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Fluidized-Bed Testing of Z-Sorb III Sorbent

Topical Report

R. P. Gupta
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August 1994

Work Performed Under Contract No.: DE-AC21-88MC25006

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

By
Research Triangle Institute
Research Triangle Park, North Carolina

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ABSTRACT

Phillips Petroleum Company (PPCo) successfully developed a fluidizable version of their proprietary Z-SORB sorbent. Z-SORB sorbent is a ZnO-based regenerable sorbent for removing hydrogen sulfide (H_2S) and carbonyl sulfide (COS). RTI conducted a life-cycle test on this sorbent in the high-temperature, high-pressure (HTHP) semi-batch fluidized-bed reactor. This test consisted of 50 cycles of sulfidation and regeneration to demonstrate the long-term chemical reactivity and mechanical strength of the Z-SORB sorbent. A simulated air-blown gasifier coal gas was used at 20 atm and 538 °C (1,000 °F).

The Z-SORB sorbent exhibited excellent sulfur removal capability; the prebreakthrough H_2S levels were below the detection limit of the analyzer (<10 ppmv). The sulfur capacity of the sorbent at breakthrough (500 ppm H_2S in reactor exit gas) was 20.2 g S/100 g sorbent in Cycle 1 and was 10 g S/100 g sorbent in Cycle 50. The sorbent loss from the reactor due to fines generation was small. While no significant change in particle size was observed, the bulk density increased by 8 percent over 51 cycles. The attrition resistance of the sorbent after the 51 cycles was slightly lower than the fresh material. The thermogravimetric analyzer (TGA) tests on fresh and reacted sorbents confirmed the sulfur capacity decline in the bench tests; however, the TGA data indicated no change in the H_2S absorption rate between the fresh and reacted sorbents. The regeneration of the sulfided sorbent was successfully carried out using 2 to 2.5 percent O_2 in N_2 at a temperature of 649 to 704 °C (1,200 to 1,300 °F) with no evidence of sulfate formation. Overall, the sorbent exhibited good performance.

ACKNOWLEDGMENTS

This work was sponsored by the Morgantown Energy Technology Center (METC) of the U.S. Department of Energy (DOE) under Contract No. DE-AC21-88MC25006. This test was carried out in close cooperation with Phillips Petroleum Company (PPCo) of Bartlesville, OK. We would like to express our appreciation to Drs. Gil J. Greenwood, Gary A. Delzer, and Donald H. Kubicek for many helpful suggestions. Valuable guidance provided by Messrs. Suresh C. Jain and Daniel C. Cicero of METC as Contracting Officer's Representatives (CORs) is gratefully acknowledged. Messrs. D.A. Ward and E.W. Johnson carried out the experimental work.

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1.0 INTRODUCTION

The Phillips Petroleum Company (PPCo) developed the Z-SORB sorbent in the late 1980s for tail-gas cleanup (Brinkmeyer and Delzer, 1990). It is a ZnO-based regenerable sorbent for capturing hydrogen sulfide (H_2S) and carbonyl sulfide (COS). Z-SORB sorbent, a proprietary sorbent of PPCo, is based on zinc oxide supported in a porous matrix and contains a nickel oxide promoter (Delzer et al., 1993).

Following extensive decrepitation of zinc titanate sorbents during multicycle testing in a fixed bed at the U.S. Department of Energy/Morgantown Energy Technology Center (DOE/METC) (Mei et al., 1993), fixed-bed tests were performed on the Z-SORB sorbent under a Cooperative Research and Development Agreement (CRADA) between DOE and the M.W. Kellogg Company (Kellogg). During these fixed-bed tests, Z-SORB sorbent exhibited excellent chemical reactivity and mechanical stability (Campbell et al., 1993).

In an ongoing sorbent research and development program, PPCo prepared a fluidized-bed version of the Z-SORB sorbent. Testing of this fluidizable Z-SORB sorbent in Kellogg's transport reactor system indicated excellent sorbent performance. The next logical step was to determine the long-term chemical reactivity and mechanical strength of the sorbent at RTI.

To this end, a life-cycle test consisting of 50 cycles of sulfidation and regeneration was performed in RTI's bench-scale high-temperature, high-pressure (HTHP) fluidized-bed reactor test facility. This report describes the details of this experimental testing and pertinent findings of this life-cycle test.

This report contains sulfur reactivity and limited physical and chemical characterization data on this test because of a secrecy and nonanalysis agreement between PPCo and RTI. As per the terms of this agreement, RTI can only report the summary information on the following to DOE/METC:

- Sorption and regeneration characteristics,
- Particle size,
- Bulk density,
- Attrition resistance, and
- Total sulfur.

2.0 EXPERIMENTAL PROCEDURES

2.1 EXPERIMENTAL APPARATUS

The HTHP bench-scale sorbent test facility employed in this test has been described in detail previously (Gupta and Gangwal, 1992; 1993). Figure 1 shows a schematic of this test facility. It is a skid-mounted system capable of operation at up to 870 °C (1,600 °F) at 20 atm (294 psia) pressure.

The reactor is constructed using a 4-in., schedule-160, 316 stainless-steel pipe. Most of the other system components are constructed with either 316 or 304 stainless steel. All hot H_2S-wetted parts are Alon-processed (a high-temperature aluminum vapor treatment) to prevent corrosion of stainless steel by sulfur gases in the presence of steam. The main components of the reactor facility are

- Gas delivery system,
- Reactor assembly,
- Data acquisition and process control,
- Gas analysis system, and
- Reactor offgas venting system.

Each component is briefly described below.

A battery of seven mass flow controllers (MFCs) capable of operation at pressures up to 100 atm controls the flow rate and composition of simulated coal gas using bottled gases for CO, H_2, CO_2, N_2, H_2S, O_2, and air. A series of positive displacement pumps feed deionized water to vaporizer at any given flow rate to generate steam. The delivery system can generate simulated coal gasifier gases representative of all types of gasifiers.

The fluidized-bed desulfurization reactor is also shown in Figure 1. The unique feature of this reactor is a removable cage for easy loading and unloading of the sorbent. The reactor can accommodate both 7.26-cm (3-in.) and 5.1-cm (2-in.) diameter sorbent cages. A removable α-alumina distributor plate is positioned at the bottom of each cage to introduce hot coal gas into the reactor. The reactor is housed inside a three-zone furnace equipped with separate temperature controllers for each zone and the furnace can heat the reactor up to 870 °C (1,600 °F). Ceramic thimble filters downstream of the reactor capture particles from the sulfidation and regeneration exit lines upstream of the condensers. The reactor exit gas, after passing through the thimble filters, is cooled using double pipe heat exchangers.

The reactor temperature is monitored at the bed inlet below the distributor, halfway in the bed, and near the bed outlet in the freeboard region of the fluidized bed using Type-K thermocouples. The thermocouples, equipped with a digital display, are connected to a data acquisition system described later. Pressure is controlled precisely by two back pressure regulators (BPRs) in series. A differential pressure indicator across the reactor provides an indication of fluidization behavior of the hot sorbent in the reactor.

A small slipstream of steam-free gas from the reactor is diverted to an online gas analysis system, which consists of three gas chromatographs (GCs): a Carle series 400 AGC with a

