium, mg/L						
Thallium, mg/L						
Sodium, mg/L						
Silver, mg/L						
Barium, mg/L						
Beryllium, mg/L						

Both the plain water and the detergent deluge removed a substantial portion of the sulfate anion. Comparison of the leachate data after deluge remediation with that with no remediation (test 1) suggests that approximately two-thirds of the sulfate was removed from the combustion zones that had undergone deluge. The retorted and unretorted zones did not show much change in the sulfate data when compared with test 1.

The analytical data (Table 11) for the leachate samples derived from the quenched retort tests (6, 8, 10, and 13) suggest that most of the organic material was removed from the unretorted zone. Visual inspection of the unretorted zones suggests that some oil is still present on the shale. However, this residual oil is not very miscible with water and will not be leached.

Table 11. Leachate Water Analysis after Quench and Deluge

Test Number Retort Zone	T6			T8			T13		
	Combusted	Retort	Unretort	Combusted	Retort	Unretort	Combusted	Retort	Unretort
TOC, mg/L	0	0	0	0	0	0			
TDS, mg/L	1050	289	78	964	462	124	940	460	190
Total Alkalinity, mg/L	950	210	42	850	380	60	930	320	85
Ammonia, mg/L	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Phenols, µg/l			<20						
Fluoride, mg/L	2.9	2.7	1.3	4	5.5	2.1	3	4.3	4.4
Sulfate, mg/L	120	14	4.6	190	46	11	110	120	62
pH	11.5	10.5	5.3	11.5	10.5	5.3			
Boron, mg/L	0.292	0.543	0.172	0.364	0.507	0.14	0.11	0.3	0.182
Molybdenum, mg/L	0.461	0.18	0.092	0.246	0.213	0.058	0.272	0.258	0.177
Arsenic, mg/L	<0.005	<0.005	0.025	<0.005	<0.005	0.015	<0.005	<0.005	0.017

Table 11. Leachate Water Analysis after Quench and Deluge (Continued)

Test Number: Retort Zone:	T10			T11		
	Combusted	Retort	Unretort	Combusted	Retort	Unretort
TOC, mg/L	0.7	0	3.2	3.6	0.5	4
TDS, mg/L	1360	267	65	990	280	76
Total Alkalinity, mg/L	1530	170	28	810	160	41
Ammonia, mg/L	<0.2	<0.2	<0.2	0.24	0.41	<0.2
Phenols, µg/l			<20			<20
Fluoride, mg/L	4.5	2.9	1.6	5.1	8.2	0.8
Sulfate, mg/L	120	25	4.6	200	28	11
pH	11.5	8.4	4.8	11.8	9.5	5.3
Boron, mg/L	0.117	0.372	<0.020	0.507	0.5	0.267
Molybdenum, mg/L	0.098	0.199	0.034	0.132	0.242	0.082
Arsenic, mg/L	<0.005	<0.005	0.005	<0.005	0.007	0.02

The TOC data for the water quench injected at the combustion front 28 suggest that some of the organic material remained on the shale. The removal is still greatly enhanced over the cool deluge (test 4) and the detergent deluge (test 9). The data for the steam injection at the combustion front suggest that about the same amount of organic material was removed from the unretorted shale as for test 10. However, the data for the combusted and retorted leachates suggest that a portion of the oil may have been steam distilled and moved up the retort to be deposited on the retorted and combusted shale. The deposited organic compounds apparently cannot be easily removed by a deluge of water.

The amount of inorganic material leached from the three retort zones of the quenched retorts is not much different than the cool deluge data (tests 4 and 9). The total alkalinity and TDS values are generally a little lower for the quenched retort zones but remain near 1000 mg/L. The sulfate data show a much wider relative variation but no real trends with respect to removal. As indicated in data from test 1 (no remediation), the inorganic material is only present in the thermally altered zones.

The analytical data (Table 12) for the leachate samples derived from the reverse combusted retort tests (2, 3, 5, 7, and 12) suggest that most of the organic material was removed from what would have been the unretorted zone in the other tests. Visual inspection of the reverse burned zones showed that no oil was present on the shale. However, the TOC value for the combusted zone in test 2 (no quench or deluge) suggests that some of the organic material condensed on this zone during the reverse combustion. The data for the same zone for tests 3 and 5 (cool deluge) suggest that the condensed organic material is easily removed by water. The organic compounds probably consisted of low molecular weight material generated by cracking during the reverse burn.

The amount of inorganic material leached from the three retort zones of the reverse-burned retorts is slightly less than the amount of inorganic material in the quenched retorts (tests 6, 8, 10, 11, and 13) and the cool-deluge retorts (tests 4 and 9). The total alkalinity and TDS values are generally less than the values in the quenched retort combustion zones (~1000 mg/L). The data for the reverse-burned zone approaches the values measured in the retorted zones in nearly all the tests.

The sulfate data for the reverse combustion tests are quite different in distribution compared with the other tests. The highest sulfate values occur in the reversed combusted zone, whereas the lowest values are in the combusted zone. The data for the combusted zones suggest that a large amount of the sulfate was removed from this zone during the reverse combustion, probably as ammonium sulfate. Further examination of the sulfate data for the combusted zones suggests that the quenching procedure in tests 7 and 12 may have made more sulfate available by thermally fracturing the spent shale.

Boron and molybdenum are also more abundant in the thermally altered, reverse-combusted zone. In this zone, boron was present in higher concentration than in the retorted zone, which was the highest

Table 12. Leachate Water Analysis after Reverse Combustion

Test Number: Retort Zone:	T2			T3			T5		
	Combusted	Retort	Reverse B	Combusted	Retort	Reverse B	Combusted	Retort	Reverse B
TOC, mg/L	19.5	0	1.2	1	2	1.5	0	0	0.3
TDS, mg/L	834	233	176	730	520	440	834	547	503
Total Alkalinity, mg/L	440	370	80	700	370	25	810	253	246
Ammonia, mg/L	<0.2	0.33	0.54	0.2	<0.2	0.3	<0.2	<0.2	0.2
Phenols, µg/l									<20
Fluoride, mg/L	21	10	14	4.6	5.7	4.3	4.6	15	5.6
Sulfate, mg/L	16	36	120	19	24	240	10	81	180
pH							11	10	8
Boron, mg/L	0.141	0.868	1.4	0.119	0.196	0.549	0.099	0.558	0.909
Molybdenum, mg/L	0.453	0.229	0.218	0.267	0.161	0.177	0.157	0.302	0.171
Zinc, mg/L									
Arsenic, mg/L	0.015	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.008	0.007

Table 12. Leachate Water Analysis after Reverse Combustion (Continued)

Test Number	T7			T12		
	Retort Zone	Combusted	Retort Reverse B	Combusted	Retort Reverse B	Reverse B
TOC, mg/L		0.1	1.1	1.2	0	0
TDS, mg/L		1010	337	411	780	550
Total Alkalinity, mg/L		950	100	130	680	180
Ammonia, mg/L		<0.2	<0.2	<0.2	0.42	0.39
Phenols, µg/l				<20		
Fluoride, mg/L		3.8	4.1	8.1	4.2	7
Sulfate, mg/L		68	73	130	58	140
pH		11	8.4	8	11	9.7
Boron, mg/L		0.239	1.85	2.52	0.133	1.52
Molybdenum, mg/L		0.502	0.307	0.251	0.372	0.328
Arsenic, mg/L		<0.005	<0.005	0.006	<0.005	<0.005

measured value in other retorts. Molybdenum is present in higher concentrations in all of the thermally affected zones compared with the unretorted shale. Arsenic concentrations are very close to the detection limit and show no differences from the previous retort tests.

Shale Rehydration

Rehydration of spent shale can greatly affect the amount of water needed in the remediation of MIS retorts. Once the shale has been thermally altered, a great deal of surface area has been made available for contact with water during the remediation process. For this reason, the rehydration characteristics of spent shale become significant when trying to minimize water use.

The thermally affected zones of a retort greatly increase their affinity for water. Fischer assay water yield data from the simulation retort tests (Figure 14) show that a large amount of water can be adsorbed by the spent shale in place. The combusted zones, which make up the majority of the shale in the retort, contain between 25 and 30 wt % water. The retorted zones generally contain between 7 to 12 wt % water. The unretorted shale only contains 2 to 4 wt % water after rehydration.

To examine the effect of temperature on shale uptake of water, sample pouches were positioned at the center thermocouples throughout the retort. The thermocouples provided a thermal history of the shale in the pouch. The amount of water that was readsorbed with respect to maximum temperature is depicted in Figure 15. The water adsorption data in this figure represents the maximum amount of water that can be taken up by the shale. The shale and the water were mixed for 24 hours, and the agitation probably broke down the friable spent shale in the combusted zone. Experimental tests 2 and 3 stand out because they show that very little water was readsorbed even under the severe test conditions. Both of these tests were reverse burned after about 60% of the shale had been retorted.

Visual inspection of the combusted zones for tests 2 and 3 showed a heavy carbon deposit. This carbon deposit may have acted as a barrier to the water and prevented it from contacting the spent shale. Tests 5, 7, and 12 were 85 to 90% retorted before reverse combustion was performed. These tests did not show as heavy of a deposit of carbon on the combusted zone as did tests 2 and 3. As a result of the agitation, the shale in tests with less carbon may have been broken down and more uncoated surface exposed to water.

To evaluate the effect of water readsorption by the spent shale in place, the Fischer assay data are presented as a comparison of the total amount of water adsorbed by the retort with respect to the percent of the retort which had been thermally affected. The amount of shale thermally affected was determined by the amount of oil remaining in the retort after the simulation test. This is shown in Figure 16. A fair correlation can be made for those retort tests that did not involve any reverse combustion (tests 4, 6, 8, 9, 10, 11, and 13).

