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STATUS OF METC INVESTIGATIONS OF COAL GAS DESULFURIZATION AT HIGH TEMPERATURE

By
G. Steinfeld

March 1984

Work Performed Under Contract No.: DE-AC21-81MC16545

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

By
Science Applications, Inc.
Morgantown, West Virginia

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SUMMARY

This report documents the continuing effort at the U.S. Department of Energy/Morgantown Energy Technology Center (METC) to develop a hot-gas desulfurization process for coal-derived gas, primarily for application to molten carbonate fuel cells. Metal oxide sorbents were tested on lab-scale test equipment, and it was determined that scale-up of the process was warranted.

A larger, skid-mounted test unit was therefore designed, constructed, and installed on a sidestream of the DOE/METC fixed-bed gasifier. A first series of tests was conducted during Gasifier Run 101. These tests served to "shake down" the test unit, and provide data on the performance of the test unit operating on coal-derived gas.

Overall, the process operated well on fixed-bed, air-blown gasifier gas. Sulfur levels in exit dry gas were reduced to less than 10 ppm. Regeneration appears to restore the sulfur-removing capacity of the sorbent. Sorbent integrity was maintained during the test period, which incorporated three sulfidations.

It is recommended that treatment of the regeneration offgas be investigated, and that testing and development of a system to reduce the sulfur in this gas to elemental sulfur be initiated. In addition, it is suggested that a multiple reactor system be planned for continuous operation, to allow for long-term tests of downstream users of desulfurized gas.

1: INTRODUCTION

The utilization of the abundant coal reserves in the United States is becoming an attractive route to promote energy independence. In light of large cost overruns and regulatory restrictions for nuclear power plants, increased attention is being given to the utilization of coal, and more conversions to coal are being implemented. Coal utilization in most cases involves either combustion or gasification. When gasification is chosen, a fuel gas is generated at temperatures generally above 1000°F. This coal-derived gas will contain sulfur compounds, primarily H_2S and CO_2 , which need to be removed to protect downstream systems such as fuel cells, catalysts, construction metals and, eventually, the atmosphere. Conventional desulfurization systems operate at low temperatures, generally below 200°F, and involve absorber stripper systems, which often remove CO_2 as well as H_2S . The primary objective of this project is the development of a high-temperature, regenerable desulfurization process for coal-derived gas. Such a system would desulfurize coal gas at temperatures of 1000-1200°F, thereby eliminating the need to cool and reheat the fuel gas for uses such as molten carbonate fuel cells (MCFC) operating at temperatures around 1200°F. This type of system would improve the overall thermal efficiency of a power plant, and would also simplify the hardware required.

The development of this process was initiated on a lab scale and is currently being tested at bench scale. This report outlines the construction, installation, and initial testing of the scaled-up sidestream test unit installed on a sidestream of the DOE/METC fixed-bed gasifier.

2: LAB-SCALE TESTING - ANALYSIS OF TEST RESULTS

Lab-scale testing using the 2-inch-diameter reactor was carried out on two zinc ferrites manufactured by United Catalysts: L-1443 and L-1504. Of the 29 tests conducted during this reporting period, 24 were done for model development by Dr. Schrodtt of the University of Kentucky. In addition, five tests were conducted using United Catalysts L-1504 zinc ferrite.

2A: Zinc Ferrite Testing for Model Development

In order to provide complete breakthrough curves for model development, a series of tests was conducted on United Catalysts L-1443 zinc ferrite at various temperatures, linear velocities, H_2S inlet concentrations, and two steam levels. Table 1 summarizes the testing conditions used in this series of tests. Appendix A contains the test summaries for these tests, and Appendix B contains plots of test data.

Figure 1 shows that the breakthrough curves at 1000°, 1200°, and 1400°F are consistent with previous test results. Higher operating temperatures result in shorter breakthrough curves, possibly due to the effect of sintering. Figure 2 shows similar tests carried out at a lower linear velocity, which results in longer plateaus, as expected. However, the shapes of the curves remain the same. Figure 3 illustrates the sulfur concentration in the reactor after Test 127. The concentration profile is typical, and was obtained by sampling pellets from various points in the reactor after completion of the test to 50 ppm breakthrough.

2B: L-1504 Zinc Ferrite Testing

The L-1504 zinc ferrite manufactured by United Catalysts was chosen to be tested in the sidestream test unit. Two sulfidations and two regenerations were conducted to check the performance of this sorbent prior to testing on the larger sidestream test unit. Performance during the two sulfidations appeared reasonable, although the second sulfidation was a little poorer than the first. This type of result has been observed in past tests, and additional multiple sulfidations would have been required to determine if a

loss of capacity occurred due to sintering. Figure 4 shows the breakthrough curves for these tests.

TABLE 1: Zinc Ferrite Testing for Model Development

TEST NUMBER	H ₂ S INLET CONCENTRATION PERCENT	LINEAR VELOCITY cm/sec	STEAM PERCENT	TEMPERATURE °F
103	6.2	16	0	1000
105	5.6	17.8	10	1000
107	5.6	17.8	10	1200
113	5.6	17.8	10	1400
115	5.6	10	10	1000
117	5.6	10	10	1200
119	5.6	10	10	1400
121	3.1	17.8	10	1000
123	3.1	17.8	10	1200
125	3.1	17.8	10	1400
127	5.6	10	10	1000

FIGURE 1

ZINC FERRITE SULFIDATION

TESTS 105, 107, 113 L-1443 UNITED CATALYSTS

FIGURE 2

ZINC FERRITE SULFIDATION

TESTS 115, 117, 119 L-1443 UNITED CATALYSTS

FIGURE 3

ZINC FERRITE SULFUR LOADING

TEST 127

FIGURE 4

ZINC FERRITE SULFIDATION

TESTS 129,131 (L1504)

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3: DESIGN MODIFICATIONS TO LAB-SCALE TEST EQUIPMENT

During the experimental testing of metal oxide sorbents, a certain amount of sulfur corrosion occurred within the reactor and sulfur accumulated on the reactor walls. When testing desulfurization sorbents at ppm levels of hydrogen sulfide, the reactor wall must be inert so that it will neither pick up nor emit sulfur. Therefore, both a quartz reactor and a metal reactor were designed and fabricated for further testing. The internal surfaces of the metal reactor were alonized in order to make the metal surface resistant to sulfur corrosion and to minimize sulfur pickup by the reactor walls. It was expected that testing conducted with both quartz and alonized metal reactors would reveal any differences between them. The metal reactor is more convenient because it is unbreakable; however, the quartz reactor is more useful for initial testing in a fluidized bed operation. Part of the test hardware was also redesigned to be compatible with the trace sulfur-handling capability of the reactors.

3A: The Test Unit - An Overview

The system flow schematic is shown in Drawing No. E830033, and a process and instruments diagram is illustrated in Drawing No. E830217, contained in Appendix C. A mixture of gases is obtained by metering N_2 , CO, CO_2 , H_2 , CH_4 , and air from individual cylinders. This allows the flexibility of simulating a variety of gas mixtures typical of several types of gasifiers. Water is added to the mixture by metering liquid water from a pressurized tank. The combined mixture is then preheated to approximately 250°F, primarily to vaporize the water in the mixture. A separate gas manifold supplies a metered mixture of H_2S and COS. The line that carries H_2S and COS to the reactor inlet is made of teflon to prevent sulfiding of metal surfaces and loss of sulfur. Both the quartz and the alonized metal reactors contain an integral preheater section and the sorbent to be tested. The reactor is situated inside an electrically heated furnace. The gas mixture, containing H_2S and COS, is heated to operating temperature in the integral preheater, fed at the bottom of the reactor, passes through the sorbent, and is cooled while passing

through the upper part of the reactor from about 1000°F to less than approximately 400°F, the melting point of teflon. The gas then exits the reactor and is piped through teflon lines to a shell-and-tube, water-cooled condenser and knockout vessel. The water contained within the gas is thus condensed. The gas continues to be sampled by a gas chromatograph and is vented.

3B: Quartz Reactor

The quartz reactor is 2.24 inches in diameter and 60 inches long. It contains a 12-inch-long integral preheater section filled with quartz wool, which is separated from the sorbent section by a quartz disc. A quartz thermowell supported in the center of the reactor contains six thermocouples which measure the temperature along the reactor's length. The reactor is supported by two discs attached on each end of the reactor. Process gas inlet and exit connections are made by a teflon plug attached to the beaded ends of the quartz reactor by metal clamps. Drawing No. D830034 (Appendix C) depicts details of this construction. The reactor temperature is maintained by a three-zone clamshell furnace in which the reactor is suspended.

3C: Metal Reactor

The metal reactor, constructed of No. 316 stainless steel, is 2 inches in diameter and 60 inches long. It contains a 12-inch-long integral preheater. All internal surfaces of the metal reactor are alonized to prevent sulfur corrosion and pickup. A No. 316 stainless steel thermowell, with six thermocouples at various points along its length, is located at the center of the reactor. The metal reactor is supported by a collar attached at each end, and is suspended on a rack (Drawing No. E830073) that is also designed to fit the quartz reactor. Drawing No. D830036 (Appendix C) depicts construction details of the metal reactor. The reactor temperature is maintained by the three-zone clamshell furnace described for the quartz reactor.

3D: Preheater

The preheater vaporizes the liquid water and preheats the gas mixture

which is metered to simulate a gasifier gas. The preheater consists of stainless steel tubing sandwiched between two 12" x 12" carbon steel blocks; each block has four cartridge heaters evenly spaced along its center line. The temperature of each block is monitored and overheating is prevented by a thermocouple wired to a controller. The temperature of gas leaving the preheater is monitored and the eight cartridge heaters are controlled accordingly. The preheater assembly has two inches of insulation and is contained in a metal box open at one end. Drawing No. E830143 (Appendix C) depicts the construction details of the preheater.

3E: Reactor Furnace

The reactor furnace maintains reactor temperatures at high levels and preheats the inlet gas mixture to operating temperature. The three-zone hinged furnace allows flexibility in temperature control, as each zone has separate controls. The center zone of the furnace is 24 inches long and each end zone is 6 inches long. The furnace is rated for operation up to 1000°C and 4300 watts.

3F. Condensate Collection System

The liquid condensed in the condenser is collected in a knockout vessel. A three-float system in the knockout vessel serves to monitor the liquid level, and to control three (3) solenoid valves which are automatically actuated to drain liquid from the knockout vessel. Minimum and maximum level indications prevent draining too much condensate or overfilling the knockout vessel by actuating designated solenoid valves. The knockout vessel is shown in Drawing No. D830305 in Appendix C.

4: CONSTRUCTION AND INSTALLATION OF SKID-MOUNTED TEST UNIT ON SIDESTREAMS OF METC GASIFIERS

The design parameters of the various components of the hot gas desulfurization test unit were outlined in the previous annual report (December 1982). During this reporting period, the test unit was fabricated and assembled at the DOE/METC machine shop, and was then transported to Building 4 and installed at its operating location.

The first step of construction was fabrication of the 12' x 4' skid to provide structural support for the test unit. The cage supporting the reactor furnace and reactor was then added, and the four-zone Mellen furnace was installed on this framework. The six-inch-diameter incoloy 800H reactor was later mounted to fit inside the hinged furnace on the same framework, and space was allowed for opening the furnace.

The two preheater boxes were then mounted on channel supports. Face plates were fabricated to cover the open ends of the preheaters and to provide a mounting surface for the preheater coils. The two condensers were mounted and the process of installing piping between them was initiated. The sliding disc valves, control valves, flow transmitters, pressure transmitters, and thermocouples were mounted. The control valves, flow transmitters, and orifice runs for regeneration steam, air, and N₂ were installed. Interface boxes for thermocouples and various control units were installed at various locations as needed. Plates 1, 2, and 3 show the test unit under construction.

The 7000-pound assembled unit was moved from Building 3 to Building 4 by the use of two forklifts. A 10-ton crane hoisted the unit to the mezzanine floor, in the northeast corner of Building 4.

Parallel to this effort, the pipeline for the hot gas sidestream was installed. The hot gasifier gas was tapped at the gasifier cyclone exit; an existing 8-inch Kamyr valve was utilized as the on/off valve for the sidestream; and a 1-1/4-inch 309 stainless steel line was installed from that point. A secondary cyclone was installed for additional particulate removal. The pipeline is about 120 feet long, reaching inside Building 4 to the hot gas desulfurization test unit. Eighteen tubular heaters, wired in sets of three in a Wye configuration, were strapped to the pipeline to heat it along its

PLATE 1: HOT GAS DESULFURIZATION TEST UNIT UNDER CONSTRUCTION

PLATE 2: HOT GAS DESULFURIZATION TEST UNIT UNDER CONSTRUCTION

PLATE 3: HOT GAS DESULFURIZATION TEST UNIT: VALVES, CONTROLS. INSTRUMENTATION

entire length. Six controllers were used, each controlling a set of three heaters by reading the pipe temperature at midpoint of the three heaters. In addition, the pipeline temperatures are monitored by thermocouples at approximately 10-foot intervals along the line, and these temperatures are read and recorded on a strip chart by a 24-point recorder. The pipeline was insulated with four inches of insulation and was covered with aluminum sheeting.

Connections were made between the test unit and pipeline. The pipeline from the gasifier was connected to the test unit with a bypass to vent through a rupture disc. Steam, air, nitrogen, water, and water drain lines were attached. All electrical connections were made to power the furnaces and heaters. Air lines to valve actuators were installed and all instrumentation wiring was completed, including the ADACS (automatic data acquisition system) hookup. Sampling lines were installed for gas analysis and the baseline chromatograph was set up to operate near the test unit. Calibration gas was supplied initially by bottled gas. The condenser knockout vessels were fabricated and installed, and load cells and two 55 gallon drums were installed for collecting and weighing condensate from the test unit.

Upon completion of plumbing, electrical connections, and instrumentation, the test unit was pressurized so any leaks (i.e., in the piping, connections, fittings) could be located. All threaded connections were backwelded to eliminate leaks and the unit was made as leak-tight as possible. It appeared that some leakage in the area of valve packings could not be avoided. However, the leakage rate was considered tolerable as good ventilation was available. Plate 4 shows the test unit installed at the test location.