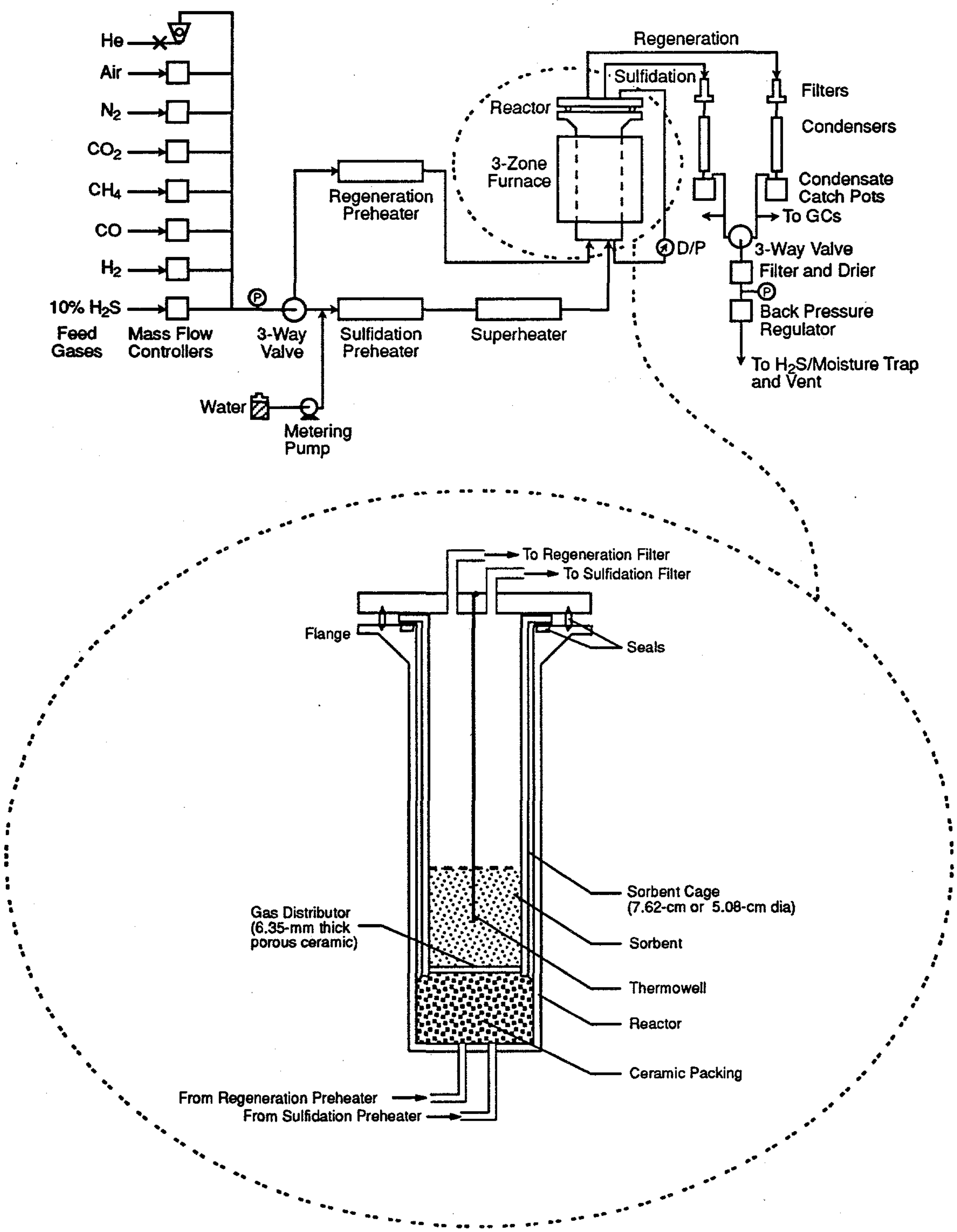

Figure 1. Bench-scale fluidized-bed sorbent test facility.

thermal conductivity detector (TCD), a Varian 3300 with a *flame photometric detector* (FPD), and a Hewlett Packard 5890 equipped with thermal conductivity and photoionization detectors. The Varian 3300 FPD is connected to an HP 3365 Series II Hewlett-Packard Chem Station. Equipped with an extensive range of sampling, column, and detector options, the HP 3365 Chem Station automates sample injections. The Carle TCD is connected to a Spectra Physics SP4270 integrator to intermittently measure the gas composition. Multiple GC sampling valves and dual loops in the Varian FPD measure H_2S and COS from 1,500 ppmv to <0.1 ppmv every 5 min. High concentrations of H_2S, SO_2, and other bulk gases (H_2, CO_2, N_2, O_2, CH_4, and CO) are measured every 30 min with the Carle TCD to evaluate mass balance. Formation of small quantities of methane during sulfidation and carbon dioxide during regeneration is measured every 2 min by the HP 5890 GC connected to HP3396 Series II integrator. An online continuous SO_2 analyzer, Model 721 AT2 from Western Research, is used to measure the SO_2 evolution during sorbent regeneration.

A state-of-the-art digital data acquisition system is employed for collection of a real-time data using a digital OM-480 Modular Data Logging System from Omega Instruments (Model No. 163568-96A). This system permits continuous data acquisition via a LabTech Note-Book software using an IBM personal computer. Audible alarms for low and high limits on various gas flow rates and temperatures can be programmed, allowing efficient and uninterrupted operation of the test facility.

This data-logging module has 16 separate channels. Each channel accepts one input. The input can be a voltage signal (0 to 5V), a direct thermocouple input (no transducer required), or a current signal (mA). This allows connection of the output signal from the MFCs and thermocouples directly into the module without introducing any system noise. Out of 16 channels, eight channels are used for monitoring the flow rates from MFCs (N_2, H_2, CO, CO_2, air H_2S/H_2, CH_4, and an additional N_2). Six channels are used for monitoring temperatures. Temperature is monitored in the sorbent bed, reactor freeboard, superheater, sulfidation condenser, and sulfidation and regeneration filters. One channel is used to gather SO_2 concentration data from the online SO_2 analyzer. The remaining channel is used for logging the data from a mass flow meter which measures the total dry gas flow.

The output from the OM-480 module is connected to an IBM personal computer via an RS232 serial cable. The LabTech Note-Book software is a real-time data-acquisition program which controls inflow and outflow of information from the various channels. It is an integrated software package for data acquisition, process control, monitoring, and data analysis. Using this software, collected data can be processed in real time through an extensive list of mathematical functions, displayed to the screen, and logged to a disk in a format directly compatible with most spreadsheet programs.

The typical frequency of data collection in the bench run is 30 sec. Various temperatures and flow rates are digitally and graphically displayed on the computer screen during the entire run. Any system disturbances are instantly displayed on the screen in addition to activation of an audible alarm.

The sulfidation exit gas containing toxic CO and H_2S and regeneration off-gas containing toxic SO_2 are properly disposed. A high-powered blower is used to dilute the gases by a factor of 100 before emitting them into the atmosphere. The calculations of total CO and S emissions from these tests indicated no environmental problems.

2.2 Sorbent Characteristics

The sorbent used in this study was a proprietary Z-SORB III sorbent. It was supplied to RTI by PPCo. Table 1 lists various physical properties of this sorbent.

Table 1. Physical Properties of Z-SORB III Sorbent

Particle size range	100 to 300 μm
Average particle size	174.3 μm[a]
Compacted bulk density	66.54 lb/ft^3
Loss of volatiles	~7%
Attrition Resistance	
5-h loss	16.0%
20-h loss	32.4%
Particle Size Distribution	
MESH	wt%
+ 50	0
- 50 + 60	10.44
- 60 + 80	57.00
- 80 + 100	10.75
- 100 + 120	7.28
-120 + 140	5.68
- 140 + Pan	8.85
Total	100.0

[a] Based on Harmonic mean.

Figure 2 shows the TGA reactivity profile of the fresh Z-SORB III sorbent. The initial decline in the weight (A→B) is due to loss of volatiles (~7%) from the sorbent. The second weight loss (B→C) is probably due to reduction of metal oxides in the presence of H_2S-free clean coal gas, followed by a rapid weight gain (C→D) during sulfidation at 550 °C (1,022 °F). From point D to E, sorbent was regenerated at 650 °C (1,202 °F), thus completing the first cycle. During the reduction in the second cycle, sorbent again lost the weight (E→F), followed by a rapid weight gain during the extended sulfidation (F→G). Based on the above TGA data, sulfur capture capacity of the sorbent is about 22 wt%.

2.3 EXPERIMENTAL APPROACH

Table 2 lists the nominal test conditions for the 50-cycle test. The composition of simulated U-Gas gasifier gas is specified in Table 3.

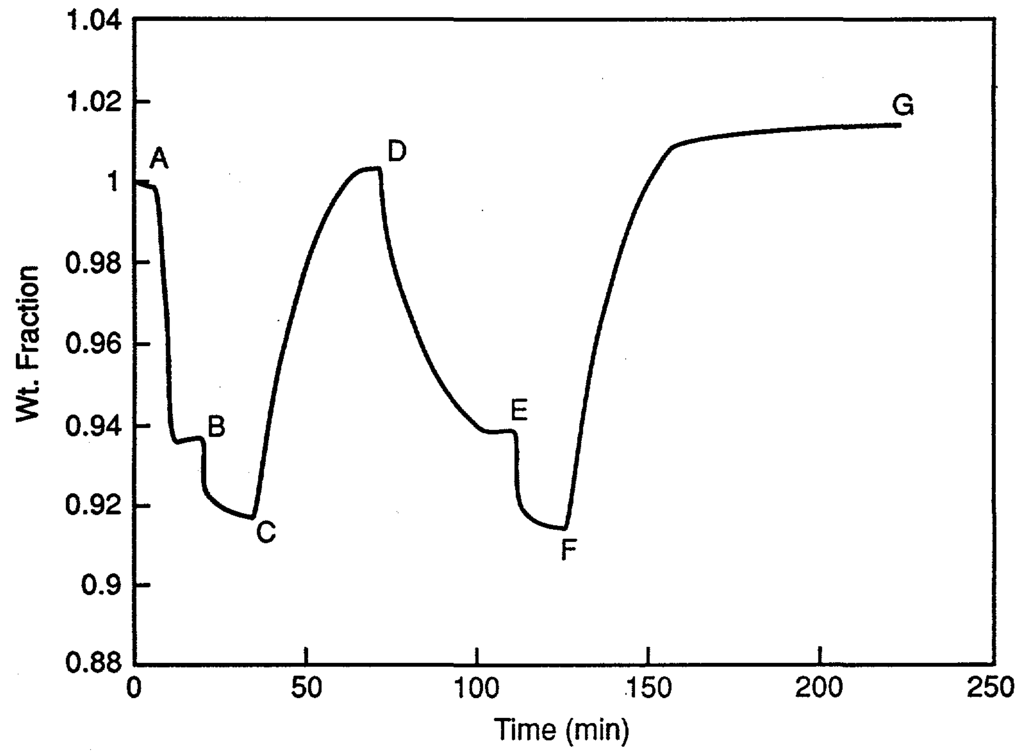

Figure 2. TGA chemical reactivity of the fresh Z-SORB III sorbent.