Figure 14. Fischer Assay Water Yield Data for Experimental Tests 1-13

Figure 15. Water Adsorption Versus Shale Temperature of Sample Pouches

Figure 16. Water Adsorption Versus Amount of Shale Thermally Altered

Tests 3, 5, and 12 deviate from the line in Figure 16. All of these tests were reverse combusted after retorting had concluded. The amount of water readsorption in tests 5 and 12, compared with the nonreverse-combusted samples, is much less than in the sample pouch data. This may occur because the carbon layer was not destroyed by mechanical agitation, as it appeared to be for the sample pouches. These data suggest that the smaller carbon deposit still retards water readsorption to some extent.

Reverse combustion appears to prevent the readsorption of water by the spent shale; however, the effectiveness of this method varies with each test. We examined the conditions for each of these tests to explain some of the differences in the results. Test 3 was only 60% retorted before the air flow was reversed and the remaining shale reverse combusted. After the retort was unloaded, visual inspection of the combusted shale showed a heavy deposit of carbon. Tests 5, 7, and 12 were reverse burned after approximately 85% of the shale had been retorted. Less water was readsorbed in tests 5 and 12, but in test 7 readsorption is about the same as retorts without reverse combustion.

The higher water adsorption for test 7 is difficult to explain based on the argument that even a small carbon deposit can reduce the water uptake of the spent shale. Both tests 7 and 12 were quenched by water after or during the reverse combustion stage of the experiment. If the carbon was stripped from the spent shale by steam-char reaction during the quench, then water uptake for both tests should very closely resemble water uptake in the nonreverse combustion tests. Test 12 was water quenched during the reverse burn, which may have prevented steam-char reactions from taking place.

The difference between tests 7 and 12 can be explained by examining the gas data obtained during the quench portions of these tests. These data are listed in Table 13. The average gas analysis for test 7 shows a large amount of hydrogen. This indicates that steam-char reaction is prevalent. This reaction removes the carbon from the spent shale. The gas analysis for test 12 does not show a high concentration of hydrogen and, thus, does not suggest a steam-char reaction. The presence of the above methane hydrocarbons and lower hydrogen concentration is more like the gas produced during the presence of oil during the reverse combustion. This means that less of the carbon deposit was affected by the steam char reaction in test 12, thus, reducing the amount of water that could contact the spent shale.

Table 13. Average Gas Analysis for Retort Quench

Experiment Number:	7	12
Hydrogen	21.45	8.58
Nitrogen	53.28	71.40
Oxygen	9.61	0.10
Argon	0.65	0.70
Carbon Monoxide	0.17	2.84
Carbon Dioxide	14.16	12.85
Methane	0.15	0.98
Ethane	0.00	0.19
Ethylene	0.07	1.01
Propane	0.00	0.00
Propylene	0.00	1.31
Butanes	0.00	0.04
Hydrogen Sulfide	0.46	0.02

CONCLUSIONS

Reverse combustion is the most effective method for removing organic material on the unretorted shale at the bottom of the retort. Hot quench and deluge after the reverse combustion removed very little additional organic material. Visual inspection of the bottom portion of the retort showed no evidence of organic components after reverse combustion.

Hot quench was effective in removing most of the organic material near the bottom of the retort. Although the deluge water analyzed after the hot quench suggested that this method was as effective as the reverse combustion, visual examination of the unretorted zone showed that heavy oil was still present. This heavy oil may act as a source of organic contaminants if it undergoes chemical changes that increase its water solubility.

Cool deluge was not as effective as reverse combustion or quench in removing organic material. Water used as a cool deluge removed the least amount of organic material from the unretorted portion of the reactor. The addition of a biodegradable detergent to the cool deluge water was slightly better than using plain water, but it still left a lot of organic material on the unretorted shale.

Reverse combustion reduced water readsorption by spent shale. A carbon layer deposited on the spent shale as a result of reverse combustion provided a barrier to water and reduced the water uptake by the spent shale substantially. A hot quench after reverse combustion removed part of the carbon deposit. The removal of the carbon layer was a result of steam-char reaction taking place during the quench.

The amount of inorganic material leached from the shale was slightly less in the reverse combusted experiments than in other tests. This is probably related to the water barrier provided by the carbon deposit from the reverse combustion.

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DISCLAIMER

Mention of specific brand names of models of equipment is for information only and does not imply endorsement.

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APPENDIX A
Retorting Gas Analysis

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Table A-1. OXY-T1-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	0.97	% C	=	13.28	N ₂ +Ar (In), g	=	1223				
Nitrogen	64.46	% H	=	0.46	Gas (Out), g	=	2175				
Oxygen	0.24	% O	=	30.005	Air (In), g	=	1592				
Argon	0.73	% N	=	55.32	Gas Produced, g	=	583				
Carbon Monoxide	2.08	% Ar	=	0.89							
Carbon Dioxide	29.36	Total:		100.00							
Methane	0.96										
Ethane	0.28										
Ethylene	0.09										
Propane	0.28										
Propylene	0.27										
Butanes	0.13										
Hydrogen Sulfide	0.16										
		Average MW = 32.6									
		Flow = 0.09 scfm									
		Exp Len = 8.5 hours									
Data Set											
1	2	3	4	5	6	7	8	9	10		
Hydrogen	1.78	0.00	1.35	3.11	0.00	0.00	0.00	1.74	0.00	1.74	
Nitrogen	74.25	73.21	69.70	55.44	62.05	72.08	72.31	51.76	53.79	59.98	
Oxygen	0.94	1.10	0.34	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Argon	0.92	0.91	0.78	0.60	0.68	0.82	0.82	0.55	0.59	0.61	
Carbon Monoxide	1.70	1.50	1.72	3.72	3.86	1.46	1.19	2.23	1.60	1.80	
Carbon Dioxide	17.43	21.21	23.68	34.24	32.01	24.96	23.33	41.26	42.33	33.16	
Methane	1.31	1.00	1.17	1.30	0.52	0.34	1.07	1.03	0.65	1.18	
Ethane	0.41	0.28	0.33	0.34	0.16	0.09	0.32	0.29	0.20	0.35	
Ethylene	0.38	0.06	0.07	0.07	0.03	0.02	0.07	0.13	0.04	0.08	
Propane	0.35	0.29	0.31	0.31	0.21	0.16	0.30	0.28	0.24	0.31	
Propylene	0.34	0.30	0.31	0.33	0.24	0.00	0.31	0.32	0.26	0.32	
Butanes	0.19	0.14	0.18	0.19	0.03	0.00	0.15	0.12	0.08	0.18	
Hydrogen Sulfide	0.00	0.00	0.06	0.35	0.2	0.07	0.12	0.28	0.23	0.3	

Table A-2. OXY-T2-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
1	2	3	4	5	6	7	8	9	10		
Hydrogen	1.39	1.17	1.35							N ₂ +AR (In), g	= 935
Nitrogen	70.58	66.51	67.25							Gas (Out), g	= 1541
Oxygen	0.00	0.00	0.13							Air (In), g	= 1218
Argon	0.81	0.76	0.75							Gas Produced, g	= 323
Carbon Monoxide	1.57	2.12	1.60								
Carbon Dioxide	23.46	27.43	25.96								
Methane	1.04	0.89	1.07								
Ethane	0.29	0.24	0.32								
Ethylene	0.05	0.05	0.07								
Propane	0.32	0.28	0.83								
Propylene	0.31	0.30	0.44								
Butanes	0.14	0.13	0.22								
Hydrogen Sulfide	0.03	0.11	0.00								

% C	=	12.19									
% H	=	0.52									
% O	=	26.60									
% N	=	59.72									
% Ar	=	59.72									
Total:		100.00									
Average MW = 31.9											
Flow = 0.09 scfm											
Exp Len = 6.5 hours											

Table A-3. OXY-T3-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %										
Hydrogen	2.11	% C	=	15.21	N ₂ +Ar (In), g	=	1115					
Nitrogen	56.85	% H	=	0.44	Gas (Out), g	=	2285					
Oxygen	0.00	% O	=	35.55	Air (In), g	=	1452					
Argon	0.66	% N	=	48.00	Gas Produced, g	=	833					
Carbon Monoxide	3.36	% N	=	0.79								
Carbon Dioxide	35.17											
Methane	1.06	Total:	100.00									
Ethane	0.29											
Ethylene	0.06											
Propane	0.12	Average MW = 33.2										
Propylene	0.08	Flow = 0.09 scfm										
Butanes	0.03	Exp Len = 7.75 hours										
Hydrogen Sulfide	0.20											
		Data Set										
	1	2	3	4	5	6	7	8	9	10		
Hydrogen	1.99	0.52	2.36	1.73	2.79	3.29						
Nitrogen	68.22	68.23	61.25	51.38	46.11	45.93						
Oxygen	0.00	0.00	0.00	0.00	0.00	0.00						
Argon	0.80	0.80	0.74	0.58	0.53	0.52						
Carbon Monoxide	1.71	1.99	2.02	4.61	3.95	5.90						
Carbon Dioxide	25.71	27.21	31.12	40.29	44.26	42.39						
Methane	0.99	0.83	1.44	0.79	1.22	1.11						
Ethane	0.27	0.22	0.41	0.21	0.36	0.30						
Ethylene	0.05	0.04	0.09	0.04	0.07	0.06						
Propane	0.13	0.09	0.17	0.09	0.15	0.09						
Propylene	0.10	0.06	0.11	0.06	0.10	0.08						
Butanes	0.00	0.00	0.14	0.00	0.06	0.00						
Hydrogen Sulfide	0.03	0.02	0.17	0.23	0.41	0.35						