PLATE 4: HOT GAS DESULFURIZATION TEST UNIT AT TEST LOCATION

5: TESTING OF SKID-MOUNTED TEST UNIT ON SIDESTREAMS OF METC GASIFIERS,
GASIFIER RUN 101

The installation of the sidestream test unit in a sidestream of the DOE/METC fixed-bed gasifier provided the opportunity to test the hot gas desulfurization process on a real coal gas with all the associated tars, oils, particulates, and additional sulfur compounds present in coal gas. The primary differences between the lab-scale and sidestream testing are outlined in Table 2. The purpose of the scaled-up testing was to demonstrate the capability of this process to desulfurize gasifier gas to ppm levels at temperatures of 1000-1200°F. Items of particular interest were as follows:

- Effect of tars
- Effect of particulates
- Effect of sorbent on organic sulfur compounds such as thiophene
- Regeneration characteristics of the larger reactor

During this reporting period two series of tests were conducted on the DOE/METC gasifier. These tests will be identified as Gasifier Run 101 (June, 1983) and Gasifier Run 102 (September 1983). Test results for Gasifier Run 102 will not be discussed in this report and will be deferred to future reports.

Gasifier Run 101 comprised the first series of tests on the sidestream test unit. The objectives were as follows:

1. To shake down the hot gas desulfurization skid-mounted test unit and put it into operating condition on a sidestream of the fixed-bed gasifier.
2. To conduct a sulfidation test on hot producer gas to determine effects, if any, of tar vapors, particulates, and other trace constituents.
3. To conduct a regeneration with minimum effect on the integrity of the sorbent.
4. To conduct a second sulfidation on producer gas to determine any loss of desulfurization capacity, after regeneration.

During Gasifier Run 101, five tests were conducted on the sidestream test

TABLE 2: Operating Conditions of Lab-Scale and Sidestream Test Unit

CONDITIONS	LAB-SCALE TEST UNIT	SIDESTREAM TEST UNIT
<u>Sulfidation:</u>		
Gas Composition:		
1. Majors (CO, CO ₂ , H ₂ , N ₂ , CH ₄)	Gases from cylinders simulating air-blown Lurgi gasifier	Actual DOE/METC fixed-bed gasifier; both air-blown and oxygen-blown compositions
2. Moisture	27% as added water	4-5% on air-blown gas 53% on oxygen-blown gas
3. Sulfur	2.7% H ₂ S from cylinder	2300-3800 ppm H ₂ S (on air-blown) plus COS, thiophenes, CS ₂ and others
Tars	None	Typical of DOE/METC fixed-bed gasifier
Particulates	None	Typical of DOE/METC fixed-bed gasifier after primary and secondary cyclone
Pressure	Atmospheric	Gasifier pressure (91-160 psig)
<u>Regeneration:</u>		
Gas Composition:		
1. Steam	50-75%	80%
2. Air	25-50%	10-20%
3. Nitrogen	0	0-8%
Pressure	Atmospheric	5 psig

unit, outlined in Table 3. Appendix D contains the data obtained from the five tests outlined in Table 3. Plates 5 and 6 illustrate the flow pattern used for absorption and regeneration.

TABLE 3: Hot-Gas Desulfurization Tests - Gasifier Run 101

TEST NO./ TYPE	DURATION (Hrs)	GASIFIER COAL TYPE	GASIFIER OXIDANT	HGD REACTOR TEMP. (°F)	HGD REACTOR PRESSURE (PSIG)	INLET SULFUR (ppm H ₂ S)	EXIT SULFUR (ppm H ₂ S)
001/ Sulfidation	29.6	Arkwright	Air	1025	160	2300	2
002/ Regeneration	14.4						
003/ Sulfidation	24.6	Arkwright	Air	1018	143	3600	1.6
004/ Regeneration	18.8						
005/ Sulfidation	23.2	Arkwright	Air/O ₂	1012	91	3800	3

5A: Desulfurization Capacity

Lab-scale testing of zinc ferrite (reported in this and previous reports) showed that the level to which H₂S concentration in the treated gas can be reduced is limited by equilibrium constraints. Tables 4 and 5 show the levels to which H₂S can be reduced using zinc oxide and iron oxide at various temperatures and for various steam concentrations. Since zinc ferrite contains both zinc oxide and iron oxide, the lower equilibrium H₂S levels shown for zinc oxide are obtained.

Test results from sidestream Test 001 are shown in Figure 5. The results indicate that equilibrium H₂S levels are obtained at approximately 1 to 3 ppm. These results match the results previously obtained on lab-scale tests on

PLATE 5: HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT ABSORPTION

LEGEND

HOT PRODUCER GAS WITH SULFUR
HOT PRODUCER GAS WITHOUT SULFUR
COLDER PRODUCER GAS WITHOUT SULFUR
COLDER PRODUCER GAS WITHOUT SULFUR

PLATE 6: HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT REGENERATION

LEGEND

- III REGENERATION GAS
- III REGENERATION GAS WITH SO₂

GENERAL GAS-SOLID EQUILIBRIUM CODE

THERMODYNAMIC DATA

ZNS HEAT OF FORMATION (25 C) =	-49.800	KCAL MOL-1	MILLS
H2O HEAT OF FORMATION (25 C) =	-57.795	KCAL MOL-1	KUBASCHEWSKI
ZNO HEAT OF FORMATION (25 C) =	-83.800	KCAL MOL-1	KUBASCHEWSKI
H2S HEAT OF FORMATION (25 C) =	-4.900	KCAL MOL-1	MILLS
ZNS ENTROPY (25 C) =	13.800	CAL K-1 MOL-1	MILLS
H2O ENTROPY (25 C) =	45.106	CAL K-1 MOL-1	KUBASCHEWSKI
ZNO ENTROPY (25 C) =	18.430	CAL K-1 MOL-1	KUBASCHEWSKI
H2S ENTROPY (25 C) =	40.150	CAL K-1 MOL-1	MILLS

DATA SOURCE

HEAT CAPACITY EQUATION IS

$$CP = A + BT + CT^2 + DT^3 + ET^{-2}$$

CAL K-1 MOL-1

HEAT CAPACITY COEFFICIENTS

	A	B	C	D	E	SOURCE
ZNO	11.71	0.00122000	0.000000000	0.000000000	-210000.00	KUBASCHEWSKI
H2S	7.81	0.00205000	0.000000000	0.000000000	-46000.00	MILLS
ZNS	11.77	0.00126000	0.000000000	0.000000000	-116000.00	MILLS
H2O	7.17	0.00256000	0.000000000	0.000000000	8000.00	KUBASCHEWSKI

TEMPERATURE
DEGREES C

DELTA G
CAL/GMOLE

EQUILIBRIUM
CONSTANT

EQUILIBRIUM H2S LEVEL IN PPM
PERCENT STEAM

			5%	10%	15%	20%	25%	30%
200.	-17802.21	16.80E+07	20.8E-05	50.5E-05	80.3E-05	11.0E-04	14.0E-04	17.0E-04
250.	-17778.15	26.85E+06	18.6E-04	37.2E-04	55.0E-04	74.5E-04	93.1E-04	11.2E-03
300.	-17753.17	50.04E+05	84.7E-04	16.0E-03	25.4E-03	33.0E-03	42.3E-03	50.8E-03
350.	-17726.84	16.54E+05	30.2E-03	60.5E-03	90.7E-03	0.12	0.15	0.18
400.	-17698.91	55.90E+04	80.4E-03	0.10	0.27	0.36	0.45	0.54
450.	-17659.21	21.03E+04	0.23	0.46	0.68	0.91	1.1	1.4
500.	-17637.63	96.06E+03	0.52	1.0	1.5	2.1	2.6	3.1
550.	-17604.13	47.20E+03	1.1	2.1	3.2	4.2	5.3	6.3
600.	-17568.67	25.01E+03	2.0	4.0	6.0	8.0	10.	12.
650.	-17531.23	14.16E+03	3.5	7.1	11.	14.	18.	21.
700.	-17491.82	84.00E+02	5.0	12.	18.	24.	29.	35.
750.	-17450.44	53.45E+02	9.4	19.	28.	37.	47.	56.
800.	-17407.11	35.11E+02	14.	28.	43.	57.	71.	85.
850.	-17361.84	23.02E+02	21.	42.	63.	84.	10.5E+01	12.5E+01

FORTRAN STOP
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TABLE 4: ZINC OXIDE EQUILIBRIUM

*
H2S GAS-SOLID EQUILIBRIUM CODE - EQUILIBRIUM CONSTANT METHOD
REACTION IS FE3O4 + 3H2S + H2 GOING TO 3FES + 4H2O

THERMODYNAMIC DATA				DATA SOURCE	
FES	HEAT OF FORMATION (25 C) =	-24.000	KCAL MOL-1	KUBASCHIEWSKI	
H2O	HEAT OF FORMATION (25 C) =	-57.000	KCAL MOL-1	CRC	
FE3O4	HEAT OF FORMATION (25 C) =	-266.000	KCAL MOL-1	KUBASCHIEWSKI	
H2S	HEAT OF FORMATION (25 C) =	-4.015	KCAL MOL-1	CRC	
H2	HEAT OF FORMATION (25 C) =	0.000	KCAL MOL-1	CRC	
FES	ENTROPY (25 C) =	14.410	CAL K-1 MOL-1	KUBASCHIEWSKI	
H2O	ENTROPY (25 C) =	45.110	CAL K-1 MOL-1	CRC	
FE3O4	ENTROPY (25 C) =	35.000	CAL K-1 MOL-1	KUBASCHIEWSKI	
H2S	ENTROPY (25 C) =	40.150	CAL K-1 MOL-1	CRC	
H2	ENTROPY (25 C) =	31.210	CAL K-1 MOL-1	CRC	
HEAT CAPACITY EQUATION IS CP = A + BT + CT2 + DT3 + ET-2				CAL K-1 MOL-1	

HEAT CAPACITY COEFFICIENTS

	A	B	C	D	E	RANGE	SOURCE	TRANSFORMATION
FE3O4	21.88	0.04820000	0.00000000	0.00000000	0.00	200. - 900.	KUBASCHEWSKI	ALPHA
FE3O4	48.00	0.00000000	0.00000000	0.00000000	0.00	900. - 1800.	KUBASCHEWSKI	BETA
H2S	7.01	0.00206000	0.00000000	0.00000000	-46000.00	200. - 2000.	KUBASCHEWSKI	
H2	6.52	0.00070000	0.00000000	0.00000000	12000.00	200. - 3000.	KUBASCHEWSKI	
FES	5.10	0.02640000	0.00000000	0.00000000	0.00	200. - 411.	KUBASCHEWSKI	ALPHA
FES	17.40	0.00000000	0.00000000	0.00000000	0.00	411. - 500.	KUBASCHEWSKI	BETA
FES	12.20	0.00230000	0.00000000	0.00000000	0.00	500. - 1460.	KUBASCHEWSKI	GAMMA
H2O	7.17	0.00256000	0.00000000	0.00000000	0000.00	200. - 2500.	KUBASCHEWSKI	

TEMPERATURE DEGREES C	DELTA G CAL/GMOLE	EQUILIBRIUM CONSTANT	EQUILIBRIUM H2S LEVEL IN PPM PERCENT STEAM					
			5%	10%	15%	20%	25%	30%
350.	-29130.37	16.55E+00	12.	31.	53.	70.	10.6E+01	13.5E+01
400.	-29053.06	40.50E+00	10.	47.	80.	11.7E+01	15.0E+01	20.2E+01
450.	-30527.46	16.00E+00	26.	67.	11.4E+01	16.0E+01	22.6E+01	28.0E+01
500.	-31147.46	63.90E+07	37.	92.	15.0E+01	23.2E+01	31.3E+01	39.0E+01
550.	-31710.64	26.34E+07	40.	12.4E+01	21.3E+01	31.2E+01	42.0E+01	53.6E+01
600.	-32214.22	11.60E+07	65.	16.3E+01	27.0E+01	41.0E+01	55.2E+01	70.4E+01
650.	-32660.07	54.12E+06	83.	21.0E+01	36.0E+01	52.0E+01	71.2E+01	90.0E+01
700.	-33086.10	27.01E+06	10.5E+01	26.5E+01	45.4E+01	66.7E+01	80.8E+01	11.4E+02
750.	-33400.50	14.33E+06	13.0E+01	32.7E+01	56.1E+01	82.3E+01	11.1E+02	14.1E+02
800.	-33800.50	03.20E+05	15.7E+01	30.6E+01	60.1E+01	90.0E+01	13.4E+02	17.2E+02
850.	-34200.77	47.13E+05	10.0E+01	47.3E+01	81.3E+01	11.0E+02	16.1E+02	20.5E+02

FORTRAN STOP

TABLE 5: IRON OXIDE EQUILIBRIUM

FIGURE 5: H₂S BREAKTHROUGH CURVE
ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 001

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simulated gas, indicating that tars and oils and/or particulates in the coal gas do not inhibit the level to which H_2S is reduced in the gas. Figure 6 shows the breakthrough curve for Tests 001 and 003. Test 003 was carried out after a regeneration. The data show that H_2S is reduced to the same low levels after a regeneration. However, the data also indicate that it took approximately 4 hours to obtain less than 4 ppm H_2S . This is attributed to sulfur remaining in the sorbent after regeneration, and is a function of the quality and duration of the regeneration. The time to breakthrough also appears to have been reduced from approximately 29 hours to 22 hours. However, during Test 003, the sulfur concentration of the inlet gas was higher (0.36% during Test 003 versus 0.23% during Test 001). The total sulfur removed (as calculated by inlet sulfur minus exit sulfur) was actually higher for Test 003 (8.06 lb. S_8) than for Test 001 (6.49 lb. S_8).

Removal of thiophene from the gasifier gas is shown in Figure 7. Thiophene levels of 4-8 ppm were measured at the reactor exit and appeared to increase during breakthrough, as with the other sulfur compounds (H_2S and COS). COS levels were typically below 1 ppm prior to breakthrough.

Sulfidations that followed the process of regeneration exhibited an SO_2 leakage phenomenon. This is attributed to unregenerated sulfide, or to sulfate in the reactor which released SO_2 when exposed to a reducing gas. Figure 8 shows the level and duration of SO_2 leakage during Test 005. The figure shows that the SO_2 level dropped to 1 ppm within the first hour.