2.3.1 Test Procedure

Based on the previous 100-cycle bench run with ZT-4 zinc titanate sorbent as described in our earlier topical report (Gupta and Gangwal, 1993), the test procedure followed is described in Sections 2.3.1.1 through 2.3.1.5.

2.3.1.1 Startup

1. Five hundred grams of as-received prescreened sorbent in the particle diameter range of 100 to 300 μm were loaded into the 2-in. ID sorbent cage. The cage was then placed in the reactor shell and the entire reactor system (described previously) was assembled. New thimble filters were mounted on sulfidation and regeneration lines. Following this assembly, the reactor was leak-tested.

2. The reactor was pressurized to 280 psig and heated to 538 °C (1,000 °F) with a continuous flow of nitrogen through the reactor.

2.3.1.2 Run Procedure

1. Once the sorbent bed attained the desired temperature (538 °C [1,000 °F]), it was sulfided by flowing the simulated coal gas through it. The composition of the gas exiting the reactor was measured continuously using the series of GCs described previously.

Table 2. Nominal Test Conditions Used in 50-Cycle Test

Sulfidation	
Sorbent	Z-SORB III
Temperature	538 °C (1,000 °F)
Pressure	20 atm (294 psia)
Sorbent particle size	100 to 300 µm (d_{50} ≈ 174.3 µm)[a]
Sorbent inventory	500 g (Cycles 1 to 25)[b] 400 g (Cycles 26 to 51)[c]
Total gas flow rate	75 slpm (Cycles 1 to 25) 60 slpm (Cycles 26 to 35) 75 slpm (Cycles 36 to 51)
Superficial gas velocity	9.14 cm/sec (0.3 ft/sec) [Cycles 1 to 25] 7.31 cm/sec (0.24 ft/sec) [Cycles 26 to 35] 9.14 cm/sec (0.3 ft/sec) [Cycles 36 to 51]
Sulfidation gas	Simulated U-Gas gasifier gas[d]
H_2S content of coal gas	5,000 ppmv (0.5 mol%)
Reactor tube diameter	5.08-cm (2 in.-ID)
Regeneration	
Initial temperature	580 to 650 °C (1,076 to 1,202 °F)
Maximum temperature	690 to 780 °C (1,274 to 1,436 °F)
Pressure	20 atm (294 psia)
Gas flow	Same as sulfidation[e]
Regeneration gas	2 to 2.5% O_2 in N_2

[a] d_{50} is the harmonic mean (see Table 1).
[b] Weight of as-received sorbent. On heating, sorbent lost 35 g weight due to loss of volatiles.
[c] Weight of the sorbent in sulfided state containing 12.15 wt% S. The estimated weight of regenerated sorbent would be 375.7 g.
[d] Complete gas composition is specified in Table 3.
[e] The same flow rates were used during regeneration as sulfidation (i.e., 75, 60, 75 slpm).

Table 3. Nominal Gas Composition of the Simulated U-Gas

Component	Vol%
H_2	14.2
CO_2	5.8
H_2O	6.6
H_2S	0.5 (5,000 ppmv)
N_2	49.8
CO	23.1
Total	100.0

Sulfidation of the sorbent was continued until the H_2S concentration in the reactor exit gas reached 500 ppmv, defined as the breakthrough point. At this point, the flow of coal gas was stopped and switched to nitrogen to purge the reactor. Purging of the reactor was continued for about 60 min. The furnace settings were increased to about 600 °C (1,112 °F) for regeneration.

2. Once the sorbent bed attained a temperature in the vicinity of 600 °C (1,112 °F), the sulfided sorbent was regenerated using a gas mixture containing air and nitrogen. The flow rates of air and nitrogen were adjusted to obtain an oxygen concentration of about 2 to 2.5 mol%.

   Sorbent regeneration was continued until the SO_2 concentration in the regeneration tail gas fell below 250 ppmv. At this point, the regeneration gas flow was switched to N_2 purge.

3. Steps 1 and 2 were repeated for subsequent cycles.

2.3.1.3 Shutdown

In this long-duration run, the reactor was kept hot with a continuous nitrogen purge for the first 24 complete cycles (including weeknights and weekends). After completion of the 25th sulfidation, the reactor was shut down. The shutdown procedure consisted of the following five steps:

1. The main reactor furnace, preheater, superheater, and variacs controlling various heat tapes were switched off;

2. Nitrogen purge was continued for the entire duration of reactor cooling;

3. The reactor was depressurized by opening the BPR to the fully open position;

4. The sorbent bed temperature was continuously monitored during the cooling process; and

5. When the sorbent bed temperature reached nearly ambient, the reactor was dismantled for removal of the sorbent.

2.3.1.4 Restart of the Reactor

Following the sorbent characterization after the 25th sulfidation, the test was resumed. First the regeneration of the sulfided sorbent was carried out. The usual test procedure was then followed for the remaining 25 cycles.

2.3.1.5 Changes In Run Conditions

As mentioned previously, the run was stopped after Sulfidation 25 to perform the sorbent characterization. When the run was resumed, only 400 g of the sulfided sorbent was loaded in the reactor because other 100 g of the sorbent was used for characterization. To obtain approximately the same gas residence time, the coal gas flow rate was reduced by 20 percent to 60 slpm, thus keeping the estimated breakthrough time approximately the same as in previous cycles.

It was found, however, that a flow rate of 60 slpm (during Cycles 26 to 35), sorbent bed was not fluidizing as vigorously as it did in the first 25 cycles. Thus, the total coal gas flow was restored to 75 slpm, thereby reducing the gas residence time through the bed. This, in turn, reduced the breakthrough time, which necessitated normalization of breakthrough data as will be discussed in the next section. In all the cycles, the total gas flow during the regeneration was kept identical to that of the sulfidation.

3.0 EXPERIMENTAL RESULTS

3.1 BREAKTHROUGH BEHAVIOR

H_2S and COS concentrations in reactor outlet gas were measured every 5 min to determine the sorbent's desulfurization efficiency. The raw breakthrough data for H_2S, COS, and SO_2 concentrations are listed in Appendix A for Cycles 1 to 50. Figure 3 shows the H_2S breakthrough curves for selected cycles. As mentioned in Table 2 and Section 2.3, the amount of the sorbent and the coal gas flow rates were changed twice during the 50 cycles, which necessitated normalization of the breakthrough data. Therefore, the abscissa in Figure 3 is shown in terms of dimensionless time. This dimensionless time is obtained by dividing the actual time (t) by a characteristic time (T), defined as

$$T = \frac{W_o \lambda_o}{G Y_{H_2S,\ inlet}}$$

W_o = Sorbent charge in the reactor
λ_o = Moles of ZnO per g of the sorbent
G = Total molar flow rate of coal gas
$Y_{H_2S,\ inlet}$ = Inlet H_2S concentration of coal gas in mole fraction units.

Table 4 shows the values of various parameters during the run.

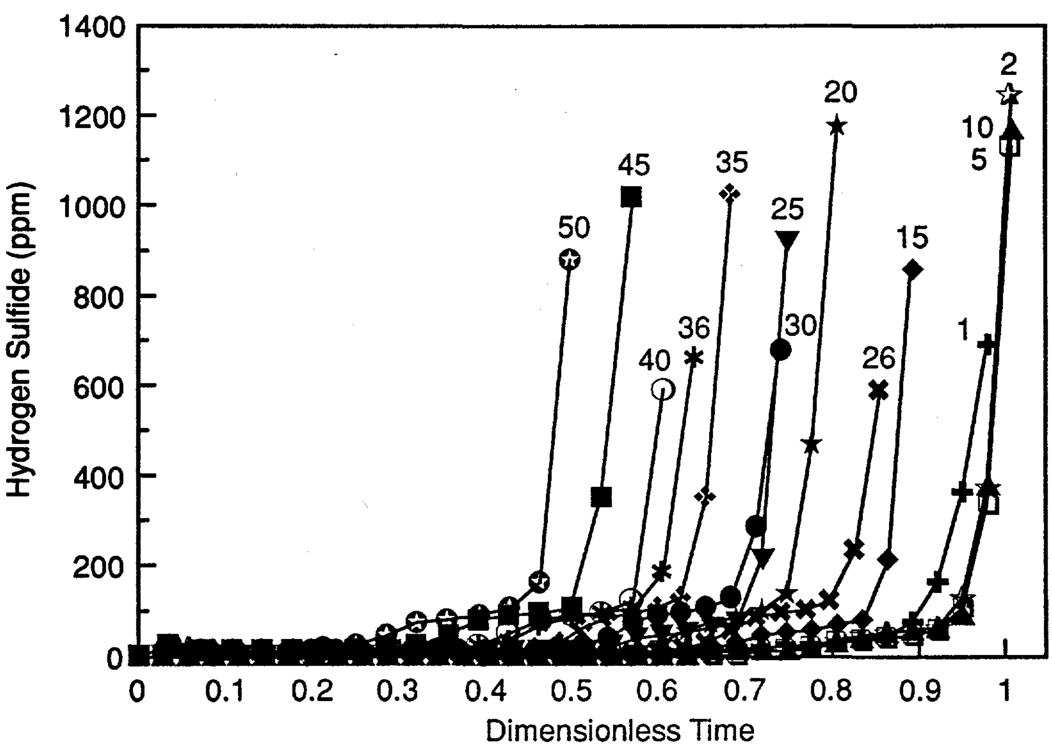

Figure 3. Breakthrough behavior of Z-SORB III sorbent during selected cycles.

