DOE/MC/21098--2247-Vol.2

DE87 001074

Summary and Assessment of METC Zinc Ferrite Hot Coal Gas Desulfurization Test Program, Volume II: Appendices

Final Report

V.S. Underkoffler

December 1986 (Revised June 1991)

Work Performed Under Contract No.: DE-AC21-84MC21098

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

By
Gilbert/Commonwealth
Reading, Pennsylvania

DOE/MC/21098-2247, Vol. 2
Distribution Category UC-106
(DE87001074)

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ABSTRACT

The Morgantown Energy Technology Center (METC) has conducted a test program to develop a zinc ferrite-based high temperature desulfurization process which could be applied to fuel gas entering downstream components such as molten carbonate fuel cells or gas turbines. As a result of prior METC work with iron oxide and zinc oxide sorbents, zinc ferrite evolved as a candidate with the potential for high capacity, low equilibrium levels of H_2S, and structural stability after multiple regenerations. The program consisted of laboratory-scale testing with a two-inch diameter reactor and simulated fixed-bed gasifier gas; bench-scale testing with a six-inch diameter reactor and actual gas from the METC 42-inch fixed bed gasifier; as well as laboratory-scale testing of zinc ferrite with simulated fluidized bed gasifier gas. Optimum operating parameters for zinc ferrite such as temperatures, gas compositions, and space velocities are discussed. From the test results, salient features of zinc ferrite were derived and discussed in regard to system implications, issues raised, and technical requirements.

An assessment of the technical and economic implications of the zinc ferrite process is presented as well as suggested programs for potential commercialization.

ACKNOWLEDGMENTS

This report was prepared under Contract No. DE-AC21-84MC21098, Department of Energy, Morgantown Energy Technology Center.

We acknowledge the interest and constructive suggestions of Dr. Vijendra P. Kothari and Dr. Thomas Grindley of Morgantown Energy Technology Center.

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APPENDIX A

Method for Preparing Zinc Ferrite Sorbent

Screened (-60 mesh, 250 micron) equimolar amounts of Fisher "Certified" ferric oxide and Sherwin-Williams "High Surface Area" zinc oxide were mixed dry with 1 percent of Fisher "Laboratory Grade" bentonite powder in a split sleeve mixer for 30 minutes. The dry mixture was placed in a small cement mixer and sprayed with water from an atomizer until the "plastic" state was reached. The wet mixture was extruded through a pipe with a die face plate using a ram rod pushed with a hydraulic jack. The resulting sorbents were dried at 200°F for several hours then baked at 1,500°F for 3 hours.

APPENDIX B

Process Modeling and Equilibrium Calculations

Process modeling consisted primarily of the calculation of equilibrium H_2S levels for various sorbents at high temperatures. This can be done either by minimization of the GIBBS free energy function for a group of reactants, or by direct calculation of the equilibrium constant for a specified reaction. The former method is more complex and allows for the interaction of all possible reactions among the specified components, whereas the latter is simpler and requires that a reaction be specified in terms of products and reactants. The ASPEN system, using the RGIBBS routine, was utilized for the more complex GIBBS free energy minimization calculations. A code (THERMOG) was written for the simpler equilibrium constant method of calculation.

A. ASPEN - RGIBBS

The RGIBBS routine computes the phase and/or chemical equilibrium compositions at a user specified temperature and pressure when any number of feed streams are mixed. The output consists of one vapor phase and any number of liquid and solid phases. The method of calculation is minimization of GIBBS free energy using an extension of the Rand algorithm (Gautram and Seider, Part I 1979). After the equilibrium compositions are computed, an overall energy balance is performed to compute the heat duty. Since the sulfidation reaction was the primary area of interest, and it is expected that this reaction is essentially isothermal, heat effects were not considered at this point. Heat effects will be an important parameter, however, when regeneration reactions are studied. Sulfidation reactions of zinc oxide with a Lurgi-type gas were simulated and the results compared with other sources. In addition, the effect of equilibrium at ambient pressure on major components was simulated. Of primary interest at this point was the level of H_2 and CO, since these make up the Molten Carbonate Fuel Cell (MCFC) useable fuel.

Equilibrium values of H_2S were obtained for the reaction of H_2S with ZnO. A Lurgi air-blown gasifier gas was used in the input file, and one atmosphere pressure specified. Runs were made for temperatures of 500 to 650°C in 50°C increments, and for a feed gas with 25 percent steam and no steam (H_2S at 2.7 percent).

Attempts were made to run ASPEN on the METC DEC/VAX-11 without success. Due to time limitations, the runs were not repeated and reliance was placed in calculations from equilibrium constants.

B. <u>THERMOG - A code that calculates equilibrium values of the components of the sulfidation reaction of any metal oxide</u>

As mentioned previously, the simpler method of calculating equilibrium values of a reaction of known components is by the equilibrium constant method. The equilibrium constant of any reaction can be calculated from the thermodynamic properties of the reactants and products of the reaction with proper accounting of the stoichiometry of the reaction. Once the equilibrium constant is calculated at a given temperature, equilibrium values of the components of the reaction can be readily calculated in terms of known values of the remaining components of the reaction.

A Fortran code written to carry out this calculation on the METC VAX-11 is currently operational. Input requirements include the thermodynamic properties of all components in the reaction, and the reaction itself has to be specified. The output is displayed in tabular form as in Table 1.

Initially, the code was used to determine the effects of various sources of thermodynamic data on the equilibrium constant for the zinc oxide reaction. Figure 1 shows the equilibrium constants obtained using various sources of data. It was generally found that small discrepancies in thermodynamic data affected the results: however, results remained within a reasonable range. The user can choose input

GENERAL GAS-SOLID EQUILIBRIUM CODE (ZnO)

THERMODYNAMIC DATA			DATA SOURCE
ZNS HEAT OF FORMATION (25 C) =	-48.500	KCAL MOL-1	CRC HANDBOOK
H2O HEAT OF FORMATION (25 C) =	-57.800	KCAL MOL-1	CRC HANDBOOK
ZNO HEAT OF FORMATION (25 C) =	-83.170	KCAL MOL-1	CRC HANDBOOK
H2S HEAT OF FORMATION (25 C) =	-4.815	KCAL MOL-1	CRC HANDBOOK
ZNS ENTROPY (25 C) =	13.800	CAL K-1 MOL-1	CRC HANDBOOK
H2O ENTROPY (25 C) =	45.110	CAL K-1 MOL-1	CRC HANDBOOK
ZNO ENTROPY (25 C) =	10.500	CAL K-1 MOL-1	CRC HANDBOOK
H2S ENTROPY (25 C) =	49.150	CAL K-1 MOL-1	CRC HANDBOOK

HEAT CAPACITY EQUATION IS CP = A + BT + CT2 + DT3 + ET-2 CAL K-1 MOL-1

HEAT CAPACITY COEFFICIENTS	A	B	C	D	E	SOURCE
ZNO	11.40	0.00145000	0.000000000	0.000000000	-182400.00	PERRY'S P.3-
H2S	7.81	0.00296000	0.000000000	0.000000000	-46000.00	MILLS
ZNS	12.81	0.00095000	0.000000000	0.000000000	-194600.00	PERRY'S P3-1
H2O	7.17	0.00256000	0.000000000	0.000000000	8000.00	KUBASCHEUSKI

TEMPERATURE DEGREES C	DELTA G CAL/GMOLE	EQUILIBRIUM CONSTANT	EQUILIBRIUM H2S LEVEL IN PPM PERCENT STEAM					
			5%	10%	15%	20%	25%	30%
200.	-17999.88	80.73E+07	24.1E-05	48.2E-05	72.3E-05	96.5E-05	12.1E-04	14.5E-04
250.	-17981.13	32.64E+06	15.3E-04	30.6E-04	46.0E-04	61.3E-04	76.6E-04	91.9E-04
300.	-17964.58	71.09E+05	70.3E-04	14.1E-03	21.1E-03	28.1E-03	35.2E-03	42.2E-03
350.	-17949.70	19.80E+05	25.3E-03	50.5E-03	75.8E-03	0.10	0.13	0.15
400.	-17938.09	66.75E+04	74.9E-03	0.15	0.22	0.30	0.37	0.45
450.	-17923.45	26.17E+04	0.19	0.38	0.57	0.76	0.96	1.1
500.	-17911.49	11.59E+04	0.43	0.86	1.3	1.7	2.2	2.6
550.	-17900.01	56.66E+03	0.88	1.8	2.6	3.5	4.4	5.3
600.	-17888.80	30.08E+03	1.7	3.3	5.0	6.6	8.3	10.
650.	-17877.71	17.10E+03	2.9	5.8	8.8	12.	15.	18.
700.	-17866.59	10.30E+03	4.9	9.7	15.	19.	24.	29.
750.	-17855.31	65.83E+02	7.7	15.	23.	31.	38.	46.
800.	-17843.76	43.09E+02	12.	23.	35.	46.	58.	70.
850.	-17831.84	29.53E+02	17.	34.	51.	68.	85.	10.2E+01

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TABLE 1

ZNO EQUILIBRIUM CONSTANT

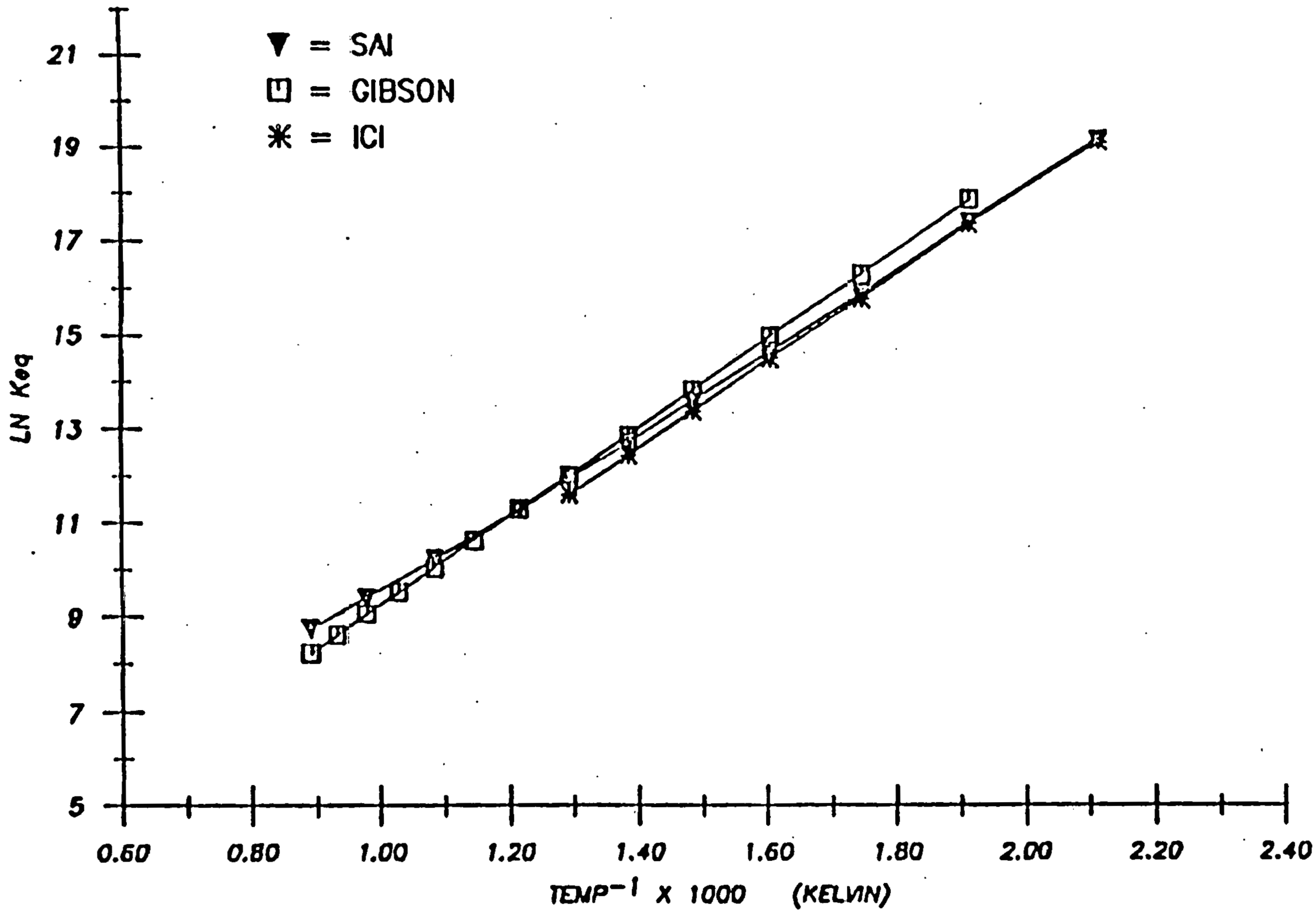

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FIGURE 1

data felt to be most accurate and obtain equilibrium values based on this chosen input data.

The effect of steam on the H_2S equilibrium level is shown in Figure 2. This plot indicates that sufficiently low levels of H_2S (below 1 ppm) can be obtained at up to approximately 7 percent steam at 1,000°F. As temperature increases, the equilibrium H_2S level increases. Additional calculations using two different sets of data were made to determine the effect of small variations in the thermodynamic properties of zinc oxide and zinc sulfide on H_2S equilibrium values. The results, shown in Tables 1 and 2 indicate differences in the H_2S levels obtained, with larger discrepancies at higher temperature and steam levels.

The equilibrium code for calculating equilibrium levels of H_2S with zinc oxide was generalized to allow equilibrium calculations for any gas-solid reaction. This was done primarily to determine equilibrium levels of H_2S with V_2O_3. Tables 3 and 4 show the results of equilibrium calculations using two different values for the heat of formation of V_2S_3. Using the National Bureau of Standards' heat of formation value for V_2S_3, of 230 Kcal/mol, equilibrium calculations indicate that H_2S levels far below 1 ppm can be achieved. However, if a correlation by Mills is used to obtain the heat of formation of V_2S_3 of 125 Kcal/mol, the results indicate that no desulfurization will take place. This discrepancy makes it difficult to draw conclusions as to the capabilities of V_2S_3 in low level desulfurization. This must be determined experimentally.

In conclusion, two methods have been used to calculate the equilibrium H_2S values expected at Hot Gas Desulfurization conditions. Results are reasonable for the most part and come reasonably close to experimental data obtained in this project and others. Additional calculations for simultaneous shift/methanation equilibrium calculations based on equilibrium constants were conducted for various temperatures. These are included in the third section of Appendix B.

H2S EQUILIBRIUM VALUES

ZINC OXIDE SYSTEM

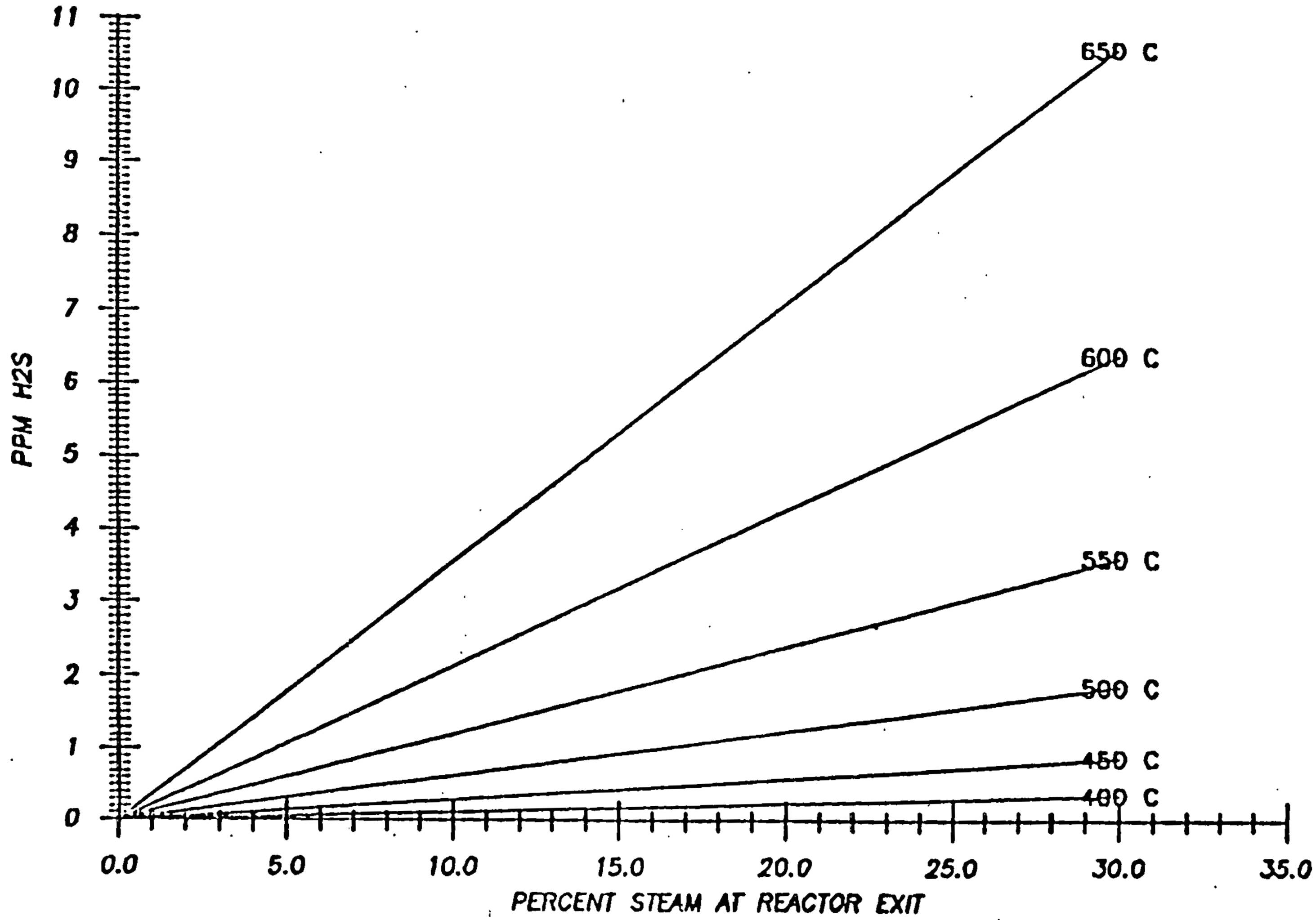

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FIGURE 2

GENERAL GAS-SOLID EQUILIBRIUM CODE (ZnO)

THERMODYNAMIC DATA DATA SOURCE

ZNS HEAT OF FORMATION (25 C) =	-49.000	KCAL MOL-1	MILLS
H2O HEAT OF FORMATION (25 C) =	-57.796	KCAL MOL-1	KUBASCHEWSKI
ZNO HEAT OF FORMATION (25 C) =	-83.820	KCAL MOL-1	KUBASCHEWSKI
H2S HEAT OF FORMATION (25 C) =	-4.930	KCAL MOL-1	MILLS

ZNS ENTROPY (25 C) =	13.800	CAL K-1 MOL-1	MILLS
H2O ENTROPY (25 C) =	45.100	CAL K-1 MOL-1	KUBASCHEWSKI
ZNO ENTROPY (25 C) =	10.430	CAL K-1 MOL-1	KUBASCHEWSKI
H2S ENTROPY (25 C) =	49.150	CAL K-1 MOL-1	MILLS

HEAT CAPACITY EQUATION IS CP = A + BT + CT2 + DT3 + ET-2 CAL K-1 MOL-1

HEAT CAPACITY COEFFICIENTS	A	B	C	D	E	SOURCE
ZNO	11.71	0.00122000	0.00000000	0.00000000	-218000.00	KUBASCHEWSKI
H2S	7.81	0.00296000	0.00000000	0.00000000	-46000.00	MILLS
ZNS	11.77	0.00126000	0.00000000	0.00000000	-116000.00	MILLS
H2O	7.17	0.00256000	0.00000000	0.00000000	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	29.8E-05	59.5E-05	89.3E-05	11.9E-04	14.9E-04	17.9E-04
250.	-17778.15	20.85E+06	18.6E-04	37.2E-04	55.9E-04	74.5E-04	93.1E-04	11.2E-03
300.	-17753.17	59.04E+05	84.7E-04	16.9E-03	25.4E-03	33.9E-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	89.4E-03	0.18	0.27	0.36	0.45	0.54
450.	-17669.21	21.93E+04	0.23	0.46	0.68	0.91	1.1	1.4
500.	-17637.63	96.96E+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.	-17491.03	84.80E+02	5.9	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	23.98E+02	21.	42.	63.	84.	10.5E+01	12.6E+01

FORTRAN STOP
$

TABLE 2

GENERAL GAS-SOLID EQUILIBRIUM CODE (V_2O_3)

THERMODYNAMIC DATA			DATA SOURCE
V2S3HEAT OF FORMATION (25 C) =	-239.000	KCAL MOL-1	NBS
H2O HEAT OF FORMATION (25 C) =	-57.795	KCAL MOL-1	KUBASCHEWSKI
V2O3HEAT OF FORMATION (25 C) =	-291.300	KCAL MOL-1	KUBASCHEWSKI-JANAF 1975
H2S HEAT OF FORMATION (25 C) =	-4.960	KCAL MOL-1	MILLS
V2S3ENTROPY (25 C) =	31.000	CAL K-1 MOL-1	MILLS
H2O ENTROPY (25 C) =	45.106	CAL K-1 MOL-1	KUBASCHEWSKI
V2O3ENTROPY (25 C) =	23.449	CAL K-1 MOL-1	KUBASCHEWSKI-JANAF 1975
H2S ENTROPY (25 C) =	49.160	CAL K-1 MOL-1	MILLS

HEAT CAPACITY EQUATION IS CP = A + BT + CT2 + DT3 + ET-2 CAL K-1 MOL-1

HEAT CAPACITY COEFFICIENTS	A	B	C	D	E	SOURCE
V2O3	29.35	0.00476000	0.00000000	0.00000000	-542000.00	B. OF MINES
H2S	7.81	0.00296000	0.00000000	0.00000000	-46000.00	MILLS
V2S3	28.00	0.01000000	0.00000000	0.00000000	0.00	MILLS(AVG)
H2O	7.17	0.00256000	0.00000000	0.00000000	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.	-95398.06	11.97E+43	10.1E-11	20.3E-11	30.4E-11	40.6E-11	50.7E-11	60.9E-11
250.	-95252.88	83.58E+38	27.0E-10	54.0E-10	81.0E-10	10.8E-09	13.6E-09	18.2E-09
300.	-95114.52	18.94E+35	40.4E-09	80.8E-09	12.1E-08	16.2E-08	20.2E-08	24.2E-08
350.	-94981.42	20.85E+32	39.1E-08	78.3E-08	11.7E-07	15.7E-07	19.6E-07	23.5E-07
400.	-94852.61	83.37E+29	27.0E-07	54.0E-07	81.1E-07	10.8E-06	13.5E-06	16.2E-06
450.	-94727.65	43.04E+27	14.3E-06	28.5E-06	42.8E-06	57.1E-06	71.3E-06	85.6E-06
500.	-94605.95	56.99E+25	60.7E-06	12.1E-05	18.2E-05	24.3E-05	30.3E-05	36.4E-05
550.	-94487.69	12.33E+24	21.6E-05	43.3E-05	64.9E-05	86.6E-05	10.8E-04	13.0E-04
600.	-94372.78	42.19E+22	66.7E-05	13.3E-04	20.0E-04	26.7E-04	33.3E-04	40.0E-04
650.	-94261.22	20.84E+21	18.2E-04	36.3E-04	54.5E-04	72.7E-04	90.8E-04	10.9E-03
700.	-94153.16	14.05E+20	44.6E-04	89.3E-04	13.4E-03	17.9E-03	22.3E-03	26.8E-03
750.	-94048.71	12.36E+19	10.0E-03	20.1E-03	30.1E-03	40.2E-03	50.2E-03	60.2E-03
800.	-93948.02	13.86E+18	20.9E-03	41.8E-03	62.8E-03	83.7E-03	0.10	0.13
850.	-93851.83	18.38E+17	40.8E-03	81.6E-03	0.18	0.16	0.20	0.24

FORTRAN STOP
$

TABLE 3

GENERAL GAS-SOLID EQUILIBRIUM CODE (V_2O_3)

THERMODYNAMIC DATA | DATA SOURCE

V2S3 HEAT OF FORMATION (25 C) =	-125.000	KCAL MOL-1	MILLS (CORRELATION)
H2O HEAT OF FORMATION (25 C) =	-57.795	KCAL MOL-1	KUBASCHEWSKI
V2O3 HEAT OF FORMATION (25 C) =	-291.300	KCAL MOL-1	KUBASCHEWSKI-JANAF 1975
H2S HEAT OF FORMATION (25 C) =	-4.900	KCAL MOL-1	MILLS
V2S3 ENTROPY (25 C) =	31.000	CAL K-1 MOL-1	MILLS
H2O ENTROPY (25 C) =	45.106	CAL K-1 MOL-1	KUBASCHEWSKI
V2O3 ENTROPY (25 C) =	23.440	CAL K-1 MOL-1	KUBASCHEWSKI-JANAF 1975
H2S ENTROPY (25 C) =	49.150	CAL K-1 MOL-1	MILLS

HEAT CAPACITY EQUATION IS CP = A + BT + CT2 + DT3 + ET-2 CAL K-1 MOL-1

HEAT CAPACITY COEFFICIENTS	A	B	C	D	E	SOURCE
V2O3	29.35	0.00476000	0.00000000	0.00000000	-542000.00	B. OF MINES
H2S	7.81	0.00296000	0.00000000	0.00000000	-46000.00	MILLS
V2S3	28.00	0.01600000	0.00000000	0.00000000	0.00	MILLS(AVG)
H2O	7.17	0.00256000	0.00000000	0.00000000	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.	9601.94	36.60E-06	15.1E+05	30.1E+05	45.2E+05	60.2E+05	75.3E+05	90.4E+05
250.	9747.14	84.52E-06	11.4E+05	22.8E+05	34.2E+05	45.6E+05	57.0E+05	68.4E+05
300.	9885.48	16.97E-05	90.3E+04	18.1E+05	27.1E+05	36.1E+05	45.2E+05	54.2E+05
350.	10018.58	39.59E-05	74.2E+04	14.8E+05	22.3E+05	29.7E+05	37.1E+05	44.5E+05
400.	10147.39	50.60E-05	62.7E+04	12.5E+05	18.8E+05	25.1E+05	31.4E+05	37.6E+05
450.	10272.45	78.50E-05	54.2E+04	10.8E+05	16.3E+05	21.7E+05	27.1E+05	32.5E+05
500.	10394.05	11.62E-04	47.7E+04	95.4E+04	14.3E+05	19.1E+05	23.9E+05	28.6E+05
550.	10512.31	16.16E-04	42.6E+04	85.2E+04	12.8E+05	17.0E+05	21.3E+05	25.6E+05
600.	10627.24	21.86E-04	38.5E+04	77.1E+04	11.6E+05	15.4E+05	19.3E+05	23.1E+05
650.	10738.78	28.66E-04	35.2E+04	70.4E+04	10.6E+05	14.1E+05	17.6E+05	21.1E+05
700.	10846.84	36.62E-04	32.4E+04	64.9E+04	97.3E+04	13.0E+05	16.2E+05	19.5E+05
750.	10951.29	45.76E-04	30.1E+04	60.2E+04	90.4E+04	12.0E+05	15.1E+05	18.1E+05
800.	11051.98	56.16E-04	28.1E+04	56.3E+04	84.4E+04	11.3E+05	14.1E+05	16.9E+05
850.	11143.77	67.66E-04	26.4E+04	52.9E+04	79.3E+04	10.6E+05	13.2E+05	15.9E+05

FORTRAN STOP
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TABLE 4

EVALUATION OF EQUILIBRIUM CONSTANTS AS A FUNCTION OF TEMPERATURE FROM BASIC THERMODYNAMIC PRINCIPLES FOR THE ZINC OXIDE SYSTEM

The reaction is:

$$ZnO + H_2S \rightleftharpoons ZnS + H_2O$$

The equilibrium constant can be expressed in terms of the free energy change (Hougen, Watson and Ragatz part II p. 983) as follows:

$$K_{eQ} = e^{\frac{-\Delta G^\circ}{RT}}$$

The equilibrium constant can therefore be calculated by finding the value of ΔG^O, the standard free energy change for the above reaction. The standard free energy change for the above reaction is the difference between the sum of the ΔG^O for the products and the reactants, at a specified temperature.

$$\Delta G_{25^oC} = \Sigma\Delta G^\circ_{PRODUCTS} - \Sigma\Delta G^\circ_{REACTANTS}$$

The standard free energy change for the products and reactants have been taken from the CRC Handbook of Chemistry and Physics 61st edition 1980-81 pages D-71, D-76, and D-78. The values quoted in the CRC Handbook are from circular of the National Bureau of Standards 500, "Selected Values of Chemical Thermodynamic Properties", issued February 1, 1952.

Products		Reactants	
	ΔG_{25^oC} Kilocal/gmole		ΔG_{25^oC} Kilocal/gmole
ZnS	-47.4	ZnO	-76.05
H_2O	-54.64	H_2S	-7.892
	-102.04		-83.942

$$\Delta G_{25^oC} = -102.04 - (-83.942) = -18.1 \ \frac{\text{Kilocal}}{\text{gmole}}$$

This is the standard free energy change for the above reaction at 25°C. In order to get the ΔG^o at higher temperatures the procedure in Hougen, Watson and Ragatz part II p 986-987 is used. This procedure utilizes the heat capacities, heats of formations, and entropies of the reactants and products at a known temperature to calculate two integration constants. Once these are known ΔG^o can be calculated at any temperature using the following equation (HWR pt II p. 986 equation 14).

$$\frac{\Delta G^o}{T} = \frac{I_H}{T} + (\Delta a - Is) - \Delta a \ lnt - \frac{\Delta bT}{2} - \frac{\Delta cT^2}{6} - \frac{\Delta dT^3}{12} - \frac{\Delta e}{2T^2} \qquad (1)$$

where I_H and I_s are the integration constants to be calculated. I_H is calculated from the following equation (HWR part II p 986 equation 12)

$$\Delta H_T^\circ = I_H + \Delta aT + \frac{1 \ \Delta bT^2}{2} + \frac{1 \ \Delta cT^3}{3} + \frac{1}{4} \Delta dT^4 - \frac{\Delta e}{T} \qquad (2)$$

where a, b, c are the coefficient "deltas" in the empirical expression for heat capacities of products less reactants.

$$c_p^\circ + a + bT + cT^2 + dT^3 + \frac{e}{T^2}$$

these coefficients are required for each of the products and reactants in the reaction, and are listed in Table 1.

$$ZnO + H_2S \rightarrow ZnS + H_2O$$

$$\Delta H_{25^oC} = \Sigma\Delta H^\circ_{PRODUCTS} - \Sigma\Delta H^\circ_{REACTANTS}$$

The standard heat of formation for products and reactants was obtained from the CRC Handbook of Chemistry and Physics 61st edition 1980-81 pag D-71, D-76 and D-78.

Table 1

Source		a	b	c	Range
ZnO	Bulletin 324 U.S. Bureau of Mines p. 39	6.63	11.26×10^{-3}	-47.22×10^{-7}	0-1300°C
ZnS		7.05	15.15×10^{-3}	-9.17×10^{-7}	0-1000°C
H_2S	Hougen Watson & Ragatz pt II Table D Appendix	7.07	0.3128×10^{-2}	0.1364×10^{-5}	0-1527°C
H_2O		7.700	0.04594×10^{-2}	0.2521×10^{-5}	0-1527°C

$\Delta a = 14.75 - 13.7 = 1.05$

$\Delta b = 1.56 \times 10^{-2} - 1.4388 \times 10^{-2} = 1.2214 \times 10^{-3}$

$\Delta c = 1.604 \times 10^{-6} - (-3.358 \times 10^{-6}) = 4.962 \times 10^{-6}$

Products		Reactants	
	$\Delta H^{\circ}_{25^{o}C}$ Kilocal/gmole		$\Delta H^{\circ}_{25^{o}C}$ kilocal/gmole
ZnS	-48.5	ZnO	-83.17
H_2O	-57.8	H_2S	-4.815
	-106.3		-87.985

$$\Delta H^{\circ}_{25^{o}C} = -106.3 + 87.985 = -18.315 \ \frac{\text{Kilocal}}{\text{gmole}}$$

This value can now be used in equation (2) to find the integration constant I_H.

$$I_H = -(-18,315)+1.05(298.16)+\frac{1.2214 \times 10^{-3}(298.16)^2}{2}+\frac{4.962 \times 10^{-6}(298.16)^3}{3} + \frac{1}{4}\Delta dT^4 - \frac{\Delta e}{T}$$

$$I_H = -18,726$$

The second integration constant comes from the entropy equation (HWR pt. II p. 986 equation 13)

$$\Delta S^{\circ}_T = I_S + \Delta a \ln T + \Delta bT + \frac{1}{2}\Delta cT^2 + \frac{1}{3}\Delta dT^3 - \frac{\Delta e}{2T^2} \qquad (3)$$

$$\Delta S^{\circ}_{25^{o}C} = \Sigma S^{\circ}_{PRODUCTS} - \Sigma S^{\circ}_{REACTANTS}$$

The entropy of the products and reactants of the reaction are from the CRC Handbook of Chemistry and Physics 61st edition 1980-81 pages D-71, D-76 and D-78.

Products		Reactants	
	$S^{\circ}_{25^{o}C}$ cal/degmole		$S^{\circ}_{25^{o}C}$ cal/degmole
ZnS	13.8	ZnO	10.5
H_2O	45.11	H_2S	49.15
	58.91		59.65

$$\Delta S^{\circ}_{25^{o}C} = 58.91 - 59.65 = -0.74 \text{ cal/degmole}$$

using equation (3) above

$$-I_S = -(-0.74)+1.05\ \ln(298.16)+1.2214\times10^{-3}(298.16)+\frac{4.962}{2}\times10^{-6}\times(298.16)^2$$

$$I_S = -7.3072$$

Having calculated the two integration constants:

$$I_H = -18{,}726$$
$$I_S = -7.3072$$

The free energy change at any temperature can now be calculated using equation (1)

$$\frac{\Delta G^o}{T} = \frac{I_H}{T} + (\Delta a - I_S) - \Delta a\ \ln T - \frac{\Delta bT}{2} - \frac{\Delta cT^2}{6} - \frac{\Delta dT^3}{12} - \frac{\Delta e}{2T^2}$$

multiply by T

$$\Delta G^o = I_H + (\Delta a - I_S)T - \Delta aT\ \ln T - \frac{\Delta bT^2}{2} - \frac{\Delta cT^3}{6} - \frac{\Delta dT^4}{12} - \frac{\Delta e}{2T}$$

$$\Delta G_T^o = -18{,}726 + 8.3572T - 1.05\ T\ \ln T - 6.107\times10^{-4}T^2 - 8.27\times10^{-7}T^3 \qquad (4)$$

Using equation (4) the free energy change of the reaction can be calculated for any temperature within the valid range of the heat capacity values. Table 2 lists the ΔG_T^o calculated in this way.

A comparison was then made of this data with other published data to compare equilibrium constants. Table 3 shows the data calculated here, the data calculated by Gibson (Louisiana State University, Ph.D. thesis 1977, "Kinetics of the reactions of hydrogen sulfide and carbonyl sulfide with spherical zinc oxide particles"), and data from ICI published in the catalyst Handbook (1970).

Table 2

^oC	^oK	ΔG^o cal/gmole	K_{eQ}
200	473	-18,056	2.20×10^8
250	523	-18,078	3.58×10^7
300	573	-18,114	8.10×10^6
350	623	-18,166	2.36×10^6
400	673	-18,232	8.31×10^5
450	723	-18,313	3.43×10^5
500	773	-18,410	1.60×10^5
550	823	-18,524	8.29×10^4
600	873	-18,653	4.67×10^4
650	923	-18,800	2.83×10^4
700	973	-18,964	1.82×10^4
750	1023	-19,145	1.23×10^4
800	1073	-19,346	8.71×10^3
850	1123	-19,564	6.41×10^3

Table 3

^oC	SAI Keq	GIBSON Keq	ICI Keq
200	2.20×10^8		2.08×10^8
250	3.58×10^7	5.77×10^7	3.48×10^7
300	8.10×10^6	1.20×10^7	7.12×10^6
350	2.36×10^6	3.21×10^6	2.04×10^6
400	8.31×10^5	1.04×10^6	6.65×10^5
450	3.43×10^5	3.97×10^5	2.64×10^5
500	1.60×10^5	1.71×10^5	1.15×10^5
550	8.29×10^4	8.18×10^4	
600	4.67×10^4	4.25×10^4	
650	2.82×10^4	2.37×10^4	
700	1.82×10^4	1.40×10^4	
750	1.23×10^4	8.72×10^3	
800	8.72×10^3	5.67×10^3	
850	6.42×10^3	3.82×10^3	

Table 3 indicates some small differences in the equilibrium constants calculated by the three sources. These are probably due to different heat capacity values used in the calculations. Heat capacities can be found in the literature from various sources, and some differences do exist in the numbers obtained and the form of the empirical equation used. Figure 1 is a plot of the three sources of equilibrium constants in the form of an arrhenius plot. The relative differences of the equilibrium constants can be seen in the plot by the differences in the slope of the curves and their absolute values. The Gibson data appears to have a larger slope than either the SAI data or the ICI data, however it coincides with the SAI data at about 550°C. The ICI values almost match the SAI values at low temperatures (200-250°C) but deviate from both the Gibson and SAI values at higher temperatures.

Since it is difficult to assess the impact of these discrepancies in equilibrium values on the actual H_2S level to be expected without actually calculating these values this was done in Table 4.

Table 4 shows that the largest discrepancies between the SAI values and Gibsons values are at the higher temperatures and at higher steam levels. In the temperatures range of interest for Hot Gas Desulfurization (approximately 550-650°C), the largest discrepancy is 2.02 ppm. The ICI values, which are not available above 500°C, would indicate larger discrepancies if extrapolated to higher temperatures.

Since experimental data at METC have verified the levels predicted by Gibson and SAI, it is felt that these values are valid, and can be used in experimental data analysis and process simulation. The calculation sequence outlined here provides a means of calculating equilibrium constants at any temperature based on heat capacity, free energy, heat of reaction, and entropy data, using basic thermodynamic principles. The accuracy of the results obtained are therefore directly related to the accuracy of the data used. The source of the data used here was specified, and is believed to be accurate.

In order to represent the H_2S equilibrium values at varying temperatures and steam levels graphically, figure 2 was drawn. This plot was drawn using the equilibrium constants calculated here. It can be seen in the plot that at

Table 4

Equilibrium H_2S Values in PPM									
°C	SAI Steam			Gibson Steam			ICI Steam		
	5%	15%	30%	5%	15%	30%	5%	15%	30%
250	.0014	.004	.008	.0008	.0026	.005	.0014	.0043	.0086
350	.0212	.063	.127	.0155	.0467	.093	.0245	.0735	.1470
450	.1457	.437	.875	.1259	.3778	.755	.1892	.5675	1.135
550	.6031	1.809	3.618	.6112	1.83	3.667			
650	1.773	5.319	10.63	2.10	6.33	12.65			
750	4.064	12.19	24.39	5.73	17.20	34.40			
850	7.79	23.36	46.72	13.08	39.26	78.53			

temperatures up to 450^oC equilibrium H_2S levels are below 1 ppm for all steam levels up to 30%. As temperatures increase above 450^oC there is an accelerated increase of H_2S with temperature, with a large increase between 550^oC-650^o at the high steam level. This plot can be used to determine the tradeoff between steam level and temperature in order to stay below 1 ppm H_2S.

A computer code has been written to carry out the calculations outlined here and to produce a table containing the information shown in Table 2 and 4. The required input includes the following:

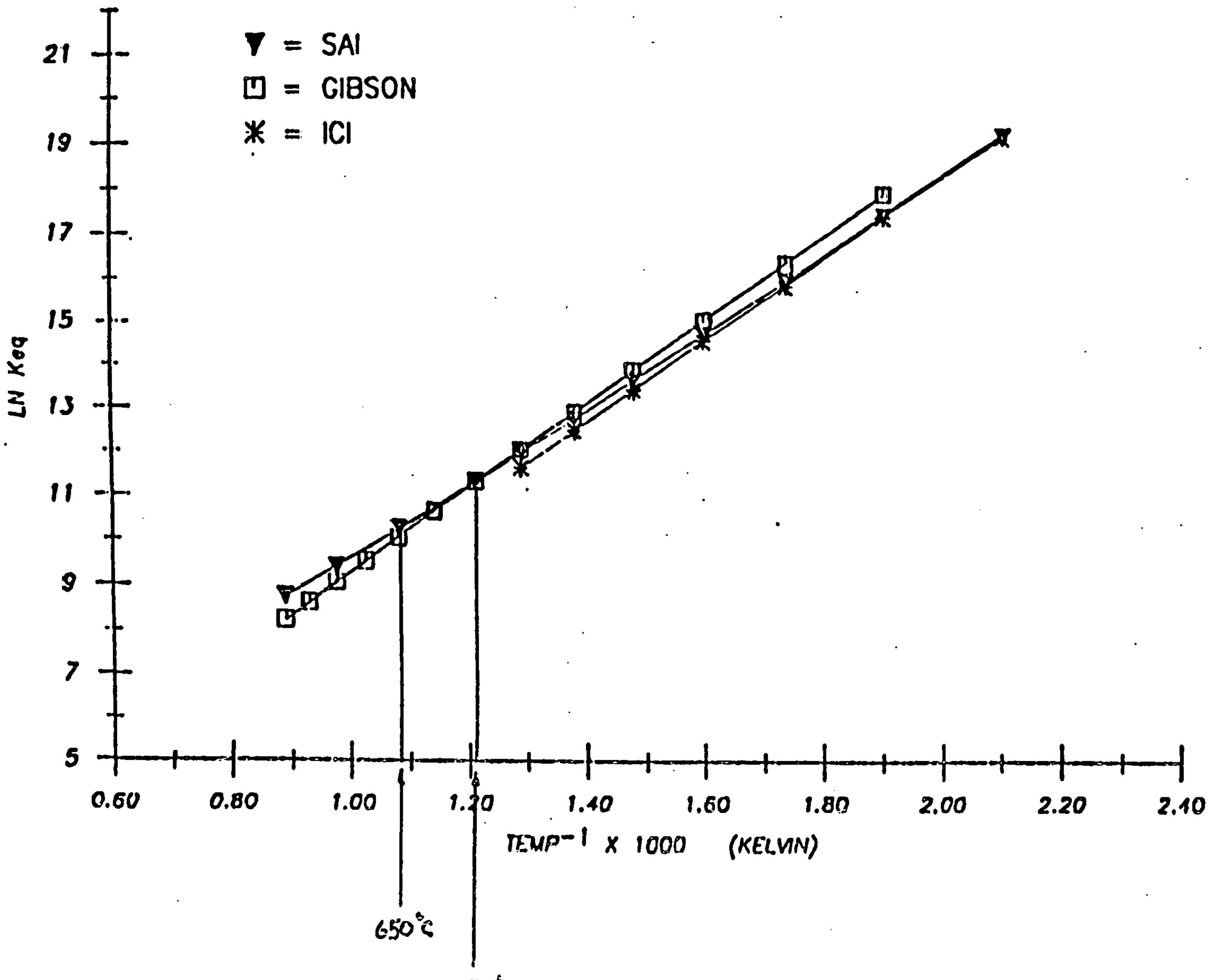
- o Heat of formation of products and reactants at 25^oC
- o Entropy of products and reactants at 25^oC
- o Heat capacity coefficients for products and reactants

Using this input data the code calculates the free energy change at various temperatures, the equilibrium constant, and equilibrium H_2S levels at various steam levels. This code can now be used to test the effect of various sources and values of thermodynamics data and their effect on the equilibrium H_2S levels calculated. A sample output is shown in Table 5.

FIGURE 1

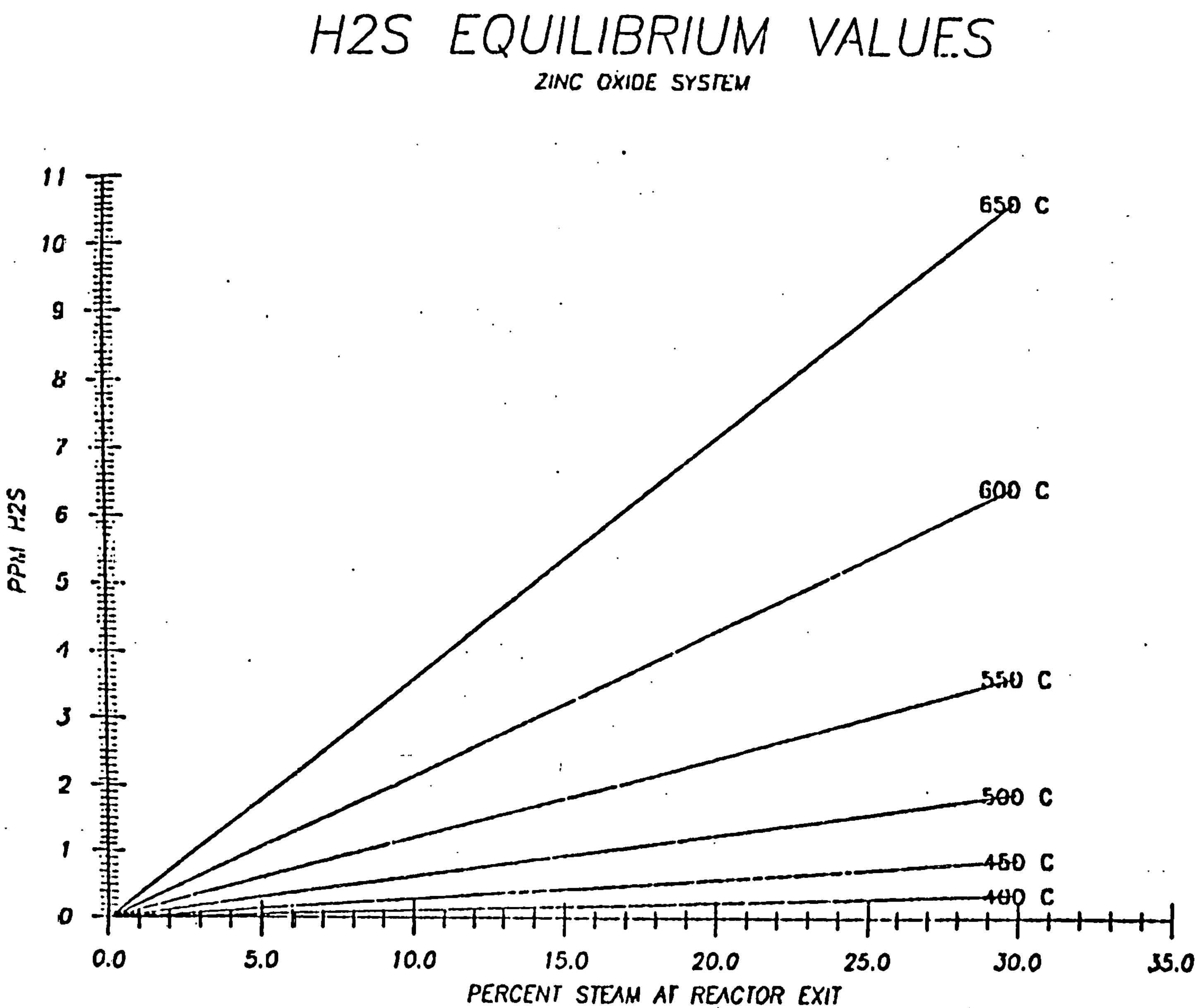
P1

ZNO EQUILIBRIUM CONSTANT



10-NOV-81 11:35:58

FIGURE 2



10-NOV-81 15:15:17

THERMOG CODE

```
C        THIS PROGRAM CALCULATES THE EQUILIBRIUM CONSTANT FOR THE
C        REACTION:    ARSOLID + BRGAS GOING TO CPSOLID + DPGAS
C        AT ANY TEMPERATURE WHERE  A, B, C, D, ARE STOICHIOMETRIC
C        COEFFICIENTS.  RSOLID IS REACTANT SOLID, RGAS IS REACTANT
C        GAS,PSOLID IS PRODUCT SOLID, AND PGAS IS PRODUCT GAS.
C
C        READ STOICHIOMETRIC COEFFICIENTS A,B,C,D
C
C        READ HEAT OF FORMATION OF PRODUCTS AND REACTANTS IN UNITS OF
C        CALORIES/GMOLE AT 25 DEGREES CENTIGRADE.
C
C        READ ENTROPY OF PRODUCTS AND REACTANTS IN UNITS OF CAL/DEGMOLE
C        AT 25 DEGREES CENTIGRADE.
C
         IMPLICIT REAL*8(A-H,O-Z)
         CHARACTER*80 SRCHPSOLID,SRCHPGAS,SRCHRSOLID,SRCHRGAS
         CHARACTER*80 SRCSPSOLID,SRCSPGAS,SRCSRSOLID,SRCSRGAS
         CHARACTER*80 RSLD,PSLD
         CHARACTER*80 SRC1,SRC2,SRC3,SRC4
         REAL DELTAH,DELTAS,IH,IS,DELA,DELB,DELC,DELD,DELE
         REAL*8 KEQ
         EXTERNAL LIB$EMULATE
         DIMENSION TEMP(20),DELG(20),POWER(20), KEQ(20)
         DIMENSION H2S5(20),H2S10(20),H2S15(20),H2S20(20),H2S25(20),H2S30(20)
         CALL LIB$ESTABLISH(LIB$EMULATE)
         READ(9,*) A,B,C,D
         READ(9,*)  HPSOLID,HPGAS,HRSOLID,HRGAS
         READ(9,*) SPSOLID,SPGAS,SRSOLID,SRGAS
C        SPECIFY SOURCE OF THIS DATA
         READ(9,5) SRCHPSOLID,SRCHPGAS,SRCHRSOLID,SRCHRGAS
         READ(9,5) SRCSPSOLID,SRCSPGAS,SRCSRSOLID,SRCSRGAS
5        FORMAT(A80)
         READ(9,6) RSLD,PSLD
6        FORMAT(A80)
         WRITE(6,500)
500      FORMAT(T40,'GENERAL GAS-SOLID EQUILIBRIUM CODE',//,
     1   T10,'THERMODYNAMIC DATA',T80,'DATA SOURCE',/)
         WRITE(6,50) PSLD,HPSOLID,SRCHPSOLID,HPGAS,SRCHPGAS,RSLD,HRSOLID,
     1   SRCHRSOLID,HRGAS,SRCHRGAS
50       FORMAT(5X,A5,T10,'HEAT OF FORMATION (25 C) = ',1X,F8.3,3X,
     1   'KCAL MOL-1',T65,A60,/,5X,'H2O HEAT OF FORMATION (25 C) = '
     1   ,1X,F8.3,3X,'KCAL MOL-1',T65,A60,/,5X,A5,T10,
     1   'HEAT OF FORMATION (25 C) = ',1X,F8.3,3X,'KCAL MOL-1',T65,A60,/,
     1   5X,'H2S HEAT OF FORMATION (25 C) = ',1X,F8.3,3X,'KCAL MOL-1',T65
     1   ,A60)
         WRITE(6,60) PSLD,SPSOLID,SRCSPSOLID,SPGAS,SRCSPGAS,RSLD,SRSOLID,
     1   SRCSRSOLID,SRGAS,SRCSRGAS
60       FORMAT(//,5X,A5,T10,'ENTROPY (25 C) = ',F7.3,3X,'CAL K-1 MOL-1',
     1   T65,A60,/,5X,'H2O ENTROPY (25 C) = ',F7.3,3X,'CAL K-1 MOL-1',
     1   T65,A60,/,5X,A5,T10,'ENTROPY (25 C) = ',F7.3,3X,'CAL K-1 MOL-1',
     1   T65,A60,/,5X,'H2S ENTROPY (25 C) = ',F7.3,3X,'CAL K-1 MOL-1',
     1   T65,A60)
```

```
C
C       READ HEAT CAPACITY COEFFICIENTS FOR PRODUCTS AND REACTANTS
C       USING THE FOLLOWING CORRELATION:  CP = A+BT+CT2+DT3+E/T2
C       ASSIGN SUBSCRIPTS AS FOLLOWS:
C
C       RSOLID   A1   B1   C1   D1   E1
C       H2S      A2   B2   C2   D2   E2
C
C       PSOLID   A3   B3   C3   D3   E3
C       H2O      A4   B4   C4   D4   E4
C
        READ(9,*)   A1,B1,C1,D1,E1
        READ(9,*)   A2,B2,C2,D2,E2
        READ(9,*)   A3,B3,C3,D3,E3
        READ(9,*)   A4,B4,C4,D4,E4
C       READ SOURCE FOR HEAT CAPACITY COEFFICIENTS
        READ(9,69)   SRC1
        READ(9,69)   SRC2
        READ(9,69)   SRC3
        READ(9,69)   SRC4
69      FORMAT(A80)
        WRITE(6,70)
70      FORMAT(//,5X,'HEAT CAPACITY EQUATION IS
     1  CP = A + BT + CT2 + DT3 + ET-2',10X,'CAL K-1 MOL-1')
        WRITE(6,80)
80      FORMAT(//,5X,'HEAT CAPACITY COEFFICIENTS',12X,'A',13X,'B',13X
     1  ,'C',13X,'D',13X,'E',15X,'SOURCE')
        WRITE(6,90) RSLD,A1,B1,C1,D1,E1,SRC1,A2,B2,C2,D2,E2,
     1  SRC2,PSLD,A3,B3,C3,D3,E3,SRC3,A4,B4,C4,D4,E4,SRC4
90      FORMAT(//,15X,A5,T41,F5.2,5X,F10.8,5X,F11.9,5X,F11.9,5X,F11.2,5X,A12
     1  ,/,15X,'H2S ',21X,F5.2,5X,F10.8,5X,F11.9,5X,F11.9,5X,F11.2,5X,A12
     1  ,/,15X,A5,T41,F5.2,5X,F10.8,5X,F11.9,5X,F11.9,5X,F11.2,5X,A12,/,
     1  15X,'H2O ',21X,F5.2,5X,F10.8,5X,F11.9,5X,F11.9,5X,F11.2,5X,A12)
C
C       CALCULATE DELTA A ,DELTA B DELTA C, DELTA D,DELTA E
C       PRODUCTS MINUS REACTANTS
C
        DELA = ((C*A3)+(D*A4))-((A*A1)+(B*A2))
        DELB = ((C*B3)+(D*B4))-((A*B1)+(B*B2))
        DELC = ((C*C3)+(D*C4))-((A*C1)+(B*C2))
        DELD = ((C*D3) +(D*D4))-((A*D1)+(B*D2))
        DELE = ((C*E3)+(D*E4))-((A*E1)+(B*E2))
C
C       CALCULATE DELTA H FOR THE REACTION FROM INPUT DATA
C
        DELTAH = (((C*HPSOLID)+(D*HPGAS))-((A*HRSOLID)+(B*HRGAS)))*1000.
C
C       CALCULATE INTEGRATION CONSTANT IH USING EQUATION 12 IN
C       HOUGEN WATSON AND RAGATZ PART II  P. 986
C
        IH = DELTAH - DELA*(298.16) - (DELB/2.)*(298.16**2) - (DELC/3.)*
     1  (298.16**3) - (DELD/4.)*(298.16**4) + DELE/298.16
```

```
C
C       CALCULATE DELTA S FOR THE REACTION FROM INPUT DATA
C
        DELTAS = ((C*SPSOLID) + (D*SPGAS)) - ((A*SRSOLID) + (B*SRGAS))
C
C       CALCULATE INTEGRATION CONSTANT IS USING EQUATION 13 IN
C       HOUGEN WATSON AND RAGATZ PART II P. 986
C
        IS = DELTAS - DELA*LOG(298.16) - DELB*(298.16) - (DELC/2.)*
     1  (298.16**2) - (DELD/3.)*(298.16**3) + DELE/(2.*(298.16**2))
C
C       HAVING CALCULATED INTEGRATION CONSTANTS IH AND IS
C       CALCULATE DELTA G FOR THE DESIRED TEMPERATURE RANGE
C       200 TO 850 CENTIGRADE
C
        DO 10 I=1,14
        TEMP(I) = 473 + (I-1)*50
        DELG(I) = IH + (DELA - IS)*TEMP(I)-DELA*TEMP(I)*LOG(TEMP(I))
     1  -DELB*((TEMP(I)**2)/2.) - DELC*((TEMP(I)**3)/6.)
     1  -DELD*((TEMP(I)**4)/12.)-DELE/(2.*TEMP(I))
10      CONTINUE
C
C       CALCULATE EQUILIBRIUM CONSTANT
C
        DO 20 I=1,14
        POWER(I) = -DELG(I)/(1.9872*TEMP(I))
        KEQ(I) = EXP(POWER(I))
20      CONTINUE
C
C       CALCULATE EQUILIBRIUM H2S COMPOSITIONS WITH VARIOUS STEAM LEVELS
C
        DO 25 I=1,14
        H2S5(I) = (((0.05**D)/KEQ(I))**(1/B))*(1E6)
        H2S10(I)= (((0.10**D)/KEQ(I))**(1/B))*(1E6)
        H2S15(I)= (((0.15**D)/KEQ(I))**(1/B))*(1E6)
        H2S20(I)= (((0.20**D)/KEQ(I))**(1/B))*(1E6)
        H2S25(I)= (((0.25**D)/KEQ(I))**(1/B))*(1E6)
        H2S30(I)= (((0.30**D)/KEQ(I))**(1/B))*(1E6)
25      CONTINUE
C
C       CONVERT TEMPERATURE FROM KELVIN TO CENTIGRADE
C
        DO 30 I=1,14
        TEMP(I) = TEMP(I) -273.
30      CONTINUE
C
C       PRINTOUT TABLE OF DELTA G AND EQUILIBRIUM CONSTANTS GENERATED
C
        WRITE(6,200)
200     FORMAT(1H,//,T4,11HTEMPERATURE,T20,7HDELTA G,T37,11HEQUILIBRIUM,
     1  T73,28HEQUILIBRIUM H2S LEVEL IN PPM/1H,T4,9HDEGREES C,T20,
     1  9HCAL/GMOLE,T37,8HCONSTANT,T80,13HPERCENT STEAM//1H,T62,2H5%,
     1  T70,3H10%,T80,3H15%,T90,3H20%,T100,3H25%,T110,3H30%/)
```

```
      DO 40 I=1,14
      WRITE(6,300) TEMP(I),DELG(I),KEQ(I),H2S5(I),H2S10(I),H2S15(I),
    1 H2S20(I),H2S25(I),H2S30(I)
300   FORMAT(1H,T8,F4.0,T20,F10.2,T37,2PG10.3,T57,6(2PG10.2),/)
40    CONTINUE
      STOP
      END
```

Simultaneous Shift/Methanation
Equilibrium Calculations

Reference:

H.P. Meissner, C. L. Kusik, W. H. Dalzell
Equilibrium Compositions with Multiple Reactions

Note: The following are results obtained from a computer code used to calculate simultaneous shift/methanation equilibria

```
RUN
RGEQ                    3-AUG-1982 08:54

? .5029
? 2.527
? .0001
? 1112
                        GAS COMPOSITIONS 1112 F

                           INLET          OUTLET

                           MOL %           MOL %


H2S =                      2.700           0.0
H2O =                     25.00           20.19
CO2 =                      9.79           14.73
CO  =                     11.69            7.59
H2  =                     17.13           26.27
CH4 =                      2.91            1.35
N2  =                     30.78           29.87
Ready
```

```
RUN
RGEQ                    3-AUG-1982 08:55

? 2.686
? 1.923
? .001
? 1202
                        GAS COMPOSITIONS 1202 F

                           INLET          OUTLET

                           MOL %           MOL %


H2S =                      2.700           0.0
H2O =                     25.00           19.97
CO2 =                      9.79           13.32
CO  =                     11.69            9.53
H2  =                     17.13           27.50
CH4 =                      2.91            0.37
N2  =                     30.78           29.30
Ready
```

RUN
RGEQ 3-AUG-1982 08:52

? .009442
? 4.878
? .000001
? 932

GAS COMPOSITIONS 932 F

	INLET MOL %	OUTLET MOL %
H2S =	2.700	0.0
H2O =	25.00	24.60
CO2 =	9.79	17.46
CO =	11.69	2.52
H2 =	17.13	17.35
CH4 =	2.91	5.68
N2 =	30.78	32.39

Ready

RUN
RGEQ 3-AUG-1982 08:53

? .07741
? 3.434
? .00001
? 1022

GAS COMPOSITIONS 1022 F

	INLET MOL %	OUTLET MOL %
H2S =	2.700	0.0
H2O =	25.00	21.84
CO2 =	9.79	16.34
CO =	11.69	4.91
H2 =	17.13	22.55
CH4 =	2.91	3.33
N2 =	30.78	31.02

Ready

RUN
RGEQ 3-AUG-1982 08:57

? .009442
? 4.878
? .000001
? 932

GAS COMPOSITIONS 932 F

	INLET MOL %	OUTLET MOL %
H2S =	2.70	0.0
H2O =	0.0	4.38
CO2 =	9.79	13.62
CO =	11.69	5.34
H2 =	17.13	8.37
CH4 =	2.91	7.58
N2 =	55.78	60.70

Ready

RUN
RGEQ 3-AUG-1982 08:57

? .07741
? 3.434
? .00001
? 1022

GAS COMPOSITIONS 1022 F

	INLET MOL %	OUTLET MOL %
H2S =	2.70	0.0
H2O =	0.0	4.20
CO2 =	9.79	10.97
CO =	11.69	9.28
H2 =	17.13	12.19
CH4 =	2.91	5.18
N2 =	55.78	58.17

Ready

RUN
RGEQ 3-AUG-1982 09:00

? .5029
? 2.527
? .0001
? 1112

GAS COMPOSITIONS 1112 F

	INLET MOL %	OUTLET MOL %
H2S =	2.70	0.0
H2O =	0.0	3.99
CO2 =	9.79	8.31
CO =	11.69	13.26
H2 =	17.13	16.08
CH4 =	2.91	2.75
N2 =	55.78	55.61

Ready

RUN
RGEQ 3-AUG-1982 09:01

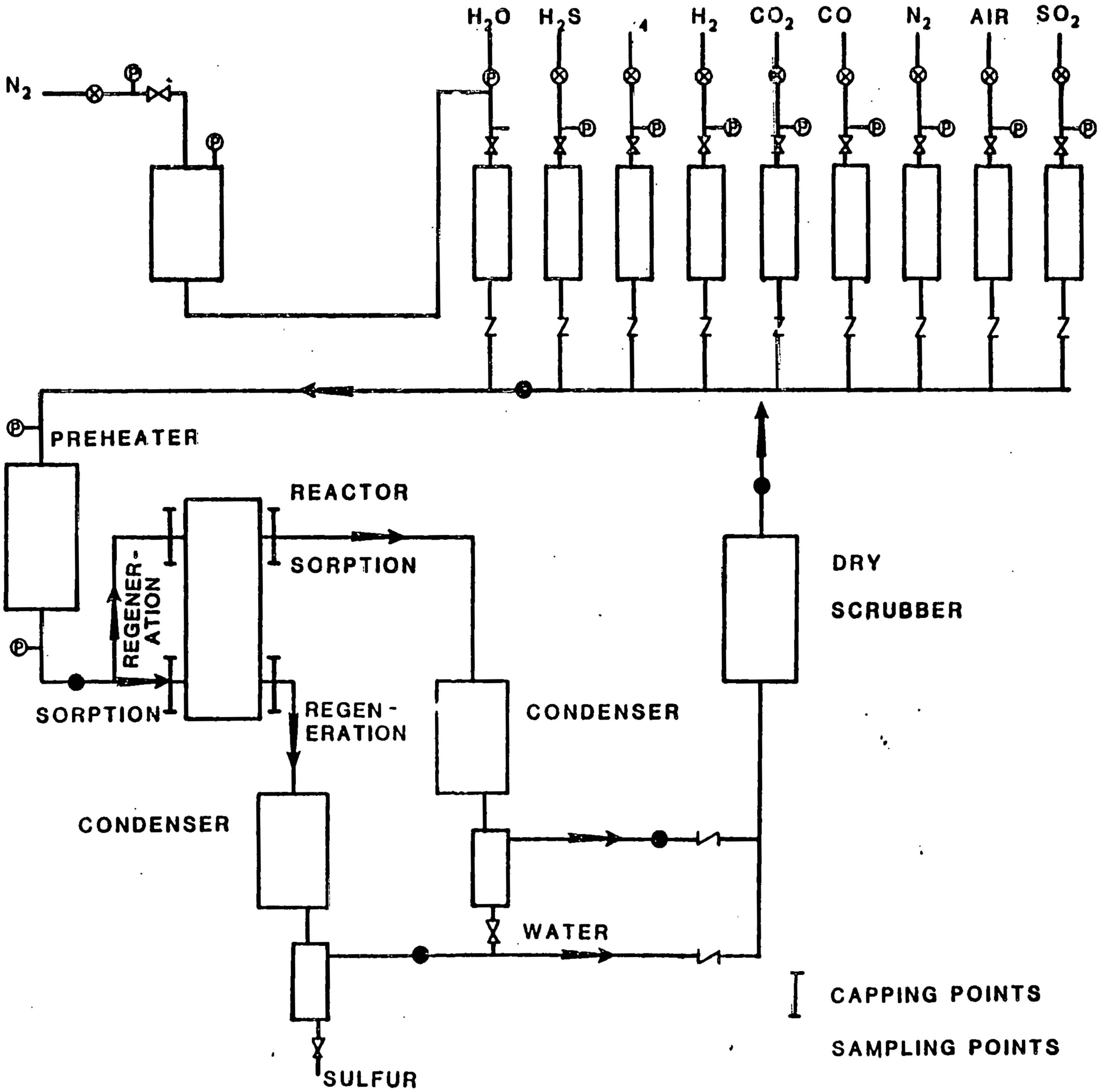
? 2.686
? 1.923
? .001
? 1202

GAS COMPOSITIONS 1202 F

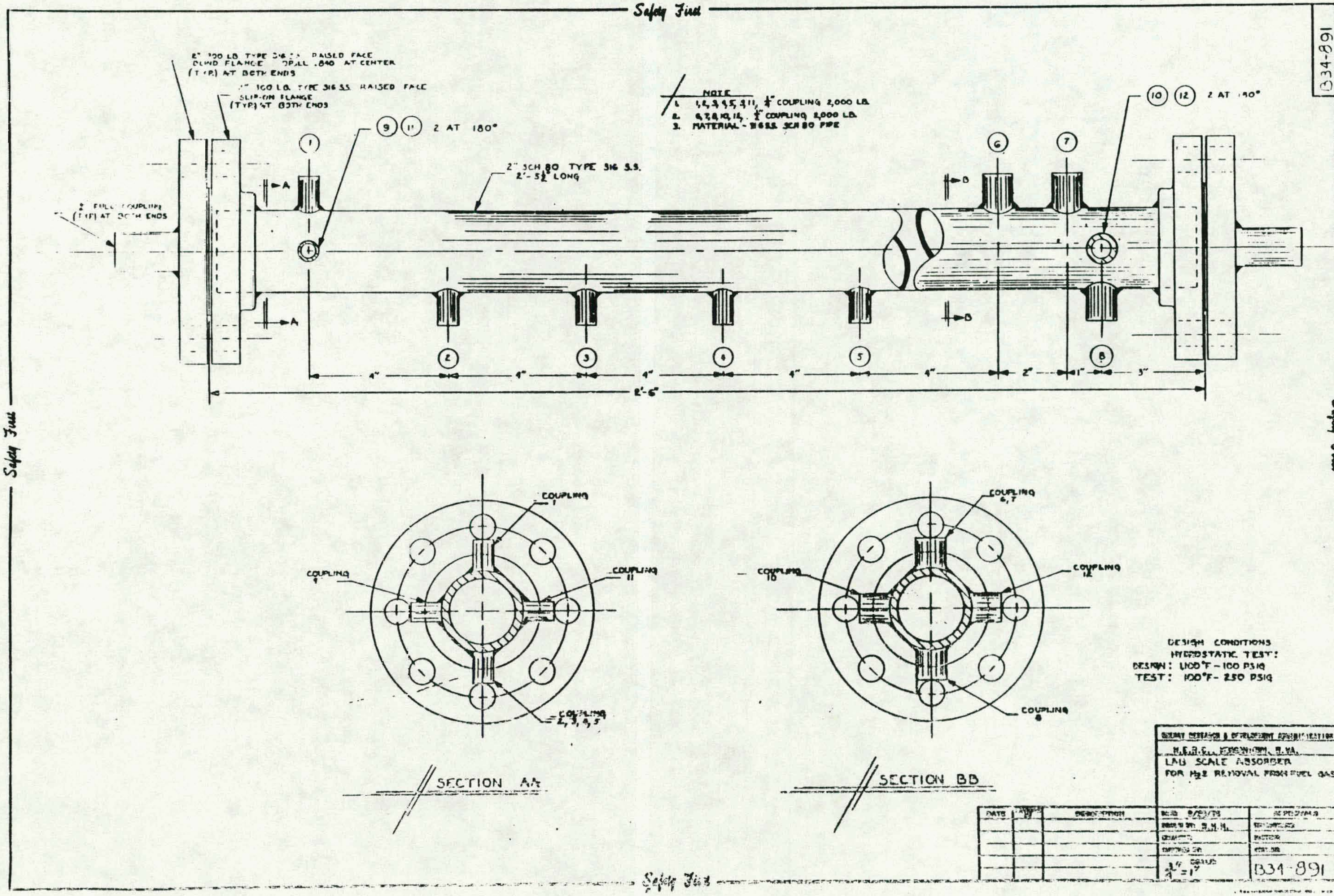
	INLET MOL %	OUTLET MOL %
H2S =	2.70	0.0
H2O =	0.0	3.88
CO2 =	9.79	6.39
CO =	11.69	16.11
H2 =	17.13	18.80
CH4 =	2.91	1.03
N2 =	55.78	53.80

Ready

HOT GAS CLEANUP FLOW SCHEMATIC



Schematic Drawing of Test Unit



APPENDIX D

Laboratory-Scale Test Summary Sheets and Data Curves

TEST SUMMARY SHEET

TEST NO. : 001
DATE STARTED : 7/30/80
DATE ENDED : 7/31/80
TOTAL HOURS : 28
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF IRON OXIDE EXTRUSIONS FOR REGENERATION TESTS TO VERIFY AIR PRODUCTS TEST RESULTS IN WHICH ELEMENTAL SULFUR WAS PRODUCED DURING REGENERATION

SORBENT TYPE/WEIGHT: IRON OXIDE/936 G
SORBENT NO. : 811X3-1X1
SORBENT COMPOSITION: 42% IRON OXIDE (YOUNGSTOWN SHEET & TUBE) ON SILICA
SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR LOADING : 4.7 % (GM SULFUR/GM FRESH SORBENT) TO 400 PPM BREAKTHROUGH

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (XW/W):	0.05	6.42 (EXIT)
SURFACE AREA (M2/G):	0.75	1.07
DENSITY (G/CM3):	3.15	3.37
PORE VOLUME (MM3/G)	0.4	2.5
MINERAL ANALYSIS :	SEE APPENDIX	
ELEMENTAL ANALYSIS :	SEE APPENDIX	

Note: Please disregard references to Appendix for all Test Summary Sheets included herein.

OPERATING CONDITIONS

TEMPERATURE: 1000 F(538C)

PRESSURE : AMBIENT

SPACE VELOCITY: 800 HOURLY

	FLOW RATE		MOLE
H2S	(0.07DM3/MIN)	0.14 SCFH	0.7%
H2O		0.52 CM3/MIN	7%
AIR			
CO2			
CO			
H2	(1.38DM3/MIN)	2.92 SCFH	15%
CH4			
N2	(7.13DM3/MIN)	15.11 SCFH	77.3%

DATA: (DETECTOR TUBE)

EXIT H2S: 200-400 PPM PLATEAU
3000 PPM AT BREAKTHROUGH

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. SULFUR LOADING CORRESPONDS TO BREAKTHROUGH FROM PLATEAU.
2. REACTOR TEMPERATURE PROFILE WAS NOT MAINTAINED WITHIN 50 DEGREES OF 1000 F.
3. THIS TEST SERVED TO SHAKEDOWN THE TEST EQUIPMENT. ONLY ONE MALFUNCTION WAS EXPERIENCED (H2S REGULATOR REPLACED).

CONCLUSIONS

1. IRON OXIDE CAN REDUCE THE SULFUR LEVEL IN A HOT GAS STREAM FROM 7000 PPM TO 200-400 PPM IN THE PRESENCE OF 7% WATER.
2. SULFIDED IRON OXIDE IS READY FOR REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 002
DATE STARTED : 10/16/80
DATE ENDED : 10/24/80
TOTAL HOURS : 39
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: IRON OXIDE/936 G
SORBENT NO. : 811X3-1X1
SORBENT COMPOSITION: 42% IRON OXIDE (YOUNGSTOWN SHEET & TUBE) ON SILICA

SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H_2S		
H_2O	4.24 CM3/MIN	95 %
AIR (0.28 DM3/MIN)	0.588 SCF/HR	5 %
CO_2		
CO		
H_2		
CH_4		
N_2		

S

PURPOSE

VERIFICATION OF AIR PRODUCTS TEST RESULTS IN WHICH ELEMENTAL SULFUR WAS PRODUCED DURING REGENERATION.

ANALYSIS

	BEFORE	AFTER
TOTAL SULFUR (%W/W)	6.42 (EXIT)	0.16 (EXIT)
SURFACE AREA (M2/G):	1.07	
DENSITY (G/CM3) :	3.371	
PORE VOLUME (MM3/G):	2.5	
MINERAL ANALYSIS :	SEE APPENDIX	
ELEMENTAL ANALYSIS :	SEE APPENDIX	

DATA: (TUTWILER METHOD)

EXIT H_2S: 7.2 %

EXIT SO_2: NONE BY TUTWILER (AVG) EXCEPT AT END OF RUN

EXIT S_2: 122.1 G

EXIT H_2:

REMARKS

1. DESIRED TEMPERATURE WAS NOT MAINTAINED
2. ESSENTIALLY COMPLETE REGENERATION WAS ACHIEVED AS CALCULATED BY MATERIAL BALANCE, AND SHOWN BY ANALYSIS.

CONCLUSIONS

1. HIGH ELEMENTAL SULFUR YIELDS ARE ACHIEVABLE WITH STEAM/AIR REGENERATION (95 %/ 5 %).
2. 76.8 % OF THE SULFUR REMOVED WAS IN THE FORM OF ELEMENTAL SULFUR.

TEST SUMMARY SHEET

TEST NO. : 003
DATE STARTED : 11/3/80
DATE ENDED : 11/3/80
TOTAL HOURS : 8.5
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF IRON OXIDE EXTRUSIONS FOR SUBSEQUENT REGENERATION TESTS USING 90% STEAM AND 10% AIR.

SORBENT TYPE/WEIGHT: IRON OXIDE/936 G
SORBENT NO. : 811X3-1X1
SORBENT COMPOSITION: 42% IRON OXIDE(YOUNGSTOWN SHEET & TUBE) ON SILICA

SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR LOADING : 4.47% (GM SULFUR/GM FRESH SORBENT) TO 400 PPM BREAKTHROUGH

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):		
DENSITY (GM/CM3) :		
PORE VOLUME (MM3/G):		
MINERAL ANALYSIS :		
ELEMENTAL ANALYSIS :		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1013 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: UP TO 1900 PPM PLATEAU
11000 PPM AT BREAKTHROUGH

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE
H2S (0.26 DM3/MIN)	0.545 SCFH	2.7%
H2O	0.52 CM3/MIN	6.9%
AIR		
CO2		
CO		
H2 (1.38 DM3/MIN)	2.92 SCFH	14.6%
CH4		
N2 (7.13 DM3/MIN)	15.11 SCFH	75.8%

$

REMARKS

1. REACTOR INLET TEMP LOW BY 75-100 F DURING RUN.

CONCLUSIONS

1. AT SV=1013 AND 2.7% H2S INLET CONCENTRATION IN THE PRESENCE OF 6.9% WATER, STEADY STATES DESULFURIZATION WAS MAINTAINED FOR 5 HRS.
2. SULFIDATION CAN BE COMPLETED IN ONE DAY.

TEST SUMMARY SHEET

TEST NO. : 004
DATE STARTED : 11/05/80
DATE ENDED : 11/06/80
TOTAL HOURS : 20.5
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: IRON OXIDE/936 G
SORBENT NO. : 811X3-1X1
SORBENT COMPOSITION: 42% IRON OXIDE (YOUNGSTOWN SHEET & TUBE) ON SILICA

SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 622 HOURLY

	FLOW RATE		MOLE
H2S			
H2O	4.2	CM3/MIN	89 %
AIR (0.56 DM3/MIN)	1.18	SCF/HR	19 %
CO2			
CO			
H2			
CH4			
N2			

8

PURPOSE

REGENERATION OF IRON OXIDE EXTRUSIONS USING 90% STEAM AND 10% AIR.

ANALYSIS

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):		
DENSITY (G/CM3) :		
PORE VOLUME (MM3/G) :		
MINERAL ANALYSIS :		
ELEMENTAL ANALYSIS :		

DATA: (TUTUILER METHOD)

EXIT H2S: 5000-6000 PPM (BY DETECTOR TUBES)

EXIT SO2: GREATER THAN 3000 PPM (BY TUTUILER)

EXIT S2: 19.41 G

EXIT H2:

REMARKS

1.
2.

CONCLUSIONS

1. ELEMENTAL SULFUR YIELDS ARE SIGNIFICANTLY LOWER WITH 90%/10% STEAM-AIR REGENERATION.

2.

TEST SUMMARY SHEET

TEST NO. : 005
DATE STARTED : 1/28/81
DATE ENDED : 1/28/81
TOTAL HOURS : 9.5
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF IRON OXIDE EXTRUSIONS FOR SUBSEQUENT REGENERATION TESTS USING 50% STEAM AND 50% AIR.

SORBENT TYPE/WEIG HT: IRON OXIDE/1000 G
SORBENT NO. : 811X3-1X1
SORBENT COMPOSITION: 42% IRON OXIDE(YOUNGSTOWN SHEET & TUBE) ON SILICA

SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR LOADING : 10.4 % (GM SULFUR/GM FRESH SORBENT) TO 400 PPM BREAKTHROUGH

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR(%W/W):		
SURFACE AREA(M2/G):		
DENSITY (G/CM3) :		
PORE VOLUME (MM3/G):		
MINERAL ANALYSIS :		
ELEMENTAL ANALYSIS :		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 900 HOURLY

	FLOW RATE	MOLE
H2S (0.26 DM3/MIN)	0.545 SCFH	2.7%
H2O	0.52 CM3/MIN	6.9%
AIR		
CO2		
CO		
H2 (1.38 DM3/MIN)	2.92 SCFH	14.6%
CH4		
N2 (7.13 DM3/MIN)	15.11 SCFH	75.8%

S

DATA: (DETECTOR TUBE)

EXIT H2S: 6 PPM PLATEAU FOR 3 HOURS BREAKTHROUGH AT 4 HOURS WITH 160 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. TEMPERATURE CONTROL WITHIN 40 F OF ABSORBER OPERATING TEMPERATURE.

CONCLUSIONS

1. AT TEST CONDITIONS IRON OXIDE CAN REDUCE EXIT H2S TO BELOW 10 PPM FOR 3 HOURS AND BELOW 1000 PPM FOR FOR 6 HOURS.

TEST SUMMARY SHEET

TEST NO. : 806
DATE STARTED : 1/29/81
DATE ENDED : 1/29/81
TOTAL HOURS : 7.5
TYPE : REGENERATION

PURPOSE

REGENERATION OF IRON OXIDE EXTRUSIONS USING 50% STEAM AND 50% AIR.

SORBENT TYPE/WEIGHT: IRON OXIDE/1000 G
SORBENT NO. : 811X3-1X1
SORBENT COMPOSITION: 42% IRON OXIDE(YOUNGSTOWN SHEET & TUBE) ON SILICA
SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR REMOVED :

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		0.7
SURFACE AREA (M2/G):		0.94
DENSITY (GM/CM3):		3.18
PORE VOLUME (MM3/G):		1.8
MINERAL ANALYSIS :	SEE APPENDIX	
ELEMENTAL ANALYSIS :	SEE APPENDIX	

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 1000-1200 PPM

EXIT SO2: 4-7 %

EXIT S2: 2.514 G

EXIT H2:

	FLOW RATE	MOLE
H2S		
H2O	2.49 CM3/MIN	50%
AIR (3.11 DM3/MIN)	6.58 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		
S		

REMARKS

1. TEMPERATURE WAVE REACHING APPROX. 1500 F WAS OBSERVED.

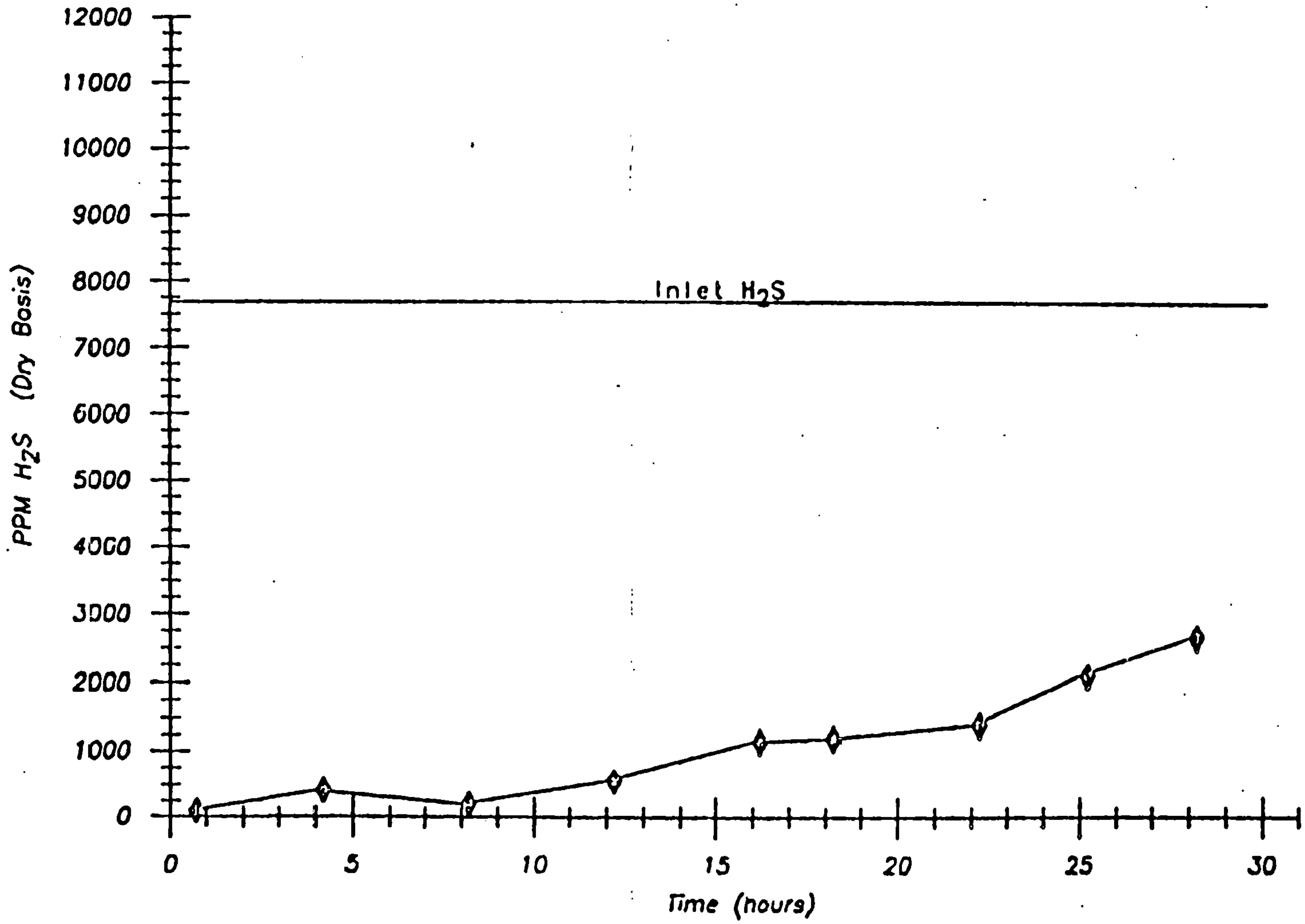
CONCLUSIONS

1. ELEMENTAL SULFUR PRODUCED IS GREATLY DECREASED WITH STEAM LEVEL REDUCED TO 50 %.

P:

IRON OXIDE SULFIDATION

TEST 001

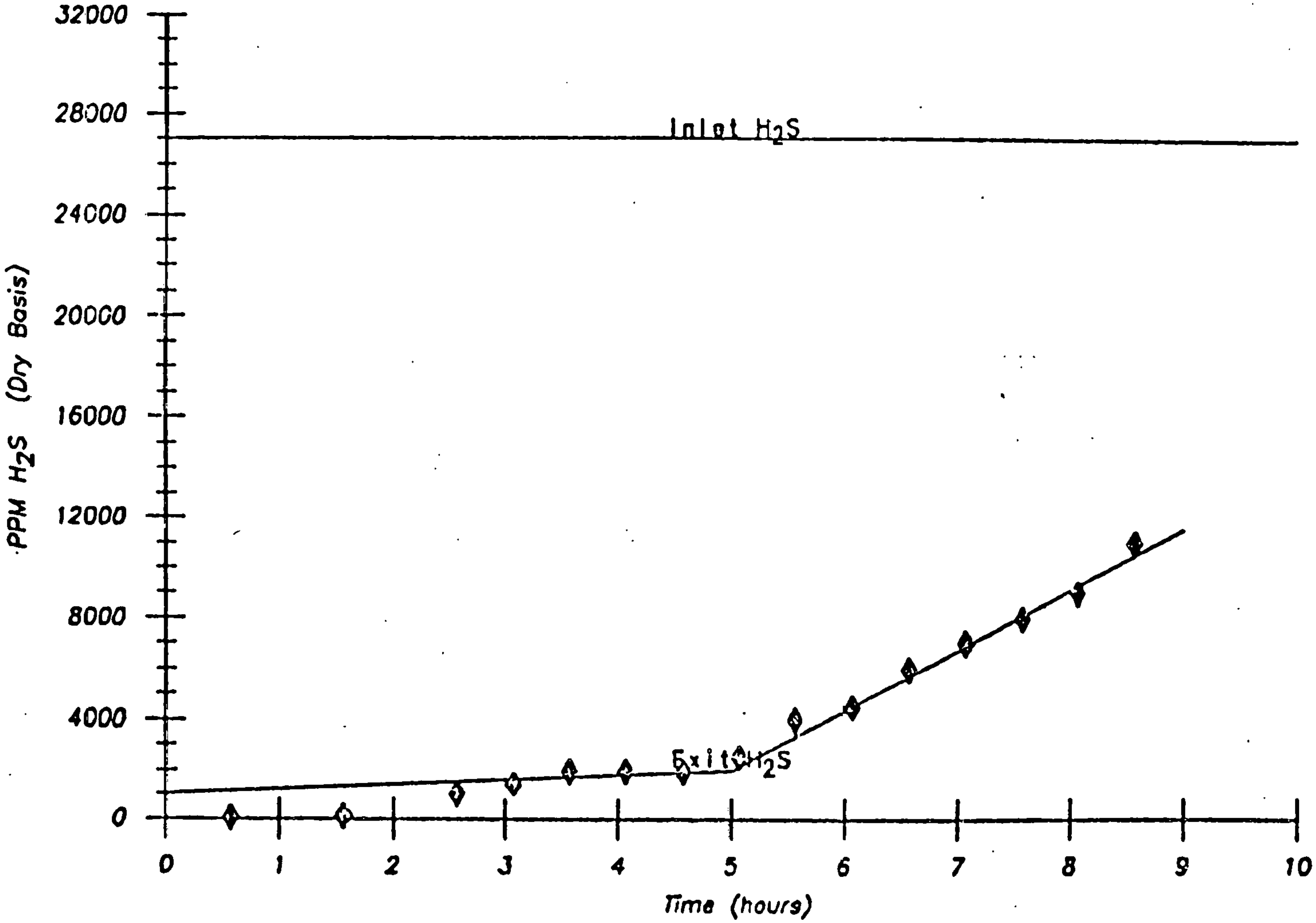


19-MAY-81 15:40:06

P:

IRON OXIDE SULFIDATION

TEST 003

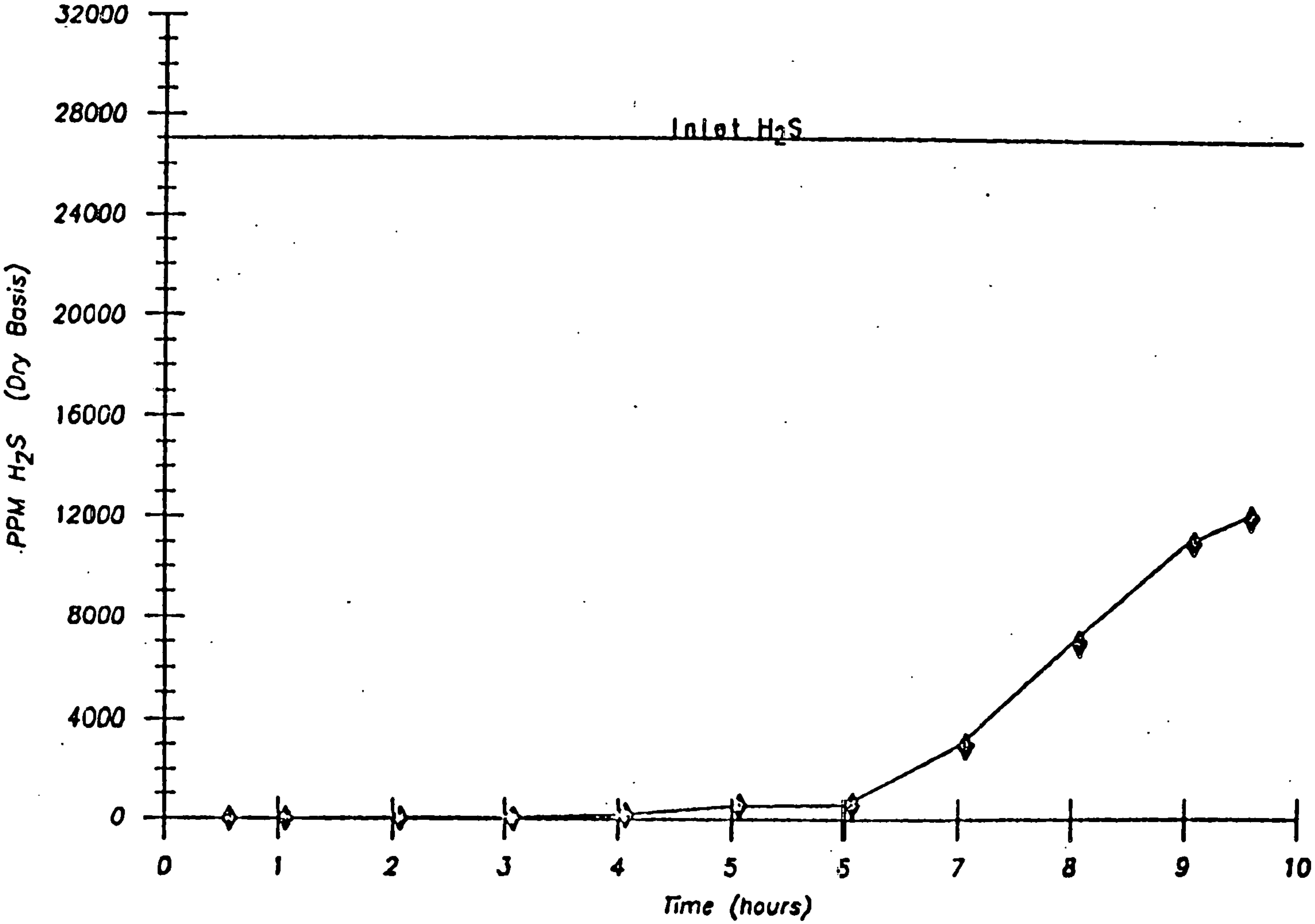


19–MAY–81 10:21:32

P1

IRON OXIDE SULFIDATION

TEST 005



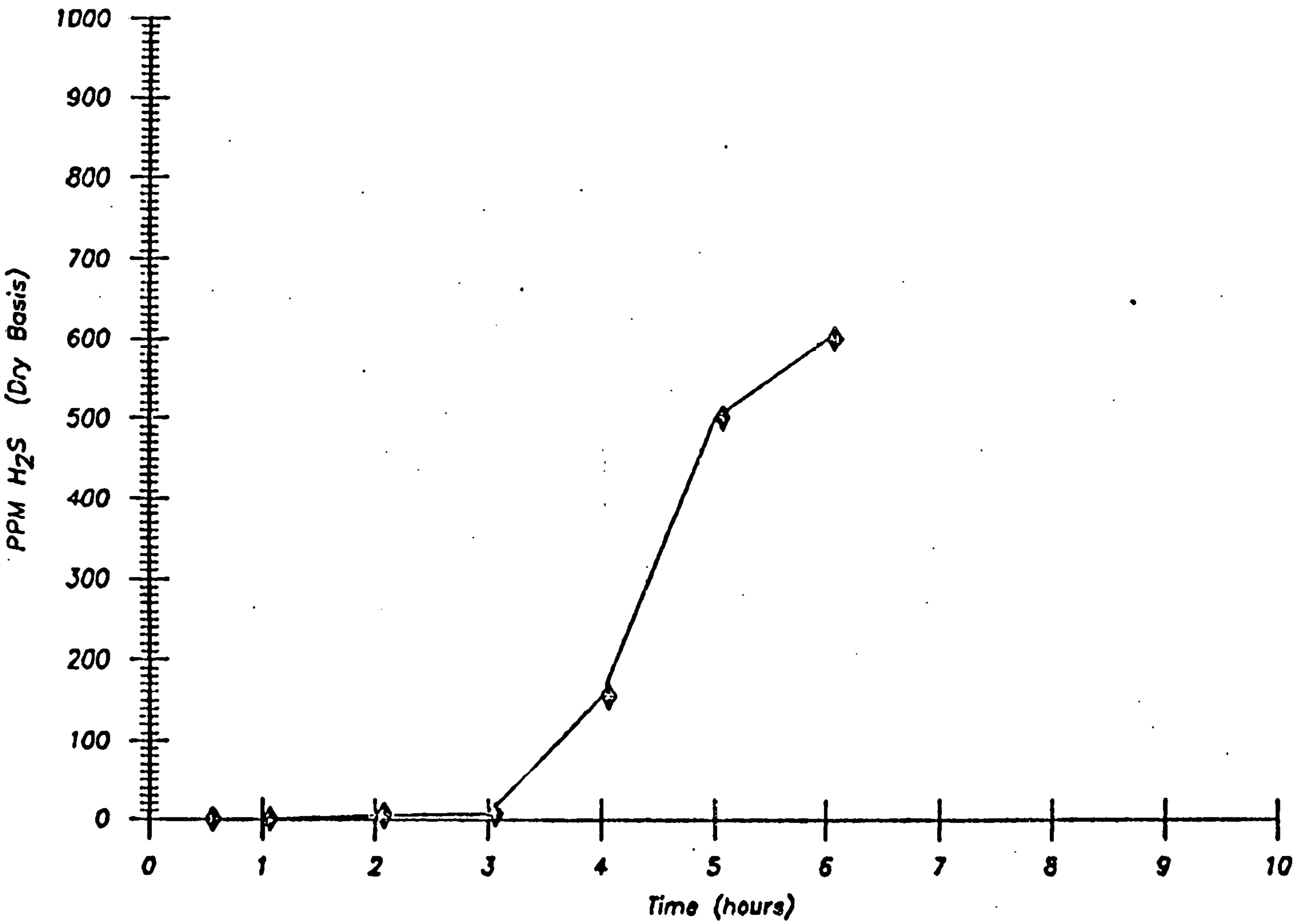
-224-

19-MAY-81 10:20:00

P:

IRON OXIDE SULFIDATION

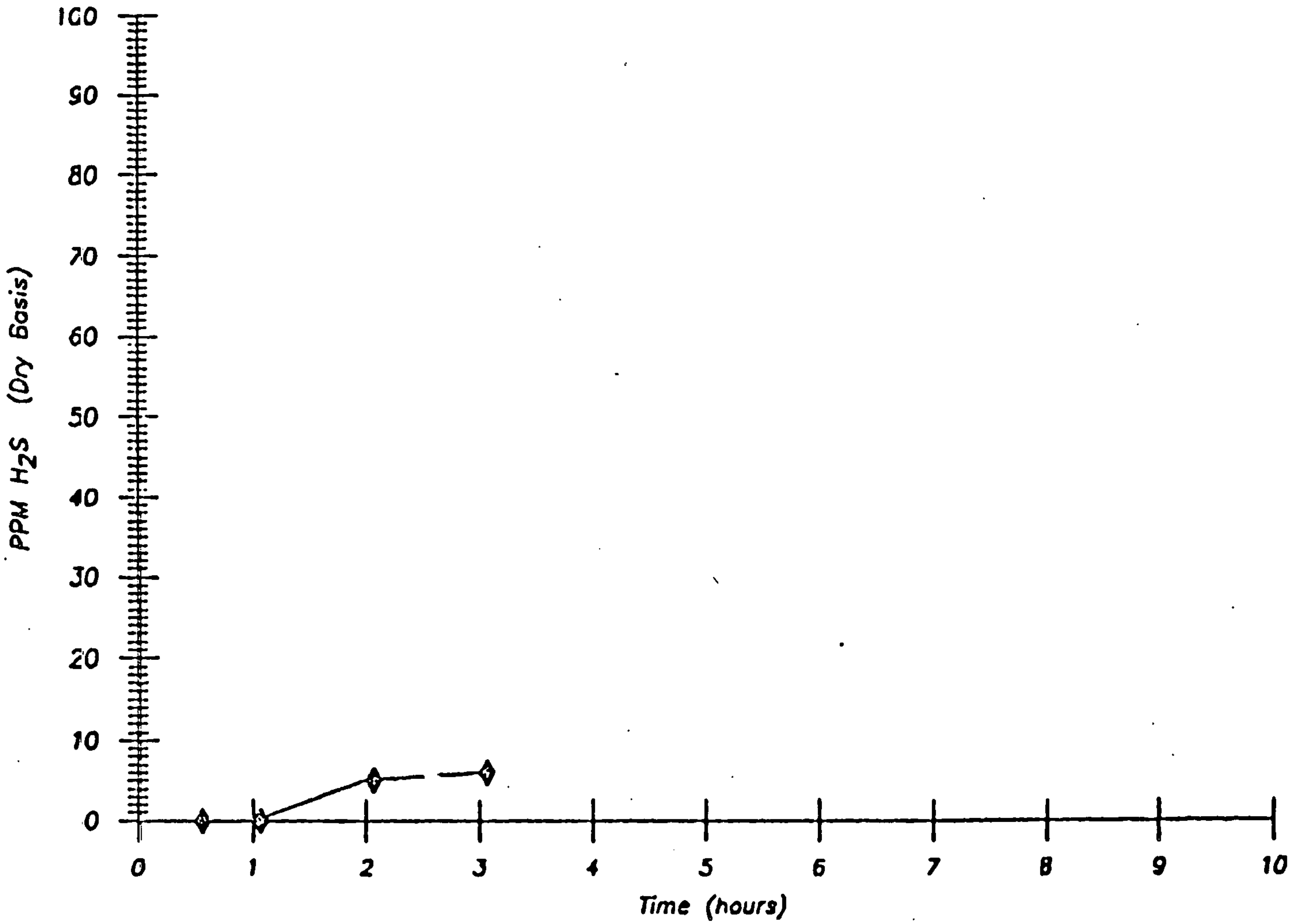
TEST 005



19-MAY-81 10:18:34

IRON OXIDE SULFIDATION

TEST 005

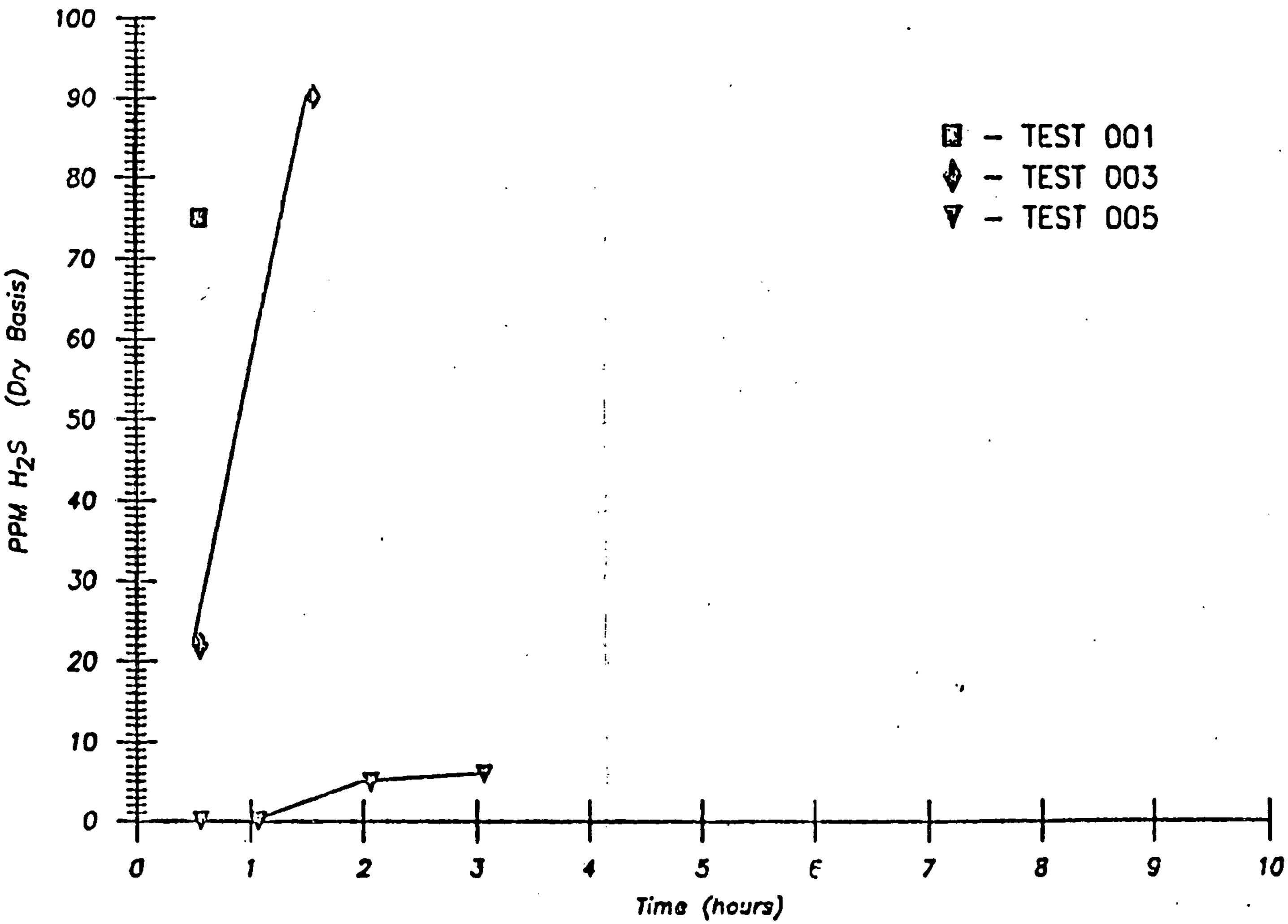


19-MAY-81 10:17:14

P1

IRON OXIDE SULFIDATION

TESTS 001,003,005

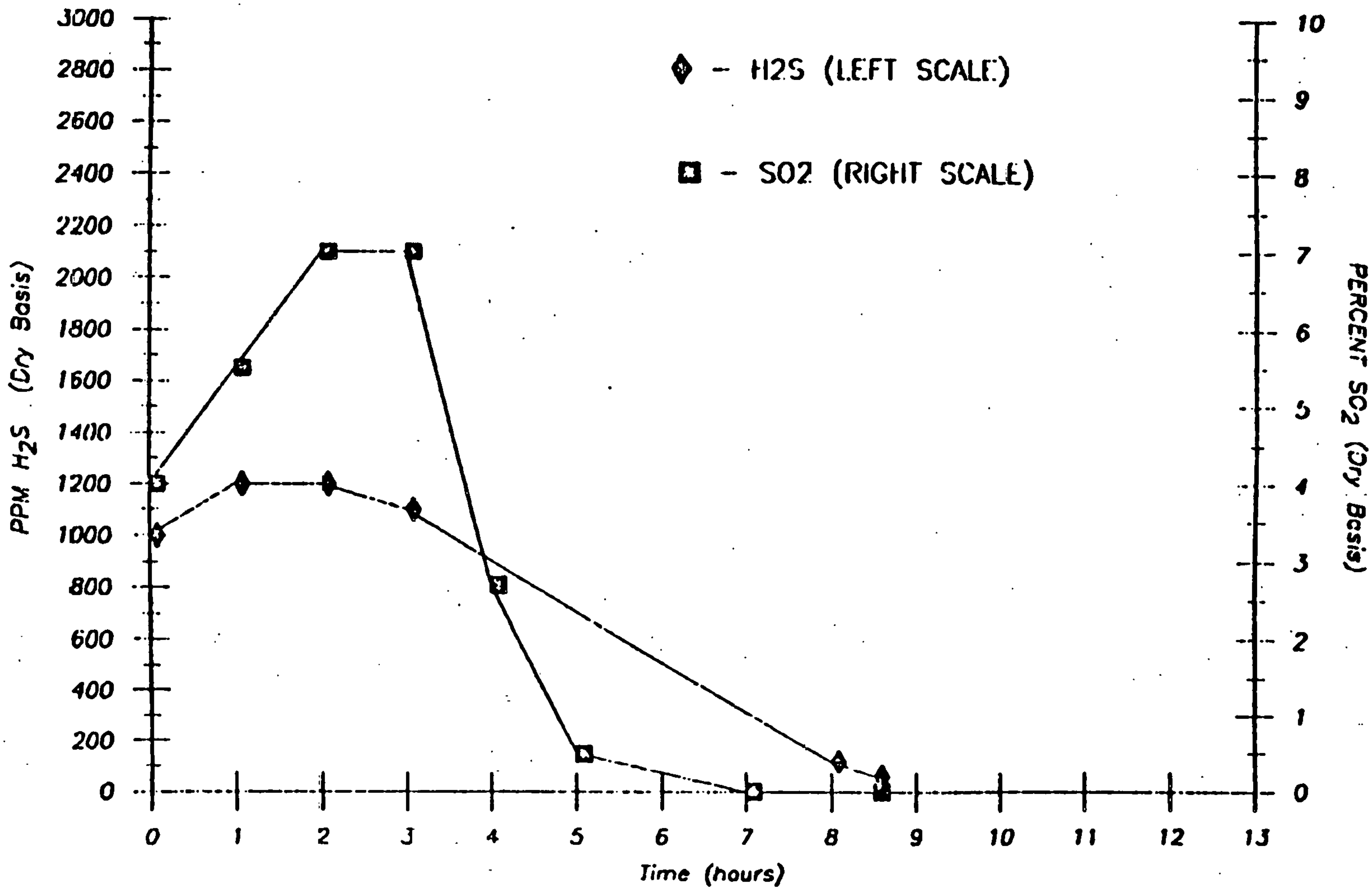


22-MAY-81 14:13:27

P1

IRON OXIDE REGENERATION

TEST 006



23-SEP-81 16:18:58

-229-

TEST SUMMARY SHEET

TEST NO.	:	007
DATE STARTED	:	1/30/81
DATE ENDED	:	1/30/81
TOTAL HOURS	:	12.5
TYPE	:	SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE SORPTION EFFICIENCY AND SUBSEQUENT REGENERATION CHARACTERISTICS.

SORBENT TYPE/WEIGHT:	(ZINC FERRITE)/300 G
SORBENT NO. :	BATCH 81/1
SORBENT COMPOSITION:	50 MOLE% IRON OXIDE (FISHER CERIFIED) 50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE :	1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT :	DRIED IN OVEN AT 200 F OVERNIGHT
SULFUR LOADING :	21.3 % (GM SULFUR/GM FRESH SORBENT) TO 10 PPM BREAKTHROUGH

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):	5.84	
DENSITY (G/CM3) :	5.19	
PORE VOLUME (MM3/G)		12.2
MINERAL ANALYSIS :	SEE APPENDIX	
ELEMENTAL ANALYSIS :		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

	FLOW RATE	MOLE
H2S (0.25 DM3/MIN)	0.529 SCFH	2.7%
H2O	0.513 CM3/MIN	6.9%
AIR		
CO2		
CO		
H2 (1.35 DM3/MIN)	2.86 SCFH	14.6%
CH4		
N2 (7.02 DM3/MIN)	14.87 SCFH	75.8%

DATA: (DETECTOR TUBE)

EXIT H2S: 2 PPM PLATEAU FOR 7 HRS
BREAKTHROUGH AT 8 HRS WITH 10 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. ABSORBER TEMPERATURES CONTROLLED WITHIN 50 F.
2. PREHEATER GRADUALLY PLUGGED DURING RUN .
3. ANALYSIS OF PLUG MATERIAL BY X-RAY DIFFRACTION SHOWS THAT THE PREHEATER TUBING (304 S/S) WAS CORRODING DUE TO H2S AND HIGH TEMP.