5B: Effects of Sorbent on Components in the Coal Gas

The primary purpose of the zinc ferrite sorbent is to remove and lower the sulfur content of the coal gas being treated. However, it was observed that in addition to removing sulfur compounds, zinc ferrite catalyzed the shift reaction. Apparently, the iron in zinc ferrite was the catalyst. Figures 9 through 11 show the extent of shift that occurred during Test 003. The initial high degree of shift was a 66°F approach to equilibrium based on a reactor temperature of 1018°F. The data indicated that most of the shift occurred during the first half of the sulfidation, and that the amount of shift gradually decreased as the sorbent was sulfided. This is because iron catalyzes the shift reaction but iron sulfide does not; i.e., as iron is sulfided by the sulfur compounds in the coal gas, its shift-catalyzing ability

FIGURE 6: H₂S BREAKTHROUGH CURVE
ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TESTS 001 AND 003

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FIGURE 7: THIOPHENE BREAKTHROUGH CURVE

ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TESTS 001 AND 003

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FIGURE 8

ZINC FERRITE SULFIDATION

INITIAL H₂S AND SO₂ CONCENTRATION

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PERCENT CO
BENDIX CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STOP TIME 15:00:00 06/27/1983

FIGURE 9: EFFECT OF SHIFT REACTION ON CO CONCENTRATION

PERCENT H₂
BENDIX CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STOP TIME 15:00:00 06/27/1983

FIGURE 10: EFFECT OF SHIFT REACTION ON H₂ CONCENTRATION

PERCENT CO₂
BENDIX CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STOP TIME 15:00:00 06/27/1983

FIGURE 11: EFFECT OF SHIFT REACTION ON CO₂ CONCENTRATION

is reduced. This phenomenon may be taken as an indication that the iron in the zinc ferrite is sulfided sufficiently to block any iron oxide surfaces. At this point, sulfur compounds have to diffuse through the sulfide layer, or be removed by the zinc oxide contained in the zinc ferrite.

5C: Sorbent Characterization

In conducting the three sulfidations and two regenerations during Gasifier Run 101, the sorbent integrity over several cycles was of interest. In order to characterize the sorbent before and after these tests, the following analyses were carried out.

- Total carbon
- Total sulfur
- BET surface area
- Crush strength

Table 6 outlines the data obtained on samples of the sorbent removed from the reactor at 10-inch intervals (except for one at midbed) after the third sulfidation. The sorbent was in the sulfided state as indicated by the total sulfur numbers. A high degree of sulfur capture is indicated by the total sulfur concentrations of 25-31 weight-percent for approximately the first half of the reactor, whereas the second half of the reactor picked up less sulfur since the concentration of sulfur in the gas was already greatly reduced. This is shown graphically in Figure 12. The utilization of the sorbent's capacity for removing sulfur is shown by these data. The factors affecting the utilization of sorbent capacity are: sulfur breakthrough concentration, space velocity, temperature, and reactor geometry. When the sorbent was removed from the reactor, it was observed that carbon formation had occurred. The sorbent was therefore analyzed for total carbon, and the data indicated a relatively low and constant level of carbon in the sorbent at an average level of 2.6 percent by weight. When the sorbent was removed from the reactor, in addition to the sorbent pellets, 2408 grams of black dust were removed. This was attributed to sorbent attrition as well as carbon formation. Since the desulfurization reaction is primarily a surface reaction, the surface-area properties of the sorbent must be maintained (as far as possible) to ensure

TABLE 6: SORBENT ANALYSIS AFTER TEST 005, ZINC FERRITE L-1504, UNITED CATALYSTS

BED HEIGHT (INCHES)	TOTAL CARBON (% WT)	TOTAL SULFUR (% WT)	BET SURFACE AREA (SQ M/G)	DENSITY ₃ (G/CC)	PORE VOLUME (CC/G) ⁵	CRUSH STRENGTH (Kg DWL) ± S.D. ⁷
0 ¹	1.97	29.88	1.4332	4.366	0.002152	7.0 ± 3.29
6	1.88	31.41	1.4903	4.548	0.002068	8.4 ± 3.55
16	2.58	27.78	2.0936	4.261	0.004003	7.3 ± 3.54
23	2.89	25.92	2.4263	4.326	0.004616	8.1 ± 3.09
26	3.48	28.97	2.2432	4.344	0.005324	4.4 ± 2.09
36	2.37	7.29	2.2978	4.063	0.003678	4.2 ± 2.57
46 ²	3.27	8.60	3.1138	5.141	0.005498	2.4 ± 1.76
FRESH --METC SORBENT --UCI	0	0.13	3.1305 3.2	6.159 85 ⁴	0.004316 0.25 ⁶	6.3 ± 2.8 7.2
ABSORBER FINES	6.93	26.29				

NOTES:

- 1 Bottom/Inlet
- 2 Top/Outlet
- 3 Helium Density
- 4 Bulk Density (Lbs/Cu Ft)
- 5 Nitrogen Adsorption
- 6 Mercury Porosimetry
- 7 Crush strength data are average and standard deviation of 15 measurements

FIGURE 12

SORBENT PELLET ANALYSIS

GASIFIER RUN 101 - THREE SULFIDATIONS

that desulfurization capacity is not lost with consecutive cycles. Table 6 shows that the surface area at the reactor exit was almost identical to that of the fresh sorbent. This is encouraging, and indicates that no loss of surface area occurred after three sulfidations and two regenerations. In fact, the pore volume for sorbent at the exit was actually somewhat higher than that of the fresh sorbent. This could be due to the expansions and contractions that occur between sulfidations and regenerations, and cracks that develop in the sorbent.

Crush strength was also measured in an attempt to learn more about possible attrition problems with the sorbent. The crush strength data obtained do not show conclusively that sorbent strength was reduced; however, the strength of the sorbent at the reactor exit was lower than that of the fresh sorbent. The sulfided sorbent at the inlet half of the reactor was stronger than the fresh sorbent. Since the standard deviation for these data is so high, it is difficult to draw firm conclusions regarding any loss of strength of the sorbent.

Table 7 shows the mineral analysis conducted on the sorbent at the same intervals as in Table 6. The mineral analysis shows the relative amounts of zinc sulfide and iron sulfide (FeS), as well as the amount of unconverted oxides. For approximately the first half of the reactor, only 12 percent (average) iron oxide was unconverted. This indicates that 88 percent of the metal oxide (present as zinc oxide and iron oxide) was converted to a sulfide. Essentially all the zinc oxide in the first half of the reactor was converted. In the last ten inches of the reactor no iron sulfide was present; and only zinc sulfide was detected. This is explained by the fact that the concentration of H_2S and COS in the gas was below the equilibrium level for iron oxide; therefore, no iron oxide was reacted. However, zinc oxide did react, and served to reduce the sulfur level in the gas to zinc oxide equilibrium levels. The analysis indicates that the last ten inches of the bed acted as a zinc oxide guard, and did not utilize the iron oxide in the sorbent. It may be desirable to pack the reactor with pure zinc oxide for the last ten inches to increase the sulfur-removing capability of that portion of the reactor, and to increase the utilization of the iron oxide.

TABLE 7: MINERAL ANALYSIS (in % TII¹) AFTER TEST 005, ZINC FERRITE L-1504,
UNITED CATALYSTS

BED HEIGHT INCHES	HEMATITE Fe ₂ O ₃	FRANKLINITE ZnFe ₂ O ₄ and/or MAGNETITE Fe ₃ O ₄ ⁴	ZINCITE ZnO	ALPHA-ZnS	BETA ZnS	PYRRHOTITE FeS
0 ²	-	10	Trace	13	51	25
6	-	4	-	12	54	29
16	-	18	Trace	19	36	27
23	-	10	-	19	42	28
26	-	19	Trace	24	25	32
36	-	68	14	10	7	-
46 ³	-	70	18	9	4	-
Fresh Sorbent	4	89	6	-	-	-

NOTES:

1. % TII = Percent Total Integrated Intensity: directly proportional to the concentration of the crystalline phase. Amorphous phase is not accounted for.
2. Bottom/Inlet
3. Top/Outlet
4. Franklinite and magnetite have the same crystal structure and therefore cannot be readily distinguished by the diffraction pattern.

6: PROCESS SIMULATION

Process simulation during this reporting period consisted primarily of equilibrium process calculations. Table 8 shows the equilibrium values of H_2S expected at various reactions with iron oxide (Fe_3O_4) in the presence of hydrogen. These were calculated by determining the equilibrium constant for the reaction shown in the table. In order to reach the high temperatures desired, two transition points were encountered for Fe_3O_4 and three transition points for FeS . These were incorporated into the calculation in order to determine equilibrium H_2S values for various steam concentrations at various temperatures for 20 percent hydrogen in the gas. The results indicate that at $550^\circ C$ iron oxide can lower H_2S levels to 57 ppm at 5 percent moisture up to a high of 620 ppm at 30 percent moisture.

A similar calculation was carried out for the reaction:

Table 9 shows the results obtained. As expected, ZnO does not reduce HCl to low levels at high temperatures, although less than 50 ppm of HCl can be obtained at $200^\circ C$. This calculation was made to verify that the sorbent will not appreciably reduce chloride levels in gasifier gas.

H2S GAS-SOLID EQUILIBRIUM CODE - EQUILIBRIUM CONSTANT METHOD
REACTION IS FE3O4 + 3H2S + H2 GOING TO 3FES + 4H2O

THERMODYNAMIC DATA

DATA SOURCE

FES	HEAT OF FORMATION (25 C) =	-24.000	KCAL MOL-1	KUBASCHIEWSKI
H2O	HEAT OF FORMATION (25 C) =	-57.800	KCAL MOL-1	CRC
FE3O4	HEAT OF FORMATION (25 C) =	-266.900	KCAL MOL-1	KUBASCHIEWSKI
H2S	HEAT OF FORMATION (25 C) =	-4.815	KCAL MOL-1	CRC
H2	HEAT OF FORMATION (25 C) =	0.000	KCAL MOL-1	CRC
FES	ENTROPY (25 C) =	14.410	CAL K-1 MOL-1	KUBASCHIEWSKI
H2O	ENTROPY (25 C) =	45.110	CAL K-1 MOL-1	CRC
FE3O4	ENTROPY (25 C) =	35.000	CAL K-1 MOL-1	KUBASCHIEWSKI
H2S	ENTROPY (25 C) =	49.150	CAL K-1 MOL-1	CRC
H2	ENTROPY (25 C) =	31.210	CAL K-1 MOL-1	CRC

HEAT CAPACITY EQUATION IS

$$CP = A + BT + CT^2 + DT^3 + ET^{-2}$$

CAL K-1 MOL-1

HEAT CAPACITY COEFFICIENTS

	A	B	C	D	E	RANGE	SOURCE	TRANSFORMATION
FE3O4	21.88	0.04020000	0.00000000	0.00000000	0.00	200.- 900.	KUBASCHEWSKI	ALPHA
FE3O4	48.00	0.00000000	0.00000000	0.00000000	0.00	900.- 1800.	KUBASCHEWSKI	BETA
H2S	7.81	0.00206000	0.00000000	0.00000000	-46000.00	200.- 2000.	KUBASCHEWSKI	
H2	6.52	0.00070000	0.00000000	0.00000000	12000.00	200.- 3000.	KUBASCHEWSKI	
FES	5.10	0.02640000	0.00000000	0.00000000	0.00	200.- 411.	KUBASCHEWSKI	ALPHA
FES	17.40	0.00000000	0.00000000	0.00000000	0.00	411.- 500.	KUBASCHEWSKI	BETA
FES	12.20	0.00230000	0.00000000	0.00000000	0.00	500.- 1468.	KUBASCHEWSKI	GAMMA
H2O	7.17	0.00256000	0.00000000	0.00000000	8000.00	200.- 2500.	KUBASCHEWSKI	

TEMPERATURE DEGREES C	DELTA G CAL/GMOLE	EQUILIBRIUM CONSTANT	EQUILIBRIUM H2S LEVEL IN PPM PERCENT STEAM					
			5%	10%	15%	20%	25%	30%
350.	-29130.37	16.55E+00	12.	31.	53.	70.	10.6E+01	13.5E+01
400.	-29853.86	49.50E+00	18.	47.	80.	11.7E+01	15.0E+01	20.2E+01
450.	-30527.46	16.80E+00	26.	67.	11.4E+01	16.8E+01	22.6E+01	28.0E+01
500.	-31147.46	63.00E+07	37.	92.	15.8E+01	23.2E+01	31.3E+01	39.9E+01
550.	-31710.64	26.34E+07	49.	12.4E+01	21.3E+01	31.2E+01	42.0E+01	53.6E+01
600.	-32214.22	11.60E+07	65.	16.3E+01	27.0E+01	41.0E+01	55.2E+01	70.4E+01
650.	-32660.87	54.12E+06	83.	21.0E+01	36.0E+01	52.0E+01	71.2E+01	90.8E+01
700.	-33086.10	27.01E+06	10.5E+01	26.5E+01	45.4E+01	66.7E+01	89.0E+01	11.4E+02
750.	-33498.50	14.33E+06	13.0E+01	32.7E+01	56.1E+01	82.3E+01	11.1E+02	14.1E+02
800.	-33890.50	80.20E+05	15.7E+01	39.6E+01	68.1E+01	99.0E+01	13.4E+02	17.2E+02
850.	-34200.77	47.13E+05	18.0E+01	47.3E+01	81.3E+01	11.0E+02	16.1E+02	20.5E+02

FORTRAN STOP

TABLE 8: HYDROGEN SULFIDE / IRON OXIDE EQUILIBRIUM

HCL GAS-SOLID EQUILIBRIUM CODE

THERMODYNAMIC DATA

ZNCL2 HEAT OF FORMATION (25 C) = -99282.0 CAL MOL-1
H2O HEAT OF FORMATION (25 C) = -57795.0 CAL MOL-1
ZNO HEAT OF FORMATION (25 C) = -83802.0 CAL MOL-1
HCL HEAT OF FORMATION (25 C) = -22063.0 CAL MOL-1

DATA SOURCE

KUBASCHEWSKI
KUBASCHEWSKI
KUBASCHEWSKI
KUBASCHEWSKI

ZNCL2 ENTROPY (25 C) = 26.640 CAL K-1 MOL-1
H2O ENTROPY (25 C) = 45.106 CAL K-1 MOL-1
ZNO ENTROPY (25 C) = 10.430 CAL K-1 MOL-1
HCL ENTROPY (25 C) = 44.643 CAL K-1 MOL-1