Table 4. Various Run Parameters

Cycle No.	W_o (g)	λ_o [a] (mol/g)	G [b] (mol/min)	$Y_{H_2S,\ inlet}$ [c] (mol fraction)	T (min)
1 to 25	465 [d]	0.00625	3.348	0.005	173.60
26 to 35	375.7 [e]	0.00625	2.678	0.005	175.33
36 to 51	375.7	0.00625	3.348	0.005	140.26

[a] Estimated assuming saturation sulfur capacity of 20 lb/100 lb of fresh sorbent.
[b] Obtained by dividing the volumetric flow rate @ STP by 22.4.
[c] 5,000 ppmv H_2S in feed.
[d] Calculated assuming 7 percent loss of volatiles during heating.
[e] Estimated assuming 12.15 percent S on the sorbent.

The above normalization permits construction of a series of breakthrough curves continuously as shown in Figure 3. Essentially, the values shown on abscissa indicate the utilization of sorbent's sulfur capture capacity at breakthrough. As can be seen, during the first 10 cycles, nearly complete capacity utilization is exhibited by the sorbent, which drops down to about 50 percent in Cycle 50, indicating a loss of sorbent reactivity with cycling. The prebreakthrough H_2S concentration was consistently below the detection limit of the analyzer.

3.2 SORBENT SULFUR CAPACITY AT BREAKTHROUGH

From the breakthrough data, the sulfur capture capacity of the sorbent was calculated for Cycles 1 to 50. Figure 4 shows the actual sulfur absorbed by the sorbent as a function of cycle number at breakthrough—500 ppmv H_2S in reactor outlet gas. During the first 10 cycles, the sulfur capacity remained nearly constant at 20.16 g S/100 g fresh sorbent, indicating nearly complete capacity utilization. A gradual decline in sulfur capacity can be seen thereafter. The sulfur capacities of the sorbent during the 25th and 50th cycles were 14.98 and 9.98 g S/100 g fresh sorbent, respectively, indicating about 25 percent drop during the first 25 cycles, increasing to about 50 percent during the remaining 25 cycles.

3.3 SORBENT LOSS FROM THE REACTOR

Initially 500 g of the as-received sorbent were charged, which during initial heating lost 35 g weight due to loss of volatiles, thus leaving 465 g of the sorbent in the reactor. After the 25th sulfidation, the reactor was cooled and dismantled to remove the sorbent. Examination of reactor inside walls and lines indicated that sulfidation and regeneration lines had considerable amount of particle buildup. The total weight of particles collected from both the lines was about 2 g, out of which about 80 percent came from sulfidation line. Examination of sulfidation filter revealed that it was black coated with a layer of black particles beneath which a layer of shiny metal was observed. The total weight of the particles collected from the sulfidation filter was 5.4 g. The regeneration filter also had a thin layer of particles, in the form of black flakes. The total weight of the extraneous material on the filter was 1.9 g. The weight of the sorbent removed from the cage was 491.2 g. Table 5 shows a material balance for the first 25 cycles.

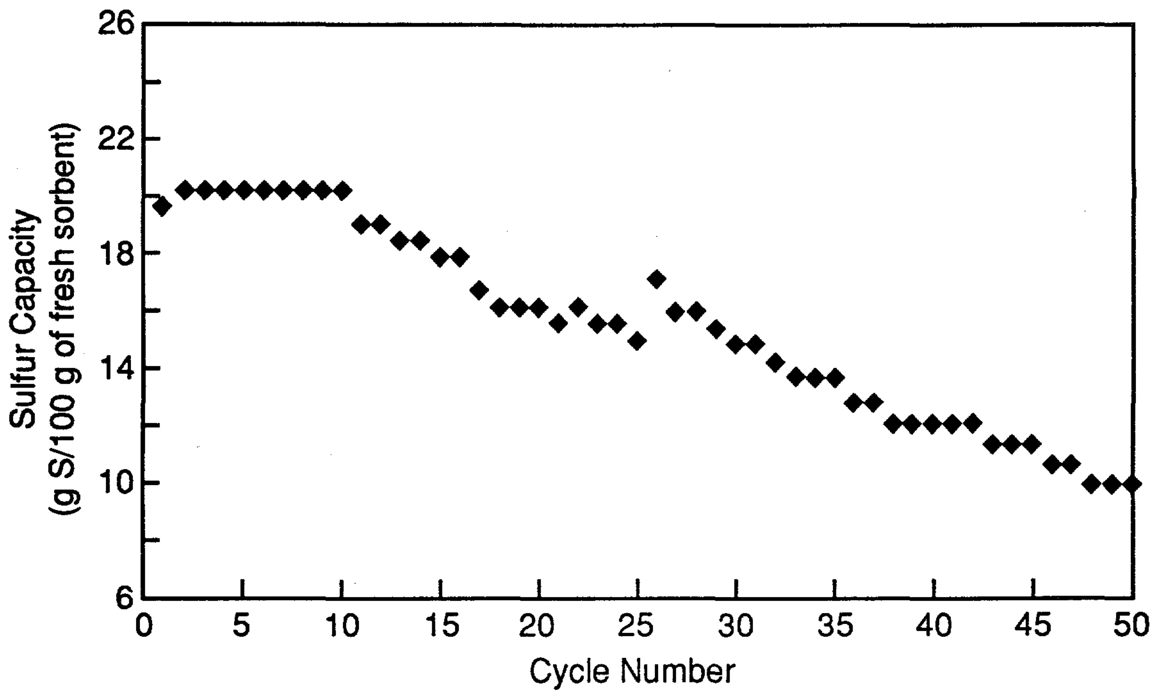

Figure 4. Sulfur capacity of Z-SORB III sorbent in U-Gas; 538 °C (1,000 °F); 20 atm; 500 ppm breakthrough.

Table 5. Sorbent Material Balance for Cycles 1 to 25

Initial	
Initial sorbent charge	500 g
Loss of volatiles during heating	35 g
Net fresh sorbent	465 g
After 25 Cycles	
Collected from cage	491.2 g
Collected from filters, thermocouples, and lines	9.3 g
Total accounted	500.5 g
Subtract amount of S on the sorbent (12.15% S on the sorbent)	29.84 g
Estimated sorbent collection (in unsulfided state)	461.36 g
Estimated net sorbent loss	3.64 g (0.78%)

Based on the values reported in Table 5, nearly all the sorbent was accounted for. The net sorbent loss was estimated to be about 0.78 percent.

After characterization, 400 g of the sulfided sorbent were loaded in the reactor and Cycles 26 to 51 were carried out. At the end of Sulfidation 51, the sorbent was removed and a material balance was performed. Table 6 shows the details of the material balance for Cycles 26 to 51.

As can be seen from Table 6, during Cycles 26 to 51, the total sorbent loss from the reactor was estimated to be 1.78%. The net total sorbent loss during Cycles 26 to 51 was estimated to be 0.64% based on the weights of the sorbent removed from the reactor and the material deposited on filters and lines. It is to be noted here that these sorbent loss estimations are based on sulfur analysis of the sulfided sorbent by combustion methods, which could be subject to errors.

Table 6. Sorbent Material Balance for Cycles 26 to 51

Initial	
Sorbent loaded after 25th sulfidation	400 g
(minus wt. gain due to sulfur)	24.3 g
Estimated unsulfided sorbent weight	375.7 g
After 51st Sulfidation	
Collected from cage	384.4 g
Subtract wt. gain due to presence of sulfur (8%)	15.4 g
Collected from filters, lines, and thermocouples	4.3 g
Estimated sorbent collection (in the unsulfided state)	369.0 g
Estimated total sorbent loss from the reactor	6.7 g (1.78%)
Estimated net sorbent loss	2.4 g (0.64%)

3.4 PHYSICAL PROPERTIES OF THE REACTED SORBENT

As per our agreement with PPCo, we measured the particle size distribution, bulk density, and attrition resistance of the fresh and the reacted sorbents. Table 7 shows the characterization results.