Table A-4. OXY-T4-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	1.17	% C	= 14.23	N ₂ +Ar (In), g	= 1115						
Nitrogen	60.58	% H	= 0.38	Gas (Out), g	= 2285						
Oxygen	0.08	% O	= 33.24	Air (In), g	= 1452						
Argon	0.69	% N	= 51.32	Gas Produced, g	= 833						
Carbon Monoxide	2.73	% Ar	= 0.83								
Carbon Dioxide	32.89	Total:	100.00								
Methane	0.93										
Ethane	0.23										
Ethylene	0.09										
Propane	0.24	Average MW	= 33.1								
Propylene	0.24	Flow	= 0.09 scfm								
Butanes	0.12	Exp Len	= 9 hours								
Hydrogen Sulfide	0.02										

		Data Set									
		1	2	3	4	5	6	7	8	9	10
Hydrogen	0.98	1.99	1.73	0.74	1.24	0.74	0.74	0.74			
Nitrogen	71.10	57.54	49.45	62.94	52.15	63.67	67.22	67.22			
Oxygen	0.44	0.00	0.00	0.00	0.00	0.00	0.12	0.12			
Argon	0.82	0.66	0.58	0.65	0.65	0.72	0.75	0.75			
Carbon Monoxide	1.60	2.73	6.69	2.37	2.57	1.76	1.37	1.37			
Carbon Dioxide	23.53	34.47	40.07	31.98	41.34	31.52	27.33	27.33			
Methane	0.95	1.61	0.67	0.56	0.98	0.57	1.16	1.16			
Ethane	0.25	0.42	0.18	0.16	0.27	0.16	0.13	0.13			
Ethylene	0.04	0.08	0.04	0.03	0.05	0.16	0.20	0.20			
Propane	0.08	0.14	0.28	0.25	0.31	0.31	0.33	0.33			
Propylene	0.07	0.13	0.25	0.24	0.29	0.34	0.33	0.33			
Butanes	0.13	0.23	0.07	0.07	0.13	0.07	0.17	0.17			
Hydrogen Sulfide	0.00	0.00	0.00	0.00	0.00	0.00	0.15	0.15			

Table A-5. OXY-T5-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %							
Hydrogen	1.41	% C	=	14.39	N ₂ +Ar (In), g	=	1396		
Nitrogen	60.66	% H	=	0.46	Gas (Out), g	=	2668		
Oxygen	0.10	% O	=	32.84	Air (In), g	=	1817		
Argon	0.68	% N	=	51.49	Gas Produced, g	=	851		
Carbon Monoxide	2.75	% Ar	=	0.82					
Carbon Dioxide	32.37								
Methane	0.91	Total:		100.00					
Ethane	0.25								
Ethylene	0.07								
Propane	0.32	Average MW	=	33.0					
Propylene	0.19	Flow	=	0.09 scfm					
Butanes	0.11	Exp Len	=	9.7 hours					
Hydrogen Sulfide	0.19								
		Data Set							
1	2	3	4	5	6	7	8	9	10
Hydrogen	1.90	0.76	0.51	1.68	1.00	2.81	1.56	0.76	0.51
Nitrogen	73.72	70.14	68.60	50.99	60.18	49.24	58.17	53.29	69.07
Oxygen	0.52	0.33	0.00	0.00	0.00	0.00	0.00	0.00	0.20
Argon	0.85	0.80	0.78	0.55	0.68	0.52	0.65	0.58	0.77
Carbon Monoxide	1.64	1.69	1.87	5.08	2.30	3.89	2.67	3.96	1.38
Carbon Dioxide	19.33	24.09	26.80	40.24	33.13	41.33	34.38	39.99	26.31
Methane	1.22	1.02	0.84	0.78	0.69	1.17	0.65	0.56	0.77
Ethane	0.35	0.28	0.22	0.21	0.20	0.34	0.19	0.16	0.23
Ethylene	0.07	0.06	0.03	0.03	0.23	0.06	0.12	0.03	0.05
Propane	0.14	0.29	0.09	0.09	0.88	0.14	0.92	0.21	0.25
Propylene	0.10	0.30	0.06	0.06	0.34	0.08	0.32	0.23	0.28
Butanes	0.18	0.16	0.11	0.10	0.13	0.00	0.10	0.02	0.06
Hydrogen Sulfide	0.00	0.08	0.09	0.21	0.25	0.41	0.27	0.22	0.13

Table A-6. OXY-T6-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	0.91	% C	= 13.48	N ₂ +Ar (In), g	= 1583						
Nitrogen	63.13	% H	= 0.40	Gas (Out), g	= 2892						
Oxygen	0.41	% O	= 31.40	Air (In), g	= 2061						
Argon	0.74	% N	= 53.83	Gas Produced, g	= 831						
Carbon Monoxide	2.17	% Ar	= 0.89								
Carbon Dioxide	30.73	Total:	100.00								
Methane	0.93										
Ethane	0.25										
Ethylene	0.04										
Propane	0.21	Average MW = 32.8									
Propylene	0.20	Flow = 0.09 scfm									
Butanes	0.11	Exp Len = 11 hours									
Hydrogen Sulfide	0.16										

		Data Set									
	1	2	3	4	5	6	7	8	9	10	
Hydrogen	0.51	1.23	0.25	1.13	2.15	0.25	1.27	0.50			
Nitrogen	73.62	69.84	69.95	69.73	52.09	60.57	55.66	53.60			
Oxygen	0.90	0.81	0.70	0.89	0.00	0.00	0.00	0.00			
Argon	0.91	0.88	0.79	0.87	0.55	0.69	0.61	0.59			
Carbon Monoxide	1.44	1.71	1.64	1.75	2.84	3.13	2.09	2.77			
Carbon Dioxide	20.92	23.54	24.58	23.41	39.76	34.46	38.40	40.77			
Methane	1.05	0.96	0.91	1.00	1.21	0.54	1.01	0.77			
Ethane	0.28	0.27	0.26	0.28	0.33	0.06	0.28	0.21			
Ethylene	0.04	0.04	0.04	0.03	0.07	0.04	0.04	0.04			
Propane	0.12	0.28	0.28	0.32	0.31	0.05	0.12	0.25			
Propylene	0.08	0.29	0.29	0.38	0.32	0.03	0.08	0.14			
Butanes	0.13	0.07	0.17	0.09	0.10	0.03	0.16	0.11			
Hydrogen Sulfide	0.00	0.07	0.14	0.10	0.28	0.15	0.29	0.25			

Table A-7. OXY-T7-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	1.20	% C	= 13.78	N ₂ +Ar (In), g	= 1511						
Nitrogen	62.61	% H	= 0.44	Gas (Out), g	= 2783						
Oxygen	0.06	% O	= 31.50	Air (In), g	= 1967						
Argon	0.71	% N	= 53.42	Gas Produced, g	= 816						
Carbon Monoxide	2.45	% Ar	= 0.86								
Carbon Dioxide	31.02	Total:	100.00								
Methane	0.93										
Ethane	0.27										
Ethylene	0.06										
Propane	0.22	Average MW	= 32.8								
Propylene	0.14	Flow	= 0.09 scfm								
Butanes	0.14	Exp Len	= 10.5 hours								
Hydrogen Sulfide	0.20										

		Data Set									
		1	2	3	4	5	6	7	8	9	10
Hydrogen	2.49	1.65	0.75	1.07	0.50	0.50	2.11	0.50	0.50		
Nitrogen	73.67	69.23	67.91	56.61	63.84	56.96	57.55	57.55	55.15		
Oxygen	0.47	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
Argon	0.90	0.78	0.78	0.62	0.72	0.62	0.62	0.63	0.60		
Carbon Monoxide	1.95	1.72	1.80	3.94	1.75	2.44	3.15	3.15	2.82		
Carbon Dioxide	17.46	24.77	26.74	36.41	30.78	35.59	36.76	36.76	39.63		
Methane	1.68	1.06	0.95	0.65	0.67	1.12	0.68	0.68	0.64		
Ethane	0.54	0.28	0.25	0.19	0.19	0.33	0.20	0.20	0.17		
Ethylene	0.12	0.04	0.05	0.04	0.09	0.05	0.04	0.04	0.04		
Propane	0.25	0.12	0.25	0.07	0.80	0.14	0.08	0.08	0.07		
Propylene	0.17	0.08	0.27	0.05	0.31	0.10	0.06	0.06	0.05		
Butanes	0.30	0.16	0.12	0.06	0.11	0.18	0.10	0.10	0.06		
Hydrogen Sulfide	0.00	0.10	0.14	0.27	0.23	0.36	0.24	0.24	0.28		