CONCLUSIONS

1. ZINC FERRITE AS TESTED CAN REDUCE A GAS STREAM CONTAINING 2.7% H2S TO 2 PPM H2S IN THE PRESENCE OF 6.9 % WATER AT 1000 F.
2. ZINC FERRITE AS TESTED CAN REDUCE A GAS STREAM CONTAINING 2.7% H2S TO LOWER LEVELS THAN POSSIBLE WITH IRON OXIDE AND WILL TAKE APPROXIMATELY TWICE AS LONG TO REACH BREAKTHROUGH.

TEST SUMMARY SHEET

TEST NO. : 008
DATE STARTED : 1/31/81
DATE ENDED : 2/1/81
TOTAL HOURS : 12.5
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 007 USING 50% AIR AND 50% STEAM.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/800 G
SORBENT NO. : BATCH 81/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT(BEFORE T007)
SULFUR REMOVED :

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		0.37 (AVG)
SURFACE AREA (M2/G):		2.65 (AVG)
DENSITY (G/CM3) :		5.22 (AVG)
PORE VOLUME (MM3/G):		7.0 (AVG)
MINERAL ANALYSIS:	SEE APPENDIX	
ELEMENTAL ANALYSIS:	SEE APPENDIX	

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 2000 PPM

EXIT SO2: 4-9 %

EXIT S2: 7.88 G

EXIT H2:

	FLOW RATE		MOLE
H2S			
H2O	2.23	CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88	SCFH	50%
CO2			
CO			
H2			
CH4			
N2			
$			

REMARKS

1. TEMPERATURE WAVE UP TO 1780 F OBSERVED.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. REGENERATION TIME APPROXIMATELY THE SAME AS FOR IRON OXIDE.

TEST SUMMARY SHEET

TEST NO. : 009
DATE STARTED : 2/03/81
DATE ENDED : 2/03/81
TOTAL HOURS : 12.75
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE SORPTION EFFICIENCY AND SUBSEQUENT REGENERATION CHARACTERISTICS. THIS IS THE SECOND SULFIDATION CYCLE OF THIS BATCH OF SORBENT.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/800 G
SORBENT NO. : BATCH 81/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT(BEFORE T007)
SULFUR LOADING : 18.7 % (GM SULFUR/GM FRESH SORBENT)
TO 13 PPM BREAKTHROUGH

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR(%W/W):		
SURFACE AREA(M2/G):		
DENSITY(G/CM3) :		
PORE VOLUME (MM3/G):		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

	FLOW RATE	MOLE
H2S (0.25 DM3/MIN)	0.529 SCFH	2.7%
H2O	0.513 CM3/MIN	6.9%
AIR		
C' '		
CO		
H2 (1.35 DM3/MIN)	2.86 SCFH	14.6%
CH4		
N2 (7.02 DM3/MIN)	14.87 SCFH	75.8%

DATA: (DETECTOR TUBE)

EXIT H2S: 2-4 PPM PLATEAU FOR 6 HOURS
BREAKTHROUGH AT 7 HOURS WITH 13 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. PREHEATER TUBE WAS REPLACED PRIOR TO THIS RUN WITH ANOTHER 304 S/S TUBE.

CONCLUSIONS

1. DESULFURIZATION WAS ACHIEVED TO THE SAME LEVEL AS DURING THE FIRST CYCLE ON THIS SORBENT.
2. BREAKTHROUGH OCCURRED ONE HOUR SOONER DURING THE SECOND CYCLE.

TEST SUMMARY SHEET

TEST NO. : 010
DATE STARTED : 2/3/81
DATE ENDED : 2/3/81
TOTAL HOURS : 12.5
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 009 USING 50% AIR AND 50% STEAM.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/800 G
SORBENT NO. : BATCH #1/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT (BEFORE T007)
SULFUR REMOVED :

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR(%W/W):		
SURFACE AREA(M2/G):		
DENSITY (G/CM3):		
PORE VOLUME(MM3/G):		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

8

DATA: (DETECTOR TUBE)

EXIT H2S: 2000-3000 PPM

EXIT SO2: 4-10 %

EXIT S2: 4.4796 G

EXIT H2:

REMARKS

1. TEMPERATURE WAVE UP TO 1730 F OBSERVED.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. THIS WAS THE SECOND REGENERATION OF THE SAME BATCH OF SORBENT. REGENERATION APPEARS NORMAL.

2. WIDE SCATTER IN SO2 DATA DURING REGENERATION

TEST SUMMARY SHEET

TEST NO. : 011A
DATE STARTED : 2/05/81
DATE ENDED : 2/05/81
TOTAL HOURS : 0.5
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE SORPTION EFFICIENCY AND SUBSEQUENT REGENERATION CHARACTERISTICS. THIS IS THE THIRD SULFIDATIOF CYCLE OF THIS BATCH OF SORBENT.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/800 G
SORBENT NO. : BATCH 81/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT(BEFORE T007)
SULFUR LOADING :

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR(%W/W):		
SURFACE AREA(M2/G):		
DENSITY(G/CM3):		
PORE VOLUME(MM3/G):		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE
H2S (0.25 DM3/MIN)	0.529 SCFH	2.7%
H2O	0.513 CM3/MIN	6.9%
AIR		
CO2		
CO		
H2 (1.35 DM3/MIN)	2.86 SCFH	14.6%
CH4		
N2 (7.02 DM3/MIN)	14.87 SCFH	75.8%

REMARKS

1. DURING HEAT UP FOR THIS RUN THE ABSORBER WAS INADVERTENTLY EXPOSED TO APPROXIMATELY 30% H2S FOR 30 MINUTES. THIS OCCURRED AT APPROX. 1000 F.

2. THE RUN WAS ABORTED AFTER IT WAS DETERMINED THAT THE H2S HAS BROKENTHROUGH, AND REGENERATION INITIATED.

CONCLUSIONS

1.

TEST SUMMARY SHEET

TEST NO. : 011B
DATE STARTED : 2/06/81
DATE ENDED : 2/06/81
TOTAL HOURS : 12.75
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/800 G
SORBENT NO. : BATCH #1/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT(BEFORE T007)
SULFUR LOADING : 21.38 % (GM SULFUR/GM FRESH SORBENT)
TO 11 PPM BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

	FLOW RATE		MOLE
H2S	(0.25 DM3/MIN)	0.529 SCFH	2.7%
H2O		0.513 CM3/MIN	6.9%
AIR			
CO2			
CO			
H2	(1.35 DM3/MIN)	2.86 SCFH	14.6%
CH4			
N2	(7.02 DM3/MIN)	14.87 SCFH	75.8%

8

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE SORPTION EFFICIENCY AND SUBSEQUENT REGENERATION CHARACTERISTICS. THIS IS THE THIRD SULFIDATION CYCLE OF THIS BATCH OF SORBENT.

ANALYSIS:

	BEFORE	AFTER	
TOTAL SULFUR (%W/W):		26.42	(AVG)
SURFACE AREA (M2/G):		1.4	(AVG)
DENSITY (G/CM3):		4.42	(AVG)
PORE VOLUME (MM3/G):		2.9	(AVG)
MINERAL ANALYSIS:	SEE APPENDIX		
ELEMENTAL ANALYSIS:	SEE APPENDIX		

DATA: (DETECTOR TUBE)

EXIT H2S: 2-6 PPM PLATEAU FOR 7 HOURS

BREAKTHROUGH AT 8 HOURS WITH 11 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. PRIOR TO THIS RUN (RUN 011A), THE SORBENT WAS INADVERTENTLY EXPOSED TO APPROXIMATELY 30% H2S FOR 30 MINUTES AND THEN IMMEDIATELY REGENERATED.

2. AFTER REGENERATION THE SORBENT SEEMS TO HAVE RECOVERED ITS ACTIVITY AS SEEN BY THE TEST DATA.

CONCLUSIONS

1. SORBENT DESULFURIZATION ACTIVITY APPEARS TO DUPLICATE THE ACTIVITY OF RUN 007 (FIRST CYCLE) AFTER A SLIGHT DECLINE IN ACTIVITY IN RUN 009 (SECOND CYCLE).

2. ZINC FERRITE APPEARS TO BE A GOOD REGENERABLE SORBENT FOR H2S AT 1000 F. THIS TEST CONCLUDES THE THIRD SULFIDATION IN THIS SERIES.

TEST SUMMARY SHEET

TEST NO. : 012A
DATE STARTED : 2/5/81
DATE ENDED : 2/5/81
TOTAL HOURS : 12.5
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 011A USING 50% AIR AND 50% STEAM. THIS REGENERATION IS CARRIED OUT TO REMOVE THE HIGH LEVEL OF H2S INADVERTENTLY ABSORBED IN RUN 011A.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/800 G
SORBENT NO. : BATCH 81/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 1/4 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT (BEFORE T007)
SULFUR REMOVED : 32.9 % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR(%W/W):		
SURFACE AREA(M2/G):		
DENSITY(G/CM3):		
PORE VOLUME(MM3/G) :		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F(538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

DATA: (DETECTOR TUBE)

EXIT H2S: 2000-3000 PPM

EXIT SO2: 4-10 %

EXIT S2:

EXIT H2:

REMARKS

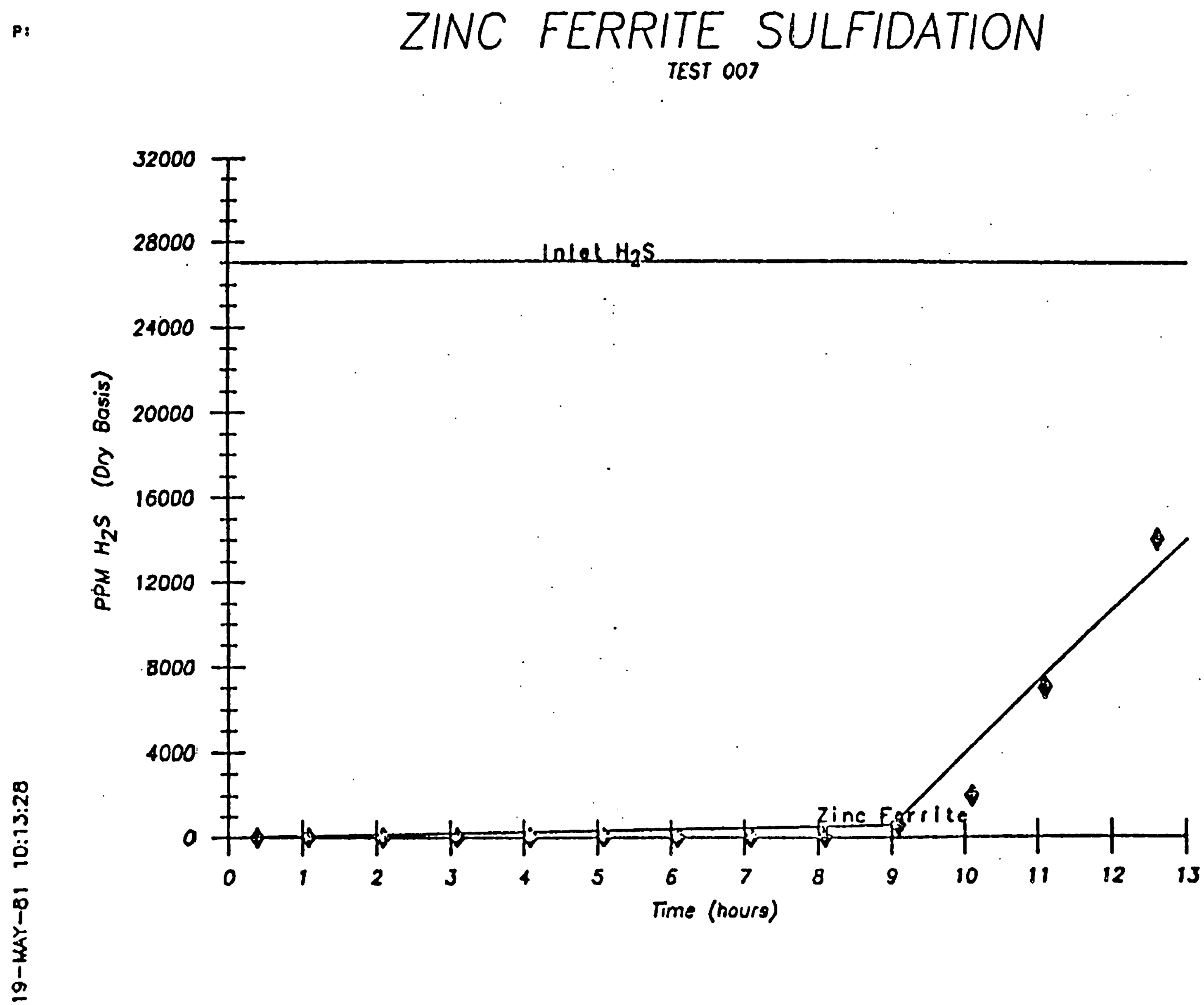
1. TEMPERATURE WAVE UP TO 1730 F OBSERVED.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. THIS WAS THE SECOND REGENERATION OF THE SAME BATCH OF SORBENT. REGENERATION APPEARS NORMAL.

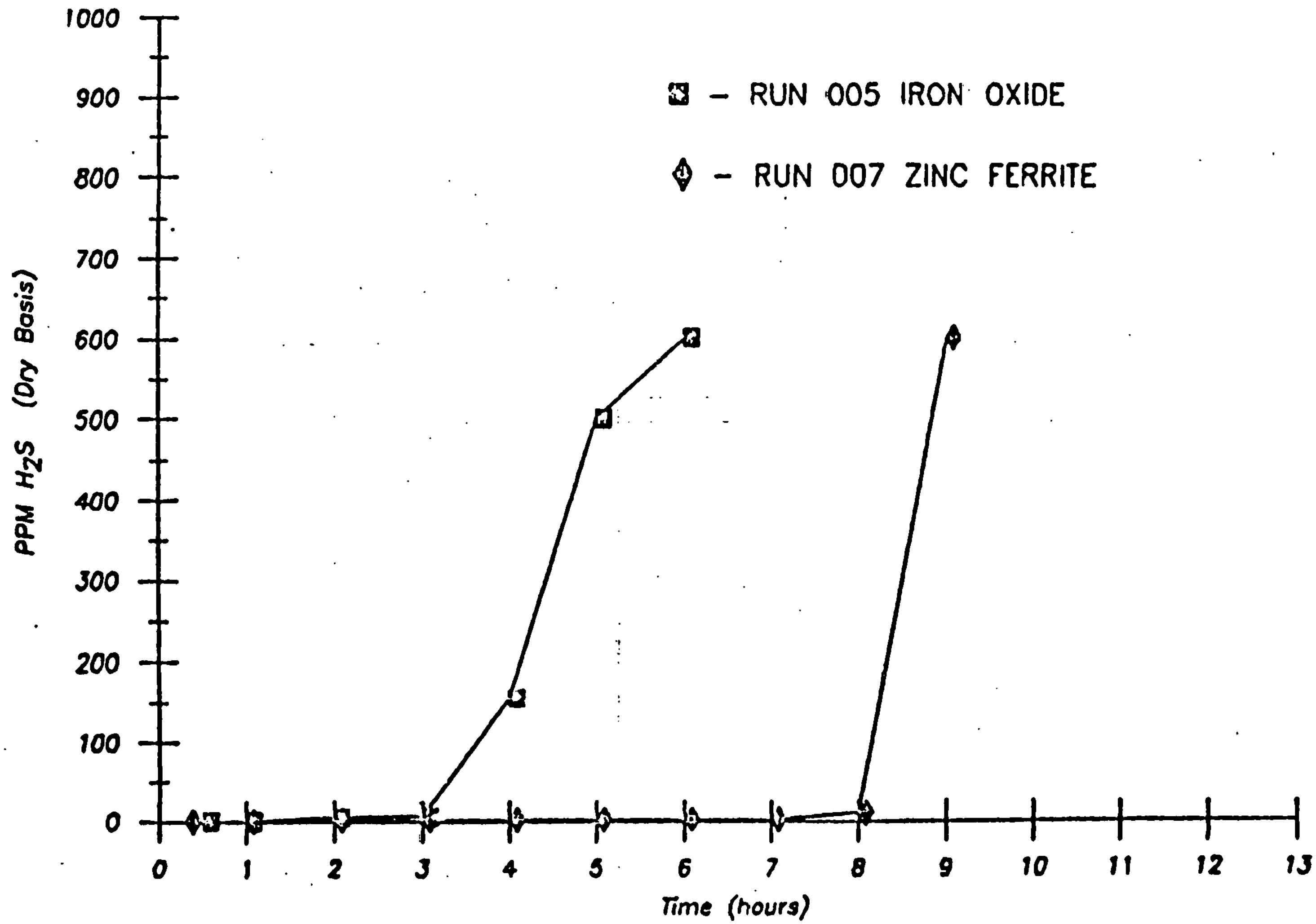
2. WIDE SCATTER IN SO2 DATA DURING REGENERATION POSSIBLY DUE TO H2S INTERFERENCE IN DETECTOR TUBE READING.


ZINC FERRITE SULFIDATION
TEST 007
PPM H_2S (Dry Basis)
32000
28000
24000
20000
16000
12000
8000
4000
0
Inlet H_2S
Zinc Ferrite
0 1 2 3 4 5 6 7 8 9 10 11 12 13
Time (hours)
19-MAY-81 10:13:28

P:

ZINC FERRITE SULFIDATION

TEST 007

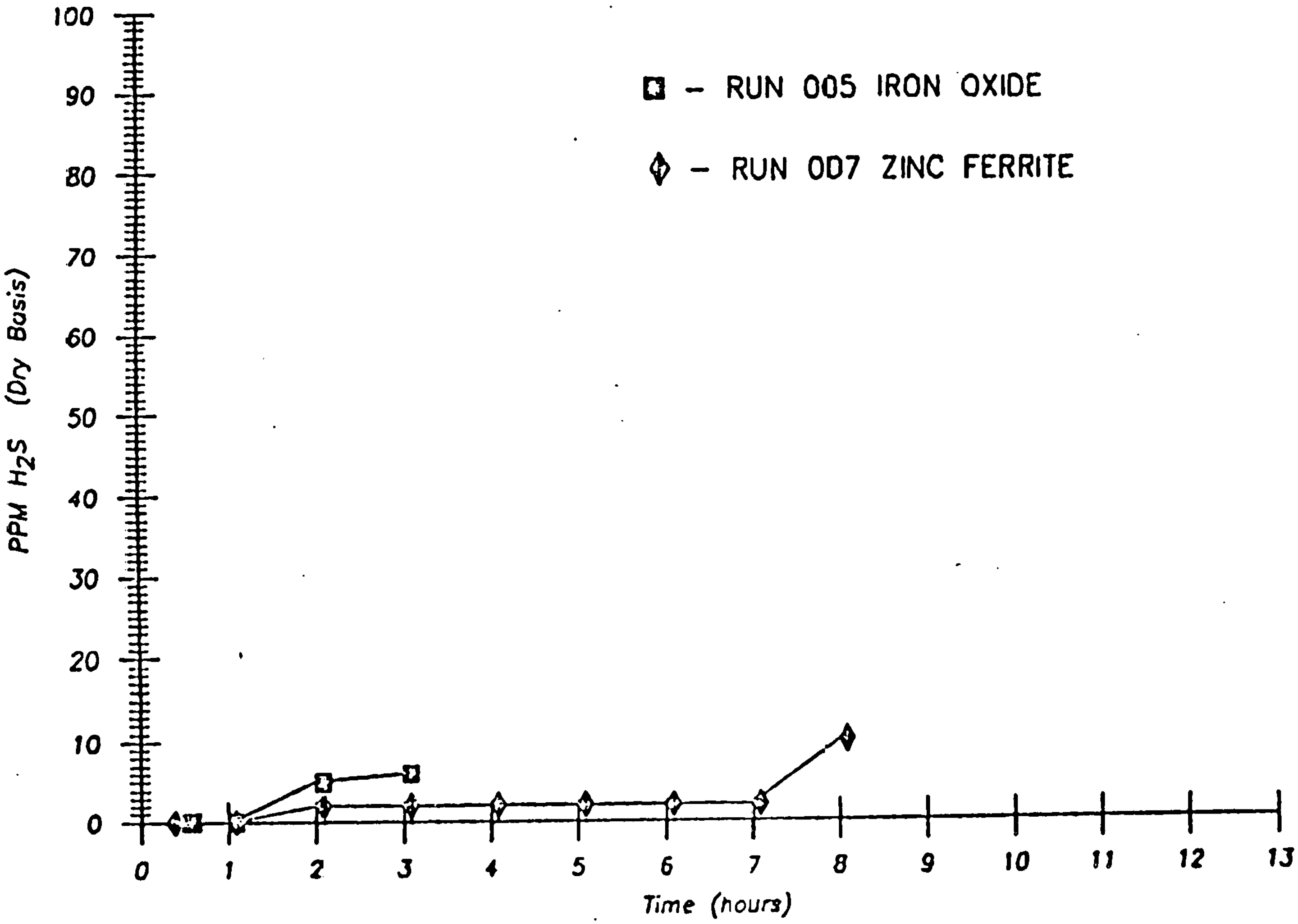


19–MAY–81 10:11:13

P1

ZINC FERRITE SULFIDATION

TEST 007

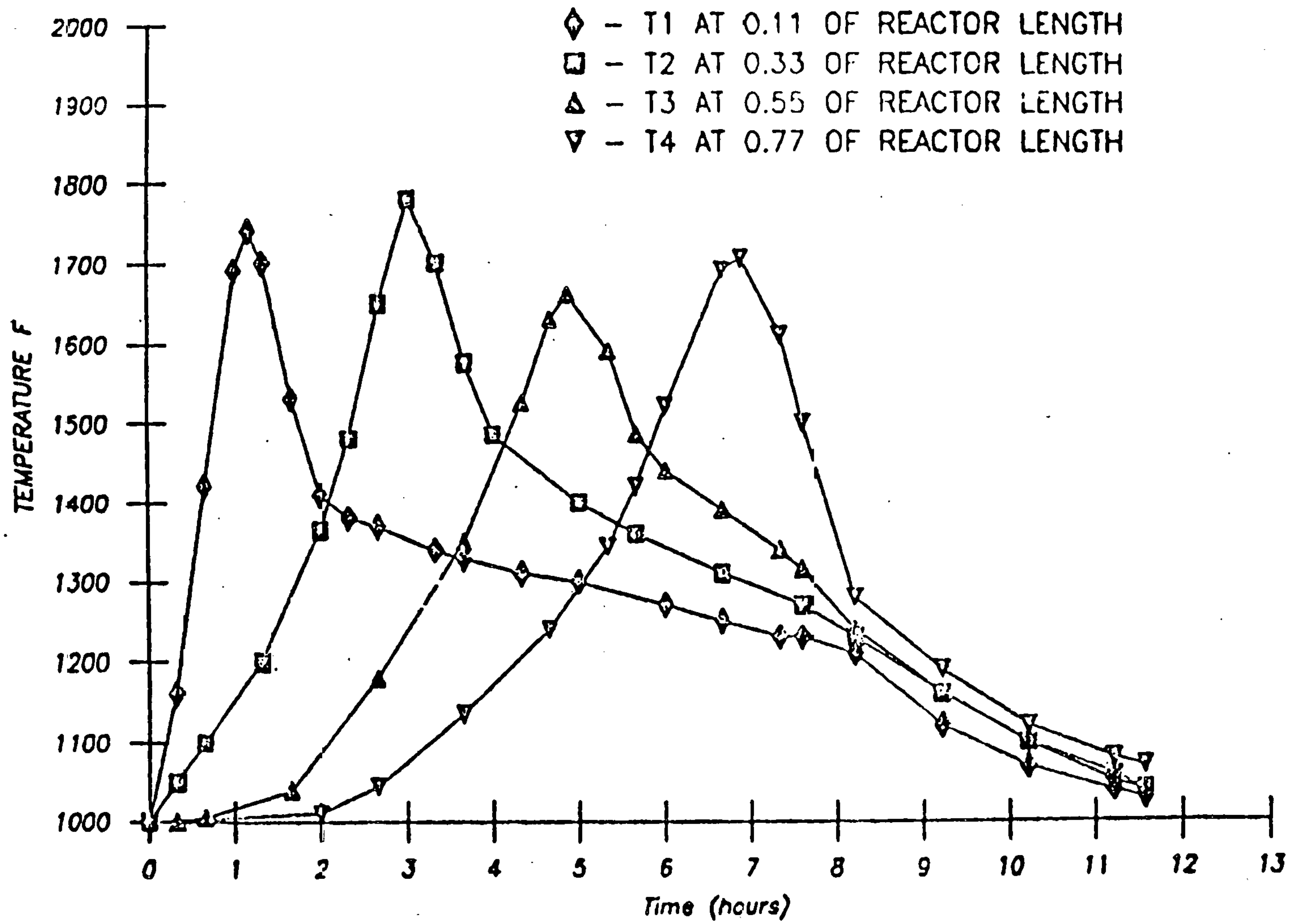


19-MAY-81 10:09:30

P:

ZINC FERRITE REGENERATION

TEST 008

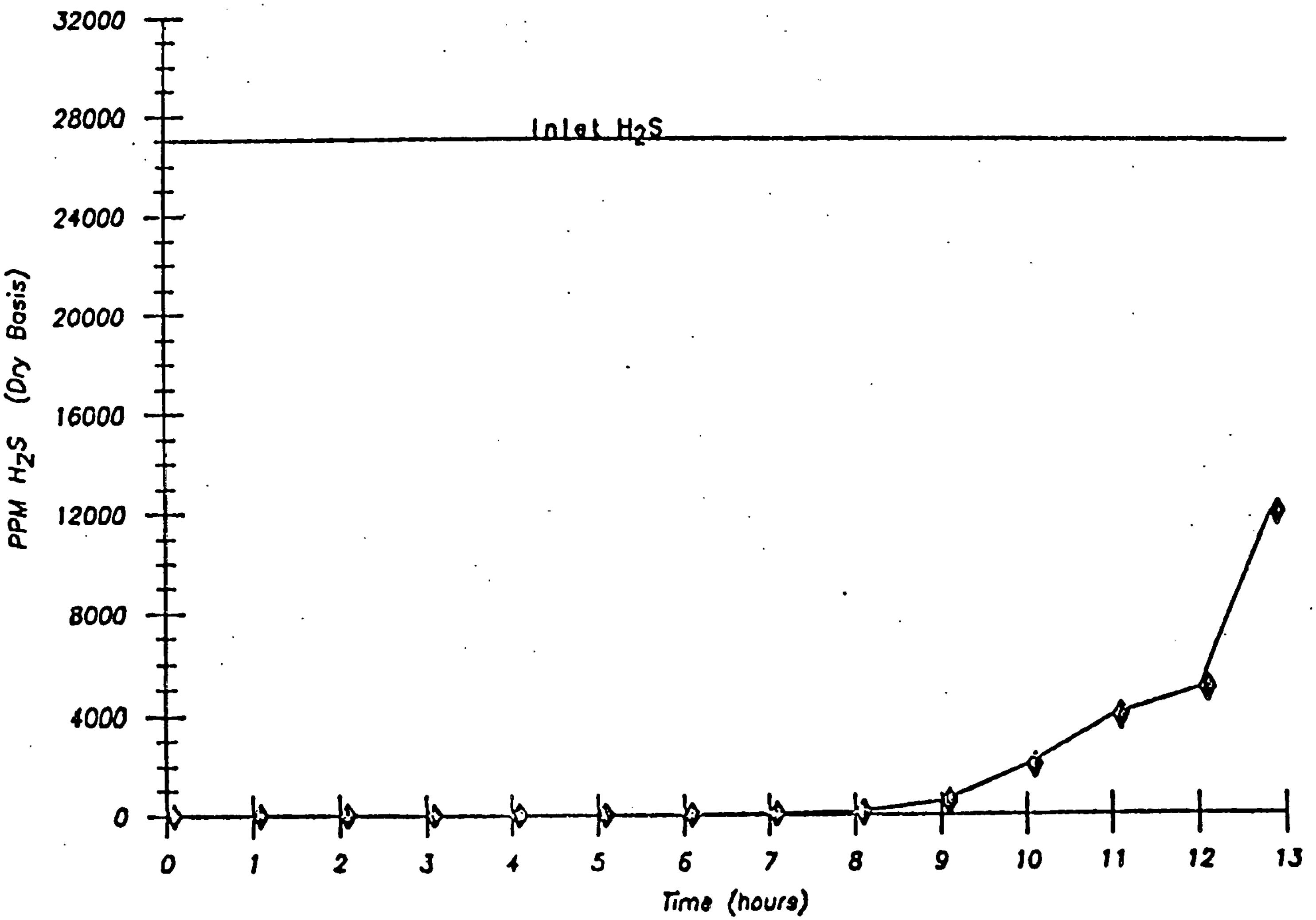


19-MAY-81 10:02:17

P1

ZINC FERRITE SULFIDATION

TEST 009 (SECOND CYCLE ON SAME SORBENT)

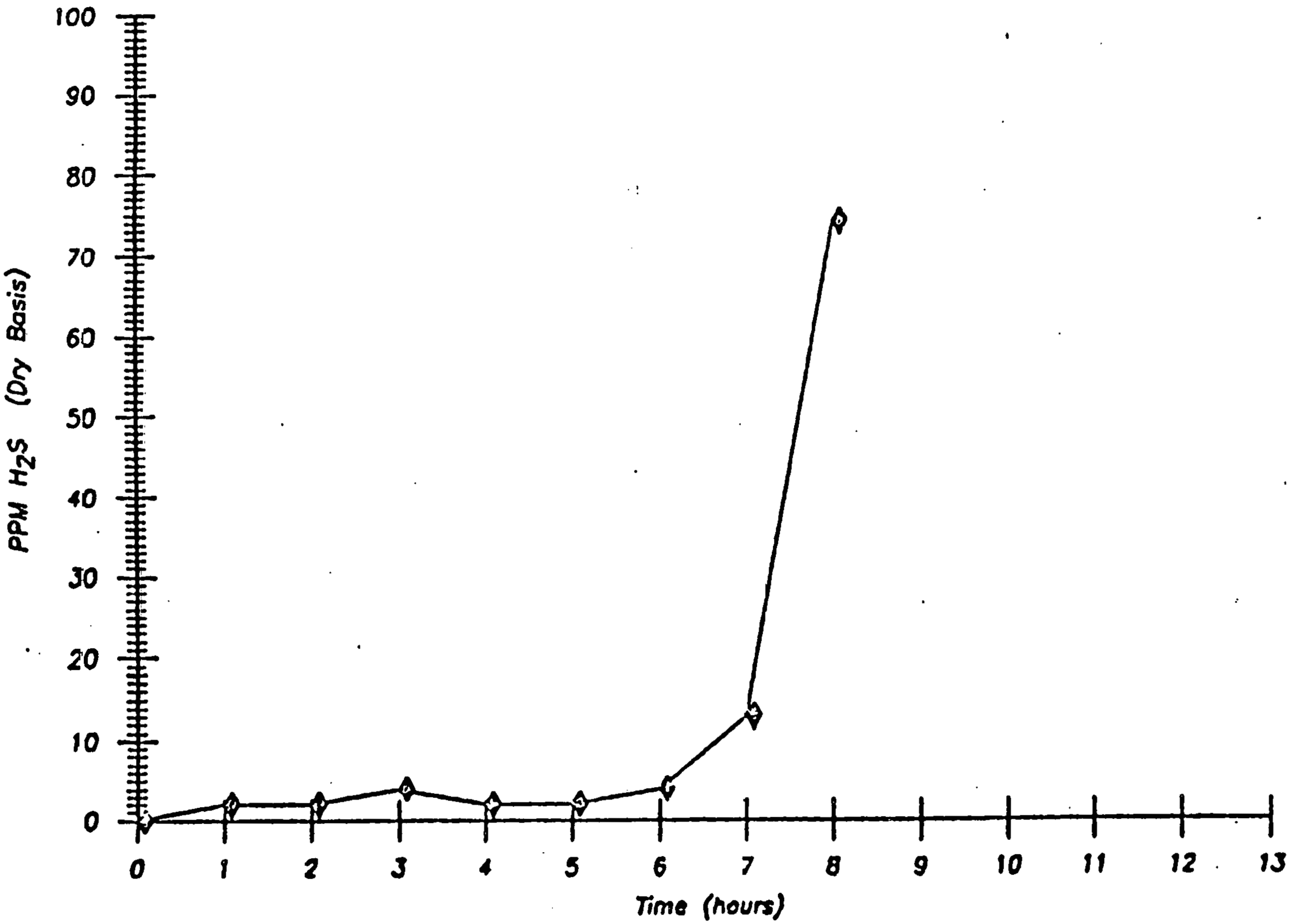


19-MAY-81 10:05:22

P1

ZINC FERRITE SULFIDATION

TEST 009 (SECOND CYCLE ON SAME SORBENT)

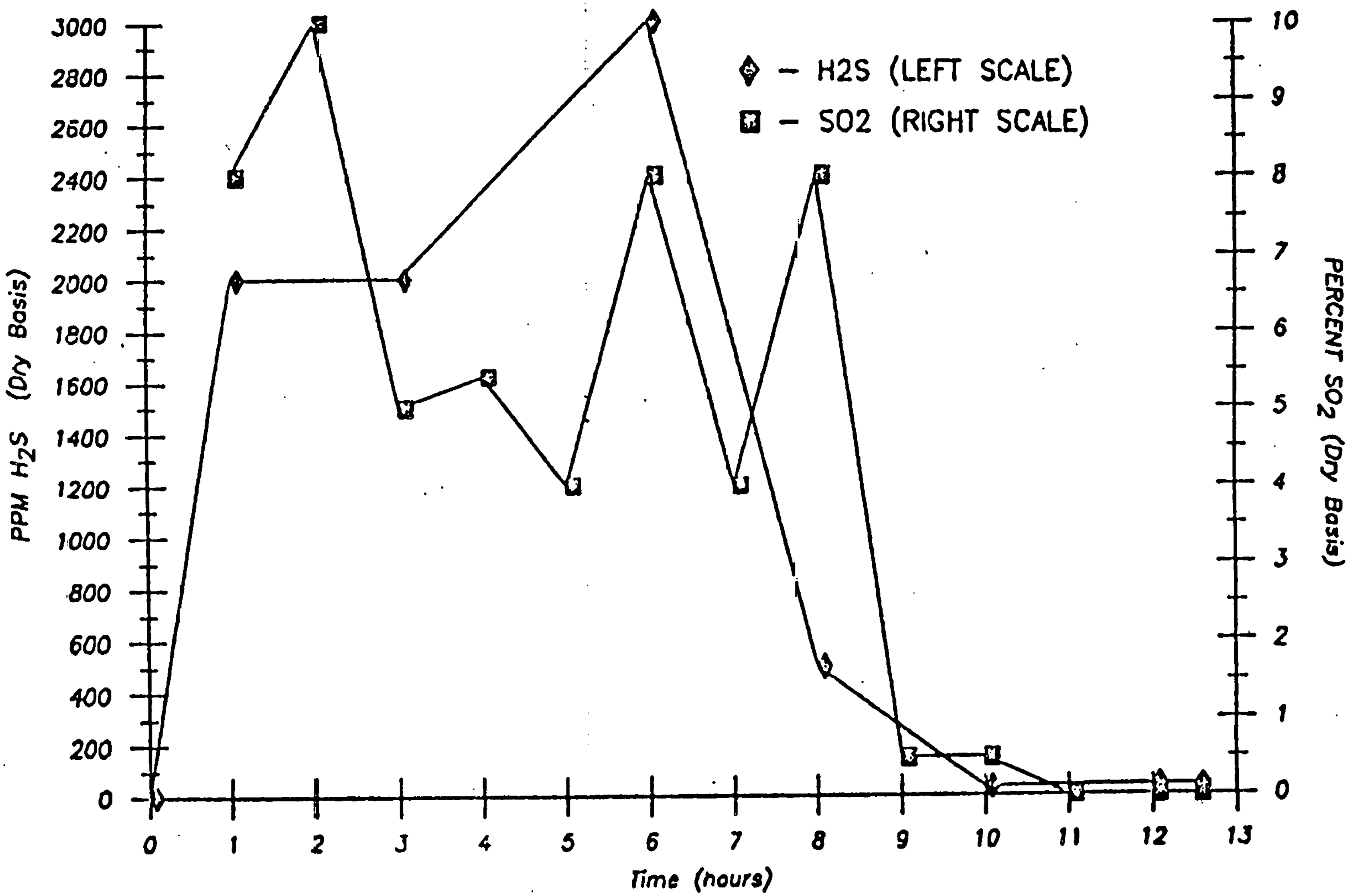


19-MAY-81 09:57:37

P1

ZINC FERRITE REGENERATION

TEST 010 (SECOND CYCLE ON SAME SORBENT)

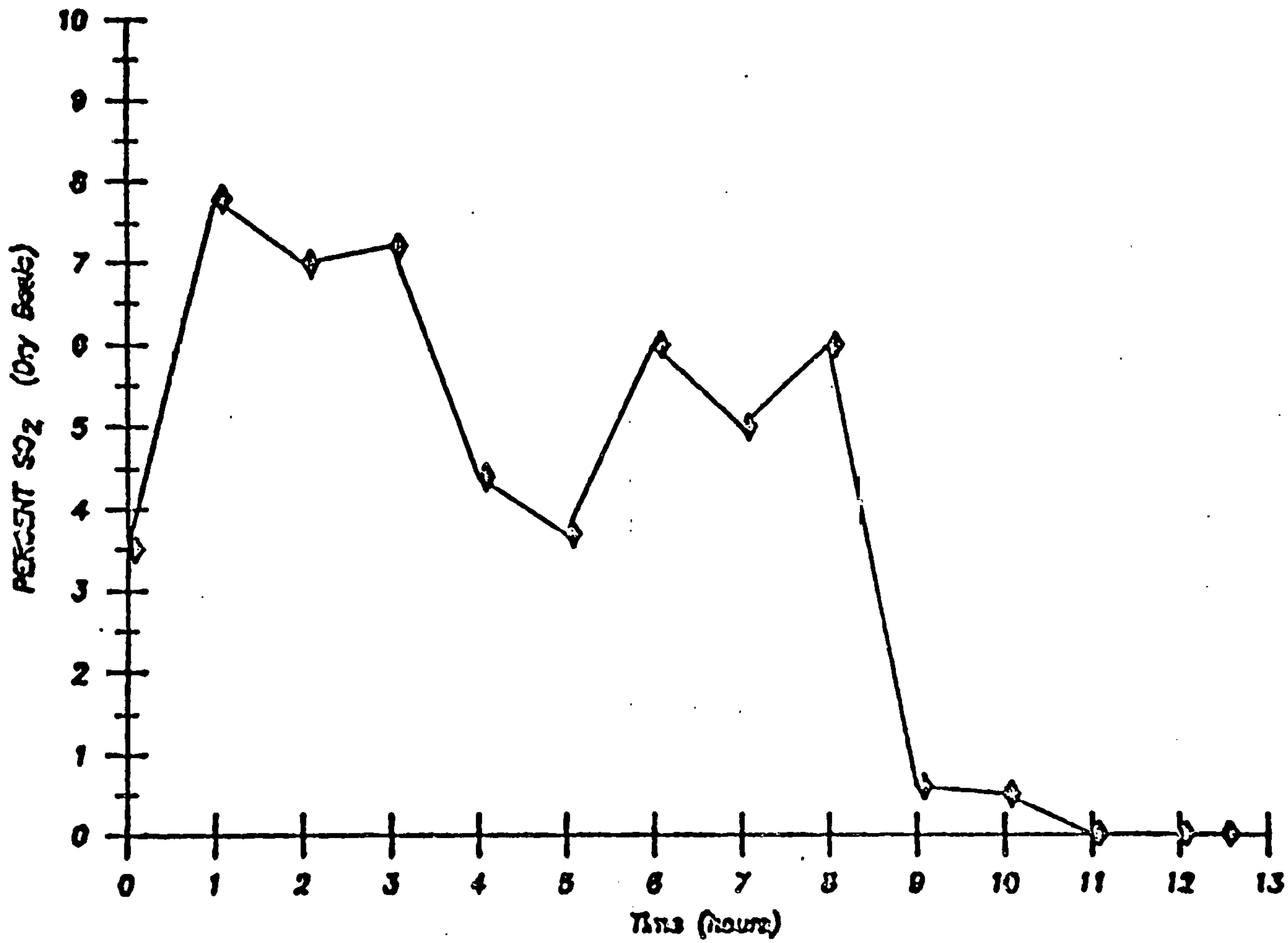


19-MAY-81 09:55:24

P:

ZINC FERRITE REGENERATION

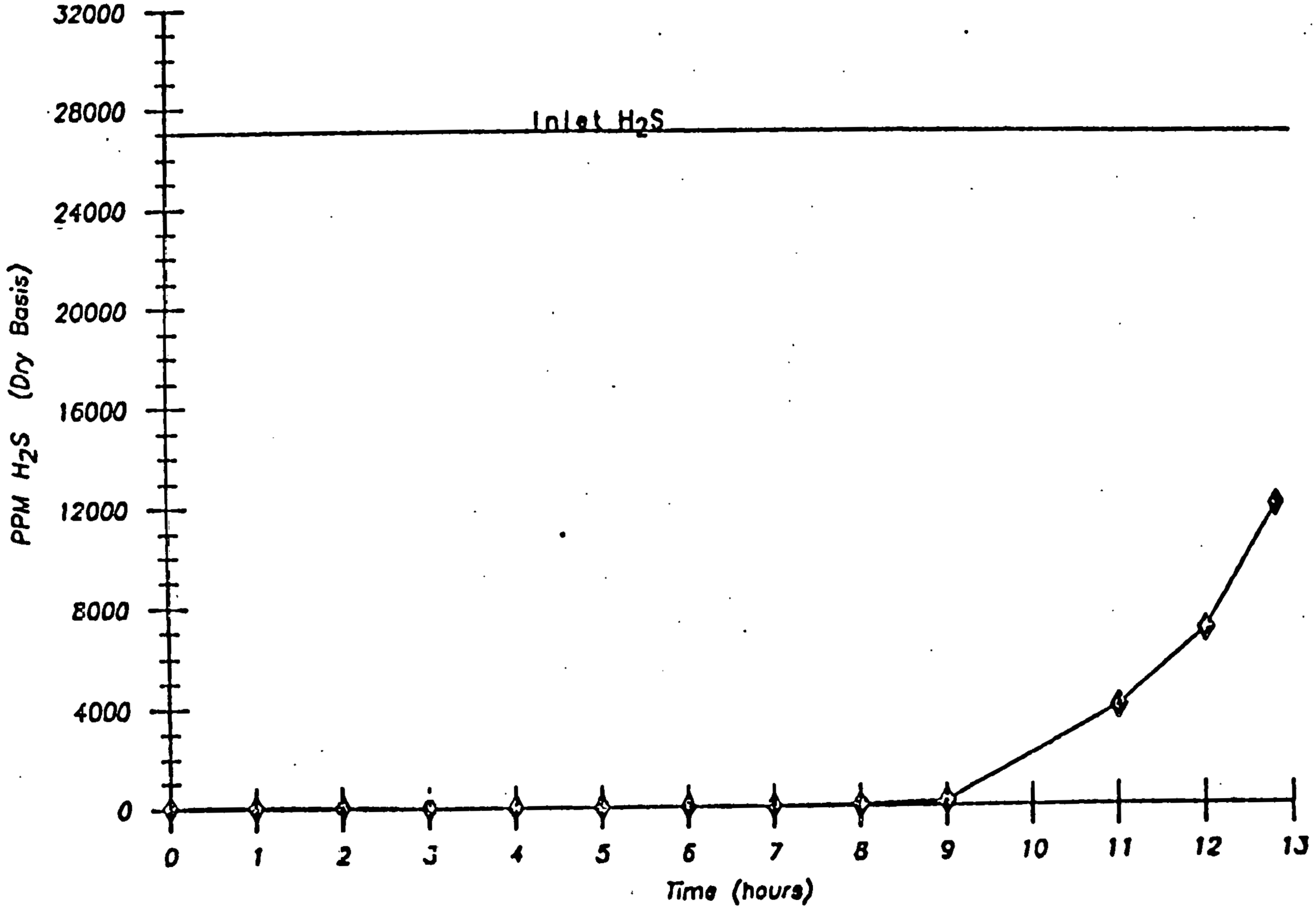
AVERAGE OF TESTS 009 AND 010



P:

ZINC FERRITE SULFIDATION

TEST 011B (THIRD CYCLE ON SAME SORBENT)

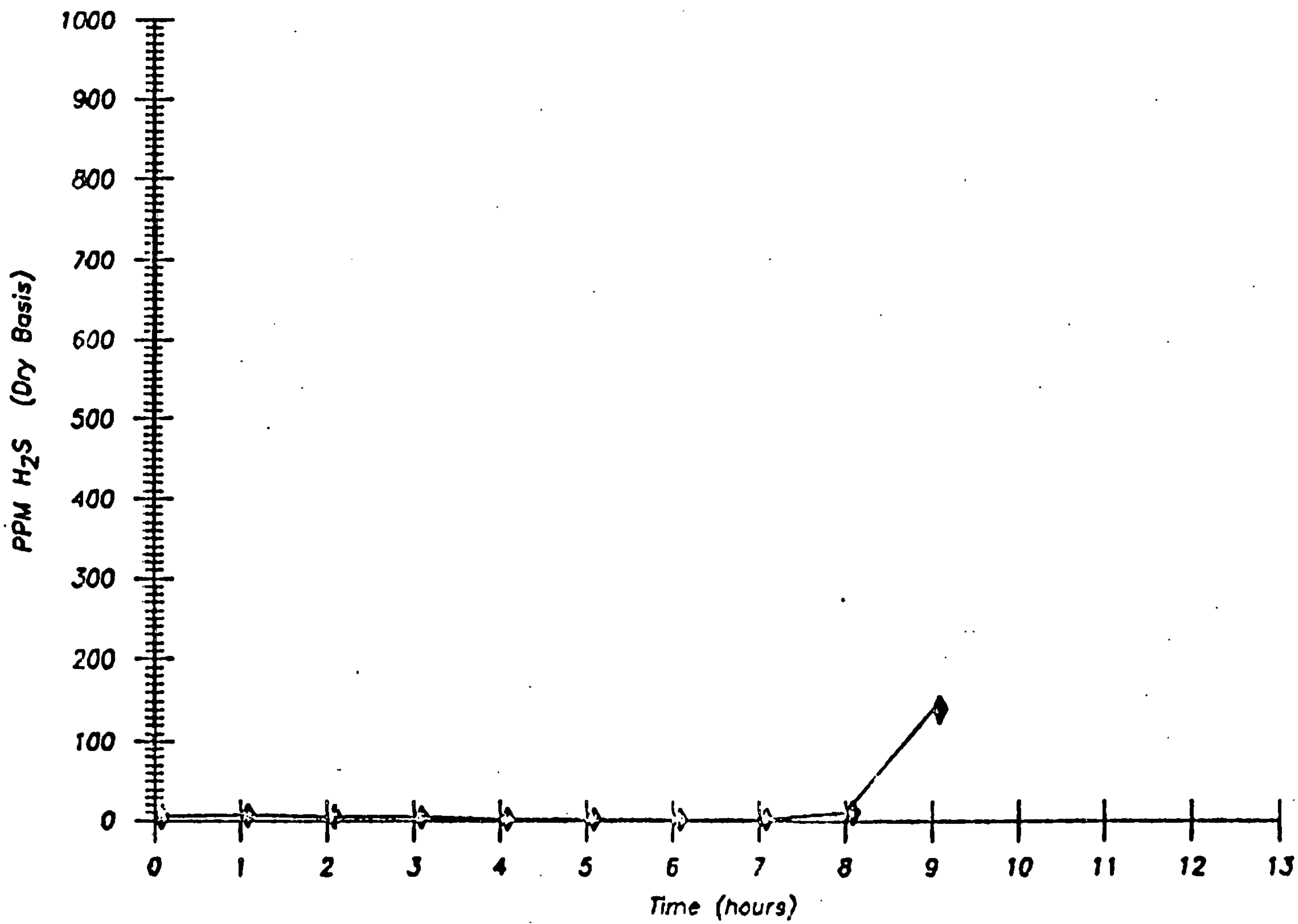


19-MAY-81 09:53:47

P1

ZINC FERRITE SULFIDATION

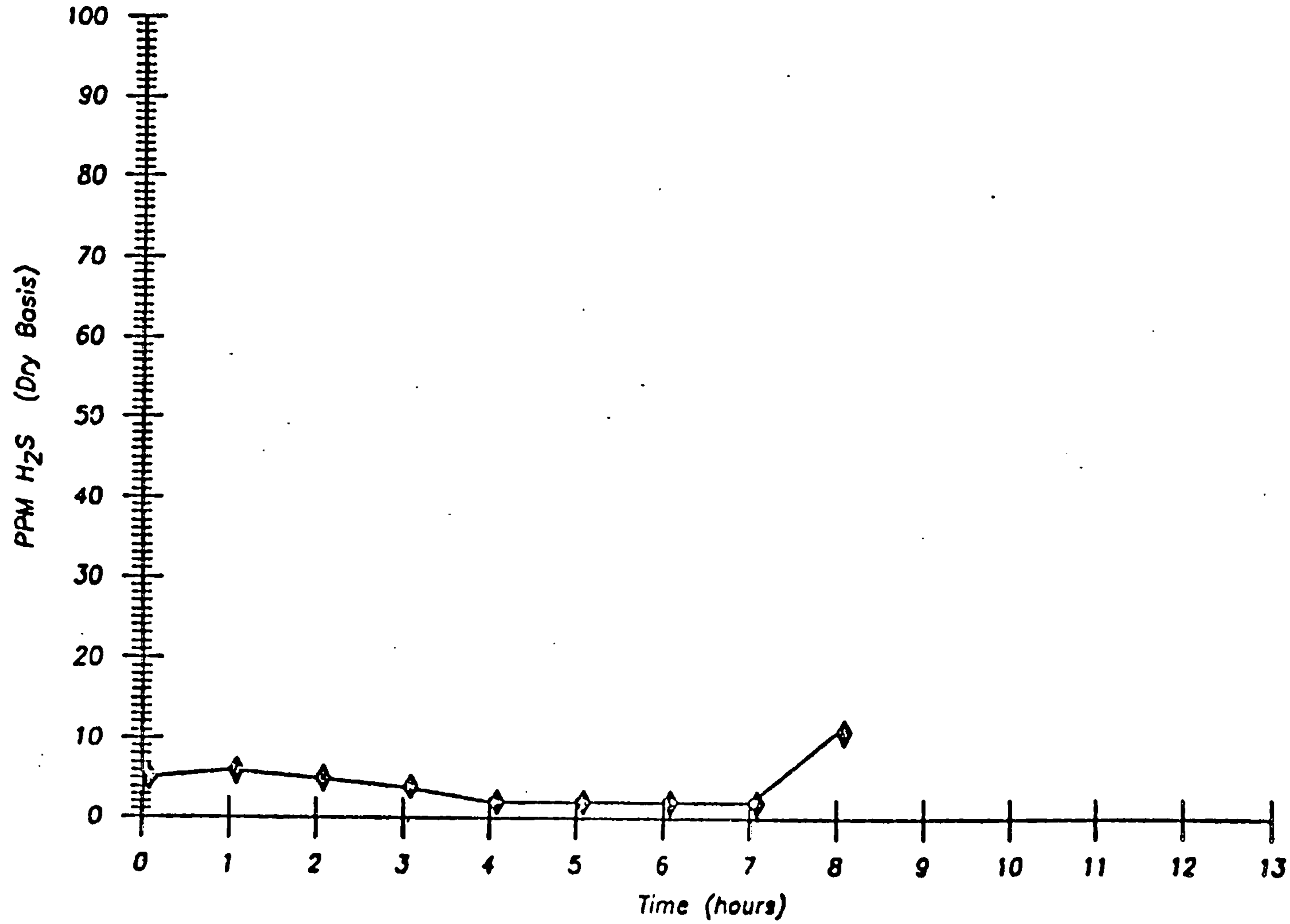
TEST 0118 (THIRD CYCLE ON SAME SORBENT)



19-MAY-81 09:52:13

ZINC FERRITE SULFIDATION

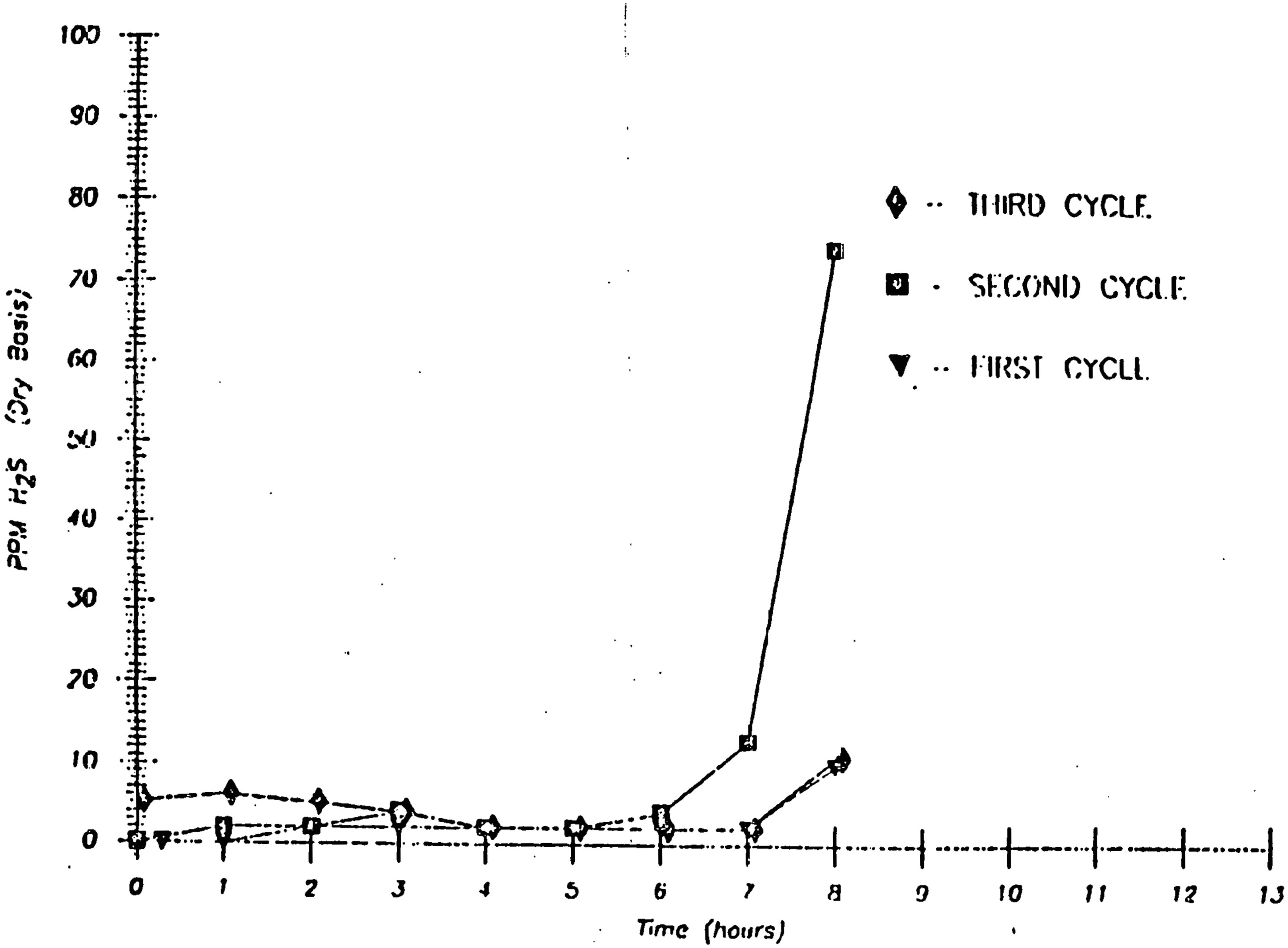
TEST 011B (THIRD CYCLE ON SAME SORBENT)



19-MAY-81 09:50:36

ZINC FERRITE SULFIDATION

TESTS 007,009,011B (THREE CYCLES ON SAME SORBENT)



18-SEP-81 08:17:37

TEST SUMMARY SHEET

TEST NO.	:	013
DATE STARTED	:	3/05/81
DATE ENDED	:	3/06/81
TOTAL HOURS	:	13
TYPE	:	SULFIDATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/1000 G
SORBENT NO. : HARSHAW ZN0401-E3/16 (SAMPLE)
SORBENT COMPOSITION: 100 % ZNO

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED AT 320 F FOR 2.5 HOURS
SULFUR LOADINGM : 18.7 % (GM SULFUR/GM FRESH SORBENT) TO 9 PPM BEFORE BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

	FLOW RATE	MOLE
H2S (0.24 DM3/MIN)	0.516 SCFH	2.7%
H20	0.500 CM3/MIN	6.9%
AIR		
CO2		
CO		
H2 (1.32 DM3/MIN)	2.79 SCFH	14.6%
CH4		
N2 (6.84 DM3/MIN)	14.50 SCFH	75.8%

8

PURPOSE

SULFIDATION OF ZINC OXIDE EXTRUSIONS TO DETERMINE SORPTION EFFICIENCY AND SUBSEQUENT REGENERATION CHARACTERISTICS.

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):	3.2	
DENSITY (G/CM3) :	6.39	
PORE VOLUME (MM3/G):	6.5	

MINERAL ANALYSIS: SEE APPENDIX
ELEMENTAL ANALYSIS: SEE APPENDIX

DATA: (DETECTOR TUBE)

EXIT H2S: 5-9 PPM PLATEAU FOR 9 HOURS
BREAKTHROUGH AT 10 HOURS WITH 960 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. H2S LEVEL REDUCED TO APPROXIMATELY THE LEVEL OBTAINED WITH ZINC FERRITE.

2.

CONCLUSIONS

1. ZNO IS A GOOD SORBENT AT HIGH TEMPERATURES WITH 6.9% WATER.

2. THIS TEST DATA VERIFIES THERMODYNAMIC CALCULATIONS INDICATING APPROXIMATELY 1 PPM H2S ACHIEVABLE WITH 6.9 % WATER AT 1000 F.

TEST SUMMARY SHEET

TEST NO.	:	014
DATE STARTED	:	3/06/81
DATE ENDED	:	3/06/81
TOTAL HOURS	:	12
TYPE	:	REGENERATION

PURPOSE

REGENERATION OF ZINC OXIDE EXTRUSIONS TO DETERMINE REGENERATION CHARACTERISTICS.

SORBENT TYPE/WEIGHT:	ZINC OXIDE/1000 G
SORBENT NO. :	HARSHAW ZN0401-E3/16 (SAMPLE)
SORBENT COMPOSITION:	100 % ZNO
SORBENT SIZE :	3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :	DRIED AT 320 F FOR 2.5 HOURS (BEFORE T013)
SULFUR REMOVED :	

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR(%W/W):		
SURFACE AREA(M2/G):		
DENSITY(G/CM3):		
PORE VOLUME(MM3/G):		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H2S		
H2O	2.17 CM3/MIN	50%
AIR (2.71 DM3/MIN)	5.735 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

DATA: (DETECTOR TUBE)

EXIT H2S: UNABLE TO READ DUE TO SO2 INTERFERENCE

EXIT SO2: 3-14%

EXIT S2: 0.1687 G

EXIT H2:

REMARKS

1. TEMPERATURE WAVE REACHED 1700 F.

2. SO2 LEVELS ARE HIGHER COMPARED TO ZINC FERRITE MUCH LESS ELEMENTAL SULFUR PRODUCED.

CONCLUSIONS

1. IT APPEARS THAT ZNO REGENERATES IN THE SAME WAY THAT IRON OXIDE AND ZINC FERRITE DO. A SIMILAR TEMPERATURE WAVE IS OBSERVED , AS EXPECTED.

2. IN SPITE OF A 50% STEAM LEVEL IN THE FEED, THE REGENERATION REACTION IS SUFFICIENTLY EXOTHERMIC TO PRODUCE A 700 F RISE IN PROCESS GAS TEMPERATURE.

TEST SUMMARY SHEET

TEST NO. : 015
DATE STARTED : 3/10/81
DATE ENDED : 3/11/81
TOTAL HOURS : 14
TYPE : SULFIDATION

PURPOSE

SECOND SULFIDATION OF ZINC OXIDE EXTRUSIONS TO DETERMINE SORPTION EFFICIENCY.

SORBENT TYPE/WEIGHT: ZINC OXIDE/1000 G
SORBENT NO. : HARSHAW ZN0401-E3/16 (SAMPLE)
SORBENT COMPOSITION: 100 % ZNO

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED AT 320 F FOR 2.5 HOURS
SULFUR LOADINGM : 18.8 (GM SULFUR/GM FRESH SORBENT) TO 14 PPM BEFORE BREAKTHROUGH

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):		1.1
DENSITY (G/CM3):		3.82
PORE VOLUME (MM3/G):		2.0
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

	FLOW RATE	MOLE
H2S (0.24 DM3/MIN)	0.516 SCFH	2.7%
H2O	0.500 CM3/MIN	6.9%
AIR		
CO2		
CO		
H2 (1.32 DM3/MIN)	2.79 SCFH	14.6%
CH4		
N2 (6.84 DM3/MIN)	14.50 SCFH	75.8%

TOTAL DRY GAS FLOW RATE = 17.80 SCFH

TOTAL WET GAS FLOW RATE = 19.11 SCFH

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DATA: (DETECTOR TUBE)

EXIT H2S: 2-7 PPM PLATEAU FOR 9 HOURS

BREAKTHROUGH AT 10 HOURS WITH 14 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SECOND SULFIDATION OF THIS SORBENT.

2. EXIT H2S WAS SLIGHTLY LOWER THAN THE FIRST SULFIDATION.

CONCLUSIONS

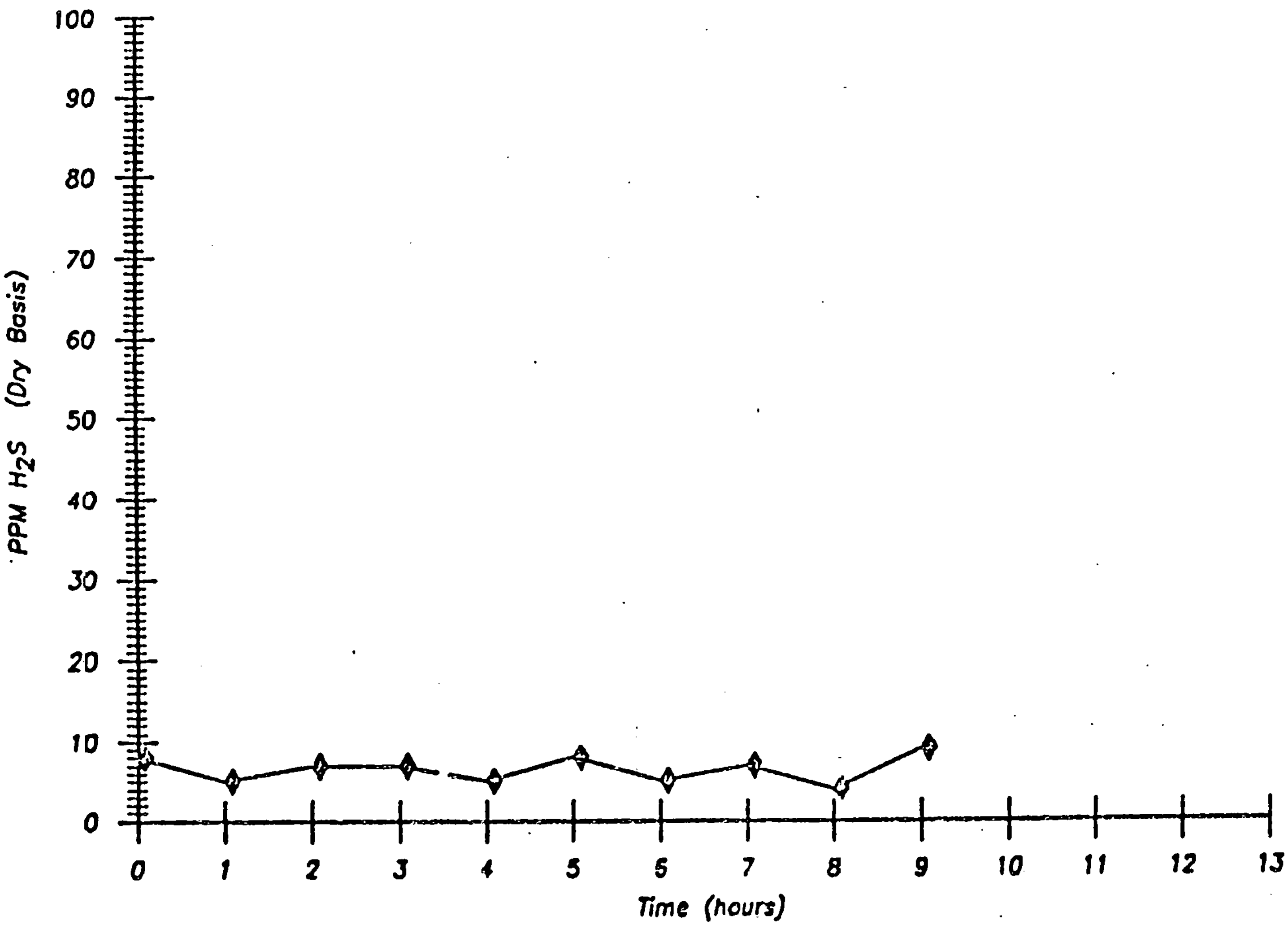
1. ZNO IS REGENERABLE AT THESE CONDITIONS.

2. NO LOSS IN ACTIVITY CAN BE DETECTED. A SLIGHT IMPROVEMENT IN SULFUR ABSORPTION IS OBSERVED.

P1

ZINC OXIDE SULFIDATION

TEST 013 (HARSHAW ZN-0401)



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TEST SUMMARY SHEET

TEST NO. : 016
DATE STARTED : 3/20/81
DATE ENDED : 3/20/81
TOTAL HOURS : 3.5
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH $2/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT
SINTERING : 3 HOURS AT 1500 F
SULFUR LOADING : 14.1 % (GM SULFUR/GM FRESH SORBENT)
TO 10 PPM BEFORE BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE		MOLE
H2S	(0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O		3.872 CM3/MIN	27.66%
AIR			
CO2	(1.70 DM3/MIN)	3.6 SCFH	9.68%
CO	(2.03 DM3/MIN)	4.3 SCFH	11.91%
H2	(2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4	(0.51 DM3/MIN)	1.07 SCFH	2.91%
N2	(4.88 DM3/MIN)	10.34 SCFH	33.09%

TOTAL DRY GAS FLOW RATE = 26.60 SCFH

TOTAL WET GAS FLOW RATE = 37.70 SCFH

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE SORPTION EFFICIENCY AND SUBSEQUENT REGENERATION CHARACTERISTICS, USING LURGI AIR/H2O GAS COMPOSITIONS AND INCREASED SPACE VELOCITIES.

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):	5.1	
DENSITY (G/CM3):	5.58	
PORE VOLUME (MM3/G):	10.9	
MINERAL ANALYSIS:	SEE APPENDIX	
ELEMENTAL ANALYSIS:		

DATA: (DETECTOR TUBE)

EXIT H2S: 9-13 PPM PLATEAU FOR 2.25 HRS
BREAKTHROUGH AT 3.25 HRS WITH 925 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. LURGI GAS COMPOSITION DOES NOT APPEAR TO PRODUCE A DIFFERENT RESULT IN THIS SULFIDATION.
2.

CONCLUSIONS

1. ACCELERATED TEST CONDITION OF SV=2000 REDUCED TIME TO BREAKTHROUGH ABOUT AS EXPECTED. TESTS CAN NOW BE COMPLETED IN ONE SHIFT.
2. H2S LEVEL AT EXIT ONLY SLIGHTLY HIGHER THAN THAT OBTAINED AT SV = 1000.

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TEST SUMMARY SHEET

TEST NO. : 017
DATE STARTED : 3/24/81
DATE ENDED : 3/24/81
TOTAL HOURS : 5
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 016 USING 50% AIR AND 50% STEAM, AND ACCELERATED SPACE VELOCITY OF 1200 HOURLY.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH 82/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED :

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):		1.4
DENSITY (G/CM3):		4.8
PORE VOLUME (MM3/G):		3.7
MINERAL ANALYSIS:	SEE APPENDIX	
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1200 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 8-13 %

EXIT S2: 7.7081 G

EXIT H2:

	FLOW RATE	MOLE
H2S		
H2O	4.19 CM3/MIN	50%
AIR (5.23 M3/MIN)	11.07 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

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REMARKS

1. TEMPERATURE WAVE UP TO 1910 F OBSERVED AT INLET, 1400-1560 THROUGHOUT THE REACTOR.

2. AFTER REMOVING THE EXTRUSIONS IT WAS OBSERVED THAT SOME AGGLOMERATION (SINTERING) HAD OCCURRED. A COATING OF HEMATITE AND ZNO WAS DETECTED ON SOME OF THE EXTRUSIONS.

CONCLUSIONS

1. SV-1200 MAY BE TOO HIGH A SPACE VELOCITY FOR REGENERATION. IT IS SUSPECTED THAT IT CAUSED SINTERING IN THIS RUN, REDUCING SORPTION ACTIVITY.

TEST SUMMARY SHEET

TEST NO. : 018
DATE STARTED : 3/27/81
DATE ENDED : 3/28/81
TOTAL HOURS : 6
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS AT 600 F TO DETERMINE SORPTION EFFICIENCY AND SUBSEQUENT REGENERATION CHARACTERISTICS, USING LURGI AIR/H2O GAS COMPOSITIONS AND INCREASED SPACE VELOCITIES.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/1
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR(%W/W):		
SURFACE AREA(M2/G):		
DENSITY(G/CM3):		
PORE VOLUME(MM3/G):		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 600 F (315 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H2S (0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2 (1.70 DM3/MIN)	3.6 SCFH	9.68%
CO (2.03 DM3/MIN)	4.3 SCFH	11.91%
H2 (2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4 (0.51 DM3/MIN)	1.07 SCFH	2.91%
N2 (4.88 DM3/MIN)	10.34 SCFH	33.09%

DATA: (DETECTOR TUBE)

EXIT H2S: 6000 PPM IMMEDIATELY

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. AFTER IMMEDIATE BREAKTHROUGH WAS OBSERVED, TEMP. WAS INCREASED TO 1000. AT THIS TEMP. 100 PPM WAS DETECTED. REGENERATION WAS CARRIED OUT & RUN MADE AT 1000 F. BREAKTHROUGH OCCURRED AFTER 50 MIN.

CONCLUSIONS

1. SULFIDATION ACTIVITY APPEARS TO BE VERY POOR AT 600 F, HOWEVER, IT IS SUSPECTED THAT THE EXTRUSIONS HAVE BEEN SINTERED IN THE LAST REGENERATION AT SV=1200.
2. THE BREAKTHROUGH OBTAINED AT 1000 F AFTER ONLY 50 MIN. INDICATES THAT SORBENT ACTIVITY HAS BEEN REDUCED.

8

TEST SUMMARY SHEET

TEST NO. : 019
DATE STARTED : 4/7/81
DATE ENDED : 4/8/81
TOTAL HOURS : 3.75
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE SORPTION EFFICIENCY AND SUBSEQUENT REGENERATION CHARACTERISTICS, USING LURGI AIR/H_2O GAS COMPOSITIONS AND INCREASED SPACE VELOCITIES.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):	4.4	
DENSITY (G/CM3):	5.72	
PORE VOLUME (MM3/G):	8.5	
MINERAL ANALYSIS:	SEE APPENDIX	
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H_2S (0.47 DM3/MIN)	0.9963 SCFH	2.7%
H_2O	3.872 CM3/MIN	27.66%
AIR		
CO_2 (1.70 DM3/MIN)	3.6 SCFH	9.68%
CO (2.03 DM3/MIN)	4.3 SCFH	11.91%
H_2 (2.97 DM3/MIN)	6.3 SCFH	17.12%
CH_4 (0.51 DM3/MIN)	1.07 SCFH	2.91%
N_2 (4.88 DM3/MIN)	10.34 SCFH	33.09%

DATA: (DETECTOR TUBE)

EXIT H_2S: 3-10 PPM PLATEAU FOR 2.5 HRS
BREAKTHROUGH WITH 60 PPM AFTER 2.5 HOURS

EXIT SO_2:

EXIT S_2:

EXIT H_2:

REMARKS

1. PREHEATER PLUGGED AFTER 1 HOUR INTO THE TEST. TEST WENT ON HOT HOLD OVER NIGHT, AND PREHEATER COIL REPLACED.
2. THIS RUN WAS STARTED WITH A FRESH BATCH OF SORBENT

CONCLUSIONS

1. NORMAL ACCELERATED SULFIDATION TEST IN TERMS OF OUTPUT DATA.

2.

TEST SUMMARY SHEET

TEST NO. : 020
DATE STARTED : 4/10/81
DATE ENDED : 4/10/81
TOTAL HOURS : 6.75
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH 82/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 019 USING SPACE VELOCITY OF 600.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 8-12 %

EXIT S2: 4.1643 G

EXIT H2:

REMARKS

1. TEMPERATURE WAVE UP TO 1600 F OBSERVED AT INLET AND 1400 F THROUGHOUT THE REACTOR.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. NORMAL REGENERAION FOR BASELINE VERIFICATION.

2.

TEST SUMMARY SHEET

TEST NO. : 021
DATE STARTED : 4/13/81
DATE ENDED : 4/13/81
TOTAL HOURS : 3.75
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO REESTABLISH BASELINE AFTER REGENERATION. THIS IS THE SECOND SULFIDATION OF THIS BATCH.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH 82/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 12.51 % (GM SULFUR/GM FRESH SORBENT) TO 3 PPM BEFORE BREAKTHROUGH

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE		MOLE
H2S	(0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O		3.872 CM3/MIN	27.66%
AIR			
CO2	(1.70 DM3/MIN)	3.6 SCFH	9.68%
CO	(2.03 DM3/MIN)	4.3 SCFH	11.91%
H2	(2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4	(0.51 DM3/MIN)	1.07 SCFH	2.91%
N2	(4.88 DM3/MIN)	10.34 SCFH	33.09%

DATA: (DETECTOR TUBE)

EXIT H2S: 3-5 PPM PLATEAU FOR 2 HRS
BREAKTHROUGH WITH 1100 PPM AFTER 3 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. SECOND SULFIDATION OF THIS BATCH OF SORBENT
2. LURGI GAS COMPOSITION USED.

CONCLUSIONS

1. NORMAL ACCELERATED SULFIDATION TEST IN TERMS OF OUTPUT DATA.

TEST SUMMARY SHEET

TEST NO. : 022
DATE STARTED : 4/14/81
DATE ENDED : 4/14/81
TOTAL HOURS : 6.75
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 021 USING SPACE VELOCITY OF 600.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED :

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 3.5-12.8 %

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

REMARKS

1. ACTUAL FLOWS WERE:2.17 CM3/MIN WATER,5.735 SCFH AIR.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. NORMAL REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 023
DATE STARTED : 4/15/81
DATE ENDED : 4/15/81
TOTAL HOURS : 6.75
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 1.56 % (GM SULFUR/GM FRESH SORBENT)
TO 5 PPM BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 800 F (427 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE		MOLE
H2S	(0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O		3.872CM3/MIN	27.66%
AIR			
CO2	(1.70 DM3/MIN)	3.6 SCFH	9.63%
CO	(2.03 DM3/MIN)	4.3 SCFH	11.91%
H2	(2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4			
N2	(5.76 DM3/MIN)	12.20 SCFH	36.03%

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 800 F (427 C).

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: INITIALLY 5 PPM
900 PPM BREAKTHROUGH AFTER 30 MIN.

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. AFTER BREAKTHROUGH AT 800 F TEMP. INCREASED TO 900 F.
2. AT 900F BREAKTHROUGH HAD OCCURRED ALSO. (900 PPM READING). REGENERATION FOLLOWED.
3. NO METHANE , N2 INCREASED TO COMPENSATE.

CONCLUSIONS

1. SORPTION ACTIVITY OF ZINC FERRITE AT 800 F IS NOT SUFFICIENTLY HIGH FOR FUEL CELL APPLICATION.

2. APPARENTLY THE KINETICS ARE TOO SLOW AT THIS TEMP TO LOWER THE H2S TO BELOW 10 PPM.

3. THIRD SULFIDATION OF THIS BATCH OF SORBENT.

TEST SUMMARY SHEET

TEST NO. : 024
DATE STARTED : 4/16/81
DATE ENDED : 4/16/81
TOTAL HOURS : 3.25
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1200 F (649 C) .

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH 82/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 15.6 % (GM SULFUR/GM FRESH SORBENT) TO 15 PPM BREAKTHROUGH

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1200 F (649 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H2S (0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2 (1.70 DM3/MIN)	3.6 SCFH	9.68 %
CO (2.03 DM3/MIN)	4.3 SCFH	11.91 %
H2 (2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4 (0.[illegible]1 DM3/MIN)	1.07 SCFH	2.91 %
N2 (4.88 DM3/MIN)	10.34 SCFH	33.09%

DATA: (DETECTOR TUBE)

EXIT H2S: 9-13 PPM PLATEAU
BREAKTHROUGH WITH 15 PPM AT 2.5 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1.

CONCLUSIONS

1. H2S LEVEL AT 1200 F ABOUT TWICE THE LEVEL OBTAINED AT 1000 F.

TEST SUMMARY SHEET

TEST NO. : 025
DATE STARTED : 4/17/81
DATE ENDED : 4/17/81
TOTAL HOURS : 5.25
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 024 USING SPACE VELOCITY OF 600.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT 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 (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 3.5-12.8 %

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

REMARKS

1. ACTUAL FLOWS WERE:2.17 CM3/MIN WATER,5.735 SCFH AIR.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. NORMAL REGENERATION.

8

TEST SUMMARY SHEET

TEST NO. : 026
DATE STARTED : 4/20/81
DATE ENDED : 4/20/81
TOTAL HOURS : 2.5
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1400 F (750 C).

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 9.4 % (GM SULFUR/GM FRESH SORBENT) TO 2 PPM BREAKTHROUGH

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1400 F (760 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H2S (0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2 (1.70 DM3/MIN)	3.6 SCFH	9.68 %
CO (2.03 DM3/MIN)	4.3 SCFH	11.91 %
H2 (2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4 (0.51 DM3/MIN)	1.07 SCFH	2.91 %
N2 (4.88 DM3/MIN)	10.34 SCFH	33.09 %

DATA: (DETECTOR TUBE)

EXIT H2S: 1-3 PPM PLATEAU
BREAKTHROUGH WITH 26 PPM AT 2 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. FOURTH SULFIDATION OF THIS SORBENT.

CONCLUSIONS

1. EXIT H2S IS LOWER AT 1400 F THAN AT 1200F, INDICATING BETTER KINETICS AND DIFFUSION AT THIS TEMPERATURE. THIS LEVEL OF ACTIVITY MAY NOT BE REPEATABLE OVER MANY CYCLES DUE TO THE EFFECT OF SINTERING.
2.

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TEST SUMMARY SHEET

TEST NO. : 027
DATE STARTED : 4/21/81
DATE ENDED : 4/21/81
TOTAL HOURS : 6.5
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 026 USING SPACE VELOCITY OF 600.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : DRIED IN OVEN AT 200 F OVERNIGHT
SULFUR REMOVED :

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 3.5-12.8 %

EXIT S2:

EXIT H2:

REMARKS

1. FOURTH REGENERATION OF THIS SORBENT.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1.

8

TEST SUMMARY SHEET

TEST NO. : 028
DATE STARTED : 4/22/81
DATE ENDED : 4/28/81
TOTAL HOURS : 1.5
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH 82/2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 6.26 % (GM SULFUR/GM FRESH SORBENT)
TO 4 PPM BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 1500 F (816 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H2S (0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2 (1.70 DM3/MIN)	3.6 SCFH	9.68 %
CO (2.03 DM3/MIN)	4.3 SCFH	11.91 %
H2 (2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4 (0.51 DM3/MIN)	1.07 SCFH	2.91%
N2 (4.88 DM3/MIN)	10.34 SCFH	33.09 %

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PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS
TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE
OF 1500 F (816 C).

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):		0.5 (SEE NOTE)
DENSITY (G/CM3):		4.99
PORE VOLUME (MM3/G):		0.7

MINERAL ANALYSIS: SEE APPENDIX
ELEMENTAL ANALYSIS:

DATA: (DETECTOR TUBE)

EXIT H2S: 4 PPM PLATEAU FOR 0.5 HR
BREAKTHROUGH AT 1 HR WITH 16,000 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. FIFTH SULFIDATION OF THIS BATCH.
2. SORBENT OVERHEATED AT REACTOR EXIT TO 1620 F DURING OVERNIGHT HOT HOLD.
3. PELLETS FUSED TOGETHER AT MID BED BETWEEN T/C 5 AND T/C 4 , DUE TO OVERHEATING.

CONCLUSIONS

1. SINTERING CAUSED REACTOR TO PLUG.
2. SULFIDATION AT 1500 F CAUSES MORE RAPID BREAKTHROUGH PROBABLY DUE TO SINTERING CAUSING LOSS OF ACTIVE SURFACE AREA.
3. INITIAL H2S LEVELS AT 1500 F ARE STILL FAIRLY LOW AT 4 PPM.

TEST SUMMARY SHEET

TEST NO. : 029
DATE STARTED : 4/24/81
DATE ENDED : 4/24/81
TOTAL HOURS : 0.6
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1500 F (816 C).

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/3
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 1.56 % (GM SULFUR/GM FRESH SORBENT) TO 2 PPM BEFORE BREAKTHROUGH

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1500 F (816 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 2 PPM INITIALLY
700 PPM AFTER 30 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE
H2S (0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2 (1.70 DM3/MIN)	3.6 SCFH	9.68 %
CO (2.03 DM3/MIN)	4.3 SCFH	11.91 %
H2 (2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4 (0.51 DM3/MIN)	1.07 SCFH	2.91%
N2 (4.88 DM3/MIN)	10.34 SCFH	33.09%

REMARKS

1. THIS TEST IS TO REPEAT TEST 028 IN WHICH OVER-HEATING WAS EXPERIENCED WITH EXCESSIVE SINTERING.
2. BREAKTHROUGH IN THIS IN TEST WAS FASTER THAN IN TEST 028 ALSO AT 1500 F.
3. NEW BATCH OF SORBENT.

CONCLUSIONS

1. RAPID BREAKTHROUGH INDICATES POSSIBILITY OF SINTERING OF SORBENT AT THIS TEMPERATURE.

2. 1500 F APPEARS TO BE THE UPPER LIMIT OF OPERATION FOR ZINC FERRITE.

TEST SUMMARY SHEET

TEST NO. : 030
DATE STARTED : 4/24/81
DATE ENDED : 4/24/81
TOTAL HOURS : 1.25
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE EXTRUSIONS FROM TEST 029 USING SPACE VELOCITY OF 600.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/3
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT 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 (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 4.0-5.0 %

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

REMARKS

1. THIS TEST IS TO REGENERATE THE LAST SULFIDATION AT 1500 F FOR ANOTHER BASELINE TEST AT 1000 F TO DETERMINE IF A PERMANENT LOSS OF ACTIVITY OCCURRED.
2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. THIS REGENERATION WAS SHORTER DUE TO THE SHORT SULFIDATION DURING TEST 029.

8

TEST SUMMARY SHEET

TEST NO.	:	031
DATE STARTED	:	4/24/81
DATE ENDED	:	4/24/81
TOTAL HOURS	:	0.5
TYPE	:	SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1000 F (538 C)

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 G
SORBENT NO. : BATCH #2/3
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):		2.2
DENSITY (G/CM3):		5.23
PORE VOLUME (MM3/G):		5.2
MINERAL ANALYSIS:	SEE APPENDIX	
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H2S	(0.47 DM3/MIN) 0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2	(1.70 DM3/MIN) 3.6 SCFH	9.68 %
CO	(2.03 DM3/MIN) 4.3 SCFH	11.91 %
H2	(2.97 DM3/MIN) 6.3 SCFH	17.12%
CH4	(0.51 DM3/MIN) 1.07 SCFH	2.91%
N2	(4.88 DM3/MIN) 10.34 SCFH	33.09 %

DATA: (DETECTOR TUBE)

EXIT H2S: 600 PPM IMMEDIATELY

3600 PPM AFTER 30 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS TEST IS TO DETERMINE IF PERMANENT LOSS OF ACTIVITY OCCURRED AT 1500 F DURING TEST 029.
2.

CONCLUSIONS

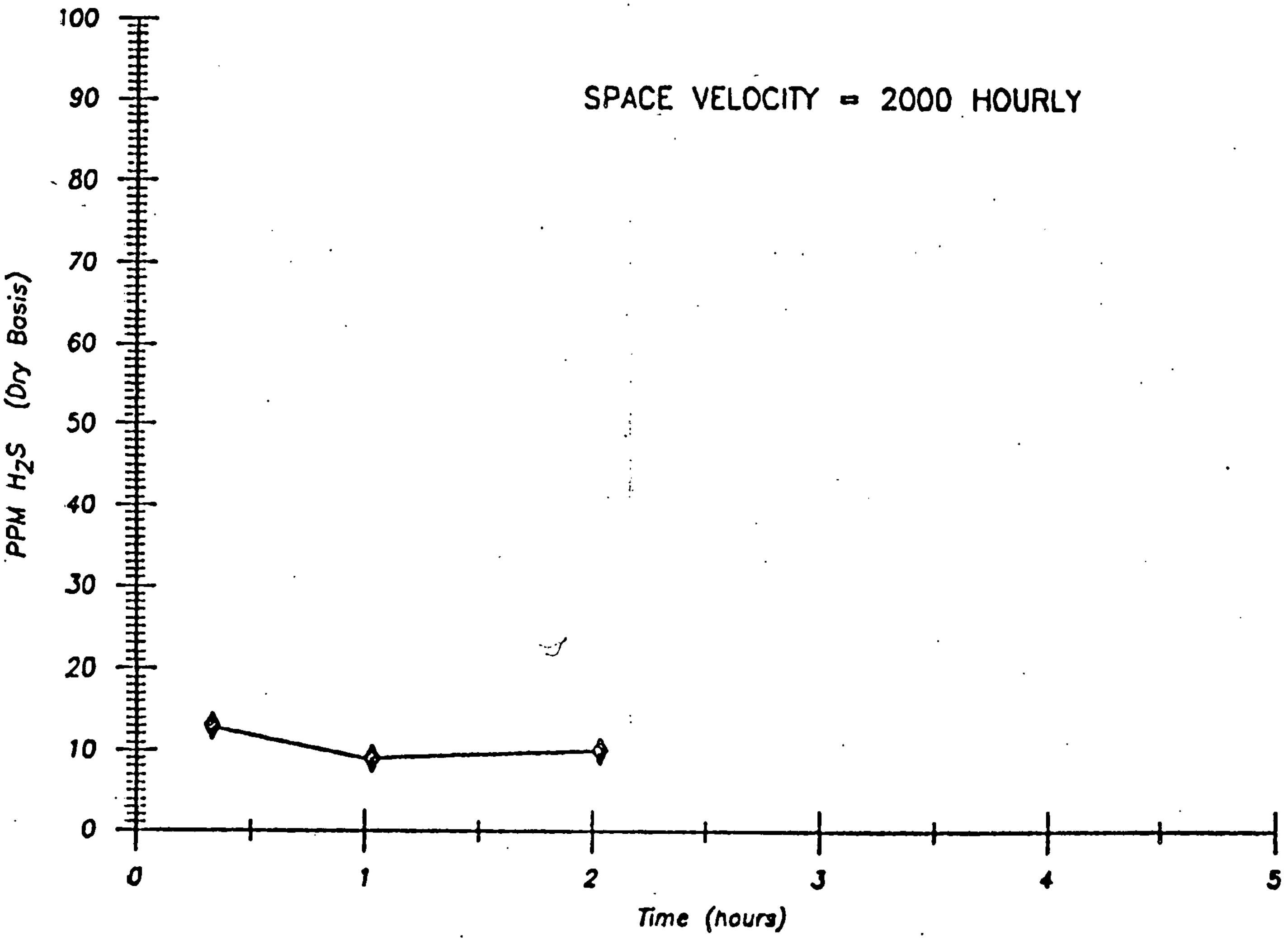
1. PERMANENT LOSS OF ACTIVITY HAS OCCURRED AS SEEN BY IMMEDIATE BREAKTHROUGH OF 600 PPM AT THE FIRST READING.

2. 1500 F APPEARS TO BE PAST THE UPPER LIMIT OF OPERATION FOR ZINC FERRITE.

8

ZINC FERRITE SULFIDATION

TEST 016

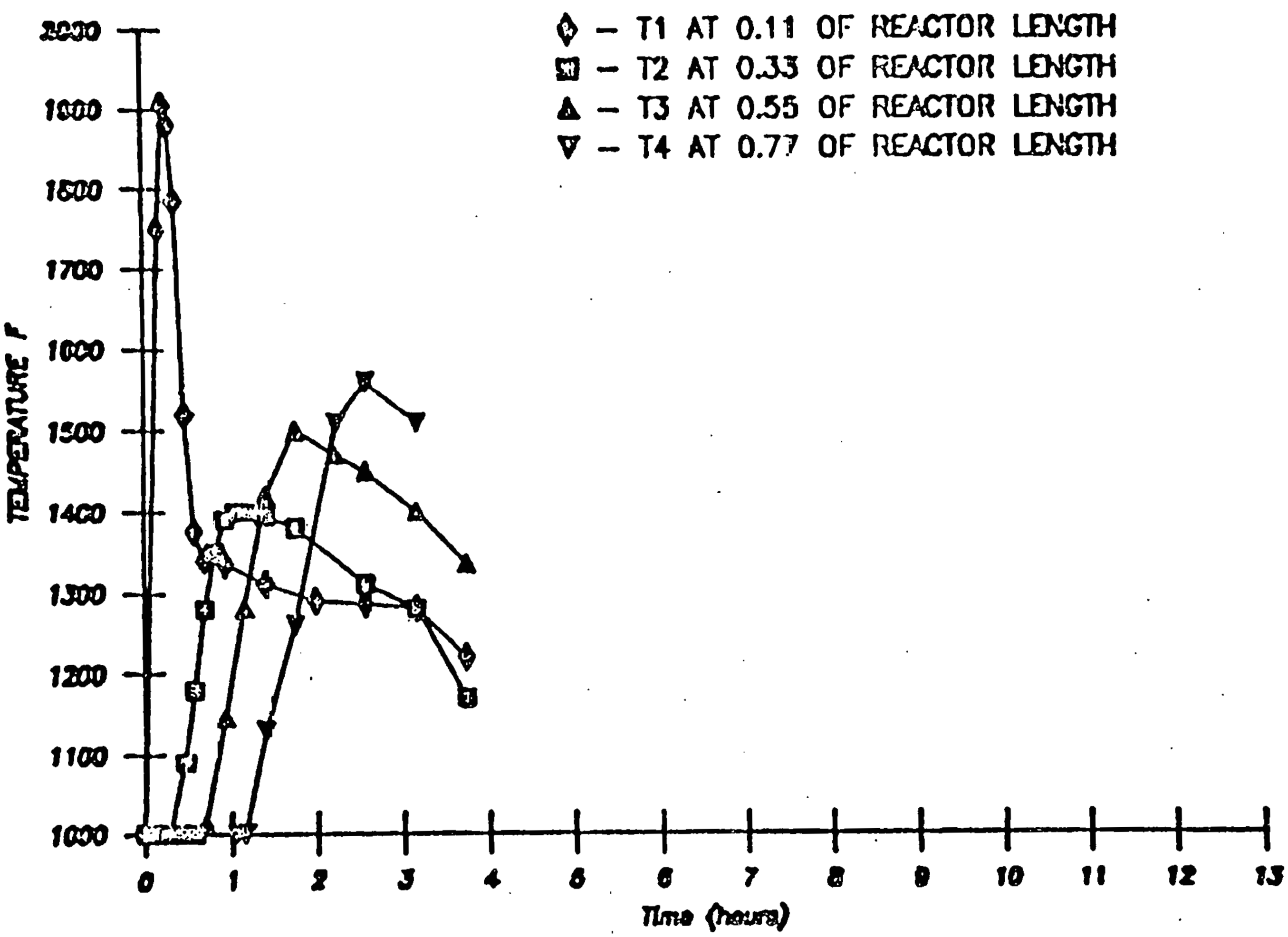


19-MAY-81 09:43:53

P:

ZINC FERRITE REGENERATION

TEST 017

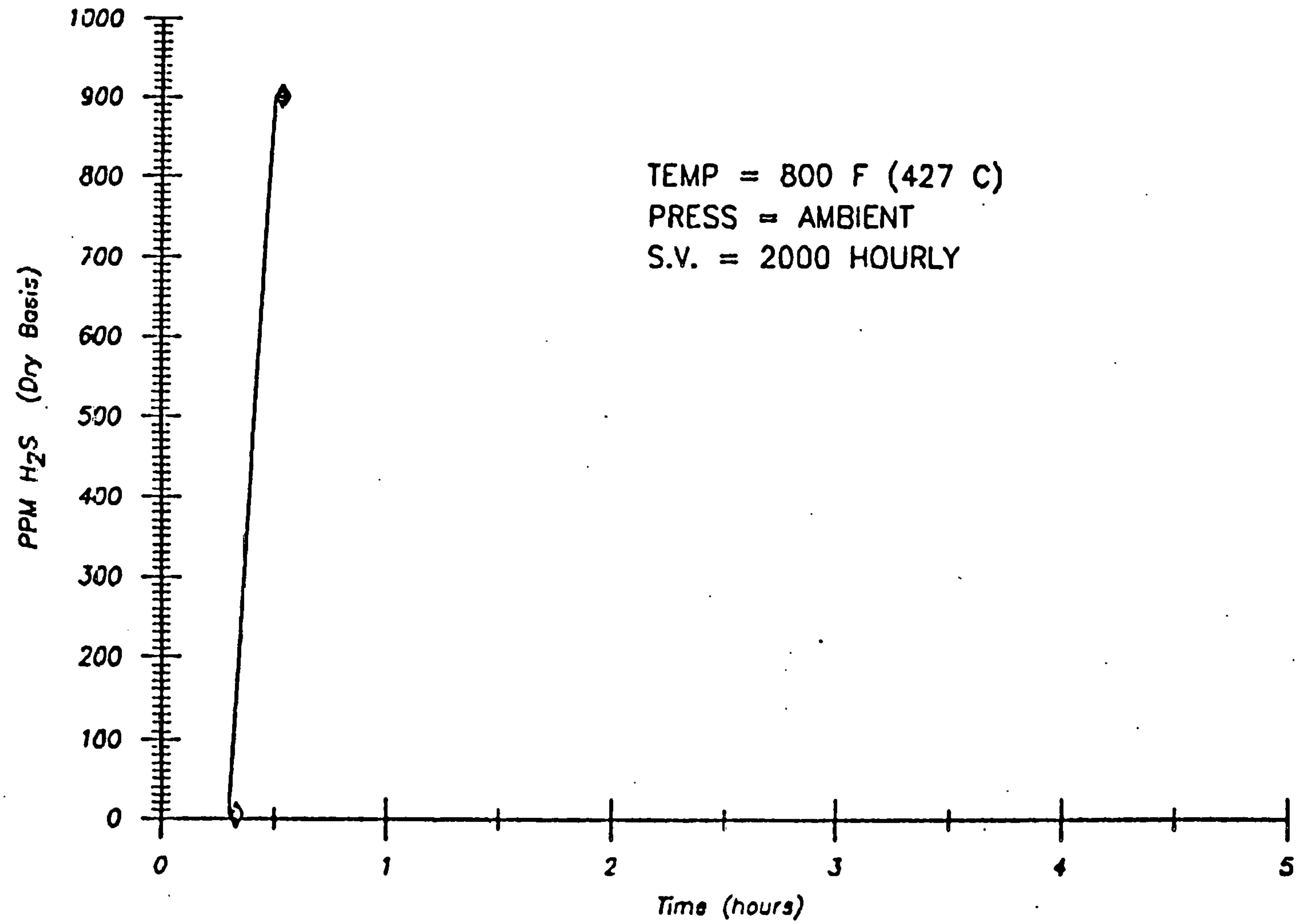


?-JUN-91 11:02:12

P1

ZINC FERRITE SULFIDATION

TEST 023

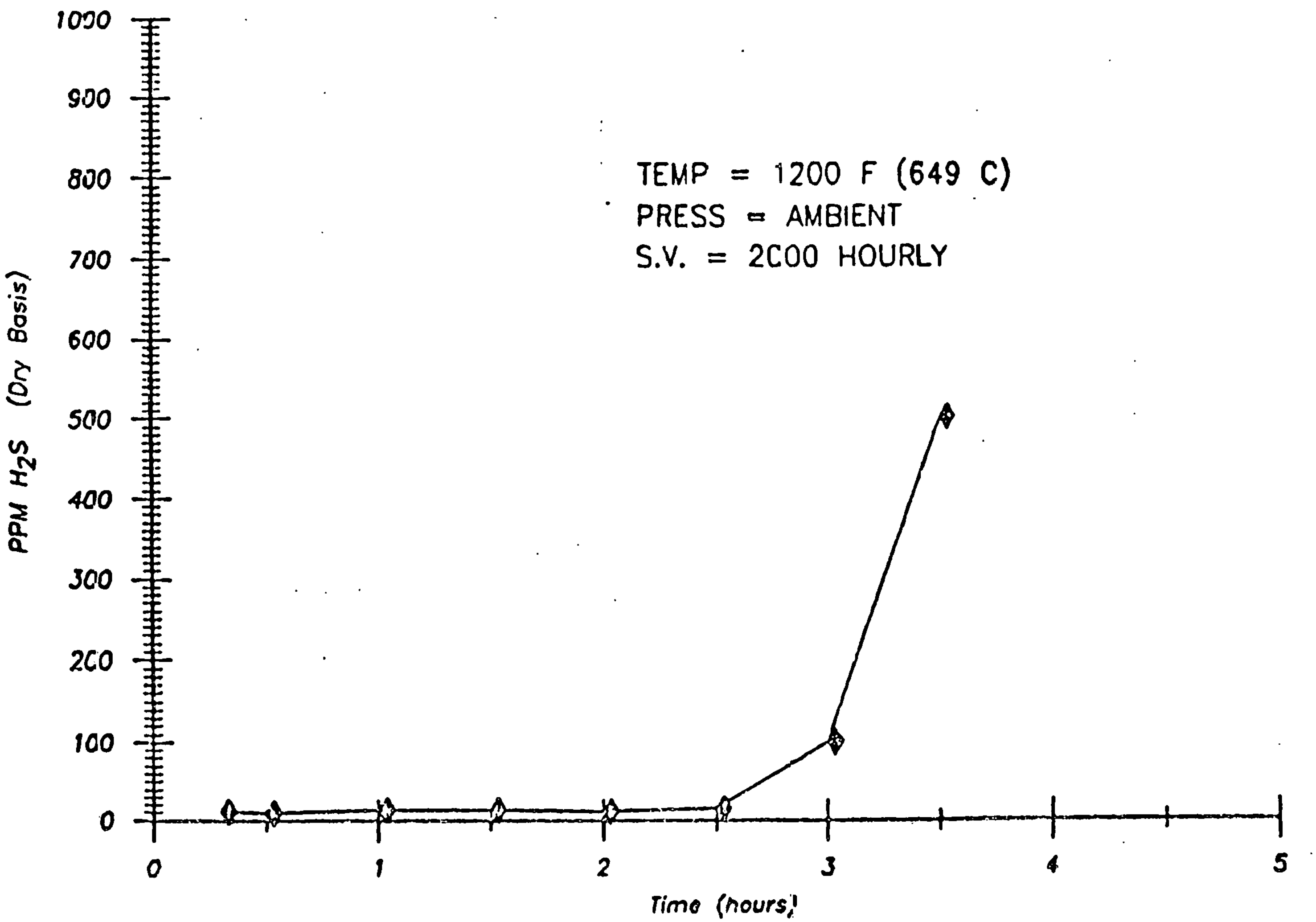


19-MAY-8' 09:42:25

P:

ZINC FERRITE SULFIDATION

TEST 024

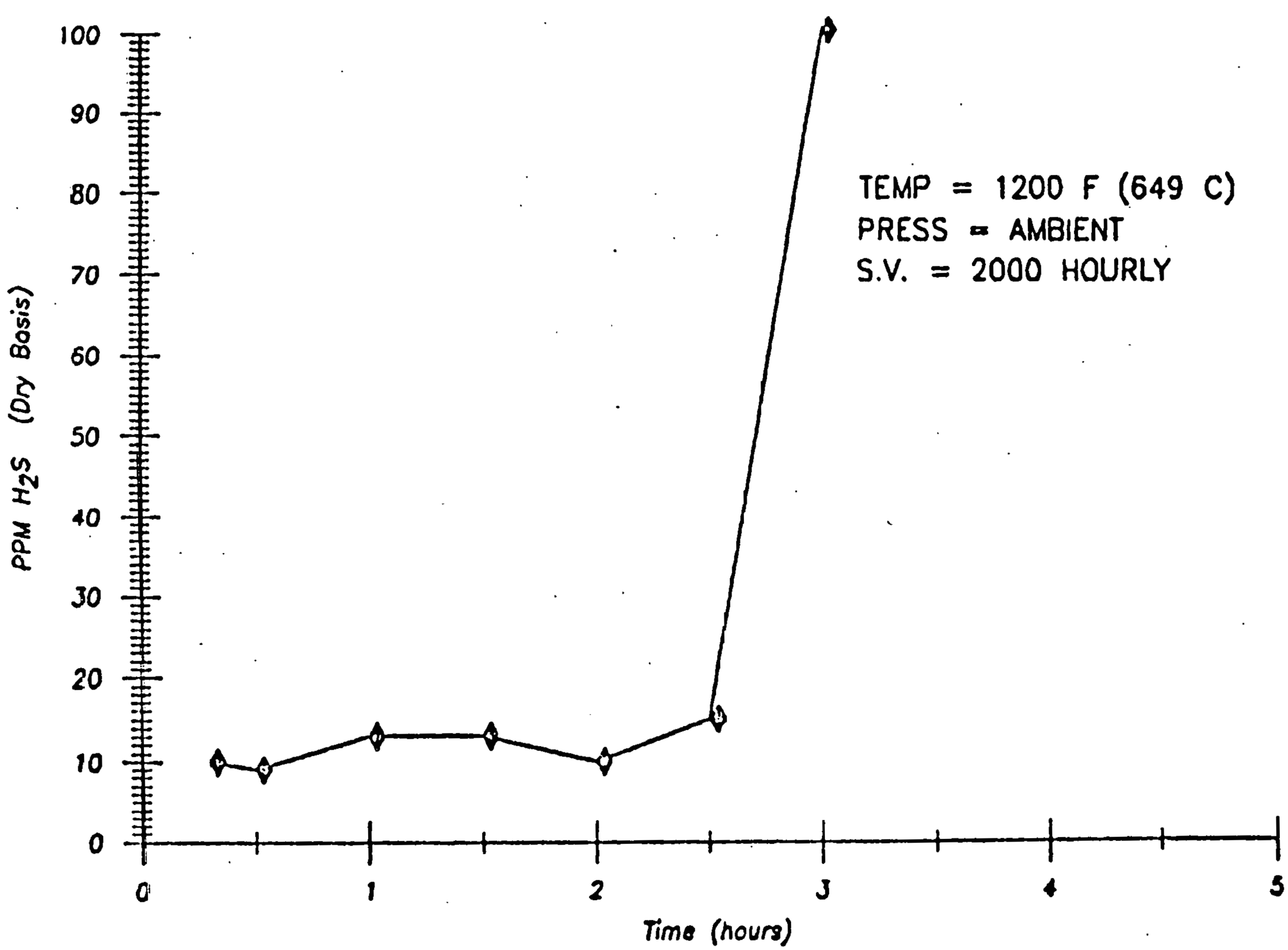


19-MAY-81 09:40:46

Pl

ZINC FERRITE SULFIDATION

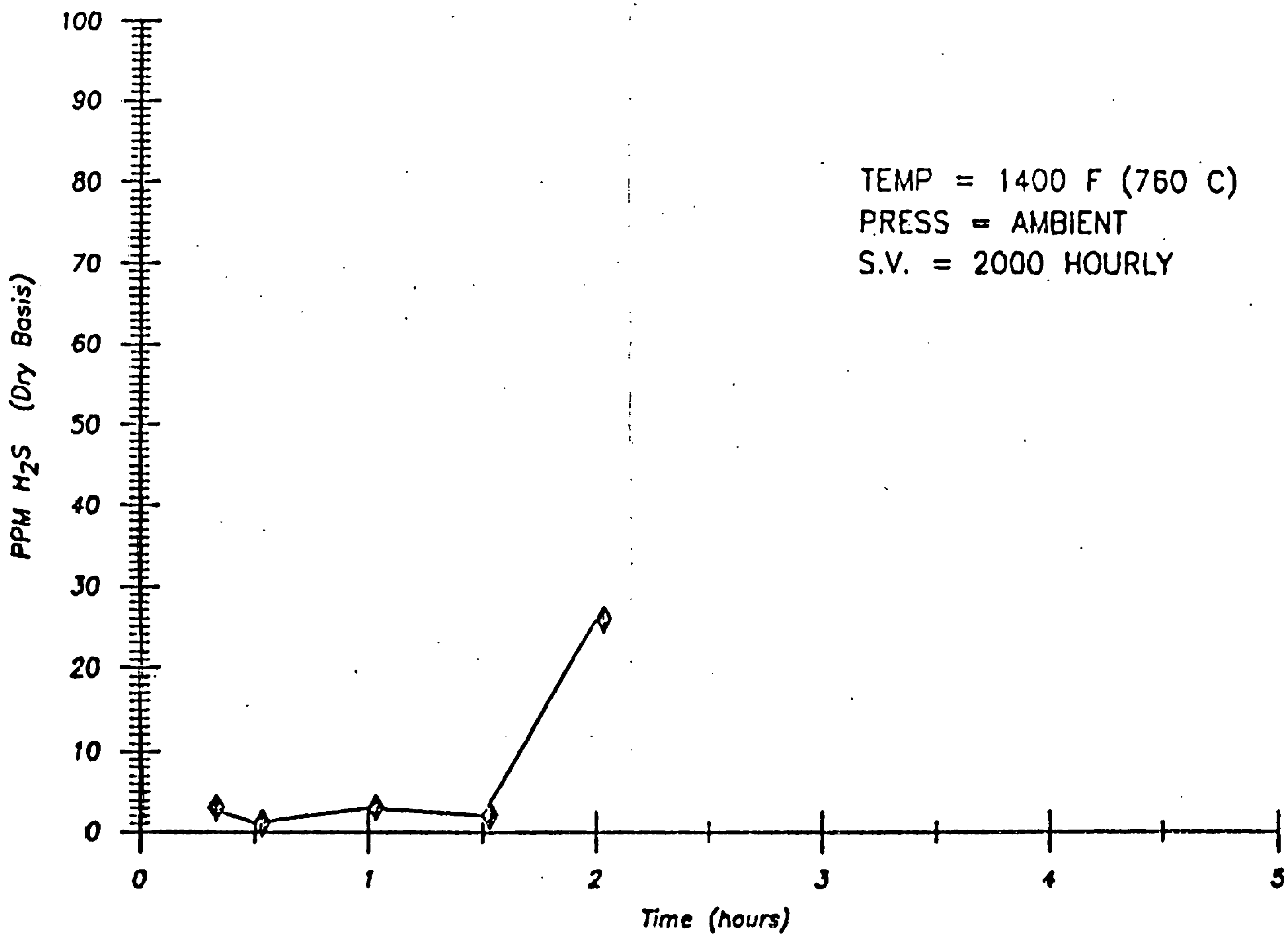
TEST 024



19-MAY-81 09:39:17

ZINC FERRITE SULFIDATION

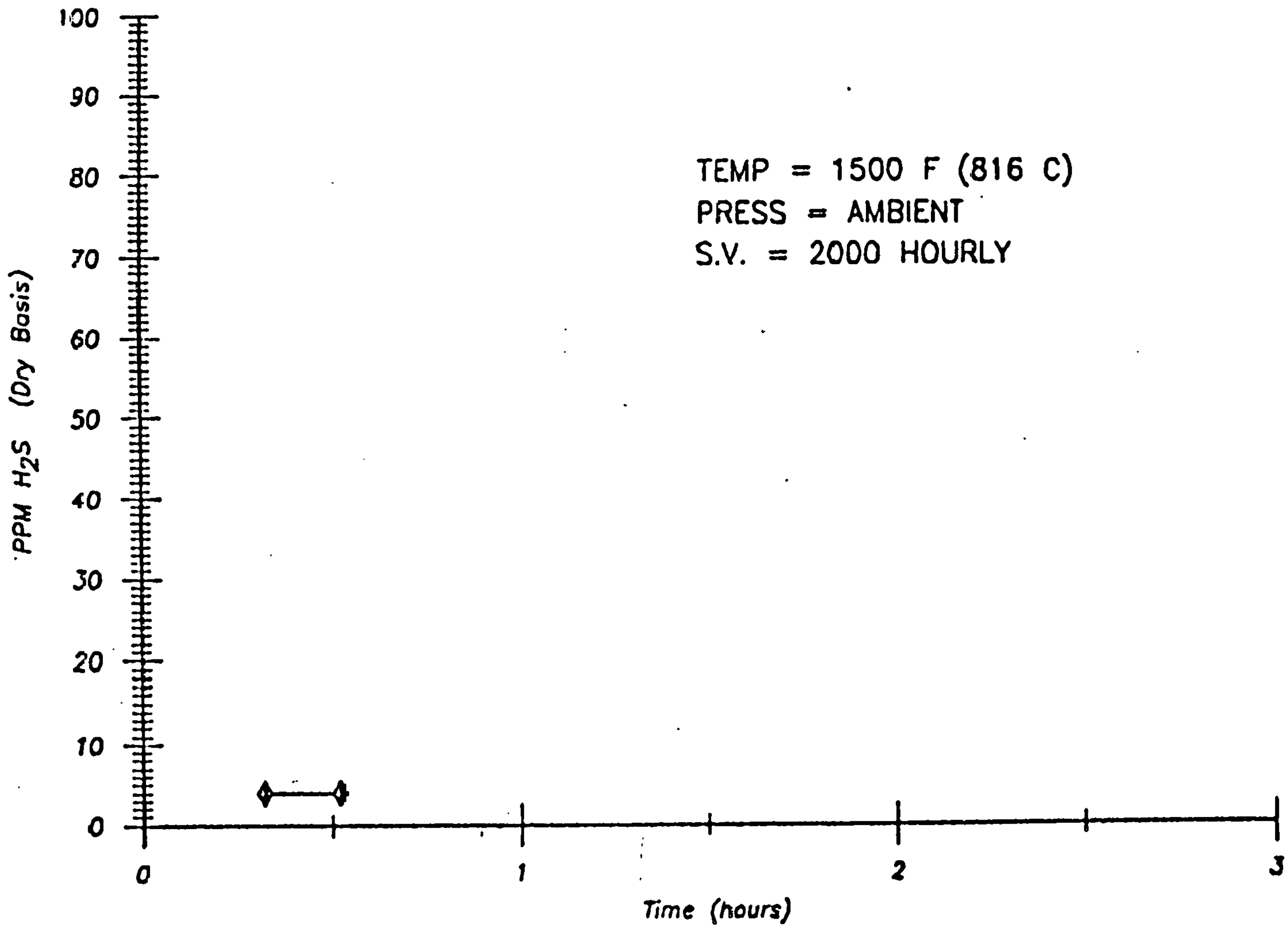
TEST 026



19-MAY-81 09:37:45

ZINC FERRITE SULFIDATION

TEST 028



19-MAY-81 09:36:19

TEST SUMMARY SHEET

TEST NO.	:	032
DATE STARTED	:	4/27/81
DATE ENDED	:	4/27/81
TOTAL HOURS	:	3
TYPE	:	SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 6.04 % (GM SULFUR/GM FRESH SORBENT) TO 7 PPM BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 800 F (427 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE		MOLE
H2S	(0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O		3.872 CM3/MIN	27.66%
AIR			
CO2	(1.70 DM3/MIN)	3.6 SCFH	9.68 %
CO	(2.03 DM3/MIN)	4.3 SCFH	11.91 %
H2	(2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4	(0.51 DM3/MIN)	1.07 SCFH	2.91%
N2	(4.88 DM3/MIN)	10.34 SCFH	33.09 %

PURPOSE

SULFIDATION OF ZINC OXIDE EXTRUSIONS
TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 800 F (427 C).