The compacted bulk density (CBD) of the sorbent was simply measured by tightly packing 50 cc of the sorbent in a graduated cylinder by periodic tapping. The weight of the 50-cc volume was then determined and the value for CBD was estimated by dividing the weight by volume and then converting into English units.

The sorbent attrition resistance was measured using a three-hole attrition tester described in detail elsewhere (Gupta and Gangwal, 1992). The attrition data reported in Table 7 and shown graphically in Figure 5 indicate that the attrition resistance of the reacted sorbent is lower than the fresh material. It is realized that attrition data from an actual reactor environment are more

Table 7. Physical Properties of Fresh and Reacted Z-SORB III Sorbent

	Fresh (wt%)	After 25th sulfidation (wt%)	After 51st sulfidation (wt%)	After 51st regeneration (wt%)
Particle Size Distribution				
MESH				
+ 50	0.00	0.00	0.00	0.8
- 50 + 60	10.44	6.98	5.00	8.8
-60 + 80	57.00	58.46	59.67	73.6
- 80 + 100	10.75	11.63	13.00	9.6
- 100 + 120	7.28	6.98	8.67	3.6
- 120 + 140	5.68	6.31	6.33	0.8
- 140 + Pan	8.85	9.64	7.33	2.8
Total	100.00	100.00	100.00	100.0
Average particle size (μm)	174.3	170.8	173.7	198.8
Compacted bulk density (lb/ft^3)	66.56	72.12	72.58	68.33
Attrition Resistance				
5-h loss (%)	16.0	20.0	23.2	19.6
20-h loss (%)	32.4	47.2	48.0	45.4

meaningful compared to simulation using the above 3-hole attrition test.

3.5 TGA CHEMICAL REACTIVITY OF FRESH AND REACTED SORBENT

The TGA reactivities of the fresh and the reacted (after 25th and 51st sulfidation) sorbents were measured using our standard 1.5 cycle TGA test described in Gupta and Gangwal (1992). Figure 6 shows the TGA reactivity profiles for the fresh as well as reacted sorbents. In this figure, the fresh sorbent was run twice to demonstrate the reproducibility of our TGA method. As expected, the ultimate sorbent sulfur capacity exhibited decline with cycling. The final weight gains closely corroborate the sulfur capacities obtained in the bench runs.

It is to be noted here that the initial rate of weight gain remains almost unchanged for the reacted sorbents, indicating good chemical reactivity albeit less capture capacity.

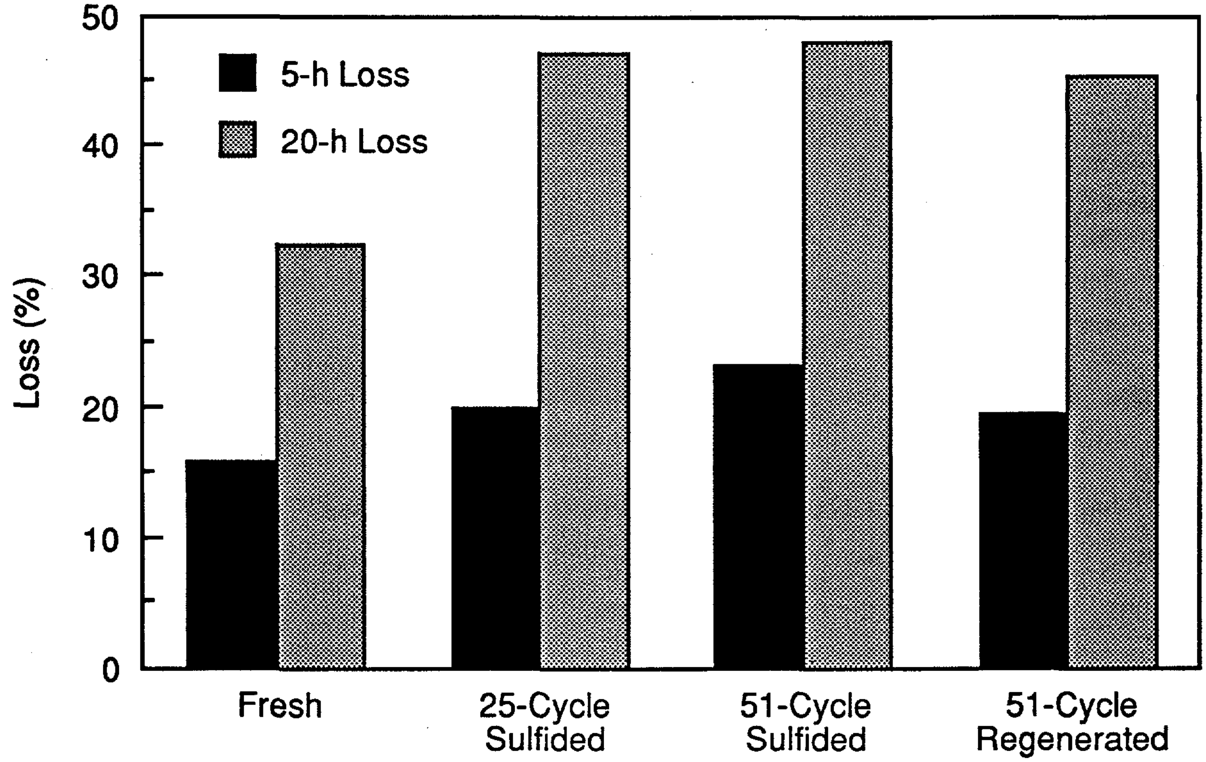

Figure 5. Attrition resistance of fresh and reacted Z-SORB III sorbent.

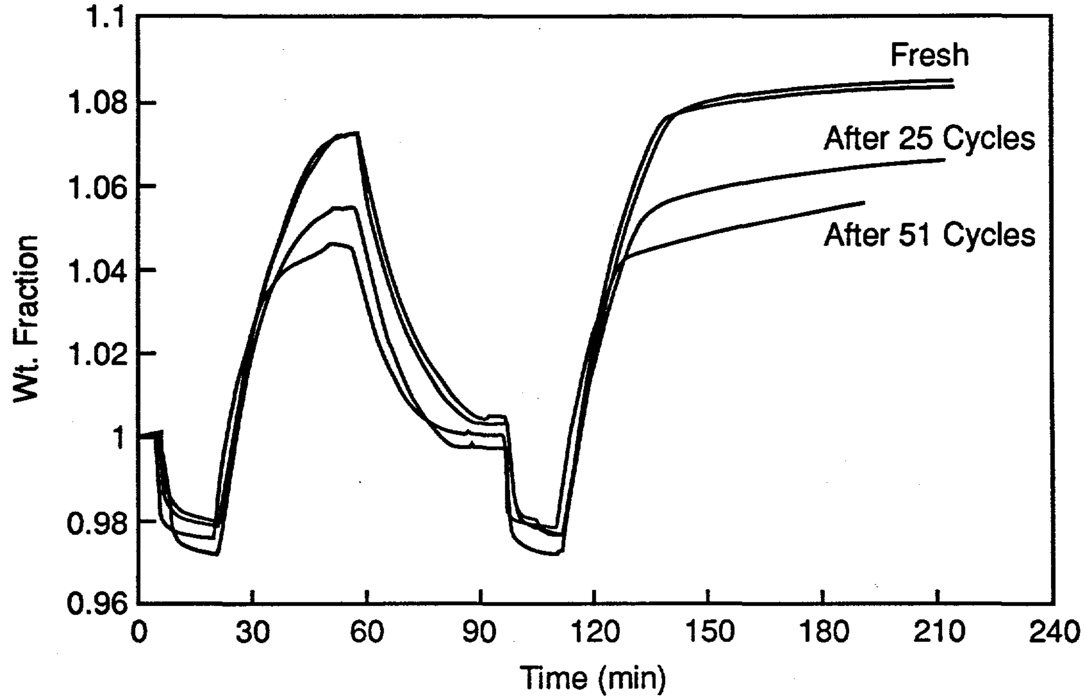

Figure 6. TGA reactivity of fresh and reacted sorbent.

4.0 SUMMARY

The following points summarize the results of this life-cycle test:

- The Z-SORB III sorbent consistently reduced the H_2S content of coal gas from 5,000 ppmv to <10 ppmv (less than the detectable limit of the analyzer) in a semibatch bench-scale fluidized bed at 538 °C (1,000 °F) and 20 atm.
- The sorbent was found to be fully regenerable, with negligible residual sulfate remaining. An oxygen concentration of 2 percent in N_2 was found to result in adequate regeneration rates.
- The sulfur capture capacity of the sorbent at breakthrough of 500 ppmv of H_2S ranged from 20.2 g S/100 g sorbent in Cycle 1 to 10 g S/100 g sorbent in Cycle 50 with an average of about 15 percent over the 50 cycles.
- The attrition resistance of the reacted sorbent as measured in 3-hole air-jet attrition tester was somewhat lower than the fresh sorbent.
- A total of 2.5 percent sorbent loss was detected over 50 cycles.
- The average particle size remained nearly constant while the bulk density increased by 8 percent over 50 cycles.
- The TGA chemical tests on the reacted sorbents confirmed the loss of sulfur capacity. However, from the TGA data, no change in H_2S absorption rate was noticeable in the reacted sorbent compared to the fresh sorbent.