KUBASCHEWSKI
KUBASCHEWSKI
KUBASCHEWSKI
KUBASCHEWSKI

HEAT CAPACITY EQUATION IS

$$CP = A + BT + CT^2 + DT^3 + ET^{-2}$$

CAL K-1 MOL-1

HEAT CAPACITY COEFFICIENTS

	A	B	C	D	E	RANGE	SOURCE	TRANSFORMATION
ZNO	11.71	0.00122000	0.00000000	0.00000000	-218000.00	200.- 1600.	KUBASCHEWSKI	
HCL	6.34	0.00110000	0.00000000	0.00000000	26000.00	200.- 2000.	KUBASCHEWSKI	
ZNCL2	14.50	0.00550000	0.00000000	0.00000000	0.00	200.- 501.	KUBASCHEWSKI	MELTING POINT AT 501 K
ZNCL2	24.10	0.00000000	0.00000000	0.00000000	0.00	501.- 1000.	KUBASCHEWSKI	
H2O	7.17	0.00256000	0.00000000	0.00000000	8000.00	200.- 2500.	KUBASCHEWSKI	

TEMPERATURE
DEGREES C

DELTA G
CAL/GMOLE

EQUILIBRIUM
CONSTANT

EQUILIBRIUM HCL LEVEL IN PPM
PERCENT STEAM

			5%	10%	15%	20%	25%	30%
200.	-15855.22	2'.17E+06	40.	60.	84.	67.	10.0E+01	11.0E+01
250.	-14464.72	1'.00E+05	21.2E+01	30.0E+01	36.0E+01	42.5E+01	47.5E+01	52.0E+01
300.	-13075.87	97. 0E+03	71.0E+01	10.1E+02	12.4E+02	14.4E+02	16.0E+02	17.6E+02
350.	-11827.00	14.00E+03	10.0E+02	26.6E+02	32.6E+02	37.7E+02	42.1E+02	46.1E+02
400.	-10670.44	20.35E+02	41.3E+02	50.4E+02	71.5E+02	82.5E+02	92.3E+02	10.1E+03
450.	-9554.66	773.	90.4E+02	11.4E+03	13.0E+03	16.1E+03	18.0E+03	19.7E+03
500.	-8453.70	246.	14.3E+03	20.2E+03	24.7E+03	28.5E+03	31.0E+03	35.0E+03
550.	-7373.81	90.8	23.5E+03	33.2E+03	40.6E+03	46.0E+03	52.5E+03	57.5E+03
600.	-6313.45	38.1	36.2E+03	51.3E+03	62.0E+03	72.5E+03	81.0E+03	88.0E+03
650.	-527'.32	17.7	53.1E+03	75.2E+03	92.0E+03	10.6E+04	11.0E+04	13.0E+04
700.	-4246.24	3.00	74.6E+03	10.5E+04	12.0E+04	14.0E+04	16.7E+04	18.3E+04

FORTRAN STOP
\$

TABLE 9: HYDROGEN CHLORIDE / ZINC OXIDE EQUILIBRIUM

7: CONCLUSIONS AND RECOMMENDATIONS

The development of a regenerable, high-temperature zinc ferrite sorbent was carried from the lab scale on simulated gas to the bench scale on actual coal-derived gas. It was found that no major problems were encountered in desulfurizing gasifier gas to the desired level of less than 10 ppm. Sidestream testing on the DOE/METC fixed-bed gasifier revealed that carbon formation was occurring in the desulfurization reactor. However, the carbon was oxidized and/or gasified during the regeneration step. It is felt that the presence of carbon is actually beneficial during the regeneration because of its capacity to reduce SO_2 to elemental sulfur in-situ. The zinc ferrite sorbent was also found to catalyze the shift reaction, thereby making the zinc ferrite a combined shift/desulfurization sorbent and catalyst.

The regenerations conducted revealed that no appreciable loss of capacity was detected for the near term, although when incomplete regenerations are conducted there will be some loss in capacity. The six-inch reactor appears to regenerate well with an 80/20 steam/air mixture.

Particulates in the feed gas did not appear to be a problem. It is felt that any particulates that may have reached the sorbent were burned during the regeneration.

The hot-gas delivery pipeline from the fixed-bed gasifier cyclone was able to transport the gasifier gas without serious blockage problems, although under certain gasifier operating conditions large dust loadings can plug the line. Tars and oils did not appear to be a problem, and were passed through the desulfurization system and condensed downstream. Carbon formation was observed, and it is believed that cracking was occurring over the sorbent. This may be due in part to tars and oils in the gas.

It is recommended that the products of regeneration be clearly identified and quantified, and that testing and development for the treatment of regeneration offgas be carried out. Reduction to elemental sulfur would be the preferred route.

Testing of small-scale fuel cell systems is recommended for the near future to obtain baseline data on the performance of fuel cells on desulfurized coal gas, and on the effect of components not removed by the zinc ferrite system.

In order to conduct longer-term tests, a continuously operating system will be required. This will require two or three reactors that can be valved to carry out the sulfidation/regeneration sequence continuously, providing full-time desulfurization. It is recommended that such a system be planned for future long-term development.

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7. DOE/MC/16441-1423 (DE83014550); D.A. Green, K.E. Leese, K.L. Mack, W.J. McMichael; Pollution Control in Coal Gasification; May 1983.

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APPENDIX A: LAB-SCALE TESTING - TEST SUMMARY SHEETS

TEST SUMMARY SHEET

TEST NO. : 183
 DATE STARTED : 9/18/82
 DATE ENDED : 9/18/82
 TOTAL HOURS : 4.4 HRS
 TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
 SORBENT NO. : L-1443

ANALYSIS:

BEFORE AFTER

SORBENT COMPOSITION:

TOTAL SULFUR (XW/W):
 SURFACE AREA (M2/G):
 DENSITY (G/CC):
 PORE VOLUME (MM3/G):
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR LOADING :

OPERATING CONDITIONS

DATA: (DETECTOR TUBE)

TEMPERATURE: 1800 F

EXIT H2S: 4.5 PPM AFTER 10 MINUTES

PRESSURE : AMBIENT

EXIT SO2:

SPACE VELOCITY: 3242 HOURLY FOR 7 INCH BED
 LINEAR VELOCITY: 16 CM/SEC

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	2.4 SCFH	6.2
H2O		
AIR		
CO2	3.6 SCFH	9.3 %
CO	4.4 SCFH	11.3 %
H2	6.3 SCFH	16.25%
CH4	1.07 SCFH	2.75%
N2	21.0 SCFH	54.2 %

REMARKS

CONCLUSIONS

1. COMPLETE BREAKTHROUGH CURVE COMPLETED IN ONE SHIFT WITH LOW INITIAL H2S.

TOTAL FLOW RATE: 38.77 SCFH

TEST SUMMARY SHEET

TEST NO. : 104
 DATE STARTED : 9/14/82
 DATE ENDED : 9/14/82
 TOTAL HOURS : 5.75 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
 EXIT SO2: MAX 3.3 PERCENT
 EXIT S2:
 EXIT H2:

FLOW RATE MOLEX

H2S
 H2O 2.23 CC/MIN 50%
 AIR 5.00 SCFH 50%
 CO2
 CO
 H2
 CH4
 N2

REMARKS

1.

CONCLUSIONS

1. REACTOR DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 185
DATE STARTED : 9/16/82
DATE ENDED : 9/16/82
TOTAL HOURS : 5.33 HRS
TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
SORBENT NO. : L-1443

ANALYSIS:

BEFORE AFTER

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

TOTAL SULFUR (XW/W):
SURFACE AREA (M2/G):
DENSITY (G/CC) :
PORE VOLUME (MM3/G):
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F

DATA: (DETECTOR TUBE)

EXIT H2S: 56 PPM AFTER 20 MINUTES

PRESSURE : AMBIENT

EXIT S02:

SPACE VELOCITY: 3600 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 17.8 CM/SEC

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	2.4 SCFH	5.6
H2O	1.63 CC/MIN	18.0 %
AIR		
CO2	3.6 SCFH	8.4 %
CO	4.4 SCFH	16.2 %
H2	6.3 SCFH	14.6%
CH4	1.07 SCFH	2.5%
N2	21.0 SCFH	48.0 %

REMARKS

CONCLUSIONS

1. COMPLETE BREAKTHROUGH CURVE COMPLETED IN ONE SHIFT.
H2S LEVEL REASONABLY LOW INITIALLY.

TOTAL FLOW RATE: 43.0 SCFH

TEST SUMMARY SHEET

TEST NO. : 106
 DATE STARTED : 9/28/82
 DATE ENDED : 9/28/82
 TOTAL HOURS : 6.0 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

ANALYSIS:

BEFORE AFTER

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 2.1 PERCENT MAX, 368 PPM AT END OF REGENERATION

EXIT S2:

EXIT H2:

FLOW RATE MOLEX

H2S
 H2O 2.23 CC/MIN 50%
 AIR 5.88 SCFH 50%
 CO2
 CO
 H2
 CH4
 N2

REMARKS

1.

2.

CONCLUSIONS

1. REACTOR WALLS DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 187
DATE STARTED : 9/21/82
DATE ENDED : 9/21/82
TOTAL HOURS : 2.5 HRS
TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHROOT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (%W/W):
SURFACE AREA (M2/G):
DENSITY (G/CC) :
PORE VOLUME (MM3/G):
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1280 F

PRESSURE : AMBIENT

SPACE VELOCITY: 3600 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 17.8 CM/SEC

DATA: (DETECTOR TUBE)

EXIT H2S: 16 PPM AFTER 15 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	2.4 SCFH	5.6
H2O	1.63 CC/MIN	10.0 %
AIR		
CO2	3.6 SCFH	8.4 %
CO	4.4 SCFH	10.2 %
H2	6.3 SCFH	14.6%
CH4	1.07 SCFH	2.5%
N2	21.0 SCFH	48.0 %

REMARKS

CONCLUSIONS

1. BREAKTHROUGH CURVE AT 1280 F IS COMPLETED SOONER THAN AT 1000F AS EXPECTED.

TOTAL FLOW RATE: 43.0 SCFH

TEST SUMMARY SHEET

TEST NO. : 188
 DATE STARTED : 9/23/82
 DATE ENDED : 9/23/82
 TOTAL HOURS : 5.5 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAM
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1800 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
 EXIT S02: 2.7 % MAX 560 PPM AT END OF RUN
 EXIT S2:
 EXIT H2:

FLOW RATE MOLEX

H2S

H2O

AIR

CO2

CO

H2

CH4

N2

2.23 CC/MIN 50%
 5.88 SCFH 50%

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFIDE REACTOR WALLS.
- 2.

CONCLUSIONS

1. REACTOR WALLS DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 100
DATE STARTED : 9/24/82
DATE ENDED : 9/24/82
TOTAL HOURS : 1.3 HRS
TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (%W/W):
SURFACE AREA (M2/G):
DENSITY (G/CC) :
PORE VOLUME (MM3/G):
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

SPACE VELOCITY: 3244 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 16 CM/SEC

DATA: (DETECTOR TUBE)

EXIT H2S: 17.5 PPM INITIALLY, DECREASING TO 5.0 PPM AT 23 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	2.4 SCFH	6.2
H2O		
AIR		
CO2	3.6 SCFH	9.3 %
CO	4.4 SCFH	11.3 %
H2	6.3 SCFH	16.25%
CH4	1.07 SCFH	2.75%
N2	21.0 SCFH	54.2 %

TOTAL FLOW RATE: 38.77 SCFH

REMARKS

TEMPERATURE WAVE DETECTED DURING SULFIDATION TEST ABORTED.

CONCLUSIONS

1. SUSPECT SORBENT REDUCED TO FE AND CATALYZED METHANATION TO PRODUCE TEMPERATURE WAVE.

TEST SUMMARY SHEET

TEST NO. : 118
 DATE STARTED : 9/20/82
 DATE ENDED : 9/20/82
 TOTAL HOURS : 5.0 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF EMPTY REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 2.25 % MAX 388 PPM AT END OF REGENERATION

EXIT S2:

EXIT H2:

FLOW RATE MOLEX

H2S

H2O 2.23 CC/MIN 50%

AIR 5.88 SCFH 50%

CO2

CO

H2

CH4

N2

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFIDE REACTOR WALLS.
- 2.

CONCLUSIONS

1. REACTOR WALLS DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 111
DATE STARTED : 9/30/82
DATE ENDED : 9/30/82
TOTAL HOURS : 8.6 HRS
TYPE : SULFIDATION

PURPOSE

REPEAT T100 FOR SULFIDATION OF " 1/2 REACTOR " TO
OBTAIN COMPLETE BREAKTHROUGH CURVE FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (XW/W):
SURFACE AREA (M2/G):
DENSITY (G/CC):
PORE VOLUME (MM3/G):
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

SPACE VELOCITY: 3244 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 16 CM/SEC

DATA: (DETECTOR TUBE)

EXIT H2S: 875 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	2.4 SCFH	5.4
H2O		
AIR		
CO2	3.6 SCFH	9.69 %
CO	4.4 SCFH	11.91 %
H2	6.3 SCFH	17.12 %
CH4	1.07 SCFH	2.91 %
N2	21.0 SCFH	56.88 %

REMARKS

CHECK GAS COMPOSITIONS AT LEFT
TEMPERATURE WAVE DETECTED DURING SULFIDATION
TEST ABORTED.

CONCLUSIONS

1. TEST ABORTED.

TOTAL FLOW RATE: 39.77 SCFH

TEST SUMMARY SHEET

TEST NO. : 112
 DATE STARTED : 10/7/82
 DATE ENDED : 10/7/82
 TOTAL HOURS : 5 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: L-1443 ZINC FERRITE/320 GRAMS
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY:

DATA: (DETECTOR TUBE)

EXIT H2S:
 EXIT SO2: UP TO 9600 PPM
 EXIT S2:
 EXIT H2:

FLOW RATE MOLEX

H2S
 H2O 2.23 CC/MIN 50%
 AIR 5.88 SCFH 50%
 CO2
 CO
 H2
 CH4
 N2

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFIDE REACTOR WALLS.
- 2.