Table A-9. OXY-T9-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	1.15	% C	= 13.70	N ₂ +Ar (In), g		= 1439					
Nitrogen	63.00	% H	= 0.49	Gas (Out), g		= 2624					
Oxygen	0.39	% O	= 30.97	Air (In), g		= 1873					
Argon	0.69	% N	= 53.98	Gas Produced, g		= 751					
Carbon Monoxide	2.46	% Ar	= 0.84								
Carbon Dioxide	30.00	Total: 100.00									
Methane	1.08	Average MW = 32.7									
Ethane	0.32	Flow = 0.09 scfm									
Ethylene	0.08	Exp Len = 10 hours									
Propane	0.35										
Propylene	0.15										
Butanes	0.18										
Hydrogen Sulfide	0.15										
Data Set											
1	2	3	4	5	6	7	8	9	10		
Hydrogen	0.26	1.70	0.51	2.03	2.09	0.26	1.56	0.25	1.30	1.54	
Nitrogen	77.56	70.65	70.12	65.08	43.63	65.10	53.80	61.77	67.44	54.81	
Oxygen	2.10	0.79	1.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Argon	0.98	0.89	0.48	0.73	0.46	0.73	0.58	0.70	0.77	0.60	
Carbon Monoxide	2.79	1.62	1.73	2.06	6.51	1.65	2.19	1.96	1.32	2.81	
Carbon Dioxide	12.23	22.51	23.64	27.35	45.47	31.10	39.03	33.23	26.93	38.52	
Methane	2.31	1.16	0.87	1.32	0.90	0.64	0.95	0.60	1.19	0.84	
Ethane	0.81	0.31	0.24	0.36	0.25	0.18	0.28	0.18	0.35	0.24	
Ethylene	0.18	0.06	0.08	0.15	0.05	0.04	0.04	0.04	0.08	0.05	
Propane	0.41	0.14	0.73	0.30	0.24	0.06	0.75	0.68	0.15	0.10	
Propylene	0.00	0.00	0.33	0.30	0.00	0.05	0.36	0.31	0.11	0.07	
Butanes	0.39	0.17	0.17	0.20	0.14	0.06	0.18	0.13	0.18	0.13	
Hydrogen Sulfide	0.00	0.00	0.07	0.13	0.26	0.13	0.27	0.14	0.20	0.30	

Table A-10. OXY-T10-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	1.23	% C	=	13.67	N ₂ +Ar (In), g	=	1439				
Nitrogen	62.80	% H	=	0.44	Gas (Out), g	=	2641				
Oxygen	0.05	% O	=	31.42	Air (In), g	=	1843				
Argon	0.70	% N	=	53.62	Gas Produced, g	=	768				
Carbon Monoxide	2.16	% Ar	=	0.86							
Carbon Dioxide	31.07	Total:		100.00							
Methane	1.00	Average MW = 32.8									
Ethane	0.28	Flow = 0.09 scfm									
Ethylene	0.07	Exp Len = 10 hours									
Propane	0.18										
Propylene	0.11										
Butanes	0.14										
Hydrogen Sulfide	0.20										

		Data Set									
		1	2	3	4	5	6	7	8	9	10
Hydrogen	1.88	0.91	1.89	2.19	1.32	1.00	0.10	0.10	1.17	0.10	
Nitrogen	74.14	69.94	65.64	51.71	61.97	55.40	66.82	66.82	54.00	67.55	
Oxygen	0.65	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Argon	0.85	0.80	0.75	0.56	0.70	0.60	0.70	0.70	0.59	0.76	
Carbon Monoxide	1.55	1.81	2.19	5.08	1.64	2.38	1.87	1.36	2.41	1.54	
Carbon Dioxide	19.02	25.04	27.10	38.58	32.40	38.88	33.87	29.01	39.68	28.88	
Methane	1.23	0.90	1.27	0.92	0.96	0.80	0.60	1.03	0.92	0.64	
Ethane	0.34	0.23	0.35	0.22	0.27	0.22	0.15	0.28	0.27	0.18	
Ethylene	0.06	0.03	0.06	0.04	0.05	0.03	0.01	0.05	0.05	0.04	
Propane	0.19	0.18	0.24	0.17	0.20	0.18	0.13	0.21	0.23	0.07	
Propylene	0.00	0.00	0.21	0.14	0.14	0.14	0.07	0.14	0.21	0.05	
Butanes	0.08	0.13	0.19	0.14	0.15	0.13	0.03	0.16	0.16	0.04	
Hydrogen Sulfide	0.00	0.02	0.12	0.24	0.20	0.24	0.13	0.08	0.30	0.16	

Table A-11. OXY-T11-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	1.39	% C	=	13.77	N ₂ +Ar (In), g	=	1439				
Nitrogen	62.69	% H	=	0.48	Gas (Out), g	=	2641				
Oxygen	0.06	% O	=	31.27	Air (In), g	=	1863				
Argon	0.70	% N	=	53.62	Gas Produced, g	=	767				
Carbon Monoxide	2.22	% Ar	=	0.86							
Carbon Dioxide	30.82	Total:		100.00							
Methane	1.03										
Ethane	0.29										
Ethylene	0.08										
Propane	0.23										
Propylene	0.11										
Butanes	0.14										
Hydrogen Sulfide	0.23										
Average MW = 32.7											
Flow = 0.09 scfm											
Exp Len = 10 hours											
Data Set											
	1	2	3	4	5	6	7	8	9	10	
Hydrogen	2.05	1.75	0.96	0.10	2.04	0.05	1.90	0.97	1.47	1.80	
Nitrogen	74.20	70.06	68.29	66.99	61.36	67.90	60.32	54.35	63.98	52.45	
Oxygen	0.51	0.17	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Argon	0.83	0.81	0.78	0.76	0.69	0.77	0.68	0.60	0.73	0.57	
Carbon Monoxide	1.93	1.64	1.92	2.20	2.84	1.64	1.85	2.69	1.35	3.02	
Carbon Dioxide	18.14	23.16	26.57	28.61	31.09	28.21	32.67	39.74	29.19	39.99	
Methane	1.54	1.20	0.83	0.82	1.06	0.74	1.29	0.81	1.14	0.99	
Ethane	0.46	0.34	0.22	0.20	0.29	0.21	0.37	0.23	0.35	0.29	
Ethylene	0.11	0.28	0.05	0.03	0.06	0.10	0.08	0.05	0.05	0.04	
Propane	0.22	0.15	0.09	0.08	0.12	0.08	0.16	0.10	0.82	0.12	
Propylene	0.00	0.10	0.06	0.00	0.09	0.06	0.11	0.07	0.36	0.08	
Butanes	0.00	0.23	0.13	0.11	0.08	0.10	0.22	0.14	0.23	0.16	
Hydrogen Sulfide	0.00	0.11	0.10	0.09	0.26	0.14	0.34	0.28	0.33	0.48	

Table A-12. OXY-T12-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	0.91	% C	=	13.07	N ₂ +Ar (In), g	=	1535				
Nitrogen	65.52	% H	=	0.50	Gas (Out), g	=	2671				
Oxygen	0.27	% O	=	28.97	Air (In), g	=	1998				
Argon	0.75	% N	=	56.54	Gas Produced, g	=	672				
Carbon Monoxide	2.14	% Ar	=	0.92							
Carbon Dioxide	28.04	Total:		100.00							
Methane	1.03										
Ethane	0.28										
Ethylene	0.08										
Propane	0.39										
Propylene	0.28										
Butanes	0.17										
Hydrogen Sulfide	0.15										
		Average MW = 32.4									
		Flow = 0.09 scfm									
		Exp Len = 10.66666 hours									
Data Set											
1	2	3	4	5	6	7	8	9	10		
Hydrogen	1.87	0.10	0.10	1.37	2.38	1.62	0.10	1.37	0.10		
Nitrogen	75.90	70.37	69.03	62.14	61.78	52.54	67.86	61.33	66.77		
Oxygen	1.90	0.35	0.31	0.00	0.00	0.00	0.00	0.00	0.00		
Argon	0.95	0.80	0.79	0.70	0.69	0.57	0.77	0.68	0.76		
Carbon Monoxide	2.02	1.99	1.87	2.83	1.75	4.27	1.15	2.06	1.31		
Carbon Dioxide	14.12	24.77	25.46	31.12	29.98	38.51	28.40	31.96	28.48		
Methane	1.57	0.94	0.88	0.85	1.62	0.77	0.72	1.19	0.86		
Ethane	0.52	0.26	0.24	0.14	0.46	0.22	0.22	0.34	0.26		
Ethylene	0.17	0.05	0.05	0.05	0.10	0.10	0.04	0.08	0.08		
Propane	0.37	0.10	0.71	0.24	0.35	0.64	0.23	0.30	0.67		
Propylene	0.32	0.07	0.31	0.25	0.34	0.32	0.26	0.31	0.35		
Butanes	0.30	0.13	0.16	0.12	0.25	0.17	0.10	0.17	0.17		
Hydrogen Sulfide	0.00	0.08	0.07	0.19	0.28	0.25	0.15	0.22	0.20		

Table A-13. OXY-T13-F Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	1.11	% C	=	13.95	N ₂ +Ar (In), g	=	1403				
Nitrogen	63.95	% H	=	0.63	Gas (Out), g	=	2517				
Oxygen	0.13	% O	=	29.69	Air (In), g	=	1826				
Argon	0.73	% N	=	54.85	Gas Produced, g	=	690				
Carbon Monoxide	2.15	% Ar	=	0.89							
Carbon Dioxide	29.08	Total:		100.00							
Methane	1.02	Average MW = 32.6									
Ethane	0.26	Flow = 0.09 scfm									
Ethylene	0.10	Exp Len = 9.75 hours									
Propane	0.83										
Propylene	0.29										
Butanes	0.20										
Hydrogen Sulfide	0.17										