5.0 RECOMMENDATIONS FOR FUTURE WORK

Delineation of the capacity declines observed following Cycle 10 suggests additional research in this area. Definition of required experiments is currently under assessment.

6.0 REFERENCES

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APPENDIX A

H_2S, COS, and SO_2 Breakthrough Data for Cycles 1 through 50

Table A-1. Breakthrough Data for Cycles 1 through 5

TIME	CYCLE #1			CYCLE #2			CYCLE #3			CYCLE #4			CYCLE #5		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0	0	0	0	0	141.9	0	0	276.9	0	0	281.6	0	0	0
5	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
15	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
20	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
25	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
30	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
35	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
40	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
45	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
50	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
55	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
60	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
65	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
70	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
75	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
80	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
85	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
90	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
95	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

(continued)

Table A-1. (continued)

TIME	CYCLE #1			CYCLE #2			CYCLE #3			CYCLE #4			CYCLE #5		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
100	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
105	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
110	6.4	0	0	0	0	0	0	0	0	0	0	0	0	0	0
115	10.7	0	0	0	0	0	0	0	0	0	0	0	6.3	0	0
120	13.6	0	0	0	0	0	0	0	0	6.3	0	0	8	0	0
125	17.2	0	0	7.7	0	0	7.2	0	0	7.1	0	0	8	0	0
130	20.7	6.2	0	9.8	0	0	8.4	0	0	8.9	0	0	11.3	0	0
135	22.9	5.8	0	16.8	0	0	12.9	0	0	13.2	0	0	15.8	0	0
140	26.2	5.5	0	25	0	0	22.9	0	0	19.7	0	0	26.5	0	0
145	31.3	6.1	0	33.1	0	0	31.9	0	0	31.1	0	0	37.4	0	0
150	39.8	6.1	0	38.9	0	0	38.8	0	0	40.1	0	0	45.5	0	0
155	68.9	9.3	0	44.9	0	0	44.1	0	0	46.1	0	0	52.8	0	0
160	159	17.9	0	52.3	6.4	0	50.7	0	0	53.3	0	0	60.3	0	0
165	359.4	35.4	0	100.7	10.5	0	65.9	6.4	0	63.4	0	0	87.2	8	0
170	686.2	65.5	0	329	34.5	0	241	16.8	0	122.2	0	0	375.3	17.8	0
175				1124.7	166.7	0	1117.1	108.8	0	712.7	29.5	0	1164.3	107.8	0

Table A-2. Breakthrough Data for Cycles 6 through 10

TIME	CYCLE #6			CYCLE #7			CYCLE #8			CYCLE #9			CYCLE #10		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0	0	346	0	0	116	0	0	41.7	0	0	144.3	0	0	277.6
5	0	0	0	0	0	0	0	0	0	0	0	0	16.5	16.5	46.5
10	0	0	0	0	0	0	0	0	0	0	0	0	14.7	12.3	0
15	0	0	0	0	0	0	0	0	0	0	0	0	11	0	0
20	0	0	0	0	0	0	0	0	0	0	0	0	8.5	0	0
25	0	0	0	0	0	0	0	0	0	0	0	0	7.1	0	0
30	0	0	0	0	0	0	0	0	0	0	0	0	5.9	0	0
35	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
40	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
45	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
50	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
55	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
60	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
65	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
70	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
75	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
80	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
85	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
90	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
95	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

(continued)

Table A-2. (continued)

TIME	CYCLE #6			CYCLE #7			CYCLE #8			CYCLE #9			CYCLE #10		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
100	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
105	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
110	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
115	6	0	0	5.6	0	0	5.5	0	0	5.5	0	0	6.1	0	0
120	6.9	0	0	5.5	0	0	9	0	0	9	0	0	7.5	0	0
125	8.3	0	0	8.2	0	0	9.2	0	0	9.2	0	0	9.1	0	0
130	9.9	0	0	10.3	0	0	12.3	0	0	12.3	0	0	11.7	0	0
135	14.1	0	0	14.2	0	0	19.7	0	0	19.7	0	0	18.2	0	0
140	23.1	0	0	24.9	0	0	32.9	0	0	32.5	0	0	29.2	0	0
145	33.4	0	0	35.7	0	0	42.1	0	0	42.1	0	0	39.9	0	0
150	42.7	0	0	43.9	0	0	49.8	0	0	49.8	0	0	45.9	0	0
155	49	0	0	50.5	0	0	57.1	0	0	57.1	0	0	52.5	0	0
160	55	0	0	57.1	0	0	65.6	0	0	65.6	0	0	59.9	0	0
165	67.8	0	0	71.6	0	0	123.2	8.1	0	123.2	8.1	0	124.1	8.6	0
170	195.6	0	0	270.5	0	0	382.4	29.5	0	382.4	29.5	0	370.1	29.2	0
175	1038.2	61.6	0	1130.3	75.6	0	639.7	139.2	0	639.7	139.2	0	1243.2	123	0

Table A-3. Breakthrough Data for Cycles 11 through 15

TIME	CYCLE #11			CYCLE #12			CYCLE #13			CYCLE #14			CYCLE #15		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0	0	78.5	0	0	1256	0	0	849.5	0	0	232.4	0	0	215.9
5	12	0	0	0	0	0	11.7	8.4	14.1	6.8	0	0	9.3	0	0
10	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
15	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
20	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
25	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
30	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
35	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
40	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
45	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
50	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
55	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
60	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
65	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
70	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
75	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
80	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
85	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
90	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
95	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

(continued)

Table A-3. (continued)

TIME	CYCLE #11			CYCLE #12			CYCLE #13			CYCLE #14			CYCLE #15		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
100	7.4	0	0	0	0	0	5.7	0	0	0	0	0	6.7	0	0
105	0.0	0	0	0	0	0	6.4	0	6.5	6.2	0	0	8.2	0	0
110	6.0	0	0	6.6	0	0	8	0	0	7.7	0	0	10	0	0
115	7.2	0	0	7.9	0	0	9.4	0	0	9.6	0	0	13.9	0	0
120	10.1	0	0	9	0	0	12.3	0	0	13	0	0	23.8	0	0
125	14.3	0	0	15.7	0	0	20.1	0	0	22.6	0	0	41.3	0	0
130	23.4	0	0	27.2	0	0	34.6	0	0	37	0	0	49.7	0	0
135	35.6	0	0	39.4	0	0	46.4	0	0	46.1	0	0	56	0	0
140	44.5	0	0	47.8	0	0	54.2	0	0	52.5	0	0	62.3	0	0
145	52.2	0	0	54.6	0	0	60.3	0	0	57.8	0	0	76.9	0	0
150	58.9	0	0	61.3	0	0	69.1	0	0	70.8	0	0	210.9	0	0
155	75.6	5.3	0	88.1	7	0	126	0	0	225.1	0	0	852.5	35.4	0
160	284.9	15.7	0	355.9	15.8	0	580.9	23.2	0	901.6	41.1	0			
165	1132.0	75.8	0	1119.7	79	0									

Table A-4. Breakthrough Data for Cycles 16 through 20

TIME	CYCLE #16			CYCLE #17			CYCLE #18			CYCLE #19			CYCLE #20		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0	0	106.9	0	0	0	0	0	263.4	0	0	30.2	0	0	0
5	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0	0	0	0	0	6.7	0	0
15	0	0	0	0	0	0	7.8	0	0	0	0	0	7	0	0
20	0	0	0	0	0	0	5.9	0	0	0	0	0	7.1	0	0
25	0	0	0	0	0	0	0	0	0	6.5	0	0	6.2	0	0
30	0	0	0	0	0	0	6.5	0	0	7.1	0	0	0	0	0
35	0	0	0	0	0	0	6.4	0	0	5.9	0	0	6.2	0	0
40	0	0	0	0	0	0	7	0	0	6.7	0	0	5.5	0	0
45	0	0	0	0	0	0	5.5	0	0	6.3	0	0	0	0	0
50	0	0	0	0	0	0	6.7	0	0	6.7	0	0	6.1	0	0
55	0	0	0	0	0	0	6	0	0	6.5	0	0	6.6	0	0
60	0	0	0	5.3	0	0	0	0	0	6.8	0	0	7.6	0	0
65	0	0	0	0	0	0	6	0	0	6.1	0	0	0	0	0
70	0	0	0	0	0	0	7.4	0	0	7.8	0	0	7.3	0	0
75	0	0	0	5.8	0	0	6.6	0	0	6.3	0	0	6.2	0	0
80	0	0	0	6.6	0	0	8.2	0	0	7.4	0	0	7.3	0	0
85	0	0	0	6.1	0	0	8.1	0	0	9	0	0	8.1	0	0
90	0	0	0	7.3	0	0	9.2	0	0	9.7	0	0	8.6	0	0
95	0	0	0	8.7	0	0	9.2	0	0	11.9	0	0	11	0	0