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 3 PPM PLATEAU FOR 1 HR

BREAKTHROUGH WITH 7 PPM AT 1.5 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FIRST SULFIDATION OF THIS SORBENT.
2.

CONCLUSIONS

1.

2.

TEST SUMMARY SHEET

TEST NO. : 033
DATE STARTED : 4/30/81
DATE ENDED : 4/30/81
TOTAL HOURS : 6
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT 95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED : % (GM SULFUR/GM FRESH SORBENT)

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

PURPOSE

REGENERATION OF ZINC OXIDE EXTRUSIONS FROM TEST 032 USING SPACE VELOCITY OF 600.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 3.5-12.8 %

EXIT S2:

EXIT H2:

REMARKS

1. TEMPERATURE WAVE REACHED 1540 F (838 C).

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. THIS IS THE FIRST REGENERATION ON THIS BATCH OF SORBENT.

TEST SUMMARY SHEET

TEST NO. : 034
DATE STARTED : 5/01/81
DATE ENDED : 5/01/81
TOTAL HOURS : 4
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 8.0 % (GM SULFUR/GM FRESH SORBENT) TO 3 PPM BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H2S	(0.47 DM3/MIN)0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2	(1.70 DM3/MIN) 3.6 SCFH	9.68 %
CO	(2.03 DM3/MIN) 4.3 SCFH	11.91 %
H2	(2.97 DM3/MIN) 6.3 SCFH	17.12%
CH4	(0.51 DM3/MIN) 1.07 SCFH	2.91%
N2	(4.88 DM3/MIN) 10.34 SCFH	33.09 %

PURPOSE

SULFIDATION OF ZINC OXIDE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1000 F (538 C).

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 0-3 PPM PLATEAU FOR 2 HRS

BREAKTHROUGH AT 2.5 HRS WITH 23 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SECOND SULFIDATION OF THIS BATCH OF SORBENT.
2.

CONCLUSIONS

1. H2S CAPACITY AT 1000 F IS BETTER THAN AT 800 F.

2.

TEST SUMMARY SHEET

TEST NO. : 035
DATE STARTED : 5/04/81
DATE ENDED : 5/04/81
TOTAL HOURS : 7.5
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC OXIDE EXTRUSIONS FROM TEST 034

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401-E3/16 LOT 95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE		MOLE
H2S			
H2O	2.23	CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88	SCFH	50%
CO2			
CO			
H2			
CH4			
N2			

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 3.7-14.8 %

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SECOND REGENERATION OF THIS BATCH OF SORBENT.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. NORMAL REGENERATION.

8

TEST SUMMARY SHEET

TEST NO.	:	036
DATE STARTED	:	5/05/81
DATE ENDED	:	5/05/81
TOTAL HOURS	:	3
TYPE	:	SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 8.0 % (GM SULFUR/GM FRESH SORBENT) TO 11 PPM BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 1200 F (649 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H2S (0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2 (1.70 DM3/MIN)	3.6 SCFH	9.68%
CO (2.03 DM3/MIN)	4.3 SCFH	11.91 %
H2 (2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4 (0.51 DM3/MIN)	1.07 SCFH	2.91%
N2 (4.88 DM3/MIN)	10.34 SCFH	33.89%

PURPOSE

SULFIDATION OF ZINC OXIDE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1200 F (649 C).

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 6-15 PPM PLATEAU FOR 2 HRS
BREAKTHROUGH WITH 184 PPM AT 2.5 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE THIRD SULFIDATION OF THIS BATCH OF SORBENT.
2.

CONCLUSIONS

1. H2S CAPACITY AT 1200 F IS LOWER THAN AT 1000 F.

2.

TEST SUMMARY SHEET

TEST NO.	:	037
DATE STARTED	:	5/07/81
DATE ENDED	:	5/07/81
TOTAL HOURS	:	6.75
TYPE	:	REGENERATION

PURPOSE

REGENERATION OF ZINC OXIDE EXTRUSIONS FROM TEST 036.

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401-E3/16 LOT 95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 3.7-14.8 %

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE THIRD REGENERATION OF THIS BATCH OF SORBENT.

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. NORMAL REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 038
DATE STARTED : 5/08/81
DATE ENDED : 5/08/81
TOTAL HOURS : 1.5
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 2.0 % (GM SULFUR/GM FRESH SORBENT) TO 2 PPM BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 1400 F (760 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE		MOLE
H2S	(0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O		3.872 CM3/MIN	27.66%
AIR			
CO2	(1.70 DM3/MIN)	3.6 SCFH	9.68 %
CO	(2.03 DM3/MIN)	4.3 SCFH	11.91 %
H2	(2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4	(0.51 DM3/MIN)	1.07 SCFH	2.91%
N2	(4.88 DM3/MIN)	10.34 SCFH	33.09 %

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PURPOSE

SULFIDATION OF ZINC OXIDE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1400 F (760 C).

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 2-3 PPM PLATEAU FOR 30 MINUTES
BREAKTHROUGH WITH 27 PPM AT 1 HR

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FOURTH SULFIDATION OF THIS BATCH OF SORBENT.
2.

CONCLUSIONS

1. H2S CAPACITY AT 1400 F IS LOWER THAN AT 1200 F.

2.

TEST SUMMARY SHEET

TEST NO. : 039
DATE STARTED : 5/15/81
DATE ENDED : 5/15/81
TOTAL HOURS : 19
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC OXIDE EXTRUSIONS FROM TEST 038.

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401-E3/16 LOT 95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

	FLOW RATE	MOLE
H2S		
H2O	2.23 CM3/MIN	50%
AIR (2.78 DM3/MIN)	5.88 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FOURTH REGENERATION OF THIS BATCH OF SORBENT.
2. REGENERATION ABORTED.
2.

CONCLUSIONS

1.

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TEST SUMMARY SHEET

TEST NO. : 040
DATE STARTED : 5/14/81
DATE ENDED : 5/14/81
TOTAL HOURS :
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 G
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A/2
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT SIZE : 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 1.0 % (GM SULFUR/GM FRESH SORBENT) TO 2 PPM BREAKTHROUGH

OPERATING CONDITIONS

TEMPERATURE: 1500 F (816 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE
H2S (0.47 DM3/MIN)	0.9963 SCFH	2.7%
H2O	3.872 CM3/MIN	27.66%
AIR		
CO2 (1.70 DM3/MIN)	3.6 SCFH	9.68 %
CO (2.03 DM3/MIN)	4.3 SCFH	11.91 %
H2 (2.97 DM3/MIN)	6.3 SCFH	17.12%
CH4 (0.51 DM3/MIN)	1.07 SCFH	2.91%
N2 (4.88 DM3/MIN)	10.34 SCFH	33.09 %

PURPOSE

SULFIDATION OF ZINC OXIDE EXTRUSIONS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1500 F (816 C).

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 2 PPM INITIALLY
BREAKTHROUGH WITH 51 PPM AT 30 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. NEW BATCH OF SORBENT. SORPENT.
2.

CONCLUSIONS

1.

2.

TEST SUMMARY SHEET

TEST NO. : 041
DATE STARTED : 5/20/81
DATE ENDED : 5/20/81
TOTAL HOURS : HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 GM
SORBENT NO. : HARSHAW ZNO 0401-E3/16
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT) AT THE START OF REGENERATION.

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

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

PURPOSE

REGENERATION OF ZINC OXIDE PELLETS FROM TEST 040

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		
DENSITY :		
POROSITY :		
X-RAY DIFFRACTION:		

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: APPROXIMATELY %

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FIFTH REGENERATION ON THIS BATCH OF SORBENT.

2.

CONCLUSIONS

1.

TEST SUMMARY SHEET

TEST NO. : 042R
DATE STARTED : 8/19/81
DATE ENDED : 8/19/81
TOTAL HOURS : 3.5 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC OXIDE PELLETS TO DETERMINE EXIT H2S COMPOSITION AT SORBTION TEMPERATURE OF 1000 F (537 C) USING DRY (NO WATER) LURGI GAS COMPOSITION .

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 GM
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:	50 M/G	
DENSITY :	G/CC	
POROSITY :		
X-RAY DIFFRACTION:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (537 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1442 HOURLY

	FLOW RATE	MOLE%
H2S	0.9963 SCFH	3.7%
H2O		
AIR		
CO2	3.6 SCFH	13.53 %
CO	4.3 SCFH	16.16 %
H2	6.3 SCFH	23.6%
CH4	1.07 SCFH	4.0%
N2	10.34 SCFH	38.87 %

DATA: (BY DETECTOR TUBE)

EXIT H2S: 1.0 PPM STEADY STATE

BREAKTHRU WITH 3.3 PPM AT 2.5 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FIRST SULFIDATION ON THIS LOAD OF SORBENT.
2. COMPOSITION OF EACH OF THE FEED GASES WAS ADJUSTED TO REFLECT REMOVAL OF H2O FROM FEED GAS.

CONCLUSIONS

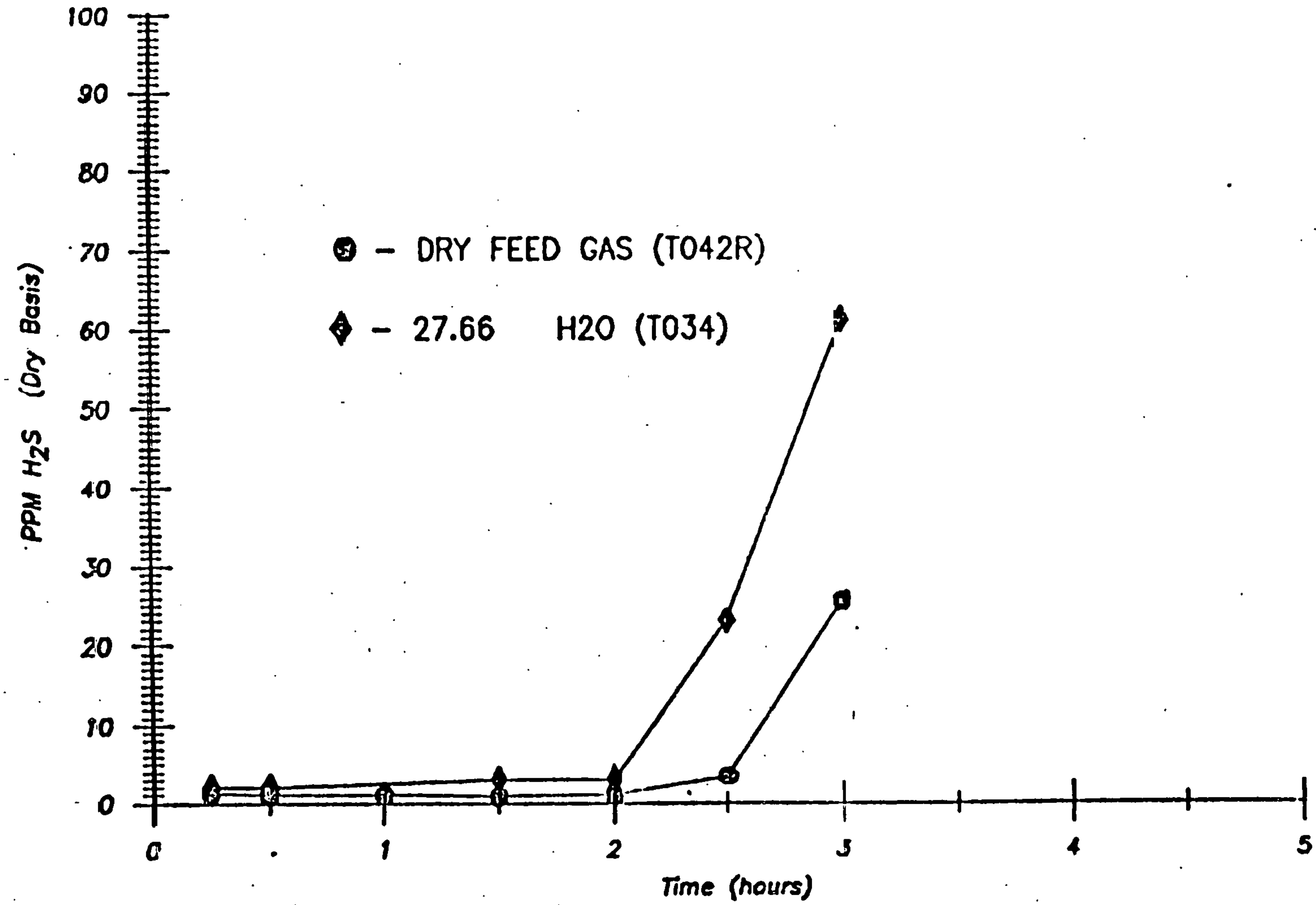
1. H2S LEVEL AT EXIT IS LOWER BY 50 % AS COMPARED TO TEST 034 WITH 27.66 % H20.

2. MEASURED EXIT H2S OF 1 PPM IS CLOSE TO THE CALCULATED EQUILIBRIUM VALUE OF 0.35 PPM (USING EQZNO)

P1

ZINC OXIDE SULFIDATION

TESTS 034,042R (HARSHAW ZN-0401)

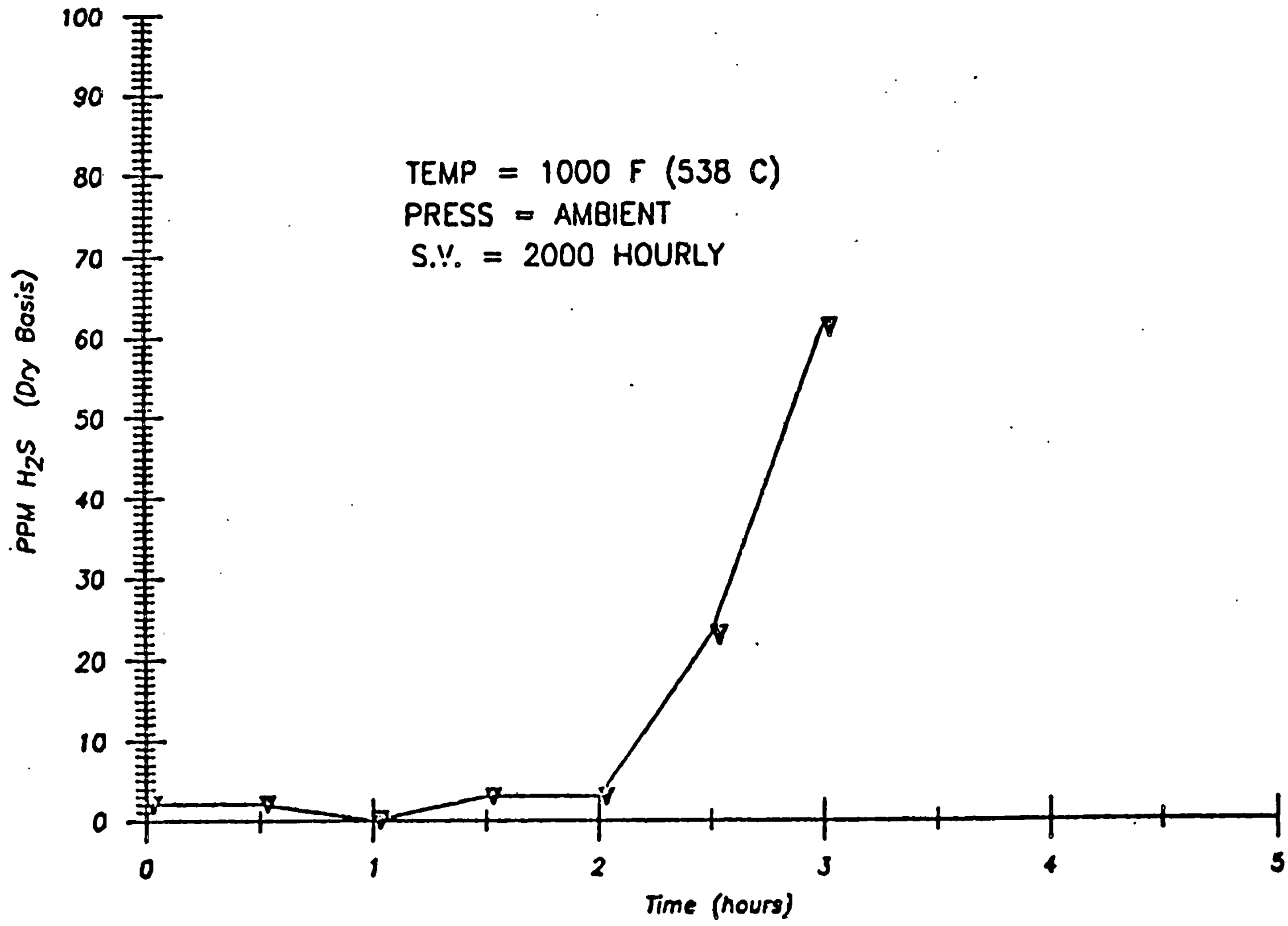


22-NOV-82 14:49:03

P:

ZINC OXIDE SULFIDATION

TEST 034 (HARSHAW ZN-0401)

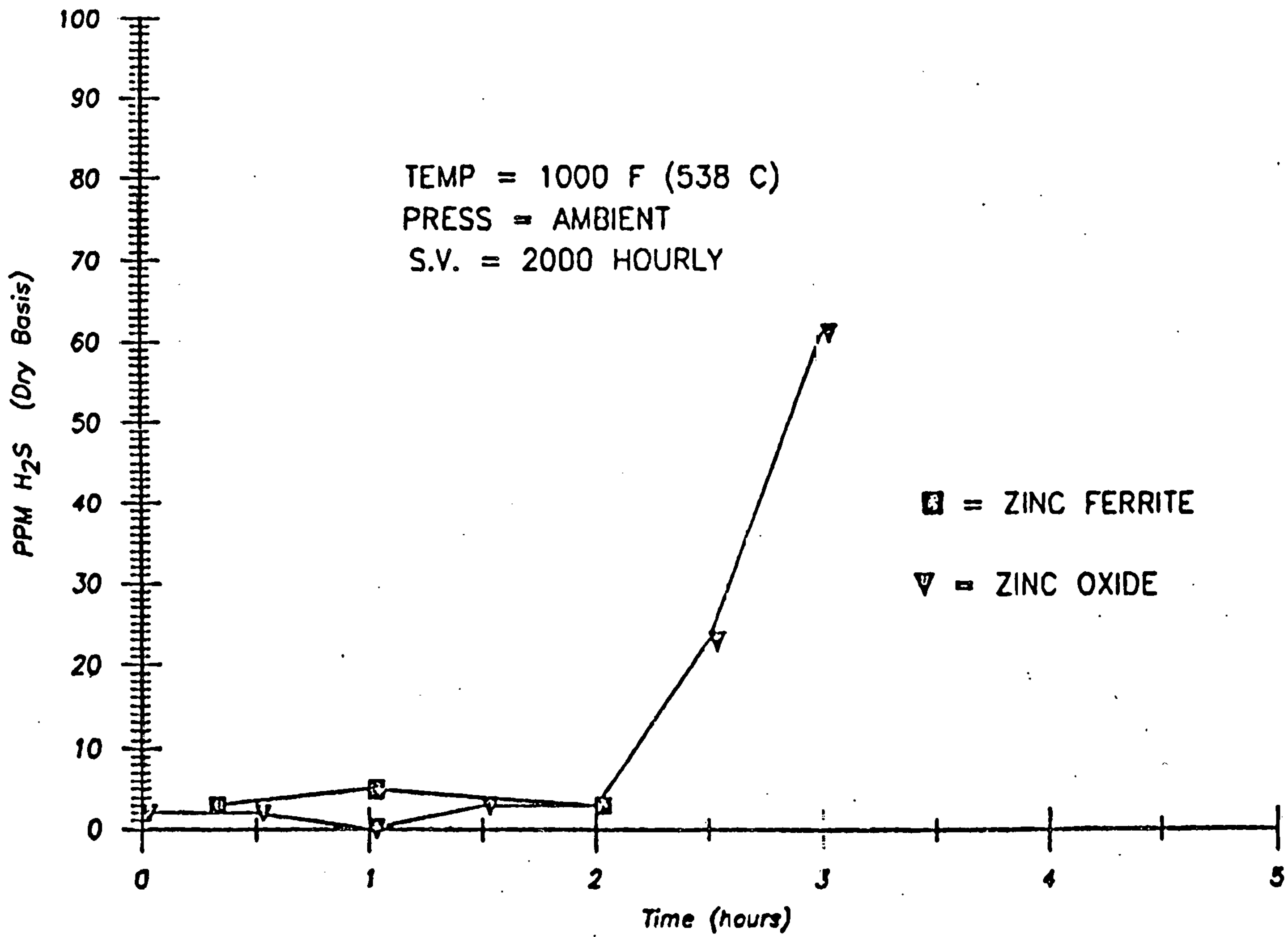


19-MAY-81 09:30:27

P1

ZINC OXIDE SULFIDATION

TESTS 034,021 (HARSHAW ZN-0401)

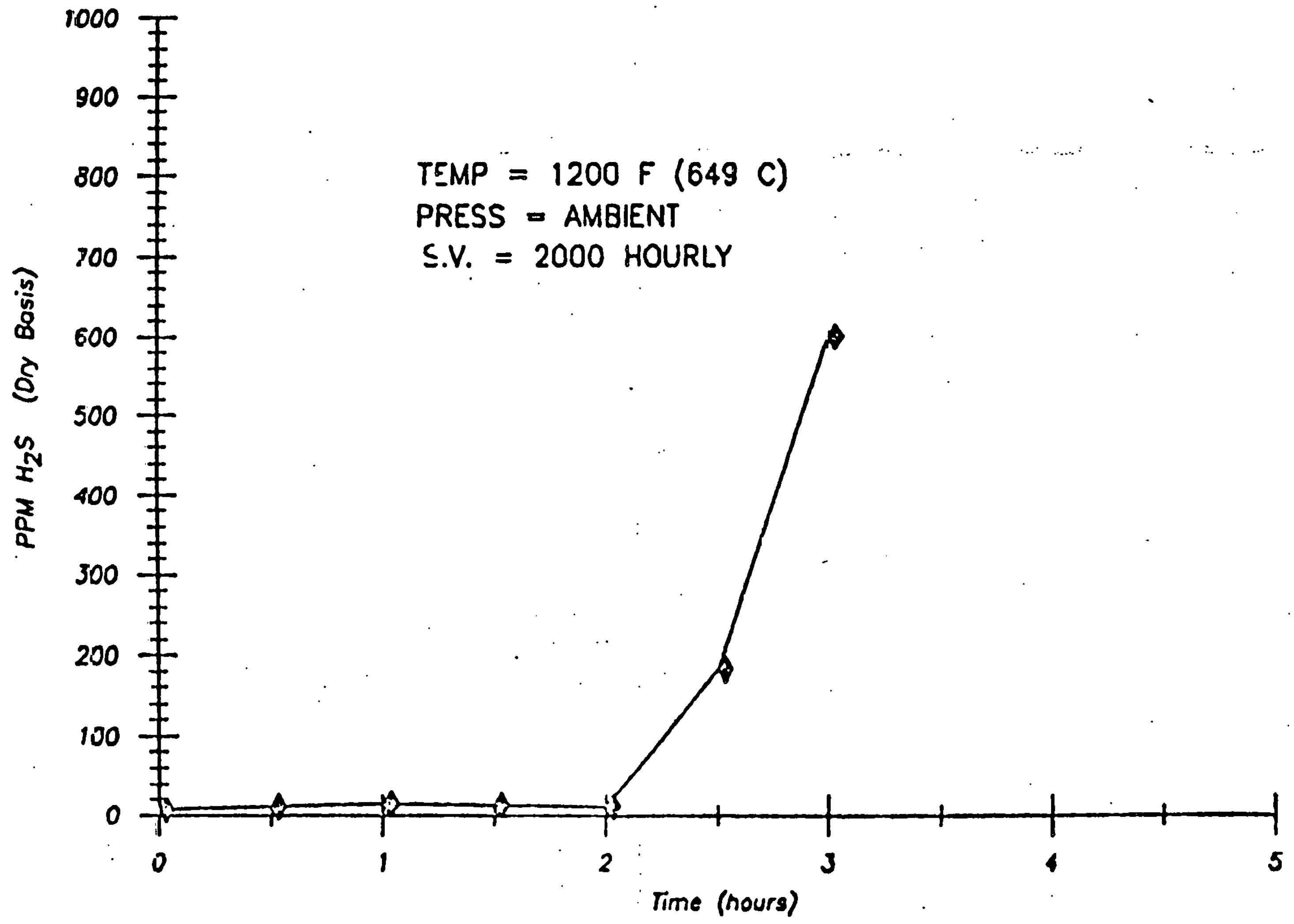


19-MAY-81 09:28:18

P1

ZINC OXIDE SULFIDATION

TEST 036 (HARSHAW ZN-0401)



19-MAY-81 09:26:42

TEST SUMMARY SHEET

TEST NO. : 043
DATE STARTED : 9/21/81
DATE ENDED : 9/21/81
TOTAL HOURS : 3.75 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #1 BATCH # 3
SORBENT COMPOSITION: 50 MOLE % IRON OXIDE (FISHER CERTIFIED)
50 MOLE % ZINC OXIDE (SHERWIN WILLIAMS)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 12.53 % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO
(AFTER 2 HOURS OF RUNNING)

OPERATING CONDITIONS

TEMPERATURE: 1000 F (537 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1442 HOURLY

	FLOW RATE	MOLE%
H2S	0.9963 SCFH	3.7%
H2O		
AIR		
CO2	3.6 SCFH	13.53 %
CO	4.3 SCFH	16.16 %
H2	6.3 SCFH	23.7%
CH4	1.07 SCFH	4.0%
N2	10.34 SCFH	38.87 %

TOTAL DRY GAS FLOW RATE = 26.6 SCFH

PURPOSE

SULFIDATION OF ZINC FERRITE PELLETS TO DETERMINE EXIT H2S COMPOSITION AT SORBTION TEMPERATURE OF 1000 F (537 C) USING DRY (NO WATER) LURGI GAS COMPOSITION .

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:	SQ M/G	
DENSITY :	G/CC	
POROSITY :		
X-RAY DIFFRACTION:		

DATA: (BY DETECTOR TUBE)

EXIT H2S: 0.7 PPM STEADY STATE

BREAKTHRU WITH 2 PPM AT 2.5 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FIRST SULFIDATION ON THIS LOAD OF SORBENT.
2. 5 STROKES USED ON DETECTOR TUBE SAMPLE PUMP.

CONCLUSIONS

1. ZINC FERRITE CAN REDUCE A DRY GAS STREAM TO LESS THAN 1 PPM.
2. ZINC FERRITE REDUCES THE H2S LEVEL DOWN TO THE SAME LEVEL AS ZINC OXIDE AT THE SAME CONDITIONS.
3. ZINC FERRITE REDUCES THE H2S LEVEL CLOSE TO THE ZINC OXIDE EQUILIBRIUM LEVEL OF 0.25 PPM

TEST SUMMARY SHEET

TEST NO. : 044
DATE STARTED : 8/14/81
DATE ENDED : 8/14/81
TOTAL HOURS : 3 HRS.
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE EXTRUSIONS WITH VANADIUM TO DETERMINE EXIT H2S COMPOSITION AT SORBTION TEMPERATURE OF 1000 F (537 C) LURGI GAS COMPOSITION

SORBENT TYPE/WEIGHT: (ZINC FERRITE + VANADIUM)/619 GM
SORBENT NO. :
SORBENT COMPOSITION: 49 MOLE % IRON OXIDE (FISHER CERTIFIED)
49 MOLE % ZINC OXIDE (SHERWIN WILLIAMS)
1.36 MOLE % ZNV2O4 (METC)
SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:	SQ M/G	
DENSITY :	G/CC	
POROSITY :		
X-RAY DIFFRACTION:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (537 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

DATA: (BY DETECTOR TUBE)

EXIT H2S: 1.2 TO 7.0 PPM STEADY STATE FOR 1.5 HRS
BREAKTHRU WITH 11.6 PPM AT 2 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE SCFH	CC/MIN	MOLE%
H2S	1.0 SCFH		3.7%
H2O		3.88	
AIR			
CO2	3.6 SCFH		9.68 %
CO	4.4 SCFH		11.91 %
H2	6.3 SCFH		17.12 %
CH4	1.1 SCFH		2.91 %
N2	12.2 SCFH		33.09 %

REMARKS

1. THIS IS THE FIRST SULFIDATION ON THIS LOAD OF SORBENT.

CONCLUSIONS

1. NO DETECTABLE EFFECT OF VANADIUM IN THE SORBENT

TEST SUMMARY SHEET

TEST NO. : 045
DATE STARTED : 8/21/81
DATE ENDED : 8/25/81
TOTAL HOURS : 10 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC OXIDE PELLETS FROM TEST 042 USING SPACE VELOCITY OF 600.

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 GM
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT 95 DRUM A/1
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 9.14 % (AVG)

EXIT S2:

EXIT H2:

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

REMARKS

1. NORMAL REGENERATION

2. AFTER COMPLETION OF THIS REGENERATION, ABSORBER TEMPERATURE WAS MAINTAINED AND SULFIDATION REPEATED

CONCLUSIONS

1. THIS IS THE FIRST REGENERATION ON THIS BATCH OF SORBENT.

TEST SUMMARY SHEET

TEST NO. : 046
DATE STARTED : 8/28/81
DATE ENDED : 8/28/81
TOTAL HOURS : 4 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/1000 GM
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A
SORBENT COMPOSITION: 100 % ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 8.09 % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

OPERATING CONDITIONS

TEMPERATURE: 1000 F (537 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 721 HOURLY

	FLOW RATE	MOLE%
H2S	0.50 SCFH	3.7%
H2O		
AIR		
CO2	1.8 SCFH	13.53 %
CO	2.2 SCFH	16.16 %
H2	3.2 SCFH	23.6%
CH4	.53 SCFH	4.0%
N2	5.17 SCFH	38.87 %

PURPOSE

SULFIDATION OF ZINC OXIDE PELLETS
TO DETERMINE EXIT H2S COMPOSITION AT SORBTION TEMPERATURE OF 1000 F (537 C) USING DRY (NO WATER) LURGI GAS COMPOSITION AT REDUCED SPACE VELOCITY (721 HOURLY)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:	SQ M/G	
DENSITY :	G/CC	
POROSITY :		
X-RAY DIFFRACTION:		

DATA: (BY DETECTOR TUBE)

EXIT H2S: 0.2 PPM STEADY STATE FOR 4 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SECOND SULFIDATION ON THIS LOAD OF SORBENT.
2. DUE TO THE LOWER SV USED, BREAKTHROUGH WAS NOT REACHED WITHIN 4 HOURS. THE STEADY STATE H2S LEVEL WAS THE PRIMARY OBJECTIVE.

CONCLUSIONS

1. THE EXIT H2S OF 0.2 PPM (AVG) COMPARES WELL WITH THE CALCULATED EQUILIBRIUM VALUE OF 0.35 PPM.

2. THIS SPACE VELOCITY (721 HOURLY) APPEARS TO ACHIEVE EQUILIBRIUM H2S AT 1000 F.

TEST SUMMARY SHEET

TEST NO. : 047
DATE STARTED : 9/ 1/81
DATE ENDED : 9/ 1/81
TOTAL HOURS : 4 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE PELLETS
TO PREPARE FOR REGENERATION AT 75% AIR AND 25 % STEAM
FOR RESOX COMPATIBILITY

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #1 BATCH #2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 12.53 % (GM SULFUR/GM FRESH SORBENT)
(FOR 2 HOURS OF RUNNING)

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: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE%
H2S	0.9963 SCFH	2.7%
H2O	3.872 CC/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	33.09 %

TOTAL DRY GAS FLOW RATE = 26.6 SCFH

DATA: (DETECTOR TUBE)

EXIT H2S: < 5 PPM FOR 2.5 HOURS
55 PPM AFTER 3 HOURS
EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS TEST IS TO PREPARE FOR REGENERATION AT 75% AIR 25% STEAM FOR RESOX COMPATIBILITY EVALUATION.

CONCLUSIONS

1. STANDARD SULFIDATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 048
DATE STARTED : 9/ 2/81
DATE ENDED : 9/ 2/81
TOTAL HOURS : 5 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 047 USING 75 % AIR AND 25 % STEAM.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #1 BATCH #2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 9.4 % (AVG)

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE%
H_2S		
H_2O	1.11 CC/MIN	25%
AIR	8.81 SCFH	75%
CO_2		
CO		
H_2		
CH_4		
N_2		

REMARKS

1. THIS TEST IS TO REGENERATE USING 75 % AIR 25% STEAM TO TEST COMPATIBILITY WITH RESOX PROCESS

2. TEMPERATURE WAVE AT INLET REACHED 1820 F. SINTERING DAMAGED SOME OF THE PELLETS AS OBSERVED VISUALLY.

CONCLUSIONS

1. 75 % AIR DURING REGENERATION AT SV = 600 HOURLY CAUSES TOO HIGH A TEMPERATURE WAVE.

2. LOWER AIR LEVELS SHOULD BE USED.

TEST SUMMARY SHEET

TEST NO. : 049
DATE STARTED : 9/11/81
DATE ENDED : 9/11/81
TOTAL HOURS : 1 HR
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE PELLETS TO CHECK FOR ANY LOSS OF ACTIVITY AFTER REGENERATION WITH 75% AIR AND 25 % STEAM FOR RESOX COMPATIBILITY

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX # 1 BATCH #2
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 3.13 % (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: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 6 PPM FOR 1/2 HOUR STEADY STATE

1150 PPM AFTER 1 HOUR

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE%
H2S	0.9953 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	33.09 %

REMARKS

1. RAPID BREAKTHROUGH OBSERVED WITH HIGH INITIAL H2S CONCENTRATION (6PPM).
2.

CONCLUSIONS

1. LOSS OF ACTIVITY OBSERVED IS DUE TO REGENERATION WITH 75 % AIR IN TEST U48 CAUSED BY SINTERING AT EXCESSIVE TEMPERATURES.

TEST SUMMARY SHEET

TEST NO. : 050
DATE STARTED : 9/22/81
DATE ENDED : 9/23/81
TOTAL HOURS : 8.25 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 043

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #1 BATCH #3/
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 10.4 - 16 %

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. NORMAL REGENERATION

CONCLUSIONS

TEST SUMMARY SHEET

TEST NO.	:	051
DATE STARTED	:	9/30/81
DATE ENDED	:	9/30/81
TOTAL HOURS	:	4.0 HRS
TYPE	:	SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #1 BATCH # 3
SORBENT COMPOSITION: 50 MOLE % IRON OXIDE (FISHER CERTIFIED)
50 MOLE % ZINC OXIDE (SHERWIN WILLIAMS)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 7.87 % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

OPERATING CONDITIONS

TEMPERATURE: 900 F (482 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 721 HOURLY

	FLOW RATE	MOLE%
H2S	0.500 SCFH	3.7%
H2O		
AIR		
CO2	1.8 SCFH	13.53 %
CO	2.15 SCFH	16.16 %
H2	3.15 SCFH	23.6%
CH4	0.535 SCFH	4.0%
N2	5.17 SCFH	38.87 %

TOTAL DRY GAS FLOW RATE = 13.3 SCFH

PURPOSE

SULFIDATION OF ZINC FERRITE PELLETS TO DETERMINE EXIT H2S COMPOSITION AT SORBTION TEMPERATURE OF 900 F (482 C) USING DRY (NO WATER) LURGI GAS COMPOSITION AT REDUCED SPACE VELOCITY OF 721 HR-1.

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:	SQ M/G	
DENSITY :	G/CC	
POROSITY :		
X-RAY DIFFRACTION:		

DATA: (BY DETECTOR TUBE)

EXIT H2S: 0.75 PPM AT STEADY STATE FOR 2.5 HRS
BREAKTHRU WITH 53 PPM AT 3.5 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SECOND SULFIDATION ON THIS LOAD OF SORBENT.
2.

CONCLUSIONS

1. H2S LEVEL APPROACHES ZNO EQUILIBRIUM LEVEL OF 0.1 PPM.
2. H2S LEVEL IS LOWER THAN TEST AT 1000 F , HOWEVER BREAKTHROUGH IS 2 HOURS SOONER.
3. KINETICS APPEAR SLOWER THAN AT 1000F, ALTHOUGH EQUILIBRIUM FAVORS LOWER H2S.

TEST SUMMARY SHEET

TEST NO. : 052
DATE STARTED : 10/01/81
DATE ENDED : 10/01/81
TOTAL HOURS : 9.75 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 051

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #1 BATCH #3/
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (FISHER CERTIFIED)
50 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 10.8-14.4 %

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

CONCLUSIONS

TEST SUMMARY SHEET

TEST NO.	:	053
DATE STARTED	:	10/14/81
DATE ENDED	:	10/14/81
TOTAL HOURS	:	5.0 HRS
TYPE	:	SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #1 BATCH # 3
SORBENT COMPOSITION: 50 MOLE % IRON OXIDE (FISHER CERTIFIED)
50 MOLE % ZINC OXIDE (SHERWIN WILLIAMS)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 14.16 % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

OPERATING CONDITIONS

TEMPERATURE: 1000 F (537C)

PRESSURE : AMBIENT

SPACE VELOCITY: 721 HOURLY

	FLOW RATE	MOLE%
H2S	0.500 SCFH	3.7%
H2O		
AIR		
CO2	1.8 SCFH	13.53 %
CO	2.15 SCFH	16.16 %
H2	3.15 SCFH	23.6%
CH4	0.535 SCFH	4.0%
N2	5.17 SCFH	38.87 %

PURPOSE

SULFIDATION OF ZINC FERRITE PELLETS
TO DETERMINE EXIT H2S COMPOSITION AT SORBTION TEMPERATURE OF 1000 F (537 C) USING DRY (NO WATER) LURGI GAS COMPOSITION AT REDUCED SPACE VELOCITY OF 721 HR-1.

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		
DENSITY :		
POROSITY :		
X-RAY DIFFRACTION:		

DATA: (BY DETECTOR TUBE)

EXIT H2S: 2.1 PPM AT STEADY STATE FOR 4.5 HRS
BREAKTHRU WITH 43 PPM AT 5 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

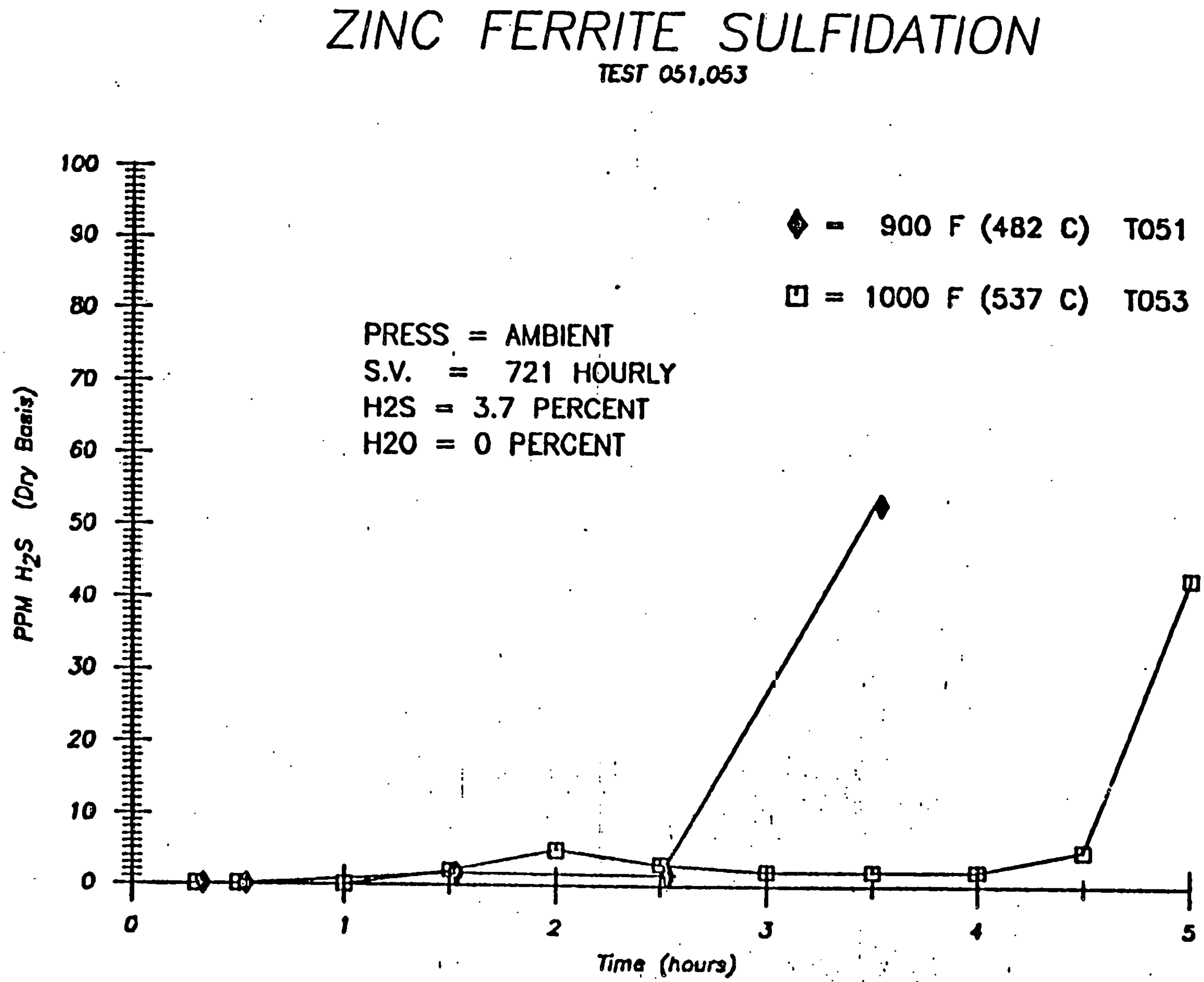
REMARKS

1. THIS IS THE THIRD SULFIDATION ON THIS LOAD OF SORBENT.

CONCLUSIONS

1. H2S LEVEL SLIGHTLY HIGHER THAN AT 900 F BUT BREAKTHROUGH IS 2 HOURS LATER.

2. LOWER S.V. INCREASES BREAKTHROUGH TIME BY 2 HOURS AS COMPARED TO S.V. 1442 HOURLY (T043).



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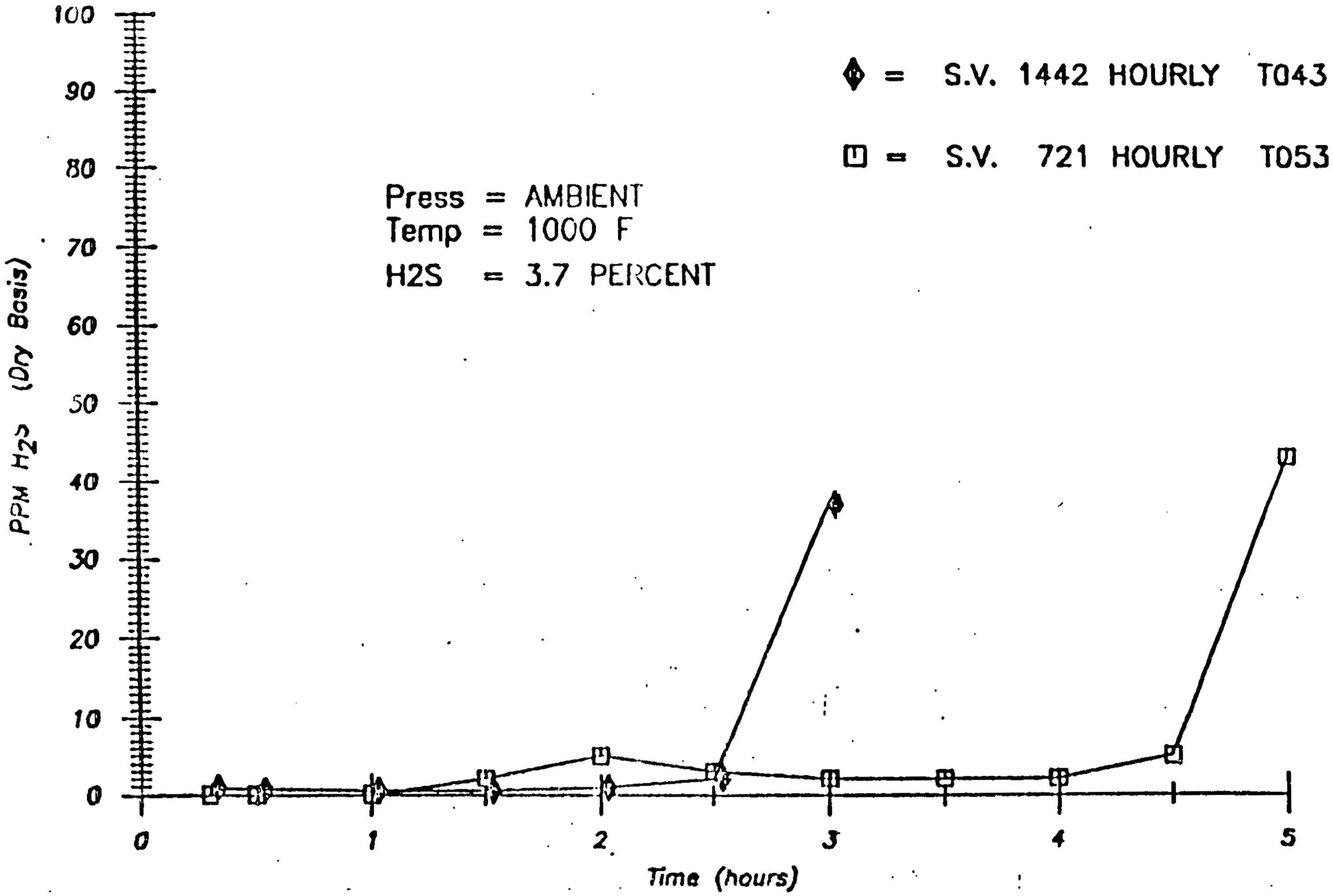
P1

ZINC FERRITE SULFIDATION

TEST 043,053

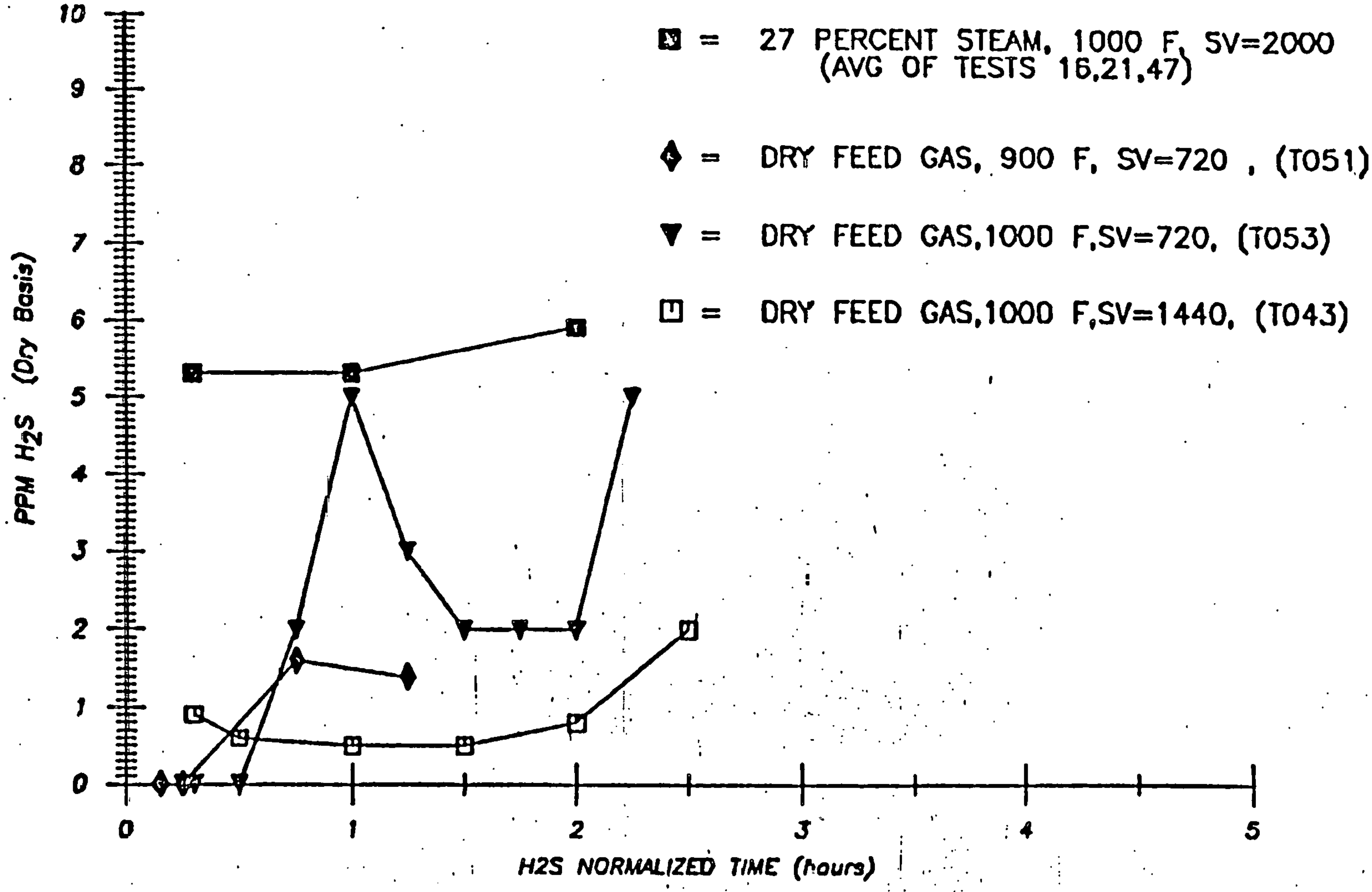


22-NOV-82 15:48:49

P1

ZINC FERRITE SULFIDATION

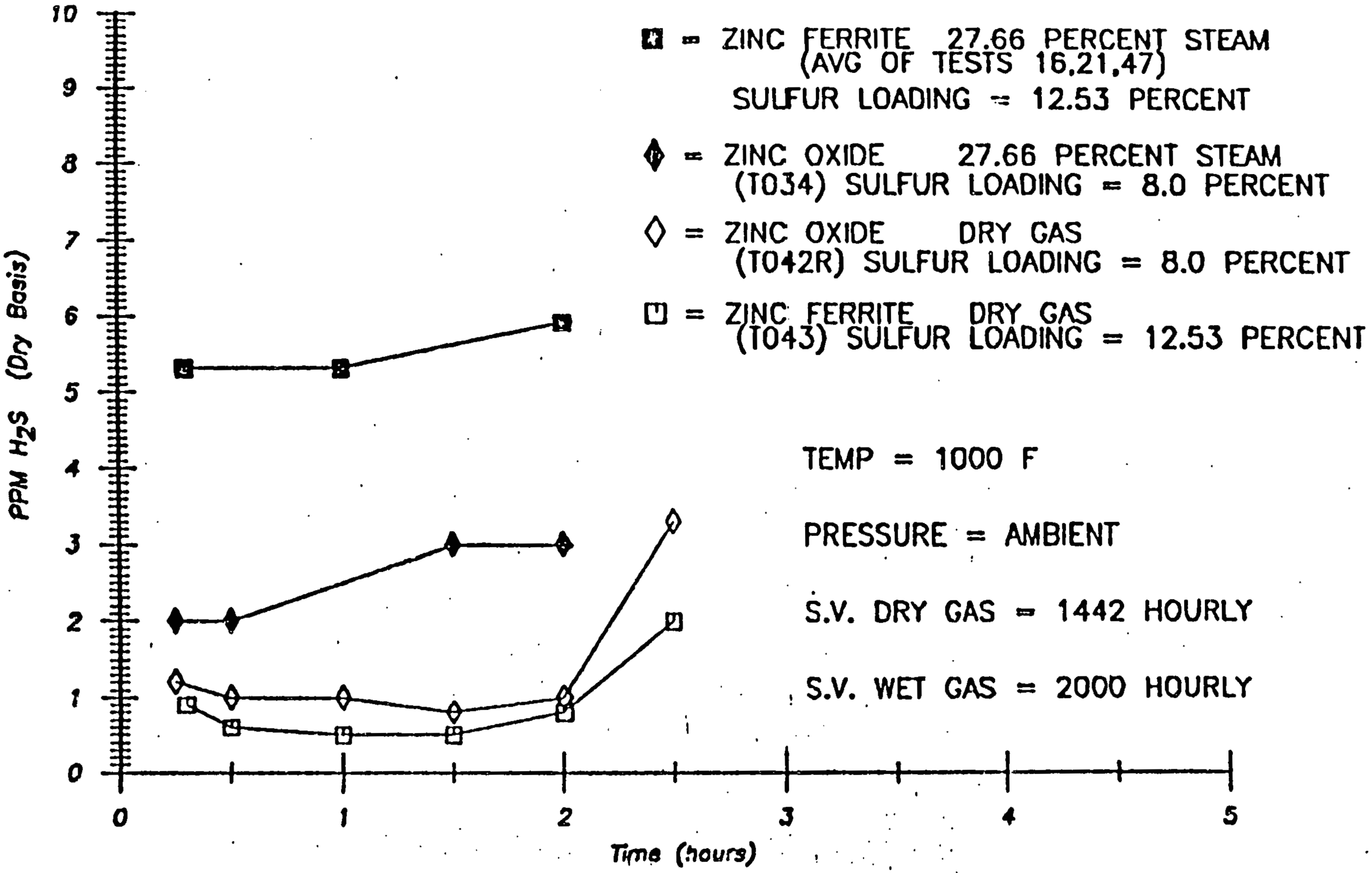
TESTS AVG(16,21,47),043,051,053



22-NOV-82 14:59:59

HOT GAS DESULFURIZATION

ZINC FERRITE AND ZINC OXIDE



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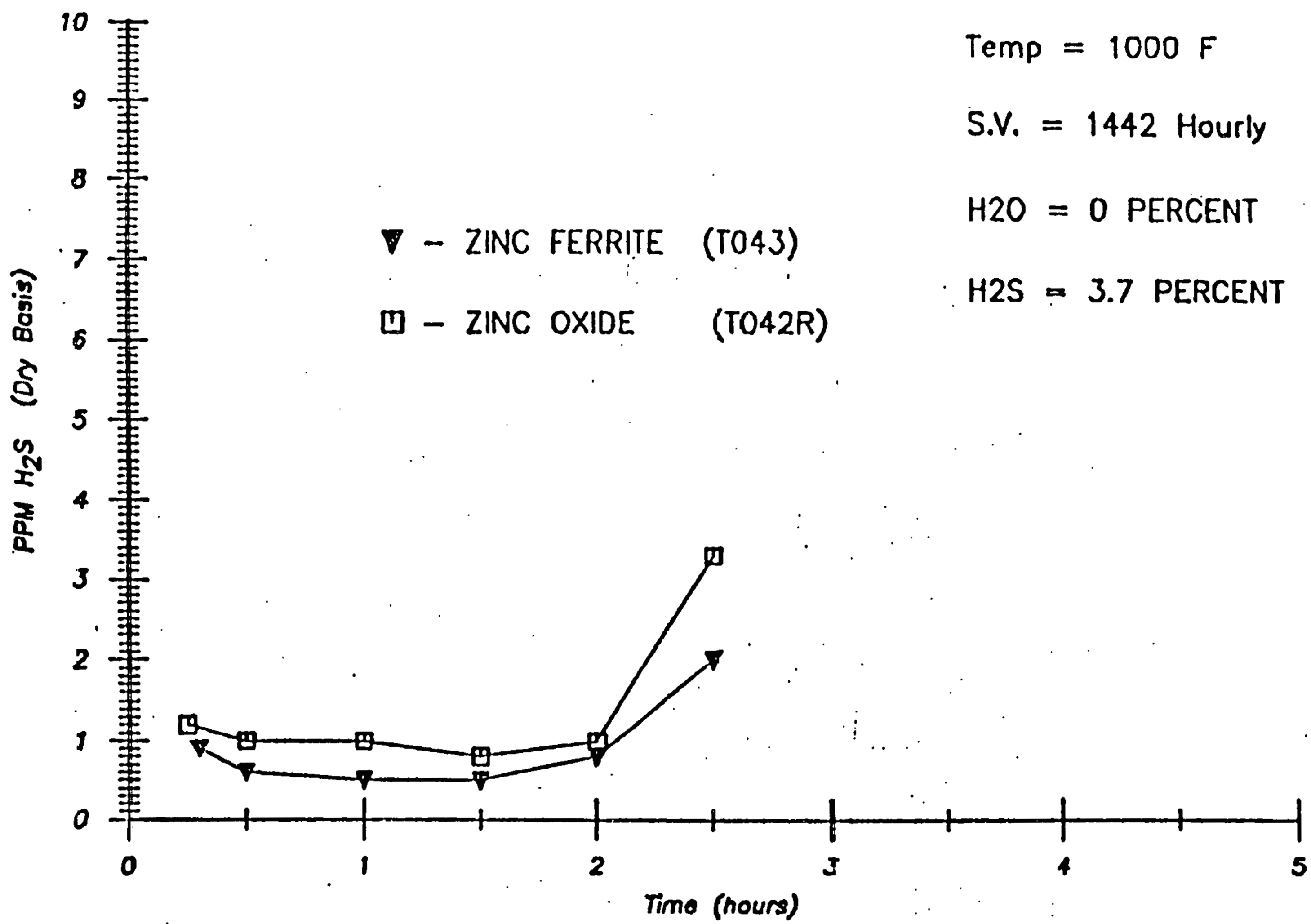
SULFIDATION

TESTS 043, 042R



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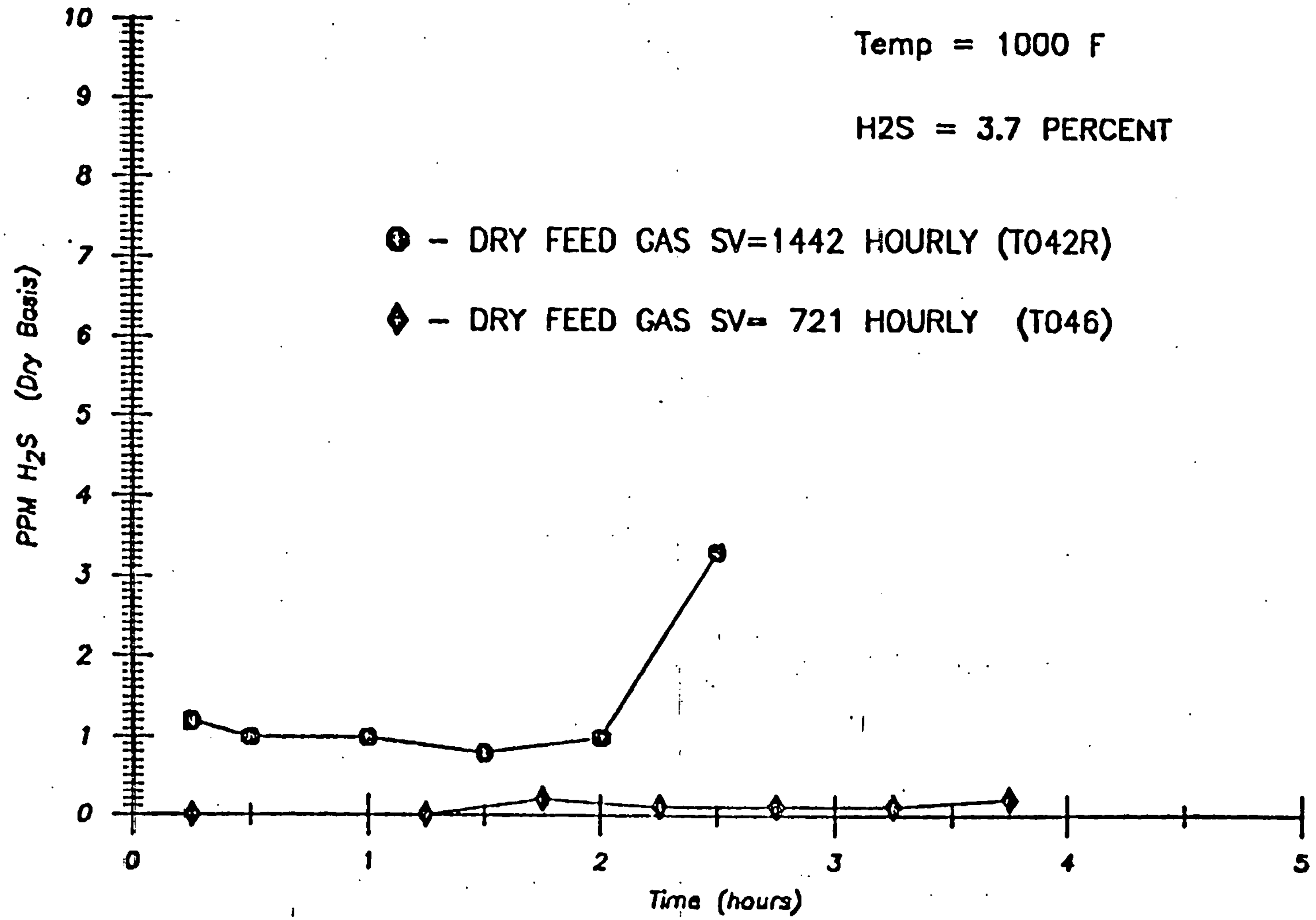
ZINC OXIDE SULFIDATION

TESTS 042R, 046 (HARSHAW ZN-0401)



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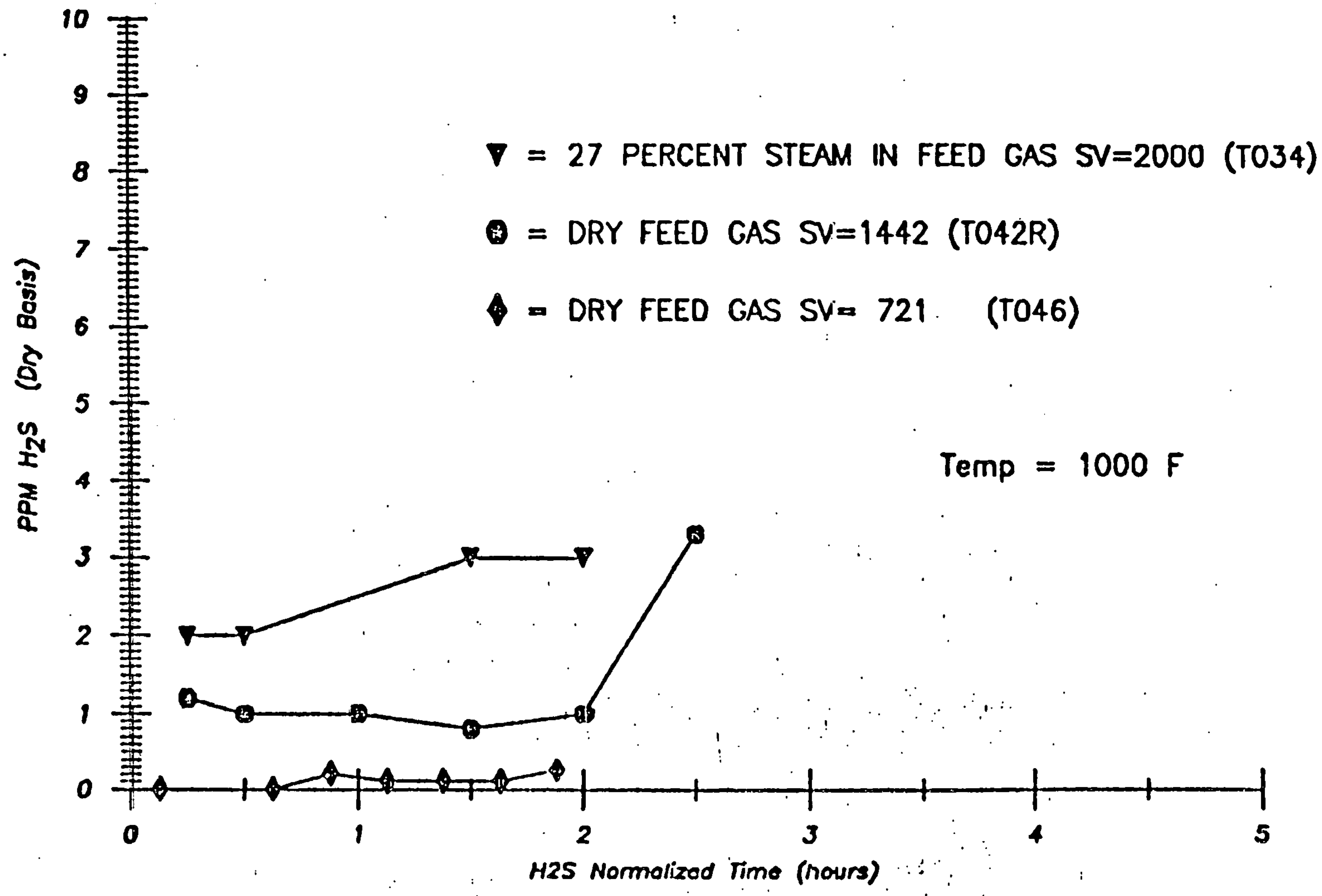
P1

ZINC OXIDE SULFIDATION

TESTS 042R, 046 , 034 (HARSHAW ZN-0401)



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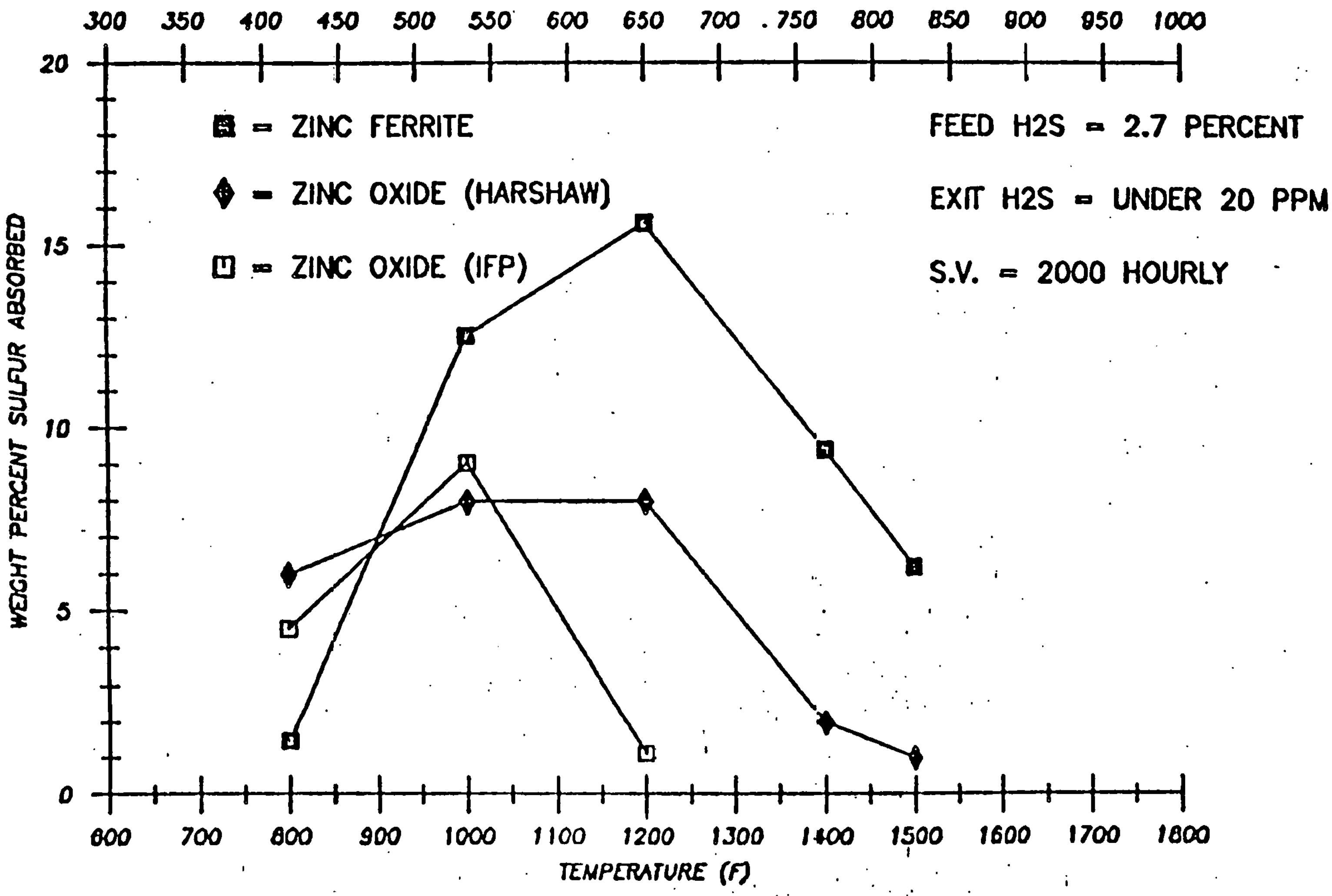
P1

SULFUR LOADING

ZINC FERRITE AND ZINC OXIDE



22-NOV-82 16:01:13

TEST SUMMARY SHEET

TEST NO. : 054
DATE STARTED : 10/16/81
DATE ENDED : 10/16/81
TOTAL HOURS : 3.25 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/500 GM
VANADIUM CATALYST/474 GM
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A
HARSHAW VANADIUM CATALYST V-0701
SORBENT COMPOSITION:

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : REDUCE VANADIUM CATALYST FOR 1 HOUR
SULFUR LOADING : 4.0 % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

OPERATING CONDITIONS

TEMPERATURE: 1200 F (649 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

	FLOW RATE	MOLE%
H2S	0.500 SCFH	2.7%
H2O	1.93 CC/MIN	27.66 %
AIR		
CO2	1.8 SCFH	9.79 %
CO	2.15 SCFH	11.69 %
H2	3.15 SCFH	17.13%
CH4	0.535 SCFH	2.91%
N2	5.17 SCFH	28.12 %

TOTAL DRY GAS FLOW RATE = 13.3 SCFH
TOTAL WET GAS FLOW RATE = 18.38 SCFH

PURPOSE

DETERMINE THE CAPABILITY OF A VANADIUM CATALYST TO LOWER THE H2S LEVEL BELOW 10 PPM AFTER A ZINC OXIDE BED.

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		
DENSITY :		
POROSITY :		
X-RAY DIFFRACTION:		

DATA: (BY DETECTOR TUBE)

EXIT H2S: 100 PPM FOR 2 HRS STEADY STATE

BREAKTHRU WITH 126 PPM AT 1.75 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. HALF THE REACTOR IS FILLED WITH ZNO AND HALF WITH VANADIUM CATALYST.
2. REDUCTION WILL BE CARRIED OUT ON THE GASES AND FLOWS SPECIFIED AT LEFT WITHOUT H2S.
3. FIRST SULFIDATION

CONCLUSIONS

1. VANADIUM CATALYST DOES NOT REDUCE H2S BELOW 10 PPM.

TEST SUMMARY SHEET

TEST NO. : 055
DATE STARTED : 10/16/81
DATE ENDED : 10/18/81
TOTAL HOURS : 3.5 HRS
TYPE : SULFIDATION

PURPOSE

DETERMINE THE CAPABILITY OF HALF A ZINC OXIDE BED TO LOWER THE H2S LEVEL BELOW 10 PPM

SORBENT TYPE/WEIGHT: ZINC OXIDE/500 GM

SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A

SORBENT COMPOSITION:

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 6.07 % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		
DENSITY :		
POROSITY :		
X-RAY DIFFRACTION:		

OPERATING CONDITIONS

TEMPERATURE: 1200 F (649 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE%
H2S	0.500 SCFH	2.7%
H2O	1.93 CC/MIN	27.65 %
AIR		
CO2	1.8 SCFH	9.79 %
CO	2.15 SCFH	11.69 %
H2	3.15 SCFH	17.13%
CH4	0.535 SCFH	2.91%
N2	5.17 SCFH	28.12 %

TOTAL DRY GAS FLOW RATE = 13.3 SCFH
TOTAL WET GAS FLOW RATE = 18.39 SCFH

DATA: (BY DETECTOR TUBE)

EXIT H2S: 7 PPM FOR 1.5 HRS STEADY STATE

BREAKTHRU WITH 13 PPM AT 1.75 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. HALF THE REACTOR IS FILLED WITH ZNO
2. THIS TEST IS TO VERIFY TE EXIT LEVEL OF H2S USING HALF A BED OF ZNO.

CONCLUSIONS

1. ZNO LOWERS H2S BELOW 10 PPM FOR 1.5 HRS AT 1200F .

TEST SUMMARY SHEET

TEST NO. : 056
DATE STARTED : 10/23/81
DATE ENDED : 10/23/81
TOTAL HOURS : 4.75 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC OXIDE AND VANADIUM CATALYST PELLETS FROM TEST 054.

SORBENT TYPE/WEIGHT: ZINC OXIDE/500 GM
VANADIUM CATALYST/474 GM
SORBENT NO. : HARSHAW ZNO C401 E3/16 LOT95 DRUM A
HARSHAW VANADIUM CATALYST V-0701
SORBENT COMPOSITION:

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : NONE
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: 600 HOURLY

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 9.2 - 14.2 %

EXIT S2:

EXIT H2:

REMARKS

1. FIRST REGENERATION OF VANADIUM CATALYST.

CONCLUSIONS

TEST SUMMARY SHEET

TEST NO. : 057
DATE STARTED : 10/26/81
DATE ENDED : 10/26/81
TOTAL HOURS : 3.5 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/500 GM
VANADIUM CATALYST/474 GM
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A
HARSHAW VANADIUM CATALYST V-0701
SORBENT COMPOSITION:

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : REDUCE VANADIUM CATALYST FOR 1 HOUR
SULFUR LOADING : 5.19 % (GM SULFUR/GM FRESH SORBENT)
(MOLE S/MOLE ZNO

OPERATING CONDITIONS

TEMPERATURE: 1200 F (649 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

	FLOW RATE	MOLE%
H2S	0.500 SCFH	2.7%
H2O	1.93 CC/MIN	27.66 %
AIR		
CO2	1.8 SCFH	9.79 %
CO	2.15 SCFH	11.69 %
H2	3.15 SCFH	17.13%
CH4	0.535 SCFH	2.91%
N2	5.17 SCFH	28.12 %

TOTAL DRY GAS FLOW RATE = 13.3 SCFH
TOTAL WET GAS FLOW RATE = 18.33 SCFH

PURPOSE

DETERMINE THE CAPABILITY OF A VANADIUM CATALYST TO LOWER THE H2S LEVEL BELOW 10 PPM AFTER A ZINC OXIDE BED.

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		
DENSITY :		
POROSITY :		
X-RAY DIFFRACTION:		

DATA: (BY DETECTOR TUBE)

EXIT H2S: 11-23 PPM FOR 2.5 HRS STEADY STATE
BREAKTHRU WITH 30 PPM AT 3 HRS
EXIT SO2:
EXIT S2:
EXIT H2:

REMARKS

1. HALF THE REACTOR IS FILLED WITH ZNO AND HALF WITH VANADIUM CATALYST.
2. REDUCTION WILL BE CARRIED OUT ON THE GASES AND FLOWS SPECIFIED AT LEFT WITHOUT H2S.
3. BOTH ZNO AND VANADIUM HAVE BEEN REGENERATED.
4. SECOND SULFIDATION.

CONCLUSIONS

1. VANADIUM CATALYST (V-0701) DOES NOT REDUCE H2S BELOW 10 PPM.

TEST SUMMARY SHEET

TEST NO. : 058
DATE STARTED : 10/30/81
DATE ENDED : 10/30/81
TOTAL HOURS : 6 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC OXIDE AND VANADIUM CATALYST PELLETS FROM TEST 057.

SORBENT TYPE/WEIGHT: ZINC OXIDE/500 GM
VANADIUM CATALYST/474 GM
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A
HARSHAW VANADIUM CATALYST V-0701
SORBENT COMPOSITION:

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : NONE
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: 600 HOURLY

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 5 - 12 %

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SECOND REGENERATION ON THIS SORBENT.

- CONCLUSIONS

TEST SUMMARY SHEET

TEST NO. : 059
DATE STARTED : 11/03/81
DATE ENDED : 11/03/81
TOTAL HOURS : 3 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/500 GM
VANADIUM CATALYST/474 GM
SORBENT NO. : HARSHAW ZNO 0401 E3/16 LOT95 DRUM A
HARSHAW VANADIUM CATALYST V-0701
SORBENT COMPOSITION:

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT : REDUCE VANADIUM CATALYST FOR 3 HOUR
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

OPERATING CONDITIONS

TEMPERATURE: 1200 F (649 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY

	FLOW RATE	MOLE%
H2S	0.500 SCFH	2.7%
H20	1.93 CC/MIN	27.66 %
AIR		
CO2	1.8 SCFH	9.79 %
CO	2.15 SCFH	11.69 %
H2	3.15 SCFH	17.13%
CH4	0.535 SCFH	2.91%
N2	5.17 SCFH	28.12 %

TOTAL DRY GAS FLOW RATE = 13.3 SCFH
TOTAL WET GAS FLOW RATE = 18.38 SCFH

PURPOSE

DETERMINE THE CAPABILITY OF A VANADIUM CATALYST TO LOWER THE H2S LEVEL BELOW 10 PPM AFTER A ZINC OXIDE BED. (SECOND TIME)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		
DENSITY :		
POROSITY :		
X-RAY DIFFRACTION:		

DATA: (BY DETECTOR TUBE)

EXIT H2S: 1000-2000 PPM DURING REDUCTION

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. HALF THE REACTOR IS FILLED WITH ZNO AND HALF WITH VANADIUM CATALYST.
2. REDUCTION WILL BE CARRIED OUT ON THE GASES AND FLOWS SPECIFIED AT LEFT WITHOUT H2S.
3. BOTH ZNO AND VANADIUM HAVE BEEN REGENERATED.

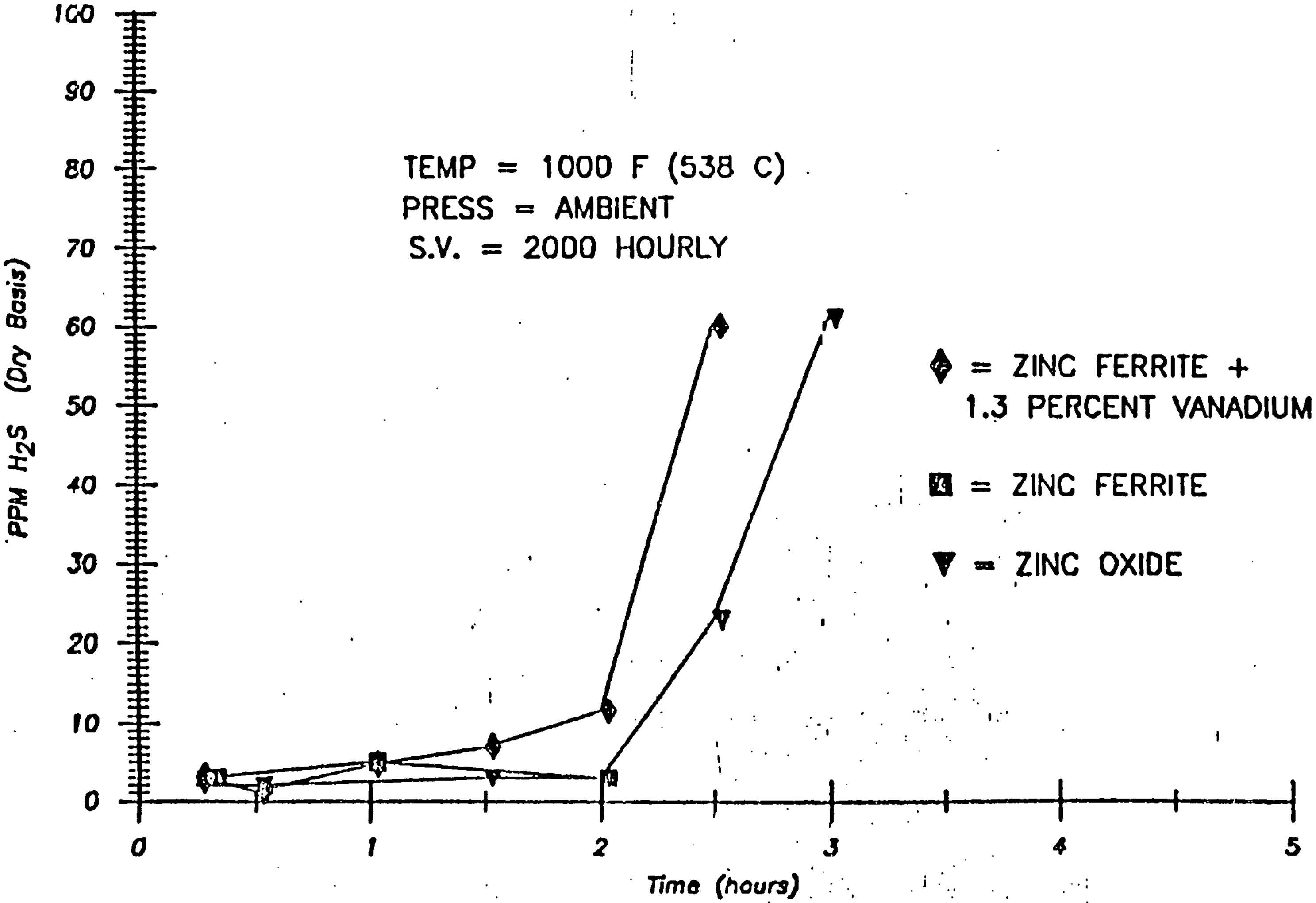
CONCLUSIONS

1. H2S LEVEL TOO HIGH (1000-2000) DURING REDUCTION.
2. TEST ABORTED.

P1

ZINC FERRITE SULFIDATION

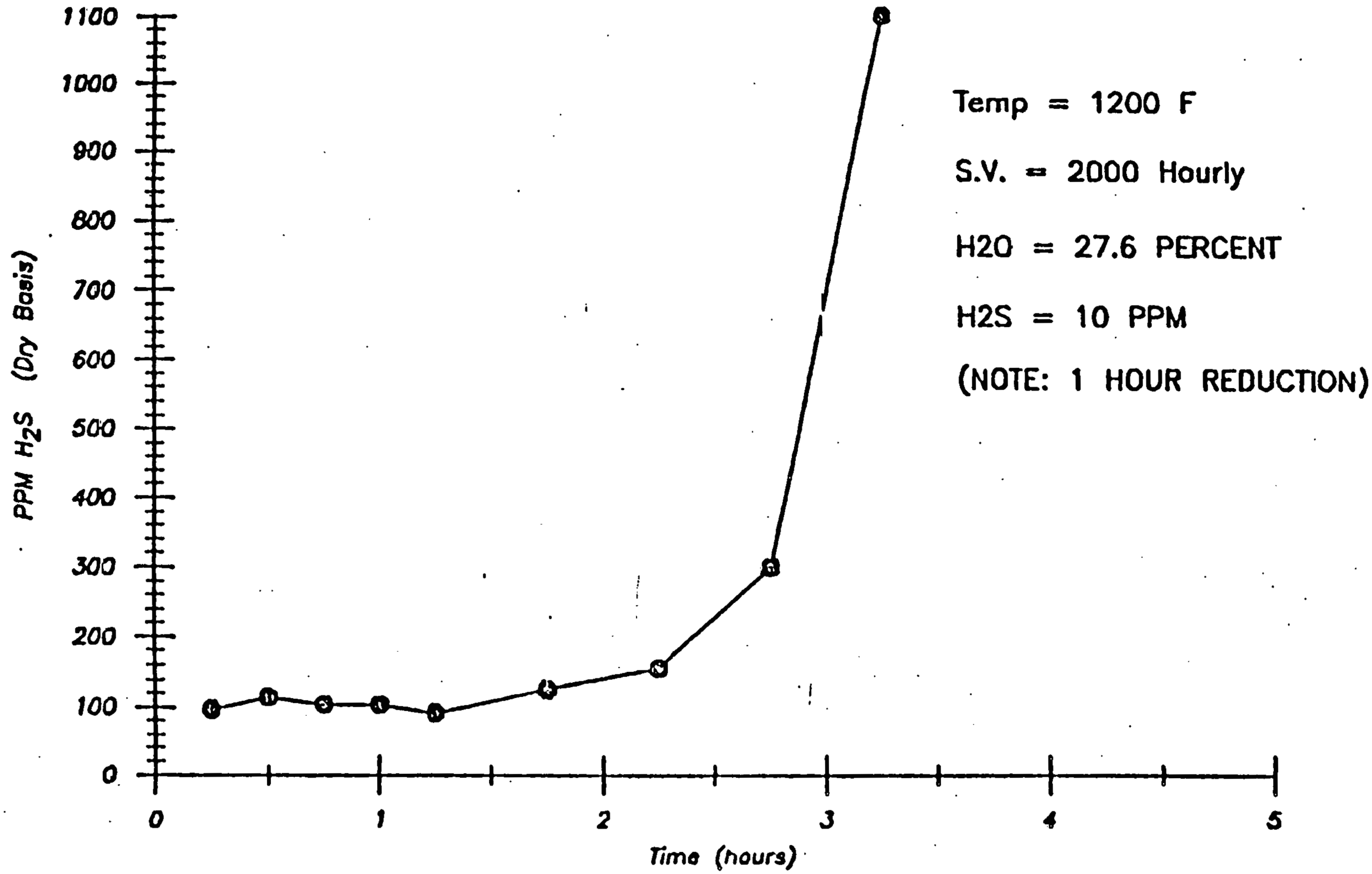
TESTS 044,034,021



22-NOV-82 15:11:50

VANADIUM SULFIDATION

TEST 054 (V-0701)

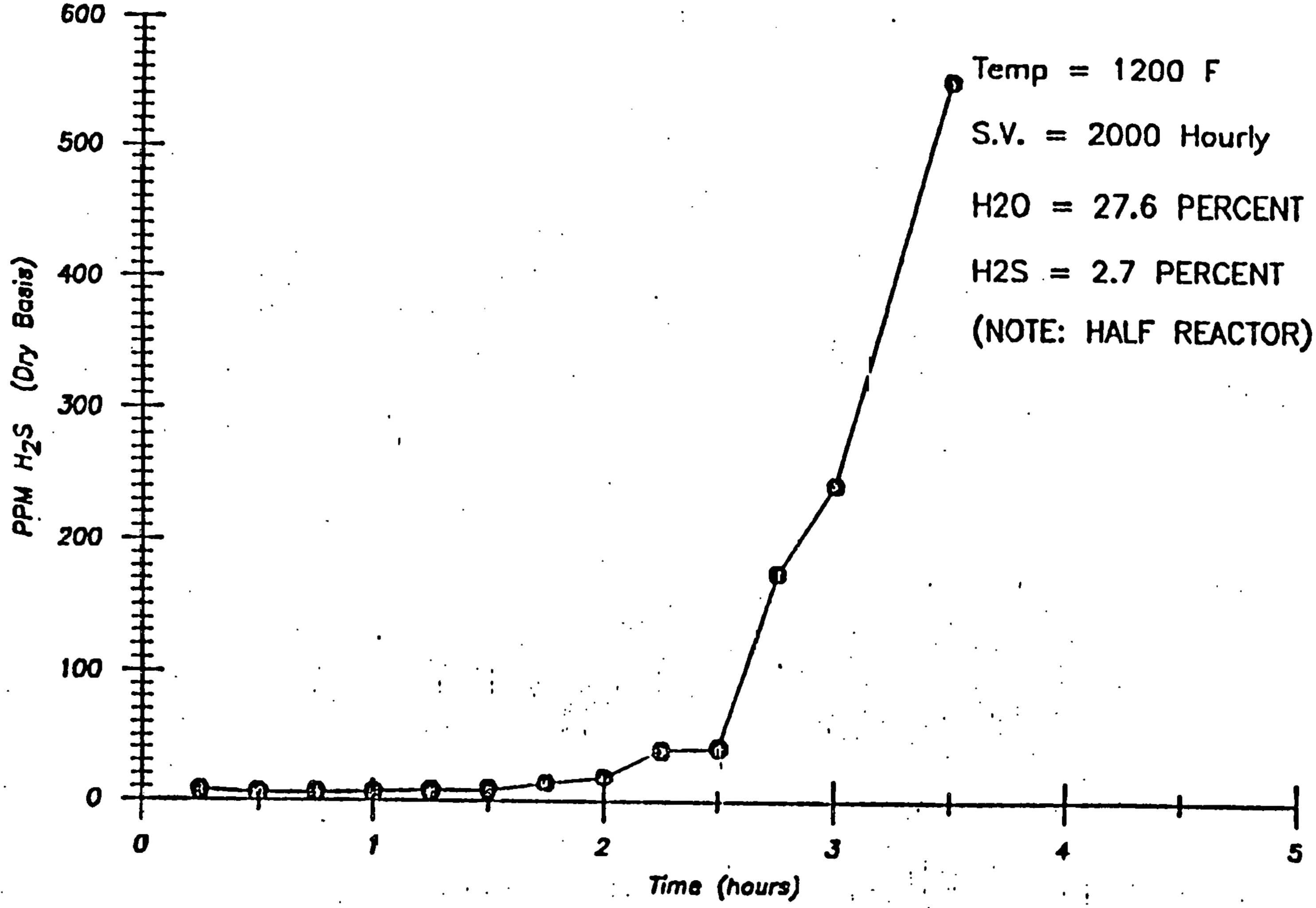


22-NOV-82 16:08:05

P1

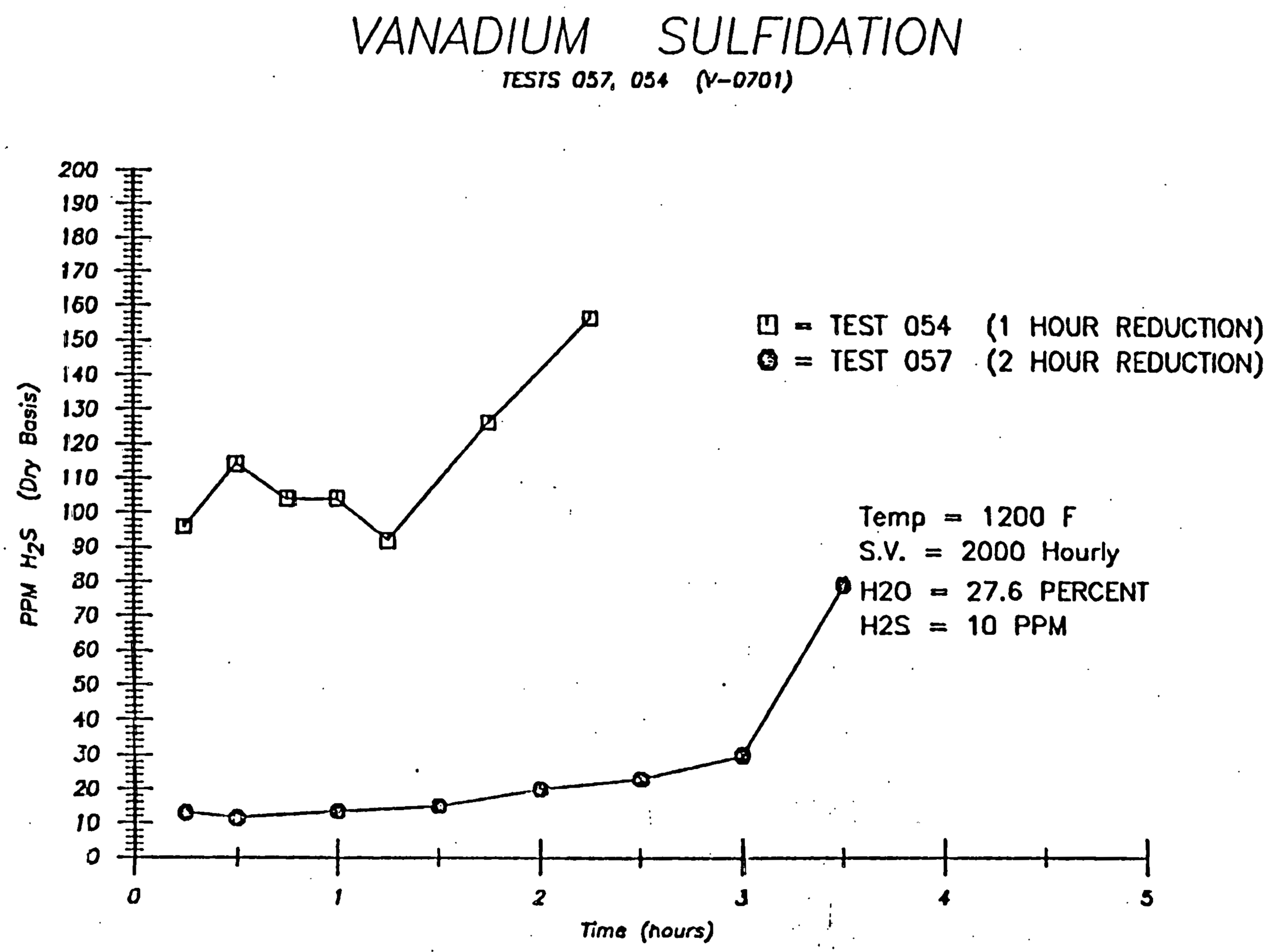
ZINC OXIDE SULFIDATION

TEST 055 (HARSHAW ZN-0401)



22-NOV-82 16:15:26

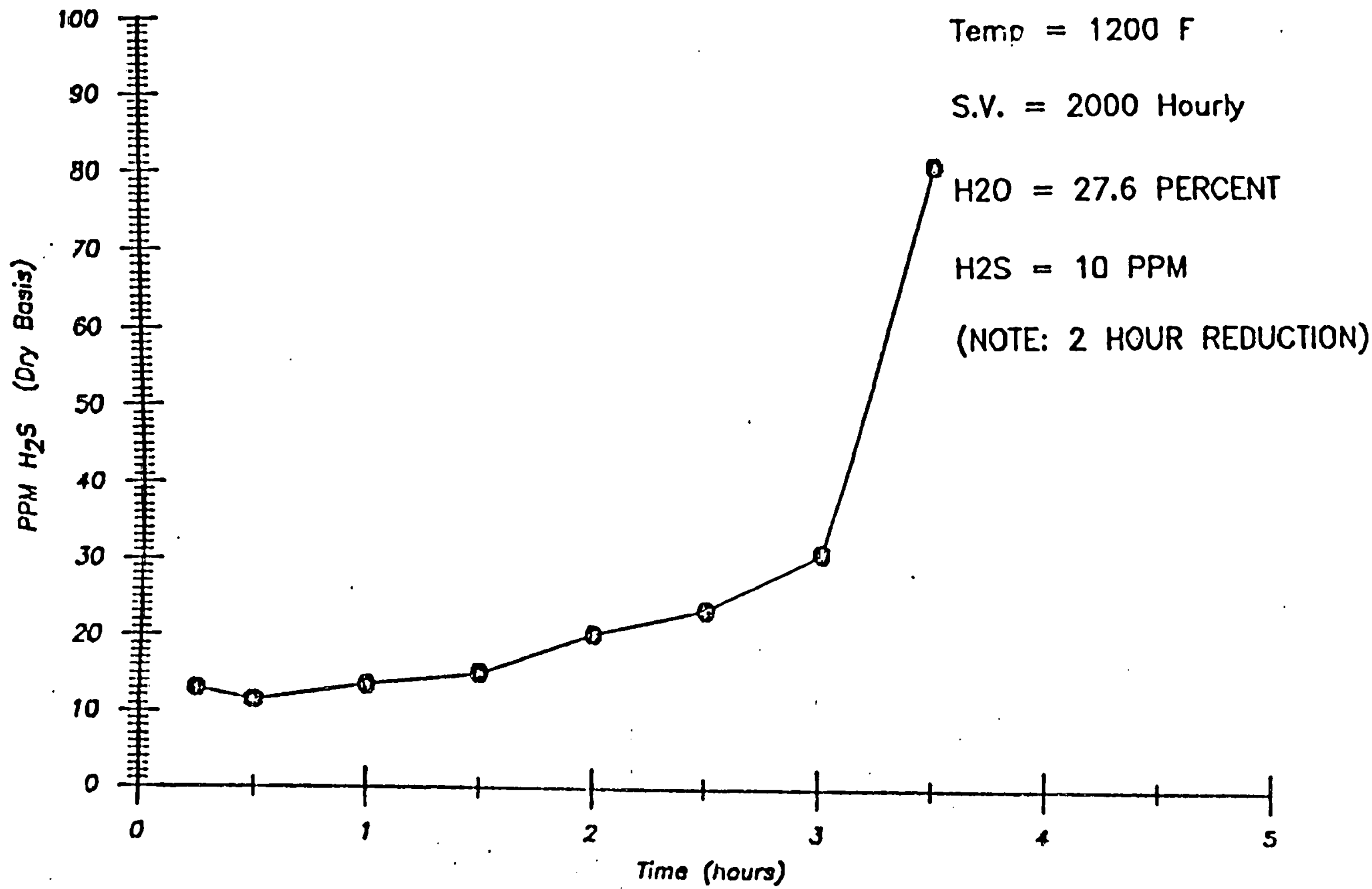
P1



22-NOV-82 16:20:49

VANADIUM SULFIDATION

TEST 057 (V-0701)



TEST SUMMARY SHEET

TEST NO. : 060
DATE STARTED : 11/05/81
DATE ENDED : 11/05/81
TOTAL HOURS : 1.5 HRS
TYPE : SULFIDATION

PURPOSE

DETERMINE THE CAPABILITY OF HALF A BED OF FRANKLINITE ORE TO LOWER THE H2S LEVEL BELOW 10 PPM

SORBENT TYPE/WEIGHT: FRANLINITE ORE/1027 GM

SORBENT NO. : MIX 2 BATCH #1

SORBENT COMPOSITION: FRANLINITE ORE SEPARATED MAGNETICALLY FROM ZINC ORE

SORBENT PELLET SIZE: SPHERES LESS THAN 1/2 INCH DIAM
PRETREATMENT : NONE
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)
MOLE S/MOLE ZNO

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		.8583 SQ M/G
DENSITY :		5.440 G/CC
POROSITY :		
X-RAY DIFFRACTION:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F (537 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

DATA: (BY DETECTOR TUBE)

EXIT H2S: 4080 PPM AT 15 MINUTES STEADY STATE

BREAKTHRU IMMEDIATE

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLES
H2S	0.500 SCFH	2.7%
H2O	1.93 CC/MIN	27.66 %
AIR		
CO2	1.8 SCFH	9.79 %
CO	2.15 SCFH	11.69 %
H2	3.15 SCFH	17.13%
CH4	0.535 SCFH	2.91%
N2	5.17 SCFH	28.12 %

TOTAL DRY GAS FLOW RATE = 13.3 SCFH
TOTAL WET GAS FLOW RATE = 18.38 SCFH

REMARKS

1. HALF THE REACTOR IS FILLED WITH FRANLINITE ORE
2. BED HIGHT IS 9 1/8 INCH.

CONCLUSIONS

1. FRANKLINITE ORE IN THIS FORM IS NOT A GOOD SORBENT.
2. SUSPECT LOW SURFACE AREA A PROBLEM.

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DIRECTION PHYSICO-CHIMIE APPLIQUEE

July 6, 1982

Object : Your letter TG-dp-512h

Dear Mr. GRINDLEY,

Thank you very much for the sample of IFP desulfurization sorbent MEP-682 and the draft report on the tests you have performed on it.

Concerning your tests and the comparison of the performances of our sorbent with those of Harshaw catalyst we have the following comments and questions:

- the IFP sorbent is designed for the removal of the bulk of the H_2S from a gazifier effluent before using it in a gas turbine for electricity production by combined cycles. It is not intended for the removal of traces of H_2S. The selection criterion used in our work was the sorbent capability to perform a great number of cycles with a good mechanical strength, a sulfur capacity in the range of 8-15 % weight and a H_2S concentration level in the treated gas of less than 300 ppm Vol.,

- you said that Harshaw sorbent is not intended for regeneration and contains no strengthening agents. Are the results on Harshaw sorbent presented in figures 5 and 6 obtained with fresh samples or after several regenerations ? We would appreciate if you could send us a copy of your report IR-N°1133 in which the results of these tests are related in detail and other reports which could be available on this subject,

- we do not believe that the degradation of IFP sorbent could be attributed to its sulfidation at 649°C. We have performed ourselves several sulfidations up to 800°C without noticing any detrimental effect on the further runs at 500°C or 600°C. We agree that the tests results are obtained in the 500°C - 600°C temperature range but we consider that the drop of the sulfur capacity versus temperature given in figure 6 is not representative of the performances of our sorbent when utilized for what it is designed,

- according to our experience the performances and the duration of a regenerable sorbent are closely related to the conditions used for its regeneration. In your test the low inlet temperature of the oxidizing gas (538°C) favorizes the formation of zinc sulfate which is later reduced in the sulfidation step

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with emission of SO_2. This emission increase with the temperature of the sulfidation step and affects the level of desulfurization.

In addition, the high oxygen content of the oxidizing gaz (10 %) allows the sorbent to reach a temperature far higher than the reported 816°C, inducing sintering.

In our tests we use an inlet temperature of 700°C and an oxygen content of 2 % vol. (maximum temperature 900°C). With these conditions we observe a rapid decrease of the sulfur capacity during the firsts cycles corresponding to the chemical stabilization of the binder (formation of Ca SO_4 and Zn Al_2O_4) and then stable performances over 50 cycles (sulfur capacity about 10 % weight for breakthrought at 200 ppm vol. H_2S, at 600°C and GHSV 1000).

- the analyses of IFP sorbent presented in table 6 are not consistent with the statement that the IFP sorbent "has been irreversibly damaged during the excursion at 1200°F". It is probably a question of level of H_2S concentration at which you consider that there is breakthrought,

- could you specify if the very surprising figures obtained for zinc carry-over are specific to the IFP sorbent or not.

We hope that these comments will lead you to modify some negative aspects of your conclusion on our MEP-682 sorbent.

In fact we do not intend to commercialize this sorbent, mainly for zinc entrainment problems due to Zn S and Zn vapor pressures. We have recently restarted our researches in this field and we are working on iron and calcium based sorbents with the target of defining the bases of a regenerative hot fuel gas desulfurization process.

In this view we are very interested in keeping in contact with you and exchanging informations.

Sincerely,

A. DESCHAMPS

TEST SUMMARY SHEET

TEST NO. : 061
DATE STARTED : 11/9/81
DATE ENDED : 11/9/81
TOTAL HOURS : 2.5 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/892 GM
SORBENT NO. : IFP /M MEP 682
SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3 MM DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR LOADING : 6.77 % (GM SULFUR/GM FRESH SORBENT)

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE%
H2S	0.9963 SCFH	2.7%
H2O	3.872CC/MIN	27.66%
AIR		
CO2	3.6 SCFH	9.69 %
	4.3 SCFH	11.91 %
H2	6.3 SCFH	17.12%
CH4	1.07 SCFH	2.91%
N2	10.34 SCFH	28.1 %

PURPOSE

SULFIDATION OF IFP ZINC OXIDE PELLETS TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1000 F

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 30 PPM PLATEAU FOR 1.5 HR
BREAKTHRU WITH 232 PPM AT 2 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FIRST SULFIDATION ON THIS SORBENT.
2. 60 F TEMP WAVE RECORDED AT INLET, AT START OF RUN.

CONCLUSIONS

1. H2S LEVELS ABOVE EQUILIBRIUM LEVELS.

TEST SUMMARY SHEET

TEST NO. : 062
DATE STARTED : 11/11/81
DATE ENDED : 11/12/81
TOTAL HOURS : 4.75 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/892 GM

SORBENT NO. : IFP ZNO MEP 682

SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3MM DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

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

PURPOSE

REGENERATION OF IFP ZINC OXIDE FROM TEST 061.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 8.8 - 14.4 %

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FIRST REGENERATION ON THIS SORBENT.

CONCLUSIONS

1. NORMAL REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 063
DATE STARTED : 11/13/81
DATE ENDED : 11/13/81
TOTAL HOURS : 3.5 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/892 GM
SORBENT NO. : IFP ZNO MEP 682
SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3 MM DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR LOADING : 9.04 % (GM SULFUR/GM FRESH SORBENT)

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLES
H_2S	0.9963 SCFH	2.7%
H_2O	3.872CC/MIN	27.66%
AIR		
CO_2	3.6 SCFH	9.68 %
CO	4.3 SCFH	11.91 %
H_2	6.3 SCFH	17.12%
CH_4	1.07 SCFH	2.91%
N_2	10.34 SCFH	28.1 %

PURPOSE

SULFIDATION OF IFP ZINC OXIDE PELLETS SECOND TIME TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1000 F

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 5 PPM PLATEAU FOR 2 HRS
BREAKTHRU WITH 20 PPM AT 2.5 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SECOND SULFIDATION ON THIS SORBENT.

CONCLUSIONS

1. LESS THAN 10 PPM H2S FOR 2 HRS.

TEST SUMMARY SHEET

TEST NO. : 064
DATE STARTED : 11/17/81
DATE ENDED : 11/17/81
TOTAL HOURS : 7 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/892 GM

SORBENT NO. : IFP ZNO MEP 682

SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3MM DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

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

PURPOSE

REGENERATION OF IFP ZINC OXIDE FROM TEST 063.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 5.6 - 11.6 %

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SECOND REGENERATION ON THIS SORBENT.