CONCLUSIONS

1. REGENERATION COMPLETE

TEST SUMMARY SHEET

TEST NO. : 113
 DATE STARTED : 10/8/82
 DATE ENDED : 10/8/82
 TOTAL HOURS : 8.41 HRS
 TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHROEDT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
 SORBENT NO. : L-1443

ANALYSIS:

BEFORE AFTER

SORBENT COMPOSITION:

TOTAL SULFUR (XW/W):
 SURFACE AREA (M2/G):
 DENSITY (G/CC) :
 PORE VOLUME (MM3/G):
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1400 F

DATA: (DETECTOR TUBE)

EXIT H2S: 0 PPM AFTER 5 MINUTES

PRESSURE : AMBIENT

EXIT SO2:

SPACE VELOCITY: 3600 HOURLY FOR 7 INCH BED
 LINEAR VELOCITY: 17.8 CM/SEC

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	2.4 SCFH	5.6
H2O	1.63 CC/MIN	10.0 %
AIR		
CO2	3.6 SCFH	8.4 %
CO	4.4 SCFH	10.2 %
H2	6.3 SCFH	14.6 %
CH4	1.07 SCFH	2.5 %
N2	21.0 SCFH	48.8 %

REMARKS

CONCLUSIONS

1. SHORTER BREAKTHROUGH CURVE AS EXPECTED.

TOTAL FLOW RATE: 43.0 SCFH

TEST SUMMARY SHEET

TEST NO. : 114
 DATE STARTED : 10/13/82
 DATE ENDED : 10/13/82
 TOTAL HOURS : 5.5 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
 EXIT SO2: TO 22,500 PPM
 EXIT S2:
 EXIT H2:

FLDW RATE MOLEX

H2S
 H2O 2.23 CC/MIN 50%
 AIR 5.08 SCFH 50%
 CO2
 CO
 H2
 CH4
 N2

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFIDE REACTOR WALLS.
- 2.

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 115
DATE STARTED : 18/14/82
DATE ENDED : 18/14/82
TOTAL HOURS : 5.73 HRS
TYPE : SULFIDATION

PURPOSE

RJN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
SORBENT NO. : L-1443

ANALYSIS:

BEFORE AFTER

SORBENT COMPOSITION:

TOTAL SULFUR (%W/W):
SURFACE AREA (M2/G):
DENSITY (G/CC):
PORE VOLUME (MM3/G):
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

DATA: (DETECTOR TUBE)

TEMPERATURE: 1000 F

EXIT H2S: UNDETECTABLE INITIALLY, 16 PPM AFTER 14 MINUTES.

PRESSURE : AMBIENT

EXIT SO2:

SPACE VELOCITY: 2026 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 10.0 CM/SEC

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	1.34 SCFH	5.6
H2O	0.02 CC/MIN	10.0 %
AIR		
CO2	2.02 SCFH	8.4 %
CO	2.5 SCFH	10.2 %
H2	3.5 SCFH	14.6 %
CH4	0.60 SCFH	2.5 %
N2	11.8 SCFH	48.8 %

REMARKS

1. EXPECTED COMPLETION OF BREAKTHROUGH CURVE IS 7 HRS.

CONCLUSIONS

1. COMPLETED BREAKTHROUGH CURVE AT 1000 F .

TOTAL FLOW RATE: 24.2 SCFH

TEST SUMMARY SHEET

TEST NO. : 116
 DATE STARTED : 10/10/82
 DATE ENDED : 10/10/82
 TOTAL HOURS : 6.16 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
 EXIT SO2: TO 10000 PPM
 EXIT S2:
 EXIT H2:

FLOW RATE MOLEX

H2S
 H2O 2.23 CC/MIN 50%
 AIR 5.88 SCFH 50%
 CO2
 CO
 H2
 CH4
 N2

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFIDE REACTOR WALLS.
- 2.

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 117
 DATE STARTED : 18/20/82
 DATE ENDED : 18/20/82
 TOTAL HOURS : 2.25 HRS
 TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
 SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR LOADING :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (XW/W):
 SURFACE AREA (M2/G):
 DENSITY (G/CC):
 PORE VOLUME (MM3/G):
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2026 HOURLY FOR 7 INCH BED
 LINEAR VELOCITY: 10.0 CM/SEC

DATA: (DETECTOR TUBE)

EXIT H2S: UNDETECTABLE INITIALLY, 13 PPM AFTER 25 MINUTES.

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	1.34 SCFH	5.6
H2O	0.02 CC/MIN	10.0 %
AIR		
CO2	2.02 SCFH	8.4 %
CO	2.5 SCFH	10.2 %
H2	3.5 SCFH	14.6%
CH4	0.60 SCFH	2.5%
N2	11.8 SCFH	48.8 %

REMARKS

1. EXPECTED COMPLETION OF BREAKTHROUGH CURVE IS 7 HRS.

CONCLUSIONS

1. COMPLETED BREAKTHROUGH CURVE AT 1200 F.

TOTAL FLOW RATE: 24.2 SCFH

TEST SUMMARY SHEET

TEST NO. : 118
DATE STARTED : 10/22/82
DATE ENDED : 10/22/82
TOTAL HOURS : 5.5 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
SORBENT NO. : L-1443
SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
SURFACE AREA:
DENSITY :
PORE VOLUME:
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
PRESSURE : AMBIENT
SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
EXIT SO2: TO 10,000 PPM
EXIT S2:
EXIT H2:

FLOW RATE MOLE%

H2S		
H2O	2.23 CC/MIN	50%
AIR	5.38 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFIDE REACTOR WALLS.
- 2.

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 110
DATE STARTED : 10/25/82
DATE ENDED : 10/25/82
TOTAL HOURS : 8.4 HRS
TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (XW/W):
SURFACE AREA (M2/G):
DENSITY (G/CC) :
PORE VOLUME (MM3/G):
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1400 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2826 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 10.0 CM/SEC

DATA: (DETECTOR TUBE)

EXIT H2S: UNDETECTABLE INITIALLY, 23 PPM AFTER 10 MINUTES.

EXIT S02:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	1.34 SCFH	5.6
H2O	0.02 CC/MIN	10.0 %
AIR		
CO2	2.02 SCFH	8.4 %
CO	2.5 SCFH	10.2 %
H2	3.5 SCFH	14.6%
CH4	0.60 SCFH	2.5%
N2	11.0 SCFH	48.0 %

REMARKS

1. EXPECTED COMPLETION OF BREAKTHROUGH CURVE IS LESS THAN 3 HOURS.

CONCLUSIONS

1. COMPLETED BREAKTHROUGH CURVE AT 1400 F.

TOTAL FLOW RATE: 24.2 SCFH

TEST SUMMARY SHEET

TEST NO. : 128
DATE STARTED : 10/25/82
DATE ENDED : 10/25/82
TOTAL HOURS : 3.25 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
SORBENT NO. : L-1443
SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
SURFACE AREA:
DENSITY :
PORE VOLUME:
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
PRESSURE : AMBIENT
SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
EXIT SO2: TO 14,000 PPM
EXIT S2:
EXIT H2:

	FLOW RATE	MOLEX
H2S		
H2O	2.23 CC/MIN	50%
AIR	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFIDE REACTOR WALLS.
- 2.

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 121
 DATE STARTED : 10/28/82
 DATE ENDED : 10/28/82
 TOTAL HOURS : 2.36 HRS
 TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
 SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR LOADING :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (XW/WI):
 SURFACE AREA (M2/G):
 DENSITY (G/CC):
 PORE VOLUME (MM3/G):
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 3600 HOURLY FOR 7 INCH BED
 LINEAR VELOCITY: 17.8 CM/SEC

DATA: (DETECTOR TUBE)

EXIT H2S: 1100 PPM INITIALLY

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	1.34 SCFH	3.1
H2O	1.63 CC/MIN	10.0 X
AIR		
CO2	3.6 SCFH	8.4 X
CO	4.4 SCFH	10.2 X
H2	6.3 SCFH	14.6X
CH4	1.07 SCFH	2.5X
N2	22.0 SCFH	46.8 X

REMARKS

CONCLUSIONS

1. COMPLETE BREAKTHROUGH CURVE COMPLETED IN ONE SHIFT.
 H2S LEVEL ABOVE EQUILIBRIUM AT THIS LINEAR VELOCITY.

TOTAL FLOW RATE: 43.0 SCFH

TEST SUMMARY SHEET

TEST NO. : 122
DATE STARTED : 11/03/82
DATE ENDED : 11/03/82
TOTAL HOURS : 3.75 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
SORBENT NO. : L-1443
SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
SURFACE AREA:
DENSITY :
PORE VOLUME:
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
PRESSURE : AMBIENT
SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
EXIT SO2: TO 17000 PPM
EXIT S2:
EXIT H2:

FLOW RATE MOLEX

H2S
H2O 2.23 CC/MIN 50%
AIR 5.08 SCFH 50%
CO2
CO
H2
CH4
N2

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFIDE REACTOR WALLS.
- 2.

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 123
DATE STARTED : 11/2/82
DATE ENDED : 11/2/82
TOTAL HOURS : 2.3 HRS
TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
SORBENT NO. : L-443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (XW/W):
SURFACE AREA (M2/G):
DENSITY (G/CC) :
PORE VOLUME (MM3/G):
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

SPACE VELOCITY: 3600 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 17.8 CM/SEC

DATA: (DETECTOR TUBE)

EXIT H2S: UNDETECTABLE INITIALLY, 4 PPM AFTER 25 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	1.34 SCFH	3.1
H2O	1.63 CC/MIN	10.0 %
AIR		
CO2	3.6 SCFH	8.4 %
CO	4.4 SCFH	10.2 %
H2	6.3 SCFH	14.6%
CH4	1.07 SCFH	2.5%
N2	22.0 SCFH	48.8 %

REMARKS

CONCLUSIONS

1. BREAKTHROUGH CURVE AT 1200 F COMPLETED.

TOTAL FLOW RATE: 43.0 SCFH

TEST SUMMARY SHEET

TEST NO. : 124
 DATE STARTED : 11/08/82
 DATE ENDED : 11/08/82
 TOTAL HOURS : 4.28 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
 EXIT SO2: TO 36,000 PPM
 EXIT S2:
 EXIT H2:

FLOW RATE MOLE%

H2S
 H2O 2.23 CC/MIN 50%
 AIR 5.88 SCFH 50%
 CO2
 CO
 H2
 CH4
 N2

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS
2. TO DESULFIDE REACTOR WALLS.

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 126
DATE STARTED : 11/9/82
DATE ENDED : 11/9/82
TOTAL HOURS : 8.28 HRS
TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" FOR COMPLETE BREAKTHROUGH
FOR SCHRÖDT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (XW/W):
SURFACE AREA (M2/G):
DENSITY (G/CC) :
PORE VOLUME (MM3/G):
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1400 F

PRESSURE : AMBIENT

SPACE VELOCITY: 3600 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 17.8 CM/SEC

DATA: (DETECTOR TUBE)

EXIT H2S: UNDETECTABLE INITIALLY, 10 PPM AFTER 7 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	1.34 SCFH	3.1
H2O	1.63 CC/MIN	10.0 %
AIR		
CO2	3.6 SCFH	8.4 %
CO	4.4 SCFH	10.2 %
H2	6.3 SCFH	14.6 %
CH4	1.07 SCFH	2.5 %
N2	22.0 SCFH	48.8 %

REMARKS

CONCLUSIONS

1. BREAKTHROUGH CURVE AT 1400 IF COMPLETED.

TOTAL FLOW RATE: 43.0 SCFH

TEST SUMMARY SHEET

TEST NO. : 126
 DATE STARTED : 11/18/82
 DATE ENDED : 11/18/82
 TOTAL HOURS : 4 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GRAMS
 SORBENT NO. : L-1443
 SORBENT COMPOSITION:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR REMOVED :

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		
DENSITY :		
PORE VOLUME:		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 984 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
 EXIT SO2: TO 17,000 PPM
 EXIT S2:
 EXIT H2:

	FLOW RATE	MOLEX
H2S		
H2O	2.23 CC/MIN	50%
AIR	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS
2. TO DESULFIDE REACTOR WALLS.

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 127
 DATE STARTED : 11/22/82
 DATE ENDED : 11/22/82
 TOTAL HOURS : 1.08 HRS
 TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/2 REACTOR" UNTIL BEGINNING OF
 BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/320 GM
 SORBENT NO. : L-1443

ANALYSIS:

BEFORE AFTER

SORBENT COMPOSITION:

TOTAL SULFUR (%W/W):
 SURFACE AREA (M2/G):
 DENSITY (G/CC):
 PORE VOLUME (MM3/G):
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

SORBENT PELLET SIZE:
 PRETREATMENT :
 SULFUR LOADING :

OPERATING CONDITIONS

DATA: (DETECTOR TUBE)

TEMPERATURE: 1000 F

EXIT H2S: UNDETECTABLE INITIALLY, 12 PPM AFTER 18 MINUTES

PRESSURE : AMBIENT

EXIT SO2:

SPACE VELOCITY: 2826 HOURLY FOR 7 INCH BED
 LINEAR VELOCITY: 10.0 CH/SEC

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	1.34 SCFH	5.6
H2O	0.02 CC/MIN	10.0 %
AIR		
CO2	2.02 SCFH	8.4 %
CO	2.5 SCFH	10.2 %
H2	3.5 SCFH	14.6%
CH4	0.60 SCFH	2.5%
N2	11.8 SCFH	48.8 %

REMARKS

1. RUN UNTIL EXIT H2S REACHES 50 PPM
2. AT END OF RUN SAMPLE SORBENT PELLETS AT FOUR LOCATIONS
 ALONG THE LENGTH OF THE REACTOR. LABEL EACH SAMPLE AND
 SEND A PORTION OF EACH SAMPLE TO:
 1. SEM/EDAX (JIM KING) 3. SURFACE AREA/POROSITY (CHIDESTER)
 2. TOTAL SULFUR (ROMANOSKY)

CONCLUSIONS

1. SAMPLES OF PELLETS TAKEN AT FOUR LOCATIONS IN THE BED
 AFTER 50 PPM BREAKTHROUGH AND ANALYZED FOR TOTAL SULFUR.

TOTAL FLOW RATE: 24.2 SCFH

TEST SUMMARY SHEET

TEST NO. : 128
DATE STARTED : 2/23/83
DATE ENDED : 2/23/83
TOTAL HOURS : 2.25 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF UNITED CATALYST L-1584
AT 1200 F.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/1000 GM
SORBENT NO. : L-1584
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE
50 MOLE% ZINC OXIDE
SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : X (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):		
DENSITY (G/CC) :		
PORE VOLUME (MM3/G):		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1200 F
PRESSURE : AMBIENT
SPACE VELOCITY: 1286 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 1.5-5 PPM FOR 1.75 HOURS

EXIT SO2: UNDETECTABLE

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE%
H2S	0.9963 SCFH	2.7%
H2O	3.872CC/MIN	27.66%
AIR		
CO2	3.6 SCFH	9.68 %
CO	4.3 SCFH	11.91 %
H2	6.3 SCFH	17.12%
CH4	1.07 SCFH	2.91%
N2	10.34 SCFH	28.1 %

REMARKS

1. BAD TEMPERATURE CONTROLLER CAUSED OVERTEMPORATING OF SORBENT. TEST RUN ABORTED.
- 2.

CONCLUSIONS

1. RUN WILL HAVE TO BE REPEATED.

TEST SUMMARY SHEET

TEST NO. : 120
 DATE STARTED : 3/1/83
 DATE ENDED : 3/1/83
 TOTAL HOURS : 6.25 HRS
 TYPE : SULFIDATION

PURPOSE

SULFIDATION OF UNITED CATALYST L-1584
 AT 1200 F.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/1000 GM
 SORBENT NO. : L-1584
 SORBENT COMPOSITION: 50 MOLE% IRON OXIDE
 50 MOLE% ZINC OXIDE
 SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
 PRETREATMENT :
 SULFUR LOADING : 1 (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (%W/W):
 SURFACE AREA (M2/G):
 DENSITY (G/CC):
 PORE VOLUME (MM3/G):
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1200 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 1286 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 0.5 - 1.6 PPM FOR 1.5 HRS
 EXIT SO2: 0 PPM
 EXIT S2:
 EXIT H2:

	FLOW RATE	MOLE%
H2S	0.9963 SCFH	2.7%
H2O	3.872CC/MIN	27.66%
AIR		
CO2	3.6 SCFH	9.68 %
CO	4.3 SCFH	11.91 %
H2	6.3 SCFH	17.12%
CH4	1.07 SCFH	2.91%
N2	10.34 SCFH	28.1 %

REMARKS

1. SAMPLE CONDENSATE FOR ZINC IRON AND SULFATE ANALYSIS
2. THIS IS A REPEAT OF TEST 120. FRESH SORBENT LOADED.

CONCLUSIONS

1.