(continued)

Table A-4. (continued)

TIME	CYCLE #16			CYCLE #17			CYCLE #18			CYCLE #19			CYCLE #20		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
100	0	0	0	8.6	0	0	13.1	0	0	13.2	0	0	14.9	0	0
105	9.4	0	0	13.6	0	0	18.1	0	0	22.9	0	0	24.2	0	0
110	11.4	0	0	25.1	0	0	36.9	0	0	50.7	0	0	53.9	0	0
115	18.8	0	0		0	0	64.9	0	0	67	0	0	69.8	0	0
120	35.5	0	0	61	0	0	83.1	0	0	76.8	0	0	79	0	0
125	47.3	0	0	72	0	0	91.4	6.2	0	84.3	0	0	95.3	0	0
130	54.5	0	0	79.6	0	0	101.3	6.5	0	108.3	0	0	135.2	0	0
135	58.8	0	0	91.8	0	0	149	0	0	404.1	0	0	472.8	16.5	0
140	67.8	0	0	24.3	0	0	556.2	18.3	0	1056.3	51.8	0	1177.2	66.2	0
145	118.1	0	0	913.2	33.6	0									
150	157.2	20.3	0												
155	1193.6	81.7	0												

Table A-5. Breakthrough Data for Cycles 21 through 25

TIME	CYCLE #21			CYCLE #22			CYCLE #23			CYCLE #24			CYCLE #25		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0	0	46.8	0	0	606.5	0	0	144.7	0	0	180.5	0	0	506.5
5	7.9	0	0	0	0	0	0	0	0	0	0	0	0	0	0
10	6.7	0	0	0	0	0	0	0	0	0	0	0	0	0	0
15	6.7	0	0	0	0	0	0	0	0	0	0	0	0	0	0
20	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
25	5.9	0	0	0	0	0	0	0	0	0	0	0	0	0	0
30	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
35	7.5	0	0	6	0	0	0	0	0	0	0	0	0	0	0
40	6.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
45	7.4	0	0	0	0	0	0	0	0	0	0	0	0	0	0
50	6.8	0	0	0	0	0	0	0	0	0	0	0	6.9	0	0
55	6.7	0	0	0	0	0	0	0	0	0	0	0	0	0	0
60	6.1	0	0	0	0	0	0	0	0	6.4	0	0	0	0	0
65	7.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
70	6.7	0	0	5.6	0	0	0	0	0	0	0	0	7	0	0
75	7.5	0	0	6.6	0	0	0	0	0	6.7	0	0	6.9	0	0
80	7.5	0	0	5.9	0	0	0	0	0	6.6	0	0	8.4	0	0
85	9.5	0	0	7	0	0	6.8	0	0	8.7	0	0	8.8	0	0
90	9.6	0	0	8.4	0	0	7.8	0	0	9.7	0	0	11.2	0	0
95	12.7	0	0	9.3	0	0	9.9	0	0	13.5	0	0	17.8	0	0

(continued)

Table A-5. (continued)

TIME	CYCLE #21			CYCLE #22			CYCLE #23			CYCLE #24			CYCLE #25		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
100	20	0	0	13.9	0	0	13.7	0	0	24.5	0	0	34.5	0	0
105	46.6	0	0	22.4	0	0	27.6	0	0	40.6	0	0	44.2	0	0
110	64.2	0	0	37.5	0	0	45.2	0	0	48.3	0	0	50.1	0	0
115	74.8	0	0	46.2	0	0	53.3	0	0	53.8	0	0	56.5	0	0
120	82.6	0	0	52.7	0	0	59.1	0	0	60.5	0	0	67.6	0	0
125	106.6	0	0	59.3	0	0	72.8	0	0	76.8	5.5	0	210.9	11.1	0
130	286.2	0	0	76.6	0	0	121.8	0	0	210.3	0	0	915.7	36.2	0
135	949.2	36	0	197.3	0	0	474.1	19.2	0	905.7	38.4	0			

Table A-6. Breakthrough Data for Cycles 26 through 30

TIME	CYCLE #26			CYCLE #27			CYCLE #28			CYCLE #29			CYCLE #30		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0	1.696	283.751	0	0	778.451	0	0	747.116	0	0	452.86	0	0	230.876
5	3.064	0	2.897	3.767	0	10.015	5.048	0.734	0	5.147	0.874	0	6.806	0.978	0
10	1.38	0	0	2.681	0	0	4.062	0	0	4.722	0	0			
15	0.926	0	0	2.089	0	0	3.407	0	0	4.462	0	0			
20	0.827	0	0	1.941	0	0	3.137	0	0	4.532	0	0	5.765	0	0
25	0.737	0	0	1.823	0	0	2.602	0	0	4.56	0	0	6.167	0	0
30	0.916	0	0	1.577	0	0	2.57	0	0	4.502	0	0	6.066	0	0
35	0.838	0	0	1.38	0	0	1.861	0	0	4.773	0	0	6.278	0	0
40	0.876	0	0	1.503	0	0	1.786	0	0	4.553	0	0	6.547	0	0
45	0.913	0	0	1.491	0	0	1.87	0	0	4.4	0	0	6.653	0	0
50	1.062	0	0	1.735	0	0	1.8	0	0	4.408	0	0	6.675	0	0
55	1.175	0	0	2.016	0	0	1.99	0	0	4.557	0	0	7.022	0	0
60	1.376	0	0	2.069	0	0	1.974	0	0	4.231	0	0	7.054	0	0
65	1.553	0	0	2.404	0	0	2.181	0	0	4.016	0	0	7.295	0	0
70	1.764	0	0	2.803	0	0	2.624	0	0	4.264	0	0	8.258	0	0
75	2.003	0	0	3.404	0	0	3.249	0	0	4.715	0	0	9.151	0	0
80	2.31	0	0	4.309	0	0	3.986	0	0	5.17	0	0	10.44	0	0
85	2.858	0	0	6.67	0	0	4.719	0	0	6.358	0	0	12.847	0	0
90	3.508	0	0	8.362	0	0	6.6	0	0	8.224	0	0	18.299	0	0
95	4.298	0	0	13.738	0	0	12.435	0	0	11.963	0	0	34.142	0.801	0

(continued)

Table A-6. (continued)

TIME	CYCLE #26			CYCLE #27			CYCLE #28			CYCLE #29			CYCLE #30		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
100	5.511	0	0	25.639	0	0	25.806	0	0	20.522	0	0	70.394	2.11	0
105	8.898	0	0	52.727	1.15	0	48.268	1.021	0	49.771	1.3	0	84.47	2.742	0
110	13.23	0	0	72.075	1.91	0	62.616	1.604	0	76.168	2.327	0	93.064	3.354	0
115	23.899	0	0	80.101	2.077	0	72.327	1.787	0	86.384	2.998	0	103.922	3.765	0
120	54.31	1.358	0	89.002	2.457	0	76.594	1.932	0	94.378	3.421	0	123.191	4.788	0
125	81.729	2.579	0	119.728	4.02	0	83.585	2.135	0	109.443	4.151	0	278.491	16.782	0
130	92.222	2.782	0	162.085	6.35	0	109.121	3.241	0	167.701	7.896	0	673.909	64.353	0
135	100.017	3.228	0	222.859	9.584	0	319.926	17.033	0	432.346	33.305	0			
140	117.631	3.848	0	323.188	17.438	0	972.286	79.685	0	998.136	117.067	0			
145	231.518	10.552	0												
150	589.374	41.773	0												