CONCLUSIONS

1. NORMAL REGENERATION

TEST SUMMARY SHEET

TEST NO. : 065
DATE STARTED : 11/18/81
DATE ENDED : 11/18/81
TOTAL HOURS : 3 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/892 GM
SORBENT NO. : IFP ZNO MEP 692
SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3 MM DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR LOADING : 9.03 % (GM SULFUR/GM FRESH SORBENT)

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	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 %

TOTAL DRY GAS FLOW RATE = 26.6 SCFH
TOTAL WET GAS FLOW RATE = 36.8 SCFH

PURPOSE

SULFIDATION OF IFP ZINC OXIDE PELLETS THIRD TIME TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1000 F

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 9 PPM PLATEAU FOR 2 HRS
BREAKTHRU WITH 200 PPM AT 2.5 HRS
EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE THIRD SULFIDATION ON THIS SORBENT.

CONCLUSIONS

1. IFP ZNO CAN REDUCE H2S TO BELOW 10 PPM.

2. NO APPARENT LOSS OF ACTIVITY AFTER 2 CYCLES.

TEST SUMMARY SHEET

TEST NO. : 066
DATE STARTED : 11/20/81
DATE ENDED : 11/20/81
TOTAL HOURS : 7.0 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/892 GM

SORBENT NO. : IFP ZNO MEP 682

SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3MM DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

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

PURPOSE

REGENERATION OF IFP ZINC OXIDE FROM TEST 065.

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:		11.6
DENSITY :		
PORE VOLUME:		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 7-15%

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE THIRD REGENERATION ON THIS SORBENT.

CONCLUSIONS

1. NORMAL REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 067
DATE STARTED : 11/23/81
DATE ENDED : 11/23/81
TOTAL HOURS : 3 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/892 GM
SORBENT NO. : IFP ZNO MEP 682
SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3 MM DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR LOADING : 6.78 % (GM SULFUR/GM FRESH SORBENT)

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	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 %

PURPOSE

SULFIDATION OF IFP ZINC OXIDE PELLETS FOURTH TIME TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1000 F

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:	11.6	
DENSITY :		
PORE VOLUME :		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

DATA: (DETECTOR TUBE)

EXIT H2S: .12 PPM PLATEAU FOR 1.5 HRS

BREAKTHRU WITH 26 PPM AT 2 HRS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FOURTH SULFIDATION ON THIS SORBENT.

CONCLUSIONS

1. POSSIBLY A SLIGHT DETERIORATION IN ACTIVITY AS SHOWN BY SLIGHTLY EARLIER BREAKTHROUGH.

TEST SUMMARY SHEET

TEST NO. : 068
DATE STARTED : 11/30/81
DATE ENDED : 11/30/81
TOTAL HOURS : 7.5 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/892 GM

SORBENT NO. : IFP ZNO MEP 692

SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3MM DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

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

PURPOSE

REGENERATION OF IFP ZINC OXIDE FROM TEST 067.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 7.5-14%

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FOURTH REGENERATION ON THIS SORBENT.

CONCLUSIONS

1. NORMAL REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 069
DATE STARTED : 12/3/81
DATE ENDED : 12/3/81
TOTAL HOURS : 2.0 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF IFP ZINC OXIDE PELLETS FIFTH TIME TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 800 F

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/892 GM
SORBENT NO. : IFP ZNO MEP 682
SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3 MM DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 4.515 % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 800 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	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 %

DATA: (DETECTOR TUBE)

EXIT H2S: 14 PPM PLATEAU FOR 0.5 HR

BREAKTHRU WITH 25 PPM AT 1 HR

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE FIFTH SULFIDATION ON THIS SORBENT.

2. ZINC METAL ANALYSIS OF CONDENSED WATER INDICATES 324 PPM ZINC.

CONCLUSIONS

1. RAPID BREAKTHROUGH AT 800 F INDICATES SLOWER KINETICS AT THE LOWER TEMPERATURE.

TEST SUMMARY SHEET

TEST NO. : 070
DATE STARTED : 01/7/82
DATE ENDED : 01/8/82
TOTAL HOURS : 7.75 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF IFP ZINC OXIDE FROM TEST 069.

SORBENT TYPE/WEIGHT: ZINC OXIDE/892 GM

SORBENT NO. : IFP ZNO MEP 682

SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3MM 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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 9.6-15.2 %

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

CONCLUSIONS

1. NORMAL REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 071
DATE STARTED : 01/12/82
DATE ENDED : 01/12/82
TOTAL HOURS : 1.0 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF IFP ZINC OXIDE PELLETS SIXTH TIME TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1200 F

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/892 GM
SORBENT NO. : IFP ZNO MEP 682
SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3 MM DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 1.12 % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

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

OPERATING CONDITIONS

TEMPERATURE: 1200 F (649 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLES%
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 %

DATA: (DETECTOR TUBE)

EXIT H2S: 15 PPM PLATEAU FOR 0.5 HR

BREAKTHRU WITH 590 PPM AT 0.5 HR

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. THIS IS THE SIXTH SULFIDATION ON THIS SORBENT.
2. ANALYSIS OF CONDENSED WATER - 92.25 PPM ZINC

CONCLUSIONS

1. LOSS OF ACTIVITY AT HIGHER TEMPERATURE. POSSIBLE SINTERING.

TEST SUMMARY SHEET

TEST NO. : 072
DATE STARTED : 01/14/82
DATE ENDED : 01/15/82
TOTAL HOURS : 7.5 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: ZINC OXIDE/892 GM

SORBENT NO. : IFP ZNO MEP 682

SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3MM DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

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

PURPOSE

REGENERATION OF IFP ZINC OXIDE FROM TEST 071.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 9.0 - 12.4 %

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. NORMAL REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 073
DATE STARTED : 1/21/82
DATE ENDED : 1/21/82
TOTAL HOURS : 0.25 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF IFP ZINC OXIDE PELLETS SEVENTH TIME TO DETERMINE EXIT H2S COMPOSITION AT SORPTION TEMPERATURE OF 1000 F AFTER SULFIDATION AT 1200 F.

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/892 GM
SORBENT NO. : IFP ZNO MEP 682
SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3 MM DIAMETER EXTRUSIONS
PRETREATMENT : NONE
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR:		
SURFACE AREA:	11.6	
DENSITY :		
PORE VOLUME :		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		
TOTAL SULFER :		34.74 (SAMPLE HAS ZNS & CASO4)

OPERATING CONDITIONS

TEMPERATURE: 1000 F (538 C)

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 28000 PPM AFTER 15 MINUTES

EXIT SO2:

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. THIS IS THE SEVENTH SULFIDATION ON THIS SORBENT.

CONCLUSIONS

1. IMMEDIATE BREAKTHROUGH INDICATES SORBENT PROBABLY WAS SINTERED IN THE 1200 F DEGREE RUN (TEST 071).

TEST SUMMARY SHEET

TEST NO. : 074
DATE STARTED : 2/8/82
DATE ENDED : 2/11/82
TOTAL HOURS : 12.75 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF IFP ZINC OXIDE FROM TEST 073.

SORBENT TYPE/WEIGHT: ZINC OXIDE/892 GM

SORBENT NO. : IFP ZNO MEP 682

SORBENT COMPOSITION: 70 % ZINC OXIDE

SORBENT PELLET SIZE: 3MM 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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 14 % - 16 %

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

CONCLUSIONS

1. NORMAL REGENERATION

TEST SUMMARY SHEET

TEST NO.	:	075
DATE STARTED	:	4/13/82
DATE ENDED	:	4/13/82
TOTAL HOURS	:	4.25 HRS
TYPE	:	SULFIDATION

PURPOSE

SULFIDATION OF ZINC FERRITE PELLETS WITH LOWER ZINC C OXIDE CONTENT.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #2 BATCH #1/1
SORBENT COMPOSITION: 71.4 MOLE% IRON OXIDE (FISHER CERTIFIED)
28.6 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 17.24 % (GM SULFUR/GM FRESH SORBENT)

ANALYSIS:

	BEFORE	AFTER
TOTAL SULFUR (%W/W):		
SURFACE AREA (M2/G):		
DENSITY (G/CC) :		
PORE VOLUME (M3/G):		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	FLOW RATE	MOLE%
H_2S	0.9963 SCFH	2.7%
H_2O	3.872CC/MIN	27.66%
AIR		
CO_2	3.6 SCFH	9.68 %
CO	4.3 SCFH	11.91 %
H_2	6.3 SCFH	17.12%
CH_4	1.07 SCFH	2.91%
N_2	10.34 SCFH	28.1 %

DATA: (DETECTOR TUBE)

EXIT H2S: 7 PPM PLATEAU FOR 2.75 HRS.

BREAKTHROUGH WITH 32 PPM AT 3.5 HRS.

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. LOWERING THE ZINC OXIDE CONTENT OF ZINC FERRITE DOES NOT APPEAR TO AFFECT ITS PERFORMANCE APPRECIABLY

TEST SUMMARY SHEET

TEST NO. : 076
DATE STARTED : 4/27/82
DATE ENDED : 4/29/82
TOTAL HOURS : HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 075

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #1 BATCH #1/1
SORBENT COMPOSITION: 71.4 MOLE% IRON OXIDE (FISHER CERTIFIED)
28.6 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 12 - 15 %

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

CONCLUSIONS

TEST SUMMARY SHEET

TEST NO. : 077
DATE STARTED : 5/12/82
DATE ENDED : 5/12/82
TOTAL HOURS : 1.5 HRS
TYPE : SULFIDATION

PURPOSE

SECOND SULFIDATION OF ZINC FERRITE PELLETS WITH LOWER ZINC OXIDE CONTENT.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : MIX #2 BATCH #1/1
SORBENT COMPOSITION: 71.4 MOLE% IRON OXIDE (FISHER CERTIFIED)
28.6 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)
SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 1.57 % (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: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 3.0 PPM FOR .25 HOURS

BREAKTHROUGH WITH 30 PPM AT .75 HOURS

EXIT SO2:

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

CONCLUSIONS

1. POOR PERFORMANCE ON SECOND SULFIDATION.POSSIBLY DUE TO INCOMPLETE REGENERATION.

TEST SUMMARY SHEET

TEST NO. : 078
DATE STARTED : 5/19/82
DATE ENDED : 5/24/82
TOTAL HOURS : 18.5 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 077

SORBENT TYPE/WEIGHT: ZINC FERRITE/643 GM
SORBENT NO. : MIX #1 BATCH #1/1
SORBENT COMPOSITION: 71.4 MOLE% IRON OXIDE (FISHER CERTIFIED)
28.6 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 13.7 PERCENT MAX 100 PPM AT END OF TEST

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. STEAM WAS SHUT OFF AFTER PASSAGE OF TEMP WAVE.

2. AIR FLOW RATE WAS INCREASED UP TO 32 SCFH, IN ORDER TO ATTEMPT LOWERING EXIT SO2.

CONCLUSIONS

1. SO2 LOWERED TO 100 PPM BY REDUCING STEAM AND INCREASING AIR FLOW RATES.

TEST SUMMARY SHEET

TEST NO. : 079
DATE STARTED : 5/25/82
DATE ENDED : 5/25/82
TOTAL HOURS : 2 HRS
TYPE : SULFIDATION

PURPOSE

THIRD SULFIDATION OF ZINC FERRITE PELLETS WITH LOWER ZINC OXIDE CONTENT.

SORBENT TYPE/WEIGHT: ZINC FERRITE/643 GM
SORBENT NO. : MIX #2 BATCH #1/1
SORBENT COMPOSITION: 71.4 MOLE% IRON OXIDE (FISHER CERTIFIED)
28.6 MOLE% ZINC OXIDE (SHERWIN WILLIAMS HSA)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : 7.84 % (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: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 9.9 PPM AVG FOR 1.5 HRS
BREAKTHROUGH WITH 1050 PPM AT 2 HRS.

EXIT SO2:

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

CONCLUSIONS

1. LOSS OF SULFIDATION CAPACITY OBSERVED IN COMPARISON TO FIRST SULFIDATION.

2' : at 1500°

L-1442 . 0/Fe_2O_3 = 1

POROSITY DETERMINATION

XRD 1. $ZnFe_2O_4$ mes > 2000 A°

SAMPLE U.S. Dept of ENERGY EXP 5156

65#/ft³ Density

2.3#/mm Crush

SAMPLE WEIGHT NORMALIZED TO 1 gram

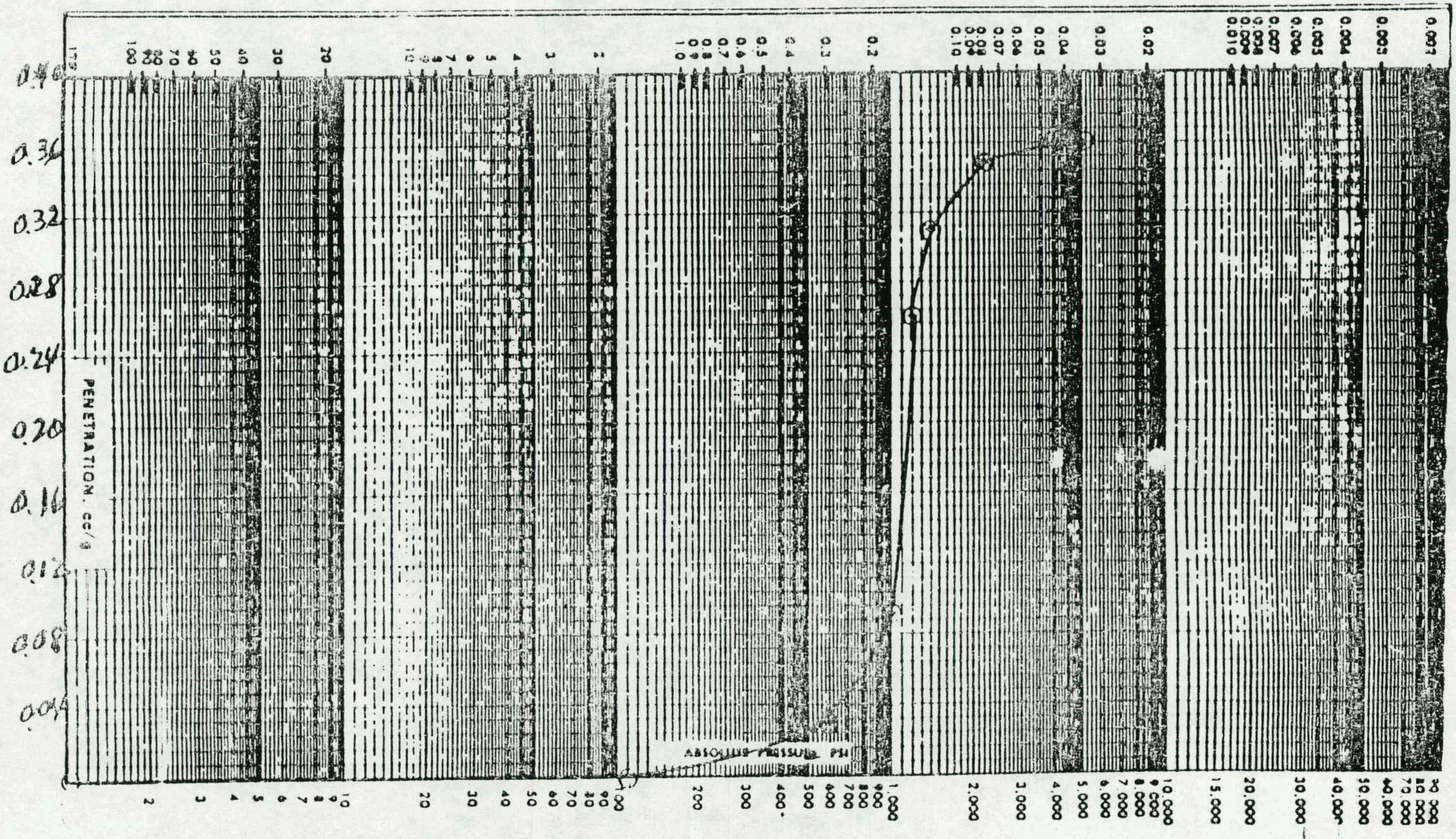
6.6 m²/g Surface Area.

DATE 5/14/82

0.36 cc/g Pore Volume > 29 A

1520 A° Av Pore Diamd.

EQUIVALENT PORE DIAMETER (MICRONS) - 175/PSI (CONTACT ANGLE = 130°)



XRD 1. $ZnFe_2O_4$ mes > 2000 A°

5.9 m²/g Surface Area

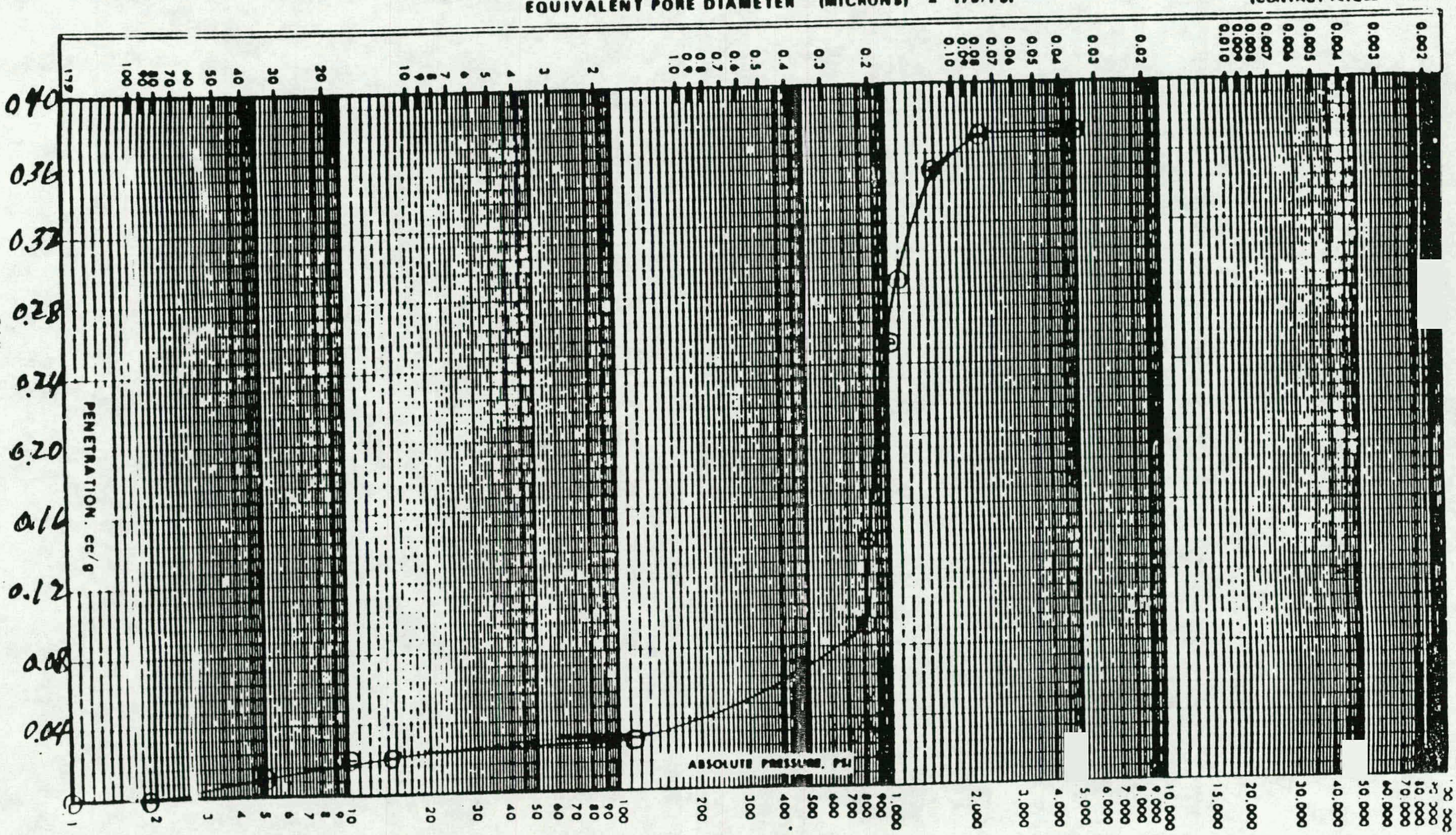
POROSITY DETERMINATION

SAMPLE USDE EKD5157

SAMPLE WEIGHT NORMALIZED TO 1 gram

DATE 5/18/82

L-1443 3x0/3 = 1
Same as L-1442 except fired 2 Hrs at 1600°F
66.5 #/ft³ Density
2.0 #/mm Crush
0.37 cc/g Pore Volume > 29 A°
2000 A° Av Pore Diameter



-343-

L-1444 ZnO/Fe_2O_3 = 1.0 with ~0% Al_2O_3
Fired 2 Hrs at 1500°F
71.#/ft³ Density
1.7#/mm Crush
0.22cc/g Pore Volume > 29Å
1095Å Av Pore Diameter

POROSITY DETERMINATION

$\boxed{XRD}$ 1. $ZnFe_2O_4$ (~67%) mcs 321Å
2. αFe_2O_3 (~33%) mcs > 2000Å
3. $ZnAl_2O_4$ mcs 80Å

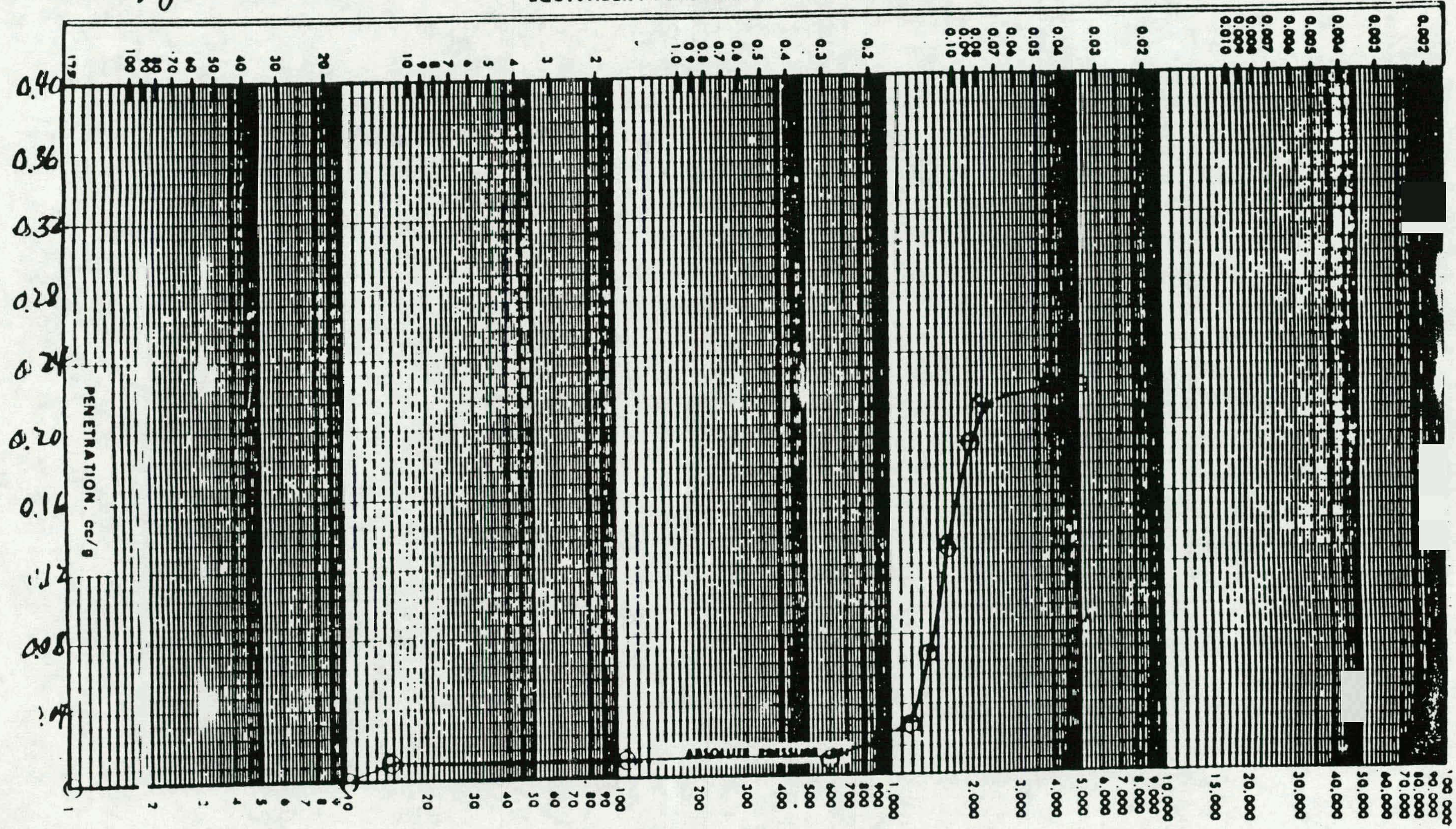
25.8 m²/g Surface Area

SAMPLE [illegible] EKP5158

SAMPLE WEIGHT NORMALIZED TO 1 gram

DATE 3/1/82

EQUIVALENT PORE DIAMETER (MICRONS) = 175/PSI (CONTACT ANGLE = 130°)



L-1445 . 1/Fe_2O_3 = 1
Fired 2 hrs at 1100°F
66.6 #/ft³ Density
0.70 #/mm Crush
0.32 cc/g Pore Volume > 29.
1400 Å Av Pore Diameter

$\boxed{\text{XRD}}$

1. $ZnFe_2O_4$ (~85%) mcs 776 Å
2. α Fe_2O_3 (~5%) mcs > 2000 Å
3. ZnO (?) —

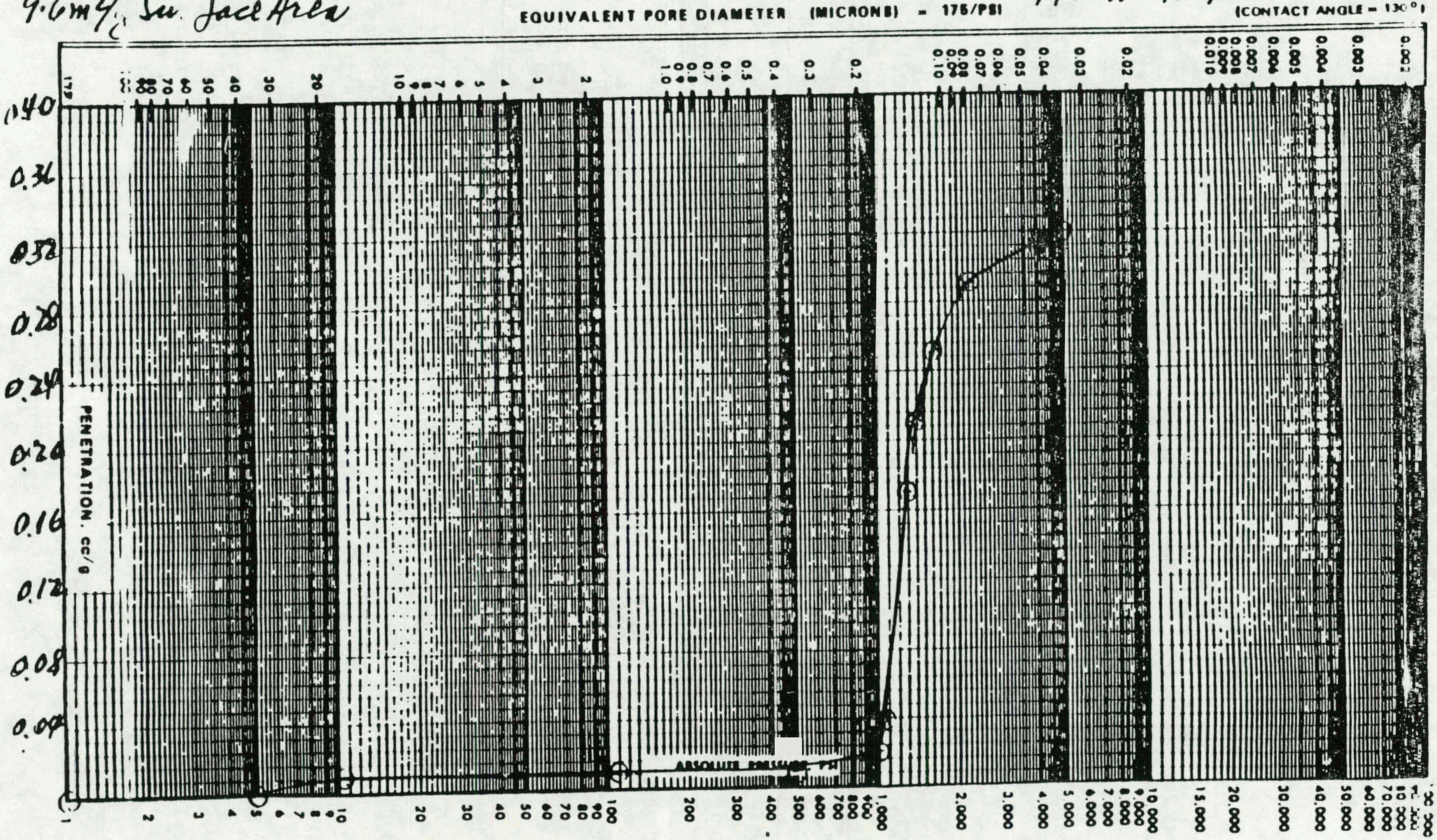
9.6 m²/g Surface Area

POROSITY DETERMINATION

SAMPLE US Dept of Energy EKD 5159

SAMPLE WEIGHT NORMALIZED TO 1 gram

DATE 5/14/82



TEST SUMMARY SHEET

TEST NO. : 080
DATE STARTED : 6/2/82
DATE ENDED : 6/2/82
TOTAL HOURS : 2.75 HRS
TYPE : SULFIDATION

PURPOSE

FIRST SULFIDATIION OF UNITED CATALYST L-1442

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : L-1442
SORBENT COMPOSITION: 50 MOLES% IRON OXIDE
50 MOLES% ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (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: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S: 330 PPM AT 15 MINUTES, DECREASING TO 74 PPM.

EXIT SO2:

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

CONCLUSIONS

1. SUSPECT IRON SULFIDES IN SORBENT DUE TO IMPURITY IN IRON OXIDE USED BY UNITED CATALYSTS.

TEST SUMMARY SHEET

TEST NO. : 081
DATE STARTED : 6/04/82
DATE ENDED : 6/7/82
TOTAL HOURS : 8.25 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 080

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/643 GM
SORBENT NO. : L-1442
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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 15.4 PERCENT MAX, 350 PPM AT END OF TEST

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

CONCLUSIONS

1. SORBENT REGENERATED.

TEST SUMMARY SHEET

TEST NO. : 082
DATE STARTED : 6/14/82
DATE ENDED : 6/14/82
TOTAL HOURS : 3.5 HRS
TYPE : SULFIDATION

PURPOSE

SECOND SULFIDATIION OF UNITED CATALYST L-1442

SORBENT TYPE/WEIGHT: UNITD CATALYSTS ZINC FERRITE)/643 GM
SORBENT NO. : L-1442
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (PFIZER CX-6139)
50 MOLE% ZINC OXIDE (FRENCH PROCESS A2066) AMERICAN SMELTING & REFINING CO.
SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (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: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	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 %

DATA: (DETECTOR TUBE)

EXIT H2S: 10.25 PPM AVG FOR 2 HOURS.

EXIT SO2: 200 PPM INITIALY 0 AFTER 45 MINUTES

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. PERFORMANCE SIMILAR TO METC ZINC FERRITE.

TEST SUMMARY SHEET

TEST NO. : 083
DATE STARTED : 6/15/82
DATE ENDED : 6/16/82
TOTAL HOURS : 6 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 082

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : L-1442
SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE (PFIZER CX-6139)
50.0 MOLE% ZINC OXIDE (FRENCH PROCESS A2066)
AMERICAN SMELTING & REFINING CO.
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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 16.4 PERCENT MAX, 6000 PPM AT END OF TEST.

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

CONCLUSIONS

1. SORBENT REGENERATED.

TEST SUMMARY SHEET

TEST NO. : 084
DATE STARTED : 6/17/82
DATE ENDED : 6/17/82
TOTAL HOURS : 3.25 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDATION OF UNITED CATALYST L-1442 AT 1200 F.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : L-1442
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (PFIZER CX-6139)
50 MOLE% ZINC OXIDE (FRENCH PROCESS A2066)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (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: 2000 HOURLY

	FLOW RATE	MOLE%
H_2S	0.9963 SCFH	2.7%
H_2O	3.872CC/MIN	27.66%
AIR		
CO_2	3.6 SCFH	9.68 %
CO	4.3 SCFH	11.91 %
H_2	6.3 SCFH	17.12%
CH_4	1.07 SCFH	2.91%
N_2	10.34 SCFH	28.1 %

DATA: (DETECTOR TUBE)

EXIT H_2S: 10 PPM AVG FOR 1.75 HOURS!!
BREAKTHROUGH AT 2.25 HOURS WITH 27 PPM

EXIT SO_2:

EXIT S_2:

EXIT H_2:

REMARKS

CONCLUSIONS

1. REASONABLE PERFORMANCE AT 1200 F. SLIGHTLY FASTER BREAKTHROUGH AS COMPARED TO SULFIDATION AT 1000F (AS EXPECTED).

TEST SUMMARY SHEET

TEST NO. : 085
DATE STARTED : 6/18/82
DATE ENDED : 6/21/82
TOTAL HOURS : 10 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 084

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/643 GM
SORBENT NO. : L-1442
SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE (PFIZER CX-6139)
50.0 MOLE% ZINC OXIDE (FRENCH PROCESS AZ066) AMERICAN SMELTING & REFINING CO.
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: 600 HOURLY

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 8.4 PERCENT MAX ,925 PPM AT END OF TEST

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

CONCLUSIONS

1. SORBENT REGENERATED.

TEST SUMMARY SHEET

TEST NO. : 086
DATE STARTED : 6/22/82
DATE ENDED : 6/22/82
TOTAL HOURS : 3.6 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/643 GM
SORBENT NO. : L-1442
SORBENT COMPOSITION: 50 MOLE% IRON OXIDE (PFIZER CX-6139)
50 MOLE% ZINC OXIDE (FRENCH PROCESS A2066)
AMERICAN SMELTING & REFINING CO.
SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	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 %

PURPOSE

SECOND SULFIDATIION OF UNITED CATALYST L-1442
AT 1200 F.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 9 PPM FOR 1 HOUR
BREAKTHROUGH WITH 200 PPM AT 2 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. SECOND SULFIDATION RESULTED IN MORE RAPID BREAKTHROUGH MAY BE AN INDICATION OF SINTERING CAUSING A DROP OFF IN PERFORMANCE.

TEST SUMMARY SHEET

TEST NO. : 087
DATE STARTED : 6/23/82
DATE ENDED : 6/24/82
TOTAL HOURS : 7.25 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : L-1442
SORBENT COMPOSITION: 50.0 MOLE% IRON OXIDE
50.0 MOLE% ZINC OXIDE

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 600 HOURLY

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

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 086

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 6.6 % MAX 1200 PPM AT END

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. SORBENT REGENERATED.

TEST SUMMARY SHEET

TEST NO. : 088
DATE STARTED : 6/25/82
DATE ENDED : 6/25/82
TOTAL HOURS : 1.75 HRS
TYPE : SULFIDATION

PURPOSE

THIRD SULFIDATIION OF UNITED CATALYST L-1442 AT 1200 F.

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/643 GM
SORBENT NO. : L-1442
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 (%W/W):		
SURFACE AREA (M2/G):		
DENSITY (G/CC) :		
PORE VOLUME (M3/G):		
MINERAL ANALYSIS:		
ELEMENTAL ANALYSIS:		

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

SPACE VELOCITY: 2000 HOURLY

	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 %

DATA: (DETECTOR TUBE)

EXIT H2S: 10 PPM AT 15 MINUTES
BREAKTHROUGH AT 1.3 HOURS WITH 520 PPM
EXIT SO2: 100 PPM AT 15 MINUTES DOWN TO 1 PPM AFTER 1.5 HRS.
EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. LOSS OF PERFORMANCE OBSERVED AS COMPARED TO FIRST TWO SULFIDATIONS. SUSPECT LOSS DUE TO SINTERING AT 1200 F.

TEST SUMMARY SHEET

TEST NO. : 089
DATE STARTED : 6/29/82
DATE ENDED : 6/29/82
TOTAL HOURS : 2 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/500 GM
(HYDROGENATION CATALYST)/333 GM
SORBENT NO. : G-72D ZINC OXIDE
G-65RS HYDROGENATION CATALYST
SORBENT COMPOSITION: 90 WEIGHT % ZNO (G-72D)
27 WEIGHT % NI (G65RS)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (GM SULFUR/GM FRESH SORBENT)

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY FOR ZNO AND G-65RS (APPROX)

	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 %

PURPOSE

DETERMINE THE CAPABILITY OF NICKEL TO REDUCE H2S LEVEL BELOW 1 PPM.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 16 PPM INITIALLY

EXIT SO2: UNDETECTABLE

EXIT S2:

EXIT H2:

REMARKS

1. BOTH SORBENTS USED ARE MANUFACTURED BY UNITED CATALYSTS.

CONCLUSIONS

1. SULFUR LEVELS WERE NOT REDUCED BELOW 1 PPM. SUSPECT POSSIBLITY OF SULFUR IN THE NICKEL CATALYST AND/OR SULFUR PICKUP FROM REACTOR WALLS.

TEST SUMMARY SHEET

TEST NO. : 090
DATE STARTED : 7/2/82
DATE ENDED : 7/2/82
TOTAL HOURS : 10 HRS
TYPE : SULFIDATION

PURPOSE

DETERMINE IF DESULFIDING OF NICKEL CATALYST OCCURS IN A LURGI GAS WITHOUT H2S.

SORBENT TYPE/WEIGHT: (ZINC OXIDE)/500 GM
(HYDROGENATION CATALYST)/500 GM
SORBENT NO. : G-72D ZINC OXIDE
G-65RS HYDROGENATION CATALYST
SORBENT COMPOSITION: 90 WEIGHT % ZNO (G-72D)
27 WEIGHT % NI (G65RS)

SORBENT PELLET SIZE: 3/16 INCH DIAMETER EXTRUSIONS
PRETREATMENT :
SULFUR LOADING : % (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: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 1000 HOURLY FOR ZNO AND G-65RS (APPROX)

DATA: (DETECTOR TUBE)

EXIT H2S: 30-40 PPM

EXIT SO2: UNDETECTABLE

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE%
H2S		
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	11.33 SCFH	30.8 %

REMARKS

1. BOTH SORBENTS USED ARE MANUFACTURED BY UNITED CATALYSTS.

CONCLUSIONS

1. DESULFIDING OF NICKEL CATALYST AND/OR REACTOR WALLS OCURRED. SUB PPM LEVEL TESTING WILL REQUIRE DEDICATED "LOW SULFUR" SYSTEM, OR QUARTZ REACTOR TO ELIMINATE POSSIBILITY OF SULFUR PICK UP FROM REACTOR WALLS

TEST SUMMARY SHEET

TEST NO. : 091
DATE STARTED : 7/20/82
DATE ENDED : 7/20/82
TOTAL HOURS : 5.5 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: NONE
SORBENT NO. :
SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY:

	FLOW RATE	MOLE%
H2S		
H2O	2.23 CC/MIN	50%
AIR	5.68 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

PURPOSE

REGENERATION OF EMPTY REACTOR PRIOR TO EMPTY REACTOR TEST.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 8000 PPM MAX, 360 PPM AT END OF TEST.

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. REACTOR WALLS DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 092
DATE STARTED : 7/21/82
DATE ENDED : 7/21/82
TOTAL HOURS : 5.5 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: NONE
SORBENT NO. :

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING : 3.524 GM S2

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY:

	FLOW RATE	MOLE%
H2S	0.036 SCFH	0.1 %
H2O	3.872CC/MIN	27.65%
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	11.3 SCFH	30.7 %

PURPOSE

SULFIDE EMPTY REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 200 PPM INITIALLY, BREAKTHROUGH COMPLETED IN 4.5 HOURS.

EXIT SO2: 14 PPM INITIALLY , DECREASED TO UNDETECTABLE AFTER 1.5 HOURS.

EXIT S2:

EXIT H2:

REMARKS

1. 48 % OF INLET H2S PICKED UP BY REACTOR WALL.
2. H2S PICKED UP BY REACTOR WALLS EQUIVALENT TO 3.524 GM S2.

CONCLUSIONS

1. SIGNIFICANT SULFUR PICK-UP BY STAINLESS STEEL REACTOR.

TEST SUMMARY SHEET

TEST NO. : 093
DATE STARTED : 7/22/82
DATE ENDED : 7/22/82
TOTAL HOURS : 4.5 HRS
TYPE :

SORBENT TYPE/WEIGHT:
SORBENT NO. :

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY:

	FLOW RATE	MOLE%
H2S		
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	11.3 SCFH	30.7 %

PURPOSE

DETERMINE H2S LEACHING FROM SULFIDED EMPTY REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 335 PPM AVERAGE

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. H2S LEACHING APPEARS TO BE NEAR EQUILIBRIUM LEVEL 335 PPM AVERAGE.

TEST SUMMARY SHEET

TEST NO. : 094
DATE STARTED : 7/23/82
DATE ENDED : 7/23/82
TOTAL HOURS : 5.75 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: NONE
SORBENT NO. :
SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY:

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

PURPOSE

REGENERATION OF EMPTY REACTOR TEST.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: UNDETECTABLE

EXIT SO2: 7 % MAX, 70 PPM AT END OF TEST

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. REACTOR DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 095
DATE STARTED : 7/26/82
DATE ENDED : 7/26/82
TOTAL HOURS : 5.75 HRS
TYPE :

SORBENT TYPE/WEIGHT:
SORBENT NO. :

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY:

	FLOW RATE	MOLES
H2S		
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	11.3 SCFH	30.7 %

PURPOSE

DETERMINE H2S LEACHING FROM REGENERATED EMPTY REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 150 - 200 PPM INITIALLY THEN DECREASED TO 30 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. H2S LEACHES FROM REACTOR WALLS EVEN AFTER REGENERATION.

2. REGENERATION APPARENTLY DOES NOT DESULFIDE REACTOR COMPLETELY.

TEST SUMMARY SHEET

TEST NO. : 096
DATE STARTED : 8/3/82
DATE ENDED : 8/3/82
TOTAL HOURS : 50 HRS
TYPE :

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/322 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 787 HOURLY
LINEAR VELOCITY: 5 CM/SEC

	FLOW RATE	MOLE%
H2S	0.0505 SCFH	0.5
H2O		
AIR		
CO2	1.17 SCFH	9.68 %
CO	1.44 SCFH	11.91 %
H2	2.07 SCFH	17.12%
CH4	0.35 SCFH	2.91%
N2	7.00 SCFH	57.88 %

TOTAL FLOW RATE: 12.1 SCFH

PURPOSE

RUN SULFIDATION OF "HALF REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 0 INITIALLY
10 PPM BREAKTHROUGH AFTER 22 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. INLET H2S FLOW WAS NOT KEPT CONSTANT.

CONCLUSIONS

1. SPACE VELOCITY TOO LOW FOR SHORT TERM TESTING.

TEST SUMMARY SHEET

TEST NO. : 097
DATE STARTED : 8/17/82
DATE ENDED : 8/17/82
TOTAL HOURS : 6.5 HRS
TYPE :

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/160 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 6780 HOURLY FOR 4 INCH BED
LINEAR VELOCITY: 25 CM/SEC

	FLOW RATE	MOLE%
H2S	0.90 SCFH	1.5
H2O		
AIR		
CO2	5.8 SCFH	9.68 %
CO	7.2 SCFH	11.91 %
H2	10.27 SCFH	17.12%
CH4	1.74 SCFH	2.91%
N2	34.120 SCFH	56.88 %

TOTAL FLOW RATE: 60.0 SCFH

PURPOSE

RUN SULFIDATION OF "1/4 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. 4 INCH BED IS TOO SHORT. POSSIBLE CHANNELING CAUSING HIGH INITIAL H2S.

2. TEST WAS COMPLETED IN ONE SHIFT.

TEST SUMMARY SHEET

TEST NO. : 098
DATE STARTED : 8/24/82
DATE ENDED : 8/24/82
TOTAL HOURS : 12 HRS
TYPE : SULFIDATION

PURPOSE

RUN SULFIDATION OF "1/4 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/160 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: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: HOURLY FOR 4 INCH BED
LINEAR VELOCITY: CM/SEC

	FLOW RATE	MOLE%
H2S	0.60 SCFH	1.5
H2O		
AIR		
CO2	3.6 SCFH	9.68 %
CO	4.4 SCFH	11.91 %
H2	6.3 SCFH	17.12%
CH4	1.07 SCFH	2.91%
N2	21.0 SCFH	56.88 %

TOTAL FLOW RATE: SCFH

DATA: (DETECTOR TUBE)

EXIT H2S: 480 PPM AFTER 15 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. LOWERED SPACE VELOCITY LOWERED INITIAL H2S SLIGHTLY BUT NOT ENOUGH. RUN TIME IS 12 HOURS WHICH IS TOO LONG FOR ONE SHIFT OPERATION. POSSIBLE CHANNELING STILL A PROBLEM.

TEST SUMMARY SHEET

TEST NO. : 099
DATE STARTED : 8/26/82
DATE ENDED : 8/26/82
TOTAL HOURS : 11 HRS
TYPE : REGENERATION

PURPOSE

REGENERATION OF EMPTY REACTOR

SORBENT TYPE/WEIGHT: L-1443 ZINC FERRITE
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:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 11.5 % MAX , 292 PPM AT END OF TEST

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. REACTOR DESULFIDED

TEST SUMMARY SHEET

TEST NO. : 100
DATE STARTED : 8/30/82
DATE ENDED : 8/30/82
TOTAL HOURS : 3.6 HRS
TYPE : REDUCTION

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/160 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: HOURLY FOR 4 INCH BED
LINEAR VELOCITY: CM/SEC

	FLOW RATE	MOLE%
H2S		
H2O		
AIR		
CO2	3.8 SCFH	10.39 %
CO	4.4 SCFH	12.03 %
H2	6.3 SCFH	17.22%
CH4	1.07 SCFH	2.92%
N2	21.0 SCFH	57.42 %

TOTAL FLOW RATE: 36.57 SCFH

PURPOSE

RUN REDUCTION OF "1/4 REACTOR" TO DETERMINE LEVEL OF H2S LEACHING FOR SCHROBT MODEL.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 4 PPM AVG

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

-

CONCLUSIONS

1. 4 PPM H2S OBTAINED DURING REDUCTION

TEST SUMMARY SHEET

TEST NO. : 101
DATE STARTED : 9/1/82
DATE ENDED : 9/1/82
TOTAL HOURS : 4.75 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: UNITED CATALYSTS ZINC FERRITE/160 GM
SORBENT NO. : L-1443

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 5500 HOURLY FOR 4 INCH BED
LINEAR VELOCITY: 15.5 CM/SEC

	FLOW RATE	MOLE%
H2S	1.2 SCFH	3.2
H2O		
AIR		
CO2	3.6 SCFH	9.6 %
CO	4.4 SCFH	11.7 %
H2	6.3 SCFH	16.8%
CH4	1.07 SCFH	2.8%
N2	21.0 SCFH	55.9 %

TOTAL FLOW RATE: 37.57 SCFH

PURPOSE

RUN SULFIDATION OF "1/4 REACTOR" FOR COMPLETE BREAKTHROUGH FOR SCHRODT MODEL.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: INITIAL H2S 5750 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. 3.5 INCH BED TOO SHORT. POSSIBLE CHANNELING.

TEST SUMMARY SHEET

TEST NO. : 102
DATE STARTED : 9/3/82
DATE ENDED : 9/3/82
TOTAL HOURS : 5.5 HRS
TYPE : REGENERATION

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

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 984 HOURLY

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

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 31200 PPM MAX, 560 PPM AT END OF REGENERATION

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. REACTOR WALLS DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 103
DATE STARTED : 9/10/82
DATE ENDED : 9/10/82
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

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: 1000 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
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

DATA: (DETECTOR TUBE)

EXIT H2S: 4.5 PPM AFTER 10 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

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

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:

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

	FLOW RATE	MOLES
H2S		
H2O	2.23 CC/MIN	50%
AIR	5.83 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: MAX 3.3 PERCENT

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. REACTOR DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 105
DATE STARTED : 9/16/82
DATE ENDED : 9/16/82
TOTAL HOURS : 5.33 HRS
TYPE : SULFIDATION

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

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
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 %

TOTAL FLOW RATE: 43.0 SCFH

PURPOSE

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

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 55 PPM AFTER 20 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

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

TEST SUMMARY SHEET

TEST NO. : 106
DATE STARTED : 9/20/82
DATE ENDED : 9/20/82
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:

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: 994 HOURLY

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

DATA: (DETECTOR TUBE)

EXIT H2S:

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

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. REACTOR WALLS DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 107
DATE STARTED : 9/21/82
DATE ENDED : 9/21/82
TOTAL HOURS : 2.5 HRS
TYPE : SULFIDATION

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

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
H2S	2.4 SCFH	5.6
H20	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 %

TOTAL FLOW RATE: 43.0 SCFH

PURPOSE

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

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 16 PPM AFTER 15 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. BREAKTHROUGH CURVE AT 1200 F IS COMPLETED SOONER THAN AT 1000F AS EXPECTED.

TEST SUMMARY SHEET

TEST NO. : 108
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:

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

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 2.7 % MAX 550 PPM AT END OF RUN

EXIT S2:

EXIT H2:

REMARKS

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

CONCLUSIONS

1. REACTOR WALLS DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 109
DATE STARTED : 9/24/82
DATE ENDED : 9/24/82
TOTAL HOURS : 1.3 HRS
TYPE : SULFIDATION

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

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
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

PURPOSE

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

ANALYSIS:

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

DATA: (DETECTOR TUBE)

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

EXIT SO2:

EXIT S2:

EXIT H2:

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. : 110
DATE STARTED : 9/29/82
DATE ENDED : 9/29/82
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

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 2.25 % MAX 380 PPM AT END OF REGENERATION

EXIT S2:

EXIT H2:

REMARKS

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

CONCLUSIONS

1. REACTOR WALLS DESULFIDED.

TEST SUMMARY SHEET

TEST NO. : 111
DATE STARTED : 9/30/82
DATE ENDED : 9/30/82
TOTAL HOURS : 0.6 HRS
TYPE : SULFIDATION

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

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
H2S	2.4 SCFH	6.4
H2O		
AIR		
CO2	3.6 SCFH	9.68 %
CO	4.4 SCFH	11.91 %
H2	6.3 SCFH	17.12%
CH4	1.07 SCFH	2.91%
N2	21.0 SCFH	56.88 %

TOTAL FLOW RATE: 38.77 SCFH

PURPOSE

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

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 075 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

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

CONCLUSIONS

1. TEST ABORTED.

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 :

	FLOW RATE	MOLE%
H2S		
H2O	2 [illegible]3 CC/MIN	50%
A	5. SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: UP TO 0600 PPM

EXIT S2:

EXIT H2:

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 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: 1400 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE %
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 %

TOTAL FLOW RATE: 43.0 SCFH

DATA: (DETECTOR TUBE)

EXIT H2S: 0 PPM AFTER 5 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. SHORTER BREAKTHROUGH CURVE AS EXPECTED.

TEST SUMMARY SHEET

TEST NO. : 114
DATE STARTED : 10/13/82
DATE ENDED : 10/13/82
TOTAL HOURS : 6.5 HRS
TYPE : REGENERATION

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

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY : 984 HOURLY

	FLOW RATE	MOLE%
H2S		
H2O	2.23 CC/MIN	50%
AIR	5.80 SCFH	50%
CO2		
CO		
H2		
CH4		
N2		

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: TO 22,500 PPM

EXIT S2:

EXIT H2:

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 : 10/14/82
DATE ENDED : 10/14/82
TOTAL HOURS : 5.73 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: 1000 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
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	40.0 %

TOTAL FLOW RATE: 24.2 SCFH

DATA: (DETECTOR TUBE)

EXIT H2S: UNDETECTABLE INITIALLY, 15 PPM AFTER 14 MINUTES.

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. EXPECTED COMPLETION OF BREAKTHROUGH CURVE IS 7 HRS.

CONCLUSIONS

1. COMPLETED BREAKTHROUGH CURVE AT 1000 F .

TEST SUMMARY SHEET

TEST NO. : 116
DATE STARTED : 10/19/82
DATE ENDED : 10/19/82
TOTAL HOURS : 6.16 HRS
TYPE : REGENERATION

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

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 984 HOURLY

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

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: TO 10230 PPM

EXIT S2:

EXIT H2:

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 : 10/20/82
DATE ENDED : 10/20/82
TOTAL HOURS : 2.25 HRS
TYPE : SULFIDATION

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

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
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 %

TOTAL FLOW RATE: [illegible] 1.2 SCFH

PURPOSE

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

ANALYSIS:

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

DATA: (DETECTOR TUBE)

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

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. EXPECTED COMPLETION OF BREAKTHROUGH CURVE IS 7 HRS.

CONCLUSIONS

1. COMPLETED BREAKTHROUGH CURVE AT 1200 F.

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

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: TO 10,000 PPM

EXIT S2:

EXIT H2:

REMARKS

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

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : [illegible] 2
DATE STARTED : 10/26/82
DATE ENDED : 10/25/82
TOTAL HOURS : 0.4 HRS
TYPE : SULFIDATION

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

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1400 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
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	40.0 %

TOTAL FLOW RATE: 24.2 SCFH

PURPOSE

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

ANALYSIS:

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

DATA: (DETECTOR TUBE)

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

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

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

CONCLUSIONS

1. COMPLETED BREAKTHROUGH CURVE AT 1400 F.

TEST SUMMARY SHEET

TEST NO. : 120
DATE STARTED : 10/25/82
DATE ENDED : 10/25/82
TOTAL HOURS : 3.25 HRS
TYPE : REGENERATION

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

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1029 F

PRESSURE : AMBIENT

SPACE VELOCITY: 984 HOURLY

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

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: TO 14,000 PPM

EXIT S2:

EXIT H2:

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 : 10/20/82
DATE ENDED : 10/20/82
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 (%W/W):		
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

	FLOW RATE	MOLE%
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 %

TOTAL FLOW RATE: 43.0 SCFH

DATA: (DETECTOR TUBE)

EXIT H2S: 1100 PPM INITIALLY

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

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

TEST SUMMARY SHEET

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

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

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 984 HOURLY

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

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: TO 17000 PPM

EXIT S2:

EXIT H2:

REMARKS

1. FRESH BATCH OF SORBENT LOADED. REGENERATION IS TO DESULFICE 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/328 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: 3600 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 17.8 CM/SEC

	FLOW RATE	MOLE%
H_2S	1.34 SCFH	3.1
H_2O	1.63 CC/MIN	10.0 %
AIR		
CO_2	3 [illegible] SCFH	8.4 %
CO	4.4 SCFH	10.2 %
H_2	6.3 SCFH	14.6%
CH_4	1.07 SCFH	2.5%
N_2	22.0 SCFH	48.8 %

TOTAL FLOW RATE: 43.0 SCFH

DATA: (DETECTOR TUBE)

EXIT H_2S: UNDETECTABLE INITIALLY, 4 PPM AFTER 25 MINUTES

EXIT SO_2:

EXIT S_2:

EXIT H_2:

REMARKS

CONCLUSIONS

1. BREAKTHROUGH CURVE AT 1200 F COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 124
DATE STARTED : 11/08/82
DATE ENDED : 11/08/82
TOTAL HOURS : 4.20 HRS
TYPE : REGENERATION

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

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 904 HOURLY

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

PURPOSE

REGENERATION OF FRESHLY LOADED REACTOR

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: TO 36,000 PPM

EXIT S2:

EXIT H2:

REMARKS

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

CONCLUSIONS

1. REGENERATION COMP_ETED.

TEST SUMMARY SHEET

TEST NO. : 125
DATE STARTED : 11/9/82
DATE ENDED : 11/9/82
TOTAL HOURS : 8.20 HRS
TYPE : SULFIDATION

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

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1400 F

PRESSURE : AMBIENT

SPACE VELOCITY: 3600 HOURLY FOR 7 INCH BED
LINEAR VELOCITY: 17.0 CM/SEC

	FLOW RATE	MOLE%
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	40.0 %

TOTAL FLOW RATE: 43.0 SCFH

PURPOSE

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

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: UNDETECTABLE INITIALLY, 10 PPM AFTER 7 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

CONCLUSIONS

1. BREAKTHROUGH CURVE AT 1400 F COMPLETED.

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

	FLOW RATE	MOLE%
H2S		
H2O	2 23 CC/MIN	50%
AIR	5.88 SCFH	50%
CO2		
CO		
CH4		
N2		

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: TO 17,000 PPM

EXIT S2:

EXIT H2:

REMARKS

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

2

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET

TEST NO. : 127
DATE STARTED : 11/22/82
DATE ENDED : 11/22/82
TOTAL HOURS : 1.00 HRS
TYPE : SULFIDATION

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

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

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

	FLOW RATE	MOLE%
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.[illegible] SCFH	14.6%
CH4	0.60 SCFH	2.5%
N2	11.8 SCFH	48.8 %

TOTAL FLOW RATE: 24.2 SCFH

PURPOSE

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

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: UNDETECTABLE INITIALLY, 12 PPM AFTER 18 MINUTES

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. RUN UNTIL EXIT H2S REACHES 50 PPM

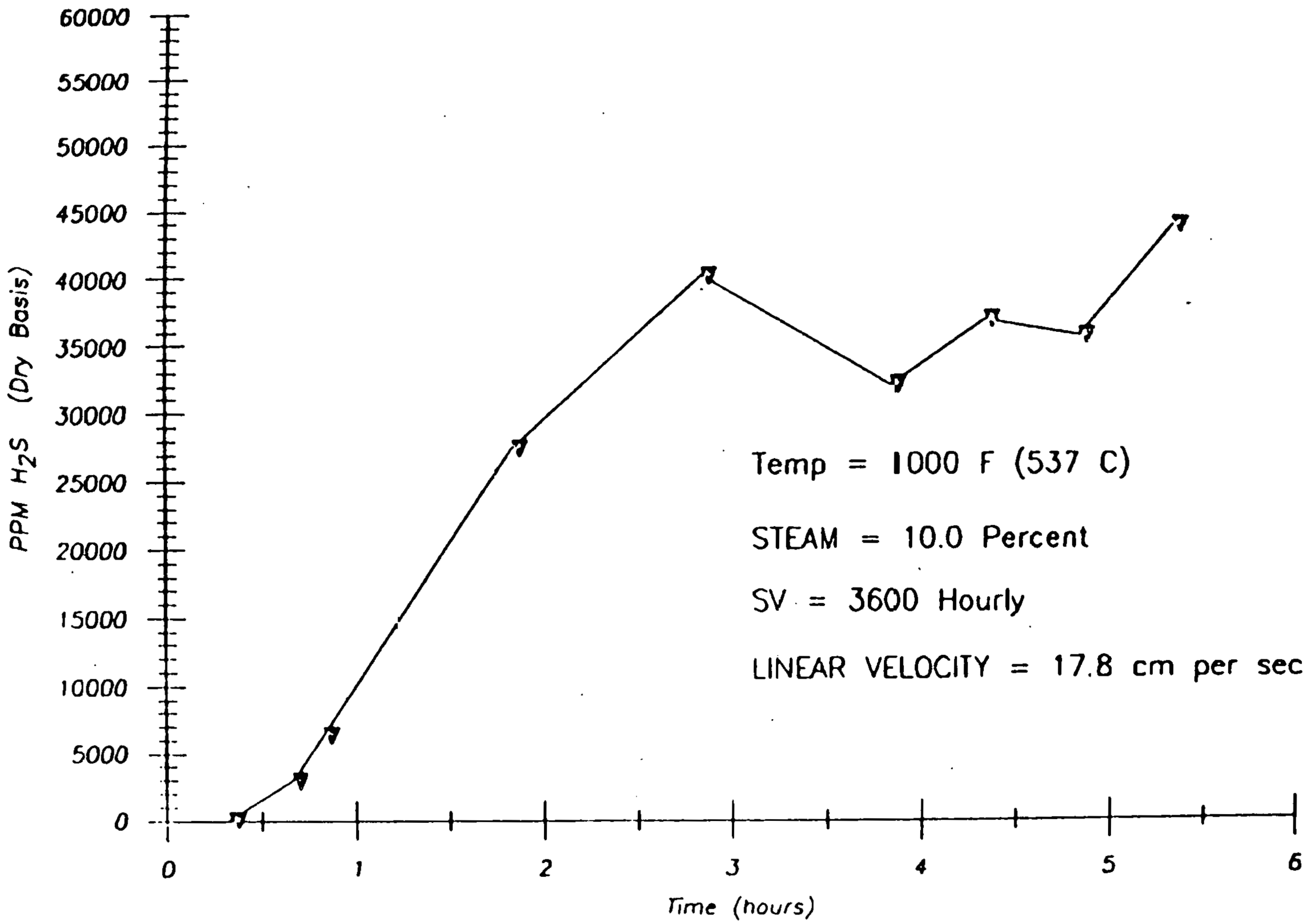
2. AT END OF RUN SAMPLE SORBENT PELLETS AT FOUR LOCATIONS ALONG THE LENGTH OF THE REACTOR. LABEL EACH SAMPLE AND SEND A PORTION OF EACH SAMPLE TO :
   1. SEM/EDAX (JIM KING) 3. SURFACE AREA/POROSITY (CHIDESTER)
   2. TOTAL SULFUR (ROMANOSKY)

CONCLUSIONS

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

ZINC FERRITE SULFIDATION

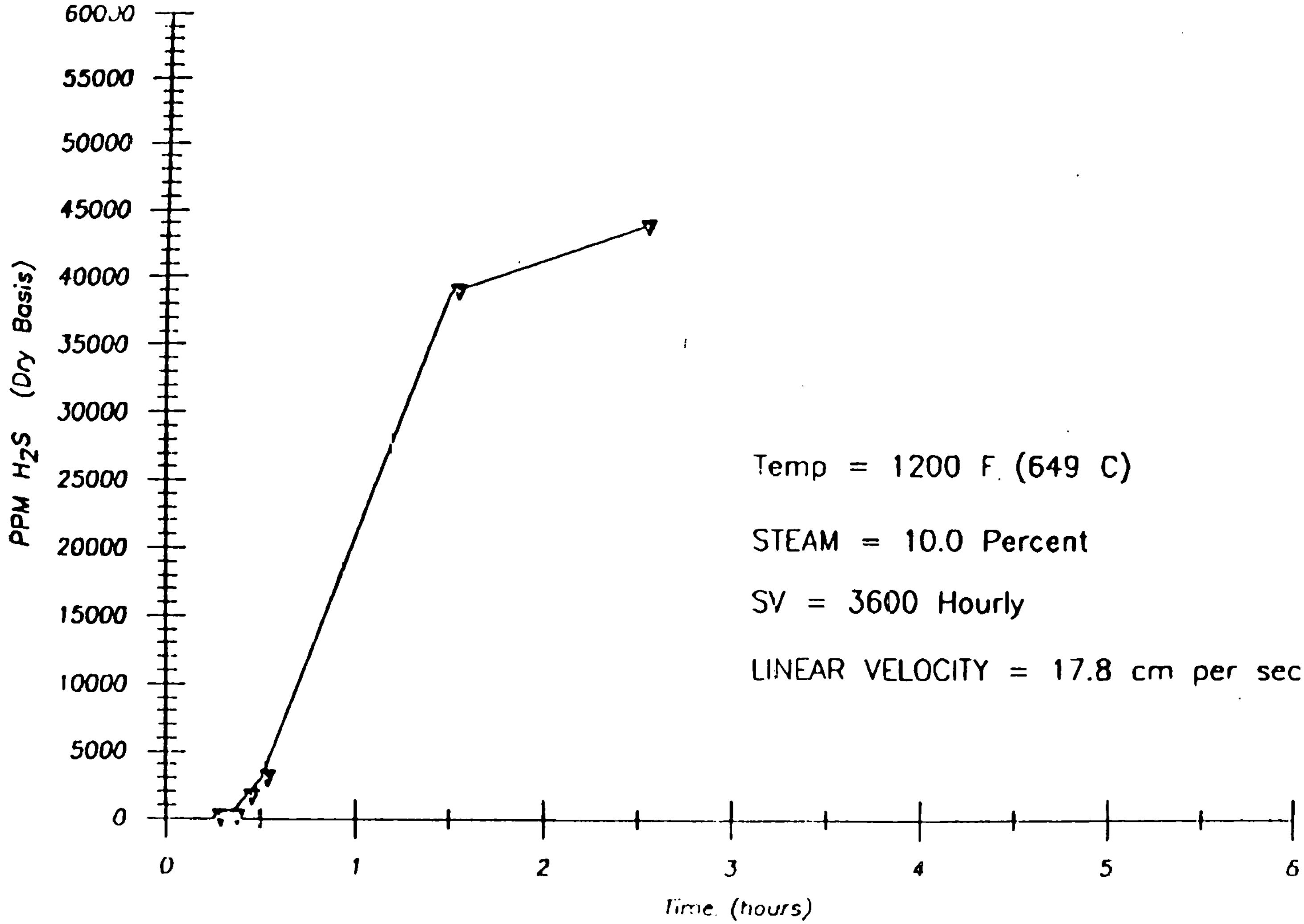
TEST 105 L-1443 UNITED CATALYSTS



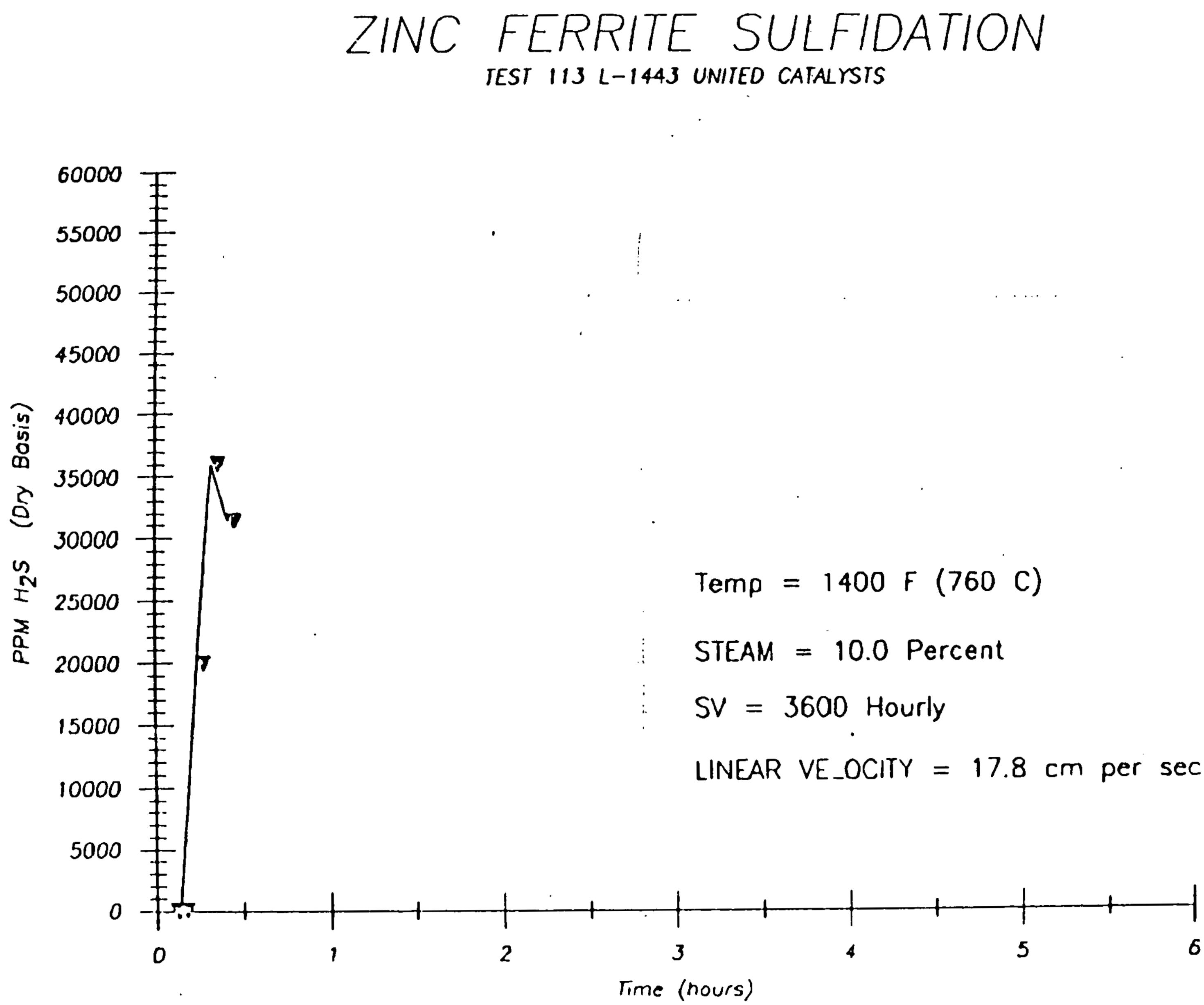
13-JAN-84 16:03:44

ZINC FERRITE SULFIDATION

TEST 107 L-1443 UNITED CATALYSTS



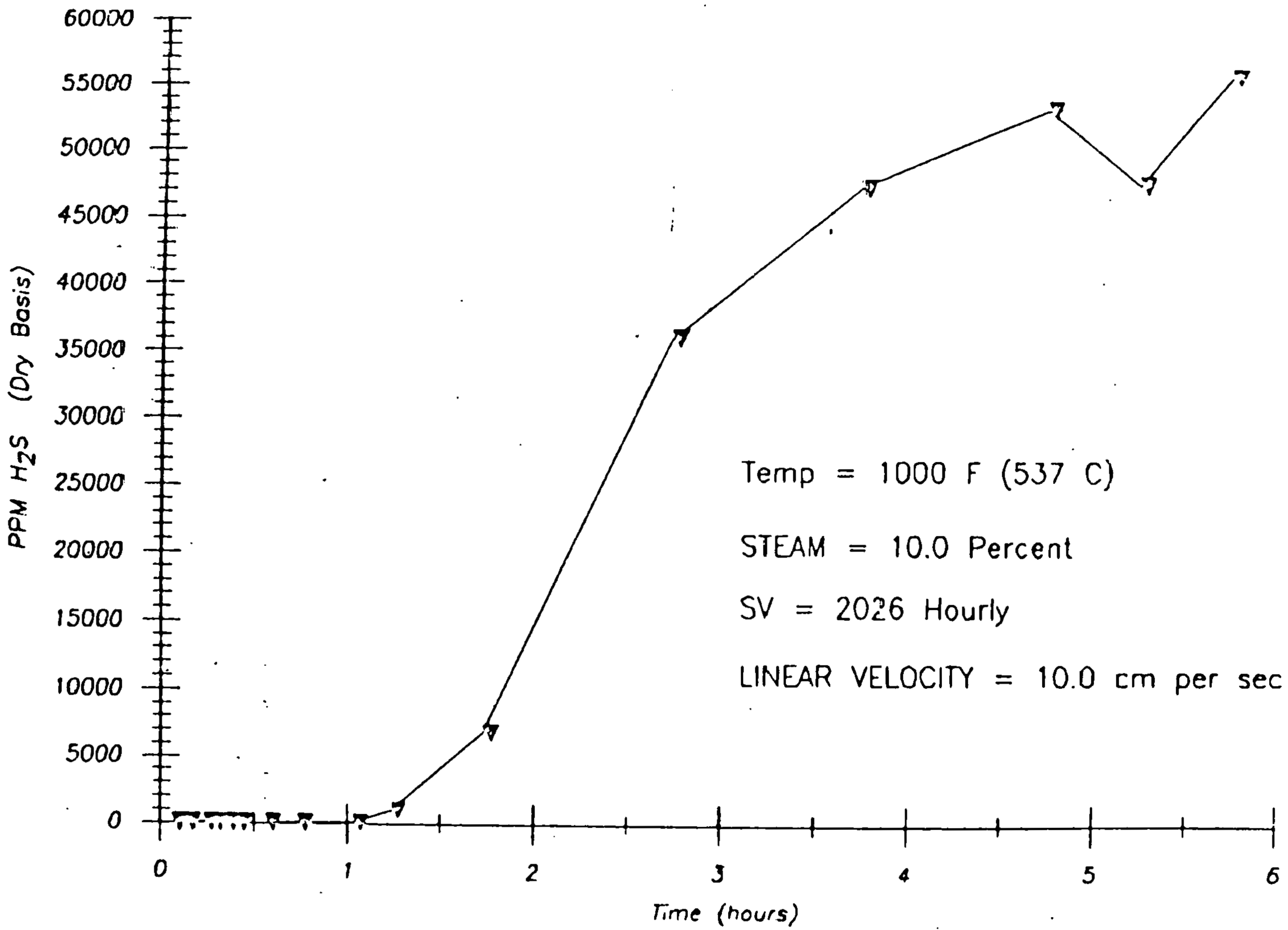
13-JAN-84 16:08:19



13-JAN-84 16:12:36

ZINC FERRITE SULFIDATION

TEST 115 L-1443 UNITED CATALYSTS

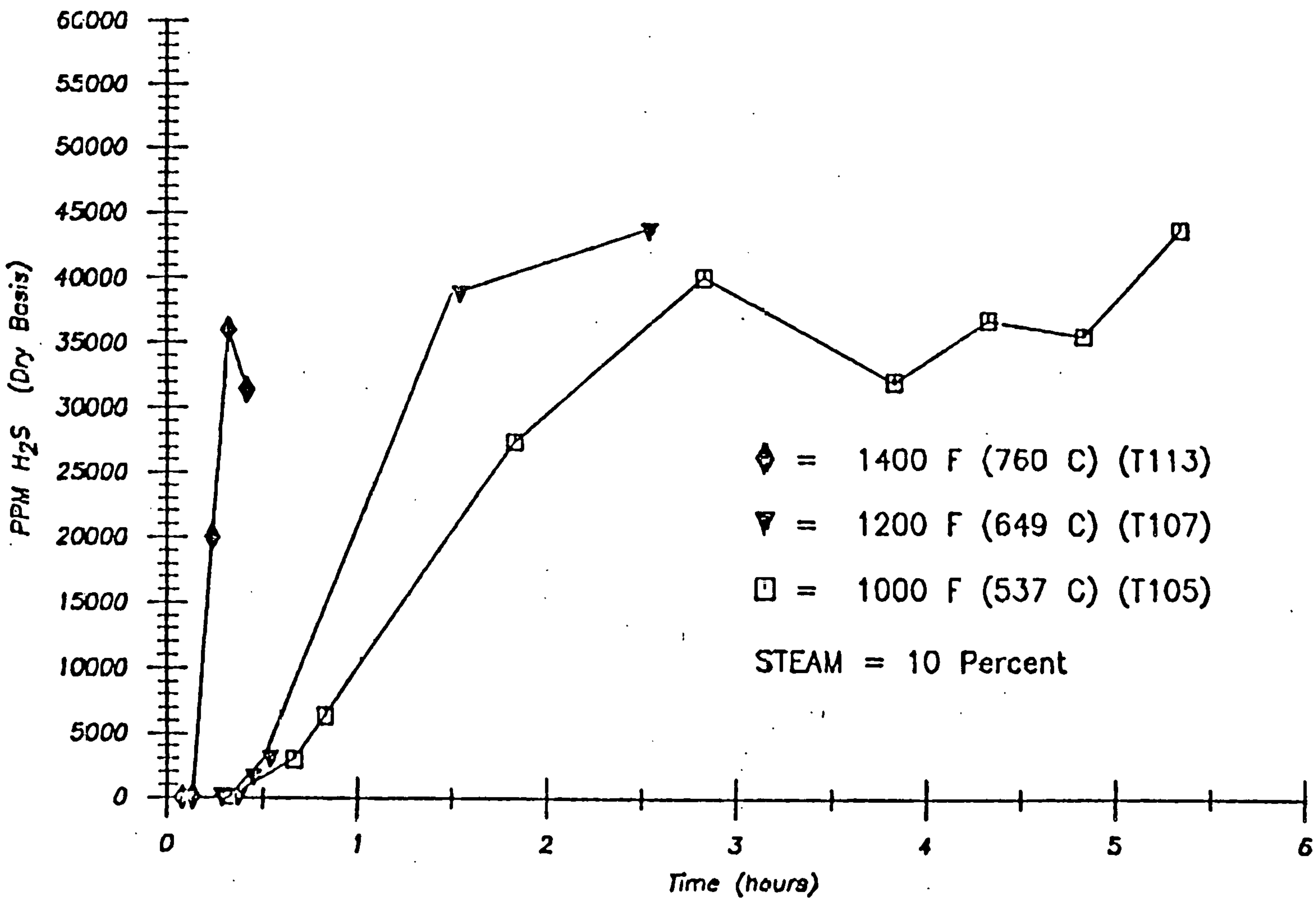


18-JAN-84 10:04:53

P1

ZINC FERRITE SULFIDATION

TESTS 105, 107, 113   L-1443 UNITED CATALYSTS

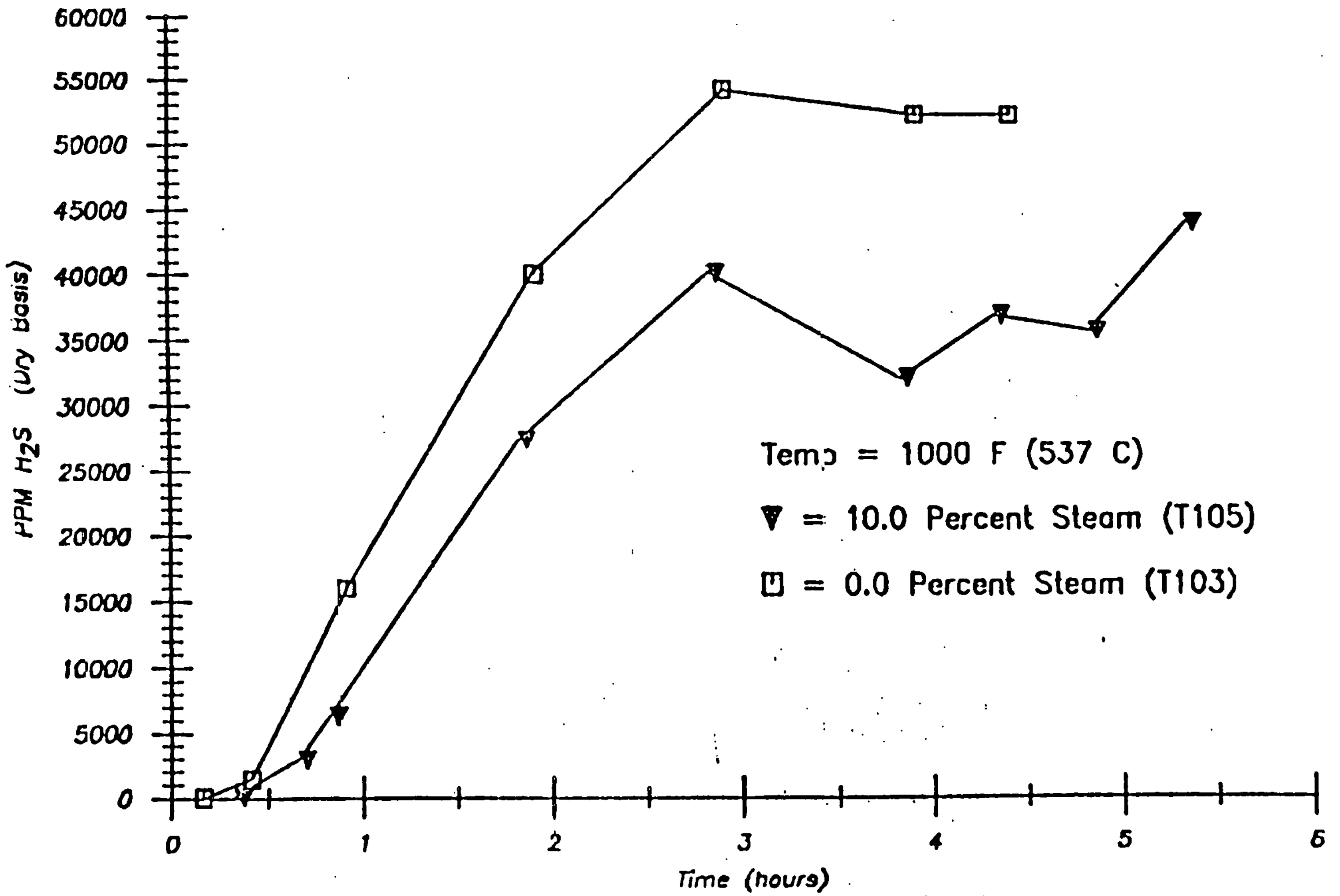


23-NOV-82 10:10:35

P1

ZINC FERRITE SULFIDATION

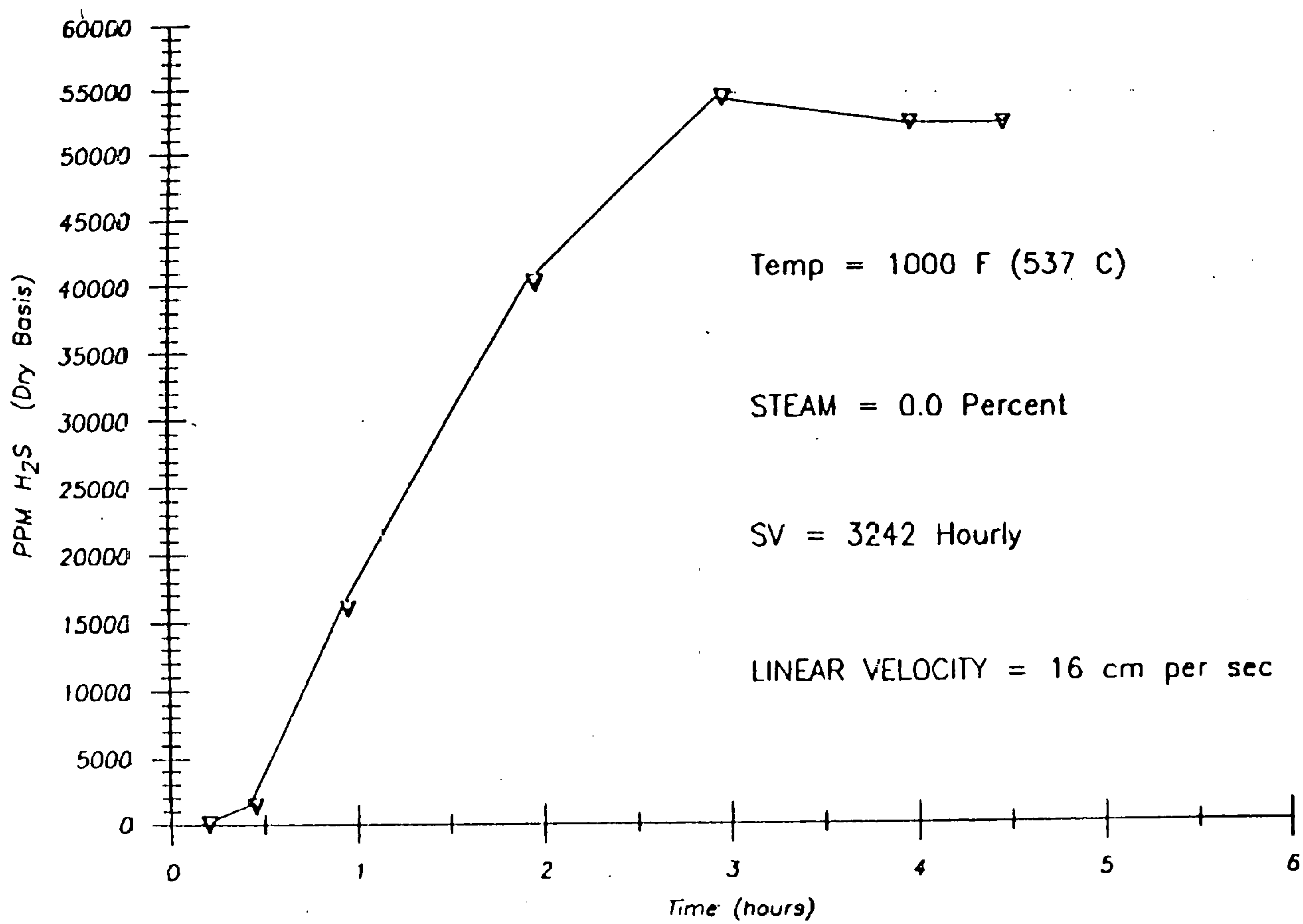
TESTS 103, 105 L-1443 UNITED CATALYSTS



23-NOV-82 09:58:40

ZINC FERRITE SULFIDATION

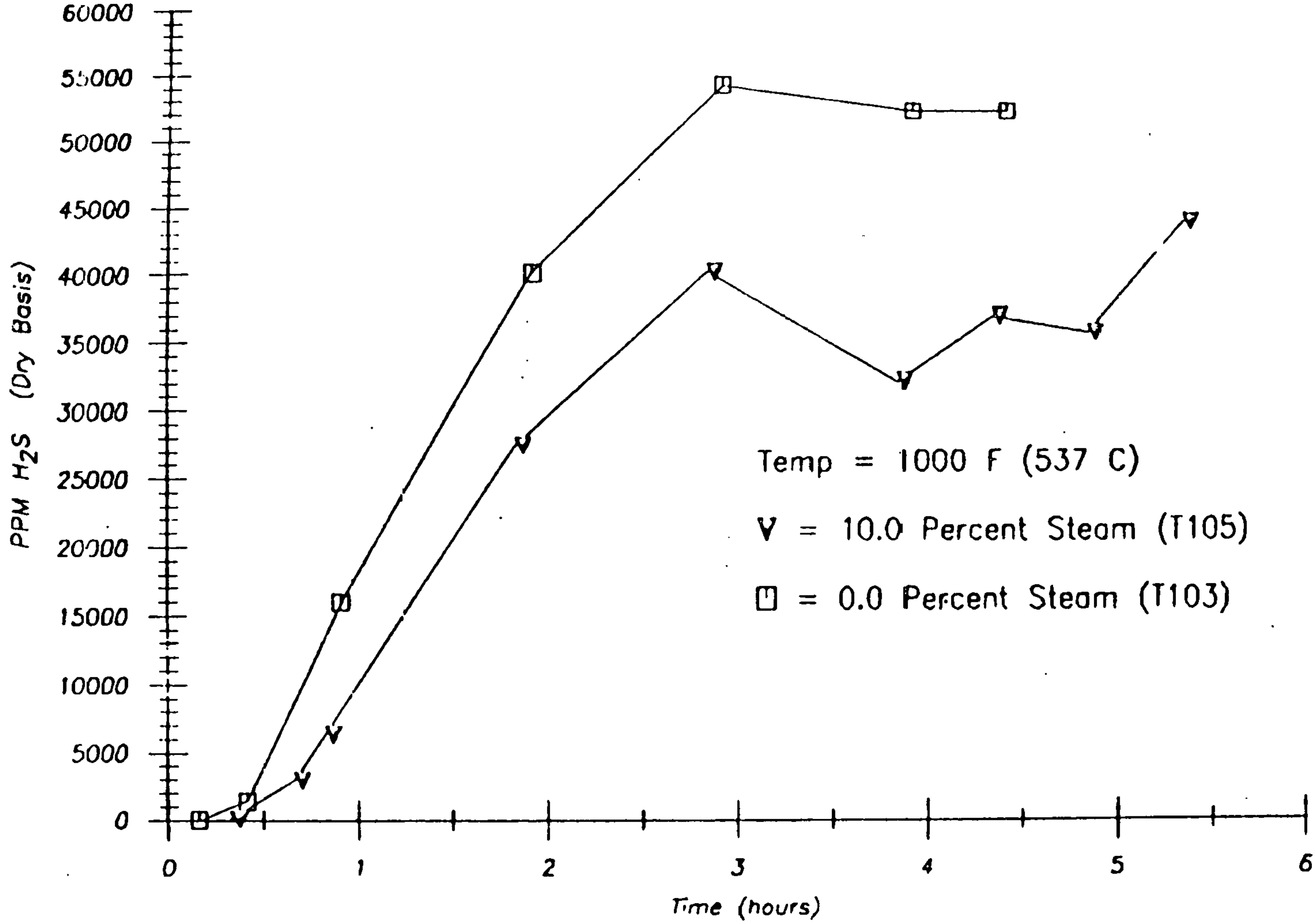
TEST 103 L-1443 UNITED CATALYSTS



13-JAN-84 16:01:37

ZINC FERRITE SULFIDATION

TESTS 103, 105   L-1443 UNITED CATALYSTS

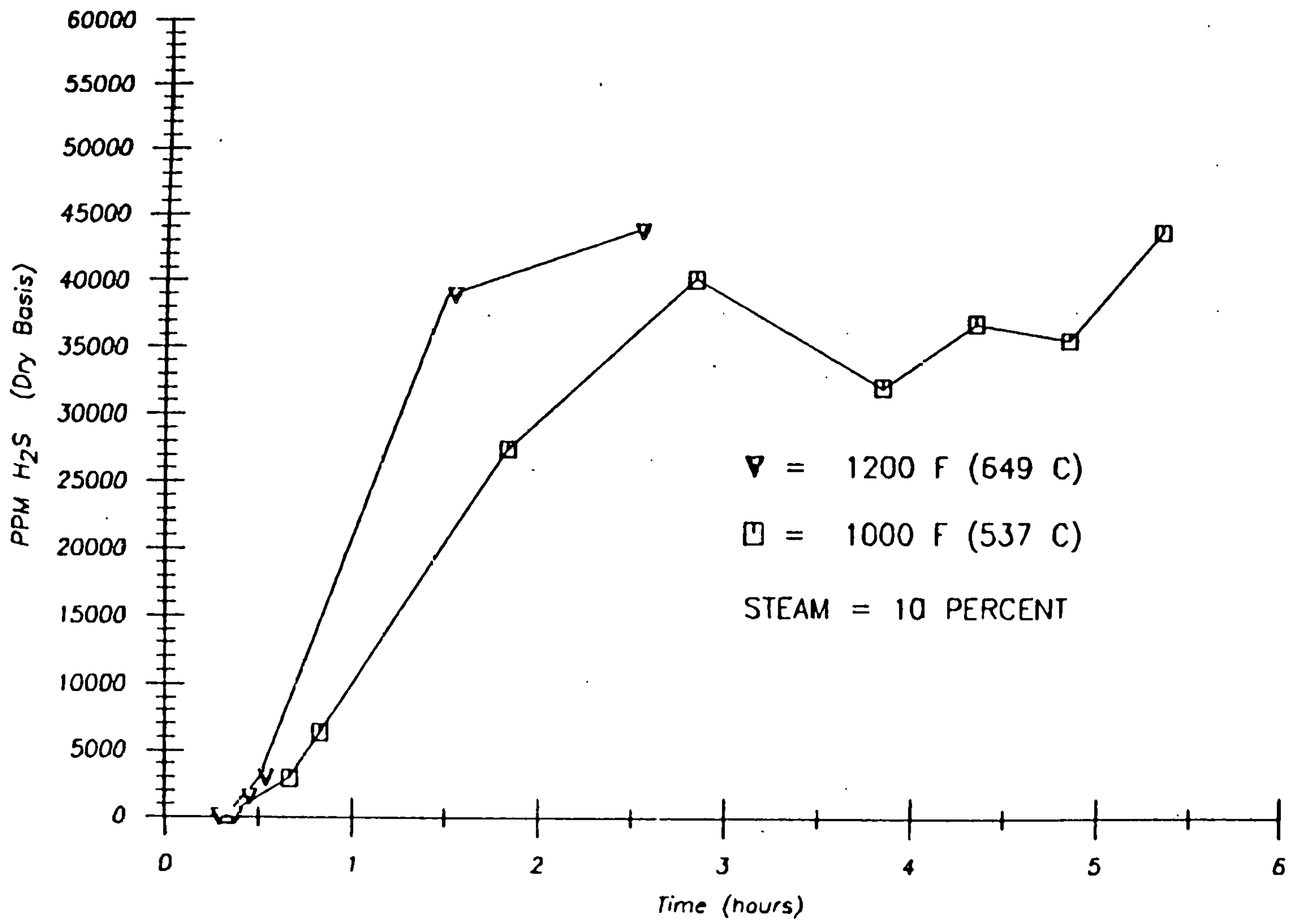


13-JAN-84 16:05:52

P1

ZINC FERRITE SULFIDATION

TESTS 105, 107 L-1443 UNITED CATALYSTS

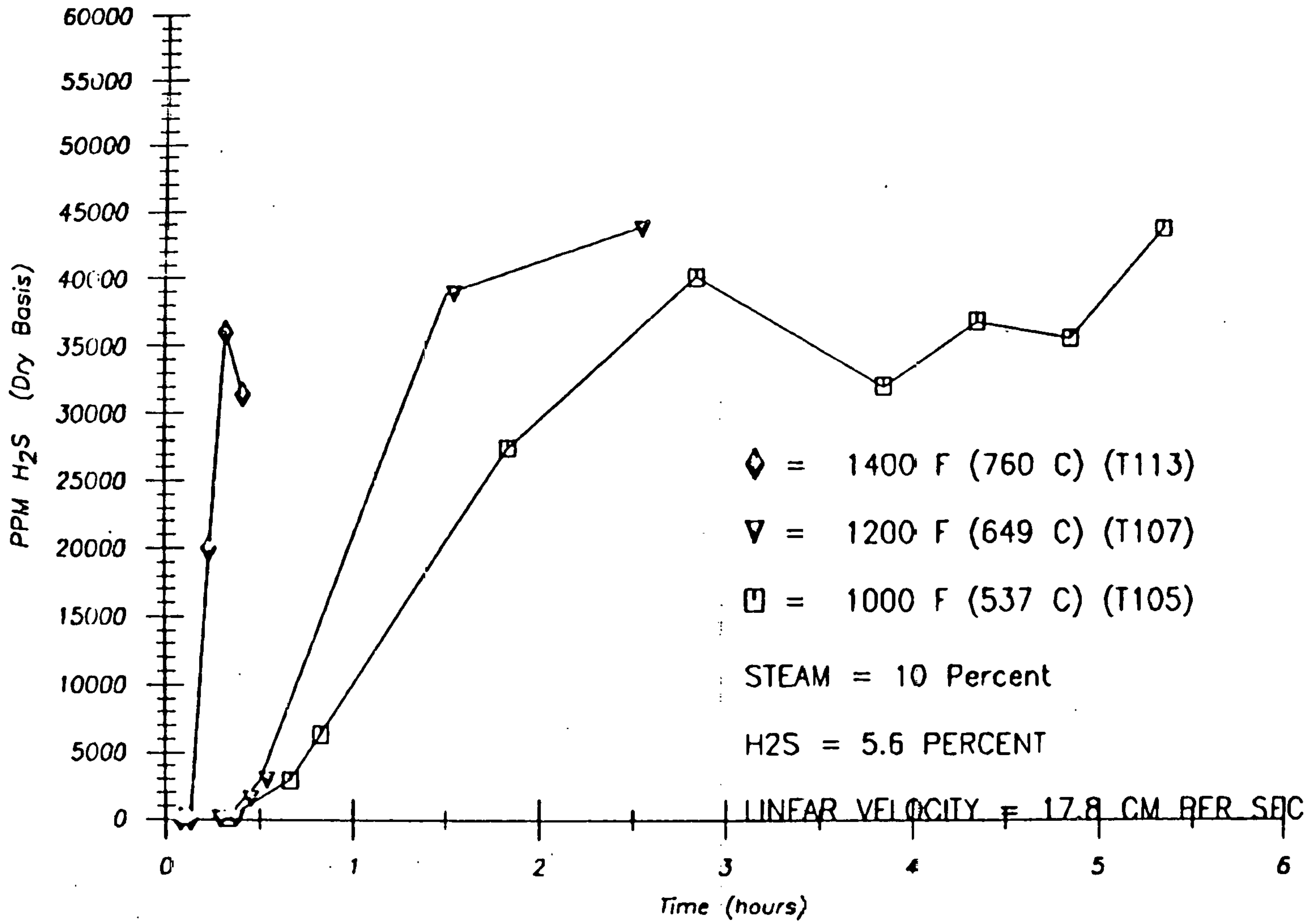


18-JAN-84 10:45:16

P1

ZINC FERRITE SULFIDATION

TESTS 105, 107, 113   L-1443 UNITED CATALYSTS

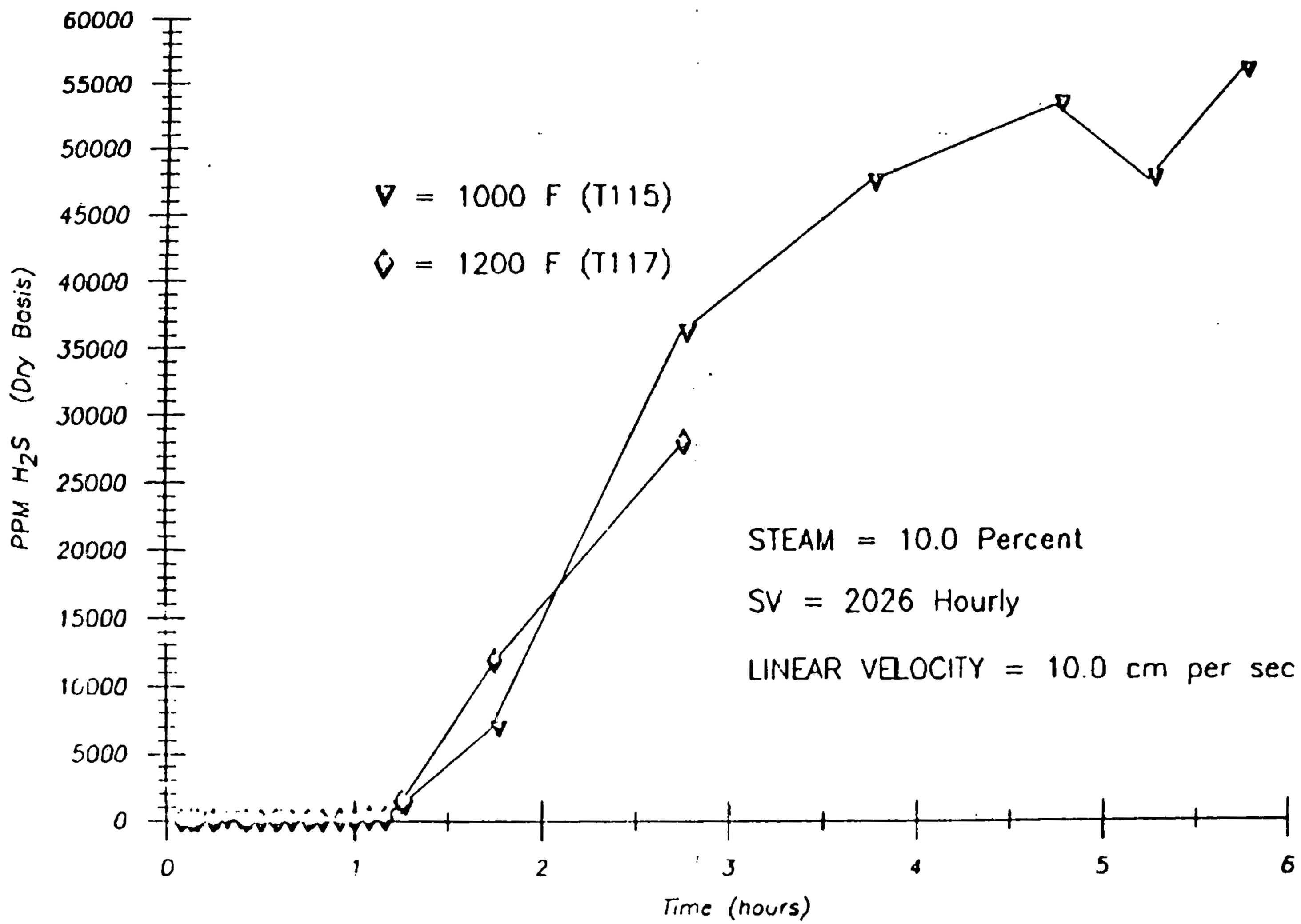


18-JAN 84 10:01:44

P1

ZINC FERRITE SULFIDATION

TEST 117 L-1443 UNITED CATALYSTS

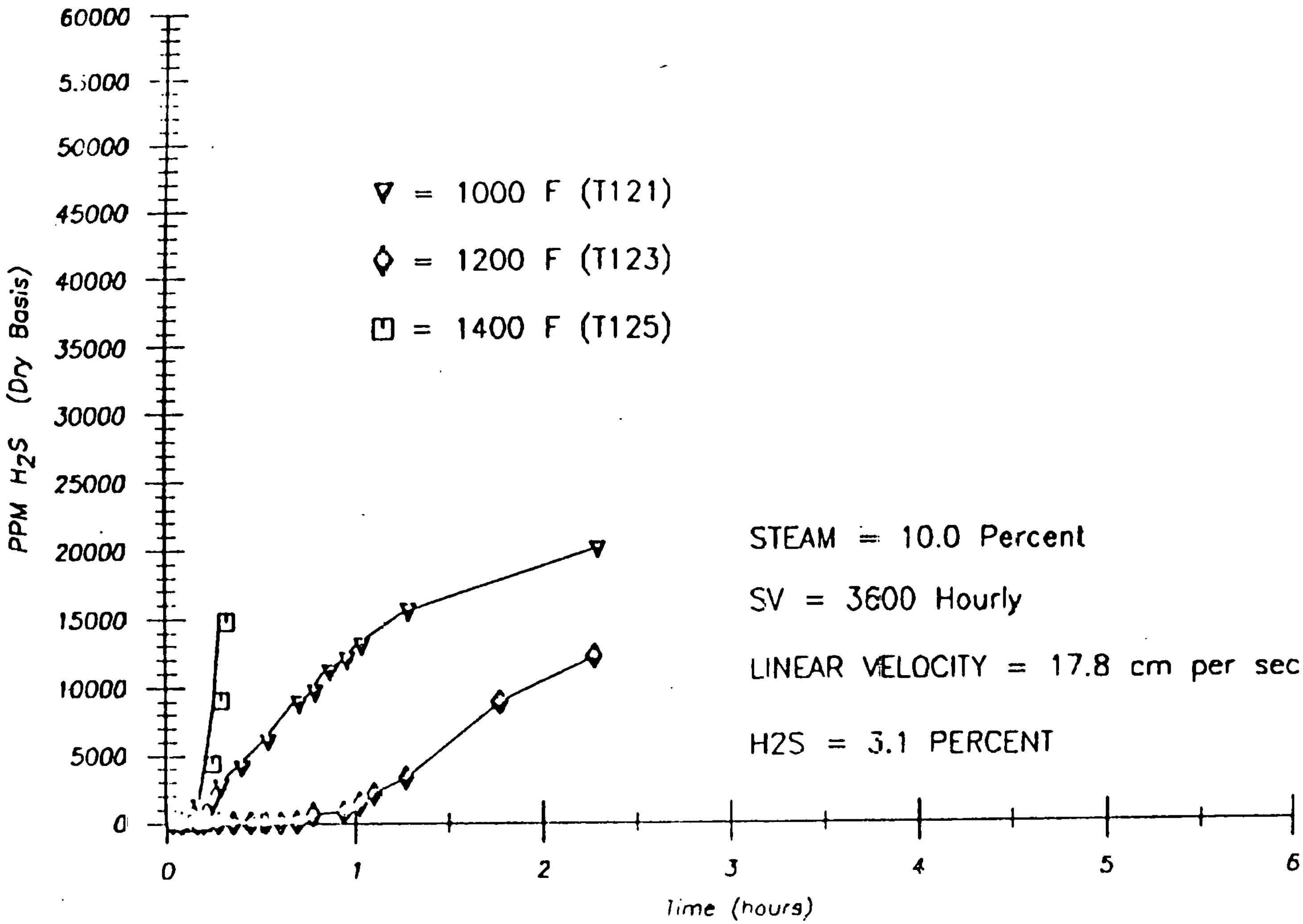


18-JAN-84 10:07:11

P1

ZINC FERRITE SULFIDATION

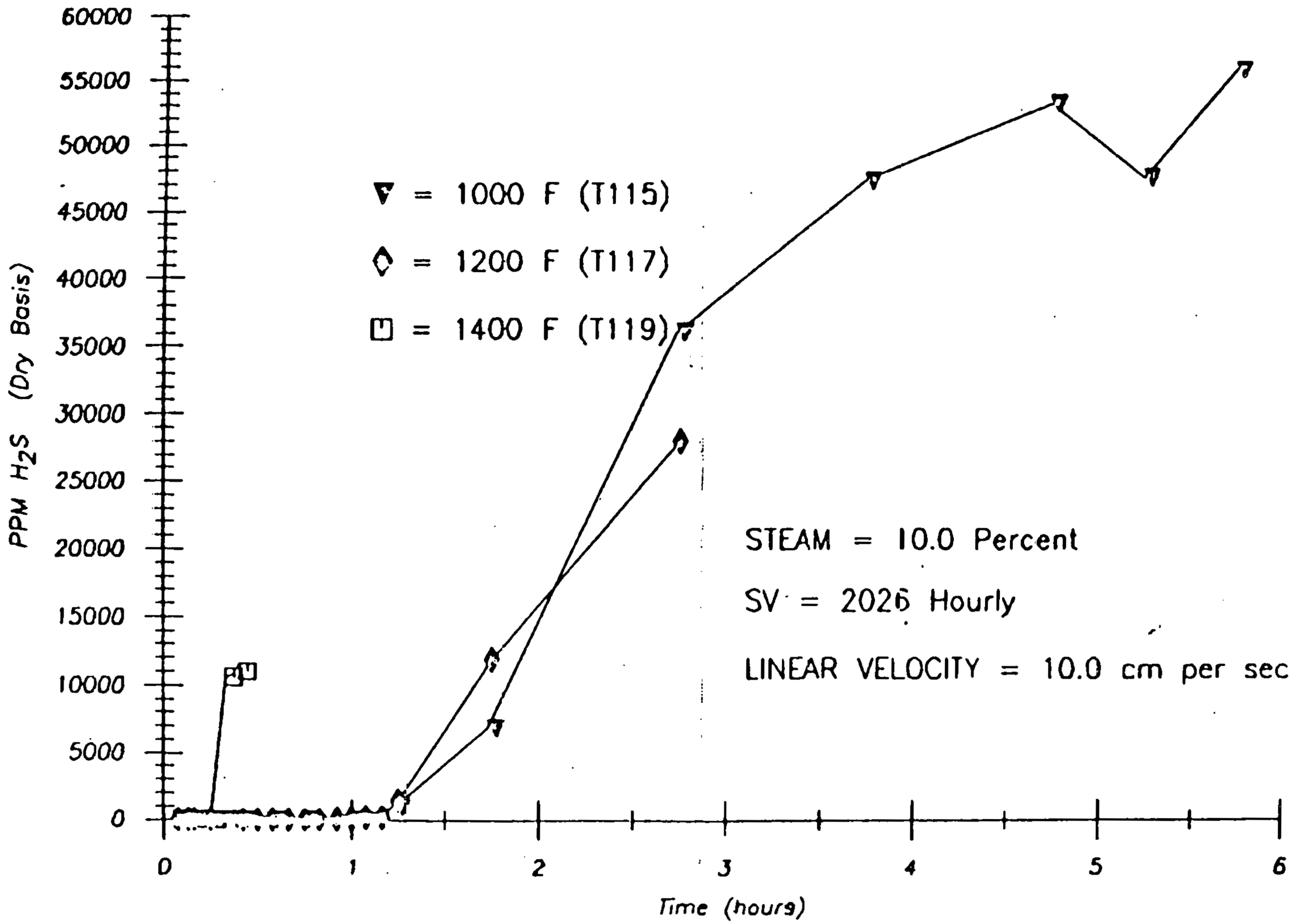
TESTS 121, 123, 125 L-1443 UNITED CATALYSTS