		Data Set									
		1	2	3	4	5	6	7	8	9	10
Hydrogen	2.96	1.52	0.88	0.10	2.04	0.10	0.10	0.10	1.32	1.00	0.10
Nitrogen	73.20	71.32	68.95	68.27	55.45	59.32	65.64	60.48	59.65	59.65	66.13
Oxygen	1.12	0.35	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Argon	0.91	0.81	0.79	0.77	0.62	0.67	0.74	0.67	0.66	0.66	0.75
Carbon Monoxide	2.43	1.66	1.79	1.97	3.23	2.72	1.78	1.80	2.08	2.08	1.58
Carbon Dioxide	15.07	22.41	24.86	26.17	35.10	35.56	29.13	32.33	33.80	33.80	29.34
Methane	2.27	1.07	0.85	0.79	1.13	0.63	0.70	1.06	0.78	0.78	0.89
Ethane	0.36	0.15	0.24	0.22	0.33	0.18	0.21	0.32	0.23	0.23	0.26
Ethylene	0.24	0.05	0.11	0.11	0.15	0.04	0.04	0.09	0.09	0.09	0.06
Propane	0.55	0.29	1.12	1.16	1.14	0.26	1.08	1.05	0.97	0.97	0.32
Propylene	0.35	0.14	0.20	0.22	0.30	0.22	0.32	0.39	0.36	0.36	0.33
Butanes	0.52	0.17	0.16	0.14	0.22	0.09	0.14	0.22	0.16	0.16	0.13
Hydrogen Sulfide	0.00	0.05	0.07	0.09	0.31	0.18	0.13	0.27	0.23	0.23	0.13

APPENDIX B

Retort Zones Fischer Assay Data

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Table B-1. Fischer Assay of Retort Zones for Test 1

	Composite	Top	Middle	Bottom
Weight % Oil	9.14	0.01	1.62	11.49
Weight % Water	1	0.2	0.45	1.03
Weight % Spent Shale	87.5	99.74	97.29	84.65
Weight % Gas + Loss	2.36	0.05	0.64	2.83
Specific Gravity @ 60/60 F	0.9141	0.9161	0.9151	0.9191
GPT Oil	24.01	0.03	4.25	30.02
GPT Water	2.40	0.48	1.08	2.47

Table B-2. Fischer Assay of Retort Zones for Test 2

	Composite	Top	Middle	Bottom
Weight % Oil	9.65	0	0.03	0.02
Weight % Water	0.9	0.2	0.2	0.3
Weight % Spent Shale	87.16	99.67	99.4	99.32
Weight % Gas + Loss	2.29	0.13	0.33	0.35
Specific Gravity @ 60/60 F	0.9081	0.9161	0.9161	0.908
GPT Oil	25.51	0.00	0.08	0.05
GPT Water	2.16	0.48	0.48	0.72

Table B-3. Fischer Assay of Retort Zones for Test 3

	Composite	Top	Middle	Bottom
Weight % Oil	10.26	0.09	0	0
Weight % Water	0.99	18.89	2.3	10.44
Weight % Spent Shale	86.39	80.83	97.53	88.9
Weight % Gas + Loss	2.35	0.18	0.17	0.67
Specific Gravity @ 60/60 F	0.9071	0.9081	0.908	0.9081
GPT Oil	27.16	0.24	0.00	0.00
GPT Water	2.38	45.35	5.52	25.07

Table B-4. Fischer Assay of Retort Zones for Test 4

	Composite	Top	Middle	Bottom
Weight % Oil	9.47	0.01	3.41	10.58
Weight % Water	0.8	25.79	6.99	3.47
Weight % Spent Shale	87.68	73.87	88.73	84.05
Weight % Gas + Loss	2.05	0.33	0.88	1.9
Specific Gravity @ 60/60 F	0.90278	0.91455	0.91767	0.91142
GPT Oil	25.19	0.03	8.92	27.87
GPT Water	1.92	61.92	16.78	8.33

Table B-5. Fischer Assay of Retort Zones for Test 5

	Composite	Top	Middle	Bottom
Weight % Oil	9.71	0.07	0.09	0.01
Weight % Water	0.7	24.87	10.59	10.83
Weight % Spent Shale	87.53	74.83	89.12	88.7
Weight % Gas + Loss	2.07	0.22	0.2	0.45
Specific Gravity @ 60/60 F	0.90509	0.92	0.92	0.92
GPT Oil	25.76	0.18	0.23	0.03
GPT Water	1.68	59.71	25.43	26.00

Table B-6. Fischer Assay of Retort Zones for Test 6

	Composite	Top	Middle	Bottom
Weight % Oil	9.71	0.18	0.11	5.31
Weight % Water	0.7	29.9	15.25	9.85
Weight % Spent Shale	87.53	69.64	84.43	93.42
Weight % Gas + Loss	2.07	0.28	0.22	1.42
Specific Gravity @ 60/60 F	0.90509	0.92	0.92	0.91574
GPT Oil	25.76	0.47	0.29	13.92
GPT Water	1.68	71.79	36.61	23.65

Table B-7. Fischer Assay of Retort Zones for Test 7

	Composite	Top	Middle	Bottom
Weight % Oil	9.705	0.45	0	0.22
Weight % Water	0.8	27.81	13.42	20.67
Weight % Spent Shale	87.31	71.4	86.57	78.55
Weight % Gas + Loss	2.17	0.33	0	0.56
Specific Gravity @ 60/60 F	0.90278	0.92	0.92	0.92
GPT Oil	25.81	1.17	0.00	0.57
GPT Water	1.92	66.77	32.22	49.63

Table B-8. Fischer Assay of Retort Zones for Test 8

	Composite	Top	Middle	Bottom
Weight % Oil	9.8	0	0.48	10.29
Weight % Water	0.8	27.74	12.96	3
Weight % Spent Shale	87.31	72.24	85.87	84.96
Weight % Gas + Loss	2.09	0.02	0.68	1.78
Specific Gravity @ 60/60 F	0.90503	0.92	0.92	0.92373
GPT Oil	26.00	0.00	1.25	26.75
GPT Water	1.92	66.60	31.12	7.20

Table B-9. Fischer Assay of Retort Zones for Test 9

	Composite	Top	Middle	Bottom
Weight % Oil	9.47	0.14	3.73	12.06
Weight % Water	0.9	28.46	9.98	2.46
Weight % Spent Shale	87.4	70.59	85.24	83.33
Weight % Gas + Loss	2.24	0.81	1.06	2.15
Specific Gravity @ 60/60 F	0.90509	0.92	0.9221	0.91403
GPT Oil	25.12	0.37	9.71	31.68
GPT Water	2.16	68.33	23.96	5.91

Table B-10. Fischer Assay of Retort Zones for Test 10

	Composite	Top	Middle	Bottom
Weight % Oil	9.47	0.21	3.05	11.57
Weight % Water	0.9	24.39	11.96	3.59
Weight % Spent Shale	87.4	75.17	84.68	82.59
Weight % Gas + Loss	2.24	0.42	0.31	2.14
Specific Gravity @ 60/60 F	0.9061	0.92	0.92	0.9144
GPT Oil	25.09	0.55	7.96	30.38
GPT Water	2.16	58.56	28.72	8.62

Table B-11. Fischer Assay of Retort Zones for Test 11

	Composite	Top	Middle	Bottom
Weight % Oil	9.2	1.43	0.04	10
Weight % Water	1.1	24.87	12.69	6.5
Weight % Spent Shale	87.46	73.38	86.94	81.54
Weight % Gas + Loss	2.24	0.32	0.33	1.96
Specific Gravity @ 60/60 F	0.9054	0.92	0.92	0.9168
GPT Oil	24.40	3.73	0.10	26.19
GPT Water	2.64	59.71	30.47	15.61

Table B-12. Fischer Assay of Retort Zones for Test 12

	Composite	Top	Middle	Bottom
Weight % Oil	9.75	0.09	0.02	0.91
Weight % Water	1	25.76	7.49	8.88
Weight % Spent Shale	86.94	73.82	92.13	89.6
Weight % Gas + Loss	2.3	0.33	0.36	0.6
Specific Gravity @ 60/60 F	0.905	0.92	0.92	0.92
GPT Oil	25.87	0.23	0.05	2.37
GPT Water	2.40	61.85	17.98	21.32

Table B-13. Fischer Assay of Retort Zones for Test 13

	Composite	Top	Middle	Bottom
Weight % Oil	9.68	0.07	0.02	6.17
Weight % Water	0.9	30.08	26.6	9.27
Weight % Spent Shale	87.25	68.35	72.94	82.31
Weight % Gas + Loss	2.17	1.5	0.43	2.25
Specific Gravity @ 60/60 F	0.9046	0.92	0.92	0.92
GPT Oil	25.69	0.18	0.05	16.10
GPT Water	2.16	72.22	63.87	22.26

APPENDIX C

Quench and Deluge Total Organic Analysis

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Table C-1. Total Organic Carbon Analysis for Quench and Deluge Tests 3 and 4

Test Number T3			Test Number T4		
Tube Volume, cm ³	4350		Tube Volume, cm ³	4350	
Volume Shale, cm ³	2285		Volume Shale, cm ³	2327	
Void Volume, cm ³	2064		Void Volume, cm ³	2023	
Deluge Flow Rate, cm ³ /min	44.3		Deluge Flow Rate, cm ³ /min	44.6	
Percent Voids	47		Percent Voids	47	