Table A-7. Breakthrough Cycles 31 through 35

TIME	CYCLE #31			CYCLE #32			CYCLE #33			CYCLE #34			CYCLE #35		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0	0	436.355				0	0	1623.312						
5	8.974	1.622	2.847				9.727	3.522	12.13	13.405	2.517	0	15.059	1.863	4.826
10	6.564	0	0	9.278	0	0	8.913	0.917	0	7.621	0	0	10.774	0.671	0
15	5.534	0	0	7.418	0	0	7.425	0	0	6.425	0	0	8.492	0	0
20	5.29	0	0	7.247	0	0	7.446	0	0	6.125	0	0	7.506	0	0
25	5.554	0	0	7.073	0	0	7.266	0	0	5.77	0	0	7.191	0	0
30	5.466	0	0	7.361	0	0	7.282	0	0	5.996	0	0	6.84	0	0
35	5.592	0	0	7.385	0	0	7.543	0	0	6.048	0	0	6.666	0	0
40	5.772	0	0	7.467	0	0	7.611	0	0	6.281	0	0	6.759	0	0
45	6.028	0	0	7.698	0	0	7.807	0	0	6.641	0	0	6.799	0	0
50	6.132	0	0	7.923	0	0	8.09	0	0	6.943	0	0	7.256	0	0
55	6.519	0	0	8.279	0	0	8.564	0	0	7.309	0	0	7.495	0	0
60	6.866	0	0	8.908	0	0	9.213	0	0	7.654	0	0	8.295	0	0
65	7.275	0	0	9.208	0	0	9.805	0	0	8.133	0	0	8.924	0	0
70	7.553	0	0	9.674	0	0	10.686	0	0	8.983	0	0	9.688	0	0
75	8.644	0	0	10.81	0	0	12.185	0	0	10.244	0	0	11.63	0	0
80	9.633	0	0	12.289	0	0	15.058	0	0	11.975	0	0	15.155	0	0
85	11.711	0	0	14.508	0	0	21.105	0	0	15.242	0	0	24.446	0	0
90	16.748	0	0	19.639	0	0	38.79	1.021	0	25.429	0	0	58.063	1.369	0
95	25.792	0	0	35.937	0.917	0	79.48	2.482	0	61.778	1.626	0	81.354	2.308	0

(continued)

Table A-7. (continued)

TIME	CYCLE #31			CYCLE #32			CYCLE #33			CYCLE #34			CYCLE #35		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
100	59.274	1.762	0	77.542	2.589	0	93.489	3.168	0	80.334	2.478	0	91.702	2.772	0
105	79.737	2.611	0	93.412	3.172	0	103.214	3.58	0	90.524	2.856	0	105.371	3.585	0
110	89.965	3.009	0	105.213	3.697	0	115.405	4.28	0	99.479	3.414	0	126.19	4.601	0
115	99.078	3.326	0	120.419	4.639	0	143.804	5.964	0	150.107	6.211	0	345.255	21.094	0
120	125.698	4.931	0	184.253	8.742	0	415.934	29.861	0	474.313	35.687	0	1018.639	109.66	0
125	305.213	19.291	0	687.865	65.838	0	961.308	107.638	0	1065.039	121.396	0			
130	659.745	61.496	0												
135	1121.067	148.423	0												

Table A-8. Breakthrough Data for Cycles 36 through 40

TIME	CYCLE #36			CYCLE #37			CYCLE #38			CYCLE #39			CYCLE #40		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0				0	0.94	1085.727	0	18.948	559.078	0	1.162	320.501	0	0	1347.083
5	15.878	2.212	0	11.604	1.184	0	13.639	1.956	0	21.448	4.67	0	17.779	3.864	0
10	8.76	0	0	7.36	0	0	8.917	0	0	9.562	0	0	9.538	0	0
15	7.516	0	0	6.853	0	0	7.612	0	0	8.164	0	0	7.896	0	0
20	7.488	0	0	6.869	0	0	7.321	0	0	7.915	0	0	7.576	0	0
25	7.838	0	0	7.013	0	0	7.966	0	0	7.898	0	0	7.691	0	0
30	8.066	0	0	7.374	0	0	7.675	0	0	8.369	0	0	7.979	0	0
35	8.778	0	0	8.009	0	0	8.038	0	0	8.665	0	0	8.512	0	0
40	9.589	0	0	8.545	0	0	9.003	0	0	9.204	0	0	9.127	0	0
45	10.809	0	0	9.548	0	0	10.382	0	0	10.622	0	0	10.382	0	0
50	12.89	0	0	11.212	0	0	13.108	0	0	13.467	0	0	13.867	0	0
55	17.879	0	0	14.658	0	0	19.576	0	0	21.22	0.782	0	22.74	0.778	0
60	31.409	0.99	0	25.243	0.724	0	40.315	1.205	0	43.447	1.375	0	48.971	1.665	0
65	64.533	2.403	0	55.129	1.778	0	75.149	3.073	0	74.095	2.792	0	75.332	2.891	0
70	83.878	3.45	0	77.922	2.839	0	87.156	3.402	0	84.167	3.561	0	84.596	3.481	0
75	92.489	4.04	0	86.74	3.375	0	97.819	4.001	0	91.497	3.984	0	94.267	4.046	0
80	101.202	4.642	0	97.793	4.116	0	118.684	5.341	0	119.578	5.649	0	118.59	5.712	0
85	183.325	10.783	0	200.811	11.699	0	446.951	37.603	0	520.949	49.58	0	587.294	58.841	0
90	658.084	72.699	0	739.113	81.335	0	1115.034	171.316	0						

Table A-9. Breakthrough Data for Cycles 41 through 45

TIME	CYCLE #41			CYCLE #42			CYCLE #43			CYCLE #44			CYCLE #45		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0.634	34.505	486.64	0	0	1673.616	0	58.109	951.552	1.6	113.845	1439.935			
5	18.805	2.533	0	24.198	2.83	0	18.511	3.878	0	18.774	2.067	0	19.832	3.298	0
10	9.446	0	0	12.927	0.679	0	9.967	0	0	8.875	0	0	9.717	0	0
15	8.44	0	0	10.149	0	0	9.235	0	0	7.322	0	0	8.631	0	0
20	8.072	0	0	9.05	0	0	8.571	0	0	6.912	0	0	8.877	0	0
25	8.191	0	0	8.562	0	0	8.63	0	0	6.903	0	0	8.948	0	0
30	8.509	0	0	8.573	0	0	9.057	0	0	7.386	0	0	9.428	0	0
35	8.866	0	0	8.954	0	0	9.541	0	0	8.522	0	0	10.911	0	0
40	9.845	0	0	9.815	0	0	10.689	0	0	11.172	0	0	13.746	0	0
45	11.51	0	0	11.941	0	0	12.735	0	0	17.664	0	0	20.526	0.756	0
50	15.082	0	0	16.908	0	0	18.387	0	0	32.711	1.071	0	45.071	1.614	0
55	24.588	0.785	0	30.932	0.939	0	38.734	1.276	0	53.887	1.993	0	75.068	3.146	0
60	53.956	1.941	0	65.305	2.334	0	75.358	3.239	0	63.35	2.456	0	84.545	3.783	0
65	81.219	3.203	0	80.426	3.177	0	86.28	3.897	0	68.5	2.688	0	91.264	4.17	0
70	91.683	3.75	0	88.092	3.562	0	95.159	4.355	0	77.965	3.16	0	105.848	5.213	0
75	100.652	4.357	0	97.737	4.098	0	110.017	5.314	0	108.281	5.015	0	348.942	29.687	0
80	140.664	7.094	0	193.941	11.512	0	359.874	29.828	0	579.52	60.517	0	1012.535	158.858	0
85	658.455	69.655	0	919.204	117.362	0	1048.905	166.255	0						

Table A-10. Breakthrough Data for Cycles 46 through 50

TIME	CYCLE #46			CYCLE #47			CYCLE #48			CYCLE #49			CYCLE #50		
	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2	H_2S	COS	SO_2
0	0	0	714.741	0	3.961	229.968	0	0	1357.188	0	0	21.301			
5	25.028	3.34	0	24.579	5.714	0	32.979	2.524	3.51	13.083	1.482	0			
10	12.753	0	0	10.9	0	0	17.314	0.746	0	9.305	0	0	10.525	0	0
15	10.506	0	0	9.229	0	0	13.111	0	0	8.962	0	0	9.567	0	0
20	9.99	0	0	9.021	0	0	11.94	0	0	9.353	0	0	9.613	0	0
25	10.467	0	0	9.333	0	0	12.156	0	0	10.712	0	0	10.913	0	0
30	11.33	0	0	10.472	0	0	13.622	0	0	11.874	0	0	13.575	0	0
35	13.494	0	0	13.225	0	0	18.165	0	0	16.095	0	0	20.753	0	0
40	18.745	0	0	19.989	0	0	31.348	0.832	0	29.76	0.919	0	41.369	1.395	0
45	34.243	1.157	0	37.266	1.355	0	60.957	2.123	0	62.035	2.179	0	67.861	2.462	0
50	69.156	2.818	0	63.18	2.586	0	79.83	2.99	0	79.079	3.009	0	75.816	2.861	0
55	83.501	3.569	0	74.901	3.275	0	88.549	3.335	0	89.501	3.537	0	85.727	2.354	0
60	91.455	0	0	82.592	3.706	0	96.052	3.757	0	96.362	3.925	0	102.409	1.269	0
65	102.633	4.842	0	90.078	4.169	0	113.862	4.698	0	125.034	5.764	0	161.048	8.157	0
70	179.206	10.686	0	112.778	5.678	0	442.477	35.537	0	592.835	57.299	0	875.614	98.923	0
75	932.278	122.458	0	473.662	47.029	0	1153.187	161.503	0						
80				1044.29	172.282	0									