18-JAN-84 10:14:50

ZINC FERRITE SULFIDATION

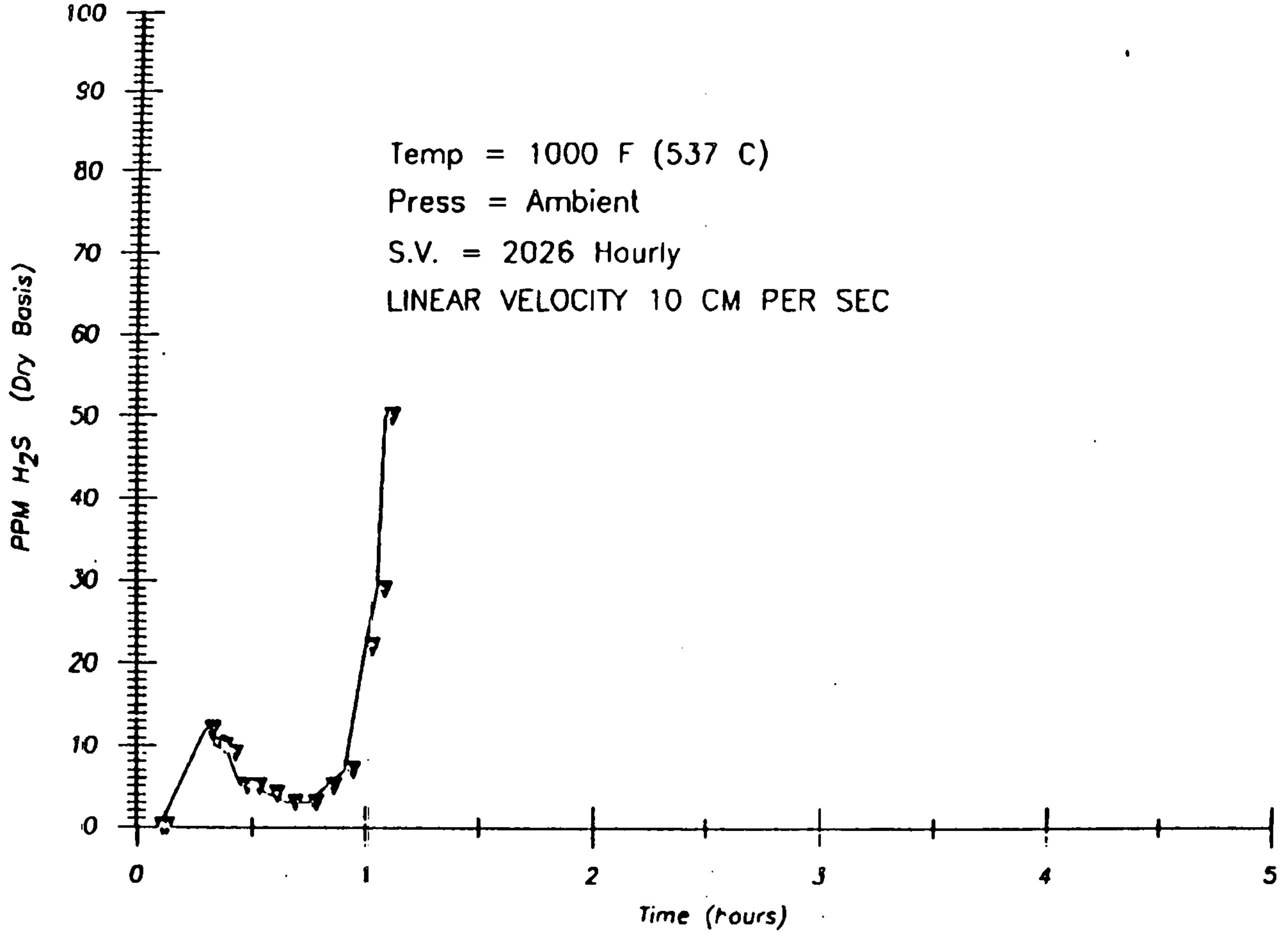
TESTS 115, 117, 119 L-1443 UNITED CATALYSTS



18-JAN-84 10:10:19

ZINC FERRITE SULFIDATION

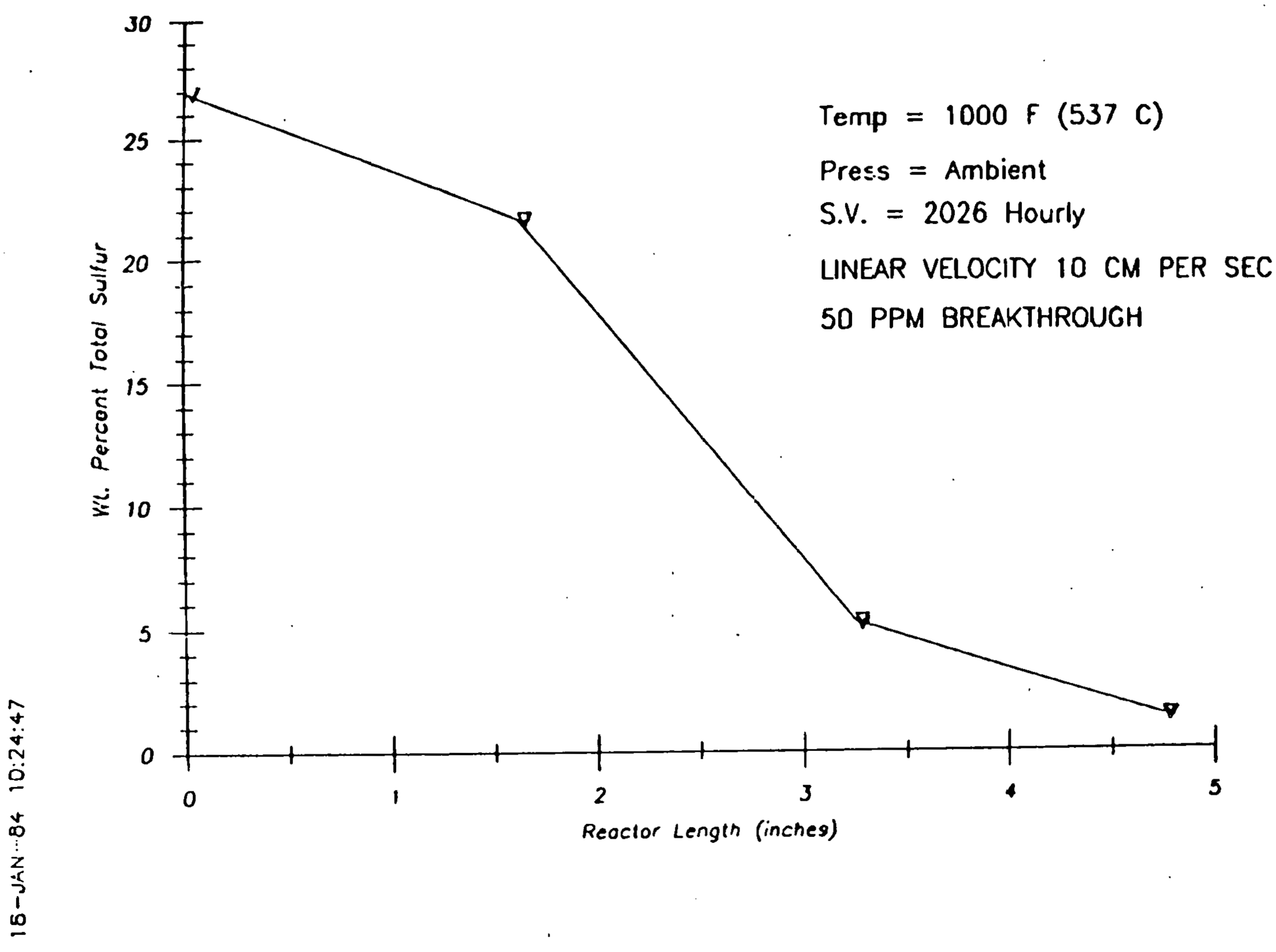
TEST 127



18-JAN-84 10:22:42

ZINC FERRITE SULFUR LOADING

TEST 127



POROS. Y DETERMINATION

L-1504

SAMPLE WSDF EK[illegible]

1. $ZnFe_2O_4$ (~92%) >2[illegible]

SAMPLE WEIGHT NORMALIZED TO 1 gram

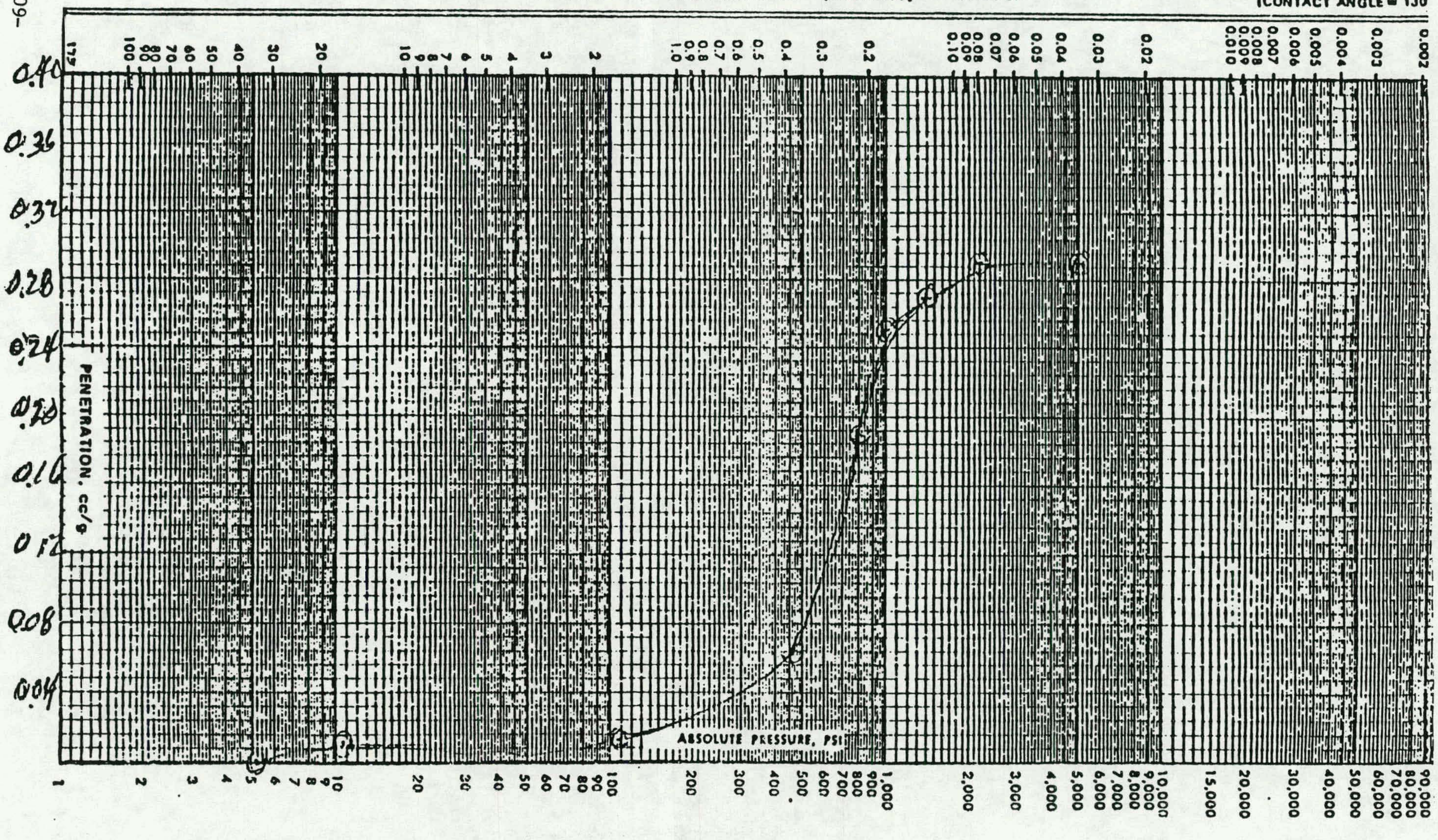
2. αFe_2O_3 (~4%) >20[illegible]

3. ZnO (~4%) >20[illegible]

DATE [illegible]

6.1 m²/g

EQUIVALENT PORE DIAMETER (MICRONS) = 175/PSI (CONTACT ANGLE = 130



L-1505

1. $ZnFe_2O_4$ (96%) 72000 Å
2. αFe_2O_3 (~2%) 72000 Å
3. ZnO (~2%) 72000 Å

3.9 m²/g

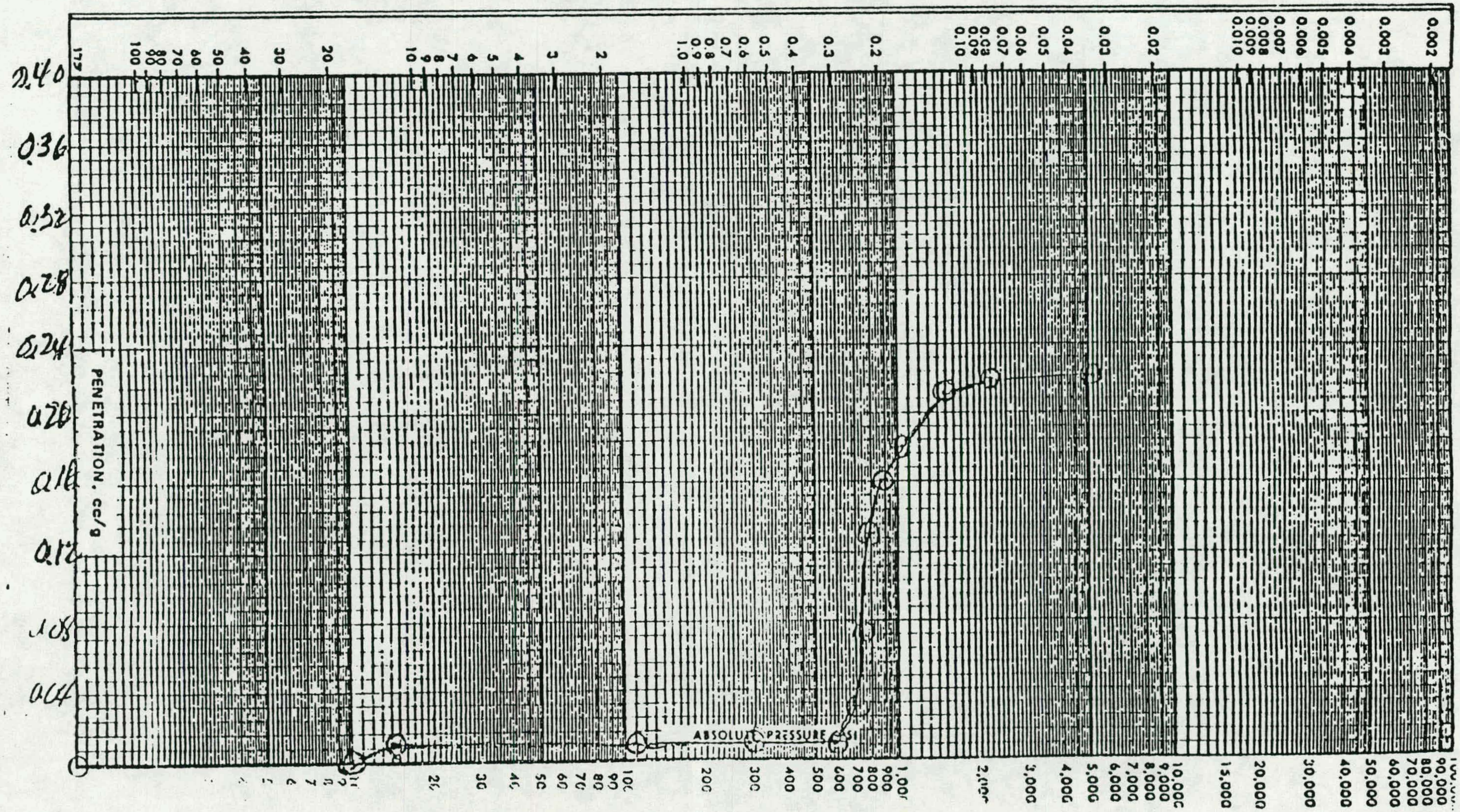
POROSITY DETERMINATION

SAMPLE USDE EKD 5281

SAMPLE WEIGHT NORMALIZED TO 1 gram

DATE 7/26/83

EQUIVALENT PORE DIAMETER (MICRONS) = 175/PSI (CONTACT ANGLE = 130°)



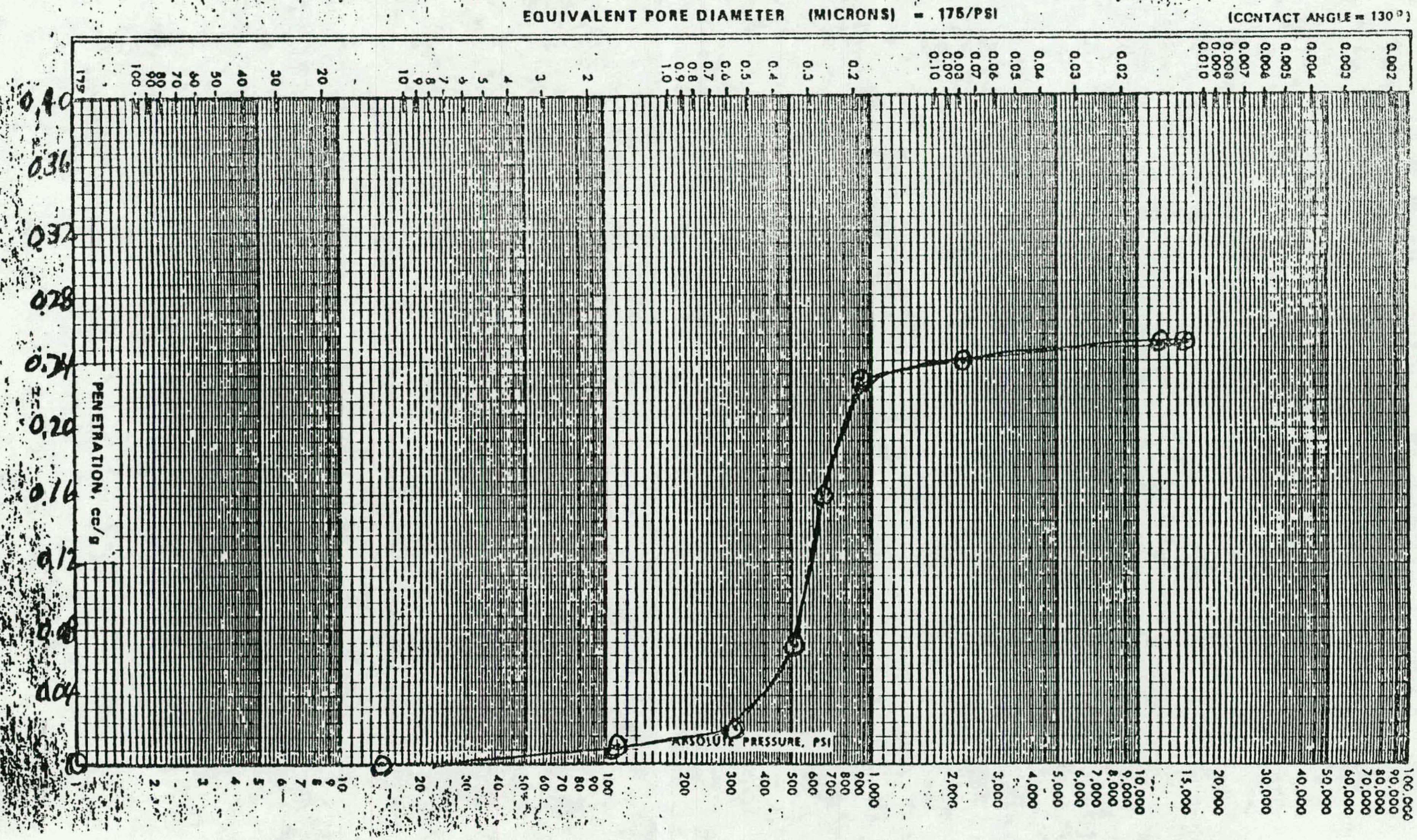
SR-1c 189 (5ft^3)
USDOE
Morgantown W.Va
L-1504

POROSITY DETERMINATION

SAMPLE G64 Type USDE EKD 7442

SAMPLE WEIGHT NORMALIZED TO 1 gram

DATE 11/23/82



TEST SUMMARY SHEET

TEST NO. : 120
DATE STARTED : 2/23/83
DATE ENDED : 2/23/83
TOTAL HOURS : 2.25 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/1000 GM
SORBENT NO. : L-1504
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)

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

SPACE VELOCITY: 1206 HOURLY

	FLOW RATE	MOLE%
H_2S	0.9963 SCFH	2.7%
H_2O	3.072CC/MIN	27.66%
AIR		
CO_2	3.6 SCFH	9.68 %
CO	4.3 SCFH	11.91 %
H_2	6.3 SCFH	17.12%
CH_4	1.07 SCFH	2.91%
N_2	10.34 SCFH	28.1 %

PURPOSE

SULFIDATION OF UNITED CATALYST L-1504
AT 1200 F.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H_2S: 1.5-5 PPM FOR 1.75 HOURS

EXIT SO_2: UNDETECTABLE

EXIT S_2:

EXIT H_2:

REMARKS

1. BAD TEMPERATURE CONTROLLER CAUSED OVERTEMPERATURING 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

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/1000 GM
SORBENT NO. : L-1504
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)

PURPOSE

SULFIDATIION OF UNITED CATALYST L-1504
AT 1200 F.

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

	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	11.01 %
H2	6.3 SCFH	17.12%
CH4	1.07 SCFH	2.01%
N2	10.34 SCFH	28.1 %

DATA: (DETECTOR TUBE)

EXIT H2S: 0.5 - 1.6 PPM FOR 1.5 HRS

EXIT SO2: 0 PPM

EXIT S2:

EXIT H2:

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

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 :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY: 385 HOURLY

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

PURPOSE

REGENERATION OF ZINC FERRITE PELLETS FROM TEST 129

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: MAX OF 7.6 PERCENT 610 PPM AT END

EXIT S2:

EXIT H2:

REMARKS

1. AIR FLOW RATE INCREASED TO +6.6 SCFH AT 4.75 HRS.

2. DID NOT OBTAIN TYPICAL TEMP WAVE AT 5.00 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

SORBENT TYPE/WEIGHT: (ZINC FERRITE)/1030 GM
SORBENT NO. : L-1504
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)

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : AMBIENT

SPACE VELOCITY: 1286 HOURLY

	FLOW RATE	MOLE%
H2S	0.0063 SCFH	2.7%
H2O	3.872CC/MIN	27.66%
AIR		
CO2	3.6 SCFH	9.68 %
CO	4.3 SCFH	11.01 %
H2	6.3 SCFH	17.12%
CH4	1.07 SCFH	2.91%
N2	10.34 SCFH	28.1 %

PURPOSE

SULFIDATIION OF UNITED CATALYST L-1504
AT 1200 F A SECOND TIME.

ANALYSIS:

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

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:

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 : 3/16/83
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)/1020 GM
SORBENT NO. : L-1584
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

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 8 % MAX

EXIT S2:

EXIT H2:

REMARKS

1. STOP REGENERATION WHEN TEMP WAVE IS AT MID BED.

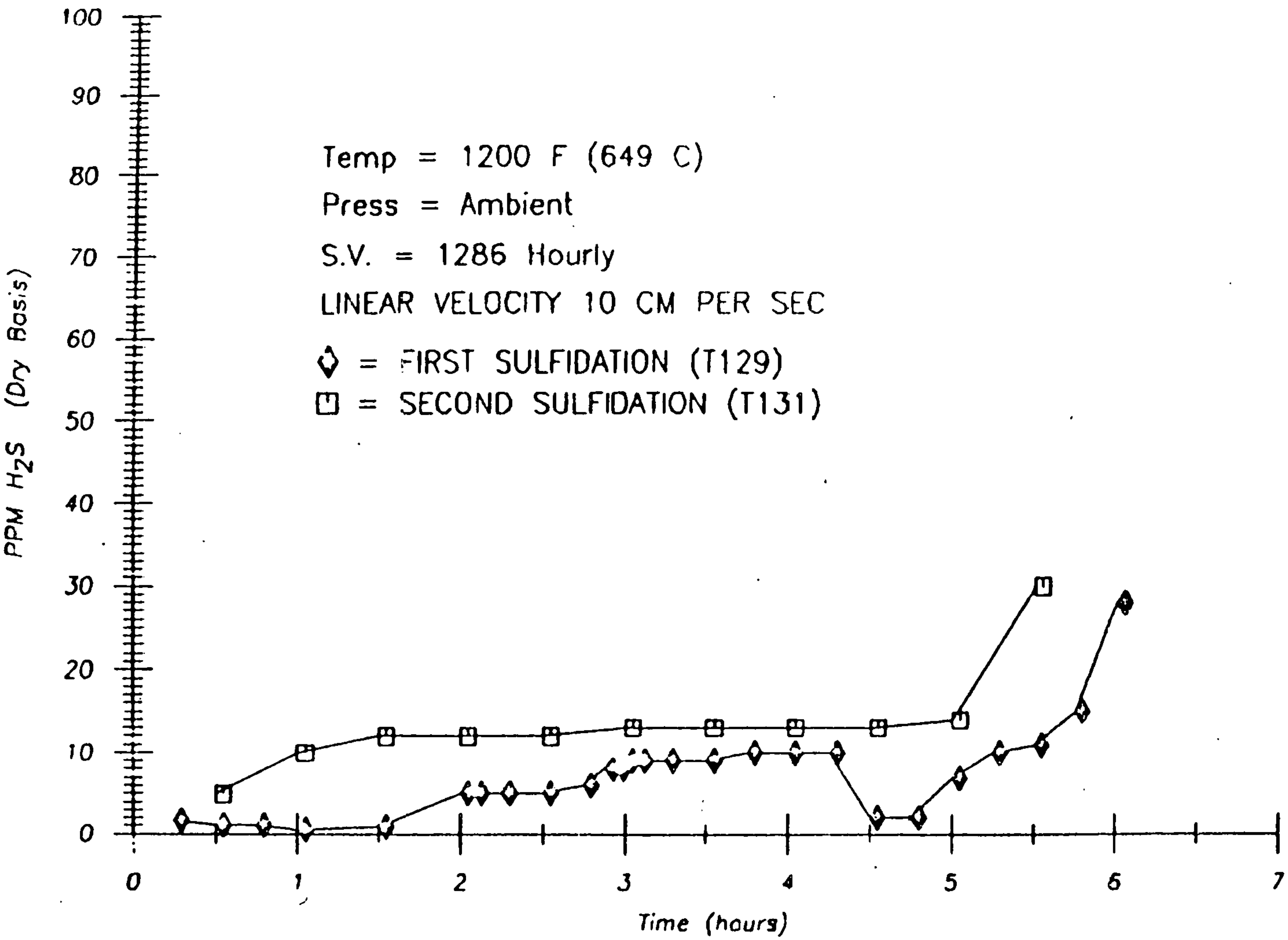
2. SAMPLE PELLETS BEFORE , AT , AND AFTER TEMP WAVE AND SEND TO JIM KING FOR ELECTRON MICROSCOPE ANALYSIS.

CONCLUSIONS

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

ZINC FERRITE SULFIDATION

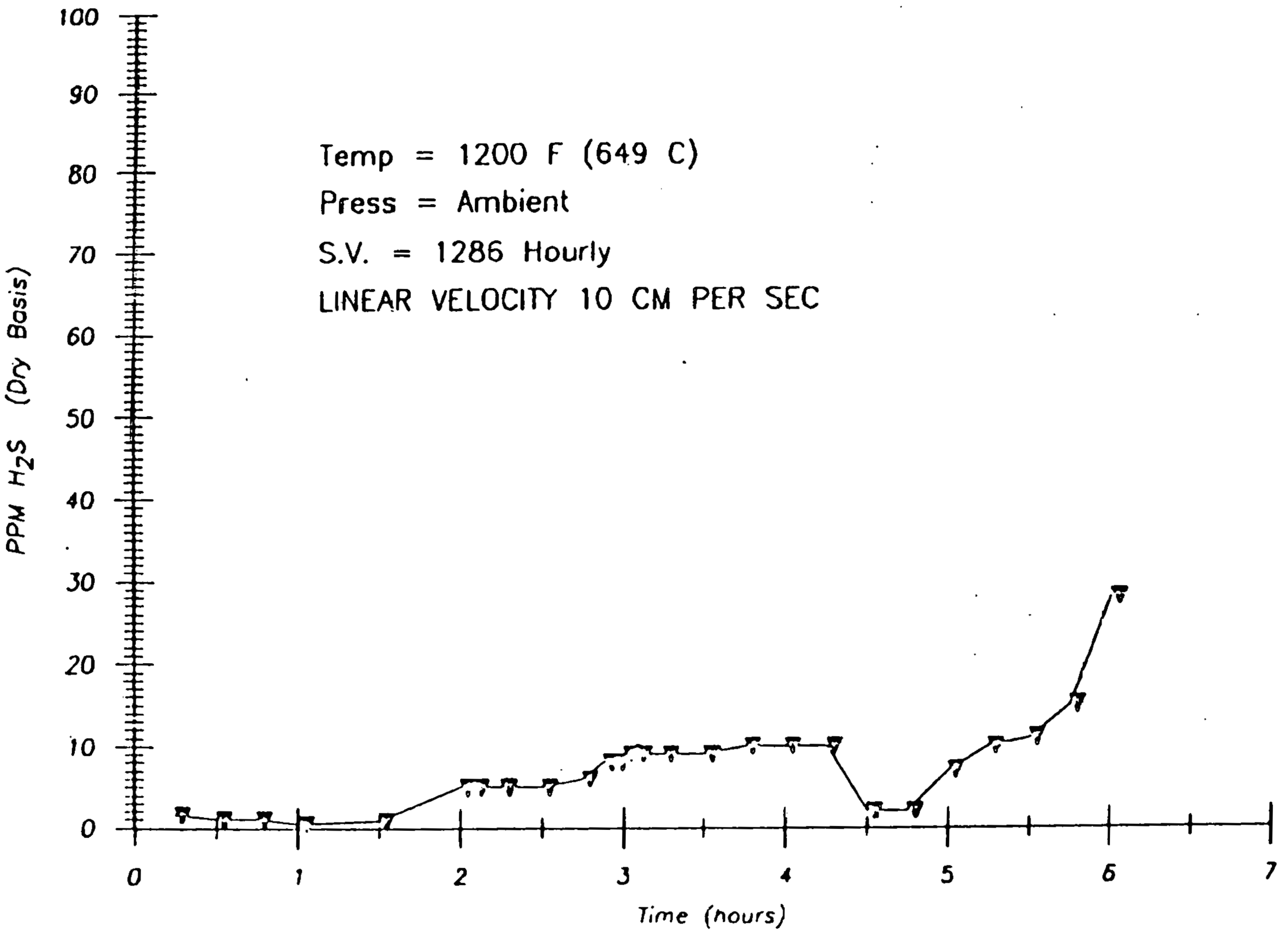
TESTS 129,131 (L1504)



18-JAN-84 10:29:17

ZINC FERRITE SULFIDATION

TEST 129 (L1504)



APPENDIX E

Sidestream Testing - Gasifier Run 101

Hot Gas Desulfurization Sidestream Test Unit, Fixed-Bed Gasifier Run 101

Introduction

Starting in 1980, laboratory-scale evaluation of developmental, regenerable solid sorbents to remove sulfur compounds from cool gas at high temperatures has been carried on at METC (1,2,3,4,5,6). Tests were performed, utilizing simulated gases, for sorbent screening and development, with the long-range objective of testing the most promising sorbents in a bench-size test unit at a somewhat larger scale in sidestreams of METC coal gasifiers. Initially, this would be in a sidestream of METC's 24 t/d fixed-bed gasifier and later, the ~ 2 t/d advanced fluid-bed gasifier.

The test unit was designed, and procurement of components practically completed during FY 82. During FY 83, the unit was assembled and installed in a location where it could be connected to either gasifier. Skid-mounting of the unit gives it portability should it be desired to remove it to another test site.

The design throughput of the sidestream test unit is an order of magnitude larger than that of the laboratory-scale unit. The bigger size of the sidestream test unit and the treating by it of an actual hot raw coal gas were expected to establish with much greater confidence the capability of sorbents under test to achieve the desired goal of removing sulfur compounds to low levels (1-10 ppm) of withstanding the effects of other contaminants and of being regenerable.

In particular, the sorbent, zinc ferrite, which showed promise in the laboratory-scale tests, was singled out for evaluation.

Design of Sidestream Test Unit

The process and instrument drawing of the sidestream test unit is shown in Figure 1. Figures 2 and 3 are perspective schematics which illustrate the operation of the unit in absorption and regeneration modes. Figure 4 is a photograph of the unit and its control panel installed at its test location. The detailed design basis was by Science Applications Inc. (4). Detailed mechanical engineering design was by Tech Engineering and Design of Morgantown and assembly, fabrication of subsidiary components and installation was by METC staff.

The principal component is a 6-inch diameter, 6-foot long reactor fabricated from Incoloy 800H by National Annealing Box Company and treated internally with aluminum (alonized) by Alon Processing, Inc. It is designed for operation up to a pressure of 300 psig and a temperature of 1,800°F. The reactor, which has six thermocouples inserted along its length, is enclosed by a four-zone, clamshell type electrically heated furnace made by Mellen Company.

Coal gas and regeneration gas entering the reactor are heated by passage through helical coils located in two electrically heated cylindrical furnaces made by Thermcraft, Inc. Gases leaving the reactor are cooled by water-cooled condensers in two stages: the first to condense the higher boiling tars and the second to condense steam and remaining tars. On-off valves for the hot gases are of the sliding disc type made by Everlasting Valve Company. The valves and piping are of 316 stainless steel except for the 1¼-inch diameter line from the fixed-bed gasifier which is of 309 stainless steel. A 1¼-inch line of alonized 304 stainless steel from METC's advanced fluid-bed gasifier is presently at the engineering stage. The piping is electrically heated by Chromolox tubular heaters.

Cooled exiting gas can be continuously sampled and analyzed by a Bendix Corporation on-line chromatograph for major species and by a Baseline Industries on-line chromatograph for H_2S, COS, and SO_2. The inlet hot coal gas can also be continuously analyzed by the Bendix chromatograph after passage through an apparatus which condenses out steam and tars.

Additional provision is made to sample hot coal gas at the reactor inlet and outlet for particulate characterization.

Condensates can be drained via holding vessels into drums so that periodic sampling and weighing can be carried out.

Process data are logged by means of an automatic data acquisition and control system (ADACS) and can be displayed on a local terminal.

Operation of Sidestream Test Unit

The sidestream desulfurization unit was operated for the first time during Run 101 of METC's 42-inch, fixed-bed gasifier (7,8,9). This run took place from June 22 to July 1. During this 9-day period while Arkwright coal was fed to the gasifier, the unit was operated in the absorption mode with upward flow for three spells with two intermediate spells in the regeneration mode with downward flow. The schedule of operation is shown in Figure 5. The first two absorptions were with air-blown (low-Btu) gas and the third with intermittently air-blown and oxygen-blown (medium-Btu) gas.

Reactor furnace temperatures were adjusted to give a temperature profile along the reactor as close as possible to 1,000°F.

The reactor contained an approximately 4-foot deep layer of zinc ferrite extrusions weighing 20.4 kg, supplied by United Catalysts, Inc., with 6-inch layers of ½-inch ceramic spheres above and below, the whole held in place by support and cover plates.

Average pressures, temperatures and flow rates of the various streams for the three sulfidation runs are shown on ADACS process flow schematics in Figures 6A, 6B, 7, and 8.

During sulfidation Runs 001 and 003, approximate reactor conditions were: pressure 150 psig, temperature 1,000°F, and space velocity 2,000 hr^{-1}, based on condenser exit gas. In sulfidation Run 005 for air-blown operation, conditions were the same except for a lower pressure of about 90 psig. For oxygen-blown operation, owing to the large steam content of the gas, the space velocity was considerably greater since the exit gas flow rate was maintained constant. The duration of Run 001 was about 29 hours, interrupted by a few hours when the gasifier was off stream. Run 003 was about 25 hours long. The length of Run 005 is not comparable because of the intermittent operation with air- and oxygen-blown gas. But, taking into account the differing rates of mass flow of sulfur compounds into the reactor, the equivalent run length is estimated to be roughly the same as that of Runs 001 and 003.

Reactor exit gases were cooled to about 600°F by the first water-cooled condenser and then to about 130°F by the second water-cooled condenser except during oxygen-blown operation when the temperature was somewhat higher because of the greatly increased heat load. The pressure drop across the reactor was initially less than 1 psi at the beginnings of the first two sulfidations increasing to about 5 psi towards the ends. Regeneration restored the pressure drop approximately to its original level. The pressure drop at the beginning of the third sulfidation was somewhat greater than that of the first two because of the greater flow rate and only increased to about 2 psi at the end because of the shorter run length. The increases in pressure drop are attributed mainly to particulate deposition. Coke, resulting from pyrolysis of tars and/or from carbon monoxide by the Boudouard reaction: $2CO \rightarrow C + CO_2$, is probably also a contributory factor.

The pressure drop across the small secondary cyclone gradually increased from 0.2 to 0.4 psi during Runs 001 and 003 and then rapidly increased during the oxygen-blown intervals of Run 005 so that, at the end of this run, the combined pressure drops across the cyclone and the inlet gas line were about 30 psi. This blockage is attributed mainly to deposition of coke from tars in the gas. When the run was over, the blockage was cleared by blowing an air-steam mixture through the line.

The zinc ferrite sorbent was regenerated twice in Runs 002 and 004 with a regeneration gas typically consisting of an about 80/20 vol. percent steam/air mixture at a temperature of 1,000°F. Space velocity was usually 500-1,000 hr^{-1} based on reactor flow. Some difficulty was experienced in controlling the air and steam flows, so that the sorbent temperature occasionally exceeded the design maximum of about 1,600°F. Figures 9 and 10 are ADACS plots of the sorbent temperature along the reactor throughout the regenerations. A somewhat lower air flow rate during Run 004 resulted in a rather longer regeneration time of 19 hours compared to 15 hours for Run 002.

The Baseline Industries gas chromatograph became fouled towards the end of the first sulfidation by oil droplets in the exit gas from the final condenser. For future tests a special Permapure Filter System was subsequently acquired.

During the sulfidation and regeneration runs, hydrogen sulfide and/or sulfur dioxide concentrations of exit gas were measured hourly by detector tubes. Grab samples of exit gas and condensates were taken generally at 4-hour intervals for laboratory analysis. Major species in the exit gas during the sulfidations were measured continuously by the Bendix chromatograph. Also, during the sulfidation runs, occasional samples of hot gas at the reactor inlet and outlet were taken via 1-inch Kamyr valves through a particle sampling system to determine particle loadings and sizes. Liquids, which were condensed from these samples, were analyzed (10).

At the conclusion of the last sulfidation run, which was continued well beyond breakthrough, the sulfidized sorbent was removed, samples being taken at points along its length. Fines were removed by screening. A lump of agglomerated sorbent extrusions about 4 inches deep was found near the top of the bed. This appeared to be caused by coke deposition.

Results

- Data Summary

  The various process conditions and data collected in Runs 001 to 005 are summarized in Figures 11 to 15.

- Absorption of Sulfur Compounds

  Figure 16 is a plot of the hydrogen sulfide concentration of exit gas, after condensation of tar and water, against on-stream time for Run 001. Measurements were made with both on-line gas chromatograph and detector tube which agreed quite well. Until breakthrough after about 28 hours, H_2S levels were below 5 ppm averaging about 2 ppm. After Run 001, the gas chromatograph became fouled with tar droplets and was subsequently inoperative for this series of runs.

  Figure 17 compares H_2S concentrations for Runs 001 and 003 as measured by detector tube. The levels are very similar except at the beginning of Run 003 in which the H_2S is initially high, presumably because of residual sulfur compounds left in the sorbent bed after regeneration. The on-stream time before breakthrough for Run 003 is about 5 hours less than for Run 001, accounted for by the higher inlet gas H_2S concentrations. The sulfur loadings of the sorbent in Runs 001 and 003, computed from the mass flow and H_2S concentration of the inlet gas, were 14.4 and 17.9 wt percent respectively (see Figures 11 and 13). Because other sulfur compounds including COS were neglected, the actual values would be somewhat greater. Also neglected was sulfur pick-up by the stainless steel delivery line.

  Figure 18 plots H_2S concentration in exit gas against time on stream for Run 005. H_2S levels while desulfurizing air-blown gas are comparable to those in Runs 001 and 003. With oxygen-blown gas, the H_2S level is about 20 ppm corresponding to the greater quantity of steam in the gas. As in Run 003, H_2S is initially high which is attributed to residual sulfur compounds in the sorbent bed. Some sulfur dioxide was also detected in the initial stages of Runs 003 and 005 but fell off more rapidly than the initially high H_2S. This is shown in Figures 19 and 20.

  The H_2S levels were corroborated through analysis of grab samples by laboratory gas chromatograph which also measured COS, CH_3SH, CS_2, and thiophene. These are shown in Table 1 which also includes major gas species. COS, CH_3SH, and CS_2 are much less than H_2S, but significant quantities of thiophene were detected. This is plotted in Figure 21 for Runs 001, 003, and 005 and varies from about 5 to 10 ppm which is not much less than the about 10 ppm typically found in raw coal gas. Table 2 shows the analysis of grab samples of gas taken at the exit of the gasifier primary cyclone for mostly the same species as in Table 1.

- Regeneration

As described above, it was attempted to carry out regeneration Runs 002 and 004 in such a way that the reaction zone temperature was generally between 1,500 and 1,600°F and the SO_2 concentration on termination less than about 0.5 vol. percent. The sample-gas analyses in Table 1 suggest that, during the first half of the regenerations, coke deposited in the sorbent due to cracking of tar was gasified and the gasification products reacted with the products of oxidation of zinc and iron sulfides. Thus, in the early stages, hydrogen exceeded 8 vol. percent and carbon dioxide 16 vol. percent (on a dry gas basis). An alternative explanation may be that magnetite was reduced to wustite, iron, and cementite to some extent by the coal gas which, being produced at a low steam/air ratio of 0.2 wt./wt., had high reducing power. Under these conditions, sulfur in the gas appeared to be mostly in the form of H_2S and elemental sulfur. When most of the coke had been burned from the sorbent and/or iron was oxidized, sulfur dioxide predominated, as shown by the detector tube readings in Figure 22, the concentration attaining about 6 vol. percent in gas exiting the unit.

- Carbon Monoxide Conversion (Shift) Reaction

It was generally found that the carbon monoxide shift reaction proceeded during the sulfidation runs as was expected, since the sorbent under the prevailing conditions was initially predominantly magnetite, a well-known shift reaction catalyst. Thus, Table 1 and Table 2 indicate for Runs 001 and 003 that CO in the early stages was lowered to about 14 vol. percent from about 22 vol. percent. In the latest stages, CO shift was significantly less, perhaps owing to coke deposition, and the CO concentration was about 20 vol. percent. Consumption of CO in reducing the sorbent to iron may also be a factor. Corresponding changes in H_2 and CO_2 concentrations occurred. The results were corroborated by on-line gas chromatograph analysis as shown in Figures 23, 24, and 25 for Run 003.

It would be expected that the sum of the CO and H_2 concentrations at the unit inlet and outlet respectively would be the same. In the early stages of Runs 001 and 003 outlet CO + H_2 was significantly lower than inlet CO + H_2 suggesting that it was being consumed in sorbent reduction.

- Sorbent Characterization

Characterizations of the sorbent, on removal from the reactor, at seven points along the reactor length are shown in Tables 3 and 4. From Table 3, it can be seen that, at the bottom of the sorbent bed, virtually all the zinc and 80 to 90 percent of the iron has been converted to sulfide; whereas at the top of the bed, the zinc is partially converted to sulfide, but no iron sulfide has been formed. It is apparent also from the data at the top of the bed that the original zinc ferrite has been first converted to magnetite and zinc oxide owing to reduction by the coal gas. There is a sharp transition between points where iron sulfide is formed and where it is absent. At the bottom of the bed, zinc sulfide is mostly in the form of beta-ZnS (cubic sphalerite); whereas at the top, there is more alpha-ZnS (hexagonal wurtzite).

Table 4 shows that at the bottom of the bed the sorbent sulfur loading is about 30 wt. percent, not much less than the value of about 35 percent for complete sulfidation. The sorbent can be seen to contain 2 to 4 wt. percent of carbon, there being slightly more at the top of the bed than at the bottom. The higher concentration of 7 wt. percent carbon in the 2.4 kg of fines, which were screened through a No. 8 (2.38 mm) screen from the total sorbent removed from the reactor, demonstrates that coke deposition occurs preferentially at the sorbent pellet surfaces. Although the amount of fines exceeds 10 percent of the sorbent, it is difficult to differentiate between that originally in the reactor and that formed during sorbent removal by suction with a vacuum hose.

The sorbent surface area and pore volume at the bottom of the bed are about half the values at the top, which are about the same as those for fresh sorbent. Thus, after three sulfidations and two regenerations, there is not a marked change in these parameters.

The fully sulfidized sorbent at the bottom of the bed has about twice the crush strength of that at the top of the bed which itself is significantly, though not a lot, less than the crush strength of fresh sorbent.

Since the sorbent prior to removal had been in contact with oxygen-blown gas it should not be inferred that its characterization after Runs 001 and 003 with air-blown gas is the same.

- <u>Particulate Analysis</u>

In the course of the operation of the hot gas desulfurization unit, a sample of particulates knocked out in the secondary cyclone during the first two sulfidations was taken and its particle size distribution determined. Details of the findings are shown in Table 5. Also included is a chemical analysis in terms of carbon, sulfur, and ash. Particle size varied from 4 to 100 microns with a median of about 30 microns. The carbon content of more than 80 wt. percent is typical for fixed-bed gasifiers.

Other particulate samples were taken at the inlet and outlet of the desulfurization unit by means of alundum extraction thimbles (8). A summary of the measurements made of these samples are given in Table 6. The particle loading is of the order of 1 g/Nm^3 being somewhat greater at the exit. Mean particle size is generally higher at the inlet (3 to 15 microns) than at the outlet (~ 3 microns). Chemical analysis indicates that the inlet particles contain about 15 wt. percent carbon suggesting that most of the particulate carbon leaving the gasifier is gasified in the ~ 100-ft line between the secondary cyclone and the desulfurization unit. Analysis of the outlet particles is very similar to that of sorbent fines suggesting that inlet particles are mostly trapped in the reactor and sorbent fines are entrained in the gas leaving the reactor. Particulate size distributions are given in Tables 7. to 13.

- Condensate Analysis

Liquor was collected periodically from the condenser through which the continuous sample of gases at the exit of the gasifier primary cyclone (sampling point S-4) was passed at gasifier pressure. This condensate, as collected, was in two fractions, one mainly water and the other tars. The water fraction was analyzed for inorganic ions of interest as shown in Table 14. An ultimate analysis of the tars was carried out and is shown in Table 15.

As previously described, gases leaving the hot-gas desulfurization reactor pass through condenser CIC immediately before exiting the unit. The liquor which condenses out drains into a collection vessel and is dumped, generally every 4 hours, into steel drums. During sulfidations the liquor separates into water and tar layers. In a few cases, the liquor from the collection vessel before dumping was sampled and the aqueous layer analyzed for the same constituents as the corresponding liquor from S-4 sample point. The analyses are shown in Table 16.

The dumped condensate from CIC was collected in three batches: that during air-blown operation from Runs 001, 003, and 005; that during oxygen-blown operation from Run 005; and that during regeneration from Runs 002 and 004. The first two batches were separated into water and tar layers, weighed, and sampled. The third batch, which consisted only of an aqueous phase, was also weighed and sampled.

The aqueous samples were analyzed for inorganic ions as above and are shown in Table 17 and 18, respectively. The first sample was also analyzed for comparison by IHI-KEMRON, who measured for the additional ions, sulfide, sulfite, cyanide, thiocyanate, antimony, and lead, but did not include nickel, cromium, aluminum, selenium, mercury, nitrate, phosphate, and bromide. This analysis is also shown in Table 17. Included in Table 18 is a corresponding analysis of the steam used for regeneration. Ultimate analyses of the two tar samples are shown in Table 19.

Perusal of Tables 14, 16, 17, and 18 permits some tentative deductions to be made concerning the absorption of certain elements and ions by the sorbent during sulfidation and their subsequent release during regeneration, bearing in mind that the amount of aqueous condensate collected from sulfidation Runs 001 and 003 was about 122 lbs and that from regeneration Runs 002 and 004 about 250 lbs (see Figures 11 to 14).

The halides F^-, Cl^-, and Br^- appear to be removed by as much as 70 percent from the air-blown gas, virtually all of this being extracted from the sorbent during regeneration. A similar proportion of the alkali metals Na and K also appears to be removed during sulfidation and is subsequently released into the regeneration condensate.

Additionally, the regeneration condensate, as analyzed, was enriched in vanadium, calcium, aluminum, mercury, and ammonium ion indicating that these may also have been removed from the coal-gas stream to some extent.

Zinc ion measured in both sulfidation and regeneration condensates was encouragingly low at less than about 2 ppm.

Iron ion in the sulfidation condensate was quite low at about 20 ppm, indicating relatively little carry-over from the sorbent. The high value in the regeneration condensate in Table 11 does not agree with that in Table 16, suggesting possible pickup from the steel collection drum.

There is some evidence that selenium and sulfate ion were removed from the coal gas to a large extent but virtually no arsenic. The fairly large amount of sulfate ion in the regeneration condensate was, of course, expected. A small quantity of inorganic ash was contained in the tar condensates (see Table 19) translating to about 10 ppm in the gas.

- <u>Sulfur in Condensate</u>

In addition to the sulfur compounds, H_2S, etc., measured by gas chromatograph in the exit gas from the hot-gas desulfurization unit, sulfur compounds were found in the exit gas condensate, both in the water and tar components. Table 17 indicates that the water condensate contained as much as 350 ppm of sulfur translating to nearly 20 ppm in the gas. From Table 19 it can be seen that the tar condensate contained about 2 wt percent of sulfur translating to about 100 ppm in the gas. Thus, taking this sulfur into account, the hot gas leaving the hot-gas desulfurization reactor contained about 100 ppm above that measured by gas chromatograph. Most of this appears to be organically bound sulfur. It is of interest to note that the sulfur concentration in the tar condensate at the unit exit does not appear to be less than at the inlet (see Table 15) contrary to expectations if the sorbent possessed hydrodesulfurization activity.

- <u>Composition of Reactor Exit Gas</u>

In Tables 20 and 21 the components found in the aqueous condensate, Tables 16 and 17, are expressed as a concentration of the reactor exit gas. Components from the tar condensate are not included. The chloride level of the coal gas considerably exceeds the expected tolerable level of about 1 ppm for molten carbonate fuel cells; the arsenic level may be acceptable. Little is known about the effect of the other components, except for sulfur compounds.

The levels of sodium, potassium, and vanadium may be acceptable should the coal gas be used as gas turbine fuel.

Conclusions

The first series of tests to evaluate the performance of zinc ferrite, hot-gas desulfurization sorbent in a side-stream of METC's 24 t/d, fixed-bed coal gasifier was carried out successfully with only minor difficulties. The concentration of sulfur compounds in the exit gas from the hot-gas desulfurization unit was generally less than 10 ppm. However, this disregards sulfur compounds condensing out in the tar and aqueous condensates which, expressed as equivalent gas concentration, amounts to about 100 ppm.

Detailed data were obtained characterizing the sorbent performance at various stages in the adsorption/regeneration cycles. Generally, the sorbent behaved as anticipated, based on the previous laboratory-scale investigation. The main difference was due to the presence of tars in the coal gas which appeared to crack in the sorbent, forming coke. An additional complication may have been reduction of magnetite iron to lower valence states. The apparent gradual buildup of particulates and coke caused the pressure drop across the reactor of the hot-gas desulfurization unit to increase but it was of manageable proportions and mostly decreased during regeneration. The coke appeared to act as a reducing agent partially reducing sulfur oxides liberated during regeneration to hydrogen sulfide, sulfur, etc., and thus could be of advantage in a scheme for sulfur recovery. However, part of this effect may have been the result of reduction of magnetite to iron and the consequent formation of hydrogen by the steam-iron reaction.

The tests established that desulfurization performance of the sorbent was satisfactory in the temperature range 1,000 to 1,100°F, at pressures up to 150 psig, and at a space velocity of 2,000 h^{-1}.

On removal from the reactor, the sorbent contained about 10 percent of fines but most of this may be the result of breakage in the removal process. The strength of the sorbent extrusions appeared to be unimpaired.

The sorbent was active in promoting the carbon monoxide "shift" conversion reaction to hydrogen. It also absorbed alkali metals and halides to a large extent, subsequently releasing them during regeneration. There was some evidence that vanadium, calcium, and mercury were similarly removed to some extent.

Particulate loading at the reactor exit was measured at about 1 g/Nm^3, the particles typically having a mean size of about 3 µm.

References

1. Grindley, T., "Development and Testing of Regenerable Hot Coal Gas Desulfurization Sorbents for Molten Carbonate Fuel Cell Application," METC, IR No. 1133 (1981).

2. Grindley, T., "Report on Testing of Institut Francais DuPetrole (IFP) Desulfurization Sorbent MEP-682," METC, IR No. 1328 (1982).

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8. Pater, K., Preliminary Summary for Run No. 101. IR 1644 August 1983, METC.

9. Romanosky, R. R., J. J. Kovach, Analytical Data Producer Run No. 101. METC, IR No. 1861 (1984).

10. Bagnell, R. D., C. K. McCurry, and E. E. Childers, "Process Stream Characterization for the Hot Gas Desulfurization Unit During Gasification Run 101." METC, IR No. 1764 (1984).

1:910318ds.1jj

The Baseline G.C. on-line H_2S, COS, and SO_2 readings as plotted are correct only for the points: H_2S: 3 ppm; COS: 1.7 ppm; and SO_2: 5.1 ppm. Calibration curves are available for correction. Corrected plots are given in Volume 1.

Refer to letter to T. Grindley from E. E. Gorski and P. Johnson, EG&G Washington Analytical Services Center, Inc. January 30, 1986. Subject: DOE-METC/EG&G Contract No. DE-AC21-85MC21353; WBS No. 9KEX-10. HGD Project: Baseline GC/Integrator Conversion Curves.

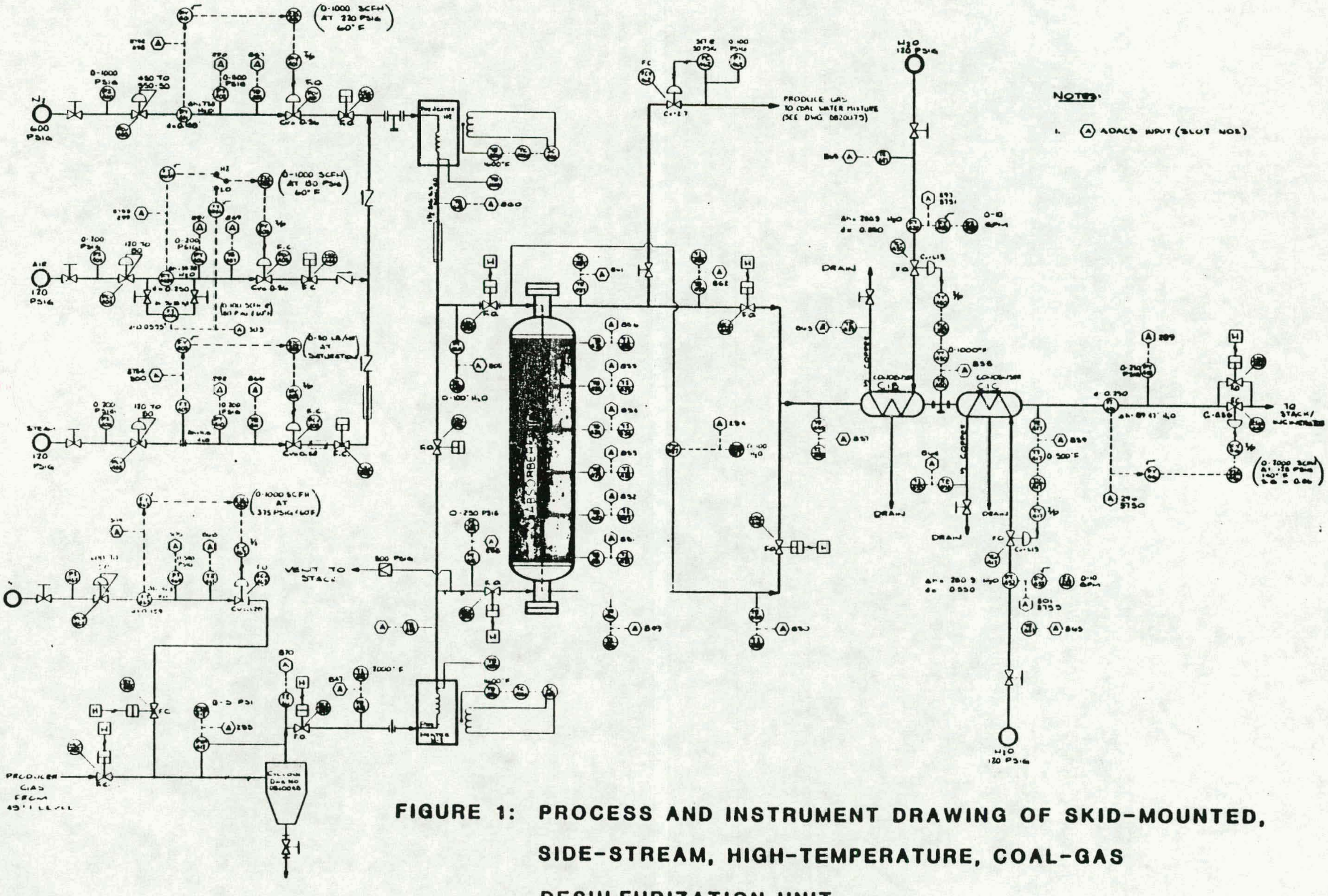


FIGURE 1: PROCESS AND INSTRUMENT DRAWING OF SKID-MOUNTED, SIDE-STREAM, HIGH-TEMPERATURE, COAL-GAS DESULFURIZATION UNIT

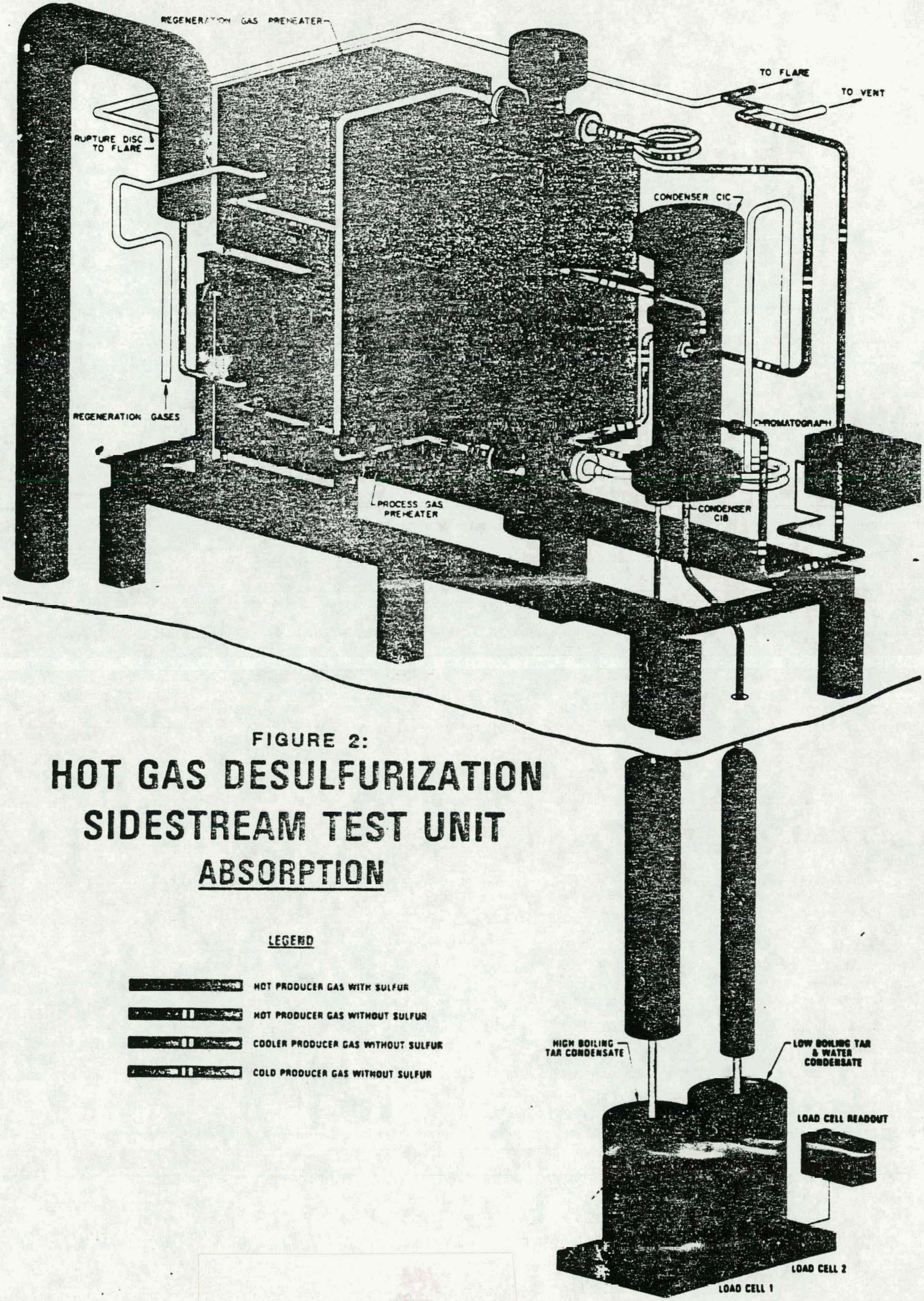


FIGURE 2:
HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT
ABSORPTION

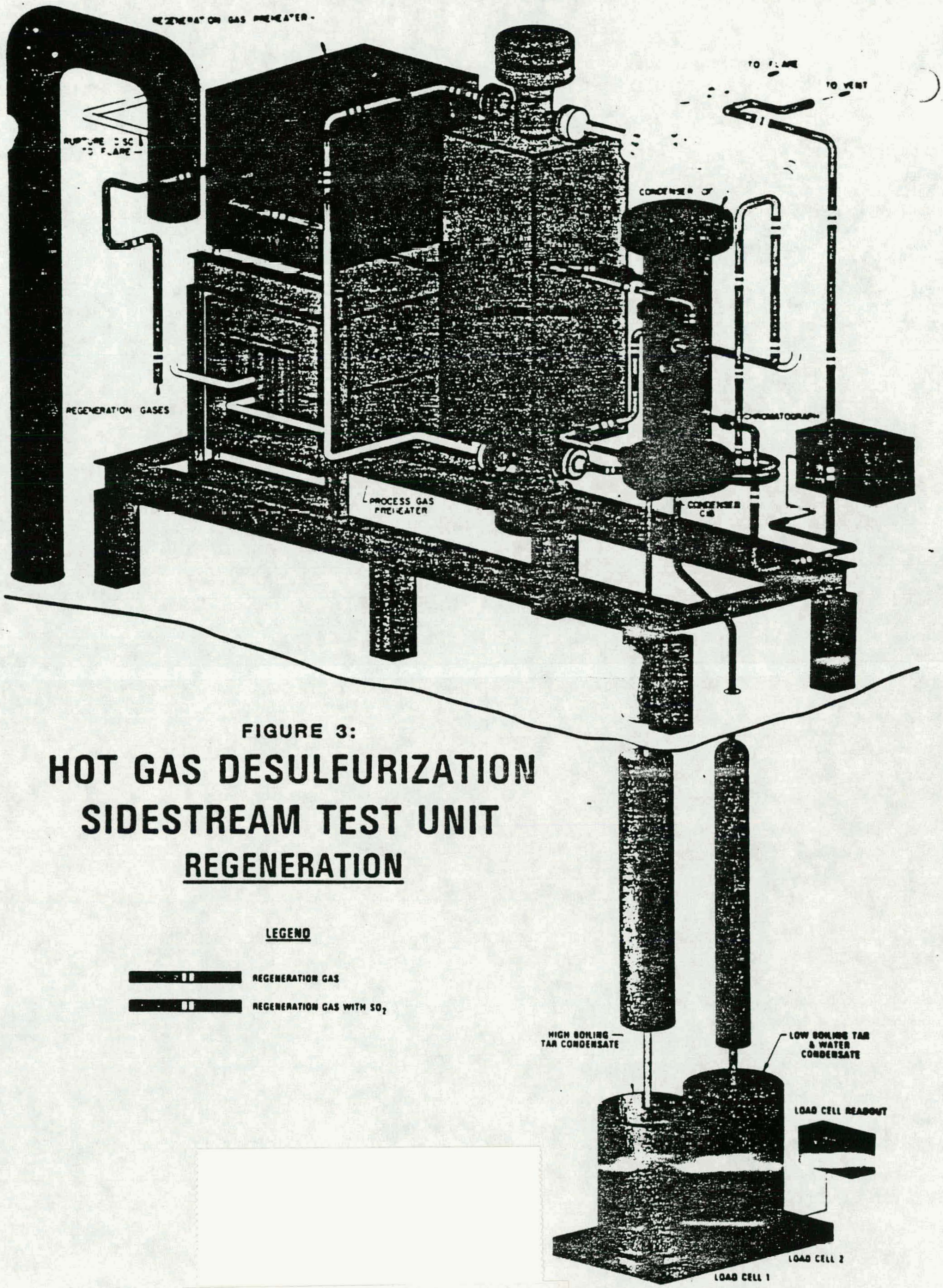


FIGURE 3:
HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT
REGENERATION

LEGEND

REGENERATION GAS

REGENERATION GAS WITH SO_2

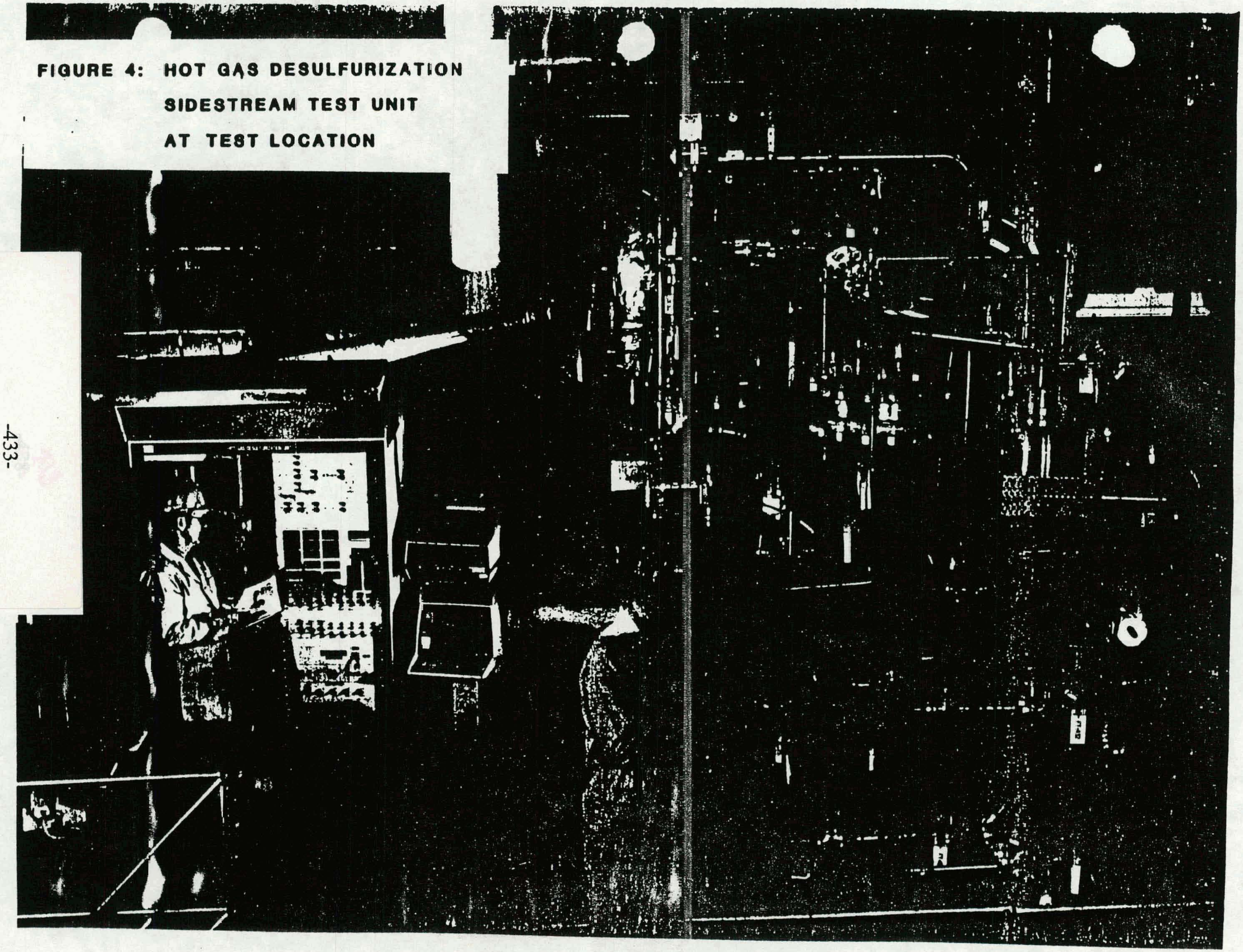
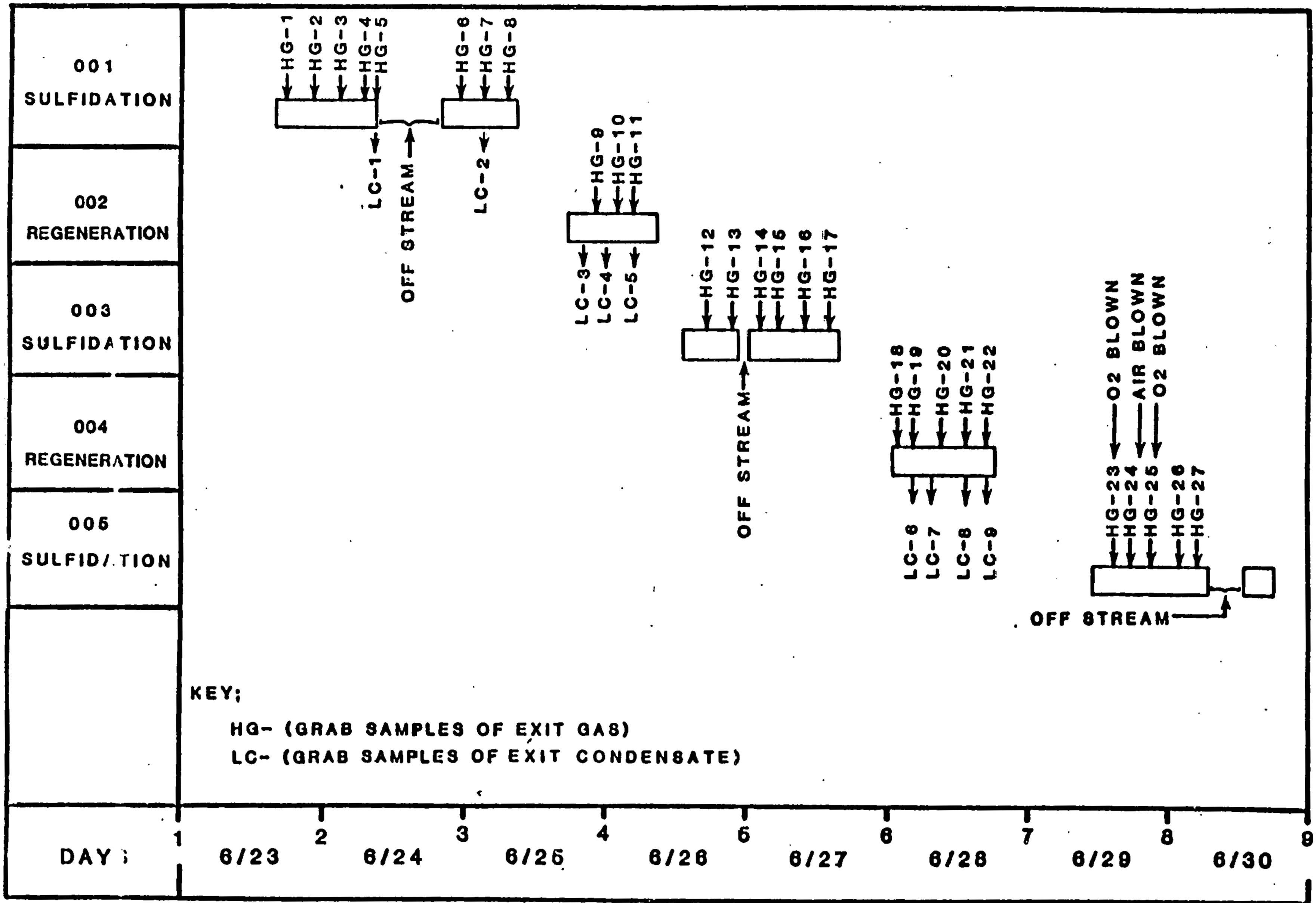


FIGURE 4: HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT AT TEST LOCATION

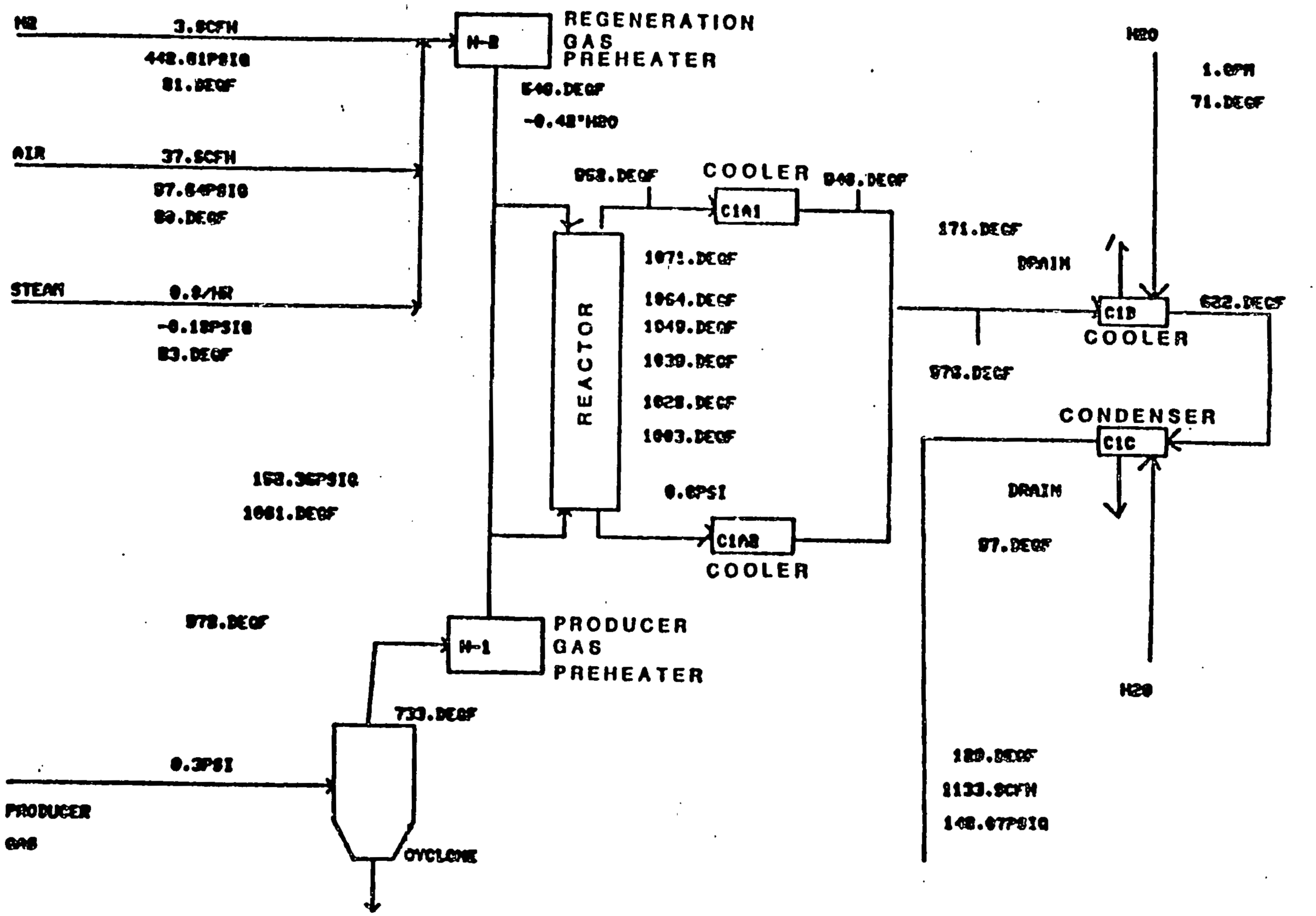


SCHEDULE OF EVENTS

FIGURE 5: HOT GAS DESULFURIZATION SIDESTREAM TESTING 1983

RUN 001A

AVERAGE OPERATING PARAMETERS



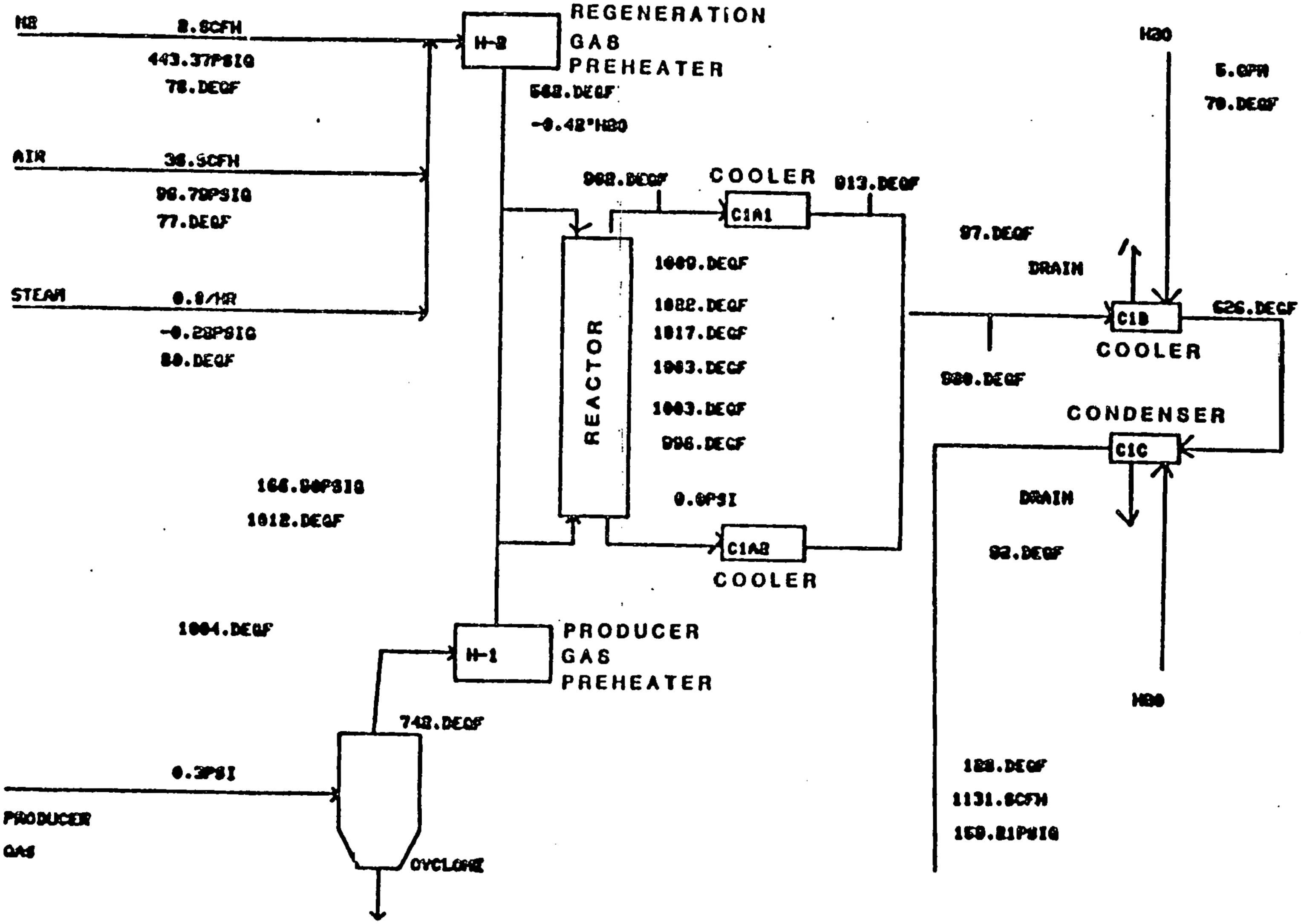
AVERAGE(S) FOR PERIOD FROM: 16:15:17 TO 7:48: 4

FIGURE 6A

-436-

RU.. J01B

AVERAGE OPERATING PARAMETERS



AVERAGE(S) FOR PERIOD FROM: 10:30:27 6/24/1983 TO 8:34:20 6/25/1983

FIGURE 6B

RUN J3

AVERAGE OPERATING PARAMETERS

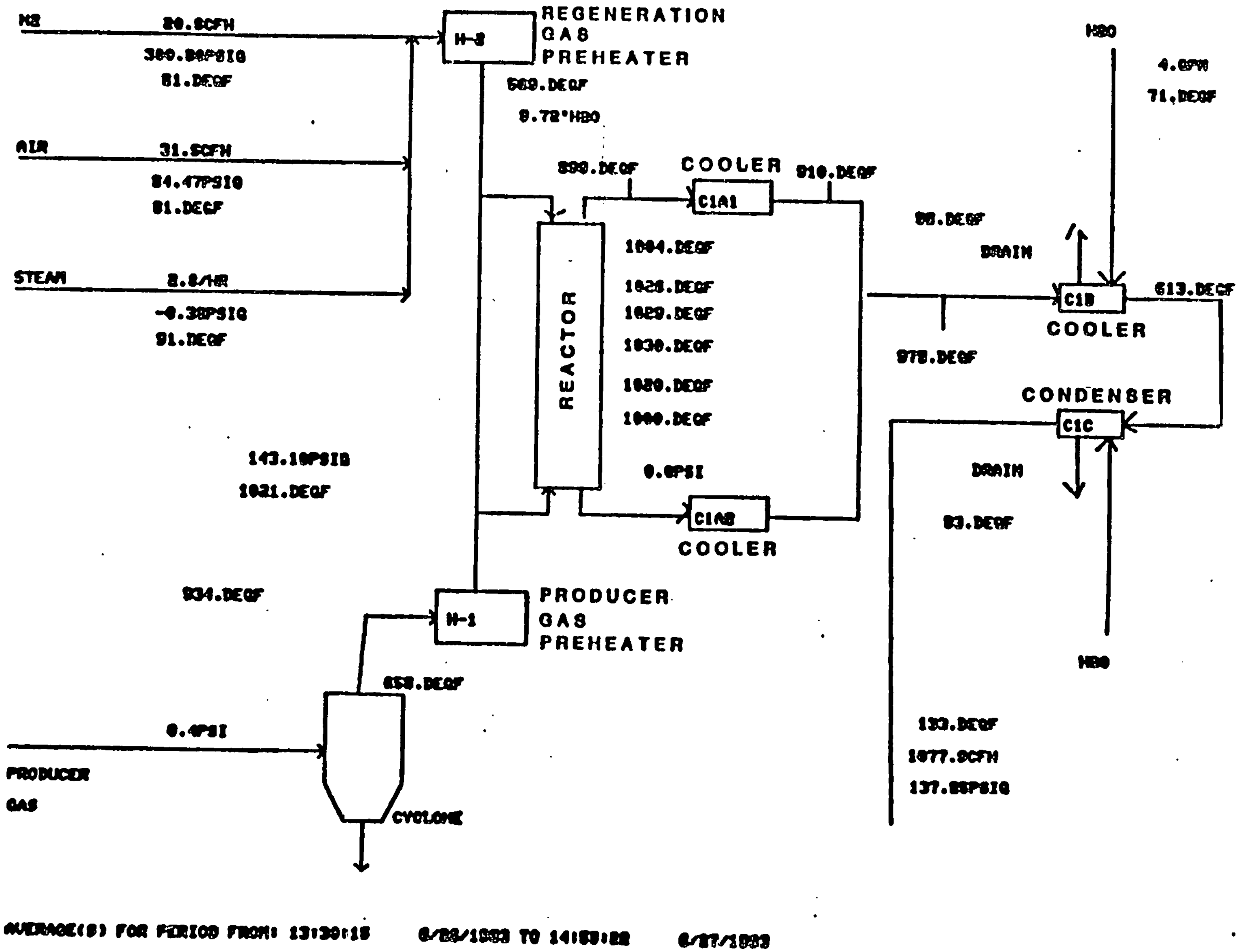
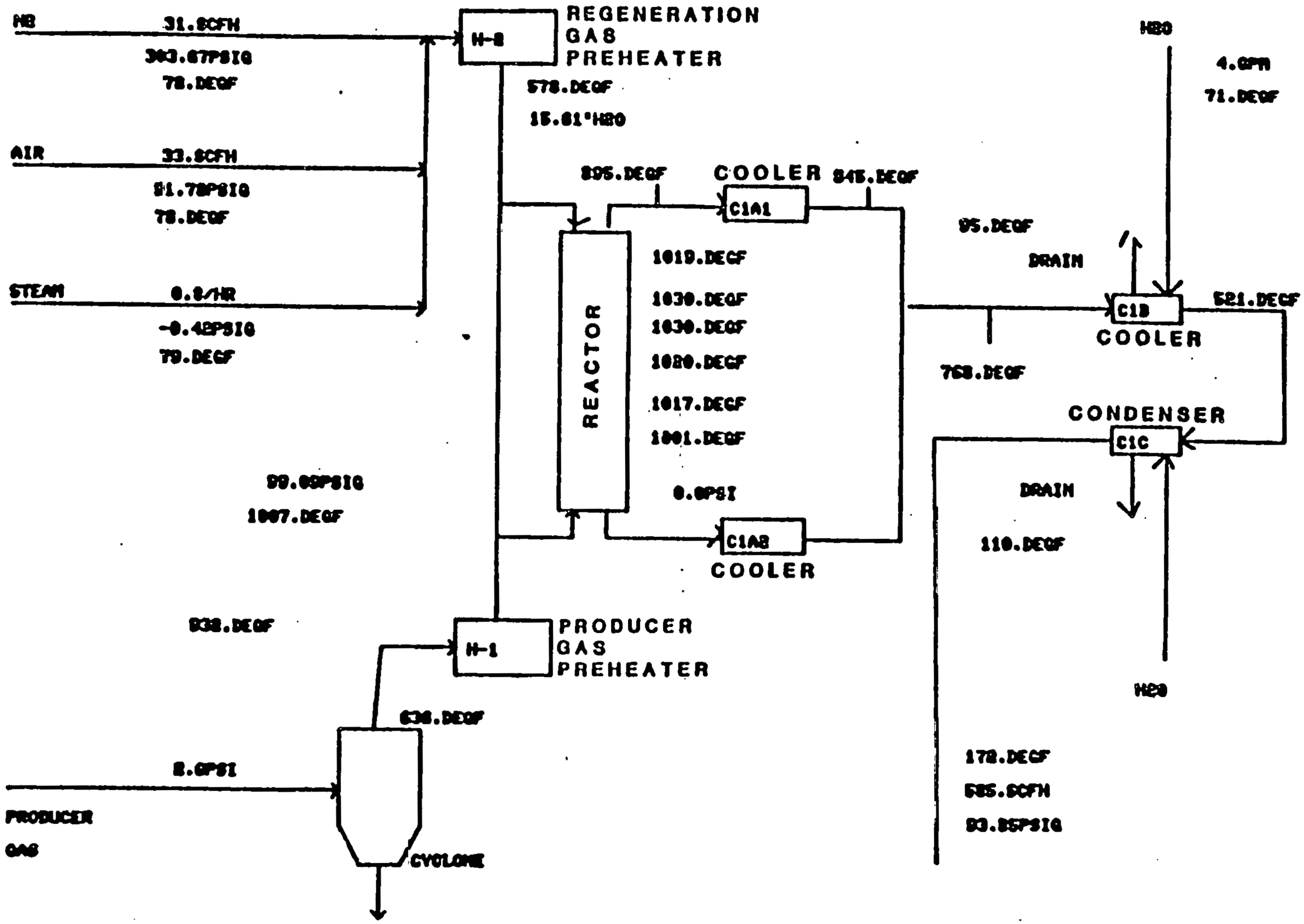


FIGURE 7

RUN 005A

AVERAGE OPERATING PARAMETERS

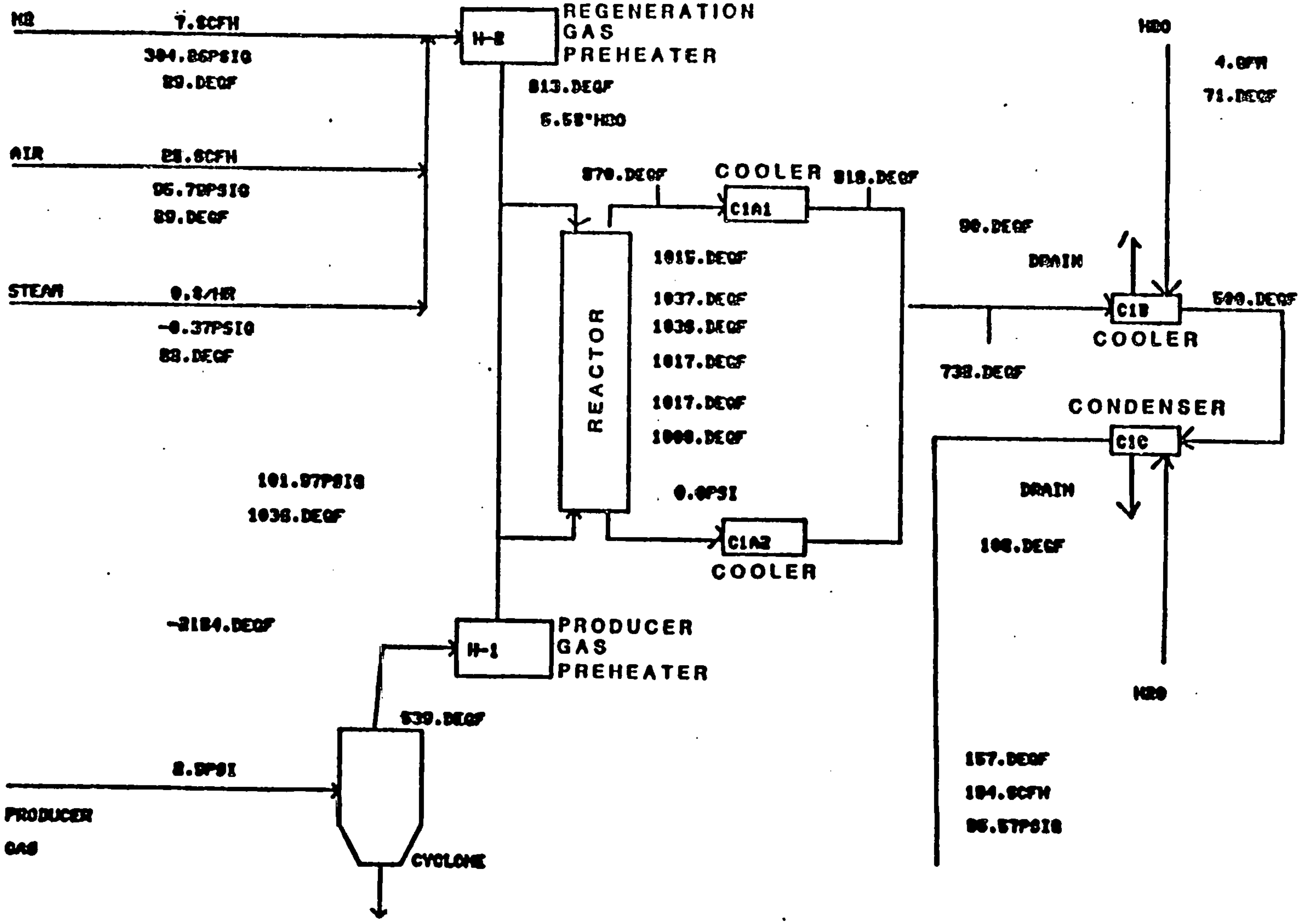


AVERAGE(S) FOR PERIOD FROM: 11: 0:18 6/28/1983 TO 8:24:11 6/30/1983

FIGURE 8A

RUI 05B

AVERAGE OPERATING PARAMETERS



AVERAGE(S) FOR PERIOD FROM: 13:45:18 6/30/1983 TO 17:29:11 6/30/1983

FIGURE 8B

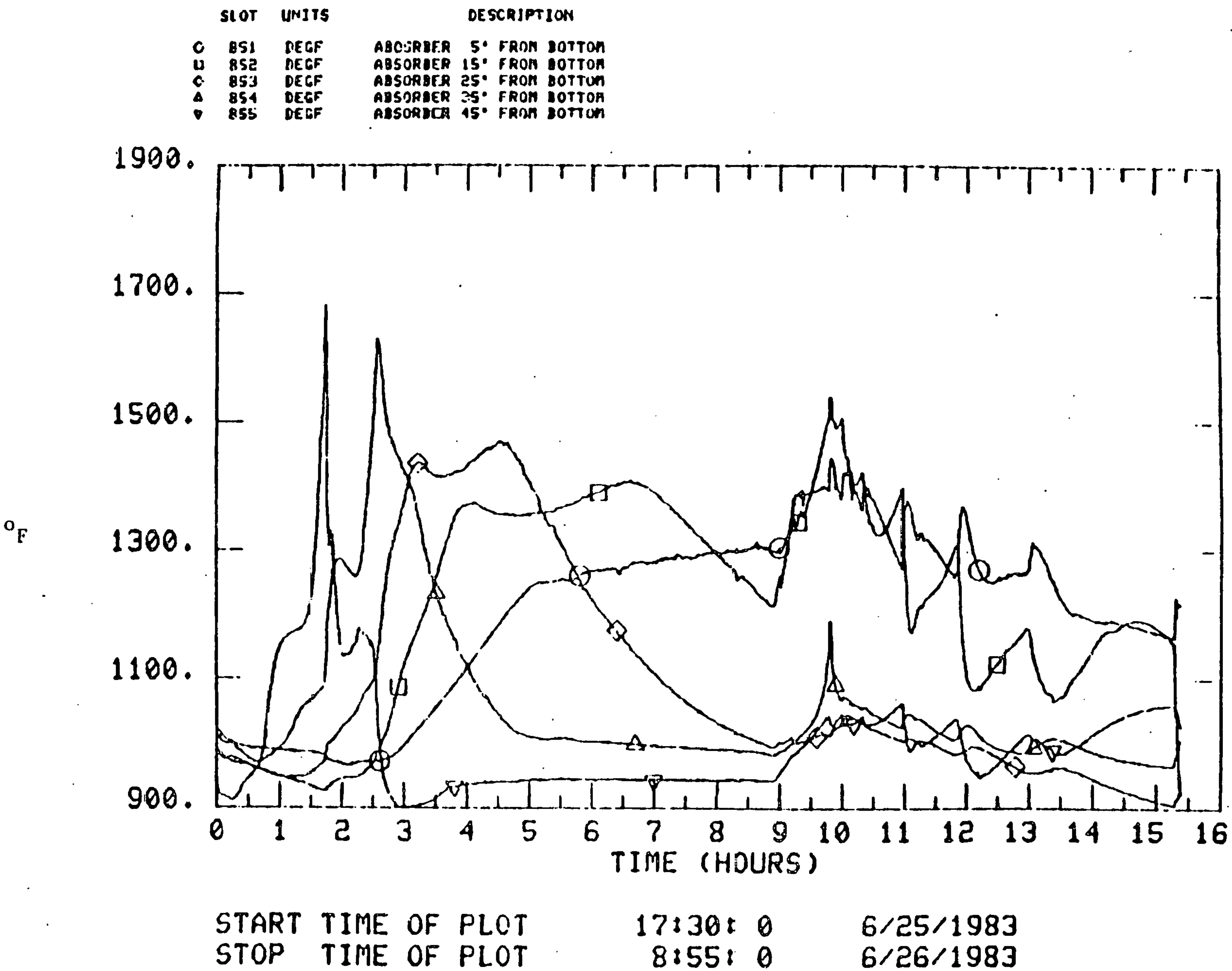


Figure 9 Run 002 Regeneration Reactor Temperatures

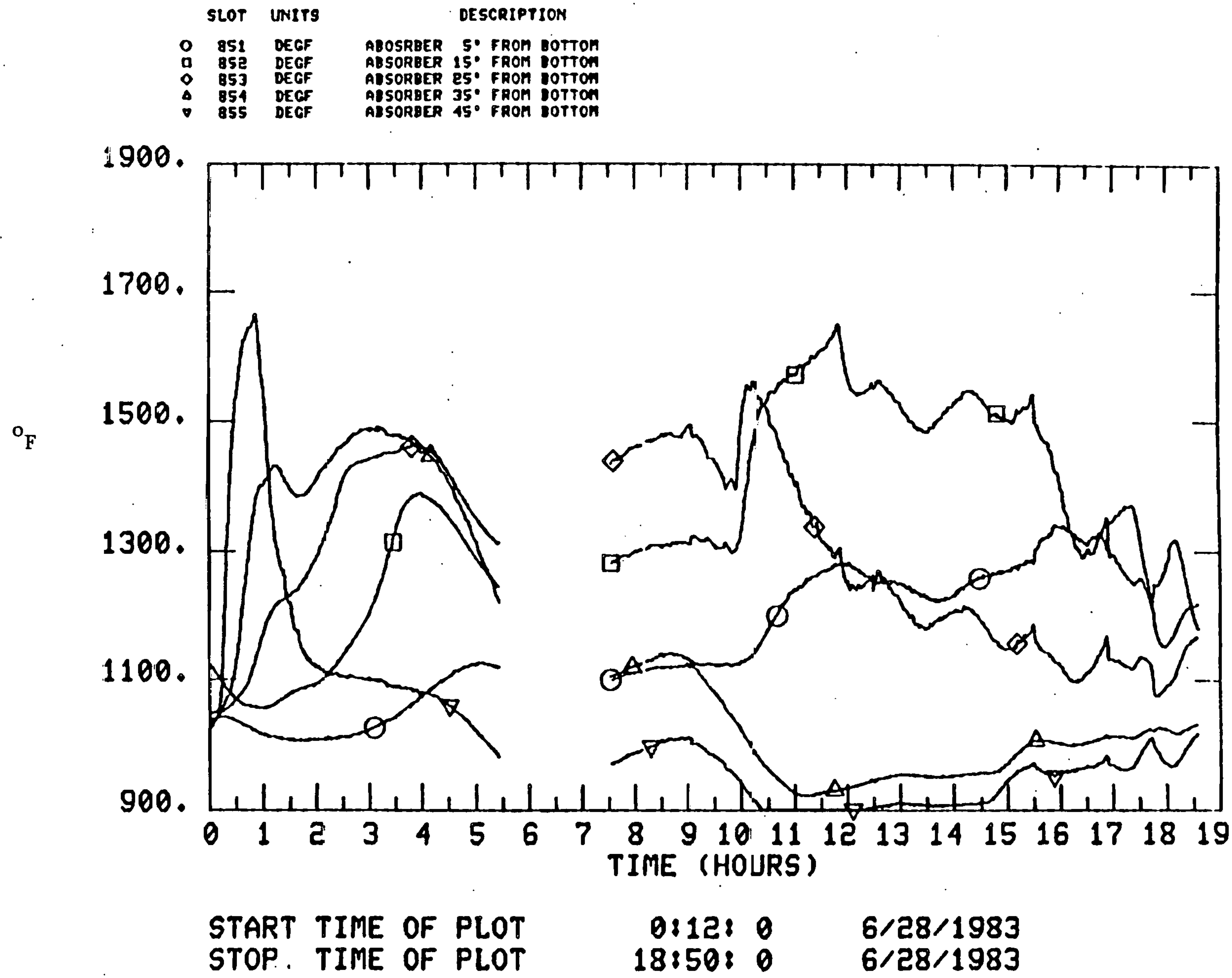


Figure 10 Run 004 Regeneration Reactor Temperatures

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 001
DATE STARTED : 16:15 6/23/83
DATE ENDED : 08:35 6/25/83

TOTAL HOURS : 29.68
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: 0.526
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	2.58			
N2	47.34			
O2	0.63			
C2H6	0.83			
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: (%WT)	0.13	
SURFACE AREA: (SQ M/G)	3.13	
DENSITY : (G/CC)	6.16	
PORE VOLUME: (CC/G)	0.004316	
ELEMENTAL ANALYSIS:		
TOTAL CARBON: (%WT)	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-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 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.
6. GAS COMPOSITION AT INLET IS AVERAGE OF DATA OBTAINED BY BENDIX GC 7000 84 SAMPLING AT S4 GASIFIER CYCLONE EXIT.

CONCLUSIONS:

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

Figure 11

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 008
DATE AND TIME STARTED : 17:30 6/25/83
DATE AND TIME ENDED : 08:55 6/28/83
TOTAL HOURS : 14.4
TYPE : REGENERATION
SULFUR REMOVED:

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

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-10789 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 OVERTEMPERATURING THE SORBENT.

Figure 12

S

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 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: 0.480
STEAM/AIR RATIO : 0.19

HGD OPERATING CONDITIONS

TEMPERATURE: 1018 F (AVERAGE)

PRESSURE : 143 PSIG (AVERAGE)

SPACE VELOCITY: 1925 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	0.36		1.6 PPM	1.5 PPM
H2O				4.40
CO2	6.93		10.35	9.91
CO	22.06		17.61	16.85
H2	14.84		16.30	15.60
CH4	2.68		2.62	2.50
N2	47.03		49.83	47.70
O2	0.46		0.61	0.58
C2H6	0.25		0.03	0.03
TOTAL FLOW:	28,473 SCF		26,473 SCF	27,553 SCF
TOTAL LB MOLES:	69.84		69.84	72.69
FLOW RATE:	1077 SCFH		1077 SCFH	1181 SCFH

PURPOSE

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

SORBENT ANALYSIS:

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

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-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 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.
5. GAS COMPOSITION AT INLET IS AVERAGE OF DATA OBTAINED BY BENDIX GC 7000 #4 SAMPLING AT S4 GASIFIER CYCLONE EXIT.
6. GAS COMPOSITION AT EXIT (DRY BASIS) IS AVERAGE OF DATA OBTAINED BY BENDIX GC 6000 #2 SAMPLING A HGD EXIT.
7. GAS COMPOSITION AT EXIT (WET BASIS) IS CALCULATED FROM DRY BASIS DATA AND WATER OBTAINED AS IN ITEM 4 .

CONCLUSIONS:
1. SORBENT CAN DESULFURIZE GASIFIER GAS CONTAINING TARS AND OILS DOWN TO LESS THATN 10 PPM TOTAL SULFUR.
2. PERFORMANCE IS REASONALBLY GOOD AFTER A REGENERATION. BREAKTHROUGH TIME DECREASED FROM 29 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.

Figure 13

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 004
DATE AND TIME STARTED : 00:18 8/28/83
DATE AND TIME ENDED : 18:50 8/28/83
TOTAL HOURS : 18.8
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	82	
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: (%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 : 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 1/8 S SR-10789 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.559 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 1687 F.

CONCLUSIONS

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

Figure 14

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 005
DATE STARTED : 11:00 6/29/83
DATE ENDED : 17:30 6/30/83
TOTAL HOURS : 83.16
TYPE : SULFIDATION
SULFUR REMOVED: 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: 0.500 ON AIR BLOWN 2.344 ON OXYGEN BLOWN
STEAM/AIR RATIO : 0.193
STEAM/OXYGEN RATIO: 3.366
HGD OPERATING CONDITIONS
TEMPERATURE: 1012 F (AVERAGE)
PRESSURE : 91 PSIG (AVERAGE)
SPACE VELOCITY: 1046 HOURLY FIRST PORTION
347 HOURLY SECOND PORTION

	INLET AIR BLOWN MOLE% (OIL FREE)		INLET O2 BLOWN MOLE% (OIL FREE)		EXIT AIR BLOWN MOLE% (OIL FREE)		EXIT O2 BLOWN MOLE% (OIL FREE)	
	DRY	WET	DRY	WET	DRY	WET	DRY	WET
H_2S	0.38		0.66		3 PPM	2.85 PPM		
H_2O						4.28		53
CO_2	7.16		18.88		12.59	12.13		
CO	24.81		24.92		16.33	15.73		
H_2	15.06		29.50		17.86	17.21		
CH_4	3.14		6.36		2.84	2.73		
N_2	51.12		1.01		47.20	45.49		
O_2	0.54		0.01		0.50	0.48		
C_2H_6	0.40		0.62		0.04	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	949	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)		4.55 (AVG)
PORE VOLUME: (CC/G)		0.0039 (AVG)
ELEMENTAL ANALYSIS:		
TOTAL CARBON: (%WT)		2.63 (AVG)

EXIT SULFUR DATA:
H_2S : 3 PPM ON AIR BLOWN GAS
C_4H_4S: 4 PPM ON AIR BLOWN GAS

CONDENSATE		AIR BLOWN (7.15 HRS)	O2 BLOWN (16 HRS)
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-10789 UNITED CATALYSTS.
2. OVERALL PACKED LENGTH IS 58 7/8 INCHES.
3. THIRD SULFIDATION ON THE SAME SORBENT.
4. PORTIONS OF THE RUN WERE AIR BLOWN, PORTIONS O2 BLOWN.
5. WATER IN EXIT GAS DETERMINED BY CONDENSATE COLLECTED AND EXIT GAS SATURATION LEVEL BY STEAM TABLES.
6. AIR BLOWN GAS COMPOSITION AT INLET IS AVERAGE OF DATA OBTAINED BY BENDIX GC 6000 #1 SAMPLING AT HGD INLET.
7. OXYGEN BLOWN GAS COMPOSITION AT INLET IS AVERAGE OF DATA OBTAINED BY BENDIX GC 6000 #1 SAMPLING AT HGD INLET.
8. AIR BLOWN DRY GAS COMPOSITION AT EXIT IS AVERAGE OF DATA OBTAINED BY BENDIX GC 6000 #2 SAMPLING AT HGD EXIT.
9. AIR BLOWN WET GAS COMPOSITION AT EXIT IS CALCULATED FROM DRY GAS COMPOSITION AND WATER AS DETERMINED IN ITEM 5.

CONCLUSIONS:
1. SORBENT CAN DESULFURIZE GASIFIER GAS CONTAINING TARS AND OILS DOWN TO LESS THAN 10 PPM TOTAL SULFUR.
2. PERFORMANCE ON AIR BLOWN GAS IS REASONABLY GOOD AFTER TWO REGENERATIONS, BUT POOR ON OXYGEN BLUON 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 PPMAFTER FORTY MINUTES.