TEST SUMMARY SHEET

TEST NO. : 130
 DATE STARTED : 3/04/83
 DATE ENDED : 3/04/83
 TOTAL HOURS : 16 HRS
 TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 120

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/1000 GM
 SORBENT NO. : L-1504
 SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE
 50.0 MOLE% ZINC OXIDE
 SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
 PRETREATMENT :
 SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
 SURFACE AREA:
 DENSITY :
 PORE VOLUME:
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 385 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
 EXIT SO2: MAX OF 7.6 PERCENT 610 PPM AT END
 EXIT S2:
 EXIT H2:

	FLOW RATE	MOLE%
H2S		
H2O	2.23 CC/MIN	50%
AIR	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

REMARKS

1. AIR FLOW RATE INCREASED TO +6.6 SCFH AT 4.75 HRS.
2. DID NOT OBTAIN TYPICAL TEMP WAVE AT 5.88 AIR FLOW .

CONCLUSIONS

1. REGENERATION COMPLETE.

TEST SUMMARY SHEET

TEST NO. : 131
 DATE STARTED : 3/11/83
 DATE ENDED : 3/11/83
 TOTAL HOURS : 6 HRS
 TYPE : SULFIDATION

PURPOSE

SULFIDATION OF UNITED CATALYST L-1584
 AT 1200 F A SECOND TIME.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/1200 GM
 SORBENT NO. : L-1584
 SORBENT COMPOSITION: 50 MOLE% IRON OXIDE
 50 MOLE% ZINC OXIDE
 SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
 PRETREATMENT :
 SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR (XW/W):
 SURFACE AREA (M2/G):
 DENSITY (G/CC):
 PORE VOLUME (MM3/G):
 MINERAL ANALYSIS:
 ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1200 F
 PRESSURE : AMBIENT
 SPACE VELOCITY: 1286 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 5-13 PPM FOR 4.5 HOURS

EXIT SO2: 17-2 PPM FOR 2.5 HOURS

EXIT S2:

EXIT H2:

	FLOW RATE	MOLEX
H2S	8.9963 SCFH	2.7%
H2O	3.872CC/MIN	27.66%
AIR		
CO2	3.6 SCFH	9.68 %
CO	4.3 SCFH	11.91 %
H2	6.3 SCFH	17.12%
CH4	1.07 SCFH	2.91%
N2	10.34 SCFH	28.1 %

REMARKS

1. SAMPLE CONDENSATE FOR ZINC IRON AND SULFATE ANALYSIS
2. SECOND SULFIDATION ON THIS BATCH.

CONCLUSIONS

1. EXIT H2S COMPOSITIONS SLIGHTLY ABOVE THOSE OF THE FIRST SULFIDATION, BUT BREAKTHROUGH TIME IS SIMILAR.

TEST SUMMARY SHEET

TEST NO. : 132
DATE STARTED : 3/15/83
DATE ENDED : 3/15/83
TOTAL HOURS : 1.75 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 131

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/1000 GM
SORBENT NO. : L-1504
SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE
50.0 MOLE% ZINC OXIDE
SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED :

ANALYSIS:

BEFORE AFTER

TOTAL SULFUR:
SURFACE AREA:
DENSITY :
PORE VOLUME:
MINERAL ANALYSIS:
ELEMENTAL ANALYSIS:

OPERATING CONDITIONS

TEMPERATURE: 1000 F
PRESSURE : AMBIENT
SPACE VELOCITY: 305 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:
EXIT SO2: 0 % MAX
EXIT S2:
EXIT H2:

	FLOW RATE	MOLE%
H2S		
H2O	2.23 CC/MIN	50%
AIR	5.00 SCFH	50%
CG2		
CG		
H2		
CH4		
H2		

REMARKS

1. STOP REGENERATION WHEN TEMP WAVE IS AT MID BED.
2. SAMPLE PELLETS BEFORE , AT , AND AFTER TEMP WAVE AND SEND TO JIM KING FOR ELECTRON MICROSCOPE ANALYSIS.

CONCLUSIONS

1. REGENERATION STOPPED WHEN TEMP WAVE WAS AT MID-BED.

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APPENDIX B: LAB-SCALE TESTING - TEST DATA

ZINC FERRITE SULFIDATION

TEST 103 L-1443 UNITED CATALYSTS

13-JAN-84 16:01:37

ZINC FERRITE SULFIDATION

TEST 105 L-1443 UNITED CATALYSTS

13-JAN-84 16:03:44

ZINC FERRITE SULFIDATION

TESTS 103, 105 L-1443 UNITED CATALYSTS

ZINC FERRITE SULFIDATION

TEST 107 L-1443 UNITED CATALYSTS

13-JAN-84 16:08:19

ZINC FERRITE SULFIDATION

TESTS 105, 107 L-1443 UNITED CATALYSTS

ZINC FERRITE SULFIDATION

TEST 113 L-1443 UNITED CATALYSTS

ZINC FERRITE SULFIDATION

TESTS 105, 107, 113 L-1443 UNITED CATALYSTS

P:

ZINC FERRITE SULFIDATION

TEST 115 L-1443 UNITED CATALYSTS

ZINC FERRITE SULFIDATION

TEST 117 L-1443 UNITED CATALYSTS

ZINC FERRITE SULFIDATION

TESTS 115, 117, 119 L-1443 UNITED CATALYSTS

ZINC FERRITE SULFIDATION

TESTS 121, 123, 125 L-1443 UNITED CATALYSTS

18-JAN-84 10:14:50

ZINC FERRITE SULFIDATION

TEST 127

ZINC FERRITE SULFUR LOADING

TEST 127

ZINC FERRITE SULFIDATION

TEST 129 (L1504)

ZINC FERRITE SULFIDATION

TESTS 129,131 (L1504)

APPENDIX C: QUARTZ/METAL REACTOR SYSTEM DRAWINGS

HOT GAS DESULFURIZATION LAB SCALE TEST UNIT
PROCESS FLOW DIAGRAM-DRAWING NO. E830033

DESIGN SPECIFICATIONS:
 DESIGN PRESSURE — 30 PSIA MAX
 DESIGN TEMPERATURE — 1800°F MAX
 OPERATING PRESSURE — 30 PSIA
 OPERATING TEMPERATURE — 1000-1200°F

HOT GAS DESULFURIZATION LAB SCALE TEST UNIT
P&I DIAGRAM-DRAWING NO. E830217

DESIGN SPECIFICATIONS:

Design Pressure — 30 psig Min
Design Temperature — 1000°F Min
Operating Pressure — 0-30 psig
Operating Temperature — 1000°-1200°F

1. THE SIZE OF HOLES IN DISC, PART #6, AND THE METHOD OF FASTENING TO TUBE, PART #1, IS TO BE DETERMINED BY QUARTZ VENDOR.
 2. THE METHOD OF FASTENING DISC, PART #3, TO TUBE, PART #1, IS TO BE DETERMINED BY QUARTZ VENDOR.
 3. THE UPPER DISC, PART #5, IS TO BE REMOVABLE. DO NOT FASTEN TO TUBE, PART #1.
 4. THE .890 φ HOLE IN DISC, PART #3, IS TO BE IN LINE WITH ONE ANOTHER SO A .890 φ ROD CAN FREELY PASS THRU HOLES.
 5. BEAD, PART #2, IS TO BE FABRICATED & FASTENED TO TUBE, PART #1, BY QUARTZ VENDOR.
1. TOLERANCES AS FOLLOWS:
 a) ALL C-RACE DIMENSIONS ±.002
 b) ALL S-RACE DIMENSIONS ±.005

NOTES: (UNLESS OTHERWISE SPECIFIED)

DESIGN SPECIFICATIONS

DESIGN PRESSURE	30 PSIG MAX
DESIGN TEMPERATURE	1800°F MAX
OPERATING PRESSURE	30 PSIG
OPERATING TEMPERATURE	1000-1800°F

HOT GAS DESULFURIZATION LAB SCALE TEST UNIT
 QUARTZ REACTOR-DRAWING NO. D830034

NOTES!

1. FASTEN WITH SELF-TAPPING SCREWS.
2. GRIND IF NEEDED.

HOT GAS DESULFURIZATION LAB SCALE TEST UNIT
SUPPORT RACK-DRAWING NO. E830073

7a

45°

2.50

.75

.4875 Ø THRU
.1875 DEEP

7b

1.75

1.00

.75 R

.1875 Ø THRU
.257 Ø & BORE
.13 DEEP

7 TOP FLANGE ASSEMBLY

25°

.422 & THRU
.250 DIA HT

5 BOTTOM FLANGE

DETAIL B-4 (10-4)

Technical drawing of a circular plate with a hexagonal hole pattern. The drawing includes the following specifications and dimensions:

- Overall Diameter:** 2.000 ϕ
- Inner Hole Diameter:** .125 ϕ Tolerances: $\pm .001$ (Top), $\pm .002$ (Bottom)
- Number of Holes:** 110 HOLES
- Spacing:** EQUALLY SPACED
- Material:** C6141-156 Titanium
- Surface Finish:** As Supplied
- Inner Hole Spacing:** .057 (Dimension from center to first hole)
- Inner Hole Diameter:** .057 ϕ Tolerances: $\pm .001$ (Top), $\pm .002$ (Bottom)
- Inner Hole Spacing:** .156 (Dimension from center to second hole)
- Inner Hole Diameter:** .156 (Dimension from center to third hole)

(15) DLSC (1.187 Tm)

⑤ INTERNAL PRECIPITATION

12.50 DIA
110 HOLES
EQUALLY SPACED
ON A 2.000 INCH
PITCH AS SHOWN

2.000 PITCH

0.125 DIA HOLES

0.005 THICK

32

12.50 DIA

SECTION A.3 (B.4 & B.5)

- ## DESIGN SPECIFICATIONS

DESIGN PRESSURE — 30.2516 MPa,
LIFE EXPECTANCY — 1000 HRS
OPERATING PRESSURE — 30.25 MPa
OPERATING TEMPERATURE — 1000°-1200°F
(in Heated Zone)

NOTES: (UNLESS OTHERWISE SPECIFIED:

HOT GAS DESULFURIZATION LAB SCALE TEST UNIT
METAL REACTOR-DRAWING NO. D83C036

NOTE: (UNLESS OTHERWISE NOTED.)

99

3 IN ASSEMBLING 'LEVEL SWITCH' TO BUSHING, FLOATS MUST BE REMOVED FIRST. IN REMOVING THE FLOATS, THE SHIRT HAS BEEN FACTORY MARKED FOR LOCATION OF STOPS. THIS CAN BE SEEN ON BOTH SIDES OF STOPS (REMOVE THREE ONLY). THREAD LEVEL SWITCH INTO BUSHING THEN REPLACE FLOATS AND STOPS (AS SHOWN)

2. ASSEMBLING SHALL BE IN ACCORDANCE WITH NORMAL SHOP PRACTICE

1 MODIFY FITTINGS (PART NO. 3) TO FIT VESSEL CYLINDER (PART NO. 1)

NOTES:

FABRICATION/TESTS REQUIREMENTS

CODE CONSTRUCTION	N/A	STAMP	U.S.
CERTIFICATION OF WELDER	YES		
HYDROSTATIC TEST	200 PSIG	TEMP.	70 °F
LEAK TEST	N/A	WITNESS	N/A
REPORT	N/A		
DESIGN CONDITIONS	30 PSIG	100 °F	
X-RAY WELDS	N/A	FULL	N/A
OR SPOT	N/A		
WELD EFFICIENCY	0.6		
MATERIAL-MILL TEST REPORT	N/A		
QUALITY ASSURANCE SPECIFICATIONS			

HCT GAS DESULFURIZATION LAB SCALE TEST UNIT
CONDENSER KNOCKOUT VESSEL-DRAWING NO. 7830305

APPENDIX D: SIDESTREAM TESTING - TEST SUMMARY SHEETS AND TEST DATA

HOT GAS DESULFURIZATION SIDE STREAM TEST UNIT PROCESS AND INSTRUMENT DIAGRAM

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 001
DATE STARTED : 16:15 6/23/83
DATE ENDED : 00:35 6/25/83

TOTAL HOURS : 20.66
TYPE : SULFIDATION
SULFUR REMOVED: 6.49 LB S8

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/45 LBS
SORBENT NO. : UNITED CATALYSTS L-1504
SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE
50.0 MOLE% ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
GASIFIER OPERATING CONDITIONS

GASIFIER TYPE : DOE/METC FIXED BED WELLMAN GALUSHA
COAL TYPE : ARKWRIGHT
STEAM/COAL RATIO: 0.525
STEAM/AIR RATIO : 0.20

HGD OPERATING CONDITIONS

TEMPERATURE: 1025 F
PRESSURE : 160 PSIG (AVERAGE)

SPACE VELOCITY: 2020 HOURLY

	INLET MOLE% DRY BASIS (OIL FREE)	INLET MOLE% WET BASIS (OIL FREE)	EXIT MOLE% DRY BASIS (OIL FREE)	EXIT MOLE% WET BASIS (OIL FREE)
H2S	0.23		2 PPM	
H2O				4.71
CO2	7.02			
CO	22.04			
H2	15.03			
CH4	2.50			
N2	47.34			
O2	0.63			
C2H6	0.23			

TOTAL FLOW:	33,515 SCF	35,010 SCF
TOTAL LB MOLES:	88.43	92.38
FLOW RATE:	1130 SCFH	1180 SCFH

PURPOSE

FIRST TEST OF SCALED-UP TEST UNIT ON SIDESTREAM
OF FIXED BED GASIFIER.