Time, min	Pore Volume	TOC		Time, min	Pore Volume	TOC
14	0.30	7.2	Deluge	12	0.26	540 Deluge
19	0.41	-0.6		19	0.42	306
24	0.52	6.2		26	0.57	182
29	0.62	8.1		38	0.84	204
49	1.05	7.6		48	1.06	112
59	1.27	16.4		53	1.17	92
69	1.48	9.3		58	1.28	115
79	1.70	21.9		63	1.39	208
89	1.91	9.3		68	1.50	195
99	2.12	9.7		75	1.65	186
109	2.34	6.6		80	1.76	244
119	2.55	8.3		85	1.87	90
129	2.77	5.1		91	2.01	199
139	2.98	1.9		97	2.14	106
149	3.20	-1.1		101	2.23	116
159	3.41	4.9		106	2.34	295
169	3.63	5		111	2.45	105
179	3.84	0.9		118	2.60	204
189	4.06	1.9		122	2.69	228
199	4.27	-0.3		127	2.80	145
209	4.49	0.8		135	2.98	142
219	4.70	-1.4		141	3.11	71
229	4.91	3.4		148	3.26	80
239	5.13	-0.9		158	3.48	20
249	5.34	1.1		168	3.70	12
259	5.56	1		180	3.97	37
				190	4.19	55
				200	4.41	59
				210	4.63	36
				220	4.85	67
				230	5.07	33
				240	5.29	46

Table C-2. Total Organic Carbon Analysis for Quench and Deluge Tests 5 and 6

Test Number	T5	Test Number	T6
Tube Volume, cm ³	4350	Tube Volume, cm ³	4350
Volume Shale, cm ³	2314	Volume Shale, cm ³	2286
Void Volume, cm ³	2036	Void Volume, cm ³	2064
Deluge Flow Rate, cm ³ /min	53.1	Deluge Flow Rate, cm ³ /min	37.5
Percent Voids	47	Percent Voids	47

Time, min	Pore Volume	TOC	Time, min	Pore Volume	TOC
20	0.52	11.4 Deluge	15	1.09	128 Quench
30	0.78	2	25	1.36	60.2 Deluge
40	1.04	2	35	1.55	76.6
50	1.30	2.8	50	1.73	52.1
60	1.56	1.5	60	2.00	16.6
70	1.83	0.3	70	2.18	34.4
80	2.09	0.3	80	2.36	39.2
90	2.35	0.9	90	2.55	30.1
100	2.61	0.1	100	2.73	27.1
110	2.87	0.2	110	2.91	39.4
125	3.26	1.4	125	3.09	35.9
140	3.65	0.1	135	3.36	31.8
155	4.04	3.1	145	3.54	16.1
170	4.43	-1.1	155	3.73	20.1
185	4.83	0.8	175	3.91	15.8
200	5.22	0.4	205	4.27	19.2
215	5.61	-1.6	215	4.82	27.2
230	6.00	2	225	5.00	17.2
245	6.39	3.9	237	5.18	25.5
260	6.78	0.4	270	5.40	10.9
275	7.17	-1.3		6.00	0.3

Table C-3. Total Organic Carbon Analysis for Quench and Deluge Tests 7 and 8

Test Number	T7	Test Number	T8
Tube Volume, cm ³	4350	Tube Volume, cm ³	4350
Volume Shale, cm ³	2228	Volume Shale, cm ³	2228
Void Volume, cm ³	2122	Void Volume, cm ³	2122
Deluge Flow Rate, cm ³ /min	51.2	Deluge Flow Rate, cm ³ /min	77.3
Percent Voids	49	Percent Voids	49

Time, min	Pore Volume	TOC		Time, min	Pore Volume	TOC	
	0.92	16.5	Quench	0	1.43	423	Quench
	0.99	37.2		30	2.52	49.6	Deluge
15	1.35	17.1	Deluge	40	2.89	15.1	
25	1.59	18.7		55	3.43	6.2	Recycle
35	1.84	8.4		70	3.98	16.1	Cond
45	2.08	6.3		85	4.53	11	
55	2.32	9.8		100	5.07	12.2	Recycle
65	2.56	10.1		130	6.17	4.9	Water
75	2.80	18.8		160	7.26	3.3	
85	3.04	19.5		200	8.72	10.4	
95	3.28	16.4					
105	3.52	9.7					
115	3.77	6.6					
125	4.01	13.5					
135	4.25	13.2					
145	4.49	8.7					
155	4.73	6.4					
165	4.97	5.8					
175	5.21	4.1					
195	5.70	2.9					
205	5.94	3.7					
215	6.18	4.8					
225	6.42	2.7					

Table C-4. Total Organic Carbon Analysis for Quench and Deluge Tests 9 and 10

Test Number			T9	Test Number			T10
Tube Volume, cm ³			4350	Tube Volume, cm ³			4350
Volume Shale, cm ³			2242	Volume Shale, cm ³			2320
Void Volume, cm ³			2108	Void Volume, cm ³			2030
Deluge Flow Rate, cm ³ /min			46.8	Deluge Flow Rate, cm ³ /min			41.4
Percent Voids			Percent Voids	Percent Voids			47

Time, min	Pore Volume	TOC		Time, min	Pore Volume	TOC
0	0.00	928.2	Surfact	0.13		1574 Quench
15	0.33	886.9	Deluge	0.33		318
25	0.56	1006.9		2.12		44 Deluge
35	0.78	714.4		35	2.84	76.2
45	1.00	650		45	3.04	10.4
55	1.22	633.7		65	3.45	8.5
70	1.55	650.2		80	3.75	5.2
80	1.78	699.1		90	3.96	8.6
90	2.00	699.1		100	4.16	6.6
100	2.22	681.8		110	4.36	7.7
110	2.44	556		120	4.57	7.1
120	2.66	554.9		130	4.77	6
135	3.00	546.9	Water	140	4.98	11.3
145	3.22	303	Deluge	150	5.18	5.6
155	3.44	233.3		160	5.38	0
165	3.66	150.8		170	5.59	4.9
175	3.89	274.2		180	5.79	7.1
185	4.11	111.6		190	6.00	2.7
195	4.33	92.9		200	6.20	2.7
205	4.55	77.9		210	6.40	2.9
215	4.77	92.5		220	6.61	0
225	5.00	69.7		230	6.81	0
235	5.22	67.8		240	7.02	0
245	5.44	76.6				
255	5.66	59.9				
265	5.88	52.8				
275	6.11	63.7				
285	6.33	42.6				
295	6.55	37.3				
305	6.77	33				
315	6.99	32.3				
325	7.22	58.2				
335	7.44	33.8				
345	7.66	29.8				
355	7.88	26.9				
365	8.10	25.1				
375	8.33	30.5				

Table C-5. Total Organic Carbon Analysis for Quench and Deluge Tests 11 and 12

Test Number	T11	Test Number	T12
Tube Volume, cm ³	4350	Tube Volume, cm ³	4350
Volume Shale, cm ³	2362	Volume Shale, cm ³	2380
Void Volume, cm ³	1988	Void Volume, cm ³	1970
Deluge Flow Rate, cm ³ /min	40.4	Deluge Flow Rate, cm ³ /min	34.9
Percent Voids	46	Percent Voids	45

Time, min	Pore Volume	TOC	Time, min	Pore Volume	TOC
10	0.07	61 Quench	0.34	113	Reverse
35	0.26	54	0.51	40	Burn
55	0.41	75	0.75	28	Quench
85	0.63	121	0.97	25	
120	0.89	248	1.03	25	
150	1.11	191	5	1.12	30.6 Deluge
190	1.41	136	20	1.38	9.3
220	1.63	148	30	1.56	21.1
250	1.85	107	40	1.74	17.1
285	2.11	78	50	1.91	16.5
325	2.40	78	60	2.09	13.4
355	2.63	52		3.16	11.2
385	2.85	46		4.22	2.3
450	3.33	47		5.28	0
480	3.55	39			
500	3.70	43			
15	4.00	80 Deluge			
25	4.21	48			
50	4.71	46			
	5.32	8.7			
	5.93	3.1			
	6.54	4.7			

Table C-6. Total Organic Carbon Analysis for Quench and Deluge Tests 13

Test Number	T13	
Tube Volume, cm ³	4350	
Volume Shale, cm ³	2339	
Void Volume, cm ³	2011	
Deluge Flow Rate, cm ³ /min	37.6	
Percent Voids	46	

Time, min	Pore Volume	TOC	Time, min	Pore Volume	TOC
	0.35	1019 Quench			
	1.29	47			
	1.42				
50	2.36	11.2 Deluge			
75	2.82	16.4			
105	3.39	5.1			
120	3.67	6.1			
135	3.95	13.5			
150	4.23	8.1			

APPENDIX D

Composite Quench and Deluge Water Analysis

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Table D-1. Composite Deluge Water Analysis for Tests 3, 4, and 5

	T3 Retort Deluge	T4 Retort Deluge	T5 Retort Deluge
TOC, mg/L	7	50	2
TDS, mg/L	576	2410	1510
Total Alkalinity, mg/L	290	830	1350
Ammonia, mg/L	<0.2	13	0.4
Phenols, µg/L	<20	910	55
Fluoride, mg/L			
Sulfate, mg/L	140	760	80
pH			
Conductivity, mS/cm	0.96	4.5	3.2
Aluminum, mg/L	7.4	36.2	139
Boron, mg/L	0.12	0.11	0.21
Cadmium, mg/L	<0.01	<0.01	<0.01
Magnesium, mg/L	0.89	0.278	0.168
Lead, mg/L	<0.004	<0.004	<0.004
Manganese, mg/L	<0.003	<0.003	0.005
Calcium, mg/L	3.58	17.4	10.3
Molybdenum, mg/L	1.11	1.69	4.05
Zinc, mg/L	0.036	0.015	0.014
Arsenic, mg/L	0.014	0.033	0.11
Selenium, mg/L	0.009	0.03	0.012
Thallium, mg/L	<0.005	<0.005	<0.10
Sodium, mg/L	198	651	471
Silver, mg/L	<0.007	<0.007	<0.007
Barium, mg/L	0.02	0.125	0.24
Beryllium, mg/L	<0.002	<0.002	<0.002