Figure 15

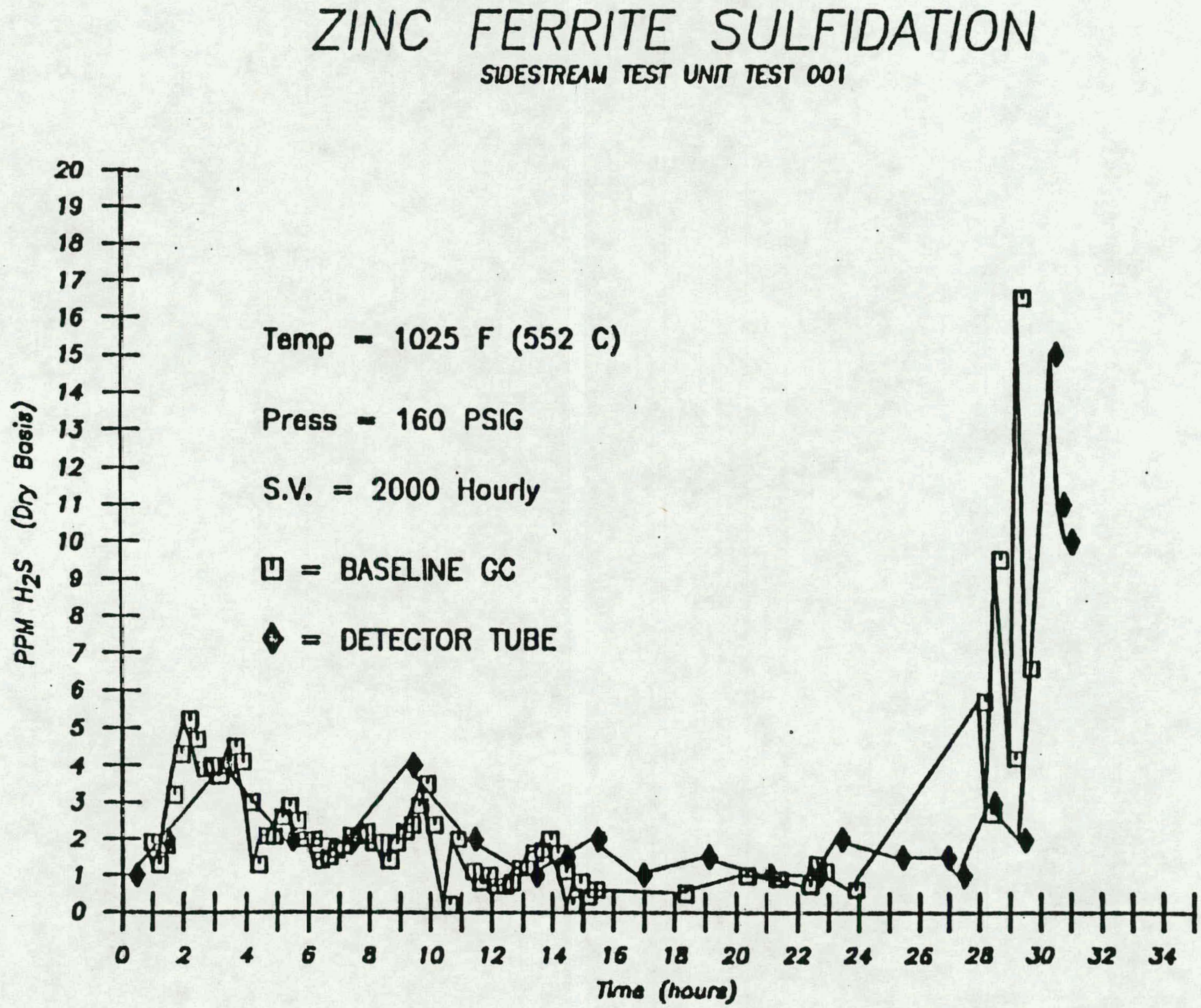
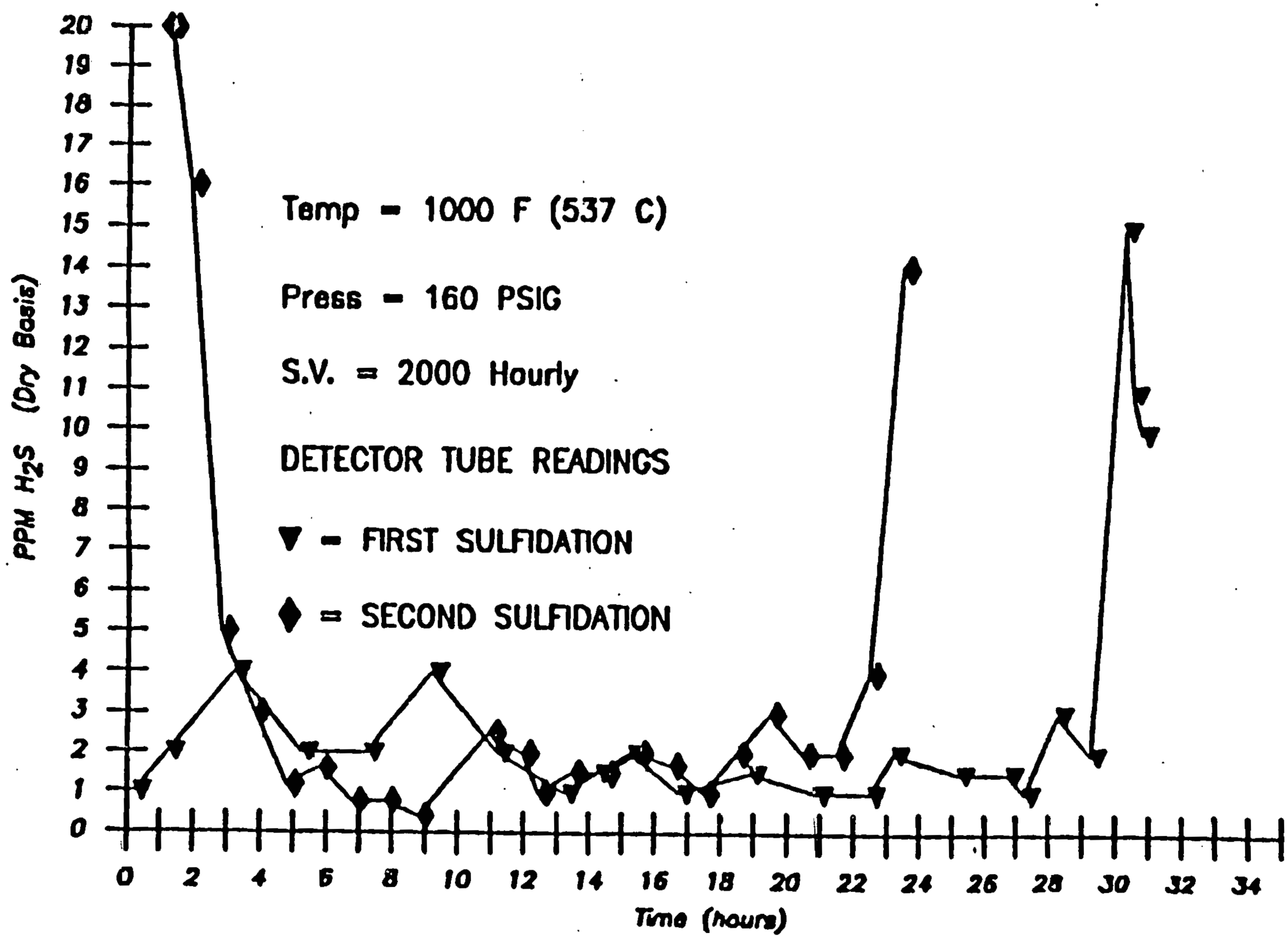


Figure 16

-447-

ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TESTS 001 AND 003



ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TEST 005

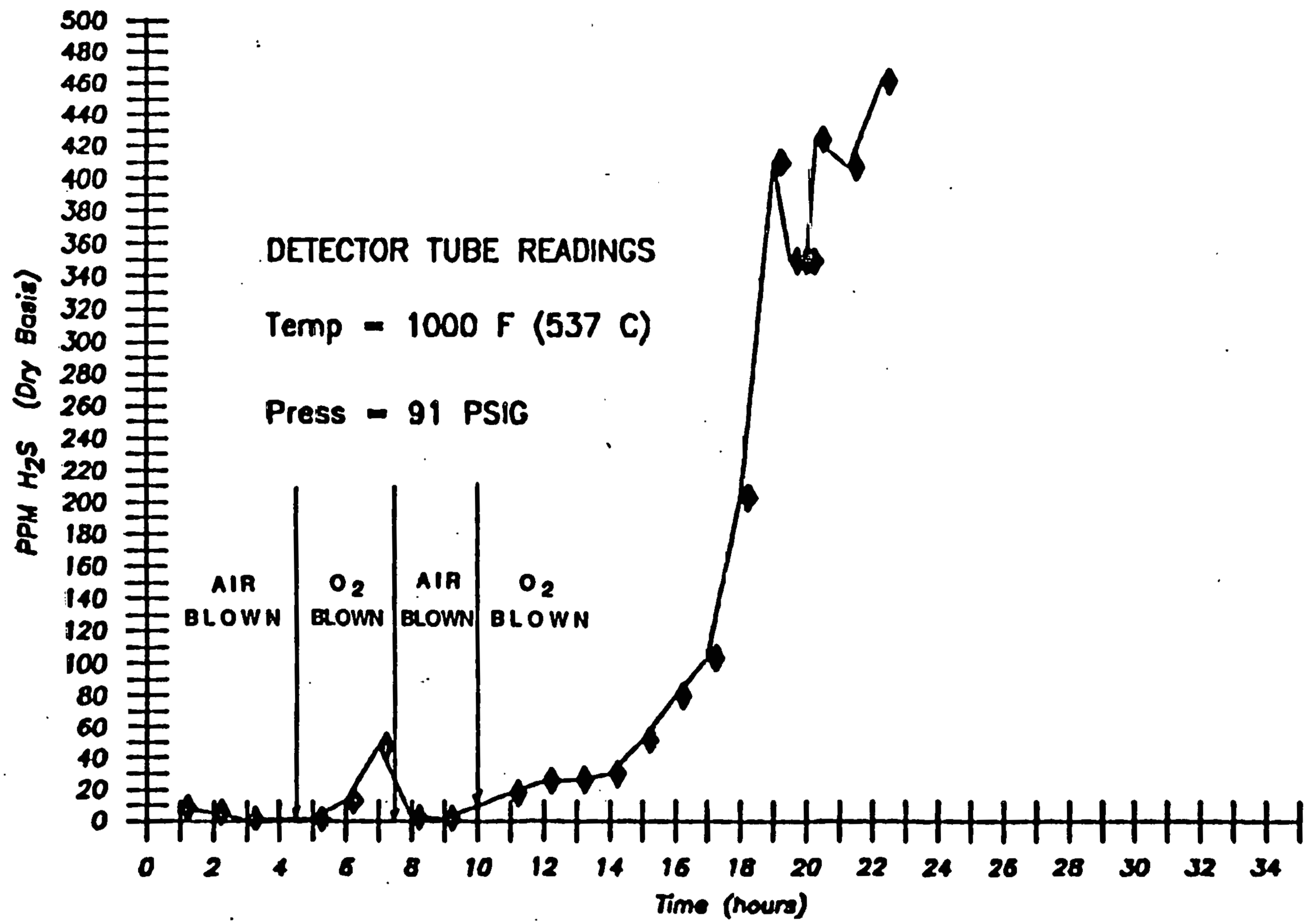
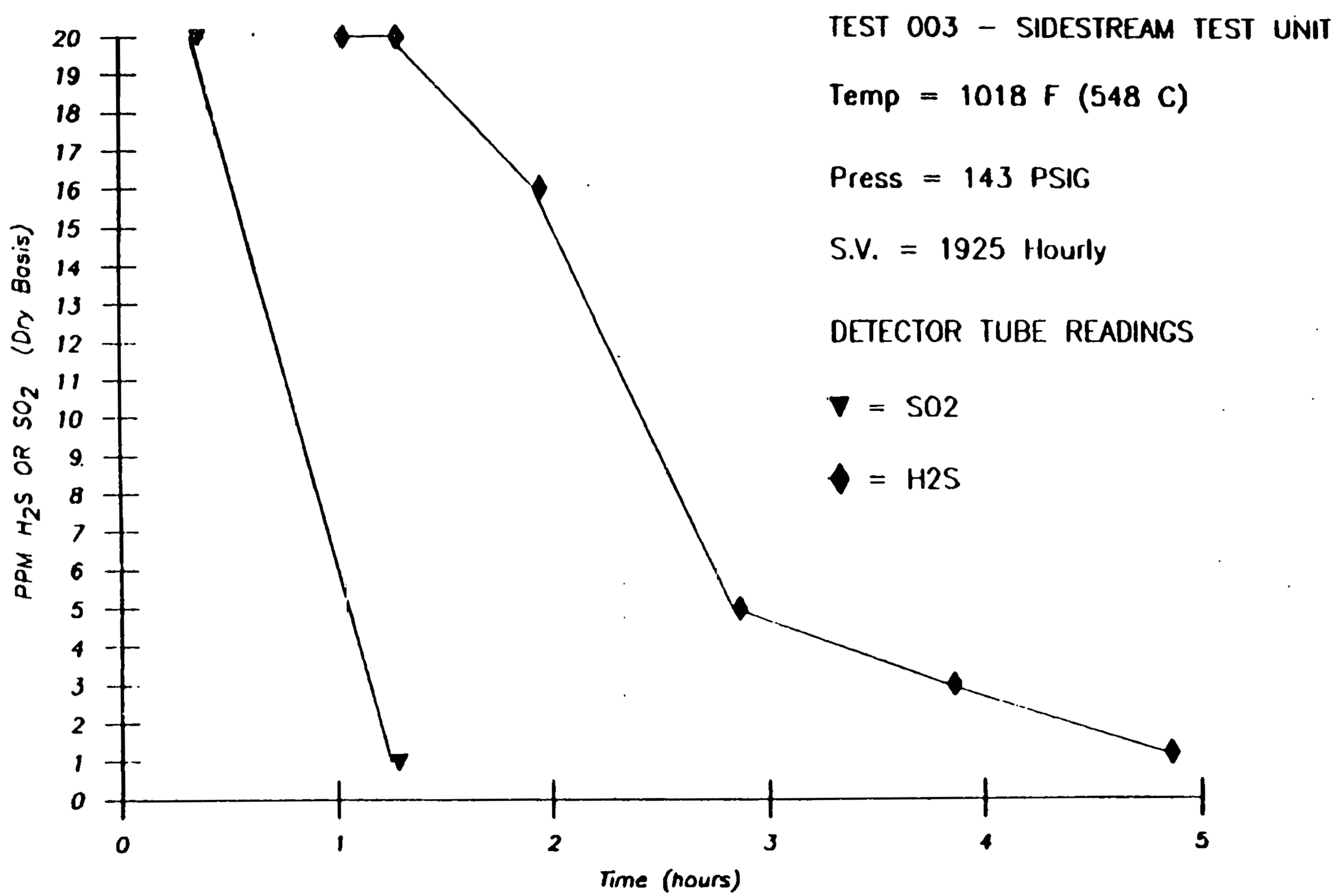


Figure 18

P1

ZINC FERRITE SULFIDATION

INITIAL H2S AND SO2 CONCENTRATIONS



19-JUN-84 10:55:07

Figure 19

ZINC FERRITE SULFIDATION

INITIAL H2S AND SO2 CONCENTRATION

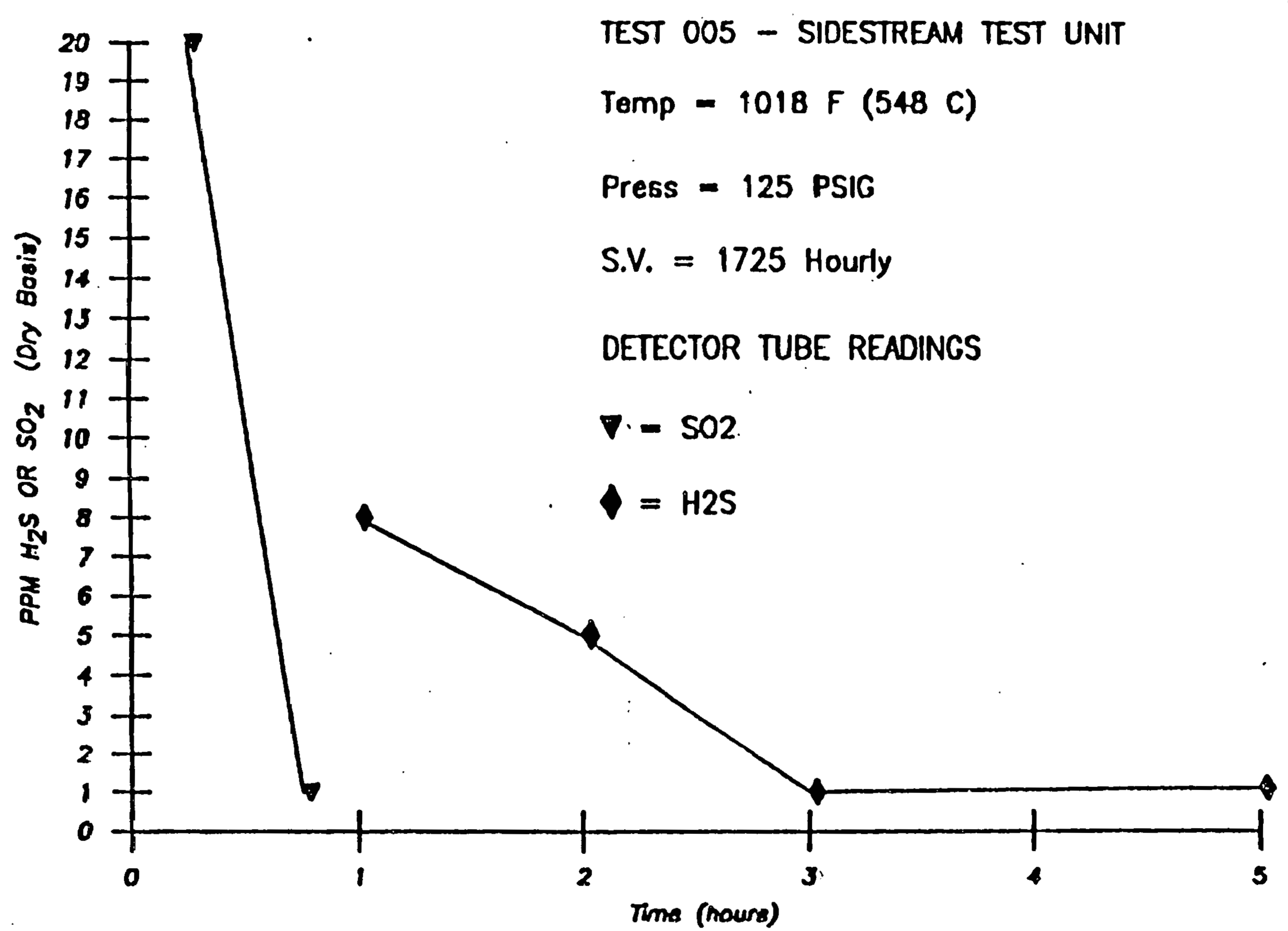


Figure 20

ZINC FERRITE SULFIDATION

THIOPHENE CONCENTRATIONS

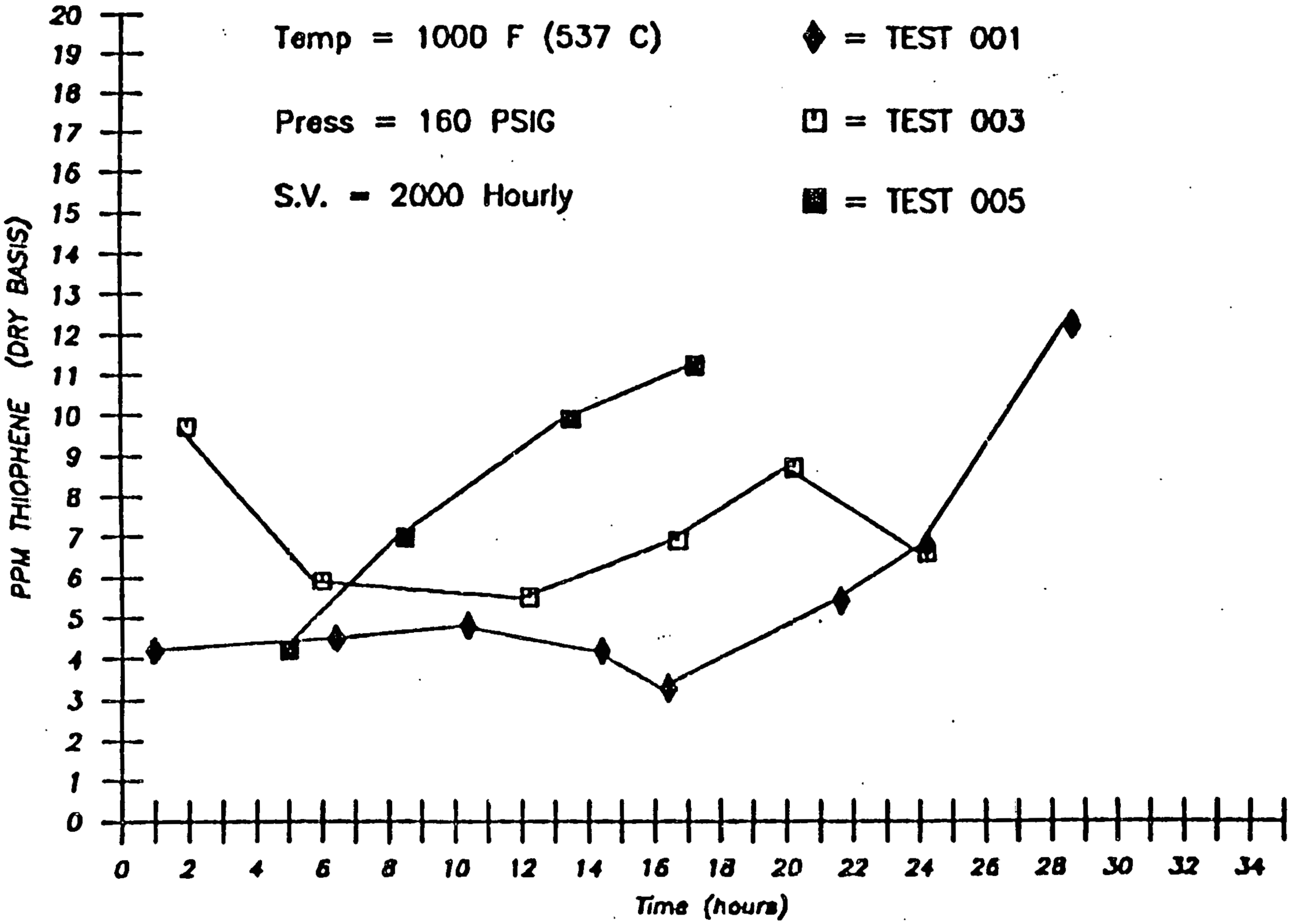
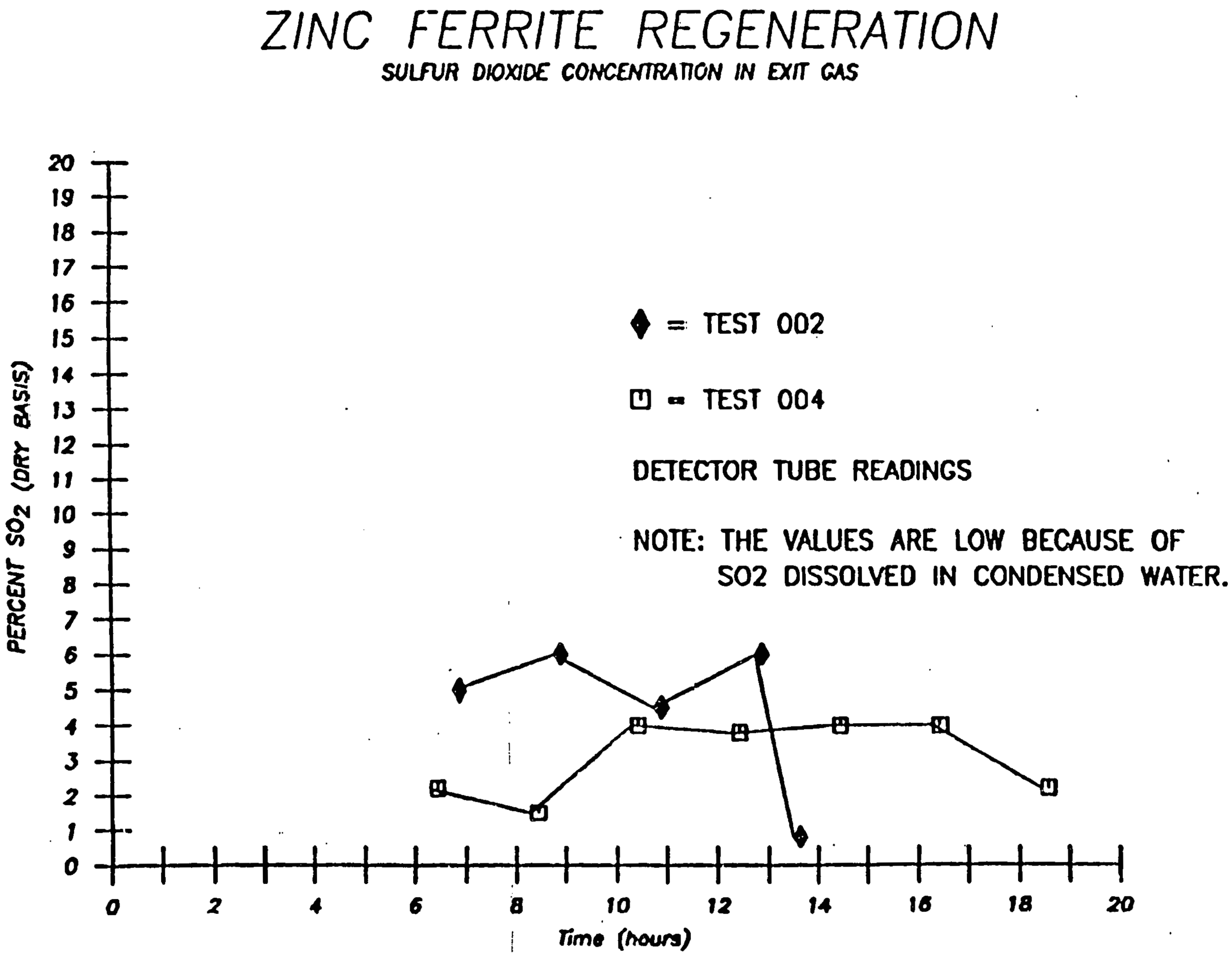


Figure 21



30-NOV-83 13:43:38

-453-

Figure 22

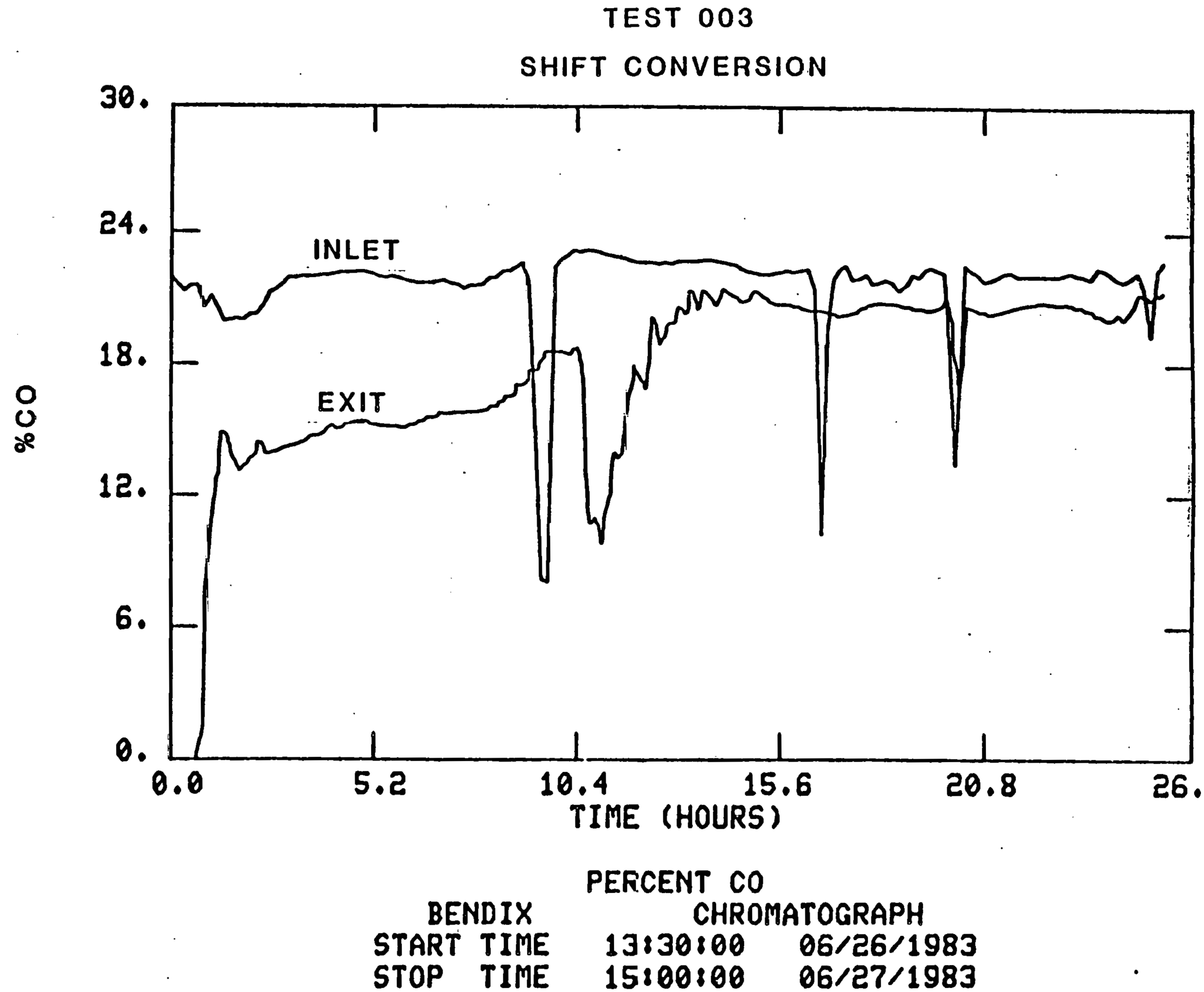
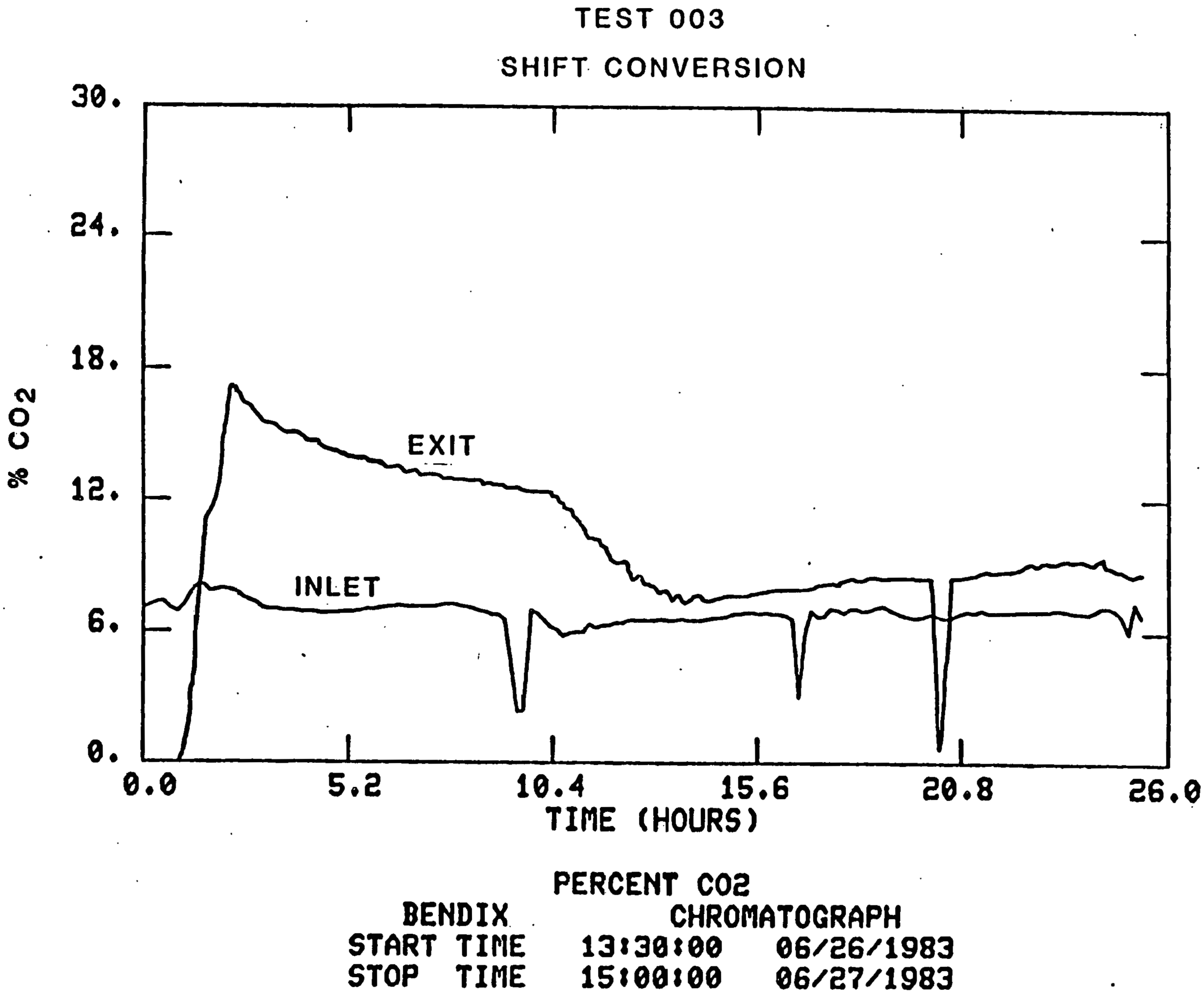
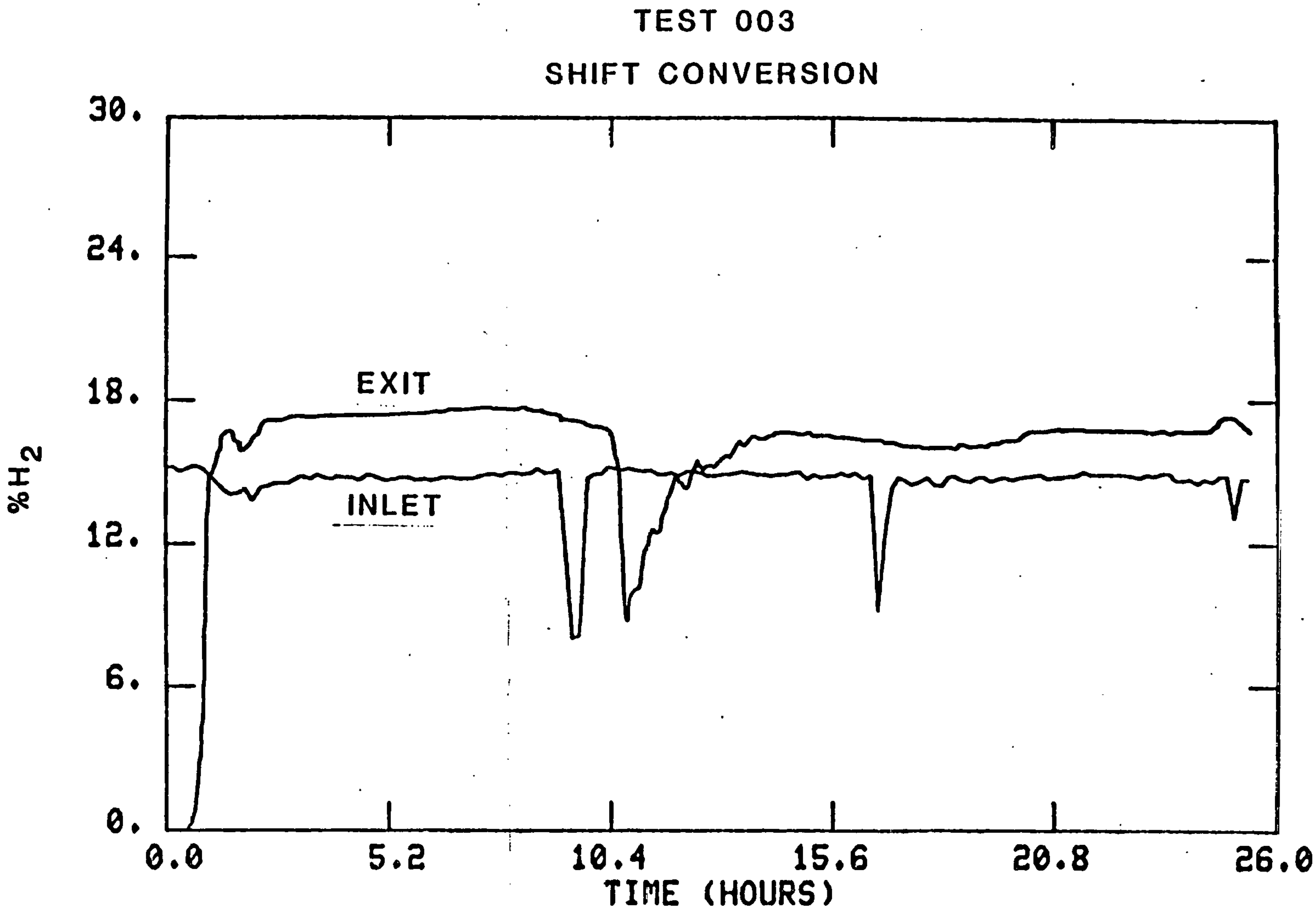

TEST 003
SHIFT CONVERSION
30.
24.
18.
12.
6.
0.
%CO
INLET
EXIT
0.0
5.2
10.4
15.6
20.8
26.
TIME (HOURS)
PERCENT CO
BENDIX CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STOP TIME 15:00:00 06/27/1983

Figure 23



TEST 003
SHIFT CONVERSION
30.
24.
18.
12.
6.
0.
% CO2
EXIT
INLET
0.0
5.2
10.4
15.6
20.8
26.0
TIME (HOURS)
PERCENT CO2
BENDIX CHROMATOGRAPH
START TIME 13:30:00 06/26/1983
STOP TIME 15:00:00 06/27/1983

Figure 24



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

Figure 25

TABLE I

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS
TESTS 001 to 005

		Sample No.	Location	Date Time	H_2	O_2	N_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	iC_4	nC_4	ppm H_2S +	ppm COS	ppm CH_3SH	ppm CS_2	ppm thio	ppm SO_2 *
SULFIDATION	T001	HG-1	Exit	6/23/83 1700	15.02	2.13	50.99	2.91	12.96	0.32	14.91	0.14	0.12	ND	ND	ND	ND	ND	ND	4.2	-
		HG-2	Exit	6/23/83 2230	16.24	2.89	50.12	2.62	15.51	0.28	11.65	0.1	0.09	ND	ND	ND	ND	ND	ND	4.5	-
		HG-3	Exit	6/24/83 0230	18.33	0.85	46.74	2.96	20.04	0.33	9.99	0.14	0.12	ND	ND	ND	ND	ND	ND	4.8	-
		HG-4	Exit	6/24/83 0630	18.76	0.97	46.20	2.90	20.98	0.32	9.13	0.12	0.11	ND	ND	ND	ND	ND	ND	4.2	-
		HG-5	Exit	6/24/83 0845	19.30	0.76	45.63	2.94	20.70	0.32	9.63	0.11	0.10	ND	ND	ND	ND	ND	ND	3.3	-
		HG-6	Exit	6/24/83 2330	14.48	2.38	52.44	2.49	18.00	0.30	9.24	0.08	0.08	ND	ND	ND	.08	ND	ND	5.4	-
		HG-7	Exit	6/25/83 0330	17.82	1.06	48.81	2.86	19.00	0.27	9.52	0.09	0.07	ND	ND	ND	0.2	ND	ND	6.8	-
		HG-8	Exit	6/25/83 0730	17.93	0.76	49.19	2.69	18.68	0.28	9.78	0.11	0.08	ND	ND	4.5	9.3	ND	ND	12.2	-
REGENER-ATION	T002	HG-9	Exit	6/25/83 2145	8.21	2.16	72.19	ND	0.53	ND	16.38	ND	0.02	ND	ND	-	-	-	-	-	4.35
		HG-10	Exit	6/26/83 0130	0.87	2.02	91.92	ND	0.02	ND	4.67	ND	ND	ND	ND	-	-	-	-	-	-
		HG-11	Exit	6/26/83 0530	ND	3.54	92.75	ND	0.11	ND	3.11	ND	ND	ND	ND	-	-	-	-	-	-
SULFIDATION	T003	HG-12	Exit	6/26/83 1515	17.39	1.65	47.72	2.99	16.21	0.31	13.00	0.14	0.09	ND	ND	16.2	2.3	2.5	ND	9.7	ND
		HG-13	Exit	6/26/83 1915	21.16	0.64	44.54	3.21	17.55	0.35	11.76	0.16	0.14	ND	ND	1.7	0.2	ND	ND	5.9	-
		HG-14	Exit	6/27/83 0130	17.53	1.35	48.53	2.75	18.79	0.32	9.95	0.15	0.14	ND	ND	ND	0.2	ND	ND	5.5	-
		HG-15	Exit	6/27/83 0530	18.98	0.86	46.28	2.88	21.56	0.32	8.34	0.14	0.14	ND	ND	ND	ND	ND	ND	6.9	-
		HG-16	Exit	6/27/83 0930	19.41	0.75	45.60	3.21	20.95	0.34	8.94	0.17	0.14	ND	ND	ND	0.7	ND	ND	8.7	-
		HG-17	Exit	6/27/83 1330	18.55	0.64	45.64	3.45	22.18	0.39	8.36	0.15	0.14	ND	ND	22.5	10.9	ND	ND	6.6	-
REGENERATION	T004	HG-18	Exit	6/28/83 0145	5.93	1.91	83.61	ND	0.21	ND	7.83	ND	ND	ND	ND	2994.9	5.5	ND	6.5	ND	10.0
		HG-19	Exit	6/28/83 0430	3.53	1.26	89.07	ND	0.17	ND	5.48	ND	ND	ND	ND	-	488.7	ND	150.0	ND	329.5
		HG-20	Exit	6/29/83 0830	ND	0.45	95.88	ND	ND	ND	3.17	ND	ND	ND	ND	-	360.8	ND	150.0	ND	4000.0
		HG-21	Exit		-	-	-	-	-	-	-	-	-	-	-	-	1350	ND	295.0	ND	1812.0
		HG-22	Exit	6/28/83 1630	0.31	1.02	98.17	ND	ND	ND	ND	ND	ND	ND	ND	-	-	-	-	-	-
SULFIDATION	T005	HG-23	Exit	6/29/83 1400	19.60	0.65	45.03	3.38	18.65	0.37	11.52	0.16	0.14	ND	ND	16.0	7.0	ND	ND	7.4	-
		HG-24	Exit	6/29/83 1545	48.96	0.08	1.44	5.34	13.22	0.49	29.59	0.14	0.24	ND	ND	29.6	6.2	ND	ND	4.2	-
		HG-25	Exit	6/29/83 1915	18.95	0.83	45.94	2.51	21.71	0.23	9.06	0.16	0.11	ND	ND	4.0	1.0	ND	ND	7.0	-
		HG-26	Exit	6/30/83 0015	37.53	1.82	7.84	5.71	15.42	0.57	30.10	0.24	0.26	ND	ND	4.0	2.0	ND	0.1	9.9	0.2
		HG-27	Exit	6/30/83 0415	42.44	1.00	4.48	5.32	15.26	0.50	30.13	0.17	0.21	ND	ND	56.8	5.4	ND	3.7	11.2	-

ND = Not Detectable

\- = Analysis not taken

* = SO_2 is suspect due to possible solubility in condensed water

\+ = H_2S Detectable level 0.2 ppm

NOTE: All analysis other than sulfur analysis is in units of % by volume on a dry gas basis. Sulfur analysis is in units of ppm by volume.

Table 2

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS, TESTS 001 to 005

	Sample No.	Location	Date Time	H_2	O_2	N_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	IC_4	nC_4	PPM H_2S	PPM COS	PPM CH_3SH	PPM CS_2	PPM thio	PPM NH_3	PPM H_2O
SULFIDATION T001	FN5	S-4	0645 6/24/83	16.57%	.77%	47.91%	3.17%	22.66%	.32%	7.79%	.15%	.15%	ND	ND	3,001.8	282.1	ND	6.1	ND	ND	555.3
	FN10	S-4	0840 6/24/83	16.57%	.76%	47.35%	3.22%	23.79%	.31%	7.18%	.16%	.15%	ND	ND	3,106.5	260.5	ND	3.8	ND	ND	864.0
	FN15	S-4	0715 6/25/83	16.88%	.73%	48.81%	3.00%	22.90%	.29%	6.64%	.16%	.10%	ND	ND	2.855.0	296.8	ND	6.5	ND	ND	1,793.9
	FN18	S-4	0825 6/25/83	17.44%	.76%	47.92%	3.21%	24.96%	.31%	4.66%	.15%	.10%	ND	ND	3,162.9	301.3	1.4	36.0	21.0	ND	1,955.9
SULFIDATION T003	FN49	S-4	13_5 6/26/83	16.67%	.61%	47.90%	3.09%	23.25%	.30%	7.39%	.15%	.14%	ND	ND	3,496.1	245.8	ND	14.7	3.0	ND	1,483.0
	FN54	S-4	0740 6/27/83	15.94%	.61%	48.63%	3.06%	23.44%	.32%	7.19%	.17%	.14%	ND	ND	4,187.3	335.1	ND	15.5	2.0	ND	1,956.7
	FN57	S-4	0855 6/27/83	16.33%	.65%	47.94%	3.15%	23.84%	.32%	6.92%	.13%	.16%	ND	ND	4,221.8	286.8	ND	16.2	ND	ND	1,931.6
	FN61	S-4	1045 6/27/83	16.44%	.70%	48.00%	3.08%	23.48%	.31%	7.19%	.17%	.15%	ND	ND	4,180.2	269.0	ND	16.7	3.0	ND	1,961.0
	FN65	S-4	1255 6/27/83	16.16%	.58%	48.36%	2.99%	23.55%	.31%	7.25%	.16%	.14%	ND	ND	3,867.4	274.0	1.5	13.9	2.8	ND	1,818.0
	FN69	S-4	1445 6/27/83	16.44%	.64%	48.10%	2.79%	24.25%	.29%	6.73%	.13%	.13%	ND	ND	3,357.9	280.4	ND	13.0	2.0	ND	1,803.0
SULFIDATION T005	FN99	S-4	1530 6/29/83	18.66%	.58%	43.80%	3.69%	24.91%	.37%	7.13%	.18%	.25%	ND	ND	3,104.2	413.2	1.5	13.7	3.0	ND	1,932.0
	FN101	S-4	0635 6/30/83	40.82%	.12%	1.52%	6.05%	23.46%	.51%	26.56%	.21%	.25%	ND	ND	5,369.1	302.1	2.6	7.9	58.0	ND	2,109.0
	FN104	S-4	0618 6/30/83	41.05%	.08%	1.58%	5.96%	23.21%	.53%	26.37%	.21%	.24%	ND	ND	5,453.3	283.4	6.3	15.6	5.9	ND	2,478.5
	FN108	S-4	0905 6/30/83	41.20%	.10%	1.34%	6.22%	24.34%	.55%	25.24%	.23%	.25%	ND	ND	5,244.0	256.1	3.3	11.7	2.6	ND	2,301.0
	FN112	S-4	1110 6/30/83	40.63%	.17%	1.08%	6.08%	24.33%	.58%	26.19%	.20%	.27%	ND	ND	5,218.9	240.4	3.1	9.3	5.0	ND	2,356.0
	FN116	S-4	1300 6/30/83	40.89%	.18%	1.19%	5.74%	26.81%	.51%	23.75%	.18%	.24%	ND	ND	5,273.4	283.8	4.4	5.5	5.1	ND	2,195.6

NOTES:

ND = Not Detectable

All analysis other than sulfur is in units of % by volume on a dry gas basis. Sulfur analysis is in units of ppm by volume.

Table 3

MINERAL ANALYSIS AFTER TEST 005
ZINC FERRITE L-1504 - UNITED CATALYSTS

BED HEIGHT (INCHES)	HEMATITE Fe_2O_3	FRANKLINITE $ZnFe_2O_4$ AND/OR MAGNETITE Fe_3O_4 (4)	ZINCITE ZnO	ALPHA ZnS	BETA ZnS	PYRRHOTITE FeS
	PERCENT TII (3)					
0 (1)	-	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 (2)	-	70	18	9	4	-
Fresh Sorbent	4	89	6	-	-	-

NOTES:

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

TABLE 4

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)	Skeletal Density (G/CC)(3)	Porosity % (5)	Crush Strength (Kg DWL) ± S.D.(7)	SkeletalDensity (G/CC)(8)	Porosity %(9)
0(1)	1.97	29.88	1.43	4.37	0.93	7.0 ± 3.3	-	-
6	1.88	31.41	1.49	4.55	0.93	8.4 ± 3.5	-	-
16	2.58	27.78	2.09	4.26	1.68	7.3 ± 3.5	3.84	27.9
23	2.89	25.92	2.43	4.33	1.96	8.1 ± 3.1	-	-
26	3.48	28.97	2.24	4.34	2.26	4.4 ± 2.1	3.84	36.1
36	2.37	7.29	2.30	4.86	1.76	4.2 ± 2.6	-	-
46(2)	3.27	8.60	3.11	5.14	2.75	2.4 ± 1.8	4.57	50.2
--METC FRESH SORBENT	0	0.13	3.13	6.16	2.59	6.3 ± 2.8	-	-
--UCI			3.2	85(4)	-	7.2	-	60.6(6)
ABSORBER FINES	6.93	26.29						

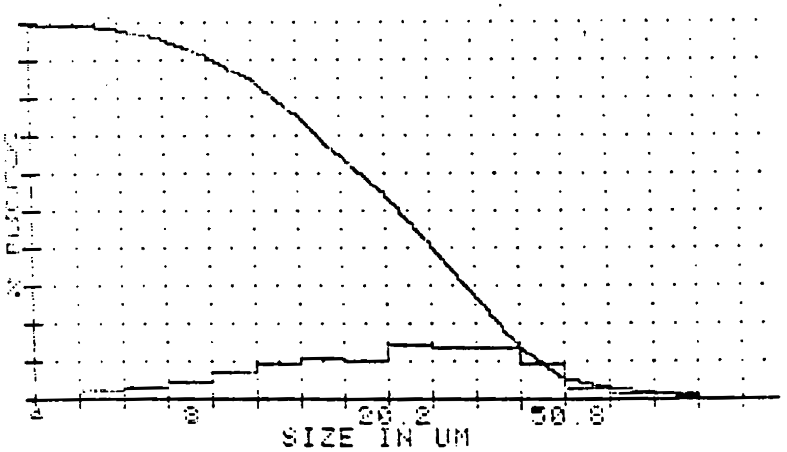
NOTES:

1. Bottom/Inlet during sulfidation - Outlet during regeneration
2. Top/Outlet during sulfidation - Inlet during regeneration
3. Skeletal Density by Helium Pycnometry
4. Bulk Density (Lbs/Cu Ft)
5. Porosity by Nitrogen Adsorption for pores up to 600Å diameter
6. Porosity by Mercury Porosimetry for pores down to 30Å diameter
7. Crush strength data are average and standard deviation of 15 measurements
8. Skeletal density by mercury porosimetry
9. Porosity by mercury porosimetry for pores down to 120Å diameter.

Table 5

DATE: 6/27/83
SAMPLE: CYCLONE #2 GRINDLEY 83-654
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA 11
APERTURES: 280
OPERATOR:REC

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	4	0	100
2	5.04	1.7	100
3	6.35	2.6	98.3
4	8	4.3	95.7
5	10.08	7	91.4
6	12.7	9.4	84.4
7	16	10.5	75
8	20.16	9.9	64.5
9	25.4	14.1	54.6
10	32	13.7	40.5
11	40.32	13.3	26.8
12	50.8	8.7	13.5
13	64	2.7	4.8
14	80.63	1.4	2.1
15	101.59	.8	.7
16	128	0	0



Total Carbon	83.12	%WT
Ash	10.23	%WT
Sulfur	1.00	%WT

VOLUME % STATISTICS

MEAN:	25.52 UM
MEDIAN:	27.37 UM
MODE:	31.32 UM
STANDARD DEVIATION:	1.89 UM
SKEWNESS:	.94 NEGATIVE
KURTOSIS:	1.51 PLATYKURTIC

Table 6

PARTICULATE SAMPLING DATA

HOT GAS DESULFURIZATION TEST UNIT

Run #	Time	Sample Duration	Sampling Flow Rate	Particle loading Inlet	Particle loading Exit	Mean particle size Inlet	Mean particle size Exit
		Minutes	liters/min.	g/Nm3		Microns	
T001	22:00 Hours 6/23/83	1	13	-	-	10.4	-
T003	16:52 Hours 6/26/83	30	13	1.6	-	15.3	-
T003	18:04 Hours 6/26/83	30	13	-	3.9	-	2.7
T003	12:40 Hours 6/27/83	30	13	-	4.6	-	2.3
T003	14:25 Hours 6/27/83	30	13	0.3(1)	-	6.0	-
T005	11:22 Hours 6/29/83	30	13	0.3	-	2.9	-
T005	16:06 Hours 6/29/83	30	11	-	2.8(2)	-	3.8

(1) Carbon 15.5% Ash 82.79%

(2) Carbon 2.79% Ash 97.19%

.e 7

Particle Distribution

DATE: 7/27/83
SAMPLE: DESULFUR BAGNELL DE#2 83-696
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA 11
APERTURES: 280
OPERATOR:REC

T001 INLET
22.00 HOURS
6/23/83

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	.5	0	100
2	.63	3.1	100
3	.79	2.8	96.9
4	1	3	94.1
5	1.26	3.6	91.1
6	1.59	3.8	87.5
7	2	3.9	83.7
8	2.52	4.3	79.8
9	3.17	4.7	75.5
10	4	5.3	70.8
11	5.04	5.4	65.5
12	6.35	4.3	60.1
13	8	4.9	55.8
14	10.08	6.1	50.9
15	12.7	6.6	44.8
16	16	7.4	38.2
17	20.16	7.1	30.8
18	25.4	8.2	23.7
19	32	6.2	15.5
20	40.32	3.2	9.3
21	50.8	.7	6.1
22	64	.6	5.4
23	80.63	.1	4.8
24	101.59	3.5	4.7
25	128	1.3	1.2

VOLUME % STATISTICS

MEAN:	8.91 UM
MEDIAN:	10.43 UM
MODE:	27.54 UM
STANDARD DEVIATION:	3.7 UM
SKEWNESS:	.79 NEGATIVE
KURTOSIS:	783.48 LEPTOKURTIC

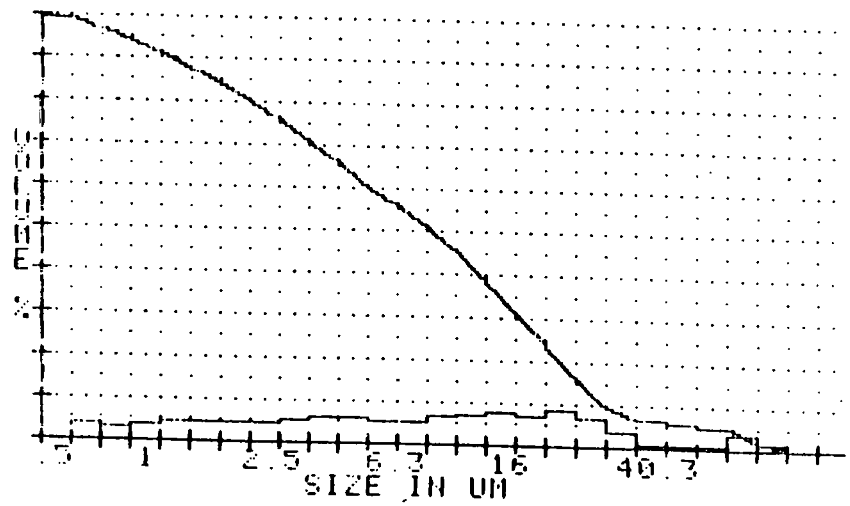


Table 8
Particle D ibution

T003 INLET
16.52 HOURS
6/26/83

DATE: 7/27/83
SAMPLE: DE #3 BAGNELL 83-697
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA 11
APERTURES: 280
OPERATOR:REC

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	4	0	100
2	5.04	.9	100
3	6.35	1.8	99.1
4	8	5	97.3
5	10.08	14.7	92.3
6	12.7	34.8	77.6
7	16	32.2	42.8
8	20.16	7.9	10.6
9	25.4	1.8	2.7
10	32	.6	.9
11	40.32	.3	.3
12	50.8	.1	0
13	64	0	0
14	80.63	0	0
15	101.59	0	0
16	128	0	0

VOLUME % STATISTICS

MEAN:	15.09 UM
MEDIAN:	15.27 UM
MODE:	15.58 UM
STANDARD DEVIATION:	1.34 UM
SKEWNESS:	1 NEGATIVE
KURTOSIS:	1.04 PLATYKURTIC

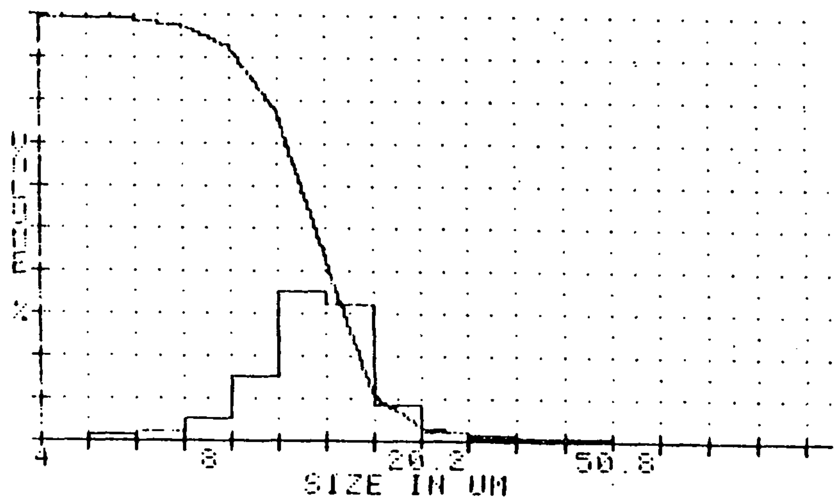


Table 9
Particle Distribution

T003 OUTLET
18.04 HOURS
6/26/83

DATE: 7/27/83
SAMPLE: DE #4 BAGNELL 280 83-698
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 280
OPERATOR:REC

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	.5	0	100
2	.63	0	100
3	.79	3.7	100
4	1	6.9	96.3
5	1.26	11.4	89.4
6	1.59	12.5	78
7	2	12.6	65.5
8	2.52	10.3	52.9
9	3.17	12	42.6
10	4	9.9	30.6
11	5.04	5.4	20.7
12	6.35	2.5	15.3
13	8	4.6	12.8
14	10.08	2.4	8.2
15	12.7	.8	5.8
16	16	.5	5
17	20.16	.6	4.5
18	25.4	.9	3.9
19	32	.9	3
20	40.32	1.1	2.1
21	50.8	.3	1
22	64	.2	.7
23	80.63	0	.5
24	101.59	0	.5
25	128	.5	.5

VOLUME % STATISTICS

MEAN:	3.12 UM
MEDIAN:	2.7 UM
MODE:	2.02 UM
STANDARD DEVIATION:	2.4 UM
SKEWNESS:	2.49 POSITIVE
KURTOSIS:	26.75 LEPTOKURTIC

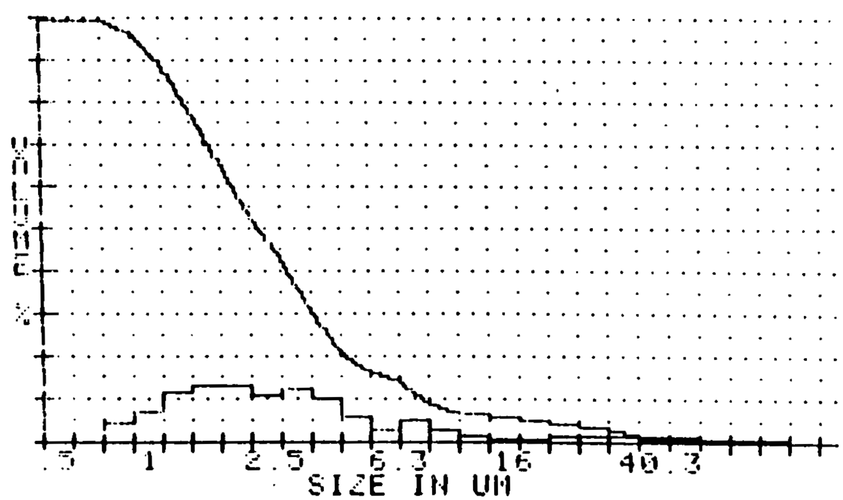


Table 10
Particle Distribution

T003 OUTLET
12.40 HOURS
6/27/83

DATE: 7/27/83
SAMPLE: DE #5 BAGNELL 280 83-699
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA 11
APERTURES: 280
OPERATOR:REC

****

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	.5	0	100
2	.63	0	100
3	.79	1.8	100
4	1	4.2	98.2
5	1.26	12.3	94
6	1.59	19.5	81.7
7	2	21.1	62.2
8	2.52	16.1	41.1
9	3.17	13	25
10	4	6.2	12
11	5.04	1.9	5.8
12	6.35	.9	3.9
13	8	1.3	3
14	10.08	.4	1.7
15	12.7	.3	1.3
16	16	.2	1
17	20.16	.2	.8
18	25.4	.2	.6
19	32	.1	.4
20	40.32	.1	.3
21	50.8	0	.2
22	64	0	.2
23	80.63	0	.2
24	101.59	0	.2
25	128	0	.2

****
VOLUME % STATISTICS
****

MEAN: 2.42 UM
MEDIAN: 2.29 UM
MODE: 2.12 UM
STANDARD DEVIATION: 1.68 UM
SKEWNESS: 1.23 POSITIVE
KURTOSIS: 1.83 PLATYKURTIC

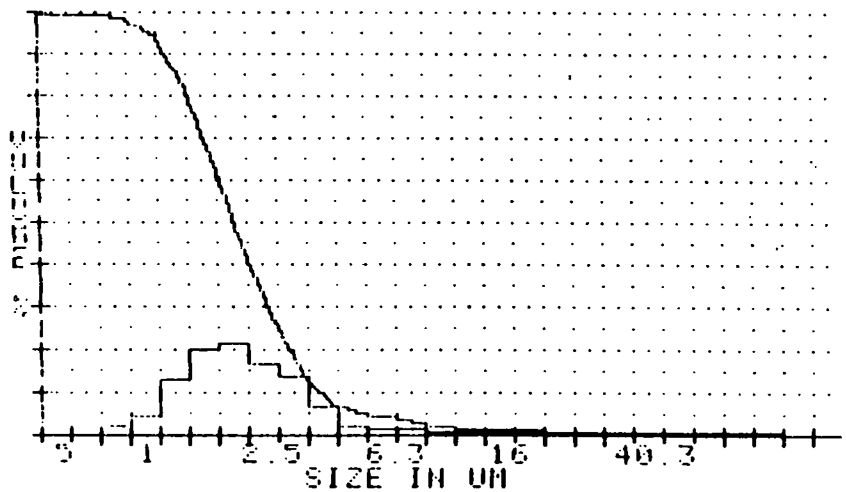


Table 11
Particle Distribution

T003 INLET

14.25 HOURS
6/27/83

DATE: 7/27/83
SAMPLE: DE #6 BAGNELL 50 83-700
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA 11
APERTURES: 50
OPERATOR:REC

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	.5	0	100
2	.63	0	100
3	.79	.9	100
4	1	1.2	99.1
5	1.26	2.2	97.9
6	1.59	3.1	95.7
7	2	4.6	92.6
8	2.52	5.5	88
9	3.17	9.9	82.5
10	4	13.1	72.6
11	5.04	13.2	59.5
12	6.35	14.1	46.3
13	8	15.6	32.2
14	10.08	9.6	16.6
15	12.7	4.1	7
16	16	2.9	2.9

VOLUME % STATISTICS

MEAN:	5.57 UM
MEDIAN:	5.96 UM
MODE:	8.38 UM
STANDARD DEVIATION:	1.88 UM
SKEWNESS:	.87 NEGATIVE
KURTOSIS:	1.61 PLATYKURTIC

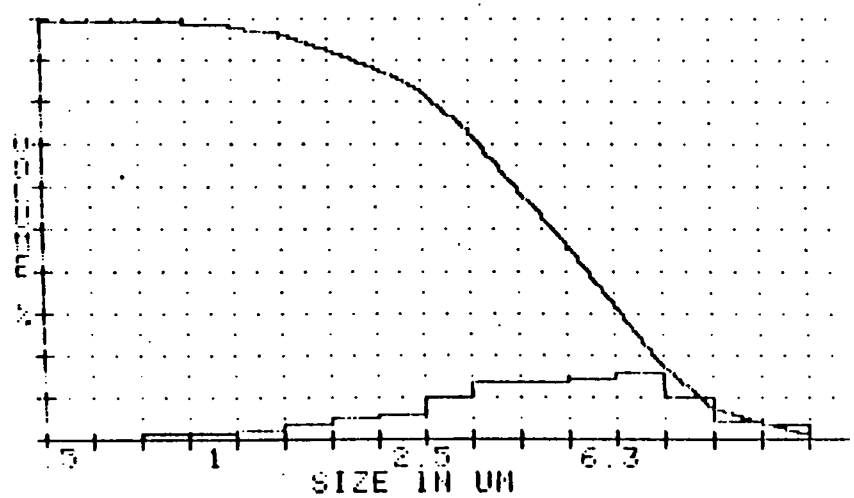


Table 12
Particle Distribution

T005 INLET
11.22 HOURS
6/29/83

DATE: 7/27/83
SAMPLE: DE #7 BAGNELL 50 83-701
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA 11
APERTURES: 50
OPERATOR:REC

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	.5	0	100
2	.63	0	100
3	.79	.2	100
4	1	.4	99.8
5	1.26	3.4	99.4
6	1.59	9.6	96
7	2	21.1	86.4
8	2.52	29.2	65.3
9	3.17	16.8	36.1
10	4	14.3	19.3
11	5.04	4	5
12	6.35	.6	1
13	8	.4	.4
14	10.08	0	0
15	12.7	0	0
16	16	0	0

VOLUME % STATISTICS

MEAN:	2.89 UM
MEDIAN:	2.84 UM
MODE:	2.76 UM
STANDARD DEVIATION:	1.42 UM
SKEWNESS:	1 POSITIVE
KURTOSIS:	1.05 PLATYKURTIC

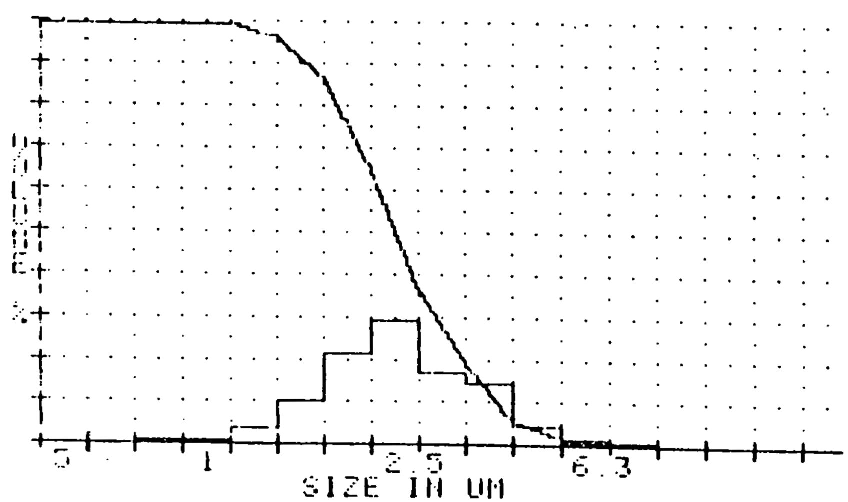


Table 13
Particle Distribution

T005 OUTLET
16.06 HOURS
6/29/83

DATE: 7/27/83
SAMPLE: DE #8 BAGHELL 50 83-702
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA 11
APERTURES: 50
OPERATOR:REC

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	.5	0	100
2	.63	0	100
3	.79	1.9	100
4	1	3.7	98.1
5	1.26	7.6	94.4
6	1.59	9.4	86.8
7	2	9.4	77.4
8	2.52	8.4	68
9	3.17	11.9	59.6
10	4	13.5	47.7
11	5.04	13	34.2
12	6.35	10.2	21.2
13	8	7.9	11
14	10.08	1.7	3.1
15	12.7	1.5	1.4
16	16	0	0

VOLUME % STATISTICS

MEAN:	3.59 UM
MEDIAN:	3.83 UM
MODE:	4.78 UM
STANDARD DEVIATION:	1.92 UM
SKEWNESS:	.96 NEGATIVE
KURTOSIS:	1.48 PLATYKURTIC

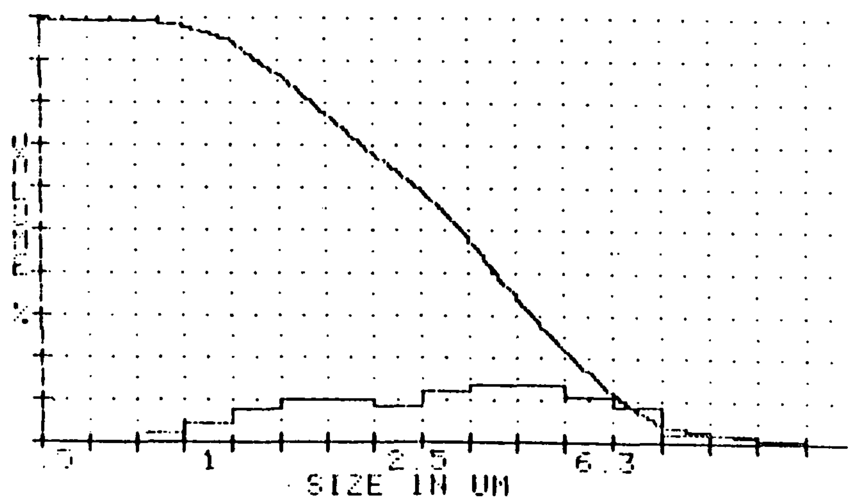


Table 14

AQUEOUS PHASE AT HOT GAS DESULFURIZATION INLET, TESTS 001 - 005

TEST TYPE AND NUMBER	LAB SAMPLE NO.	LOCATION	DATE TIME	Zn ppm	Hg ppm	Cr ppm	As ppm	Se ppm	FE ppm	Al ppm	Ni ppm	K ppm	Na ppm	NH_4 ppm	F ppm	Cl ppm	PO_4 ppm	Br ppm	NO_3 ppm	SO_4 ppm
				ANIONS, CATIONS AND METALS																
T001	27496	S-4	6/24/83 0400	<0.1	< 0.2	0.989	3.18	2.47	13.9	6.12	8.62									
T002	27501	S-4	6/25/83 1200	<0.1	< 0.2	1.42	0.75	2.35	12.0	2.62	5.01	15.6	35.8	7439	168	5259	13.9	67.7	< 2	857
T004	27524	S-4	6/28/83 1800	<0.1	< 0.2	0.110	0.442	<1.0	8.14	10.6	0.322	12.1	28.6	6277	73.3	3541	<3	41.7	< 2	257
	27525	S-4	6/28/83 1800	<0.1	0.94	0.093	1.33	2.11	7.62	10.35	0.370	13.8	54.2	6596	74.9	3393	<3	42.7	< 2	255
	27528	S-4	6/29/83 0200	<0.1	2.17	<0.007	0.551	1.62	4.57	1.50	0.497	8.34	1.0	6384	34.9	1294	< 3	20.8	< 2	324
T005	27534	S-4	6/29/83 2330	<0.1	< 0.2	<0.007	2.08	2.22	5.03	5.32	0.164	16.3	8.5	3173	99	2552	10.7	28.5	299	2065
	27539	S-4	6/30/83 1100	<0.1	< 0.2	<0.007	0.504	1.69	3.59	2.80	< 0.002	9.48	57.1	7872	27.8	1000	<3	13.5	< 2	151
	27540	S-4	6/30/83 1500	<0.1	< 0.2	<0.007	0.466	4.29	3.09	1.66	< 0.02	9.28	4.8	2538	28.8	620	<3	12.4	28.6	360
	27543	S-4	6/30/83 2100	<0.1	<0.2	<0.007	1.01	1.19	3.00	3.23	0.225	15.3	56.6	6626	41.1	2296	<3	17.2	< 2	152

NOTES:

1. Above analysis was made on samples taken at the sidestream inlet, whether gas was flowing to the test unit or not (i.e., during regeneration).
2. Sampling location S-4 is after gasifier cyclone and before humidifier.
3. Metals determined by atomic absorption/atomic emission spectroscopy at DOE/METC.
4. Anions and cations determined by ion chromatography at DOE/METC.

Table 15

TAR ULTIMATE ANALYSIS

AT HOT GAS DESULFURIZATION INLET

TEST TYPE	TEST NO.	SAMPLE LOCATION	LAB SAMPLE NO.	TIME/ DATE	ASH	S	H	T.C.	N	O	BTU
Sulfidation	T001	S-4	27943	0400 6/24/83	0.26	1.15	7.35	70.85	1.92	18.47	13060
Sulfidation	T003	S-4	28193	1300-1400 6/27/83	0.16	1.11	7.34	75.56	2.53	13.3	14467
Sulfidation	T005	S-4	27948	1100 6/30/83	0.61	1.04	7.65	65.36	1.74	23.60	14202
Sulfidation	T005	S-4	27949	1500 6/30/83	0.45	1.44	8.33	61.81	1.52	26.45	13836

NOTES:

1. Sampling location S-4 is after gasifier cyclone and before humidifier.
2. Oxygen obtained by difference and includes oxygen in the moisture present.
3. Total sulfur analysis obtained by ASTM D-3177-75 at DOE/METC.
4. Carbon and hydrogen analysis by ASTM D-3178-73 at DOE/METC.
5. Nitrogen analysis by ASTM D-3179-73 at DOE/METC.
6. Ash analysis by ASTM D-3174 at DOE/METC.
7. BTU content analysis by ASTM D-2015 at DOE/METC.

Table 16

AQUEOUS PHASE CONDENSATE AT HOT GAS DESULFURIZATION EIIT, TESTS 001-005

TEST TYPE AND NUMBER		HGD SAMPLE NO.	DATE TIME	ANIONS, CATIONS AND METALS																
				Zn ppm	Hg ppm	Cr ppm	As ppm	Se ppm	FE ppm	Al ppm	Ni ppm	K ppm	Na ppm	NH_4 ppm	F ppm	Cl ppm	PO_4 ppm	Br ppm	NO_3 ppm	SO_4 ppm
SULFI-DATION	T001	LC1	6/24/83 0825	10.73	<0.2	0.632	0.429	<1.0	5.20	0.258	0.248	1.69	53.0	16383	48.7	1847	<3	4.5	<2	34.5
		LC2	6/25/83 0230	0.590	<0.2	2.10	0.578	<1.0	9.42	1.71	4.40	29.12	54.0	7573	418	13289	5.6	42.7	<2	44.6
REGENER-ATION	T002	LC3	6/25/83 2130	9.42	<0.2	0.048	0.524	<1.0	1.60	0.824	0.284	9.72	19.1	4055	106	4121	<3	32.3	6.2	101
		LC4	6/26/83 0130	0.77	<0.2	0.880	0.69	1.99	36.8	0.49	2.70	4.50	6.1	268	38.5	496	<3	20.8	1067	521
		LC5	6/26/83 0530	1.48	8.56	11.56	0.661	<1.0	157.4	0.792	4.88	4.59	21.1	488	33.4	641	<3	29	1740	1053
REGENERATION	T004	LC6	6/28/83 0515	0.48	<0.2	0.20	0.96	2.73	1.66	<0.02	0.10	-	13.6	1019	26.8	223	<3	<4	34	187
		LC7	6/28/83 0830	6.15	<0.2	>10.0	0.79	3.55	209	1.69	12.8	-	15.5	798	20.0	2300	<3	45.7	< 4	593
		LC8	6/28/83 1230	1.75	<0.2	16.5	0.53	3.36	>10.0	1.51	4.58	-	13.6	663	50.7	2080	<3	62.4	200	1978
		LC9	6/28/83 1630	0.694	1.60	9.88	0.665	<1.0	7.35	0.695	2.76	1.11	12.7	273	12.9	488	<3	37.9	96.2	1275

NOTES:

1. Sampling location is at condenser CIC knockout.
2. Metals determined by atomic absorption/atomic emission spectroscopy at DOE/METC
3. Anions and cations determined by ion chromatography at DOE/METC.

Table 17

COMPOSITE ANALYSIS OF EXIT AQUEOUS CONDENSATE DURING SULFIDATION

COMPONENT	AIR BLOWN (RUNS 001, 003, and 005) IHI KEMRON (mg/l)	AIR BLOWN (RUNS 001, 003, and 005) DOE/METC (ppm)	OXYGEN BLOWN (RUN 005) DOE/METC (ppm)
Chloride, dissolved (Cl)	1000	1382	545
Cyanide, total (CN)	23		
Nitrogen, Ammonia, total (N)	17,000	10,709	6209
Phenolics, total (Phenol)	33		
Sulfate (SO_4)	200	264	81.0
Sulfide (S)	96		
Sulfite (SO_3)	230		
Fluoride (F)	50	47.5	30.3
Thiocyanate (SCN)	180		
Antimony, total (Sb)	-		
Arsenic, total (As)	6.75	1.45	0.191
Iron, total (Fe)	16	24.64	6.51
Lead, total (Pb)	<0.05		
Calcium, total (Ca)	7.5		
Potassium, total (K)	5.0	7.57	1.13
Vanadium, total (V)	<0.4	<0.05	0.048
Zinc, total (Zn)	1.1	1.84	0.046
Sodium, total (Na)	3.0	11.0	2.33
Chromium (Cr)		3.13	0.305
Nickel (Ni)		0.629	0.101
Phosphate (PO_4)		<3	<3
Bromine (Br)		<4	<4
Nitrate (NO_3)		18.6	<2
Aluminum (Al)		0.155	<0.02
Selenium (Se)		0.215	0.056
Mercury (Hg)		7.31	1.73

NOTES:

1. Sampling location is at condenser CIC knockout.
2. DOE/METC metals analysis was by atomic absorption/atomic emission spectroscopy.
3. DOE/METC anions and cations determined by ion chromatography.

Table 18

COMPOSITE ANALYSIS OF EXIT AQUEOUS CONDENSATE FROM REGENERATION RUNS 002 AND 004

COMPONENT	DOE/METC RUN 002 & 004 (ppm)	DOE/METC PLANT STEAM (ppm)
Zinc (Zn)	0.889	0.582
Chromium (Cr)	0.297	<0.02
Iron (Fe)	6817	0.351
Nickel (Ni)	0.861	<0.8
Potassium (K)	7.83	0.150
Sodium (Na)	10.79	0.293
Calcium (Ca)	34.56	0.873
Vanadium (V)	0.360	<0.05
Ammonia (NH_4)	458	0.54
Fluoride (F)	29.4	0.11
Chloride (Cl)	1030	1.02
Phosphate (PO_4)	<3	<0.03
Bromine (Br)	21.5	<0.03
Nitrate (NO_3)	<2	1.12
Sulphate (SO_4)	829	2.38
Aluminum (Al)	1.79	0.169
Selenium (Se)	<0.01	<0.01
Mercury (Hg)	86.3	<0.2
Arsenic (As)	<0.01	<0.01

NOTES:

1. Sampling location is at condenser CIC knockout.
2. DOE/METC metals analysis was by atomic absorption/atomic emission spectroscopy.
3. DOE/METC anions and cations determined by ion chromatography.

Table 19

COMPOSITE ULTIMATE ANALYSIS OF EXIT TAR CONDENSATE DURING SULFIDATION (GASIFIER RUN 101)

	AIR-BLOWN ARKWRIGHT COAL (RUNS 001, 003, 005)		OXYGEN-BLOWN ARKWRIGHT COAL (RUN 005)
COMPONENT	IHI KEMRON WT %	DOE/METC WT %	DOE/METC WT %
Ash	0.023	0.29	0.12
S	2.11	2.11	3.20
H	6.64	7.63	7.23
N	1.87	1.08	1.00
O	5.87	15.51	11.77
Total Carbon	83.49	73.38	76.68
BTU/lb	15,289	15,111	15,717

NOTES:

1. Oxygen obtained by difference and includes oxygen in moisture.

Table 20

TRACE COMPONENTS IN EXIT GAS DURING SULFIDATION

COMPONENT	AIR BLOWN (RUNS 001, 003, and 005) IHI KEMRON ppmw	AIR BLOWN (RUNS 001, 003, and 005) DOE/METC ppmw	OXYGEN BLOWN (RUN 005) DOE/METC ppmw
Chloride (Cl)	45	62	289
Cyanide, total (CN)	1		
Nitrogen, Ammonia, total (N)	758	478	329
Phenolics, total (Phenol)	1.5		
Sulfate (SO_4)	8.9	12	43
Sulfide (S)	4		
Sulfite (SO_3)	10		
Fluoride (F)	2.2	2	16
Thiocyanate (SCN)	8		
Antimony, total (Sb)	-		
Arsenic, total (As)	.3	.06	0.10
Iron, total (Fe)	.7	1	3.5
Lead, total (Pb)	<.002		
Calcium, total (Ca)	.3		
Potassium, total (K)	.2	.3	0.6
Vanadium, total (V)	<.02	<.002	0.03
Zinc, total (Zn)	.05	.08	0.02
Sodium, total (Na)	.13	.5	1.23
Chromium (Cr)		.1	0.16
Nickel (Ni)		.03	0.05
Phosphate (PO_4)		<.1	<1.6
Bromine (Br)		<.2	<2.1
Nitrate (NO_3)		.8	<1.1
Aluminum (Al)		.007	<0.011
Selenium (Se)		.01	0.03
Mercury (Hg)		.33	0.92

NOTES:

1. Sampling location is at condenser CIC knockout.
2. DOE/METC metals analysis was by atomic absorption/atomic emission spectroscopy.
3. DOE/METC anions and cations determined by ion chromatography.
4. Concentration in gas phase calculated based on analysis in aqueous phase.

Table 21

TRACE COMPONENTS IN EXIT GAS DURING REGENERATION

COMPONENT	DOE/METC RUN 002 & 004 ppmw	DOE/METC PLANT STEAM ppmw
Zinc (Zn)	0.72	0.582
Chromium (Cr)	0.24	<0.02
Iron (Fe)	5556	0.351
Nickel (Ni)	0.70	<0.8
Potassium (K)	6.38	0.150
Sodium (Na)	8.79	0.293
Calcium (Ca)	28.2	0.873
Vanadium (V)	0.3	<0.05
Ammonia (NH_4)	373	0.54
Fluoride (F)	24	0.11
Chloride (Cl)	839	1.02
Phosphate (PO_4)	<2.4	<0.03
Bromine (Br)	17.5	<0.03
Nitrate (NO_3)	<1.6	1.12
Sulphate (SO_4)	675	2.38
Aluminum (Al)	1.45	0.169
Selenium (Se)	<0.008	<0.01
Mercury (Hg)	70.3	<0.2
Arsenic (As)	<0.008	<0.01

NOTES:

1. Sampling location is at condenser CIC knockout.
2. DOE/METC metals analysis was by atomic absorption/atomic emission spectroscopy.
3. DOE/METC anions and cations determined by ion chromatography.
4. Concentration in gas phase calculated based on analysis in aqueous phase.

APPENDIX F

Sidestream Testing - Gasifier Run 102

Hot Gas Desulfurization Sidestream Test Unit, Fixed-Bed Gasifier Run No. 102

Introduction

The high-temperature, sidestream desulfurization unit (1) was operated for a second series of tests, during fixed-bed gasifier Run No. 102 (2,3), between September 20 and October 2.

The principal purpose of these tests was to provide hot desulfurized gas to a combustion test unit to investigate the feasibility of using it as a fuel for heat engines (4).

Operation of Sidestream Test Unit

The sidestream unit was operated in two periods with different batches of the zinc ferrite sorbent. During the first period the gasifier was fed with Arkwright coal, and during the second, with Blacksville coal briquettes. The gasifier was operated in the air-blown mode. The operational schedule is shown in Figure 1.

- First Period

  The reactor contained the customary 20.4 kg of sorbent but, contrary to the first test series, reactor furnace temperatures were set at 1,100°F, compared to 1,000°F previously, which, it was hoped, would increase the sulfur absorption rate and result in longer times to breakthrough. Gasifier pressure was about 150 psig.

  The unit was operated for two short absorption spells (Test Nos. 006 and 008), with an intermediate regeneration (Test No. 007). Average operating parameters are shown on the ADACS flow schematics in Figures 2, 3, 4A, 4B, and 4C.

  In Test No. 006, gas was passed through the unit at a space velocity of 2,000 h^{-1} for about an hour, which was then increased to 8,000 h^{-1}, the additional gas going to the combustor. After a further 2 hours, H_2S breakthrough began and gas to the combustor was cut off. A renewed breakthrough occurred after 2 more hours at a space velocity of 2,000 h^{-1}. Reactor temperatures, pressure drop, and gas flow rates are shown in Figures 5, 6, and 7, respectively. Initially there was a temperature rise in the reactor of about 100°F, falling to about 50°F later, attributed to the exothermic partial reduction of the sorbent and the "shift" reaction. The reactor pressure drop gradually increased and exceeded the maximum instrument reading of about 160 inches of water at the higher space velocity, well above the bed lifting pressure.

  The sorbent was regenerated in Test No. 007 for about 10 hours. Because of the low sulfur loading it was found necessary to increase the air/steam ratio above normal. Nonetheless, temperatures barely reached

1,300°F and pressure drop on completion still exceeded the instrument maximum. Reactor temperatures, pressure drop and flow rates are shown in Figures 8, 9, and 10, respectively.

In Test No. 008, gas was immediately supplied to the combustor at the same conditions as in Test No. 006. H_2S breakthrough again occurred after about 2 hours. Gas to the combustor was cut off and the unit continued on-stream at the lower space velocity of 2,000 h^{-1} for a further 4 hours until a renewed breakthrough occurred. At this point the reactor pressure drop exceeded 50 psig, based on unit inlet and outlet pressure indicators, and it was decided to replace the sorbent before carrying out further tests. The sorbent was found to have agglomerated to a great extent, especially at the reactor exit, apparently because of coke formation and the high differential pressures. Reactor temperatures, pressure drop (inlet and outlet pressures) and flow rates are shown in Figures 11, 12, and 13, respectively.

- <u>Second Period</u>

For this period the reactor was refilled with fresh sorbent. Reactor furnace temperatures were set at 1,000°F, a temperature which had given lower reactor pressure drops previously. Gasifier pressure was about 100 psig.

Two absorption spells (Test Nos. 009 and 011) and two regenerations (Test Nos. 010 and 012) were carried out. Average operating parameters are shown on the ADACS flow schematics in Figures 14, 15, 16, and 17.

In Test No. 009, after an initial short time at 2,000 h^{-1}, the space velocity was stepped up to 5,000 h^{-1}; the additional gas flowed to the combustor. This flow was half that in Test Nos. 006 and 008, to provide a longer on-stream time to breakthrough and a more acceptable reactor pressure drop. Breakthrough of H_2S occurred after about 6 hours, when the pressure drop was about 6 psig. Upon cutting off the flow to the combustor, reactor pressure drop fell to 1.5 psig. A renewed breakthrough occurred after a further hour, when the pressure drop had increased to 1.75 psig. Reactor temperatures, pressure drop, and flow rates are shown in Figures 18, 19, and 20.

The sorbent was regenerated in Test No. 010 by the regular procedure and reactor pressure drop returned to approximately its original level. Reactor temperatures, pressure drop, and flow rates are shown in Figures 21, 22, and 23.

In Test No. 011 no gas was supplied to the combustor, gas flow through the reactor being at a space velocity of 2,000 h^{-1} throughout. H_2S breakthrough occurred after about 19 hours. Reactor pressure drop rose from 0.5 psi to 1 psi after 10 hours and then more rapidly to about 6 psi finally, exceeding the bed lifting pressure. Reactor temperatures, pressure drop, and flow rate are shown in Figures 24, 25, and 26.

A second regeneration was carried out in Test No. 012 and the sorbent removed. No agglomeration was found. Reactor temperatures, pressure drop, and flow rates are shown in Figures 27, 28, and 29.

Results

- Data Summary

The various process conditions and data collected in Test Nos. 006 to 012 are summarized in Figures 30 to 36.

- Absorption of Sulfur Compounds

The hydrogen sulfide concentrations in exit gas from the unit during Test Nos. 006, 008, 009, and 011, measured by both detector tube and baseline gas chromatograph, are plotted in Figures 37 to 40; SO_2 levels, measured by detector tube, are also shown occasionally. H_2S levels before breakthrough are generally less than about 5 ppm. Because of the higher space velocities through the reactor, the amount of sulfur absorbed in Test Nos. 006 and 008 is less than half that found previously at a space velocity of ~ 2,000 h^{-1}, albeit at the lower temperature of ~ 1,000°F. This indicates that resistance to diffusion of H_2S into the sorbent is very significant at these conditions.

In Test Nos. 009 and 011, gas from Blacksville coal containing more H_2S than that from Arkwright coal was desulfurized; breakthrough times were correspondingly shorter.

In order to provide gas to the combustion test unit for a longer time in Test No. 009 than in Test Nos. 006 and 008, the space velocity was set at ~ 5,000 h^{-1} instead of ~ 8,000 h^{-1}, and desulfurized gas was supplied for about 6 hours instead of for about 2 hours previously. The amount of this absorbed in Test No. 009 is about three quarters of that in Test No. 011, in which the space velocity was 2,000 h^{-1} throughout, again showing that resistance to diffusion of H_2S into the sorbent is a significant factor. In Test No. 011, H_2S and SO_2 are initially high, as expected after a preceding regeneration. Figures 41 to 44 give baseline gas chromatograph readings for H_2S, SO_2, and COS in Test Nos. 006, 008, 009, and 011. H_2S and SO_2 measurements are generally in agreement with detector tube measurements (see Figures 37 to 40) in the vicinity of the calibration points: H_2S -- 3 ppm, COS -- 5 ppm, SO_2 -- 8 ppm. Nonlinearities in the gas chromatograph give rise to considerable departures between actual and measured values away from the calibration points. Calibration curves were constructed to make corrections in future tests.

Breakthrough of COS was found to occur roughly at the same time as H_2S breakthrough. The H_2S, COS, and SO_2 levels were corroborated at a few points by laboratory gas chromatograph analysis of grab samples of exit gas. These, together with values for methyl mercaptan, carbon disulfide and thiophene are shown in Table 1A. Thiophene was generally less than about 5 ppm.

Grab samples were also taken at the exit of the gasifier primary cyclone. The corresponding analyses of these are shown in Table 1B (5).

- Gas Analysis for Major Species

In sulfidation Test Nos. 006, 008, 009, and 011, gas to and from the desulfurization unit was analyzed in most cases for major species by Bendix on-line gas chromatographs. The measurements were averaged by ADACS statistical analysis and are tabulated in Tables 2, 3A, 3B, 3C, 4, and 5. Inlet gas was usually sampled at two points, one at the exit of the gasifier cyclone and the other at the immediate inlet to the desulfurization unit.

Exit gas was also analyzed during regeneration Test No. 010 and 012. These measurements are shown in Tables 6 and 7.

The above analyses for the unit exit gas were corroborated by laboratory gas chromatograph analysis of grab samples. These are shown in Table 8A. Corresponding analyses of grab samples taken at the exit of the gasifier primary cyclone are shown in Table 8B. Some of these samples are seen to contain excessive quantities of oxygen, which should not be more than about 1 vol. percent. An explanation may be that air in the sample bottles was not properly displaced.

Perusal of the gas analyses in the sulfidization tests indicate that the carbon monoxide shift conversion reaction is taking place. However, there is a substantial deficiency of total carbon oxides in the exit gas in relation to the inlet gas. This is presumably the result of being converted to carbon through the Boudouard reaction: $2CO \rightarrow CO_2 + C$.

This conclusion is substantiated by the analyses of the exit gas in the regeneration tests. It is seen to contain substantial quantities of CO_2 and CO which must have been formed from carbon remaining in the sorbent after the preceding sulfidation tests.

- Sorbent Regeneration

The two batches of zinc ferrite sorbent were regenerated in Test No. 007 and Test Nos. 010 and 012, respectively, using the same procedure as previously with a steam/air volume ratio of about 80/20 in the regeneration gas. Because the amount of sulfur which had been absorbed was relatively small, the temperature of the hot regeneration zone in Test No. 007 was initially too low (see Figure 8) and it was necessary to boost the air flow some two-fold (see Figure 9).

Regeneration Test Nos. 010 and 012 were normal, the temperature of the regeneration zone reaching about 1,500°F (see Figures 21 and 27). The reactor pressure drop during Test No. 007 (Figure 9) is uncertain because of apparent malfunctioning of the differential pressure gauge. The inlet pressure exceeded the instrument limit throughout except for a short initial period, indicating that the pressure drop was still high after the regeneration was over. Figures 22 and 28 show that the reactor pressure drop in the course of regeneration Tests Nos. 010 and

012 falls to a low value, comparable to that for fresh sorbent. All the regenerations were continued until the SO_2 content of the dry exit gas contained less than about 0.5 vol. percent. (see Figures 31, 34, and 36).

- Reactor Pressure Drop

In contrast to the series of tests during gasifier Run No. 101, the differential pressure across the reactor was directly measured in this series of tests. Figure 6 is a plot of this pressure drop during Test No. 006. The pressure is initially low, corresponding to a space velocity of about 2,000 h^{-1}. On increasing the space velocity to about 8,000 h^{-1}, after about an hour, the pressure rises to about 80 inches of water and, thereafter, gradually increases until it reaches and passes the limit of the indicator. On lowering the space velocity to ~ 2,000 h^{1} again, pressure falls to ~ 30 inches of H_2O and then gradually increases to ~ 60 inches of H_2O at the end of the test. The gradual rise of pressure drop, is attributed to the combined effect of particulate deposition and coke formation in the sorbent. Gas flow rates to the combustor and flare are plotted in Figure 7.

Possibly because of an inadequate regeneration in Test No. 007, the pressure drop in Test No. 008 (see Figure 12) was very high throughout at a level of 80 psi, corresponding to a space velocity of ~ 8,000 h^{-1}, and about 10 psi for a space velocity of ~ 2,000 h^{-1}. Gas flow rates to the combustor and flare are shown in Figure 13.

The pressure drops during Test Nos. 009 and 011 (see Figures 19 and 25) follow the same pattern as in Test No. 006 and 008. The fluctuations during Test No. 011, which lasted for a longer time, were caused by gas surges through the reactor when dumping condensate from the exit gas condenser.

- Reactor Temperatures

In Test Nos. 006 and 008, the average reactor temperatures exceed the furnace setting of 1,100°F by up to 100°F. This is attributed mainly to the exothermic "shift" reaction (see Figures 5 and 11). In the initial stages a mildly hot zone passes through the reactor which is thought to be caused by the rapid reduction of zinc ferrite to magnetite and zinc oxide.

Test Nos. 009 and 011 (see Figures 18 and 24) follow the same pattern though the furnace setting in this case was 1,000°F.

- Sorbent Characterization

The sulfidized sorbent after Test No. 008 was sampled on removal at points along the reactor length. The results are shown in Tables 9 and 10. At the reactor inlet most of the zinc oxide has been converted to sulfide, but a significant amount remains, probably as a result of the diffusional limitations discussed above. A substantial part of the sorbent's iron content is found to be in the form of cementite (iron carbide). At the reactor outlet the sorbent's zinc content is predominantly in the form of zinc oxide, as expected. All of the iron appears in the form of iron carbide and no magnetite is detected (see Table 9). The values in Table 9 represent total integrated intensities and, because of nonlinearities in their proportional relation to weight fraction, they should only be construed as showing rough amounts. These results from X-ray diffraction analyses are supported by the total sulfur and carbon analyses (see Table 10). Sulfur content is seen to fall from about 25 wt. percent at the reactor inlet to near zero at the reactor outlet. Carbon content varies from about 5 wt. percent at the inlet to about 8 wt. percent at the outlet. This can be explained by the fact that, at the inlet, a good part of the iron in the sorbent is in the form of pyrrhotite (FeS_x), whereas at the outlet, no pyrrhotite is detected.

The surface area and pore volume of the sorbent, particularly towards the outlet, are significantly greater than for the fresh material (see Table 10). This is attributed to the increased porosity resulting from reduction of magnetite in the sorbent to iron and iron carbide. The increased porosity is accompanied by a significantly lower crush strength, which may be the main reason for the large amount of fines formed in the removed sorbent. The fines which passed through a No. 12 screen amounted to about a third of the total sorbent.

Characterization of the sorbent, after regeneration in Test No. 012, is shown in Tables 11 and 12. The mineral analysis (see Table 11) indicates that the sorbent is now predominantly in the form of franklinite and magnetite with minor amounts of hematite and zincite present. There appears to be some enrichment of zincite towards the top of the reactor. Analysis for total carbon and sulfur (see Table 12) shows that virtually all of the carbon, presumed to be present before the regeneration, has been burned out. However, some residual sulfur still remains, reaching up to about 2 wt. percent at the bottom of the reactor, which was the inlet during sulfidation. Most of this residual sulfur is seen to be in the form of sulfate.

The surface area and pore volume of the regenerated sorbent a little less than for fresh material. Crush strength, on average, is much the same as for fresh sorbent, though there is a significant variation with bed depth, being less at the top of the reactor where the sorbent had undergone a smaller conversion to sulfide. In contrast to Test No. 008, only about 2.5 wt. percent of fines, passing through a No. 12 screen, were found in the removed sorbent.

- Particulate Analysis

Samples of inlet and outlet gas were taken during the series of tests to characterize the particulate content (6) as described previously. The results are summarized in Table 13. Because of the short test durations it did not prove possible to sample both inlet and outlet gas in a given test. However, one inlet sample was taken in each of Test Nos. 006 and 008 (Tables 14 and 15), and one outlet sample in each of Test Nos. 009 and 011 (Tables 16 and 17). It should be recalled that during Test Nos. 006 and 008 Arkwright coal was fed to the gasifier. Table 18 shows the size distribution of a random sample of particles removed by the primary gasifier cyclone at this time. Table 19 shows the size distribution of a similar particle sample taken when the gasifier was fed with Blacksville coal briquettes at the time of Test Nos. 009 and 011. It is noteworthy that the median particle diameter of the Arkwright dust is substantially greater than that of the Blacksville briquette dust, 64 µm compared to 38 µm. Ultimate analyses of the coals are given in Table 20.

The particle loadings in the hot gas stream passing through the hot gas desulfurization unit are seen to be in the range 0.3 to 0.8 g/Nm^3 with a smaller loading found in exit gas. Median particle size falls in the range 20 µm to 27 µm. It is not possible to say whether exit particle size is significantly different from entering particle size when taking into account the differing coal feeds to the gasifier. Chemical analyses (ultimate) of the particulate loadings (see Table 13) show that, though exiting particles contain a large amount of carbon, indicating that they are mostly the same as the entering particles, their larger ash content suggests that they contain some attrited sorbent bed particulate.

- Condensate Analysis

In another attempt to characterize the hot gas passing through the desulfurization unit for minor trace constituents, a number of condensate samples were taken and analyzed (5). Table 21 shows the analysis of a composite sample of water condensate from Test Nos. 006 and 008. The corresponding exit gas composition is calculated from a knowledge of the gas flow through the unit (see Figures 30 and 32).

Table 22 gives the ultimate analysis of a composite sample of the associated tar condensate. A number of samples of the tar condensate were taken from the S4 sample point at the gasifier exit. The analyses of these are shown in Table 23.

Condensate was collected during Test Nos. 006 and 008 at the inlet of the unit as part of the particulate sampling. Analyses of the water fractions of these condensates are shown in Table 24. These are averaged and translated to a gas composition for comparison with the analysis of the composite sample from Test Nos. 006 and 008 in Table 25. The comparison shows that roughly half of the halides and a fifth of the ammonia in the gas are removed, which accords with previous results. The molar proportions of ammonia and halide are approximately the same suggesting that the mechanism of ammonia removal may be through the intermediate formation of an amminochloride. The other metals and ions are present in relatively small amounts so that it is not possible to say with certainty to what extent their concentrations have changed. However, it seems that as much as three quarters of the alkali and alkaline earth metals are removed by the hot gas desulfurization process.

The above conclusions are corroborated by the analysis of a composite sample of condensate from regeneration Test No. 007 shown in Table 26. Corresponding analyses of condensate from regeneration Test Nos. 010

and 012 are shown in Table 27. These also exhibit an enrichment in the same constituents. The analyses of the aqueous condensates from the sulfidation tests are supported by additional analyses by IHI Kemron of spot samples taken during Test Nos. 006 and 011. These are shown in Table 28. The constituents measured vary somewhat from the ones measured at METC.

Conclusions

A second series of tests to evaluate the performance of zinc ferrite, hot gas desulfurization sorbent in a sidestream of METC's 24 t/d fixed-bed gasifier was carried out during Run No. 102. The general performance of the sorbent was similar to that in the previous series of tests during gasifier Run No. 101 with low molecular weight sulfur compounds removed to less than about 10 ppm.

It was found that, at the higher space velocities necessary to provide gas to the METC combustion test unit, diffusional resistance resulted in the sorbent picking up less than half the anticipated amount of sulfur. This diffusional resistance may be accentuated by the conversion of magnetite in the sorbent to iron carbides.

The formation of iron carbides, resulting from the high CO/CO_2 ratio in the gas which was desulfurized, appears to weaken the sorbent considerably and, upon removal in the sulfidized form, it contained a large quantity of fines.

On these grounds, it appears advisable to operate the desulfurization process at conditions of gas composition and temperature such that iron in the sorbent is not reduced from magnetite to iron and iron carbide.

Regeneration of sorbent containing iron carbides produced a tail gas containing reduced sulfur compounds, such as H_2S and COS, and also hydrogen and carbon monoxide. Treatment of this stream to fix the sulfur compounds would probably require its incineration.

The particulate loading of the gas stream to the desulfurization unit was again of the order of 1 g/Nm^3 and median particle diameter in the range 20 µm to 25 µm. This combination of particulate loading and size resulted in an unacceptable reactor pressure drop, high enough to lift the sorbent bed (operation was in an upward flow mode.)

Future tests were planned to investigate the reactor pressure drop more closely, including the effects on it of space velocity, particle loading and size, and iron carbide and coke formation.

As in previous tests, the sorbent was found to remove significant amounts of other minor and trace constituents from the hot gas, in addition to sulfur. The most noticeable of these were halides, ammonia, and alkali and alkaline earth metals. The mechanism by which ammonia is removed is not clear at present.

References

1. Grindley, T., "Hot Gas Desulfurization Sidestream Test Unit, Fixed-Bed Gasifier Run No. 101," METC, IR No. 1846 (1984).

2. "Integrated Test Plan for METC Fixed-Bed Gasifier and Cleanup System Test Run No. 102," EG&G, Morgantown, September 1983. DOE/MC/14521-1467.

3. Pater, K., Preliminary Test Summary -- Run No. 102 (FN), IR No. 1808, March 1984, METC; Process Summary Report Test Run 102 METC Fixed-Bed Gasifier and Full-Flow Cleanup System, IR No. 1983, September 1984, METC.

4. Waltermire, D. D., "METC In-House Combustion Studies on Alternate Fuels for Heat Engines Applications," First Annual Heat Engines Contractors' Meeting (1984), METC.

5. Romanosky, R. R., and J. J. Kovach, "Analytical Data -- Producer Run No. 102," IR No. 1970, August 1984, METC.

6. Bagnell, R. D., "Particle Sampling in the Hot Gas Stream During Gasifier Run No. 102," IR No. 1757, December 1983, METC.

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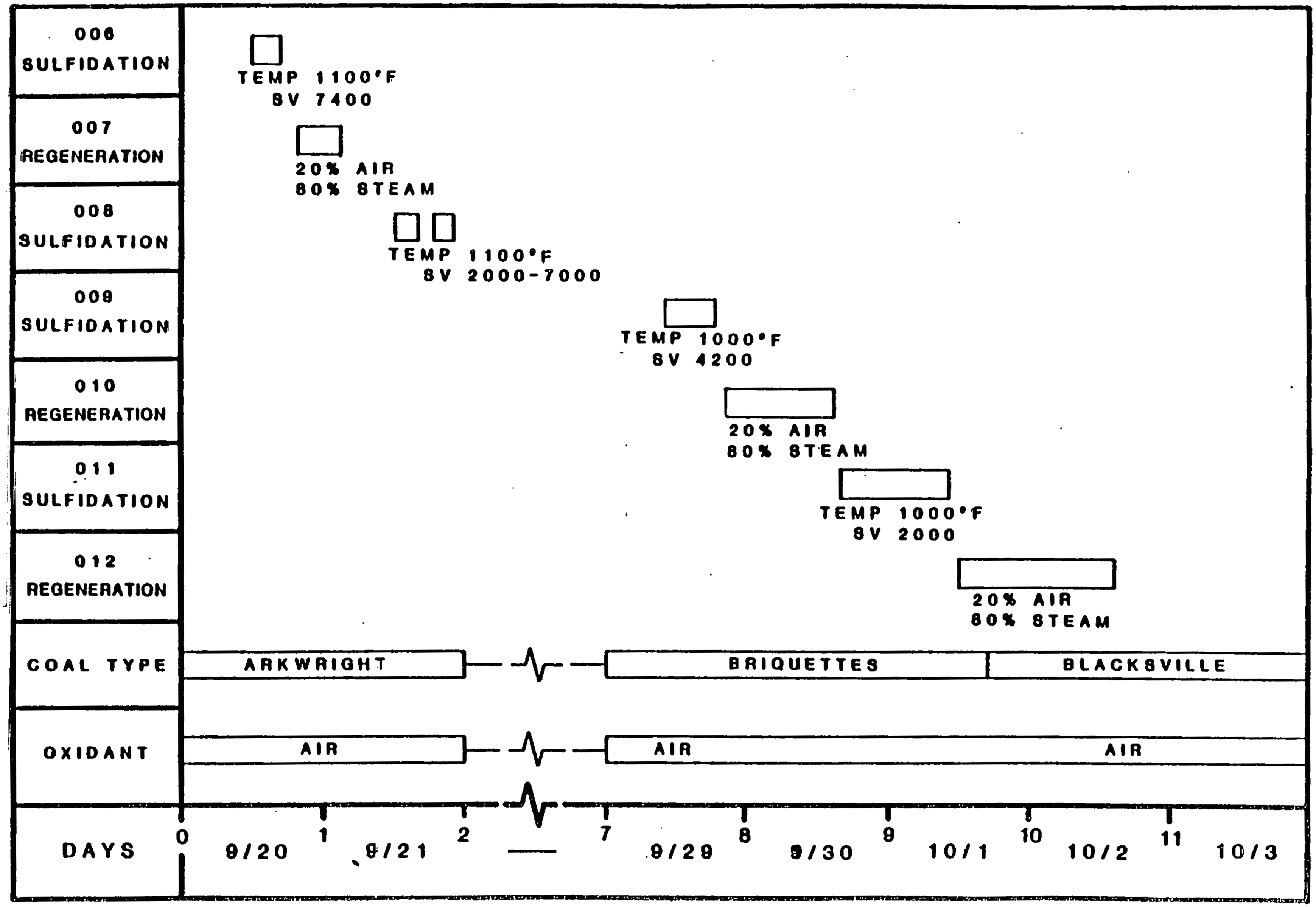
ADDENDUM TO APPENDIX F

The Baseline G.C. on-line H_2S, COS, and SO_2 readings as plotted are correct only for the points: H_2S: 3 ppm; COS: 1.7 ppm; and SO_2: 5.1 ppm. Calibration curves are available for correction. Corrected plots are given in Volume 1.

Refer to letter to T. Grindley from E. E. Gorski and P. Johnson, EG&G Washington Analytical Services Center, Inc. January 30, 1986. Subject: DOE-METC/EG&G Contract No. DE-AC21-85MC21353; WBS No. 9KEX-10. HGD Project: Baseline GC/Integrator Conversion Curves.