SORBENT ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR: (XWT)	0.13	
SURFACE AREA: (SQ M/G)	3.13	
DENSITY : (G/CC)	6.16	
PORE VOLUME: (CC/G)	0.004316	
ELEMENTAL ANALYSIS:		
TOTAL CARBON: (XWT)	0.0	

EXIT SULFUR DATA:

H2S : 2 PPM
C4H4S: 5 PPM

CONDENSATE		
AQUEOUS	:	71.2 LBS (ESTIMATED)
HYDROCARBON	:	8 LBS (ESTIMATED)
TOTAL	:	79.2 LBS (MEASURED)

REMARKS

1. PACKED LENGTH IN REACTOR IS 46 7/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10780 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 50 7/8 INCHES
3. FIRST TEST ON FRESH SORBENT
4. EXIT GC NOT OPERATING DURING THIS TEST.
5. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED AND EXIT GAS SATURATION LEVEL BY STEAM TABLES.

CONCLUSIONS:

1. SORBENT CAN DESULFURIZE GASIFIER GAS CONTAINING TARS AND OILS DOWN TO LESS THAN 10 PPM TOTAL SULFUR.

RUN 001A AVERAGE OPERATING PARAMETERS

AVERAGE(S) FOR PERIOD FROM: 11:15:17 6/21/1983 TO 7:48: 4 6/24/1983

RUN 001B

AVERAGE OPERATING PARAMETERS

AVERAGE(S) FOR PERIOD FROM: 19:30:27 6/24/1983 TO 8:34:28 6/25/1983

ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TEST 001

RUN 001 - REACTOR PRESSURE

06/23/1983

09:00:00

SLCT 293

ABSORBER INLET BOTTOM

ENGINEERING ID: PI-415

ENGINEERING UNITS: PSIG

06/25/1983

09:00:00

NOTE: Reactor ΔP reading zero due to inadvertant opening of by-pass on ΔP transmitter. Later tests show significant ΔP rise.

RUN 001 - BENDIX GAS ANALYSIS AT PRODUCER CYCLONE EXIT

H_2
 1=S4108
 CO_2
 2=S4109
 CO
 3=S4115

START TIME OF PLOT
 STOP TIME OF PLOT

9: 0: 0
 9: 0: 0

6/23/1983
 6/25/1983

RUN 001 - INLET H₂S CONCENTRATION

PERCENT H₂S

BENDIX 7000 #4 CHROMATOGRAPH AT S4

START TIME 09:00:00 06/23/1983

STOP TIME 09:00:00 06/25/1983

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 802
DATE AND TIME STARTED : 17:30 6/25/83
DATE AND TIME ENDED : 08:55 6/26/83
TOTAL HOURS : 14.4
TYPE : REGENERATION
SULFUR REMOVED:

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/45 LBS
SORBENT NO. : UNITED CATALYSTS L-1504
SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE
50.0 MOLE% ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS

HGD OPERATING CONDITIONS

INITIAL TEMPERATURE: 1000 F
PRESSURE : 152 INCHES OF WATER (AVERAGE)
SPACE VELOCITY: 961 HOURLY

	INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	19	
H2O	81	

TOTAL LB MOLES:
FLOW RATE : 543 SCFH
\$

PURPOSE

FIRST REGENERATION OF SULFIDED SORBENT ON SIDESTREAM TEST UNIT.

SORBENT ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR: (%WT)		
SURFACE AREA: (SQ M/G)		
DENSITY : (G/CC)		
PORE VOLUME: (CC/G)		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		
TOTAL CARBON: (%WT)		

EXIT SULFUR DATA:

H2S :
SO2 : 14 % INITIALLY

CONDENSATE

AQUEOUS	: 151.1 LBS
HYDROCARBON	:
TOTAL	: 151.1 LBS

REMARKS

1. PACKED LENGTH IN REACTOR IS 46 7/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10780 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 7/8 INCHES
3. FIRST REGENERATION ON SULFIDED SORBENT
4. THERE WERE SOME DIFFICULTIES CONTROLLING THE STEAM AND AND AIR FLOWS. MANUAL ADJUSTMENTS IN STEAM AND AIR FLOWS WERE MADE TO PREVENT EXCEEDING 1700 F IN THE BED.
5. STEAM AND AIR FLOWS WERE ERRATIC DURING THE LAST 7 HOURS DUE TO OPERATOR ADJUSTMENTS.

CONCLUSIONS

1. REGENERATION COMPLETED.
2. IMPROVED FLOW CONTROL FOR STEAM AND AIR ARE REQUIRED.
3. HIGHER STEAM CONTENT OR LOWER SPACE VELOCITY ARE REQUIRED TO PREVENT OVERTEMPORATING THE SORBENT.

RUN 002 AVERAGE OPERATING PARAMETERS

AVERAGE(S) FOR PERIOD FROM: 17:30: 9 6/25/1983 TO 8:54:58 6/26/1983

RUN 002 - REGENERATION REACTOR TEMPERATURES

RUN 002 - REGENERATION STEAM AND AIR FLOWS

AIR #STEAM
 1=53753 2=53754
 START TIME OF PLOT
 STOP TIME OF PLOT

18:31: 0
 8:55: 0

6/25/1983
 6/26/1983

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 003
DATE STARTED : 13:30 6/26/83
DATE ENDED : 15:00 6/27/83
TOTAL HOURS : 24.50
TYPE : SULFIDATION
SULFUR REMOVED: 8.06 LB S0

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/45 LBS
SORBENT NO. : UNITED CATALYSTS L-1584
SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE
50.0 MOLE% ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
GASIFIER OPERATING CONDITIONS

GASIFIER TYPE : DOE/METC FIXED BED WELLMAN GALUSHA
COAL TYPE : ARKWRIGHT
STEAM/COAL RATIO: 0.480
STEAM/AIR RATIO : 0.10

HGD OPERATING CONDITIONS

TEMPERATURE: 1010 F (AVERAGE)

PRESSURE : 143 PSIG (AVERAGE)

SPACE VELOCITY: 1025 HOURLY

	INLET MOLE% DRY BASIS (OIL FREE)	INLET MOLE% WET BASIS (OIL FREE)	EXIT MOLE% DRY BASIS (OIL FREE)	EXIT MOLE% WET BASIS (OIL FREE)
H2S	8.36		1.6 PPM	1.5 PPM
H2O				4.10
CO2	6.03		10.35	9.91
CO	22.06		17.61	16.85
H2	14.84		16.30	15.60
CH4	2.62		2.62	2.50
N2	47.03		49.03	47.70
O2	0.46		0.61	0.53
C2H6	0.25		0.03	0.03

TOTAL FLOW: 26,473 SCF 26,473 SCF 27,553 SCF

TOTAL LB MOLES: 69.84 69.84 72.66

FLOW RATE: 1077 SCFH 1077 SCFH 1121 SCFH

PURPOSE

SECOND SULFIDATION ON SAME BATCH OF SORBENT USING FIXED BED GASIFIER.

SORBENT ANALYSIS:

BEFORE AFTER

TOTAL SULFUR: (XWT)
SURFACE AREA: (SQ M/G)
DENSITY : (G/CC)
PORE VOLUME: (CC/G)
ELEMENTAL ANALYSIS:
TOTAL CARBON: (XWT)

EXIT SULFUR DATA:

H2S : 1.6 PPM

C4H4S: 6.7 PPM

CONDENSATE

AQUEOUS : 51.35 LBS (ESTIMATED)
HYDROCARBON : 5.74 LBS (ESTIMATED)
TOTAL : 57.1 LBS (MEASURED)

REMARKS

1. PACKED LENGTH IN REACTOR IS 46 7/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10700 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 50 7/8 INCHES
3. SECOND SULFIDATION ON SAME SORBENT
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED AND EXIT GAS SATURATION LEVEL BY STEAM TABLES.

CONCLUSIONS:

1. SORBENT CAN DESULFURIZE GASIFIER GAS CONTAINING TARS AND OILS DOWN TO LESS THAN 10 PPM TOTAL SULFUR.
2. PERFORMANCE IS REASONABLY GOOD AFTER A REGENERATION. BREAKTHROUGH TIME DECREASED FROM 20 HOURS TO 23 HOURS.
3. H2S WAS INITIALLY HIGH AND DROPPED DOWN TO LOW LEVELS AFTER 4 HOURS.
4. SO2 WAS DETECTED INITIALLY AND DROPPED DOWN TO 1 PPM AFTER FORTY MINUTES.

RUN 003 AVERAGE OPERATING PARAMETERS

AVERAGE(S) FOR PERIOD FROM: 13:30:15 6/25/1983 TO 14:59:22 6/27/1983

ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TESTS 001 AND 003

ZINC FERRITE SULFIDATION

INITIAL H₂S AND SO₂ CONCENTRATIONS

RUN 003 - H₂S CONCENTRATION AT PRODUCER CYCLONE EXIT

PERCENT H₂S
BENDIX 7000 #4 CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STCP TIME 15:00:00 06/27/1983

PERCENT CO2
BENDIX CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STOP TIME 15:00:00 06/27/1983

TEST 003
SHIFT CONVERSION

PERCENT CO
BENDIX CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STOP TIME 15:00:00 06/27/1983

TEST 003
SHIFT CONVERSION

PERCENT H₂
BENDIX CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STOP TIME 15:00:00 06/27/1983

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 004
DATE AND TIME STARTED : 08:12 6/28/83
DATE AND TIME ENDED : 18:50 6/28/83
TOTAL HOURS : 10.8
TYPE : REGENERATION
SULFUR REMOVED:

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/45 LBS
SORBENT NO. : UNITED CATALYSTS L-1504
SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE
50.0 MOLE% ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS

HGD OPERATING CONDITIONS

INITIAL TEMPERATURE: 968 F
PRESSURE : 139 INCHES OF WATER (AVERAGE)
SPACE VELOCITY: 657 HOURLY

		INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	37.5 SCFH	10.2	
H2O	14.3 #/HR	02	
N2	28.8 SCFH	7.8	

TOTAL LB MOLES:
FLOW RATE : 367 SCFH
\$

PURPOSE

SECOND REGENERATION OF SULFIDED SORBENT ON SIDESTREAM TEST UNIT.

SORBENT ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR: (XWT)		
SURFACE AREA: (SQ M/G)		
DENSITY : (G/CC)		
PORE VOLUME: (CC/G)		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		
TOTAL CARBON: (XWT)		

EXIT SULFUR DATA:

H2S :
SO2 : 16 PPM INITIALLY . HIGH OF 4 % AFTER TEN HOURS

CONDENSATE
AQUEOUS : 100.7 LBS
HYDROCARBON :
TOTAL : 100.7 LBS

REMARKS

1. PACKED LENGTH IN REACTOR IS 46 7/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10780 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.550 CUBIC FOOT
3. SECOND REGENERATION ON SULFIDED SORBENT
4. THERE WERE SOME DIFFICULTIES CONTROLLING THE STEAM AND AND AIR FLOWS.
5. MAXIMUM TEMPERATURE REACHED WAS 1667 F.

CONCLUSIONS

1. REGENERATION COMPLETED.
2. IMPROVED FLOW CONTROL FOR STEAM AND AIR ARE REQUIRED.

RUN 004 - REGENERATION

AVERAGE(S) FOR PERIOD FROM: 0:12:27 6/22/1983 TO 18:49:48 6/22/1983

RUN 004 - REGENERATION REACTOR TEMPERATURES

RUN 004 - REGENERATION

DURATION OF ANALYSIS

START	STOP
0 : 12 : 0 6 / 28 / 1983	18 : 50 : 0 6 / 28 / 1983
(0 : 12 : 27 6 / 28 / 1983)	(18 : 49 : 48 6 / 28 / 1983)