Table D-2. Composite Deluge Water Analysis for Tests 6 and 7

	T6 Retort	T7 Retort	
	Deluge	Quench	Deluge
TOC, mg/L	9	18	4.2
TDS, mg/L	1010	72	2550
Total Alkalinity, mg/L	735	101	1360
Ammonia, mg/L	1.1	47	0.47
Phenols, µg/L	89	502	25
Fluoride, mg/L	2.9	7.7	10
Sulfate, mg/L	790	33	450
pH	12.3	5.3	12
Conductivity, mS/cm		0.42	
Aluminum, mg/L		<0.045	
Boron, mg/L	0.123	0.058	0.377
Cadmium, mg/L		<0.01	
Magnesium, mg/L		0.365	
Lead, mg/L		<0.002	
Manganese, mg/L		0.007	
Calcium, mg/L		3.08	
Molybdenum, mg/L	0.201	<0.01	0.024
Zinc, mg/L		<0.003	
Arsenic, mg/L	<0.005	0.014	<0.005
Selenium, mg/L		<0.005	
Thallium, mg/L		<0.005	
Sodium, mg/L		2.38	
Silver, mg/L		<0.007	
Barium, mg/L		<0.01	
Beryllium, mg/L		<0.002	

Table D-3. Composite Deluge Water Analysis for Tests 8 and 10

	T8 Retort		T10 Retort	
	Quench	Deluge	Quench	Deluge
TOC, mg/L	423	10.4	162	6.4
TDS, mg/L	138	2750	450	4080
Total Alkalinity, mg/L	140	1590	320	1510
Ammonia, mg/L	43	2.5	31	0.5
Phenols, µg/L	2120	27	600	25
Fluoride, mg/L	14	10	3.9	16
Sulfate, mg/L	16	600	54	1170
pH	5.9	12.3	6.7	11
Conductivity, mS/cm	0.39		0.71	
Aluminum, mg/L	<0.045		0.063	
Boron, mg/L	0.201	0.329	0.226	0.363
Cadmium, mg/L	<0.01		<0.01	
Magnesium, mg/L	0.317		14.8	
Lead, mg/L	<0.002		<0.05	
Manganese, mg/L	0.006		<0.003	
Calcium, mg/L	1.5		7.38	
Molybdenum, mg/L	<0.01	1.08	0.093	1.59
Zinc, mg/L	<0.003		0.005	
Arsenic, mg/L	0.074	<0.005	<0.1	0.009
Selenium, mg/L	<0.005		<0.1	
Thallium, mg/L	<0.005			
Sodium, mg/L	13.1	109		
Silver, mg/L	<0.007		<0.007	
Barium, mg/L	<0.01		0.025	
Beryllium, mg/L	<0.002		<0.002	

Table D-4. Composite Deluge Water Analysis for Tests 11 and 12

	T11 Retort		n	T12 Retort	
	Quench	Deluge		Quench	Deluge
TOC, mg/L	84	4.4		19.5	7.8
TDS, mg/L	650	3950		200	2760
Total Alkalinity, mg/L	390	1970		220	1650
Ammonia, mg/L	3.3	3.8		69	1.1
Phenols, µg/L	150	56		570	64
Fluoride, mg/L	6.4	11.5		0.6	11
Sulfate, mg/L	98	1100		28	35
pH	9	12.3		5.9	11.5
Conductivity, mS/cm					
Aluminum, mg/L					
Boron, mg/L	0.4	0.359		0.102	0.074
Cadmium, mg/L					
Magnesium, mg/L					
Lead, mg/L					
Manganese, mg/L					
Calcium, mg/L					
Molybdenum, mg/L	0.18	1.43		<0.01	0.058
Zinc, mg/L					
Arsenic, mg/L	0.064	0.026		0.038	0.005
Selenium, mg/L					
Thallium, mg/L					
Sodium, mg/L					
Silver, mg/L					
Barium, mg/L					
Beryllium, mg/L					

APPENDIX E

Reverse Combustion Gas Analysis

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Table E-1. OXY-T2-R Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	12.91	% C	=	16.42	N ₂ +Ar (In), g	=	1301				
Nitrogen	56.65	% H	=	3.04	Gas (Out), g	=	2148				
Oxygen	0.00	% O	=	19.97	Air (In), g	=	1694				
Argon	0.65	% N	=	59.60	Gas Produced, g	=	454				
Carbon Monoxide	3.28	% Ar	=	0.97							
Carbon Dioxide	14.97	Total:		100.00							
Methane	7.21										
Ethane	1.24										
Ethylene	1.31										
Propane	0.33										
Propylene	0.91										
Butanes	0.53										
Hydrogen Sulfide	0.00										
		Average MW = 26.6									
		Flow = 0.074 scfm									
		Exp Len = 11 hours									
		Data Set									
	1	2	3	4	5	6	7	8	9	10	
Hydrogen	3.64	26.79	26.02	25.41	22.58	14.39	9.00	4.95	9.38	9.30	
Nitrogen	13.61	15.49	29.09	43.87	56.44	74.67	80.32	79.01	65.81	66.13	
Oxygen	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Argon	0.09	0.15	0.30	0.50	0.65	0.90	0.97	0.95	0.78	0.72	
Carbon Monoxide	0.79	15.02	6.78	2.38	0.96	0.31	0.59	2.91	3.20	3.11	
Carbon Dioxide	56.02	13.87	9.59	7.50	6.34	5.73	7.91	10.99	12.96	13.05	
Methane	22.95	19.96	17.24	11.08	6.73	1.90	0.59	0.69	4.35	4.28	
Ethane	0.17	3.27	3.57	2.50	1.56	0.46	0.14	0.21	1.24	1.23	
Ethylene	2.72	3.44	3.60	2.75	1.79	0.54	0.17	0.10	0.54	0.53	
Propane	0.00	0.30	0.65	0.59	0.42	0.16	0.06	0.09	0.50	0.52	
Propylene	0.00	1.51	2.61	2.33	1.60	0.51	0.18	0.10	0.52	0.55	
Butanes	0.00	0.20	0.54	1.08	0.92	0.43	0.07	0.00	0.71	0.57	
Hydrogen Sulfide	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	

Table E-2. OXY-T3-R Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	12.25	% C	=	14.60	N ₂ +Ar (In), g	=	599				
Nitrogen	55.53	% H	=	1.86	Gas (Out), g	=	1018				
Oxygen	0.00	% O	=	24.63	Air (In), g	=	781				
Argon	0.65	% N	=	57.93	Gas Produced, g	=	237				
Carbon Monoxide	9.54	% Ar	=	0.97							
Carbon Dioxide	15.89	Total:	100.00								
Methane	5.14										
Ethane	0.38										
Ethylene	0.53										
Propane	0.01	Average MW = 26.8									
Propylene	0.08	Flow = 0.05 scfm									
Butanes	0.00	Exp Len = 7.5 hours									
Hydrogen Sulfide	0.00										
Data Set											
1	2	3	4	5	6	7	8	9	10		
Hydrogen	9.71	22.49	21.64	18.33	15.25	4.98	3.54	2.09			
Nitrogen	31.87	35.68	34.10	42.76	67.20	78.61	76.37	77.65			
Oxygen	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00			
Argon	0.37	0.40	0.36	0.49	0.81	0.94	0.92	0.93			
Carbon Monoxide	7.14	18.62	18.88	19.20	7.64	1.58	1.63	1.64			
Carbon Dioxide	38.09	9.96	12.94	13.58	6.56	13.26	16.25	16.47			
Methane	11.09	10.67	10.14	5.19	2.29	0.39	0.68	0.63			
Ethane	0.75	0.82	0.73	0.19	0.12	0.07	0.17	0.16			
Ethylene	0.85	1.20	1.14	0.27	0.14	0.11	0.23	0.26			
Propane	0.00	0.00	0.00	0.00	0.00	0.00	0.06	0.00			
Propylene	0.10	0.15	0.07	0.00	0.00	0.05	0.15	0.16			
Butanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00			
Hydrogen Sulfide	0.03	0.00	0.00	0.00	0.00	0.00	0.00	0.00			

Table E-3. OXY-T5-R Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	14.23	% C	=	13.61	N ₂ +Ar (In), g	=	420				
Nitrogen	58.22	% H	=	2.00	Gas (Out), g	=	670				
Oxygen	0.00	% O	=	21.78	Air (In), g	=	546				
Argon	0.65	% N	=	61.62	Gas Produced, g	=	124				
Carbon Monoxide	6.93	% Ar	=	0.98							
Carbon Dioxide	14.54	Total:		100.00							
Methane	3.28	Average MW = 26.5									
Ethane	0.38	Flow = 0.15 scfm									
Ethylene	0.87	Exp Len = 1.8 hours									
Propane	0.02										
Propylene	0.81										
Butanes	0.06										
Hydrogen Sulfide	0.00										

Data Set										
1	2	3	4	5	6	7	8	9	10	
Hydrogen	15.58	14.49								
Nitrogen	51.77	58.87								
Oxygen	0.00	0.00								
Argon	0.57	0.66								
Carbon Monoxide	6.96	7.67								
Carbon Dioxide	17.26	12.98								
Methane	5.09	2.73								
Ethane	0.58	0.32								
Ethylene	1.34	0.71								
Propane	0.06	0.00								
Propylene	0.66	1.51								
Butanes	0.12	0.06								
Hydrogen Sulfide	0.00	0.00								