FIGURE 1



SCHEDULE OF EVENTS

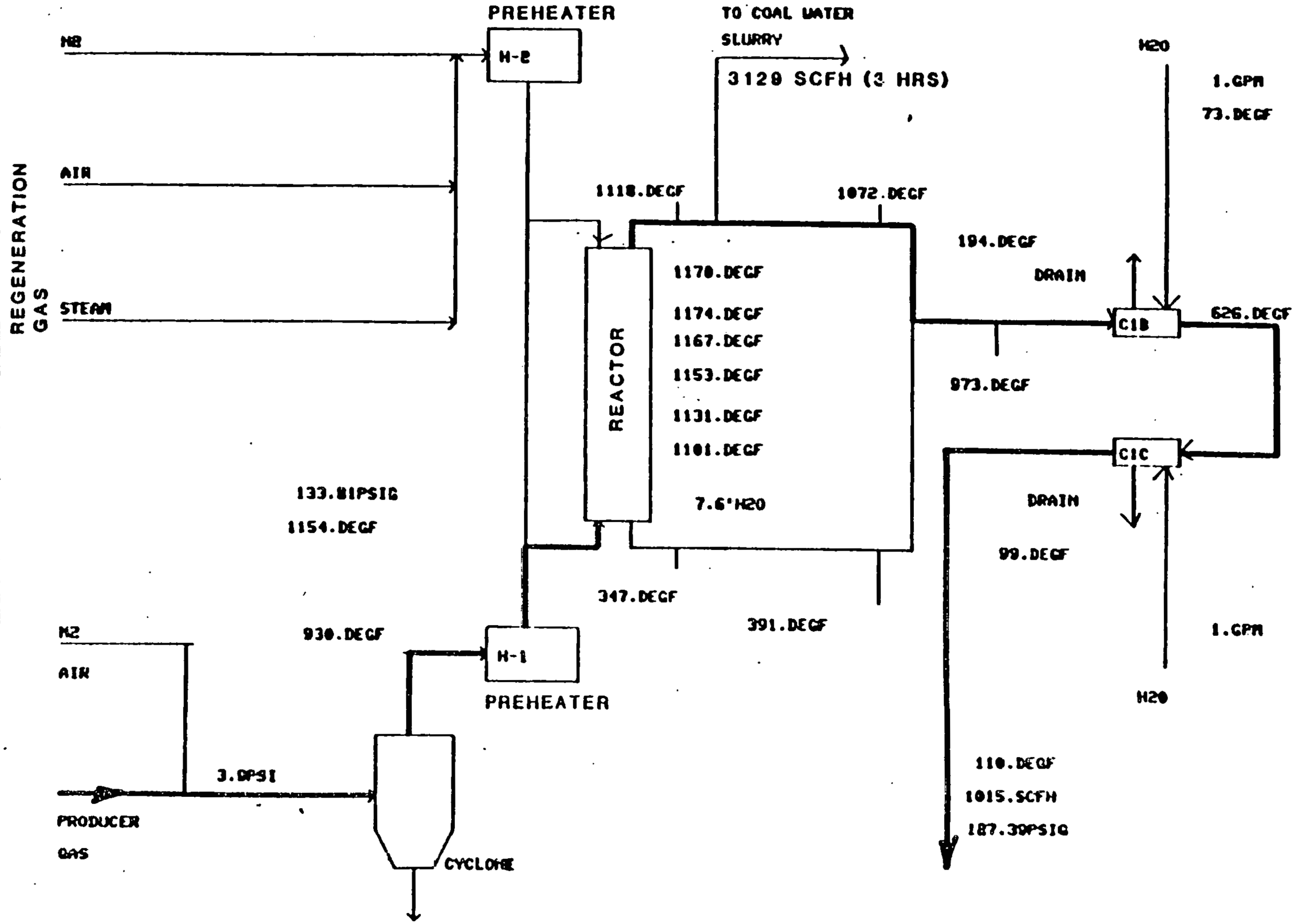
HOT GAS DESULFURIZATION SIDESTREAM TESTING 1983

GASIFIER RUN 102

FIGURE 2

TEST 006

AVERAGE OPERATING PARAMETERS

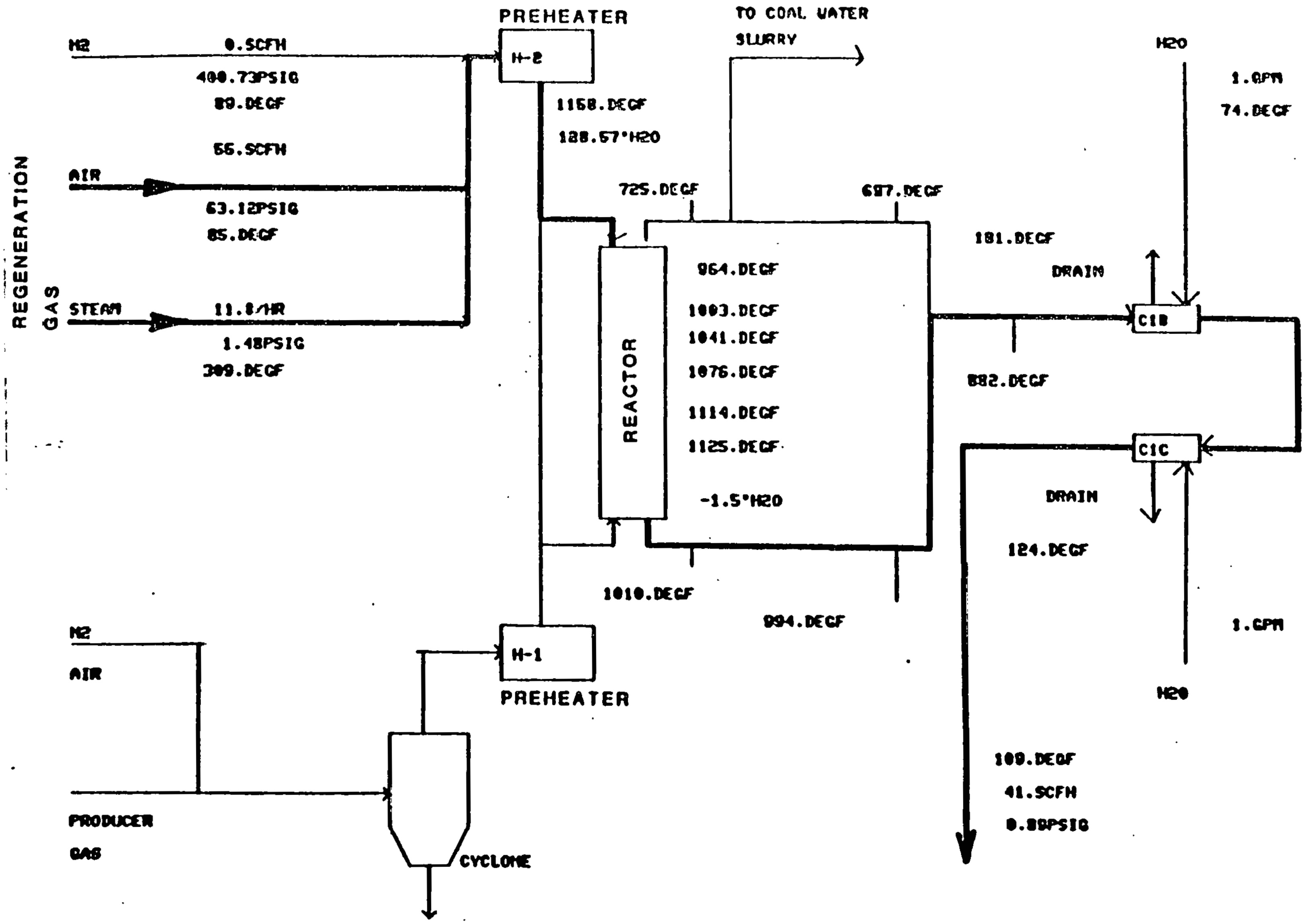


AVERAGE(S) FOR PERIOD FROM: 12: 2:18 9/20/1983 TO 18: 9:38 9/20/1983

FIGURE 3

TEST 007

AVERAGE OPERATING PARAMETERS

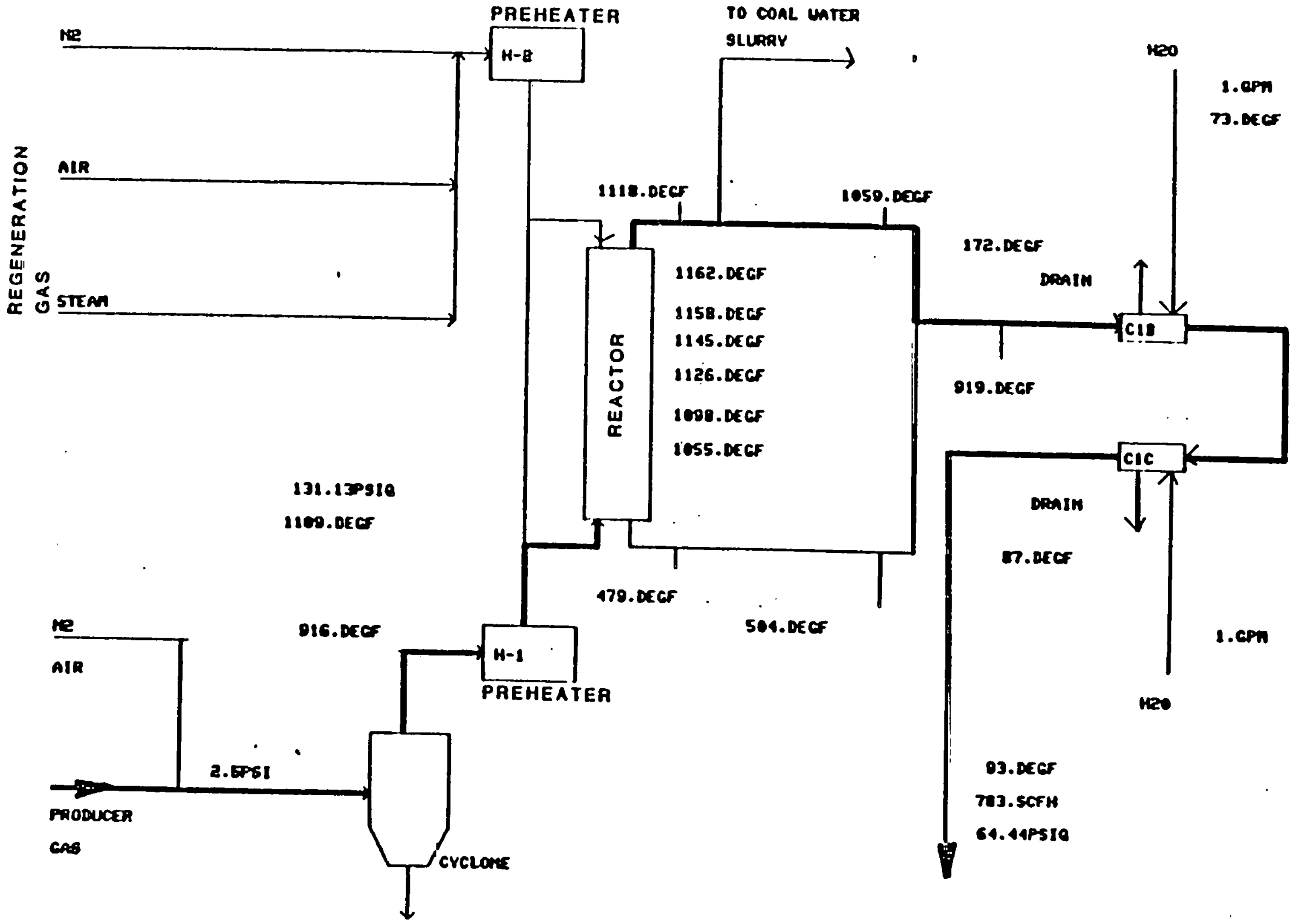


AVERAGE(S) FOR PERIOD FROM: 19:30:14 9/20/1983 TO 8:49:55 9/21/1983

FIGURE 4A

TEST 008 A

AVERAGE OPERATING PARAMETERS

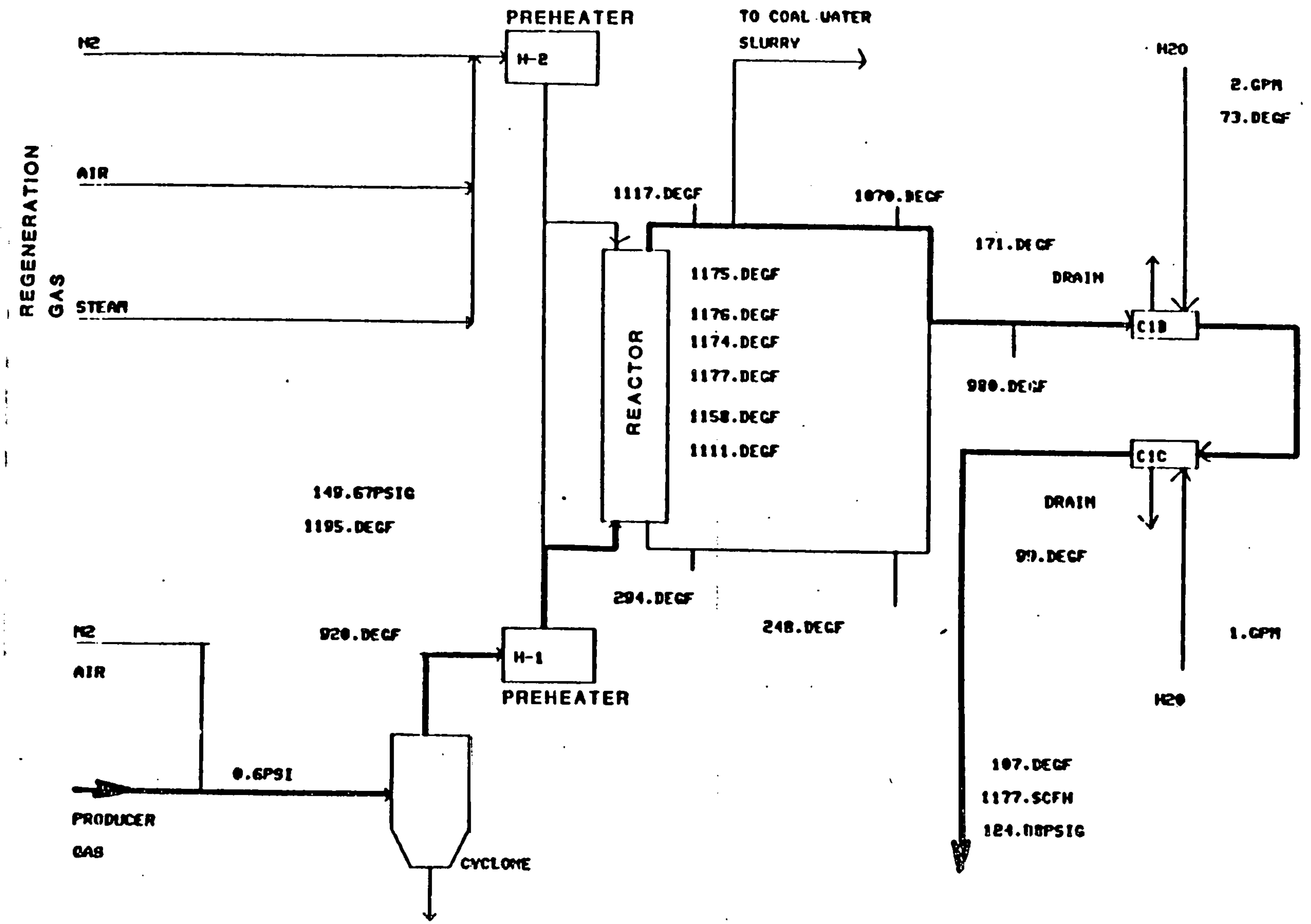


AVERAGE(S) FOR PERIOD FROM: 18:17:25 9/21/1983 TO 14: 0:28 9/21/1983

FIGURE 4B

TES: 008 B

AVERAGE OPERATING PARAMETERS

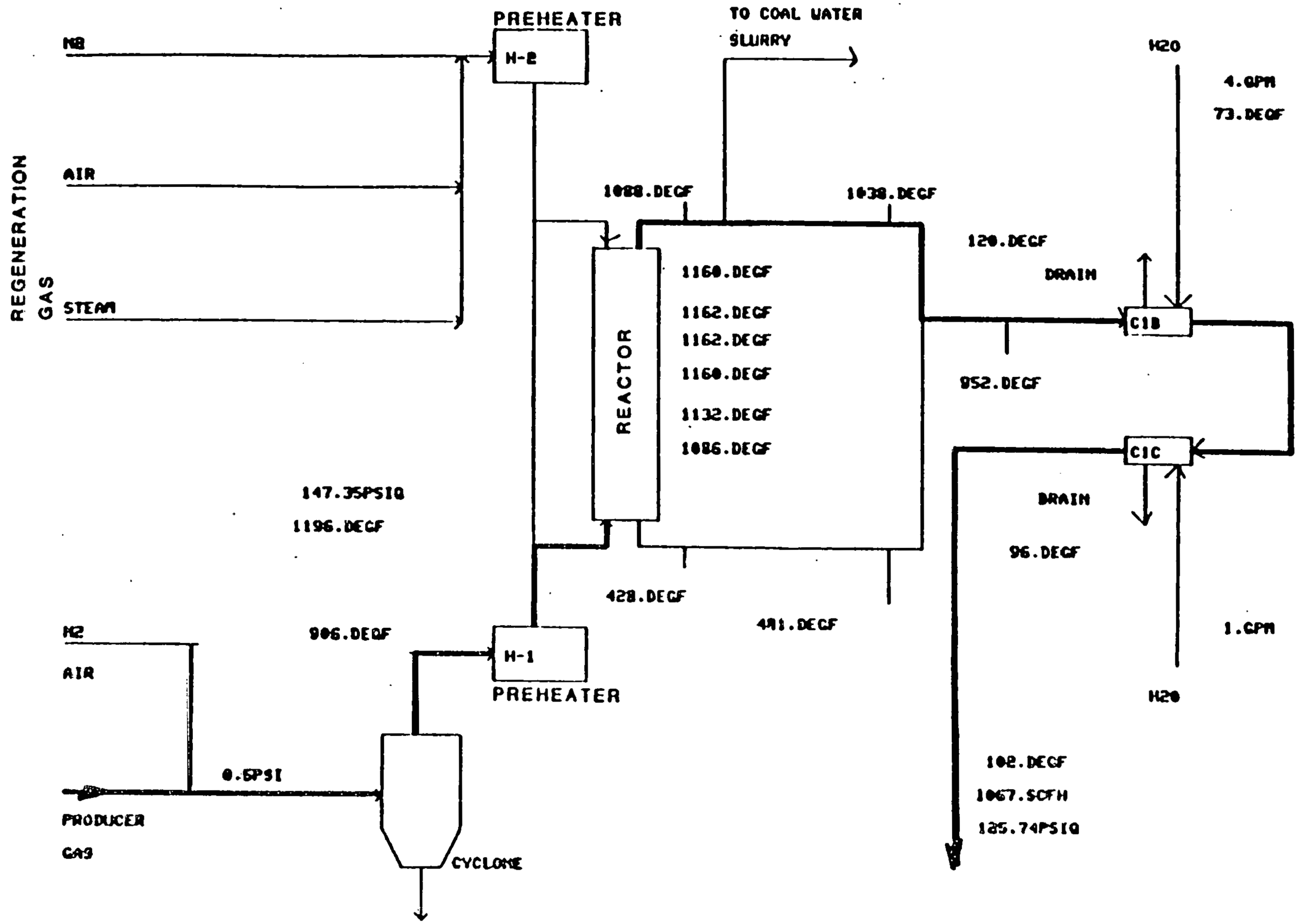


[illegible] PERIOD FROM: 14:46:30 9/21/1983 TO 15:52:35 9/21/1983

FIGURE 4C

TEST 008 C

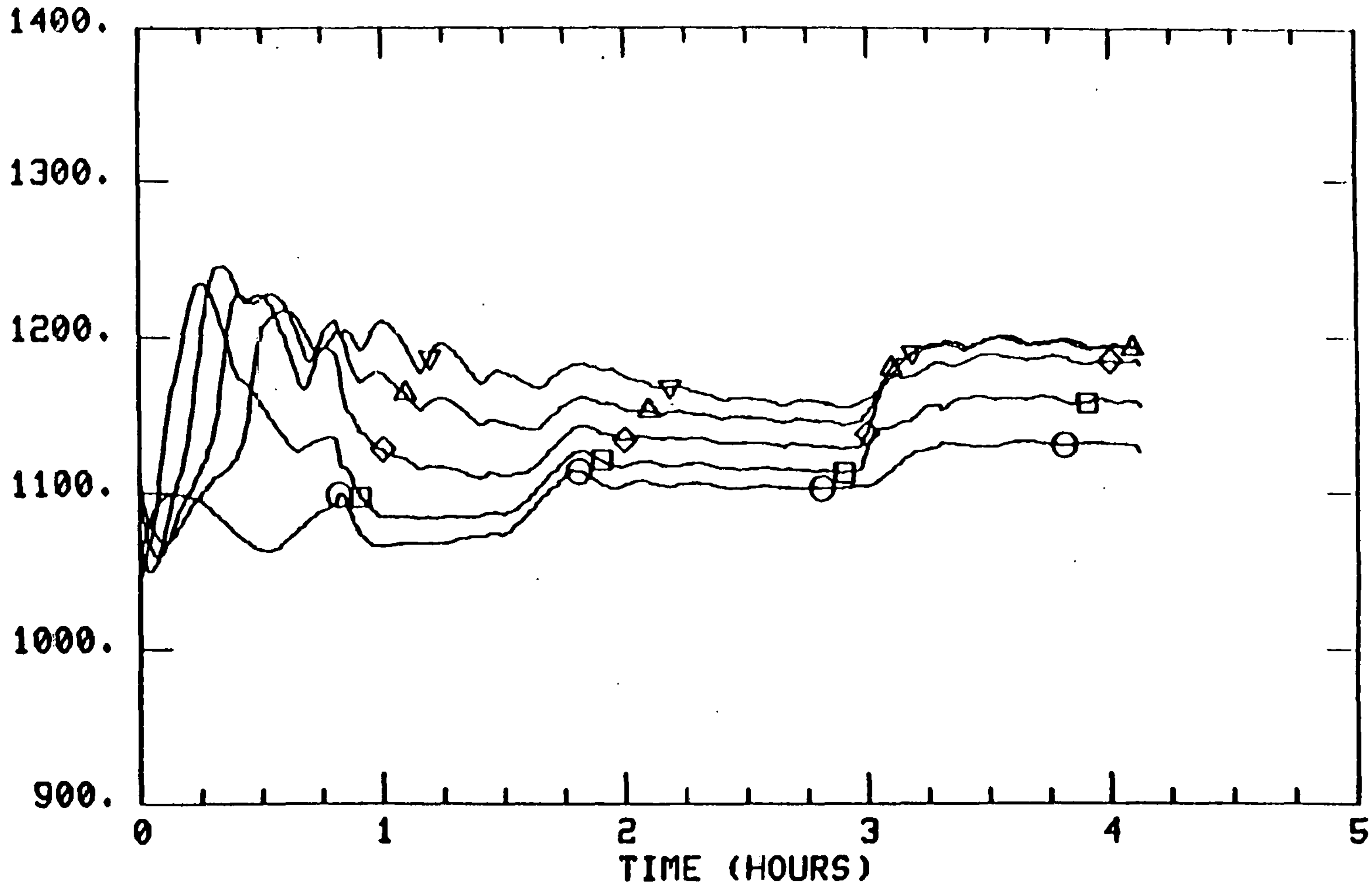
AVERAGE OPERATING PARAMETERS



AVERAGE(S) FOR PERIOD FROM: 18:29:33 9/21/1983 TO 20:28:07 9/21/1983

FIGURE 5

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN -	FREQ	MAX -	FREQ
○	851	DEGF	ABOSRBER 5° FROM BOTTOM	498	1101.	22.21	1059.	1	1134.	8
□	852	DEGF	ABSORBER 15° FROM BOTTOM	498	1131.	34.00	1045.	1	1238.	2
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	496	1153.	38.08	1049.	2	1247.	1
△	854	DEGF	ABSORBER 35° FROM BOTTOM	498	1167.	33.07	1059.	2	1228.	3
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	496	1174.	32.04	1068.	2	1218.	3



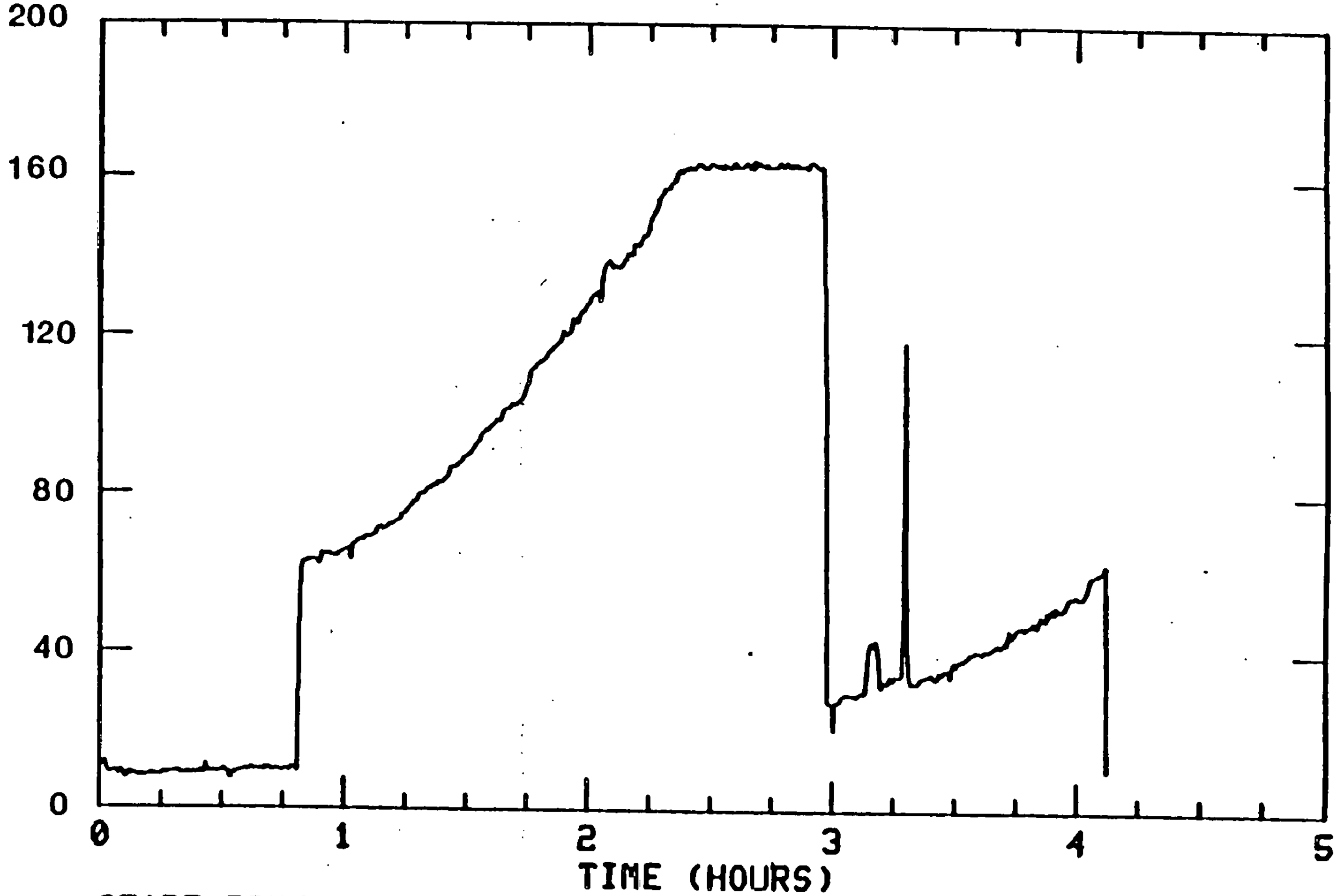
START TIME OF PLOT 12: 2: 0 9/20/1983
STOP TIME OF PLOT 16:10: 0 9/20/1983

TEST 006

REACTOR TEMPERATURES

FIGURE 6

SLOT UNITS DESCRIPTION
804 "HBO ABSORBER DIFFERENCE PRESSURE



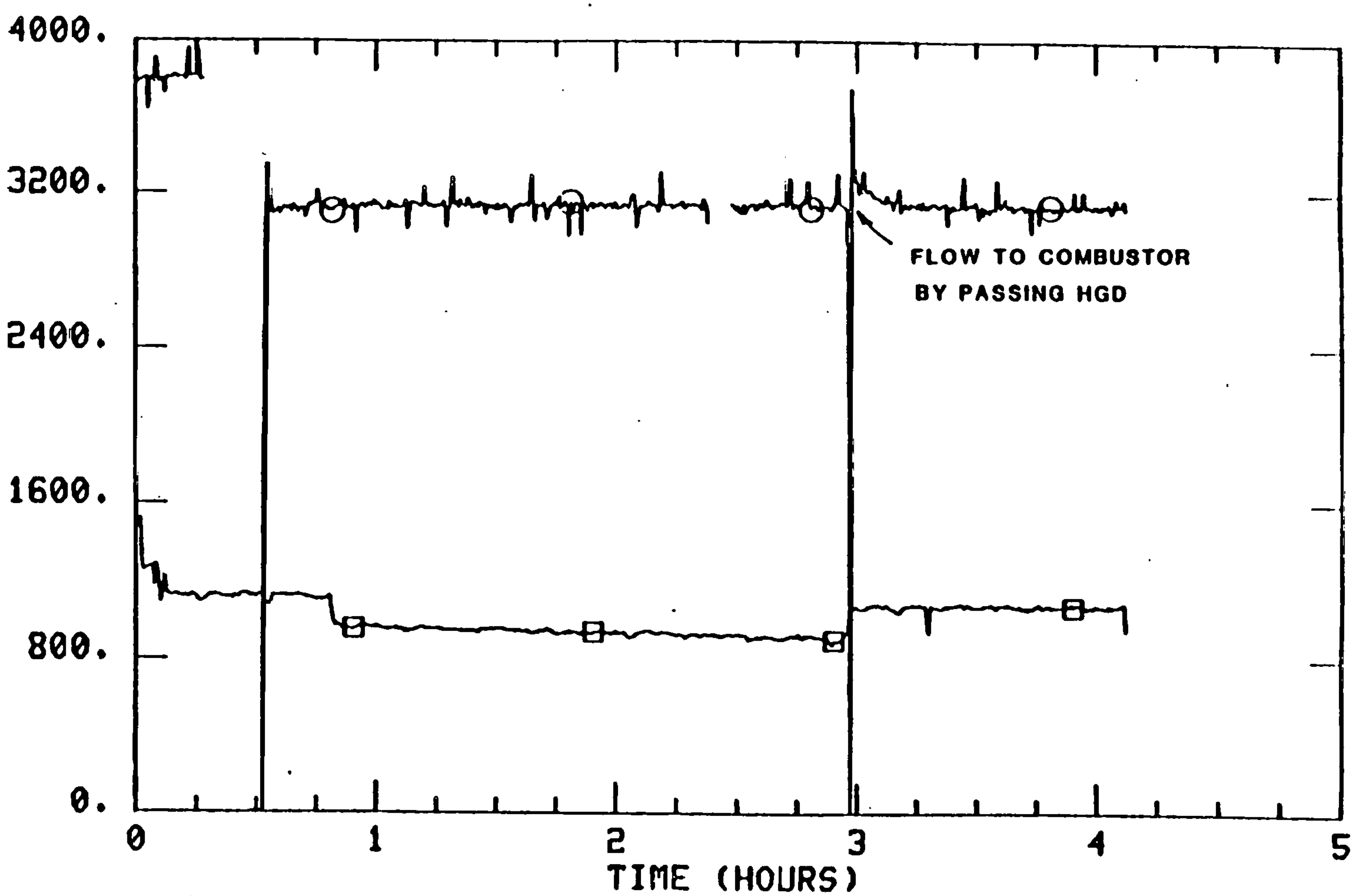
START TIME OF PLOT 12: 2: 0 9/20/1983
STOP TIME OF PLOT 16:10: 0 9/20/1983

TEST 006

REACTOR PRESSURE DROP

FIGURE 7

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	4017	SCFH	PRODUCER GAS FLOW TO COMBUSTOR	463	3138.	459.5	0.0000E+00	8	3974.	1
□	3750	SCFH	PROD. GAS OUT OF SYSTEM	496	1015.	93.39	876.0	1	1527.	1



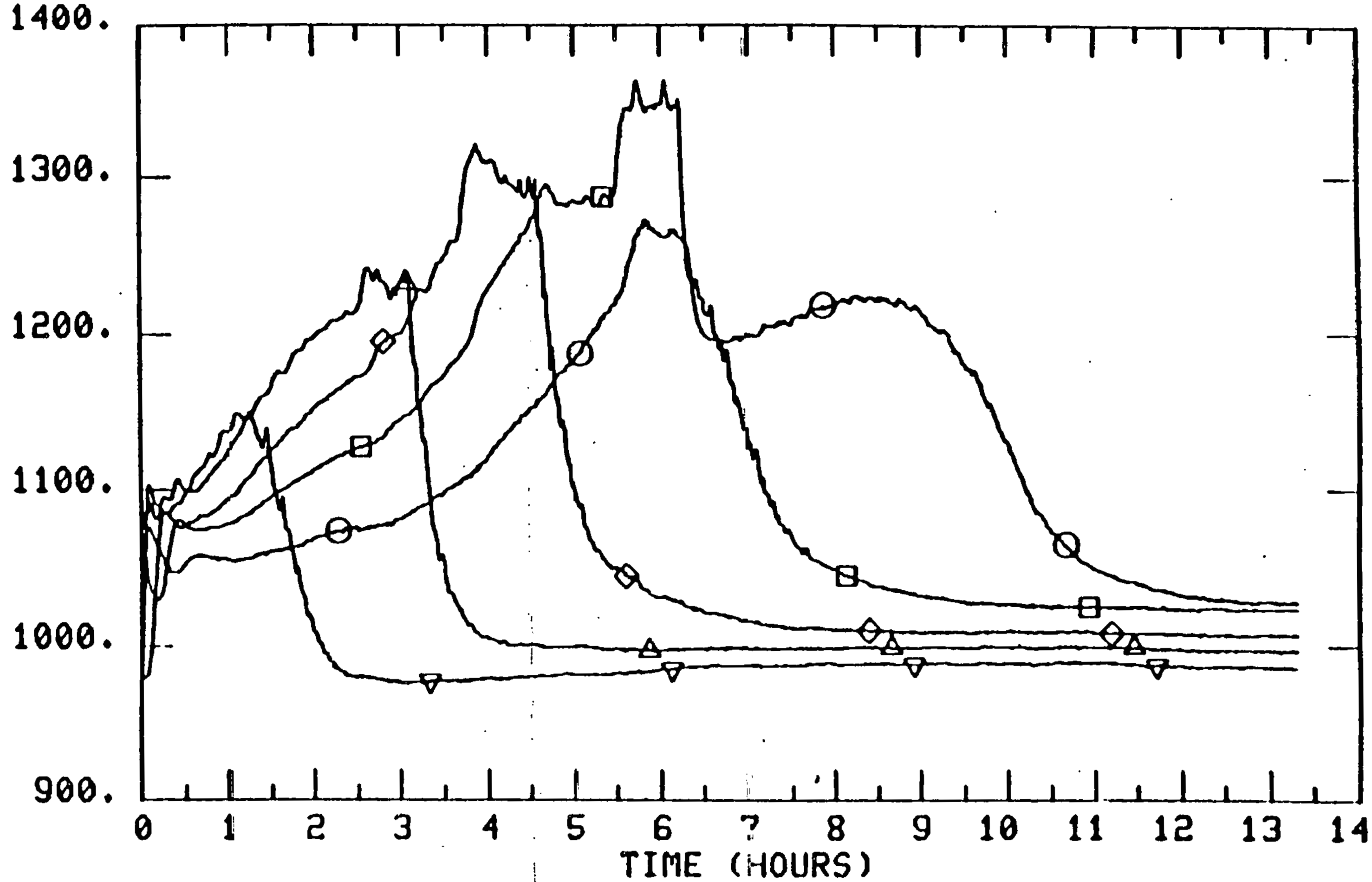
START TIME OF PLOT 12: 2: 0 9/20/1983
STOP TIME OF PLOT 16:10: 0 9/20/1983

TEST 006
EXIT FLOWS

-496-

FIGURE 8

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
○	851	DEGF	ABOSRBER 5° FROM BOTTOM	1595	1125.	78.06	1027.	11	1273.	2
□	852	DEGF	ABSORBER 15° FROM BOTTOM	1595	1114.	100.8	1023.	1	1365.	1
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	1595	1076.	92.93	979.5	2	1322.	1
△	854	DEGF	ABSORBER 35° FROM BOTTOM	1595	1041.	75.61	984.5	1	1243.	5
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	1595	1003.	44.50	975.4	5	1151.	1



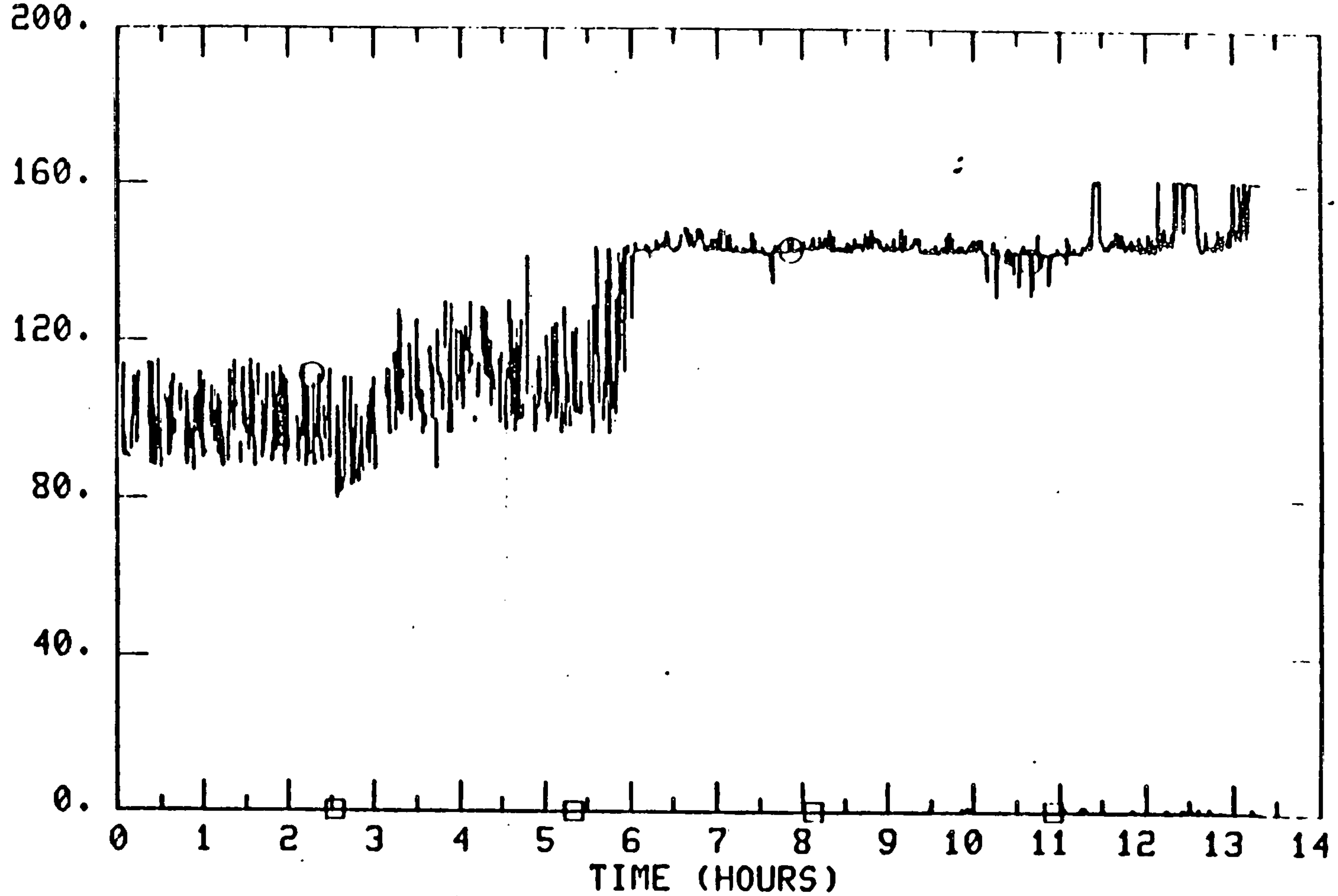
START TIME OF PLOT 19:30: 0 9/20/1983
STOP TIME OF PLOT 8:50: 0 9/21/1983

TEST 007

REACTOR TEMPERATURES

FIGURE 9

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	302	"H2O	ABSORBER INLET TOP PRESSURE	1377	131.0	20.99	80.14	1	161.9	1
□	294	"H2O	ABSORBER DIFFERENCE PRESSURE	1595	-1.457	0.3722	-1.631	1	1.383	1



START TIME OF PLOT 19:30: 0 9/20/1983
STOP TIME OF PLOT 8:50: 0 9/21/1983

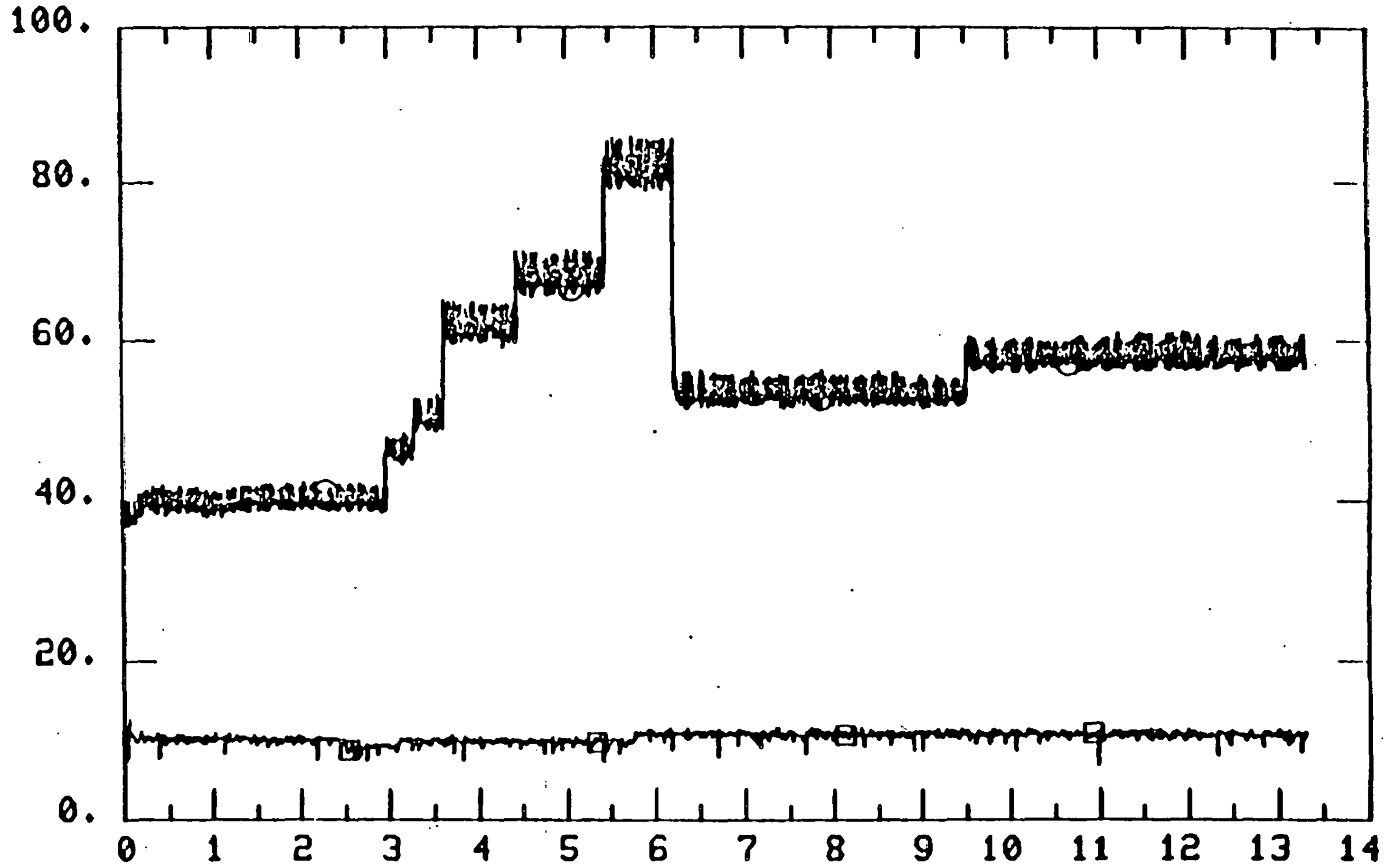
TEST 007

REGENERATION

REACTOR PRESSURE DROP AND REACTOR INLET PRESSURE

FIGURE 10

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	3756	SCFH	AIR FLOW LOW RANGE	1595	55.24	10.84	38.88	1	86.02	1
□	3754	#/HR	STEAM FLOW	1595	10.54	0.6721	8.977	1	12.94	1



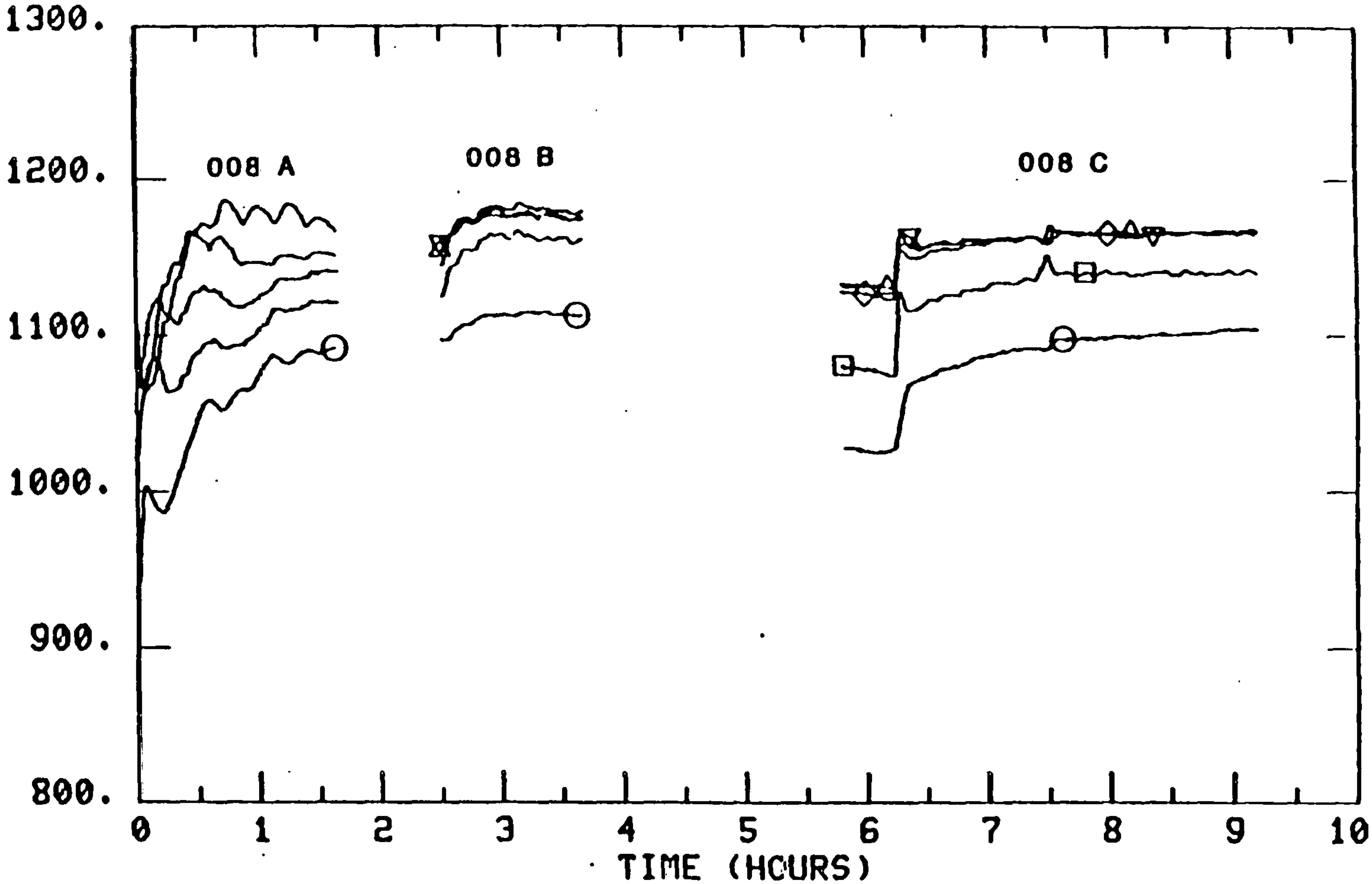
START TIME OF PLOT 19:30: 0 9/20/1983
STOP TIME OF PLOT 8:50: 0 9/21/1983

TEST 007

STEAM AND AIR FLOWS

FIGURE 11

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	BS1	DEGF	ABOSRBER 5° FROM BOTTOM	748	1081.	32.09	940.7	1	1116.	6
□	BS2	DEGF	ABSORBER 15° FROM BOTTOM	753	1126.	28.16	1023.	1	1168.	1
◇	BS3	DEGF	ABSORBER 25° FROM BOTTOM	753	1153.	22.16	1067.	1	1185.	5
△	BS4	DEGF	ABSORBER 35° FROM BOTTOM	751	1159.	17.85	1067.	1	1179.	2
▽	BS5	DEGF	ABSORBER 45° FROM BOTTOM	749	1162.	21.18	1065.	1	1188.	3



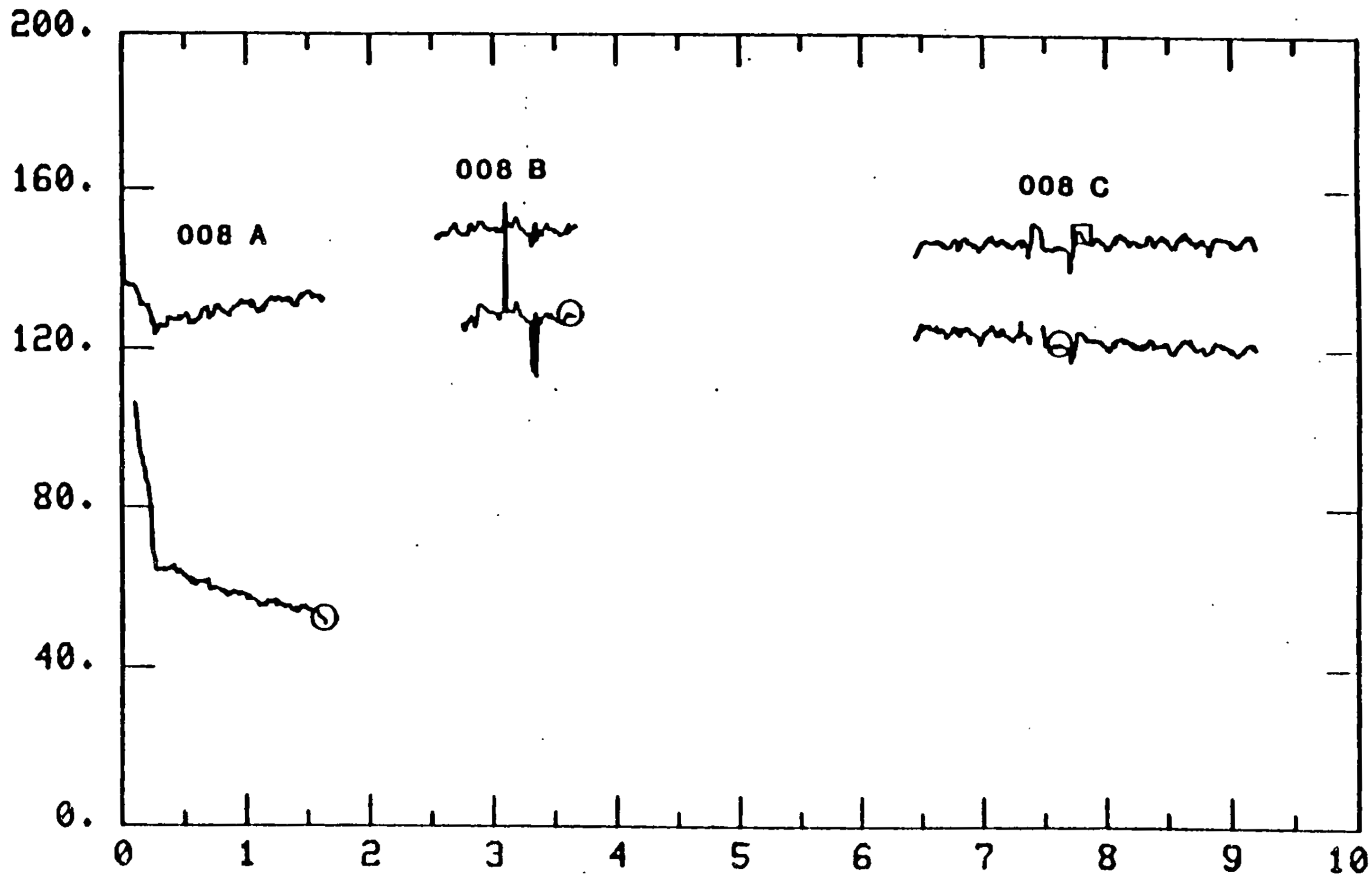
START TIME OF PLOT 12:17: 0 9/21/1983
STOP TIME OF PLOT 21:30: 0 9/21/1983

TEST 008

REACTOR TEMPERATURES

FIGURE 12

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	289	PSIG	GAS OUT OF SYSTEM	618	105.5	29.39	50.90	1	153.3	1
□	893	PSIG	ABSORBER INLET BOTTOM	665	143.8	8.219	124.0	1	156.8	1



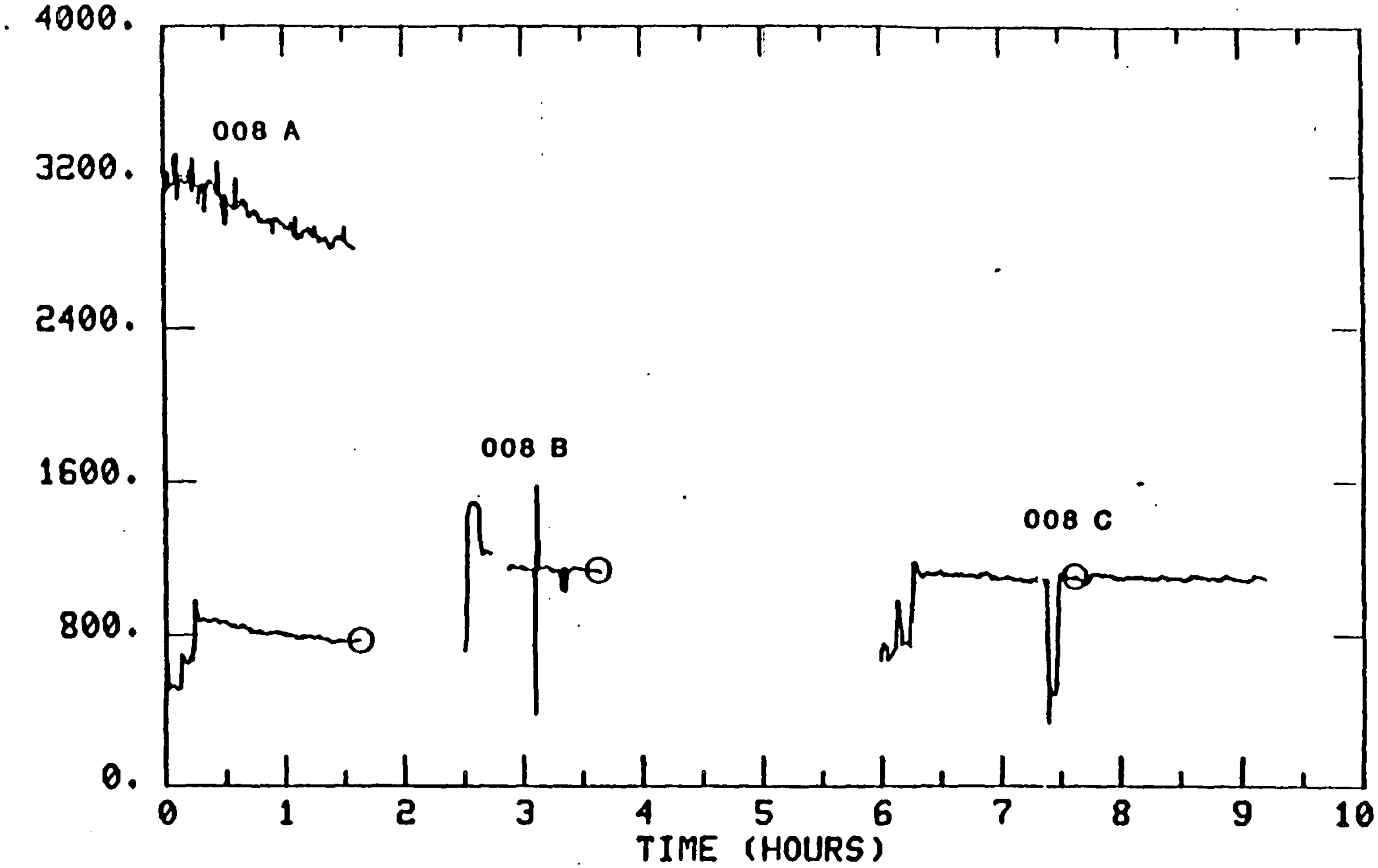
START TIME OF PLOT 12:17: 0 9/21/1983
STOP TIME OF PLOT 21:30: 0 9/21/1983

TEST 008

INLET AND EXIT PRESSURES

FIGURE 13

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN	FREQ	MAX	FREQ
O	3750	SCFH	PROD. GAS OUT OF SYSTEM	700	1000.	191.2	336.4	1	1581.	1
□	4017	SCFH	PRODUCER GAS FLOW TO COMBUSTOR	192	3010.	124.6	2818.	1	3321.	1



START TIME OF PLOT 12:17: 0 9/21/1983
STOP TIME OF PLOT 21:30: 0 9/21/1983

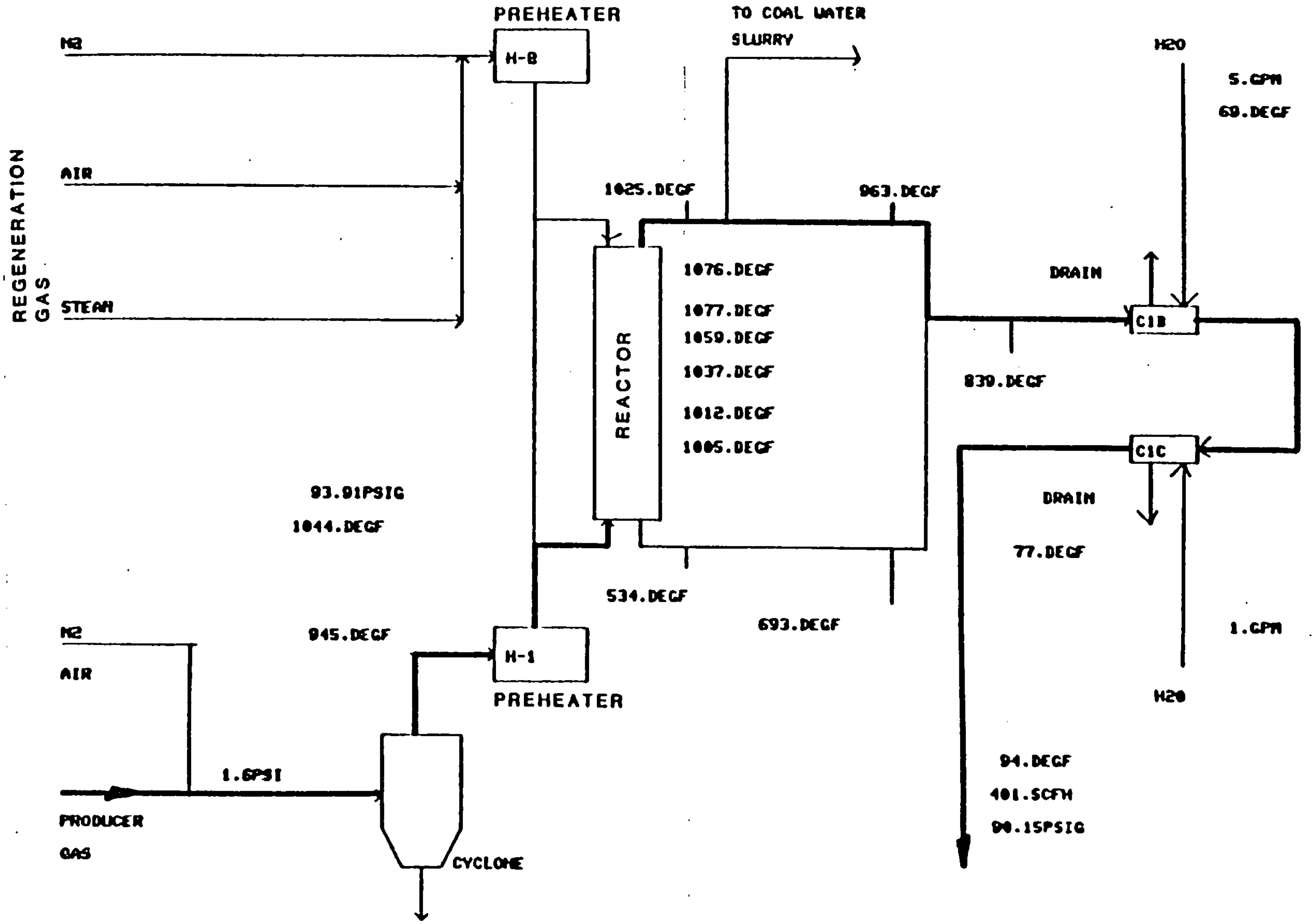
TEST 008

EXIT FLOWS

FIGURE 14

TEST 009

AVERAGE OPERATING PARAMETERS

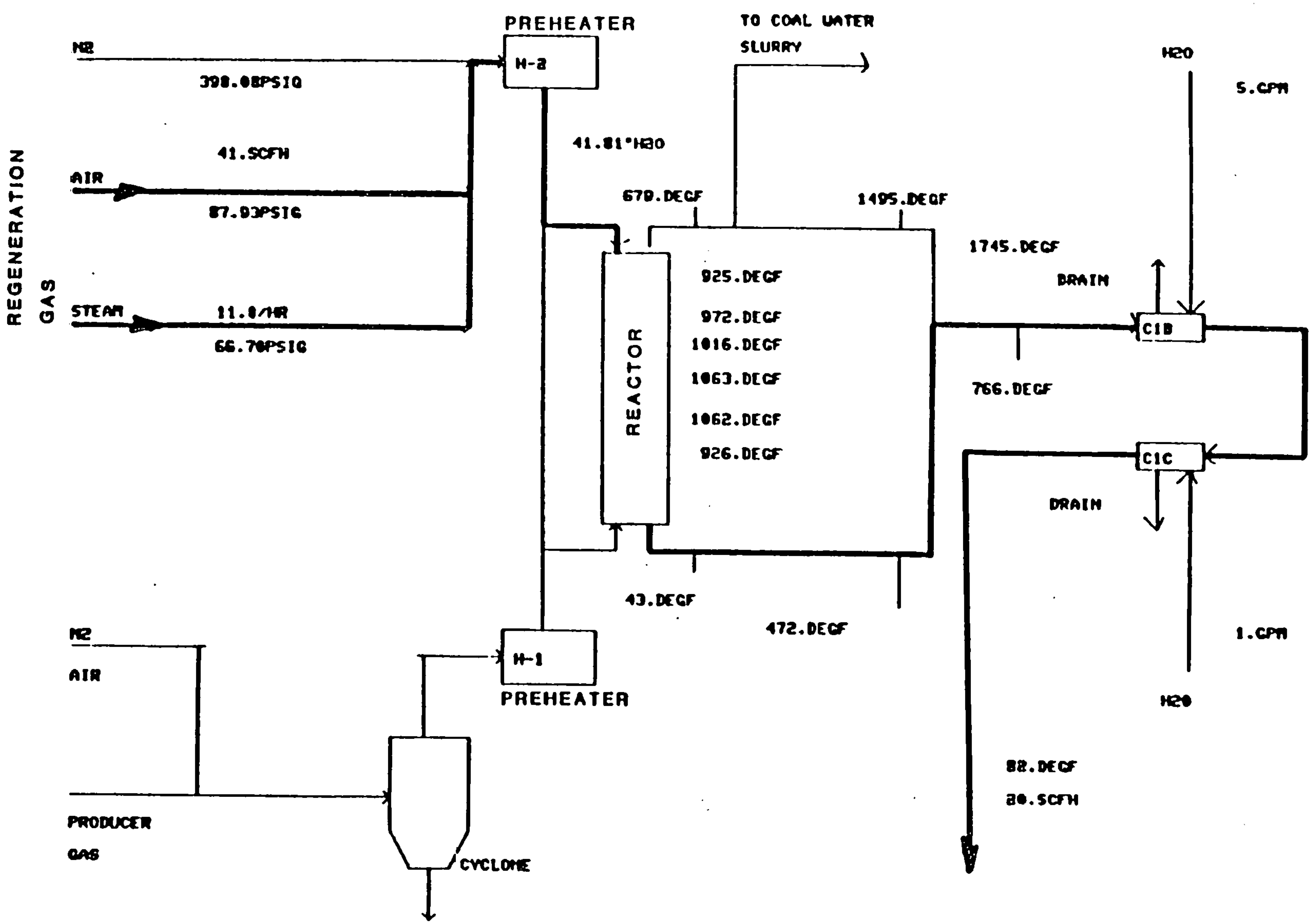


AVERAGE(S) FOR PERIOD FROM: 11:45:20 9/29/1983 TO 19:40:37 9/29/1983

FIGURE 15

TEST 010

AVERAGE OPERATING PARAMETERS

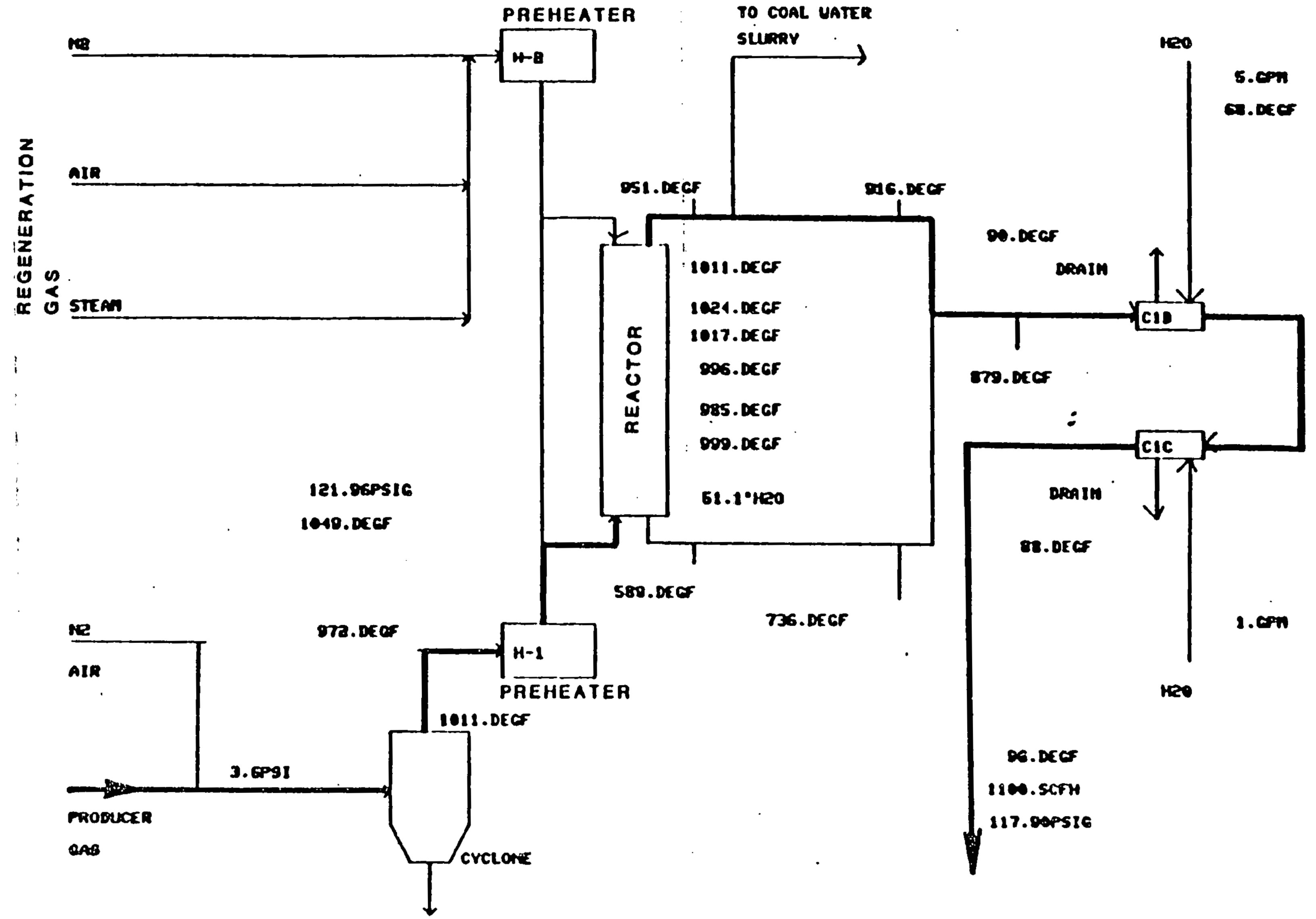


AVERAGE(S) FOR PERIOD FROM: 21:40: 1 9/29/1983 TO 15:29:59 9/30/1983

FIGURE 16

TEST 011

AVERAGE OPERATING PARAMETERS

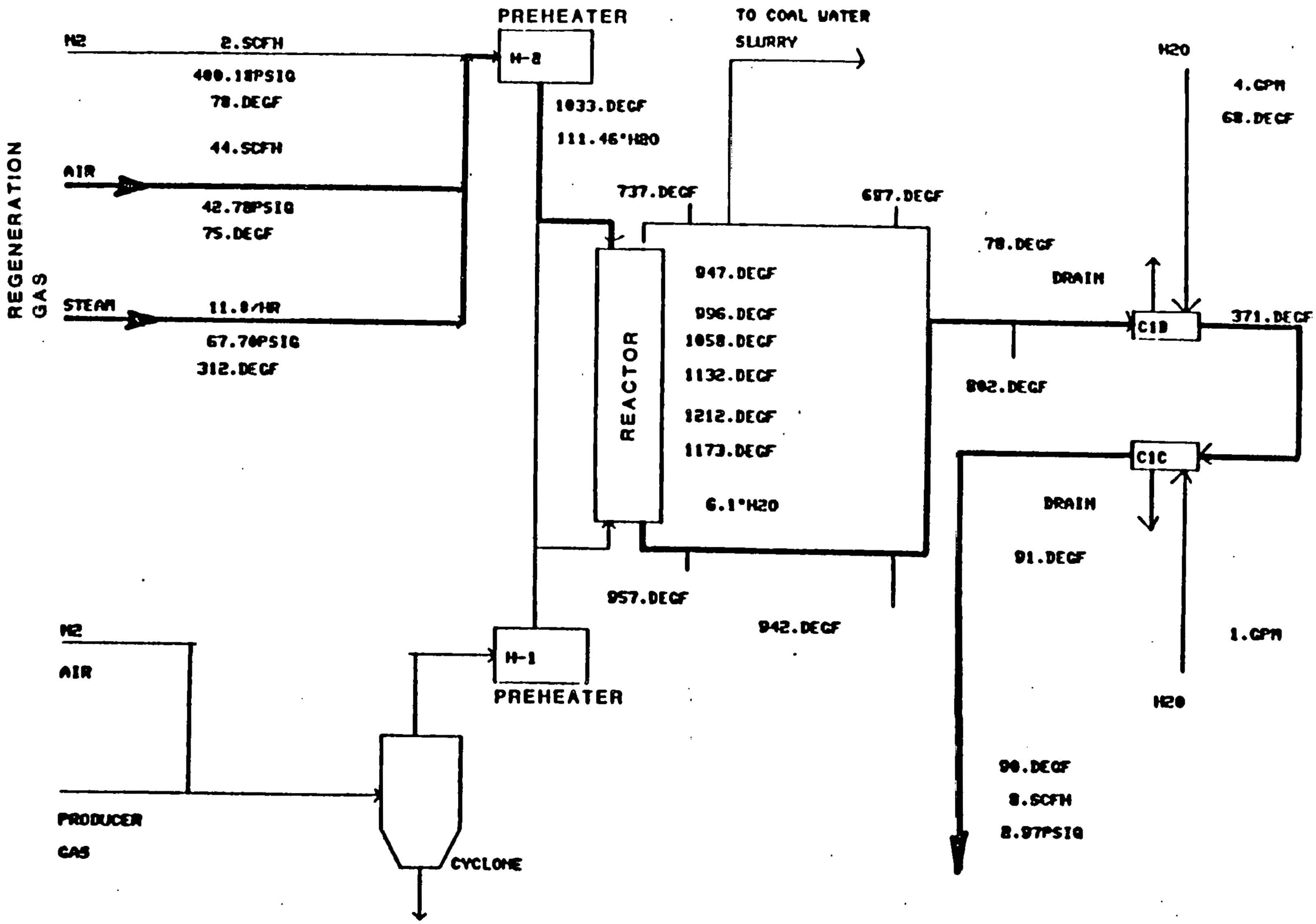


AVERAGE(S) FOR PERIOD FROM: 18:40:17 9/30/1983 TO 11:09:56 10/ 1/1983

FIGURE 17

TEST 012

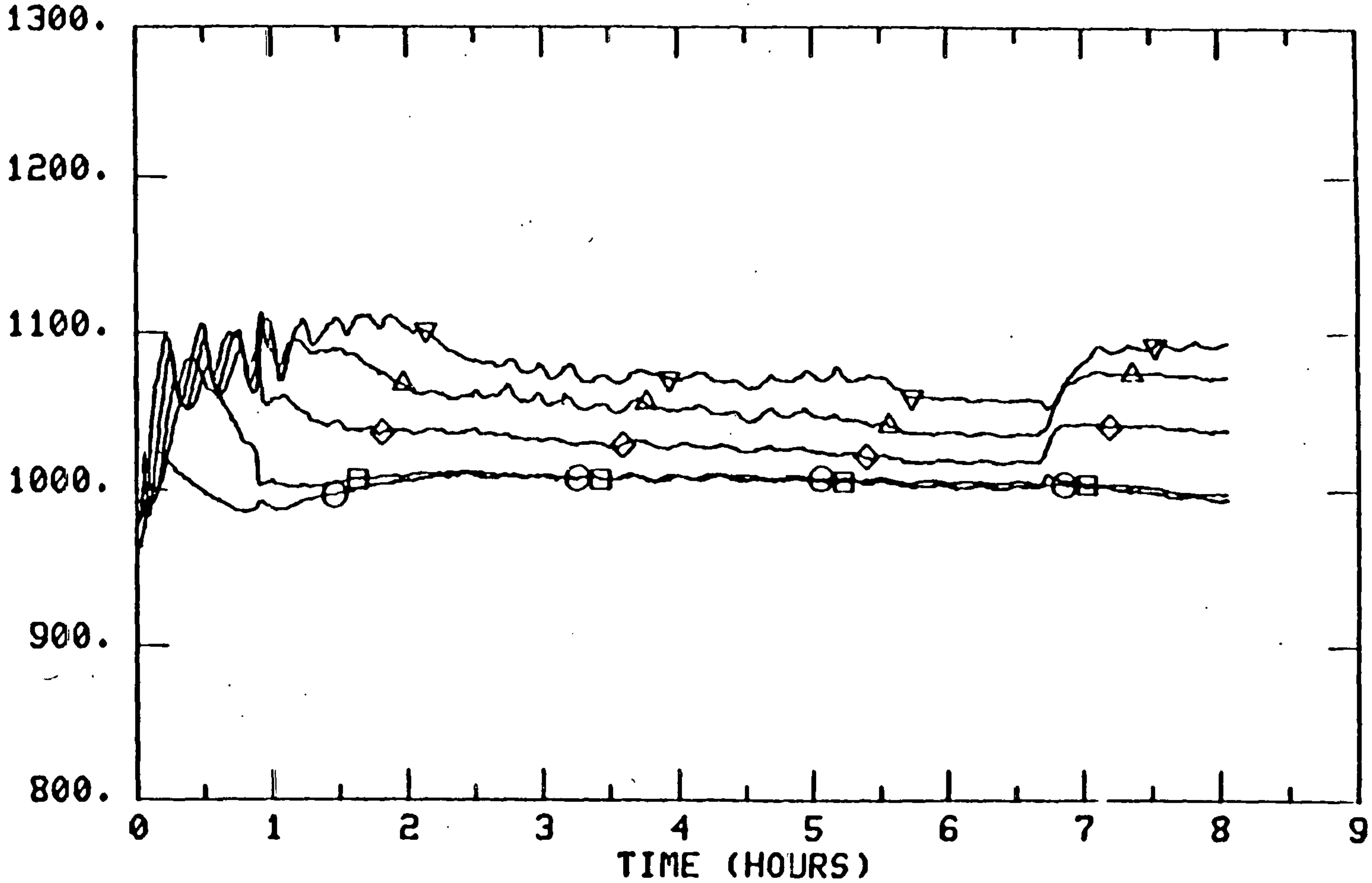
AVERAGE OPERATING PARAMETERS



AVERAGE(S) FOR PERIOD FROM: 13:20:81 10/ 1/1983 TO 5:19:38 10/ 8/1983

FIGURE 18

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
○	BS1	DEGF	ABOSRBER 5° FROM BOTTOM	970	1005.	8.915	979.7	1	1026.	1
□	BS2	DEGF	ABSORBER 15° FROM BOTTOM	970	1018.	18.67	963.1	1	1099.	2
◇	BS3	DEGF	ABSORBER 25° FROM BOTTOM	970	1037.	18.99	979.7	2	1106.	2
△	BS4	DEGF	ABSORBER 36° FROM BOTTOM	970	1059.	19.32	991.2	2	1113.	1
▽	BS5	DEGF	ABSORBER 45° FROM BOTTOM	970	1077.	19.23	1001.	1	1112.	5



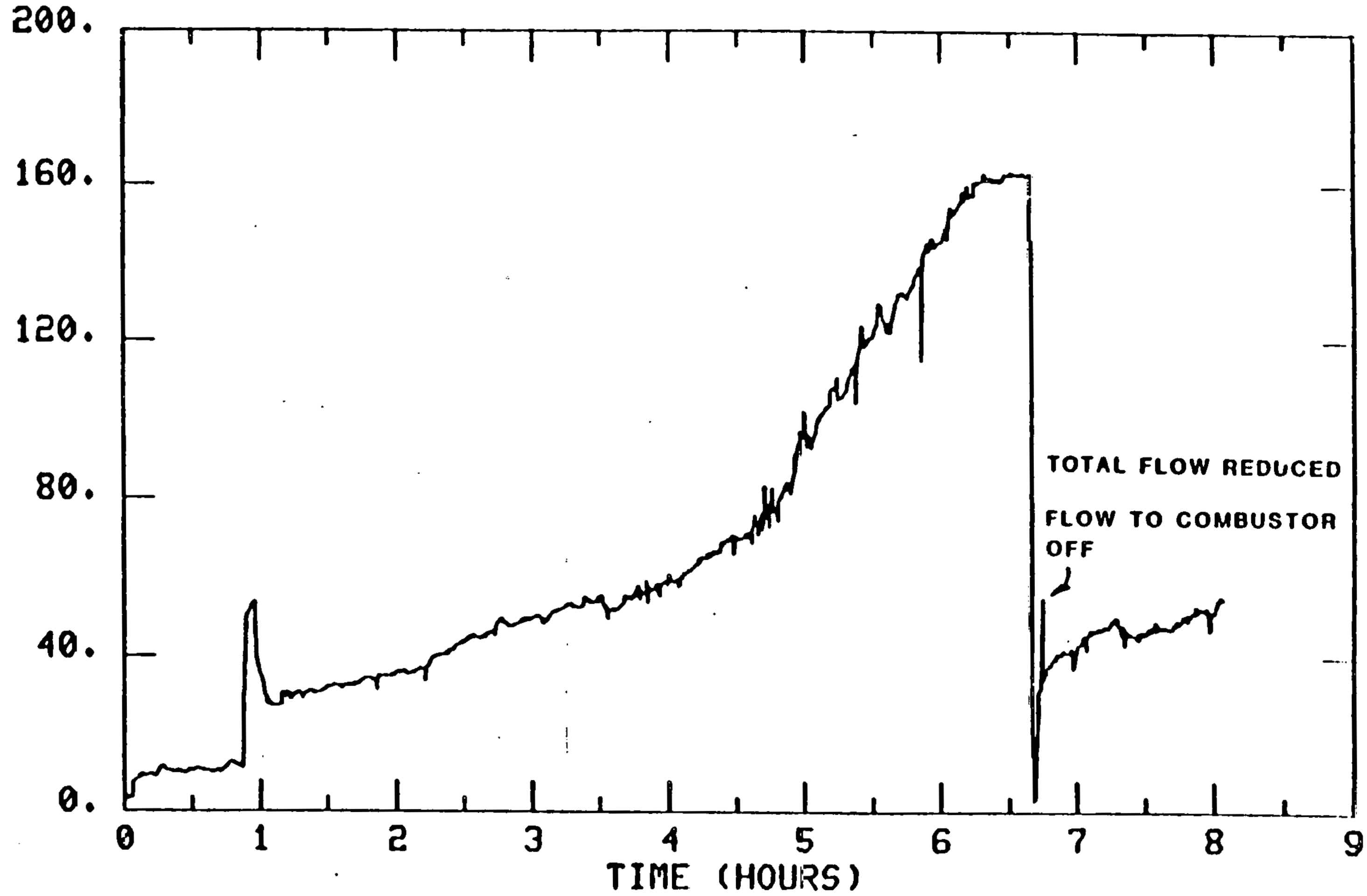
START TIME OF PLOT 11:45: 0 9/29/1983
STOP TIME OF PLOT 19:50: 0 9/29/1983

TEST 009

REACTOR TEMPERATURES

FIGURE 19

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
294	"H2O	ABSORBER DIFFERENCE PRESSURE	970	62.68	42.27	2.984	1	163.8	1



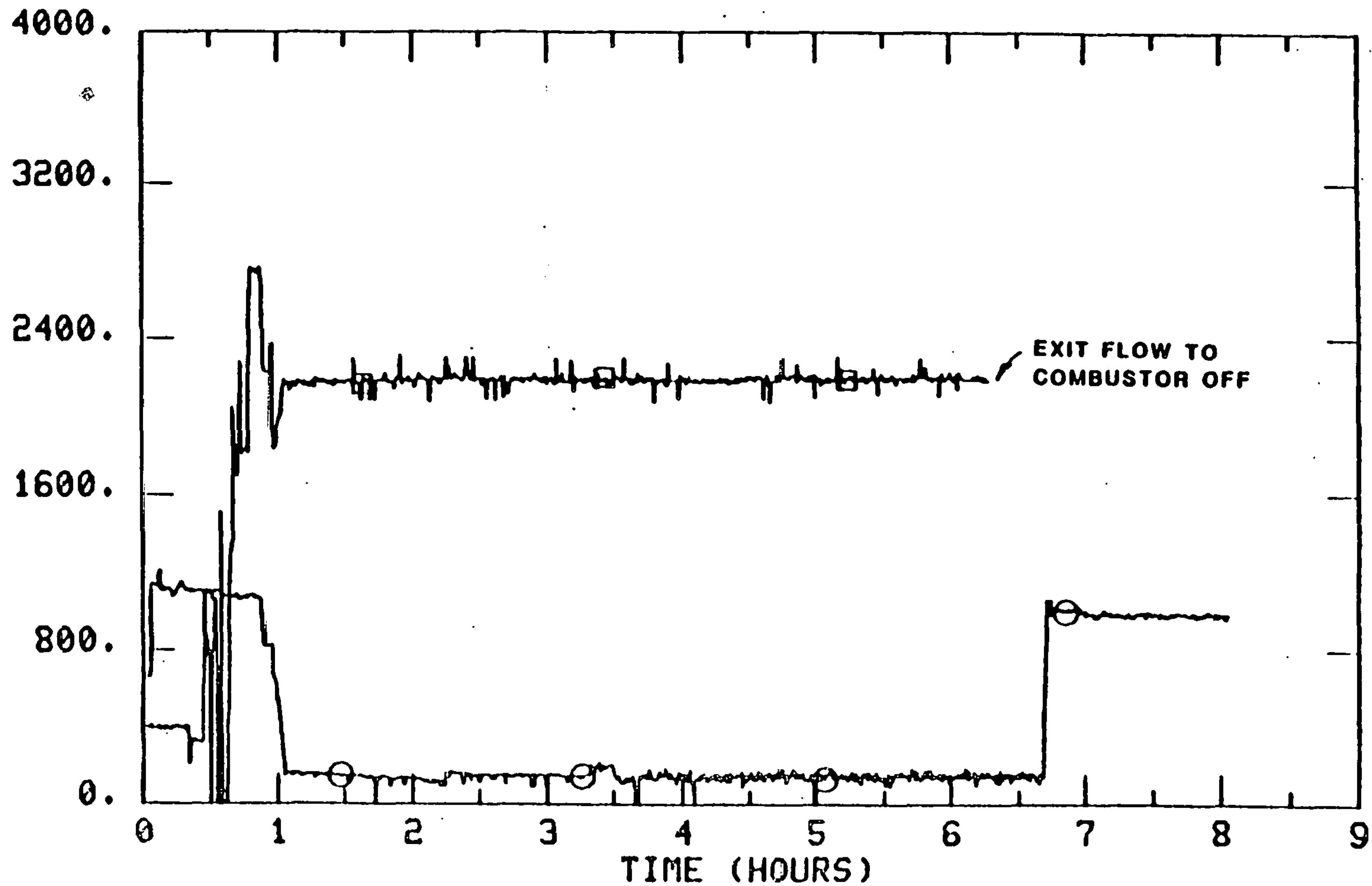
START TIME OF PLOT 11:45: 0 9/29/1983
STOP TIME OF PLOT 19:50: 0 9/29/1983

TEST 009

REACTOR PRESSURE DROP

FIGURE 20

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	3750	SCFH	PROD. GAS OUT OF SYSTEM	970	401.1	397.3	0.0000E+00	5	1216.	1
□	4017	SCFH	PRODUCER GAS FLOW TO COMBUSTOR	755	8010.	551.4	0.0000E+00	8	2770.	1



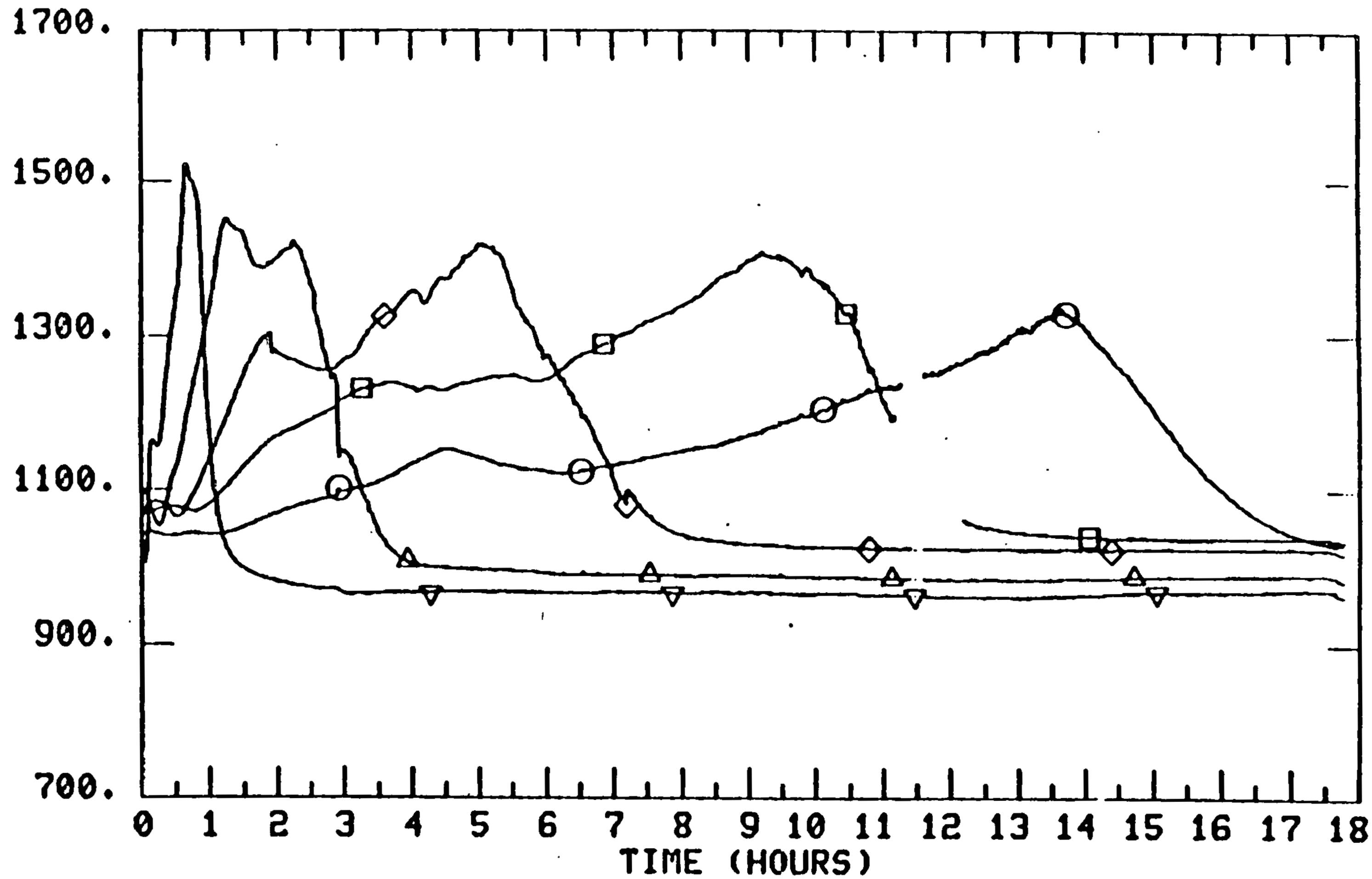
START TIME OF PLOT 11:45: 0 9/29/1983
STOP TIME OF PLOT 19:50: 0 9/29/1983

TEST 009

EXIT FLOWS

FIGURE 21

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	851	DEGF	ABOSRBER 5° FROM BOTTOM	2100	1154.	82.39	1030.	2	1339.	1
□	852	DEGF	ABSORBER 15° FROM BOTTOM	2020	1182.	125.8	1034.	1	1412.	2
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	2116	1124.	130.7	1019.	2	1421.	1
△	854	DEGF	ABSORBER 35° FROM BOTTOM	2116	1048.	128.8	983.1	1	1453.	1
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	2116	989.2	85.68	961.4	1	1525.	2



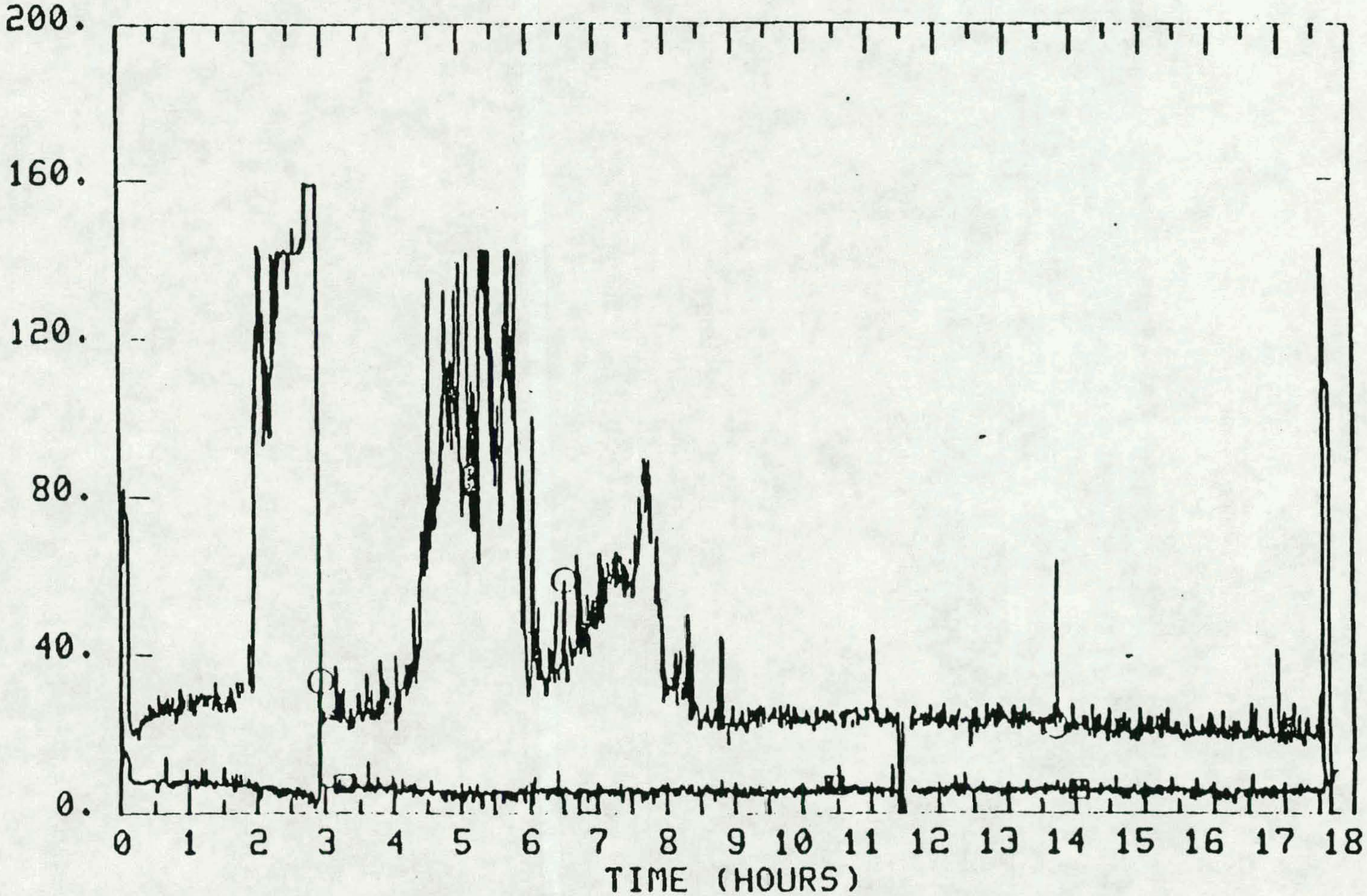
START TIME OF PLOT 21:40: 0 9/29/1983
STOP TIME OF PLOT 15:30: 0 9/30/1983

TEST 010

REACTOR TEMPERATURES

FIGURE 22

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	302	"H2O	ABSORBER INLET TOP PRESSURE	1930	41.44	35.36	-84.39	8	159.6	1
□	294	"H2O	ABSORBER DIFFERENCE PRESSURE	2124	5.769	2.703	-28.72	8	31.48	1



START TIME OF PLOT 21:40: 0 9/29/1983
STOP TIME OF PLOT 15:30: 0 9/30/1983

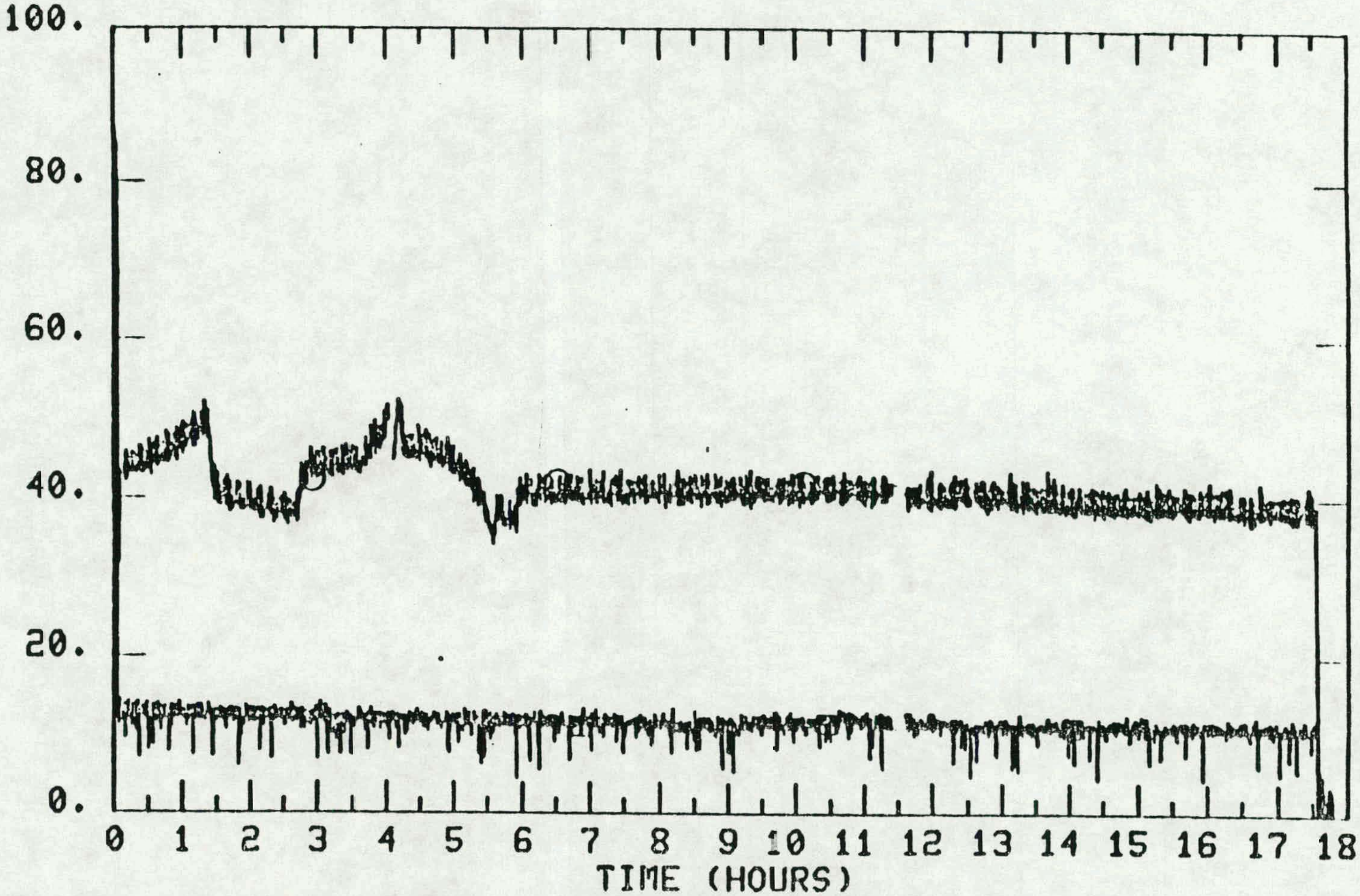
TEST 010

REGENERATION

REACTOR PRESSURE DROP AND REACTOR INLET PRESSURE

FIGURE 23

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	3756	SCFH	AIR FLOW LOW RANGE	2108	41.05	5.928	0.0000E+00	24	87.36	1
D	3754	#/HR	STEAM FLOW	2114	11.30	1.646	0.0000E+00	3	14.74	1



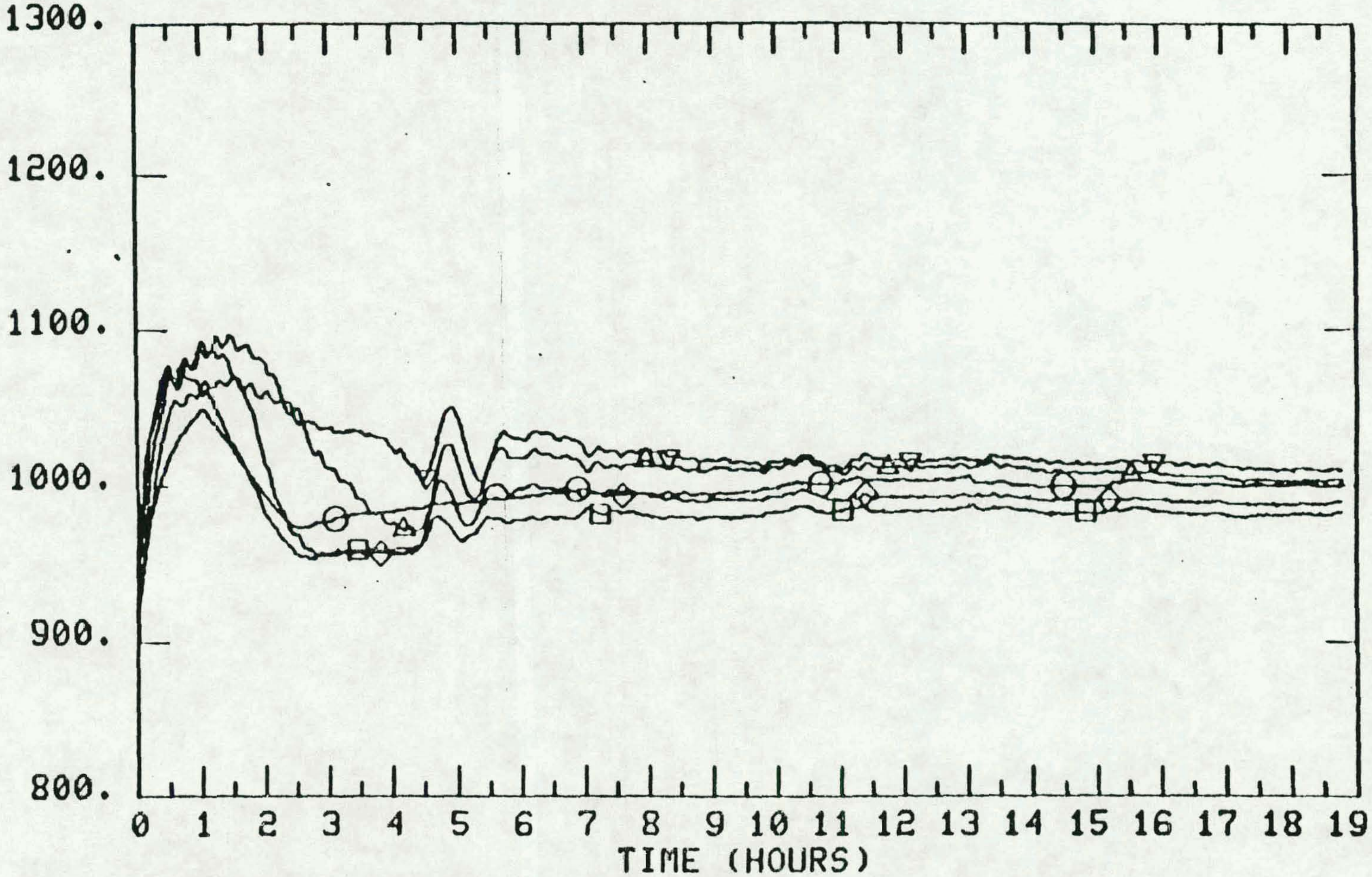
START TIME OF PLOT 21:40: 0 9/29/1983
STOP TIME OF PLOT 15:30: 0 9/30/1983

TEST 010

STEAM AND AIR FLOWS

FIGURE 24

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	851	DEGF	ABOSRBER 5° FROM BOTTOM	2262	998.6	13.25	915.2	1	1050.	1
□	852	DEGF	ABSORBER 15° FROM BOTTOM	2262	985.3	22.91	900.3	1	1077.	2
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	2262	995.8	26.72	942.4	1	1089.	2
△	854	DEGF	ABSORBER 35° FROM BOTTOM	2262	1017.	24.85	950.7	2	1098.	3
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	2262	1024.	16.78	971.3	3	1072.	1



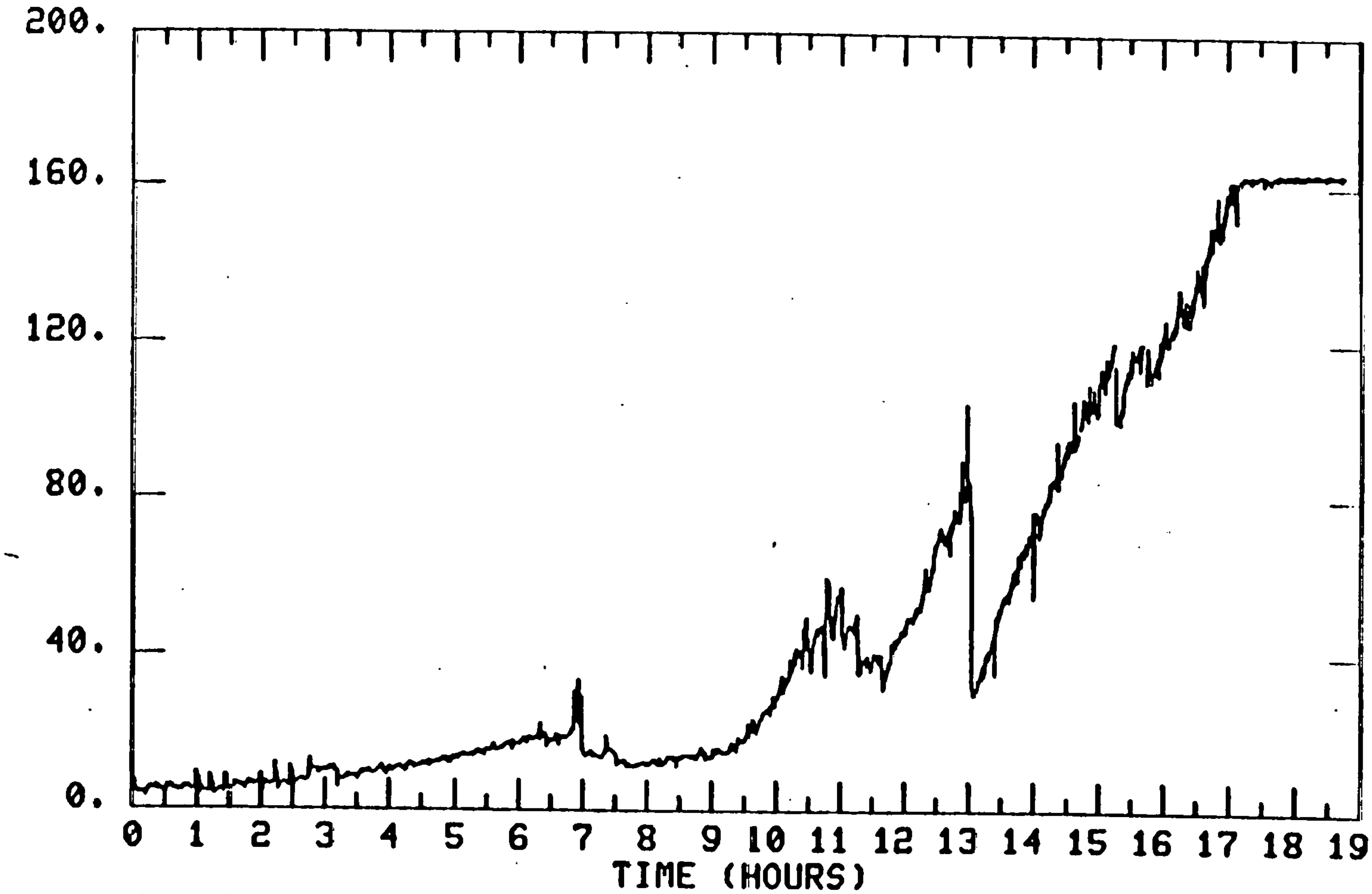
START TIME OF PLOT 16:40: 0 9/30/1983
STOP TIME OF PLOT 11:30: 0 10/ 1/1983

TEST 011

REACTOR TEMPERATURES

FIGURE 25

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
204	'H2O	ABSORBER DIFFERENCE PRESSURE	2250	50.77	58.33	3.066	1	164.7	1



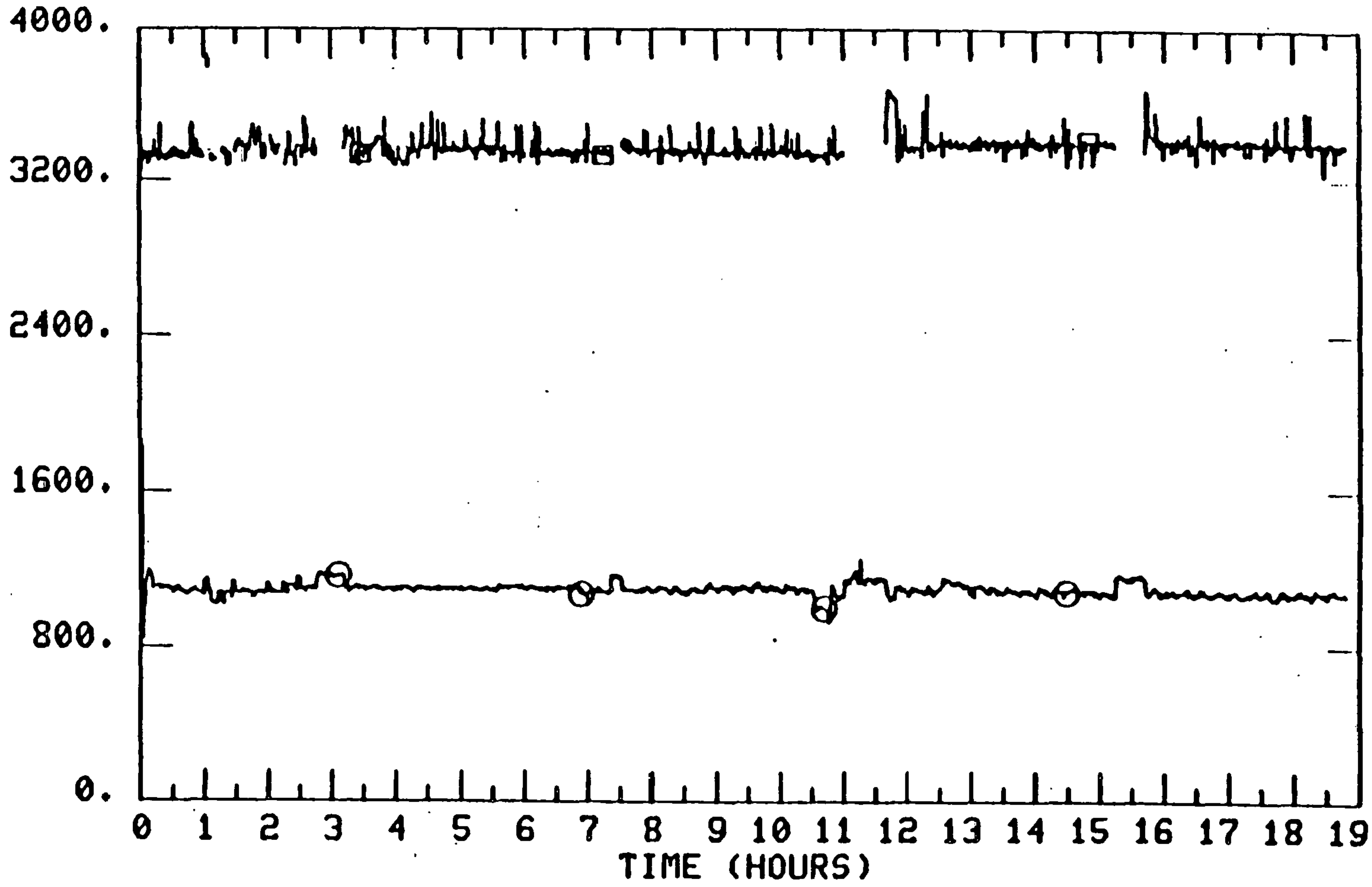
START TIME OF PLOT 16:40: 0 9/30/1983
STOP TIME OF PLOT 11:30: 0 10/ 1/1983

TEST 011

REACTOR PRESSURE DROP

FIGURE 26

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN	FREQ	MAX	FREQ
O	3750	SCFH	PROD. GAS OUT OF SYSTEM	2262	1100.	43.10	846.1	1	1869.	1
□	4017	SCFH	PRODUCER GAS FLOW TO COMBUSTOR	1954	3388.	53.70	3244.	1	3879.	1



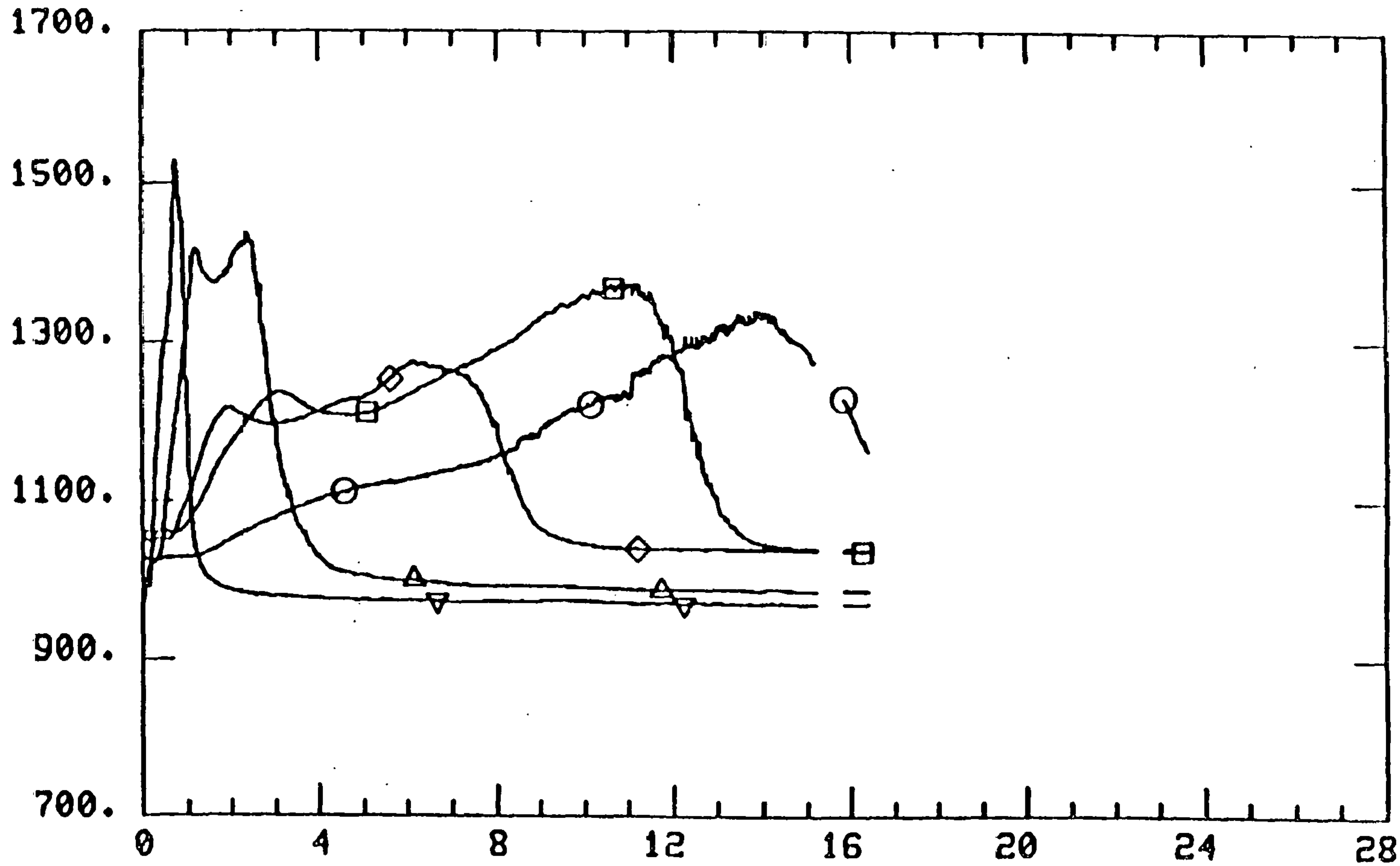
START TIME OF PLOT 16:40: 0 9/30/1983
STOP TIME OF PLOT 11:30: 0 10/ 1/1983

TEST 011

EXIT FLOWS

FIGURE 27

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN	FREQ	MAX	FREQ
○	851	DEGF	ABOSRBER 5° FROM BOTTOM	1900	1173.	95.81	972.3	1	1340.	4
□	852	DEGF	ABSORBER 15° FROM BOTTOM	1900	1207.	108.9	1036.	3	1375.	4
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	1900	1129.	90.80	1038.	1	1276.	4
△	854	DEGF	ABSORBER 35° FROM BOTTOM	1900	1058.	131.3	986.8	2	1438.	2
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	1900	995.5	83.29	969.8	2	1530.	1



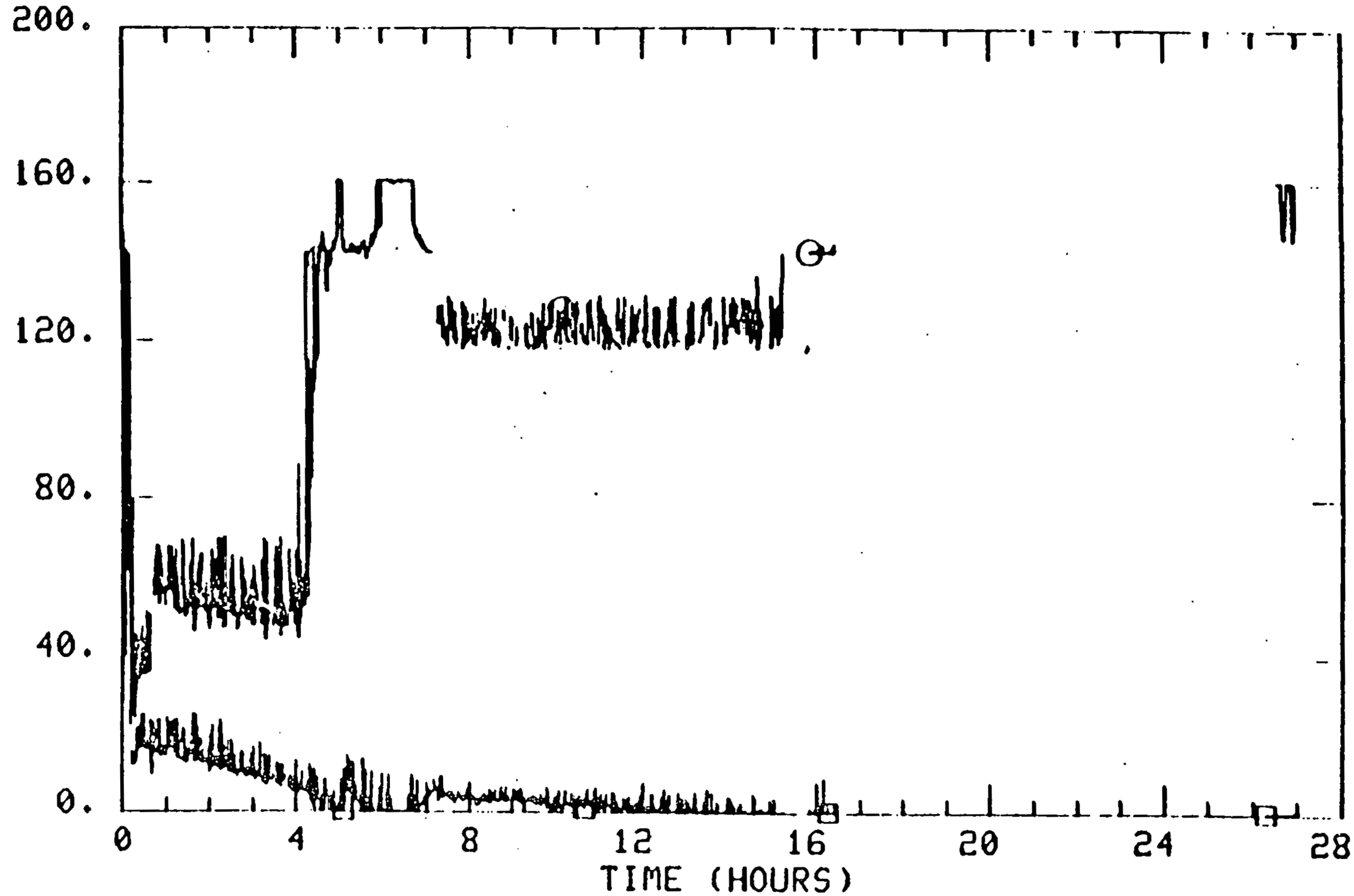
START TIME OF PLOT 13:20: 0 10/ 1/1983
STOP TIME OF PLOT 16:20: 0 10/ 2/1983

TEST 012

REACTOR TEMPERATURES

FIGURE 28

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	302	"H2O	ABSORBER INLET TOP PRESSURE	1497	118.2	38.99	23.99	1	161.1	1
□	294	"H2O	ABSORBER DIFFERENCE PRESSURE	1873	4.095	9.347	-28.72	34	113.6	1



START TIME OF PLOT 13:20: 0 10/ 1/1983
STOP TIME OF PLOT 16:20: 0 10/ 2/1983

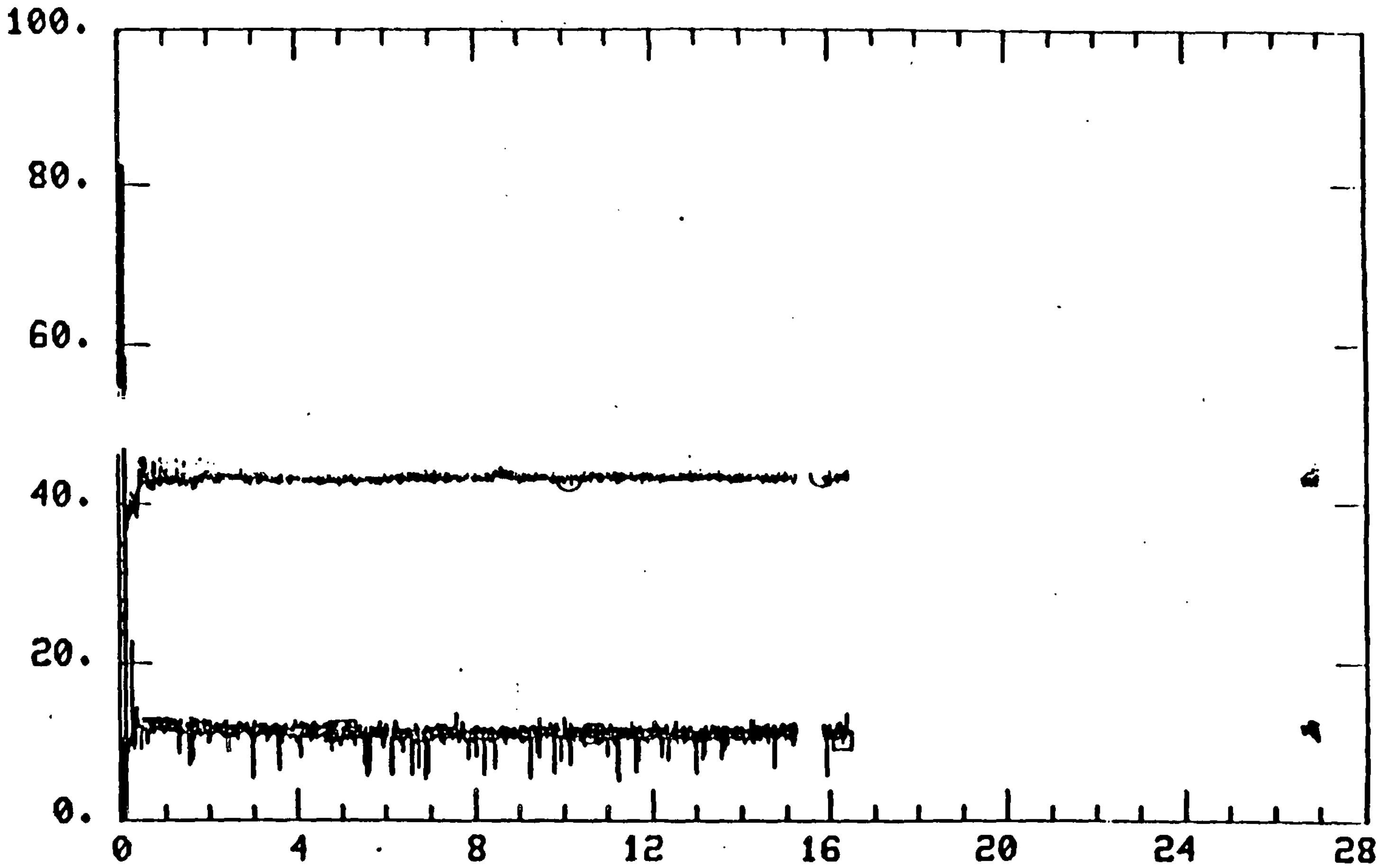
TEST 012

REGENERATION

REACTOR PRESSURE DROP AND REACTOR INLET PRESSURE

FIGURE 29

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	3756	SCFH	AIR FLOW LOW RANGE	1946	43.86	4.204	0.0000E+00	14	82.66	1
□	3754	#/HR	STEAM FLOW	1944	11.12	1.070	0.0000E+00	3	22.85	1



START TIME OF PLOT 13:20: 0 10/ 1/1983
STOP TIME OF PLOT 16:20: 0 10/ 2/1983

TEST 012

STEAM AND AIR FLOWS

Figure 30

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 006
DATE STARTED : 12:02 9/20/83
DATE ENDED : 16:10 9/20/83

TOTAL HOURS : 4.13
TYPE : SULFIDATION
SULFUR REMOVED: 2.86 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
COAL TYPE : ARKWRIGHT
STEAM/COAL MASS RATIO: 0.408
STEAM/AIR MASS RATIO: 0.19

HGD OPERATING CONDITIONS

TEMPERATURE: 1149 F (AVERAGE)
PRESSURE : 171 PSIG (AVERAGE)
SPACE VELOCITY: 2028 (0.87 HRS), 7270 (2.13 HRS), 1908 (1.13 HRS)

	INLET MOLE% DRY BASIS (OIL FREE)	INLET MOLE% WET BASIS (OIL FREE)	EXIT MOLE% DRY BASIS (OIL FREE)	EXIT MOLE% WET BASIS (OIL FREE)
H_2S	0.314			
H_2O				7.3
CO_2	14.7		NO EXIT GC DATA	
CO	24.0			
H_2	15.5			
CH_4	3.1			
N_2	44.3			
O_2	1.0			
C_2H_6	0.25			
TOTAL FLOW:				
TOTAL LB MOLES:				
FLOW RATE:	1173 SCFH (0.87 HOURS)		1135 SCFH (0.87 HRS)	
	4013 SCFH (2.13 HOURS)		4068 SCFH (2.13 HRS)	
	1063 SCFH (1.13 HOURS)		1058 SCFH (1.13 HRS)	

PURPOSE

DETERMINE EFFECT OF TEMPERATURE ON DESULFURIZATION EFFICIENCY
DETERMINE EFFECT OF HIGH SPACE VELOCITY ON OPERATING PERFORMANCE.
PROVIDE DESULFURIZED GAS TO SLURRY COMBUSTION UNIT.