SLOT #	ENG ID	UNIT	SLOT DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ	MAX - FREQ
289	PI-418	PSIG	GAS OUT OF SYSTEM	1978	-4.095	10.23	-30.40	- 1
290	PI-402	PSIG	NITROGEN PRESSURE	1976	272.0	17.03	227.3	- 2
291	PI-406	PSIG	AIR PRESSURE	1976	29.28	21.98	0.2655E-01	- 1
292	PI-410	PSIG	STEAM PRESSURE	1976	106.2	8.953	75.55	- 1
293	PI-415	PSIG	ABSORBER INLET BOTTOM	1976	19.27	25.60	-4.691	- 1
294	PDI-427	PSI	ABSORBER DIFFERENCE PRESSURE	1976	-0.8959E-01	0.1666	-0.6136	- 1
295	PDI-412	PSI	CYCLONE DIFFERENCE PRESSURE	1976	-0.5084E-01	0.6927E-01	-0.2717	- 1
296	FI-416	MV	PROD. GAS OUT OF SYSTEM	1976	1451.	509.0	773.8	- 1
297	FI-428	MV	COOL H2O TO HX C18	1976	1637.	70.88	1123.	- 1
298	FI-401	MV	NITROGEN FLOW	1976	993.7	34.26	851.3	- 1
299	FI-405	MV	AIR FLOW	1976	1013.	24.63	981.1	- 1
300	FI-400	MV	STEAM FLOW	1976	1581.	881.2	922.0	- 1
301	FI-432	MV	COOL H2O TO HX C1C	1976	1044.	21.48	945.6	- 1
302	PI-420	*H2O	ABSORBER INLET TOP PRESSURE	1976	138.9	28.30	54.12	- 1
847	TI-413	DEGF	PREHEATER #1 GAS IN	1978	297.9	4.428	252.6	- 1
848	TI-414	DEGF	PREHEATER #1 GAS OUT	1978	632.2	10.04	584.9	- 2
849	TI-456	DEGF	H.X. C1A-2 GAS IN	1976	926.5	20.28	778.7	- 1
850	TI-458	DEGF	H.X. C1A-2 GAS OUT	1976	968.1	12.19	938.1	- 4
851	TI-421	DEGF	ABSORBER 5' FROM BOTTOM	1976	1173.	109.9	1007.	- 1
852	TI-422	DEGF	ABSORBER 15' FROM BOTTOM	1976	1346.	166.4	1057.	- 1
853	TI-423	DEGF	ABSORBER 25' FROM BOTTOM	1976	1289.	144.4	1049.	- 1
854	TI-424	DEGF	ABSORBER 35' FROM BOTTOM	1976	1127.	196.0	922.0	- 8
855	TI-425	DEGF	ABSORBER 45' FROM BOTTOM	1976	1018.	151.6	870.0	- 1
856	TI-426	DEGF	ABSORBER 55' FROM BOTTOM	1976	838.2	40.83	784.0	- 1
857	TI-448	DEGF	H.X. C1B GAS IN	1976	838.0	7.772	799.8	- 1
858	TI-430	DEGF	H.X. C1B GAS OUT	1976	276.5	23.27	192.9	- 1
859	TI-417	DEGF	H.X. C1C GAS OUT	1976	91.12	6.636	80.16	- 1
860	TI-419	DEGF	PREHEATER #2 GAS OUT	1976	1100.	29.71	349.9	- 1
861	TI-457	DEGF	H.X. C1A-1 GAS IN	1976	738.9	5.743	718.2	- 1
862	TI-459	DEGF	H.X. C1A-1 GAS OUT	1976	782.1	13.57	737.2	- 3
863	TI-429	DEGF	H.X. C1B COOL H2O OUT	1976	81.89	1.242	78.69	- 2
864	TI-433	DEGF	H.X. C1C COOL H2O OUT	1976	84.11	1.808	76.09	- 1
865	TI-447	DEGF	COOL H2O TO H.X.S	1976	71.48	0.5789	69.53	- 1
866	TI-411	DEGF	STEAM	1976	344.0	4.434	339.5	- 1
867	TI-403	DEGF	N2	1976	85.79	2.630	80.84	- 1
869	TI-407	DEGF	AIR	1976	86.85	2.577	81.91	- 1
870	TI-461	DEGF	CYCLONE OUTLET	1976	259.3	18.37	231.8	- 1
3750	FI-416X	SCFH	PROD. GAS OUT OF SYSTEM	1976	147.8	137.0	0.0000E+00	- 588
3751	FI-428X	GPM	COOL H2O TO HX C1B	1976	3.986	0.2294	1.752	- 1
3752	FI-401X	SCFH	NITROGEN FLOW	1976	88.82	34.47	0.0000E+00	- 1148
3753	FI-405X	SCFH	AIR FLOW	1976	37.46	25.64	0.0000E+00	- 506
3754	FI-400X	\$/HR	STEAM FLOW	1976	14.27	12.17	0.0000E+00	- 170
3755	FI-432X	GPM	COOL H2O TO H.X. C1C	1976	0.9937	0.3556	0.0000E+00	- 188

RUN 004 - REGENERATION STEAM AND AIR FLOWS

1=53754 2=53753

START TIME OF PLOT

0:12: 0

6/28/1983

STOP TIME OF PLOT

18:50: 0

6/28/1983

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 085
DATE STARTED : 11:00 6/29/83
DATE ENDED : 17:30 6/30/83
TOTAL HOURS : 23.15
TYPE : SULFIDATION
SULFUR REMOVED: LB S8

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/45 LBS
SORBENT NO. : UNITED CATALYSTS L-1504
SORBENT COMPOSITION: 50.0 MOLEX IRON OXIDE
50.0 MOLEX ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
GASIFIER OPERATING CONDITIONS

GASIFIER TYPE : DOE/METC FIXED BED WELLMAN GALUSHA
COAL TYPE : ARKWRIGHT
STEAM/COAL RATIO: 0.500 ON AIR BLOWN 2.344 ON OXYGEN BLOWN
STEAM/AIR RATIO : 0.193
STEAM/OXYGEN RATIO: 3.366
HGD OPERATING CONDITIONS
TEMPERATURE: 1812 F (AVERAGE)
PRESSURE : 91 PSIG (AVERAGE)
SPACE VELOCITY: 1846 HOURLY FIRST PORTION
347 HOURLY SECOND PORTION

	INLET AIR BLOWN MOLEX (OIL FREE) DRY WET		INLET O2 BLOWN MOLEX (OIL FREE) DRY WET		EXIT AIR BLOWN MOLEX (OIL FREE) DRY WET		EXIT O2 BLOWN MOLEX (OIL FREE) DRY WET	
	H2S	0.38		0.66		3 PPM	2.85 PPM	
H2O					4.28		53	
CO2	7.16		18.88		12.50	12.13		
CO	24.81		24.92		16.33	15.73		
H2	15.06		29.50		17.86	17.21		
CH4	3.14		6.36		2.84	2.73		
N2	51.12		1.01		47.20	45.49		
O2	0.54		0.01		0.50	0.48		
C2H6	0.40		0.62		0.34	0.03		
TOTAL FLOW (SCF)	6405		5447	11423	6405	6645	5447	11423
LB MOLE	16.9		14.37	30.14	16.9	17.53	14.37	30.14
FLOW RATE (SCFH)	949		340	714	945	984	340	714

PURPOSE

THIRD SULFIDATION ON SAME BATCH OF SORBENT USING FIXED BED GASIFIER.

SORBENT ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR: (%WT)		8.6-31.41 (22.83 AVG)
SURFACE AREA: (SQ M/G)		2.16 (AVG)
DENSITY : (G/CC)		
PORE VOLUME: (CC/G)		0.0039 (AVG)
ELEMENTAL ANALYSIS:		
TOTAL CARBON: (%WT)		2.63 (AVG)

EXIT SULFUR DATA:

H2S : 3 PPM ON AIR BLOWN GAS
C4H4S: 4 PPM ON AIR BLOWN GAS

	AIR BLOWN (7.15 HRS)	O2 BLOWN (16 HRS)
CONDENSATE		
AQUEOUS	11.42 LBS	284 LBS
HYDROCARBON	1.28 LBS	10 LBS
TOTAL	12.7 LBS	294 LBS

REMARKS

1. PACKED LENGTH IN REACTOR IS 46 7/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10780 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 7/8 INCHES
3. THIRD SULFIDATION ON SAME SORBENT
4. PORTIONS OF THE RUN WERE AIR BLOWN, PORTIONS O2 BLOWN.
5. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED AND EXIT GAS SATURATION LEVEL BY STEAM TABLES.

CONCLUSIONS

1. SORBENT CAN DESULFURIZE GASIFIER GAS CONTAINING TARS AND CILS DOWN TO LESS THAN 10 PPM TOTAL SULFUR.
2. PERFORMANCE ON AIR BLOWN GAS IS REASONABLY GOOD AFTER A REGENERATION, BUT POOR ON OXYGEN BLOWN GAS.
3. H2S WAS INITIALLY HIGH AND DROPPED DOWN TO LOW LEVELS AFTER 3 HOURS.
4. SO2 WAS DETECTED INITIALLY AND DROPPED DOWN TO 1 PPM AFTER FORTY MINUTES.

RUN 005A

AVERAGE OPERATING PARAMETERS

AVERAGE(S) FOR PERIOD FROM: 11: 0:18 6/29/1983 TO 8:24:11 6/30/1983

RUN 005B

AVERAGE OPERATING PARAMETERS

AVERAGE(S) FOR PERIOD FROM: 12:45:18 6/20/1983 TO 17:29:11 6/20/1983

FIGURE 8B

RUN 005

HGCLEAN

DURATION OF ANALYSIS

START

11 : 0 : 0 6 / 29 / 1983
(11 : 0 : 18 6 / 29 / 1983)

STOP

17 : 30 : 0 6 / 30 / 1983
(17 : 29 : 41 6 / 30 / 1983)

SLOT #	ENG ID	UNIT	SLOT DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ	MAX - FREQ
289	PI-418	PSIG	GAS OUT OF SYSTEM	3629	85.75	25.78	0.2752	155.3
290	PI-402	PSIG	NITROGEN PRESSURE	3629	298.1	11.64	234.5	321.2
291	PI-406	PSIG	AIR PRESSURE	3629	92.95	2.907	78.88	96.77
292	PI-410	PSIG	STEAM PRESSURE	3629	-0.3998	0.3547	-1.665	0.9722
293	PI-415	PSIG	ABSORBER INLET BOTTOM	3629	91.21	25.79	5.242	161.2
294	PDI-427	PSI	ABSORBER DIFFERENCE PRESSURE	3629	0.1687E-02	0.1614E-01	-0.4796E-01	0.5454E-01
295	PDI-412	PSI	CYCLONE DIFFERENCE PRESSURE	3629	2.158	1.639	-0.8265	7.533
296	FI-416	MV	PROD. GAS OUT OF SYSTEM	3629	1528.	480.9	272.4	8179.
297	FI-428	MV	COOL H2O TO HX C1B	3629	1538.	63.87	1323.	1748.
298	FI-401	MV	NITROGEN FLOW	3629	1236.	541.3	971.6	5264.
299	FI-405	MV	AIR FLOW	3629	1010.	6.816	985.7	1032.
300	FI-409	MV	STEAM FLOW	3629	525.9	302.0	312.0	1023.
301	FI-432	MV	COOL H2O TO HX C1C	3629	1041.	0.074	1012.	1072.
302	PI-420	M2O	ABSORBER INLET TOP PRESSURE	3629	11.64	11.38	-0.2444	82.41
847	TI-413	DEGF	PREHEATER #1 GAS IN	3629	-128.8	1576.	-2941.	1665.
848	TI-414	DEGF	PREHEATER #1 GAS OUT	3629	989.4	66.10	704.6	1105.
849	TI-456	DEGF	H.X. C1A-2 GAS IN	3629	199.4	39.69	153.3	468.6
850	TI-458	DEGF	H.X. C1A-2 GAS OUT	3629	143.4	97.84	92.13	699.2
851	TI-421	DEGF	ABSORBER 5" FROM BOTTOM	3629	993.1	16.17	941.4	1022.
852	TI-422	DEGF	ABSORBER 15" FROM BOTTOM	3629	1010.	16.49	953.8	1042.
853	TI-423	DEGF	ABSORBER 25" FROM BOTTOM	3629	1014.	19.65	969.6	1072.
854	TI-424	DEGF	ABSORBER 35" FROM BOTTOM	3629	1025.	25.38	968.6	1096.
855	TI-425	DEGF	ABSORBER 45" FROM BOTTOM	3629	1023.	34.07	954.3	1117.
856	TI-426	DEGF	ABSORBER 55" FROM BOTTOM	3629	1006.	33.38	943.2	1086.
857	TI-448	DEGF	H.X. C1B GAS IN	3629	738.7	50.73	624.0	803.1
858	TI-430	DEGF	H.X. C1B GAS OUT	3627	491.9	56.43	335.1	570.0
859	TI-417	DEGF	H.X. C1C GAS OUT	3629	155.8	35.56	97.05	245.7
860	TI-419	DEGF	PREHEATER #2 GAS OUT	3629	661.5	157.8	531.2	936.6
861	TI-457	DEGF	H.X. C1A-1 GAS IN	3629	875.3	33.33	800.2	930.2
862	TI-459	DEGF	H.X. C1A-1 GAS OUT	3629	821.5	41.50	729.9	877.9
863	TI-429	DEGF	H.X. C1B COOL H2O OUT	3629	91.65	6.001	79.01	106.9
864	TI-433	DEGF	H.X. C1C COOL H2O OUT	3629	103.2	17.59	76.69	137.9
865	TI-447	DEGF	COOL H2O TO H.X.S	3629	71.31	0.5010	69.71	73.58
866	TI-411	DEGF	STEAM	3629	79.99	3.900	72.48	91.07
867	TI-403	DEGF	N2	3629	79.10	5.000	67.73	92.30
869	TI-407	DEGF	AIR	3629	79.37	4.872	71.22	91.03
870	TI-461	DEGF	CYCLONE OUTLET	3629	500.2	76.76	325.8	608.2
3750	FI-416X	SCFH	PROD. GAS OUT OF SYSTEM	3629	531.1	314.0	0.0000E+00	1473.
3751	FI-428X	GPM	COOL H2O TO HX C1B	3629	3.662	0.2196	2.840	4.325
3752	FI-401X	SCFH	NITROGEN FLOW	3629	134.0	233.9	0.0000E+00	1137.
3753	FI-405X	SCFH	AIR FLOW	3629	32.02	32.56	0.0000E+00	92.06
3754	FI-409X	\$/HR	STEAM FLOW	3629	0.7717E-03	0.8684E-01	0.0000E+00	0.9692
3755	FI-432X	GPM	COOL H2O TO H.X. C1C	3629	1.011	0.1148	0.5424	1.339

ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TEST C05

ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TEST 005

9-AUG-83 20:31:24

RUN 005 - HGD REACTOR TEMPERATURES

1-S 851	2-S 852	3-S 853	4-S 854	5-S 855
START TIME OF PLOT		11: 0: 0	6/29/1983	
STOP TIME OF PLOT		17:30: 0	6/30/1983	

RUN 005 - H₂S CONCENTRATION AT PRODUCER CYCLONE EXIT

PERCENT H₂S

BENDIX 7000 #4 CHROMATOGRAPH

START TIME 11:00:00 06/29/1983

STOP TIME 17:30:00 06/30/1983

PERCENT C₂H₆
BENDIX 7000 #4 CHROMATOGRAPH
START TIME 11:00:00 06/29/1983
STOP TIME 17:30:00 06/30/1983

PERCENT C₂H₆
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 11:00:00 06/29/1983
STOP TIME 17:30:00 06/30/1983

PERCENT H₂S
BENDIX 6000 #1 CHROMATOGRAPH
START TIME 11:00:00 06/29/1983
STOP TIME 17:30:00 06/30/1983