Table E-4. OXY-T7-R Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
1	2	3	4	5	6	7	8	9	10		
Hydrogen	12.13		% C = 17.38							N ₂ +Ar (In), g	= 599
Nitrogen	57.06		% H = 2.60							Gas (Out), g	= 999
Oxygen	0.00		% O = 20.00							Air (In), g	= 781
Argon	0.66		% N = 59.05							Gas Produced, g	= 218
Carbon Monoxide	5.18		% Ar = 0.97								
Carbon Dioxide	14.32										
Methane	2.48		Total: 100.00								
Ethane	0.68										
Ethylene	6.77										
Propane	0.04										
Propylene	0.55										
Butanes	0.14										
Hydrogen Sulfide	0.00										
<p>Average MW = 27.1 Flow = 0.15 scfm Exp Len = 2.5 hours</p>											
Data Set											
1	2	3	4	5	6	7	8	9	10		
Hydrogen	13.05										
Nitrogen	48.48										
Oxygen	0.00										
Argon	0.54										
Carbon Monoxide	5.40										
Carbon Dioxide	14.38										
Methane	3.14										
Ethane	1.11										
Ethylene	12.92										
Propane	0.07										
Propylene	0.70										
Butanes	0.21										
Hydrogen Sulfide	0.00										

Table E-5. OXY-T12-R Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	11.55	% C	=	11.91	N ₂ +Ar (In), g	=	228				
Nitrogen	64.68	% H	=	1.69	Gas (Out), g	=	338				
Oxygen	0.00	% O	=	18.92	Air (In), g	=	297				
Argon	0.74	% N	=	66.40	Gas Produced, g	=	41				
Carbon Monoxide	4.45	% Ar	=	1.08							
Carbon Dioxide	13.90	Total:		100.00							
Methane	1.97	Average MW = 27.3									
Ethane	0.37	Flow = 0.09 scfm									
Ethylene	1.33	Exp Len = 1.6 hours									
Propane	0.07										
Propylene	0.62										
Butanes	0.32										
Hydrogen Sulfide	0.00										

Data Set										
1	2	3	4	5	6	7	8	9	10	
Hydrogen	11.55									
Nitrogen	64.68									
Oxygen	0.00									
Argon	0.74									
Carbon Monoxide	4.45									
Carbon Dioxide	13.90									
Methane	1.97									
Ethane	0.37									
Ethylene	1.33									
Propane	0.07									
Propylene	0.62									
Butanes	0.32									
Hydrogen Sulfide	0.00									

APPENDIX F

Quench Gas Analysis

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Table F-1. OXY-T6-Q Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
		1	2	3	4	5	6	7	8	9	10
Hydrogen	2.39										
Nitrogen	61.15										
Oxygen	15.18										
Argon	0.72										
Carbon Monoxide	0.00										
Carbon Dioxide	14.33										
Methane	5.86										
Ethane	0.05										
Ethylene	0.31										
Propane	0.00										
Propylene	0.00										
Butanes	0.00										
Hydrogen Sulfide	0.00										
		Data Set									
Hydrogen	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Nitrogen	45.22	60.87	65.19	67.66	64.43	63.57	63.57	63.57	63.57	63.57	63.57
Oxygen	11.47	15.74	15.16	16.31	16.10	16.30	16.30	16.30	16.30	16.30	16.30
Argon	0.54	0.75	0.73	0.77	0.77	0.78	0.78	0.78	0.78	0.78	0.78
Carbon Monoxide	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Carbon Dioxide	34.93	18.14	10.37	8.84	7.38	6.31	6.31	6.31	6.31	6.31	6.31
Methane	7.17	3.95	8.08	6.34	5.49	4.12	4.12	4.12	4.12	4.12	4.12
Ethane	0.14	0.05	0.02	0.01	0.04	0.06	0.06	0.06	0.06	0.06	0.06
Ethylene	0.53	0.50	0.44	0.06	0.15	0.16	0.16	0.16	0.16	0.16	0.16
Propane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Propylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Butanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Hydrogen Sulfide	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

% C = 8.46

% H = 1.00

% O = 31.84

% N = 57.73

% Ar = 0.97

Total: 100.00

Average MW = 29.7

Flow = 0 scfm

Exp Len = 22 hours

Table F-2. OXY-T7-Q Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	21.45	% C	=	8.03							
Nitrogen	28	% H	=	1.95							
Oxygen	9.61	% O	=	30.12							
Argon	0.65	% N	=	58.88							
Carbon Monoxide	0.17	% Ar	=	1.02							
Carbon Dioxide	14.16	Total:		100.00							
Methane	0.15										
Ethane	0.00										
Ethylene	0.07										
Propane	0.00										
Propylene	0.00										
Butanes	0.00										
Hydrogen Sulfide	0.46										
		Average MW	=	25.3							
		Flow	=	0 scfm							
		Exp Len	=	25 hours							
		Data Set									
		1	2	3	4	5	6	7	8	9	10
Hydrogen	10.81	28.77	27.39	24.09	29.76	29.84	10.63	10.30			
Nitrogen	62.73	33.40	41.62	50.84	50.85	51.06	67.73	68.00			
Oxygen	1.20	1.70	5.67	10.38	11.96	11.58	17.18	17.19			
Argon	0.78	0.27	0.50	0.63	0.64	0.65	0.86	0.86			
Carbon Monoxide	0.93	0.36	0.10	0.00	0.00	0.00	0.00	0.00			
Carbon Dioxide	23.26	33.87	23.05	13.03	6.20	6.60	3.60	3.65			
Methane	0.06	0.16	0.16	0.38	0.23	0.24	0.00	0.00			
Ethane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00			
Ethylene	0.08	0.00	0.23	0.00	0.24	0.00	0.00	0.00			
Propane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00			
Propylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00			
Butanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00			
Hydrogen Sulfide	0.15	1.47	1.28	0.65	0.12	0.04	0.00	0.00			

Table F-3. OXY-T8-Q Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	2.63	% C	=	23.26							
Nitrogen	47.71	% H	=	4.69							
Oxygen	5.43	% O	=	23.21							
Argon	0.52	% N	=	48.10							
Carbon Monoxide	0.90	% Ar	=	0.75							
Carbon Dioxide	14.27	Total:		100.00							
Methane	19.82										
Ethane	3.39										
Ethylene	4.28										
Propane	0.44	Average MW	=	27.8							
Propylene	0.39	Flow	=	0 scfm							
Butanes	0.16	Exp Len	=	22.5 hours							
Hydrogen Sulfide	0.07										

		Data Set									
	1	2	3	4	5	6	7	8	9	10	
Hydrogen	1.02	2.40	3.25	3.21	3.26						
Nitrogen	32.05	35.79	38.88	60.91	70.90						
Oxygen	0.46	0.39	0.42	8.86	17.00						
Argon	0.28	0.33	0.37	0.74	0.89						
Carbon Monoxide	1.07	1.20	1.28	0.89	0.06						
Carbon Dioxide	17.42	19.06	18.69	12.33	3.83						
Methane	32.87	28.20	26.17	9.03	2.81						
Ethane	5.90	4.88	4.30	1.45	0.42						
Ethylene	7.35	6.20	5.37	2.06	0.44						
Propane	0.73	0.60	0.51	0.20	0.17						
Propylene	0.63	0.51	0.42	0.16	0.21						
Butanes	0.08	0.30	0.27	0.16	0.00						
Hydrogen Sulfide	0.14	0.15	0.07	0.02	0.00						

Table F-4. OXY-T10-Q Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
Hydrogen	39.17	% C = 18.95									
Nitrogen	31.92	% H = 5.85									
Oxygen	0.00	% O = 30.67									
Argon	0.00	% N = 44.53									
Carbon Monoxide	1.03	% Ar = 0.00									
Carbon Dioxide	18.72	Total: 100.00									
Methane	6.37	Average MW = 20.1									
Ethane	1.21	Flow = 0 scfm									
Ethylene	1.58	Exp Len = 16 hours									
Propane	0.00										
Propylene	0.00										
Butanes	0.00										
Hydrogen Sulfide	0.00										
		Data Set									
		1	2	3	4	5	6	7	8	9	10
Hydrogen	39.17										
Nitrogen	31.92										
Oxygen	0.00										
Argon	0.00										
Carbon Monoxide	1.03										
Carbon Dioxide	18.72										
Methane	6.37										
Ethane	1.21										
Ethylene	1.58										
Propane	0.00										
Propylene	0.00										
Butanes	0.00										
Hydrogen Sulfide	0.00										

Table F-5. OXY-T12-Q Experimental Gas Composition Data

Average Composition, vol %		Average Atomic Composition, wt %									
		Data Set									
		1	2	3	4	5	6	7	8	9	10
Hydrogen	8.58	9.59	8.35	7.81							
Nitrogen	71.40	70.80	71.75	71.63							
Oxygen	0.10	0.00	0.21	0.08							
Argon	0.70	0.68	0.73	0.71							
Carbon Monoxide	2.84	2.82	2.84	2.84							
Carbon Dioxide	12.85	12.68	12.89	12.96							
Methane	0.98	0.97	0.98	0.98							
Ethane	0.19	0.18	0.19	0.20							
Ethylene	1.01	1.27	0.46	1.28							
Propane	0.00	0.00	0.00	0.00							
Propylene	1.31	0.94	1.55	1.44							
Butanes	0.04	0.00	0.04	0.07							
Hydrogen Sulfide	0.02	0.07	0.00	0.00							

% C	=	9.96
% H	=	1.24
% O	=	16.41
% N	=	71.39
% Ar	=	1.00
Total:		100.00
Average MW	=	28.0
Flow	=	0 scfm
Exp Len	=	18 hours

Quenching and Stabilization of MIS Retorts:
Bench-Scale Experiments

END

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