SORBENT ANALYSIS:

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

DATA ABOVE FROM T001

EXIT SULFUR DATA:

H_2S : 5 PPM INITIALLY
CH_4S: 7 PPM

CONDENSATE

AQUEOUS : 13.7 LBS (ESTIMATED)
HYDROCARBON : 2.0 LBS (ESTIMATED)
TOTAL : 15.7 LBS (MEASURED)

REMARKS

1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 7/8 INCHES
3. FIRST TEST ON FRESH SORBENT. GASIFIER RUN 102
4. BENDIX GC AT HGD EXIT NOT FUNCTIONING.
5. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
6. AVERAGE H2S AT HGD INLET LESS THAN 0.1% AS DETERMINED BY BENDIX GC 6000 #1.
7. AN AVERAGE OF [illegible] SCFH DESULFURIZED GAS WAS PROVIDED TO THE SLURRY COMBUSTION UNIT FOR THREE HOURS.
8. REACTOR DELTA P WAS OBSERVED TO INCREASE RAPIDLY.
9. HOT GAS PIPELINE APPEARS TO HAVE PICKED UP SULFUR FOR THE FIRST TWO HOURS OF THE RUN. THIS APPEARS LIKELY SINCE THE HOT GAS DELIVERY PIPELINE WAS "REGENERATED" WITH STEAM AND AIR PRIOR TO THIS TEST.
10. GAS COMPOSITION AT INLET IS AVERAGE OF DATA OBTAINED BY BENDIX GC 7000 #4 SAMPLING AT 54 GASIFIER CYCLONE EXIT

CONCLUSIONS:

1. H2S BREAKTHROUGH AT 1149 F AND SPACE VELOCITY OF 7270 IS 25 % OF TIME TO BREAKTHROUGH AT 1000 F AND S.V. OF 2000 WHEN NORMALIZED.

Figure 31

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 007
DATE AND TIME STARTED : 19:30 9/20/83
DATE AND TIME ENDED : 08:50 9/21/83
TOTAL HOURS : 13.3
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: 935 F

PRESSURE : 129 INCHES OF WATER (AVERAGE)

SPACE VELOCITY: 512 HOURLY

		INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	55 SCFH	19 %	NO GC DATA AT EXIT
H_2O	11 #/HR	81 %	
N_2	0 SCFH		

TOTAL LB MOLES:
FLOW RATE : 287 SCFH

PURPOSE

FIRST REGENERATION OF SULFIDED SORBENT, GASIFIIER RUN 102.

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 : HIGH SO2 (> 20 %) INITIALLY.
0.1% SO2 AT END OF REGENERATION.

CONDENSATE		
AQUEOUS	:	409 LBS
HYDROCARBON	:	
TOTAL	:	409 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-10789 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.559 CUBIC FOOT
3. FIRST REGENERATION ON SULFIDED SORBENT

4. MAXIMUM TEMPERATURE REACHED WAS 1364 F.

CONCLUSIONS

1. REGENERATION COMPLETED.

Figure 32

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 008
DATE STARTED : 12:17 9/21/83
DATE ENDED : 21:30 9/21/83
TOTAL HOURS : 6.95
TYPE : SULFIDATION
SULFUR REMOVED: 4.24 LB SE

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
COAL TYPE : ARKWRIGHT
STEAM/COAL RATIO: 0.366
STEAM/AIR RATIO : 0.19

HGD OPERATING CONDITIONS

TEMPERATURE: 1147 F (AVERAGE)
PRESSURE : 143 PSIG (AVERAGE)

SPACE VELOCITY: 6507 HOURLY FOR 3 HOURS, 1803 HOURLY FOR 4 HRS

	INLET MOLE% DRY BASIS (OIL FREE)	INLET MOLE% WET BASIS (OIL FREE)	EXIT MOLE% DRY BASIS (OIL FREE)	EXIT MOLE% WET BASIS (OIL FREE)
H_2S	0.336			
H_2O				7.8
CO_2	14.14		NO GC DATA AT EXIT	
CO	24.44			
H_2	15.37			
CH_4	2.92			
N_2	42.45			
O_2	1.00			
C_2H_6	0.23			
TOTAL FLOW:				

FLOW RATE: 3809 SCFH WITH FLOW TO SLURRY COMBUSTION. APPROXIMATELY 3 HOURS
1009 SCFH WITHOUT FLOW TO COMBUSTOR APPROXIMATELY 4 HOURS

PURPOSE

DETERMINE EFFECT OF TEMPERATURE ON DESULFURIZATION EFFICIENCY
DETERMINE EFFECT OF HIGH SPACE VELOCITY ON OPERATING PERFORMANCE.
PROVIDE DESULFURIZED GAS TO SLURRY COMBUSTION UNIT.

SORBENT ANALYSIS:

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

EXIT SULFUR DATA:

H_2S : 5 PPM INITIALLY
CH_4S:

CONDENSATE
AQUEOUS : 28.3 LBS (ESTIMATED)
HYDROCARBON : 4.1 LBS (ESTIMATED)
TOTAL : 32.4 LBS (MEASURED)

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES
CS-346 1/2 S SR-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 53 7/8 INCHES
3. SECOND TEST ON FRESH SORBENT, GASIFIER RUN 102
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
5. SORBENT WAS REMOVED AFTER THIS TEST. TOP 20 INCHES WERE FUSED, WITH ANOTHER 4 INCH SECTION AT MID BED FUSED.
6. REACTOR DELTA P WAS HIGH FROM THE BEGINNING OF TEST AT MAX READING OF 160 INCHES OF WATER.
7. FLOW TO SLURRY COMBUSTION FROM HGD EXIT AT 2800 SCFH AVERAGE FOR APPROXIMATELY 3 HOURS, AFTER WHICH A PLUG DEVELOPED AND FLOW WAS SHUT OFF.

CONCLUSIONS
1. SULFUR REMOVAL BETTER DURING THIS SECOND SULFIDATION COMPARED WITH THE FIRST SULFIDATION, TEST 006.
2. APPARENTLY CARBON AND PARTICULATES WERE NOT BURNED OUT DURING THE LAST REGENERATION. HOWEVER, THIS DOES NOT APPEAR TO AFFECT THE DESULFURIZATION CAPACITY NEGATIVELY.

Figure 33

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 009
DATE STARTED : 11:45 9/29/83
DATE ENDED : 19:50 9/29/83
TOTAL HOURS : 8.09
TYPE : SULFIDATION
SULFUR REMOVED: 7.76 LB SB

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
COAL TYPE : BRIQUETTES (BLACKSVILLE + TAR)
STEAM/COAL RATIO: 0.553
STEAM/AIR RATIO : 0.27

HGC OPERATING CONDITIONS

TEMPERATURE: 1044 F (AVERAGE)
PRESSURE : 94 PSIG (AVERAGE)

SPACE VELOCITY: 4179 HOURLY (5.75 HRS), 1814 HOURLY (2.32 HRS)

	INLET MOLE% DRY BASIS (OIL FREE)	INLET MOLE% WET BASIS (OIL FREE)	EXIT MOLE% DRY BASIS (OIL FREE)	EXIT MOLE% WET BASIS (OIL FREE)
H_2S	0.583		1 PPM	
H_2O				6.5 %
CO_2	10.03		11.79	
CO	22.25		15.83	
H_2	17.14		21.34	
CH_4	2.40		2.09	
N_2	47.06		47.59	
O_2	0.65		0.86	
C_2H_6	0.16		0.00	
TOTAL FLOW:				
TOTAL LB MOLES:				

FLOW RATE: 2373 SCFH (5.75 HRS)
1015 SCFH (2.32 HRS)

PURPOSE

DETERMINE EFFECT OF HIGH SPACE VELOCITY ON OPERATING PERFORMANCE.
PROVIDE DESULFURIZED GAS TO SLURRY COMBUSTION UNIT.

SORBENT ANALYSIS:

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

DATA ABOVE FROM T001

EXIT SULFUR DATA:

H_2S : 1 PPM INITIALLY, BREAKTHROUGH AT 5 HOURS
CH_3SH:

CONDENSATE
AQUEOUS : 10.6 LBS (ESTIMATED)
HYDROCARBON : 1.6 LBS (ESTIMATED)
TOTAL : 12.2 LBS (MEASURED)

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10769 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 53 7/8 INCHES
3. GASIFIER RUN 102. FRESH SORBENT LOADED FOR T009.
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
5. AN AVERAGE OF 2193 SCFH DESULFURIZED GAS WAS PROVIDED TO THE SLURRY COMBUSTION UNIT FOR APPROX. 5.75 HOURS.
6. REACTOR DELTA P WAS OBSERVED TO INCREASE RAPIDLY UP TO A MAX OF 144 INCHES OF WATER.
7. INLET DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 7000 #4 DATA FOR THE DURATION OF THE RUN.
8. EXIT DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 6000 #2 DATA FOR THE DURATION OF THE RUN.

CONCLUSIONS:

1. TIME TO BREAKTHROUGH AT SV OF 4179 IS LESS THAN HALF OF TIME TO BREAKTHROUGH AT SV 2000.
2. SPACE VELOCITIES AS HIGH AS 4000 DO NOT APPEAR TO LOAD THE SORBENT AS WELL AS LOWER SPACE VELOCITIES.
3. COS BREAKTHROUGH APPEARS TO LEAD H2S BREAKTHROUGH BY ABOUT 1 1/2 HOURS.

Figure 34

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 010
DATE AND TIME STARTED : 21:40 9/29/83
DATE AND TIME ENDED : 15:30 9/30/83
TOTAL HOURS : 17.8
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: 1058 F

PRESSURE : 41.8 INCHES OF WATER (AVERAGE)

SPACE VELOCITY: 496 HOURLY

		INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	40.86 SCFH	21 %	
H2O	11.25 #/HR	79 %	
N2	0 SCFH		

TOTAL LB MOLES:
FLOW RATE : 278 SCFH

PURPOSE

REGENERATION OF SULFIDED SORBENT FROM T009 GASIFIIER RUN 102.

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 : 4.5 % INITIALLY . HIGH OF >16% AFTER 2.5 HOURS
0.5 % AT END OF RUN .

CONDENSATE
AQUEOUS :
HYDROCARBON :
TOTAL : 220 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-10769 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.559 CUBIC FOOT
3. FIRST REGENERATION ON SULFIDED SORBENT (SECOND BATCH)

4. MAXIMUM TEMPERATURE REACHED WAS 1524 F.

CONCLUSIONS

1. REGENERATION COMPLETED.

Figure 35

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 011
DATE STARTED : 16:40 9/30/83
DATE ENDED : 11:30 10/1/83
TOTAL HOURS : 18.8
TYPE : SULFIDATION
SULFUR REMOVED: 6.27 LB S3

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
COAL TYPE : BRICQUETTES (BLACKSVILLE + TAR),AND BLACKSVILLE RUN OF MINE
STEAM/COAL RATIO: 0.556
STEAM/AIR RATIO : 0.225

HGC OPERATING CONDITIONS
TEMPERATURE: [illegible] F (AVERAGE)

PRESSURE : 122 PSIG (AVERAGE)

SPACE VELOCITY: 1966 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)
H_2S	0.351			
H_2O				7.1 %
CO_2	8.9		11.4	
CO	23.3		15.5	
H_2	16.1		26.4	
CH_4	2.2		2.4	
N_2	48.0		50.8	
O_2	0.6		0.7	
C_2H_6	0.2		0.1	
TOTAL FLOW:				
TOTAL LB MOLES:				
FLOW RATE:	1100 SCFH			SCFH

PURPOSE

SECOND SULFIDATION ON SORBENT

SORBENT ANALYSIS:

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

EXIT SULFUR DATA:
H_2S : [illegible] PPM INITIALLLY GRADUALLY DOWN TO 2 PPM

C_nH_mS:
SO_2 : 10 PPM INITIALLY

CONDENSATE
AQUEOUS : 75.2 LBS (ESTIMATED)
HYDROCARBON : 11.4 LBS (ESTIMATED)
TOTAL : 86.6 LBS (MEASURED)

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SP-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 59 7/8 INCHES
3. SECOND SULFIDATION ON FRESH SORBENT. GASIFIER RUN 102 SORBENT REMOVED AFTER RUN 008 AND FRESH SORBENT LOADED
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
5. EXIT DRY GAS ANALYSIS TOTAL IS 107 % .
6. REACTOR DELTA P WAS OBSERVED TO INCREASE RAPIDLY UP TO A MAX OF 164 INCHES OF WATER.
7. INLET DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 6000 #1 DATA.
8. EXIT DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 6000 #2 DATA.

CONCLUSIONS:

1. SULFIDATION AT SV 1966 RESULTED IN LONGER TIME TO BREAKTHROUGH. APPROACHING THE TIMES PREVIOUSLY OBSERVED.

Figure 36

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 012
DATE AND TIME STARTED : 13:20 10/01/83
DATE AND TIME ENDED : 16:20 10/02/83

TOTAL HOURS : 27
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

PURPOSE

REGENERATION OF SULFIDED SORBENT FROM T011 GASIFIIER RUN 10?

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)		

HGD OPERATING CONDITIONS

INITIAL TEMPERATURE: 1006 F

PRESSURE : 112.3 INCHES OF WATER (AVERAGE)

SPACE VELOCITY: 496 HOURLY

			INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	44.22	SCFH	22.7 %	
H2O	11.12	#/HR	77.3 %	
N2	0	SCFH		

TOTAL LB MOLES:
FLOW RATE : 278 SCFH

EXIT SULFUR DATA:

H2S :

SO2 : 6 % INITIALLY . HIGH OF 10% AFTER 4.2 HOURS
300 PPM AT END OF RUN .

CONDENSATE
AQUEOUS : LBS
HYDROCARBON :
TOTAL : 379 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-10789 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.559 CUBIC FOOT
3. SECOND REGENERATION ON SULFIDED SORBENT (SECOND BATCH)
4. MAXIMUM TEMPERATURE REACHED WAS 1530 F.
5. LAST TEST DURING GASIFIER RUN 102. REACTOR COOLED AND
SORBENT SAMPLED AND REMOVED.
6. 118 GRAMS OF GRAY/BLACK DUST COLLECTED ON BOTTOM FLANGE
OF THE REACTOR. THIS HAS ACCUMULATED SINCE RUN T001.

CONCLUSIONS:
1. REGENERATION COMPLETED.

Figure 37

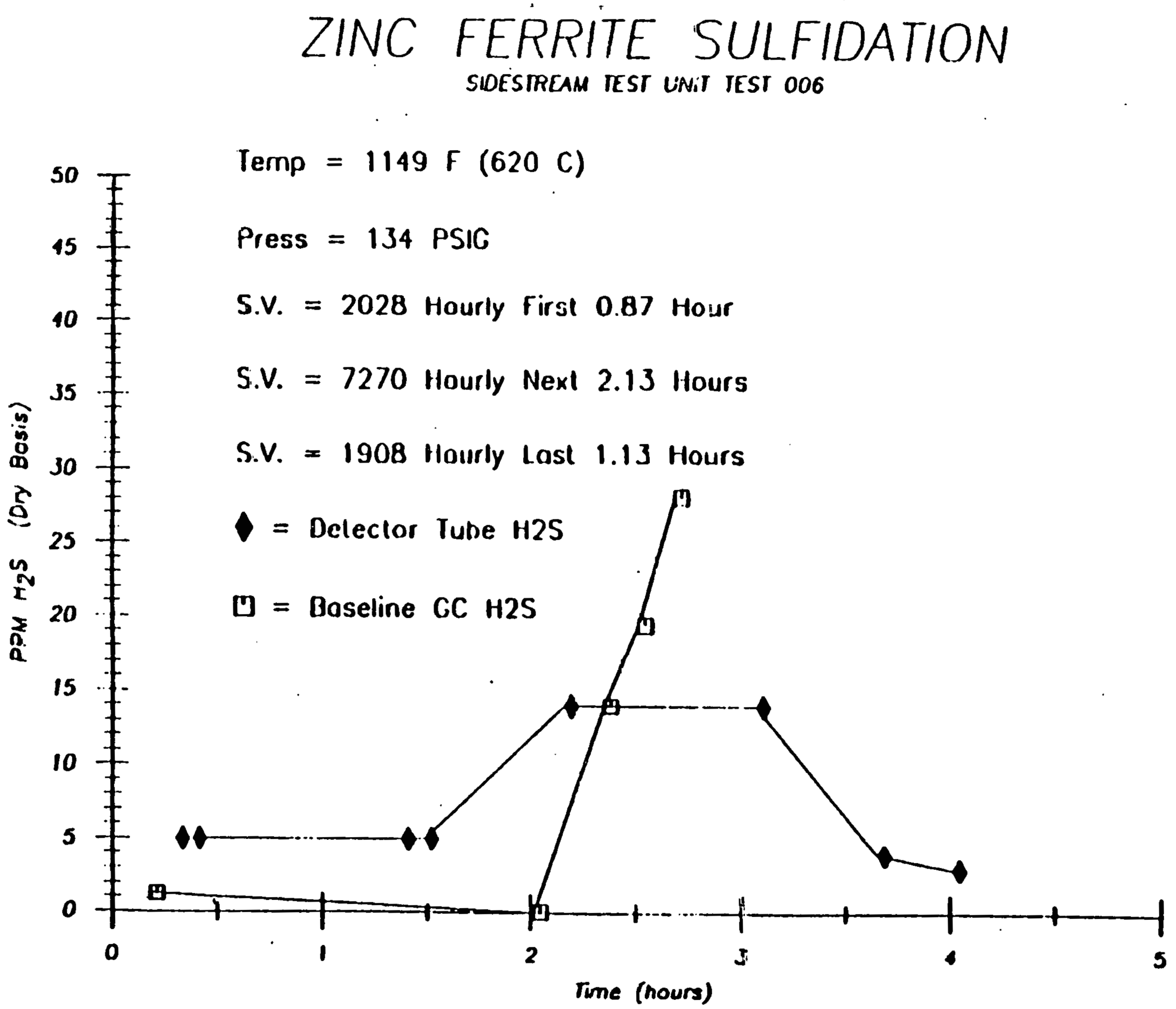

ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 006
Temp = 1149 F (620 C)
Press = 134 PSIG
S.V. = 2028 Hourly First 0.87 Hour
S.V. = 7270 Hourly Next 2.13 Hours
S.V. = 1908 Hourly Last 1.13 Hours
= Detector Tube H2S
= Baseline GC H2S
PPM H2S (Dry Basis)
50
45
40
35
30
25
20
15
10
5
0
0
1
2
3
4
5
Time (hours)

Figure 38

ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TEST ' 008

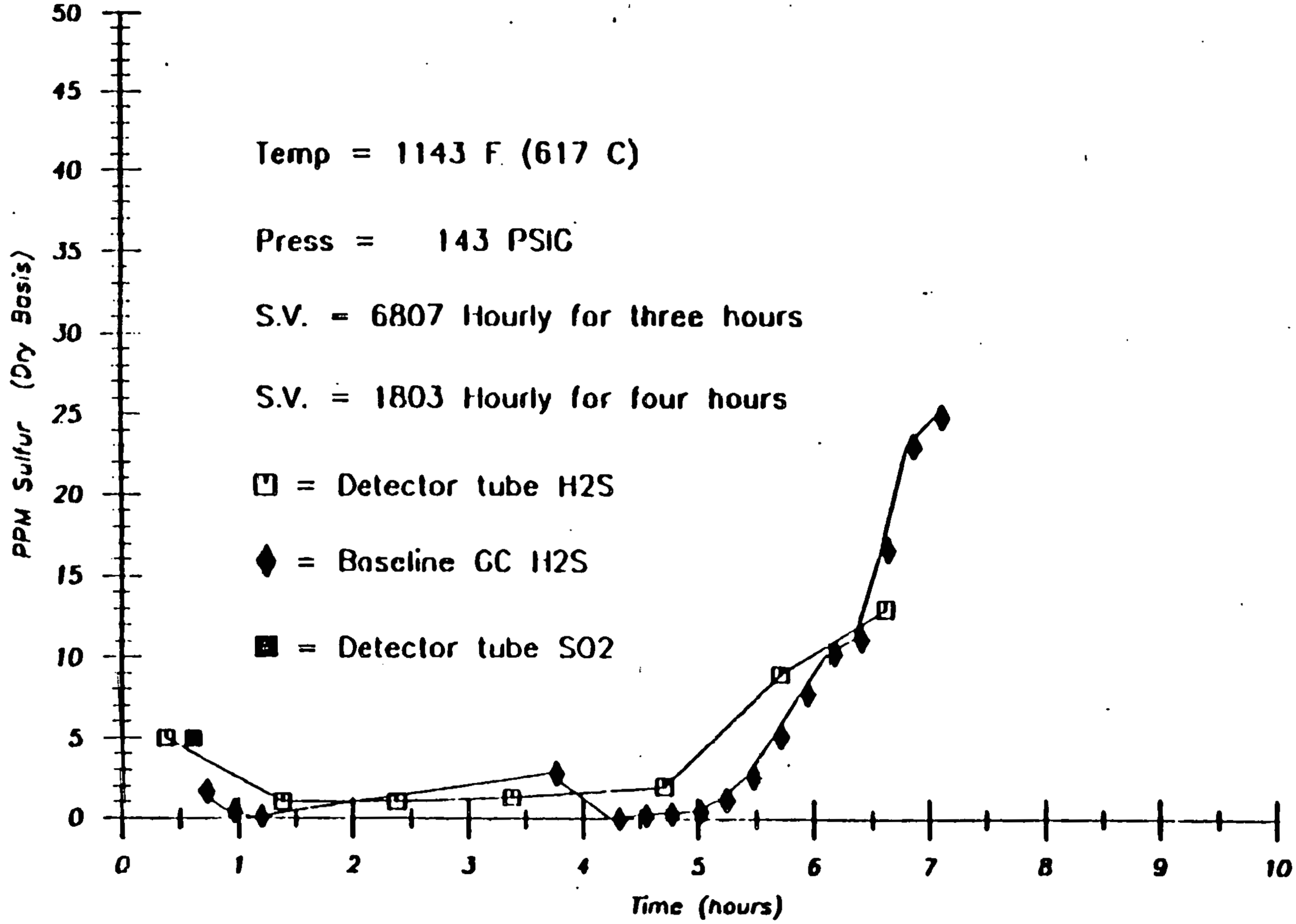


Figure 39

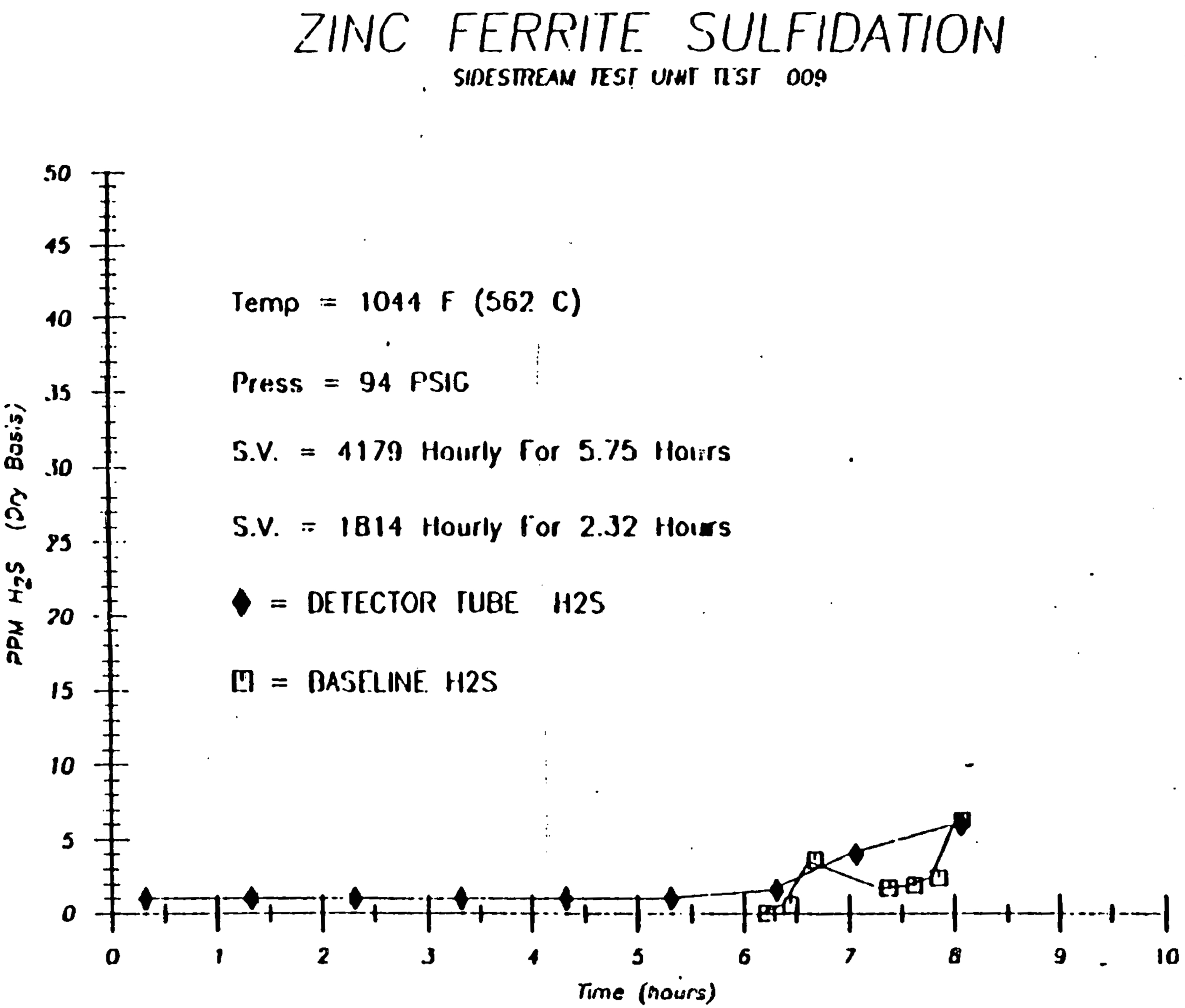

ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 009
Temp = 1044 F (562 C)
Press = 94 PSIG
S.V. = 4179 Hourly For 5.75 Hours
S.V. = 1814 Hourly For 2.32 Hours
♦ = DETECTOR TUBE H2S
□ = BASELINE H2S
PPM H2S (Dry Basis)
Time (hours)
0
5
10
15
20
25
30
35
40
45
50
1
2
3
4
6
7
8
9

Figure 40

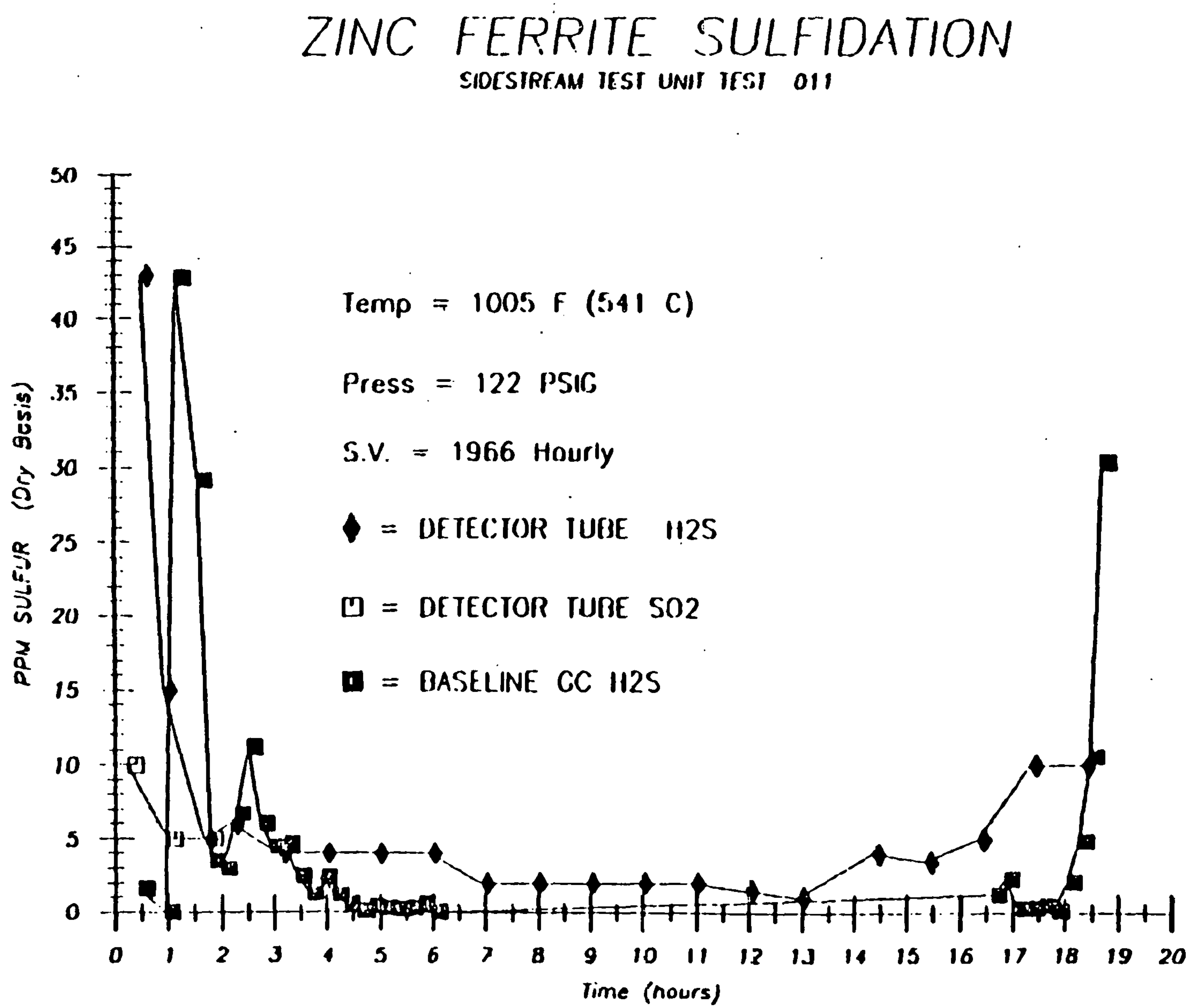

ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 011
Temp = 1005 F (541 C)
Press = 122 PSIG
S.V. = 1966 Hourly
= DETECTOR TUBE H2S
= DETECTOR TUBE SO2
= BASELINE GC H2S
PPM SULFUR (Dry Basis)
0
5
10
15
20
25
30
35
40
45
50
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
Time (hours)

Figure 41

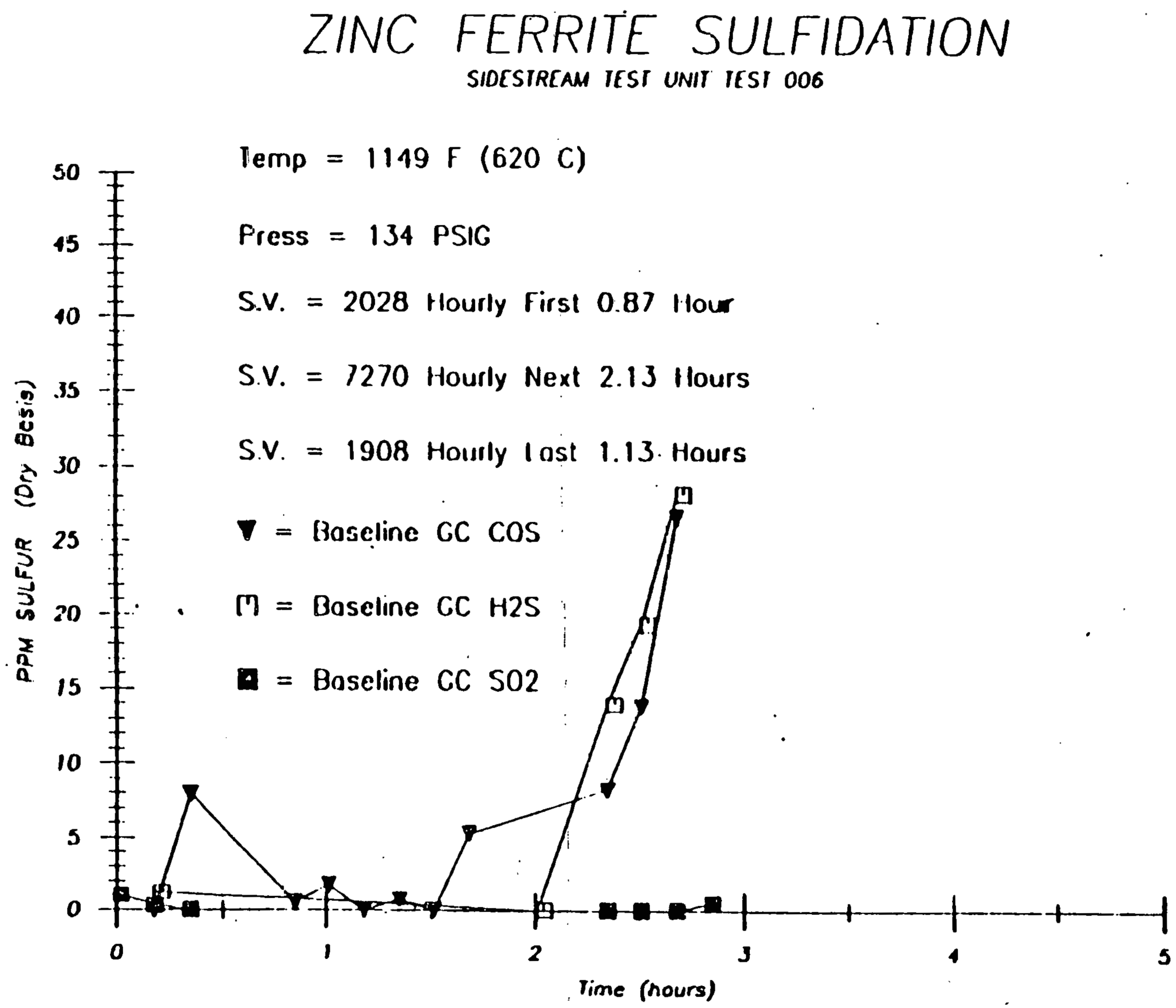
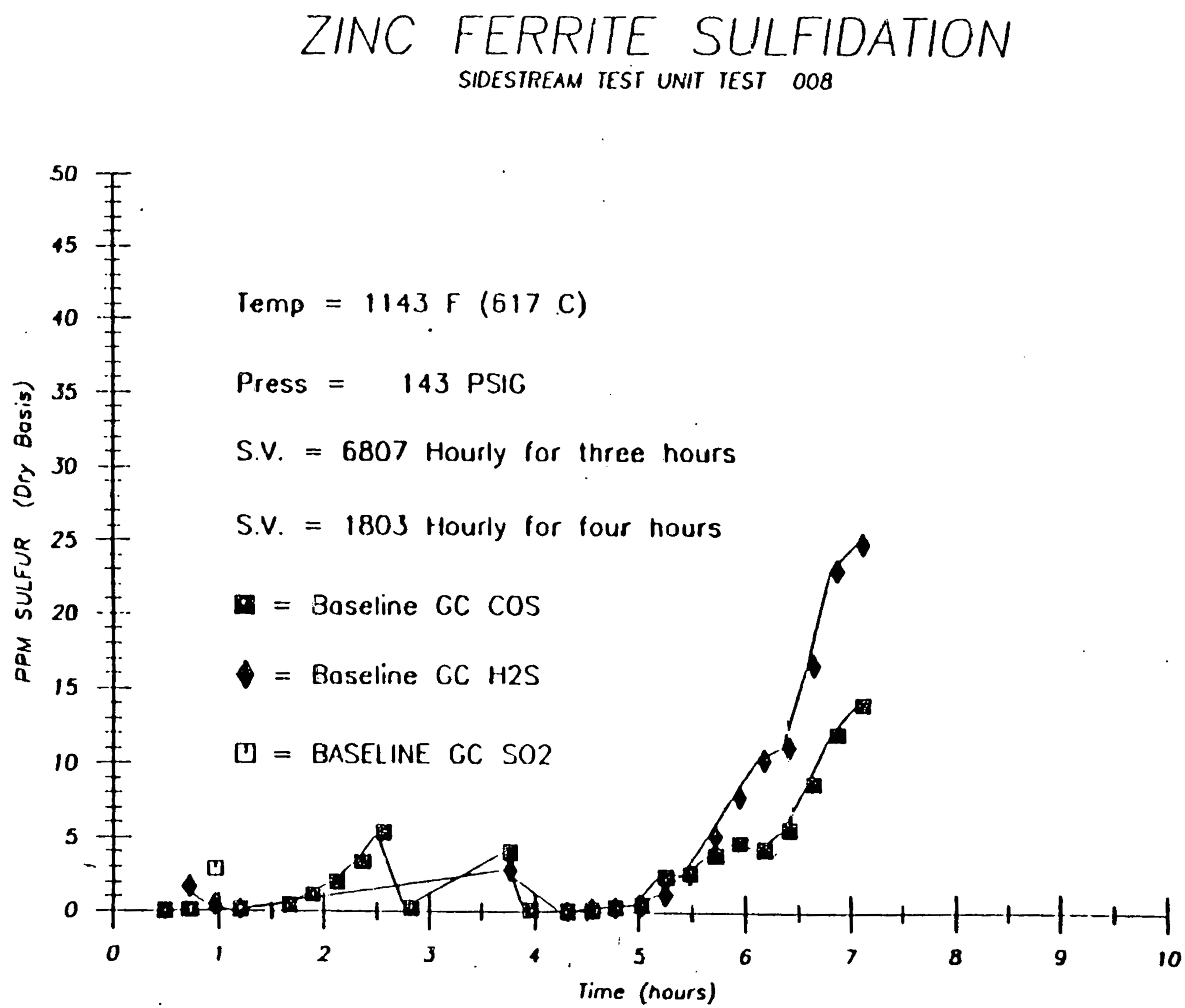

ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 006
Temp = 1149 F (620 C)
Press = 134 PSIG
S.V. = 2028 Hourly First 0.87 Hour
S.V. = 7270 Hourly Next 2.13 Hours
S.V. = 1908 Hourly Last 1.13 Hours
▼ = Baseline GC COS
□ = Baseline GC H2S
■ = Baseline GC SO2
PPM SULFUR (Dry Basis)
0
5
10
15
20
25
30
35
40
45
50
0
1
2
3
4
5
Time (hours)

Figure 42



ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 008
Temp = 1143 F (617 C)
Press = 143 PSIG
S.V. = 6807 Hourly for three hours
S.V. = 1803 Hourly for four hours
= Baseline GC COS
= Baseline GC H2S
= BASELINE GC SO2
PPM SULFUR (Dry Basis)
50
45
40
35
30
25
20
15
10
5
0
0
1
2
3
4
5
6
7
8
9
10
Time (hours)

re 43

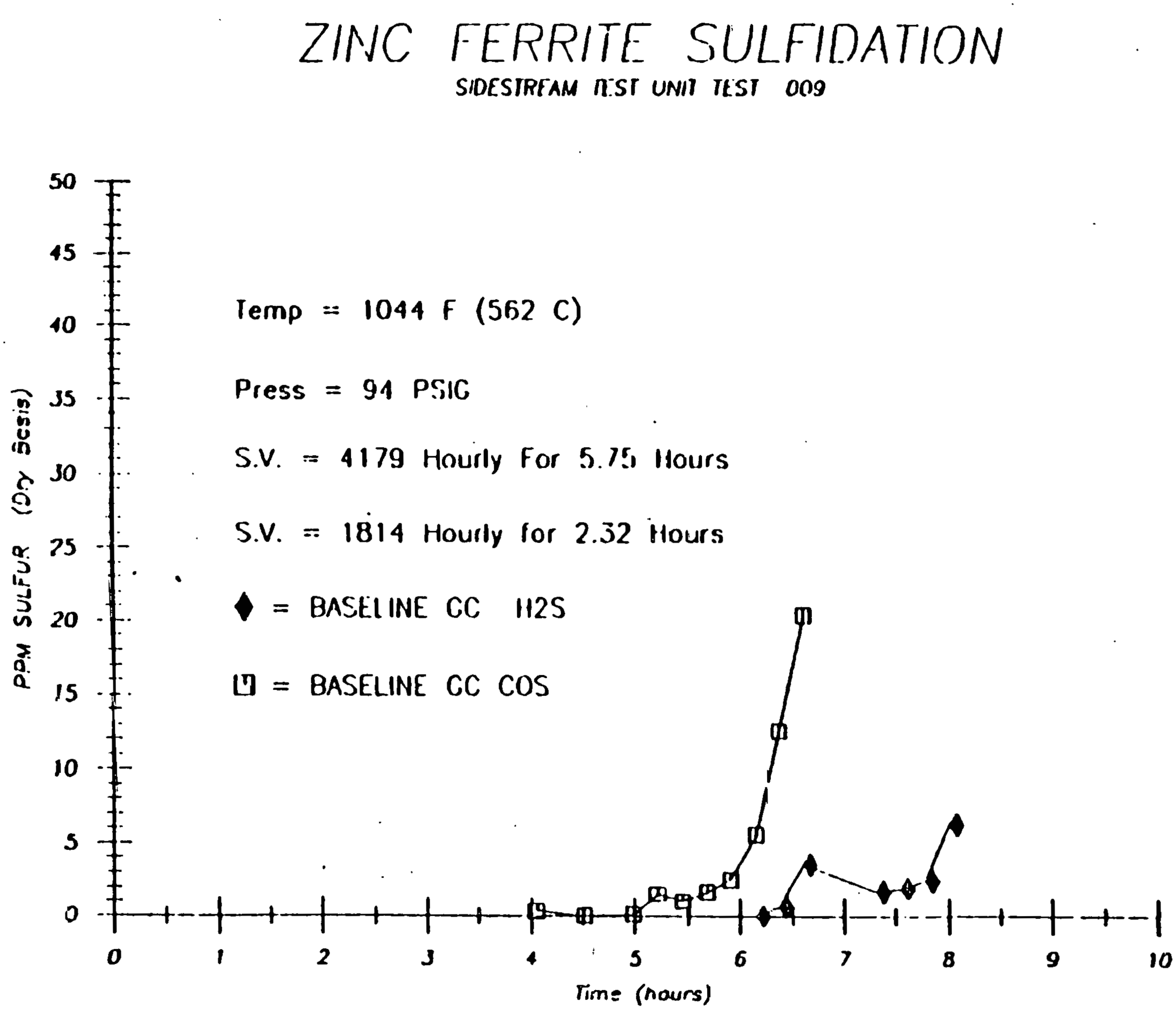

ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 009
Temp = 1044 F (562 C)
Press = 94 PSIG
S.V. = 4179 Hourly For 5.75 Hours
S.V. = 1814 Hourly for 2.32 Hours
= BASELINE GC H2S
= BASELINE GC COS
PPM SULFUR (Dry Basis)
Time (hours)
0
5
10
15
20
25
30
35
40
45
50
1
2
3
4
6
7
8
9

Figure 44

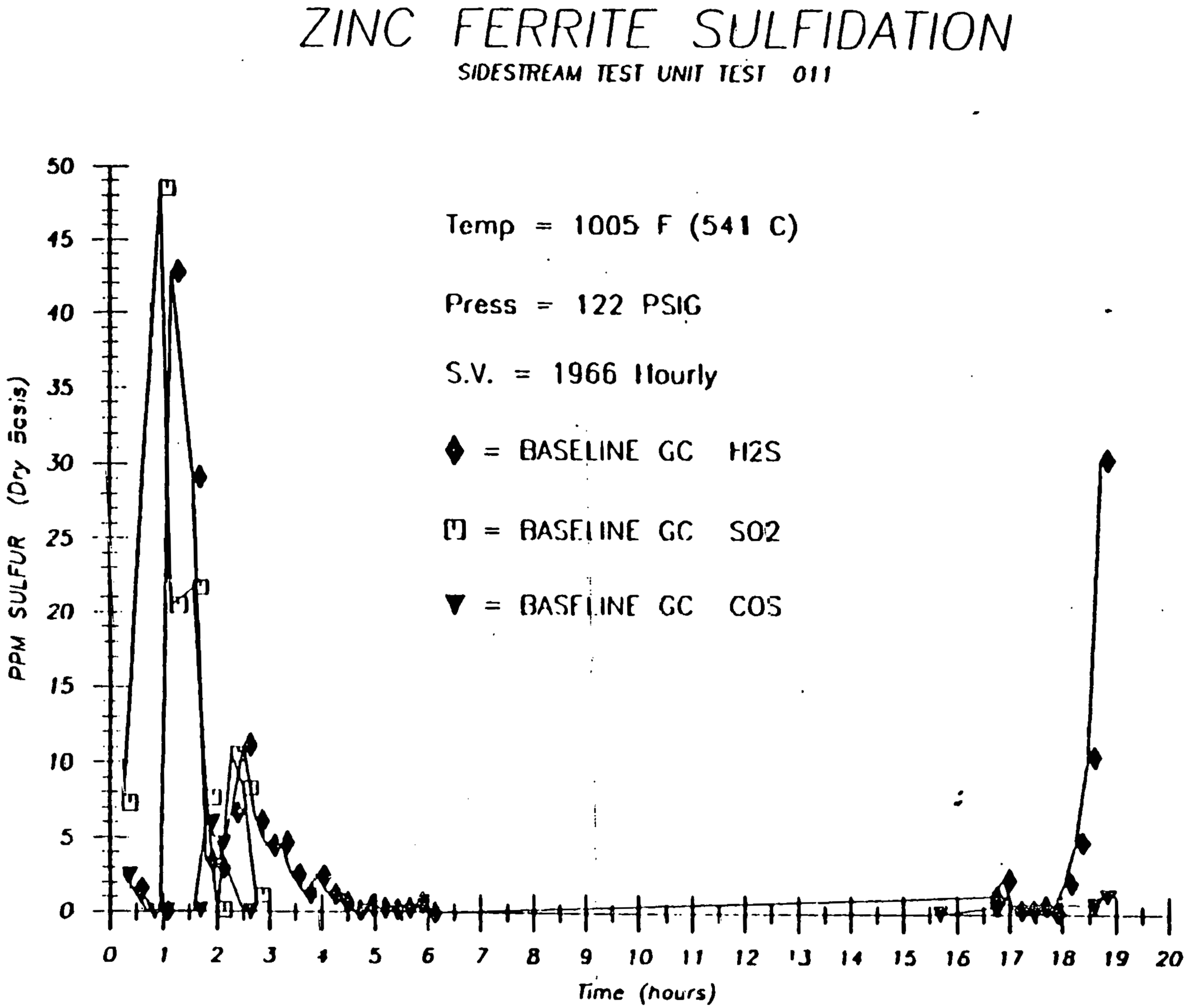

ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 011
Temp = 1005 F (541 C)
Press = 122 PSIG
S.V. = 1966 Hourly
= BASELINE GC H2S
= BASELINE GC SO2
= BASELINE GC COS
PPM SULFUR (Dry Basis)
Time (hours)
0
5
10
15
20
25
30
35
40
45
50
1
2
3
4
6
7
8
9
11
12
13
14
16
17
18
19

Table 1A

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT EXIT
GASIFIER RUN 102
TRACE COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2S	COS	CH_3SH	CS_2 PPM	Thio	NH_3	SO_2	H_2O
T006 SULFI- DATION	HG 1	9/20/83 14:30	3.84	12.4	ND	ND	6.77	70.4	ND	-
T008	HG 2	9/21/83 13:10	ND	0.51	ND	0.39	1.50	112.2	ND	5560.9
SULFI-	HG 3	9/21/83 14:10	ND	4.71	ND	0.46	1.27	432.9	ND	7771.59
DATION	HG 4	9/21/83 21:00	<.2	14.7	ND	0.24	0.96	260.4	ND	WATER IN SAMPLE
T009	HG 5	9/29/83 13:15	TRACE	ND	ND	TRACE	4.70	13.0	ND	2887.8
SULFI-	HG 6	9/29/83 14:15	ND	TRACE	ND	1.18	3.51	ND	ND	2433
DATION	HG 7	9/29/83 16:45	ND	5.71	0.06	1.42	3.34	ND	ND	1816.6
T010	HG 8	9/30/83 01:45							ND	
REGENER-	HG 9	9/30/83 03:15	3743	51.4	ND	59.6	ND	ND.	~7550	11,769
ATION	HG 10	9/29/83 23:45	>4000	62.7	10.9	36.4	3.0	99.3	116.2	6169

NOTES:
1. ND = Not Detectable.
2. Detectable level for H_2S. COS, CH_3SH, Thiophene, SO_2 is 0.2 ppm.

Table 1B

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT INLET
GASIFIER RUN 102
TRACE COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2S	COS	CH_3SH	CS_2 PPM	Thio	NH_3	SO_2	H_2O
T006	FN-15	9/20/83 12:00	--	--	--	--	--	--	--	1083
SULFI-DATION	FN-17	9/20/83 14:20	3736	356	ND	6	ND	ND	--	732
T008 SULFI-DATION	FN-41	9/21/83 13:35	4031	328	ND	5	ND	ND	--	510.7
T009	FN-93	9/29/83 13:45	4502	383	15	11	ND	--	--	660
SULFI-DATION	FN-97	9/29/83 15:15	4409	359	13	11	ND	--	--	555
T011	FN-111	10/1/83 06:55	4362	398	3	12	ND	--	--	1050
SULFI-DATION	FN-116	10/1/83 10:23	4564	466	10	10	3	--	--	912

NOTES:
1. ND = Not Detectable
2. Detectable level for H_2S, COS, CH_3SH, Thiophene, SO_2 is 0.2 ppm.

Table 2

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 006

AVERAGE GC READINGS

DURATION

FROM		TO	
12: 2: 0	9/20/1983	16:10: 0	9/20/1983

GASIFIER CYCLONE EXIT

BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
25	15.573	14.709	0.256	0.314	0.993	44.366	3.110	28.521	107.842	28.21	1.261	0.975	180.58	169.14

HGD INLET

BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
22	16.141	12.450	0.238	0.094	0.970	44.906	2.592	34.007	111.398	28.74	1.284	0.993	193.09	182.02

Table 3A

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 008A
AVERAGE GC READINGS

DURATION

FROM		TO	
12:17: 0	9/21/1983	14: 0: 0	9/21/1983

GASIFIER CYCLONE EXIT — BENDIX 7000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
10	15.509	14.450	0.250	0.406	0.996	44.890	2.992	24.642	104.174	27.16	1.214	0.939	167.16	155.83

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
9	9.700	5.564	0.243	0.208	0.401	61.823	1.736	16.734	96.408	25.20	1.126	0.871	108.69	101.63

Table 3B

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 008B

AVERAGE GC READINGS

DURATION

FROM		TO	
14:34: 0	9/21/1983	15:52: 0	9/21/1983

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
6	15.448	14.509	0.246	0.389	0.998	44.384	3.104	24.569	104.147	27.18	1.215	0.940	167.69	156.29

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
6	16.208	6.636	0.309	0.220	0.608	47.068	3.078	24.908	99.036	24.27	1.084	0.838	171.03	159.27

Table 3C

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 008C
AVERAGE GC READINGS

DURATION

FROM		TO	
18:29: 0	9/21/1983	20:27: 0	9/21/1983

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
11	15.080	13.299	0.207	0.303	0.998	41.743	2.774	24.966	99.370	25.77	1.152	0.891	163.17	152.39

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
5	9.220	1.114	0.104	0.113	0.273	68.347	1.287	13.240	93.718	23.90	1.067	0.825	88.22	62.11

Table 4

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 009

AVERAGE GC READINGS

DURATION

FROM		TO	
11:45: 0	9/29/1983	19:50: 0	9/29/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
36	21.367	11.794	-0.005	-0.001	0.864	47.598	2.089	15.828	99.524	24.00	1.073	0.830	141.49	128.72

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
48	17.141	10.030	0.160	0.583	0.655	47.062	2.396	22.247	100.273	25.02	1.118	0.865	158.25	146.75

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
41	16.479	9.658	0.194	0.378	0.610	48.060	2.078	21.731	99.189	24.85	1.110	0.859	150.51	139.70

Table 5

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 011

AVERAGE GC READINGS

DURATION

FROM		TO	
16:40: 0	9/30/1983	11:30: 0	10/ 1/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
113	26.374	11.378	0.143	0.000	0.725	50.819	2.366	15.464	107.269	24.76	1.107	0.856	162.04	146.27

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
109	17.512	9.272	0.191	0.586	0.634	45.892	2.543	23.182	99.930	24.66	1.102	0.852	164.53	152.64

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
112	16.072	8.912	0.201	0.361	0.621	47.959	2.196	23.262	99.583	24.93	1.114	0.862	155.31	144.59

Table 6

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 010

AVERAGE GC READINGS

DURATION

FROM	TO
21:40: 0 9/29/1983	15:30: 0 9/30/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
57	3.055	11.754	0.339	-0.001	0.273	78.358	-0.116	1.165	95.088	27.73	1.239	0.959	20.92	18.76

Table 7

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 012

AVERAGE GC READINGS

DURATION

FROM 13:20: 0 10/ 1/1983

TO 16:20: 0 10/ 2/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
1	-0.150	0.160	0.418	-0.005	0.038	76.296	0.317	9.144	86.219	24.19	1.080	0.835	39.63	38.76

TABLE 8A

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT EXIT
GASIFIER RUN 102
MAJOR COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2	O_2	N_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	iC_4	nC_4
						VOLUME %							
T008	HG 2	9/21/83 13:10	8.58	7.62	59.14	1.65	13.75	0.33	8.29	0.09	0.05	ND	ND
SULFI-	HG 3	9/21/83 14:10	5.18	8.89	61.96	2.27	14.13	0.24	6.68	0.06	0.09	ND	ND
DATION	HG 4	9/21/83 21:00	5.77	8.99	61.41	1.94	14.60	0.15	6.44	0.09	0.10	ND	ND
T009	HG 5	9/29/83 13:15	1.55	10.3	68.7	1.12	8.21	.13	9.36	.05	0.07	ND	ND
SULFI-	HG 6	9/29/83 14:15	6.05	5.87	62.59	1.95	10.56	.19	12.12	.10	.08	ND	ND
DATION	HG 7	9/29/83 16:45	20.99	.60	45.77	2.44	16.75	.19	12.56	.13	.08	ND	ND
REGENER-	HG 9	9/30/83 03:45	ND	7.05	89.36	ND	ND	3.0	ND	ND	ND	ND	ND
ATION	HG 10	9/29/83 23:45	1.91	7.15	75.91	.01	ND	ND	14.02	ND	ND	ND	ND

NOTES:
1. ND = Not Detectable.

Table 8B

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT INLET
GASIFIER RUN 102
MAJOR COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2	O_2	N_2	CH_4 VOLUME %	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	iC_4	nC_4
T006	FN-15	9/20/83 12:00	16.16	0.70	48.02	3.13	24.69	0.32	6.30	0.15	0.02	ND	ND
SULFI-DATION	FN-19	9/20/83 14:20	16.77	0.69	46.79	3.22	24.56	0.26	6.51	0.17	0.53	ND	ND
T008 SULFI-DATION	FN-41	9/21/83 13:35	17.30	0.63	47.28	3.10	24.33	0.23	6.42	0.05	0.13	ND	ND
T009	FN-93	9/29/83 13:45	17.29	0.63	47.54	2.42	22.47	0.21	8.69	0.12	0.13	ND	ND
SULFI-DATION	FN-97	9/29/83 15:15	17.06	0.62	48.24	1.80	22.13	0.17	9.28	0.07	0.12	ND	ND
T011	FN-111	10/1/83 06:35	15.72	0.68	50.05	2.57	21.97	0.17	8.10	0.13	0.11	ND	ND
SULFI-DATION	FN-116	10/1/83 10:25	15.17	0.71	49.69	2.67	22.29	0.09	8.60	0.15	0.12	ND	ND

NOTES:
1. ND = Not Detectable.

TABLE 9

ANALYSIS OF MINERALS BY X-RAY DIFFRACTION IN SULFIDED ZINC FERRITE GASIFIER RUN 102

BED HEIGHT (INCHES)	HEMATITE Fe_2O_3	FRANKLINITE $ZnFe_2O_4$ OR MAGNETITE Fe_3O_4 (4)	ZINCITE ZnO	ALPHA ZnS	BETA ZnS	PYRROHITE FeS	CEMENTITE Fe_3C
		PERCENT TII (3)					
0 (1)			10.9	29.1	38.1	10.1	11.9
6			11.5	32.6	33.4	8.7	13.9
16			15.9	28.6	27.4	5.3	22.7
23			27.6	18.0	19.1	1.3	34.0
26			39.5	10.3	9.8	-	40.4
36			38.4	12.2	9.8	-	39.6
46 (2)			58.6	-	1.1	-	40.3
FRESH SORBENT	4.	89.	6.0	-	-	-	-

NOTES:

(1) Bottom/Inlet.

(2) Top/Outlet.

(3) Percent TII = Percent Total Integrated Intensity; Directly proportional to the concentration of the crystalline phase. Amorphous phase is not accounted for.

(4) Franklinite and Magnetite have the same crystal structure and, therefore, cannot be readily distinguished by the diffraction pattern.

TABLE 10
SORBENT ANALYSIS OF SULFIDED ZINC
FERRITE GASIFIER RUN 102
TEST 008

Bed Height (Inches)	Total Carbon (% Wt)	Total Sulfur (% Wt)	BET Surface Area (SO M/G)	Skeletal Density (G/CC)(3)	Porosity %(4)	Crush Strength (Kg DWL) ± S.D.(5)	Skeletal Density (G/CC)(6)	Porosity %(7)
0(1)	4.88	24.61		4.39		3.1 ± 0.9	-	-
6	5.86	24.41					-	-
16	7.51	19.67	4.68	4.57	4.22	1.7 ± 1.6	4.50	46.9
23	7.65	13.15					-	-
26	7.67	7.11	4.72	5.58	6.45	1.6 ± 1.6	4.31	53.2
36	8.18	7.22	6.53	5.72	6.36	1.6 ± 1.3	-	-
46(2)	7.98	0.28	4.46	6.20	7.73	1.2 ± 0.9	5.61	69.4
								-
Fresh Sorbent	0	0.13	3.13	6.16	2.59	6.3 ± 2.8	6.16	60.6(8)

NOTES:

1. Bottom/Inlet during sulfidation - Outlet during regeneration.
2. Top/Outlet during sulfidation - Inlet during regeneration.
3. Skeletal Density by Helium Pycnometry.
4. Porosity by Nitrogen Adsorption for pores up to 600Å diameter.
5. Average and standard deviation of 15 measurements.
6. Skeletal density by mercury porosimetry.
7. Porosity by mercury porosimetry for pores down to 120Å diameter.
8. Porosity by mercury porosimetry for pores down to 30Å diameter.

TABLE 11

ANALYSIS OF MINERALS BY X-RAY DIFFRACTION IN REGENERATED ZINC FERRITE GASIFIER RUN 102

BED HEIGHT (INCHES)	HEMATITE Fe_2O_3	FRANKLINITE $ZnFe_2O_4$ OR MAGNETITE Fe_3O_4 (4)	ZINCITE ZnO	ALPHA ZnS	BETA ZnS	PYRROHITE FeS
		PERCENT TII (3)				
0 (1)	4.9	93.3	1.9			
6	4.4	93.0	2.6			
16	3.8	93.7	2.5			
23	3.9	93.7	2.5			
26	3.1	94.0	2.9			
36	2.7	93.4	3.9			
46 (2)	2.0	92.3	5.8			
FRESH SORBENT	4.	89.	6.0			

NOTES:

(1) Bottom/Inlet during sulfidation - exit during regeneration.

(2) Top/Outlet during sulfidation - inlet during regeneration.

(3) Percent TII = Percent Total Integrated Density; Directly proportional to the concentration of the crystalline phase. Amorphous phase is not accounted for.

(4) Franklinite and Magnetite have the same crystal structure and, therefore, cannot be readily distinguished by the diffraction pattern.

TABLE 12

SORBENT ANALYSIS OF REGENERATED ZINC FERRITE GASIFIER RUN 102

BED HEIGHT (INCHES)	TOTAL CARBON (% WT)	TOTAL SULFUR (% WT)	%SULFUR AS SULFATE SO_4	%SULFUR AS SULFIDE S	BET SURFACE AREA (SQ M/G)	DENSITY (G/CC)[3]	PORE VOLUME (CC/G)[4]	CRUSH STRENGTH (Kg DWL) ± S.D.[5]
0[1]	0.01	1.99						
0-6			1.21	0.0006	1.9591	5.582	0.003666	7.45 ± 3.41
6	0.08	1.43						
6-16			0.66	0.0033	1.9756	5.645	0.002679	5.66 ± 2.99
16	0.02	1.33						
23	0.01	1.36	0.71	0.0012	2.0393	5.567	0.003835	5.42 ± 3.50
26	0.01	0.97						
26-36			0.42	0.0008	1.6614	5.572	0.002274	4.20 ± 4.21
36	0.02	0.59						
36-46			0.20	0.0096	1.5306	5.769	0.001548	3.88 ± 3.68
46[2]	0.03	0.14						
FRESH SORBENT	0	0.13			3.1305	6.159	0.004316	6.3 ± 2.8

NOTES:
(1) Bottom/Inlet during sulfidation - Exit during regeneration.
(2) Top/Outlet during sulfidation - Inlet during regeneration.
(3) Helium Density.
(4) Nitrogen Adsorption.
(5) Average and standard deviation of 15 measurements.

TABLE 13

PARTICULATE SAMPLING DATA
HOT GAS DESULFURIZATION TEST UNIT
GASIFIER RUN 102

TEST #	HOURS FROM START OF TEST	PARTICLE LOADING g/Nm^3 INLET	PARTICLE LOADING g/Nm^3 EXIT	MEDIAN PARTICLE SIZE um (microns) INLET	MEDIAN PARTICLE SIZE um (microns) EXIT	CHEMICAL ANALYSIS WT % C	H	N	S	ASH
006	1.18	0.74		21.5		77.1	4.5	2.2	2.0	2.5
008	0.16	0.72		25.5		85.7	4.6	2.1	2.2	5.9
009	1.25		0.53		26.6	NM	NM	NM	NM	NM
011	6.33		0.34		20.9	58.5	3.2	1.4	1.5	33.8

NOTES:
1. NM = Not Measured due to insufficient sample size. Particles embedded in ceramic filter.

Table 14

DATE: 10/27/83
SAMPLE: DE BAGNELL 6I9201 83-872
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 280
OPERATOR:RDB

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	4	.1	100
2	5.04	2.1	99.9
3	6.35	2.3	97.8
4	8	3.4	95.5
5	10.08	7	92.1
6	12.7	13.5	85.1
7	16	17.3	71.6
8	20.16	16.6	54.3
9	25.4	13.9	37.7
10	32	9.3	23.8
11	40.32	7.1	14.5
12	50.8	2.8	7.4
13	64	2.8	4.6
14	80.63	.1	1.8
15	101.59	1.8	1.7
16	128	.1	0

PARTICULATE SIZE DISTRUTION

TEST 006 INLET

VOLUME % STATISTICS

MEAN:	22.1 UM
MEDIAN:	21.45 UM
MODE:	19.48 UM
STANDARD DEVIATION:	1.8 UM
SKEWNESS:	1.05 POSITIVE
KURTOSIS:	1.5 PLATYKURTIC

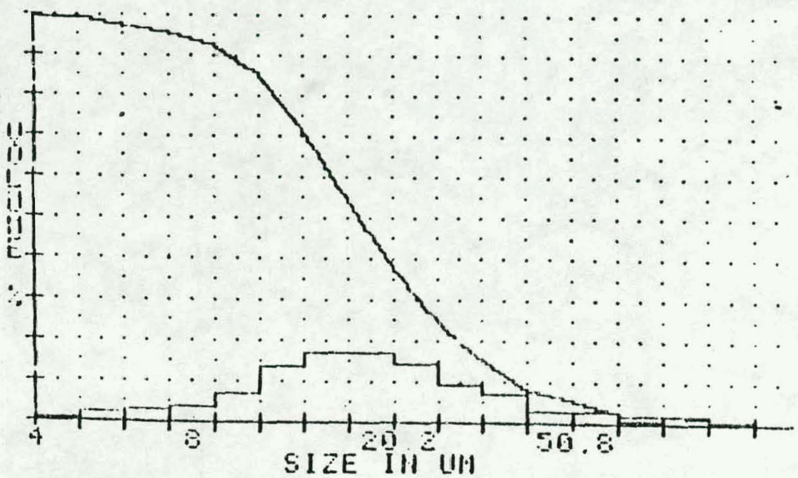


Table 15

DATE: 10/27/83
SAMPLE: DE BAGNELL 200 T2 83-881
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 200
OPERATOR:ROB

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	2.52	0	100
2	3.18	2.7	100
3	4	1.9	97.3
4	5.04	2.1	95.4
5	6.35	2.6	93.3
6	8	3.5	90.7
7	10.08	6.1	87.2
8	12.7	8.5	81.1
9	16	11.1	72.6
10	20.16	11.3	61.5
11	25.4	8.2	50.2
12	32	8.7	42
13	40.32	6.2	33.3
14	50.8	7.8	27.1
15	64	1.2	19.3
16	80.64	1.5	18.1
17	101.6	4.3	16.6
18	128.01	4.7	12.3
19	161.28	4.2	7.6
20	203.2	3.3	3.4
21	256.02	.1	.1
22	322.56	.1	0

PARTICULATE SIZE DISTRIBUTION

TEST 008 INLET

VOLUME % STATISTICS

MEAN:	29.14 UM
MEDIAN:	25.54 UM
MODE:	20.44 UM
STANDARD DEVIATION:	2.77 UM
SKEWNESS:	1.26 POSITIVE
KURTOSIS:	16.07 LEPTOKURTIC

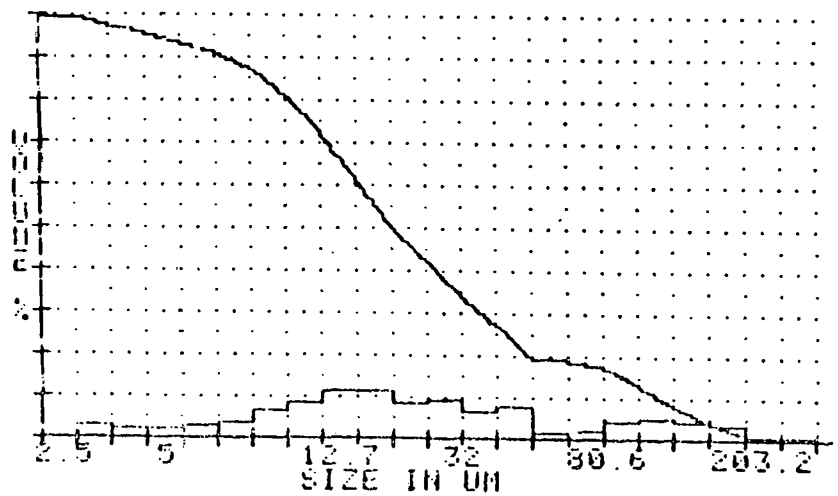


Table 16

DATE: 10/31/83
SAMPLE: CMM BAGHELL 609-29-3 83-873
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 280
OPERATOR:REC

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	4	0	100
2	5.04	3.1	100
3	6.35	3.5	96.9
4	8	4.6	93.4
5	10.08	7.1	88.8
6	12.7	9.2	81.7
7	16	10.7	72.5
8	20.16	9.4	61.8
9	25.4	11.6	52.4
10	32	10.7	40.8
11	40.32	7.1	30.1
12	50.8	5.6	23
13	64	3.4	17.4
14	80.63	4.3	14
15	101.59	4.1	9.7
16	128	5.6	5.6

PARTICULATE SIZE DISTRIBUTION

TEST 009 EXIT

VOLUME % STATISTICS

MEAN:	27.72 UM
MEDIAN:	26.62 UM
MODE:	29.93 UM
STANDARD DEVIATION:	2.31 UM
SKEWNESS:	1.14 NEGATIVE
KURTOSIS:	3.2 LEPTOKURTIC

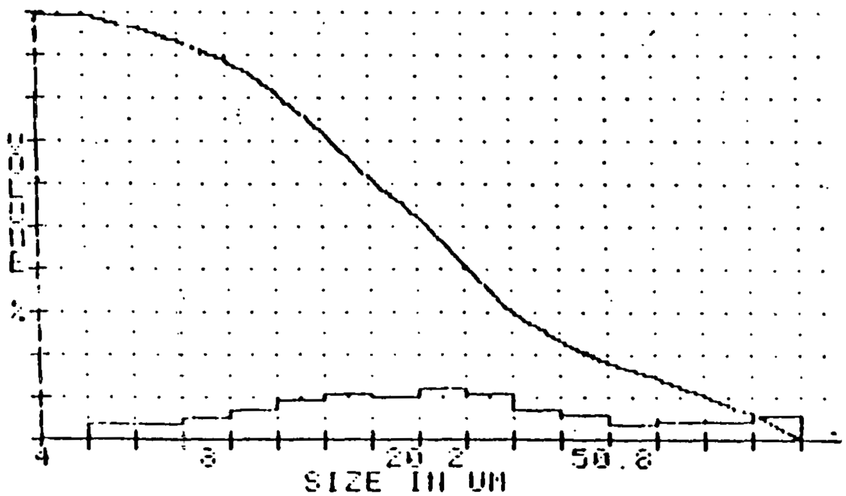


Table 17

SAMPLE: DE BAGNELL 280 609304 83-874
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 280
OPERATOR:ROB

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	4	.1	100
2	5.04	5.4	99.9
3	6.35	6	94.5
4	8	7	88.5
5	10.08	9	81.5
6	12.7	10.9	72.5
7	16	10.3	61.6
8	20.16	7.3	51.3
9	25.4	7.7	44
10	32	8.6	36.3
11	40.32	6.8	27.7
12	50.8	6.6	20.9
13	64	5.5	14.3
14	80.63	3.3	8.8
15	101.59	5.2	5.5
16	128	.1	.3

PARTICULATE SIZE DISTRIBUTION

TEST 011 EXIT

VOLUME % STATISTICS

MEAN:	22.93 UM
MEDIAN:	20.91 UM
MODE:	15.16 UM
STANDARD DEVIATION:	2.32 UM
SKEWNESS:	1.12 POSITIVE
KURTOSIS:	2.83 PLATYKURTIC

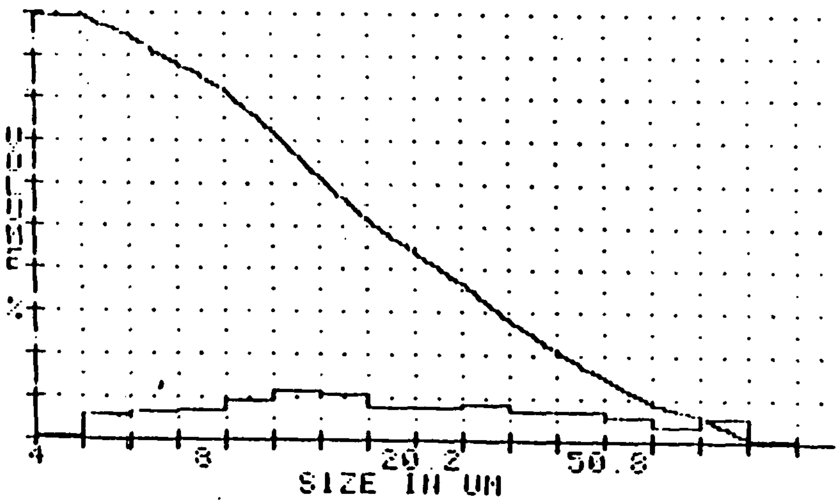


Table 18

DATE: 11/1/83
SAMPLE: HGDS BAGWELL ARKWRIGHT DUST 83-876
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 560
OPERATOR:REC

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	8	0	100
2	10.08	2.6	100
3	12.7	3.1	97.4
4	16	2.3	94.3
5	20.16	2.9	92
6	25.4	4.7	89.1
7	32	7.6	84.4
8	40.32	10.4	76.8
9	50.8	16.3	66.4
10	64	19.9	50.1
11	80.63	16.1	30.2
12	101.59	8.7	14.1
13	128	3.6	5.4
14	161.27	.7	1.8
15	203.19	1.1	1.1
16	256	0	0

PARTICULATE SIZE DISTRIBUTION

ARKWRIGHT DUST

VOLUME % STATISTICS

MEAN:	57.5 UM
MEDIAN:	64.13 UM
MODE:	71.66 UM
STANDARD DEVIATION:	1.84 UM
SKEWNESS:	-.85 NEGATIVE
KURTOSIS:	1.62 PLATYKURTIC

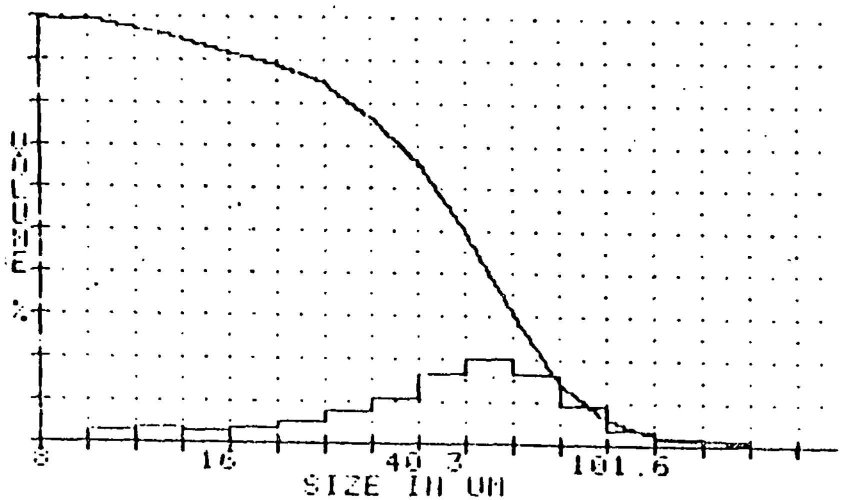


Table 19

DATE: 11/1/83
SAMPLE: HGDS BAGHELL BRIQUETTE DUST 83-878
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 560
OPERATOR:REC

CH.#	SIZE	DIFF VOL. %	CUM VOL %
1	8	0	100
2	10.08	2.8	100
3	12.7	6.4	97.2
4	16	6	90.8
5	20.16	8.7	84.8
6	25.4	13.2	76.1
7	32	16.5	62.9
8	40.32	16	46.4
9	50.8	14.6	30.4
10	64	9.5	15.8
11	80.63	4.5	6.3
12	101.59	1.1	1.8
13	128	.4	.7
14	161.27	.1	.3
15	203.19	.1	.2
16	256	.1	.1

PARTICULATE SIZE DISTRIBUTION

BRIQUETTE DUST

VOLUME % STATISTICS

MEAN:	37.06 UM
MEDIAN:	38.35 UM
MODE:	39.17 UM
STANDARD DEVIATION:	1.73 UM
SKEWNESS:	.98 NEGATIVE
KURTOSIS:	1.28 PLATYKURTIC

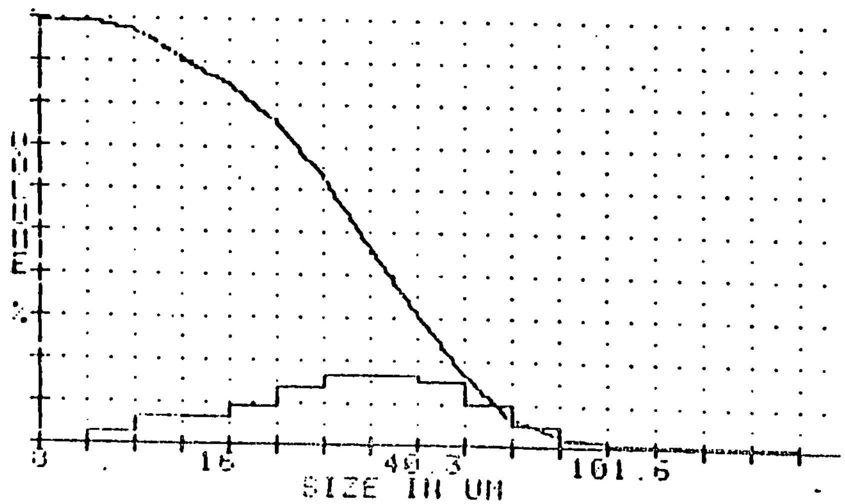


Table 20

ULTIMATE ANALYSIS OF COALS
USED DURING GASIFIER RUN 102

COMPONENT	WT %		
	PITTSBURGH SEAM COAL		
	ARKWRIGHT	BLACKSVILLE	BLACKSVILLE BRIQUETTES
Moisture	0.39	1.1	0.96
Ash	7.15	10.55	15.33
S	1.82	3.20	2.39
H	5.12	5.58	1.72
N	3.05	1.30	1.29
Total Carbon	77.10	73.17	70.49
Volatiles	38.35	38.25	34.14
Btu/lb	13715	13310	12615

Table 21

TRACE COMPONENTS IN EXIT GAS
DURING SULFIDATION
GASIFIER RUN 102

	TESTS 006 AND 008	
COMPONENT	AQUEOUS CONDENSATE ANALYSIS ppmw	CALCULATED GAS PHASE COMPOSITION ppmw
Ni	4.15	0.221
V	<0.05	<0.003
Cr	1.21	0.064
K	3.73	0.199
Na	3.06	0.064
Fe	11.68	0.622
Zn	4.89	0.260
Ca	1.77	0.094
Al	0.382	0.020
Se	0.005	0.0002
As	1.52	0.081
Hg	<0.2	<0.011
F	64	3.40
Cl	1899	101.02
Br	10.8	0.574
NO_3	13.2	0.702
SO_4	332	17.662
NO_4	<3	<0.159
NH_4	10074	535.93
Cu	<0.02	<0.001

NOTES:

1. Aqueous condensate analysis from Sample # 100011.
2. Sampling location is at condenser CIC knockout.
3. Sample is a composite sample of HGD tests 006 and 008.
4. Metals determined by atomic absorption/atomic emission spectroscopy.
5. Anions and cations determined by ion chromatography.

Table 22

COMPOSITE ULTIMATE ANALYSIS
OF EXIT TAR CONDENSATE
DURING SULFIDATION
GASIFIER RUN 102

COMPONENT	TESTS 6 AND 8 WT %
Ash	0.13
S	3.13
H	7.36
N	1.25
Total Carbon	72.94

NOTES:

1. Sample Number 28244
2. Sample Location is at Condenser CIC Knockout.

Table 23

TAR ULTIMATE ANALYSIS
AT HOT GAS DESULFURIZATION INLET

TEST # TYPE	SAMPLE NO.	DATE TIME	ASH	S	H	T.C.	N
Shortly after Test 008 Sulfidation	28338	9/22/83 04:00	0.10	0.98	8.45	66.18	1.05
Between Tests 009 and 011 Sulfidation	28340	9/30/83 14:30	0.09	1.08	7.67	66.32	1.14

NOTES:

1. Sampling location is S-4 after gasifier cyclone.
2. Sample No. 28338 obtained during operation on Oxygen Blown Arkwright coal.
3. Sample No. 28340 obtained during operation on Air-Blown Blacksville Briquettes.

TABLE 24

TRACE COMPONENTS IN INLET GAS
DURING SULFIDATION
GASIFIER RUN 102

COMPONENT	AQUEOUS CONDENSATE TEST 006	TEST 008	AVERAGE TEST 006 & 008	CALCULATED GAS PHASE COMPOSITION
	PPMW	PPMW	PPMW	PPMW
Ni	0.71	0.24	0.475	0.022
V	<0.05	<0.05	<0.05	<0.0023
Cr	4.3	2.3	3.3	0.155
K	1.5	0.79	1.9	0.089
Na	10.9	6.6	8.75	0.411
Fe	6.8	4.0	5.4	0.254
Zn	<0.1	<0.1	<0.1	<0.005
Ca	6.1	6.6	6.35	0.298
Al	1.4	2.5	1.95	0.092
Se	0.348	0.03	0.189	0.009
As	0.35	0.23	0.29	0.014
Hg	<0.2	0.23	0.215	0.010
Cu	<0.02	<0.02	<0.02	<0.0009
F	140	124	132	6.204
Cl	3445	4659	4052	190
Br	26	42	34	1.598
SO_4	155	23	89	4.183
PO_4	<3	<3	<3	<0.141
NO_3	<2	<2	<2	<0.094
NH_4	13,763	14,072	13,917	654

NOTES:

1. Gas phase composition is calculated from average of Test 006 and Test 008 aqueous condensate data and gas flow rate from which the condensate was obtained.
2. Aqueous condensate data obtained from HGD inlet particulate sampling system.
3. Test 006 aqueous condensate data from sample #GI-9-20-1.
4. Test 008 aqueous condensate data from sample #GI-9-21-2.

TABLE 25

TRACE COMPONENTS DURING SULFIDATION
GASIFIER RUN 102
HGD TESTS 006 AND 008

COMPONENT	CALCULATED GAS PHASE COMPOSITIONS INLET	EXIT	CHANGE
	PPMW	PPMW	PPMW
Ni	0.022	0.221	+0.199
V	<0.0023	0.003	+0.0007
Cr	0.155	0.064	-0.091
K	0.089	0.199	+0.11
Na	0.411	0.163	-0.240
Fe	0.254	0.622	+0.368
Zn	<0.005	0.260	+0.255
Ca	0.298	0.094	-0.204
Al	0.092	0.020	-0.072
Se	0.009	0.0002	-0.008
As	0.014	0.081	+0.067
Hg	0.010	0.011	+0.001
Cu	<0.0009	0.001	+0.0001
F	6.204	3.4	-2.8
Cl	190	101.02	-89
Br	1.598	0.574	-1.024
SO_4	4.183	17.662	+13.48
PO_4	<0.141	0.159	+0.018
NO_3	<0.094	0.702	+0.608
NH_4	654	535.93	-118

NOTES:

1. Inlet data calculated from aqueous condensate obtained at HGD inlet at particulate sampling tap. Average of samples GI-9-20-1 and GI 9-21-2 used.
2. Exit data calculated from aqueous condensate obtained on condenser CIC knockout. A composit of tests 006 and 008 was used. Sample #100011.

Table 26

TRACE COMPONENTS IN EXIT GAS CONDENSATE
DURING REGENERATION
GASIFIER RUN 102

COMPONENT	AQUEOUS CONDENSATE TEST 007 SAMPLE # 100004 ppmw	SAMPLE # 100008 ppmw	AVERAGE ppmw
Ni	1.41	1.37	1.39
V	<0.05	<0.05	<0.05
Cr	1.39	1.33	1.36
K	0.66	0.600	0.63
Na	0.71	0.641	0.67
Fe	82.4	79.0	80.2
Zn	1.83	1.77	1.8
Ca	1.54	1.43	1.48
Al	1.68	1.67	1.68
Se	0.039	<0.5 ppb	<0.02
As	0.538	1.14	0.84
Hg	<0.2	<0.2	<0.2
F	9.3	8.7	9
Cl	404	322	363
Br	<1	108	54.5
NO_3	270	<2	136
SO_4	281	259	270
PO_4	0.55	<3	<1.8
NH_4	270	200	235
Cu	0.025	<0.02	<0.022

NOTES:

1. Metals determined by atomic absorption/atomic emission spectroscopy.
2. Anions and cations determined by ion chromatography.
3. Sampling location is at condenser CIC knockout.

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Table 27 (revision)

TRACE COMPONENTS IN EXIT GAS CONDENSATE DURING REGENERATION GASIFIER RUN 102

COMPONENT	AQUEOUS CONDENSATE TEST 010 PPMW SAMPLE #100009	SAMPLE #100288	TEST 012 PPMW SAMPLE #100010	SAMPLE #100289
Ni	0.750	0.56	1.91	1.90
V	0.197	<0.05	<0.05	<0.05
Cr	1.56	1.1	3.33	3.61
K	no.sample	1.08	0.743	0.887
Na	no.sample	0.959	1.51	0.831
Fe	272.8	240.8	110.8	118.9
Zn	no.sample	6.90	2.98	3.14
Ca	no.sample	8.30	3.02	1.11
Al	no.sample	0.999	0.77	0.739
Se	<0.120	0.040	0.121	0.021
As	0.298	0.30	4.401	5.72
Hg	no.sample	0.255	<0.2	<0.2
F	26.1	25	28.9	45
Cl	695	688	545	688
Br	19.9	<4	526	415
NO_3	<2	492	<2	<2
SO_4	3059	1043	393	1469
PO_4	<3	<3	<3	<3
NH_4	226	200	261	266
Cu	<0.02	<0.02	0.022	0.031

NOTES:

1. Duplicate aqueous condensate analysis for Test 010 from sample #100009 and #100288.
2. Duplicate aqueous condensate analysis for Test 012 from sample #100010 and #100289.
3. Metals determined by atomic absorption/atomic emission spectroscopy.
4. Anions and cations determined by ion chromatography.
5. Sampling location is at condenser C1C knockout.

TABLE 28

HGD EXIT CONDENSATE
GASIFIER RUN 102

COMPONENT		IHI-KEMRON SAMPLE # HG2-2A-102 HGD TEST 011 AQUEOUS mg/l	IHI-KEMRON SAMPLE #HG2-1A-102 HGD TEST 006 HYDROCARBON mg/l
Chloride, Dissolved	(Cl)	1600	300
Cyanide, Total	(Cn)	12	88
Nitrogen, Ammonia, Total	(N)	14,000	2,500
Phenolics, Total	(Phenol)	3,200	32,000
Sulfate, Dissolved	(SO_4)	240	<100
Sulfide	(s)	530	200
Sulfite	(SO_3)	220	49
Fluoride, Total	(F)	120	85
Thiocyanate, Total	(SCN)	220	510
Arsenic, Total	(As)	0.37	0.77
Iron, Total	(Fe)	5.0	12.2
Lead, Total	(Pb)	<0.05	<0.05
Calcium, Total	(Ca)	0.8	5.9
Potassium, Total	(K)	0.32	14.5
Vanadium, Total	(V)	<0.2	<0.2
Zinc, Total	(Zn)	2.1	4.2
Sodium, Total	(Na)	-	13

NOTES:
1. Sampling location is at condenser C1C knockout.

APPENDIX G

Sidestream Testing - Gasifier Run No. 103

Hot Gas Desulfurization Sidestream Test Unit, Fixed-Bed Gasifier Run No. 103

Introduction

A third series of tests with the high-temperature, sidestream desulfurization unit (1,2) was carried out during fixed-bed gasifier Run No. 103 (3,4,5) between November 7 and 18.

The purpose of the tests was to characterize further the behavior of zinc ferrite sorbent in coal gas from various coals. A particular interest was to investigate more closely the magnitude and cause of the increase in reactor differential pressure during sulfur absorption periods.

Operation of Sidestream Test Unit

The sidestream unit was operated with a single batch of sorbent in the reactor for four sulfidation periods and three intermediate regeneration periods. The gasifier was operated in the air-blown mode and fed with Blacksville, Arkwright, and Kittanning coals. The operational schedule is shown in Figure 1.

As usual, the reactor contained 20.4 kg of zinc ferrite extrusions. Reactor furnace temperatures were set at 1,000°F throughout all the sulfidation and regeneration periods. A lower minimum space velocity than previously of 1,000 h^{-1} for the sulfidation was thought to give a more acceptable reactor pressure drop. The space velocity during the regenerations was about 500 h^{-1}.

Average operating parameters for the tests are shown on the ADACS flow schematics in Figures 2 to 13. Because of interruptions in the gasifier operation, Tests 017 and 019 are broken down into parts when the sidestream unit was on stream.

In Test No. 013, with the gasifier providing gas from Blacksville coal at about 120 psig, breakthrough of hydrogen sulfide occurred after about 46 hours during which time the H_2S level was about 5 ppm initially, but drifted to about 20 ppm later. The reactor pressure drop gradually rose from about 2 inches to more than 100 inches of water, slowly at first, but more rapidly later. Pressure drop fluctuations occurred when condensate was dumped from the reactor exit condenser. These were caused by surges of gas through the reactor and consequent movement of the sorbent extrusions. This bed movement may have caused some channeling and accounted for the higher than expected H_2S levels towards the end of the test before breakthrough. As before, an increase in reactor temperatures approaching 100°F, occurred through partial sorbent reduction and the shift reaction. Reactor temperatures, pressure drop, and gas flow rates are shown in Figures 14, 15, and 16.

Regeneration of the sorbent in Test No. 014 required about 24 hours, the steam to air volume ratio being about 85/15. Except for the reactor inlet temperature, which peaked sharply up to about 1,700°F, the reaction zone temperature reached about 1,300°F, some 100°F lower than desired. Reactor pressure drop gradually fell, more rapidly at first from about 20 to 2 inches of water. Reactor temperatures, pressure drop, and flow rates are shown in Figures 17, 18, and 19.

Test 015, the second sulfidation in which the gasifier provided gas from Arkwright coal at about 120 psig, lasted 40 hours and was terminated prematurely because of an excessive pressure drop increase from about 2 inches, initially to about 160 inches of water, finally. H_2S levels were similar to those in the first sulfidation, except initially when both H_2S and SO_2 were high because of sulfur compounds left in the sorbent after regeneration. Reactor exit condensate was dumped more frequently than in the previous sulfidation so that the resulting gas surges caused movement of the extrusions (presumably) with consequent pressure drop decrease, thus extending the test longer before an unacceptable pressure drop was reached. As in previous sulfidations, there was a small temperature rise along the reactor length. Reactor temperatures, pressure drop, and flow rate are shown in Figures 20, 21, and 22.

Test 016 was the second regeneration of the sorbent. In this case, the steam to air ratio was decreased stepwise from 85/15 initially to 75/25 during the first few hours to ensure that the temperature in the reaction zone reached the desired level of about 1,500°F. The duration was consequently rather shorter, about 20 hours. Pressure drop was about 70 inches of water for the first few hours and then rapidly fell to about 5 inches of water at the same time that the steam/air ratio was decreased, i.e., the air flow was increased. Reactor temperatures, pressure drop, and flow rates are shown in Figures 23, 24, and 25.

In Test 017, the third sulfidation of the series, gas was provided from Kittanning coal at 120 psig for about 43 hours until this coal was used up. Then, for a further 13 hours until H_2S breakthrough, the gas came from Arkwright coal at about 240 psig. H_2S levels were less than 10 ppm for the Kittanning coal gas except for a short initial period when the values were again high, resulting from residual sulfate in the sorbent; for the Arkwright coal gas H_2S levels slowly drifted up to the 20 ppm level. Very surprisingly, the reactor pressure drop remained at the low level of about 5 inches of water for the entire duration of the test. As in previous sulfidations, there was a moderate temperature increase along the reactor length because of the shift reaction. Reactor temperatures, pressure drop, and flow rate are shown in Figures 26, 27, and 28.

In Test 018, the third regeneration, the steam/air ratio was again decreased stepwise from 85/15 to 75/25 but more rapidly than before, resulting in a shorter duration of about 12 hours. The reactor pressure drop gradually fell as before but not as far and was finally about 10 inches of water compared to 5 inches previously. Reactor temperatures peaked at about 1,500°F, the design level. Reactor temperatures, pressure drop, and flow rates are shown in Figures 29, 30, and 31.

Test 019 was the fourth and final sulfidation of the test series. Gas was again derived from Arkwright coal but, in contrast to Test 015, the gasifier pressure was twice as high, at 240 psig. Because the gasifier was shut down prematurely, H_2S breakthrough did not occur and the test lasted about 24 hours. The reactor pressure drop, initially about 4 inches of water, increased at a much lower pace than in Test 015 even accounting for the lower pressure drop resulting from the higher operating pressure. H_2S levels were less than about 10 ppm. Reactor temperatures, as usual, showed a moderate increase of less than 100°F along the reactor length. Reactor temperatures, pressure drop, and flow rate are shown in Figures 32, 33, and 34.

Results

- <u>Data Summary</u>

  The various process conditions and data collected in Test Nos. 013 to 019 are summarized in Figures 35 to 41.

- <u>Absorption of Sulfur Compounds</u>

  Table 1 gives laboratory gas chromatographic analyses, including major and trace components, of grab samples of gas exiting the hot gas desulfurization unit condenser (6). Corresponding analyses for grab samples of inlet gas from the gasifier cyclone exit are given in Tables 2 and 3, covering the sulfidation tests only. The values for H_2S, COS, CS_2, and thiophene (C_4H_4S) respectively at both unit inlet and outlet during the sulfidation tests are plotted on semilog plots in Figures 42 to 45. On the average, H_2S is reduced from a few thousand ppm to a few ppm, COS from a few hundred ppm to a few ppm, and CS_2 and thiophene from a few tens of ppm to a few ppm, so that the total average level of these low molecular weight sulfur compounds is lowered to less than about 10 ppm.

  The hydrogen sulfide concentration in exit gas from the unit was also measured during the sulfidation tests by the Baseline Industries on-line gas chromatograph and by detector tubes. The values obtained are plotted against on-stream time in Figures 46 to 49, together with some values for sulfur dioxide measured by detector tubes. There is generally satisfactory agreement between the H_2S values measured by the two techniques except when they depart widely from the gas chromatograph calibration point of 3 ppm H_2S. As found previously, the H_3S and SO_2 levels in gas from regenerated sorbent are initially high but fall off fairly rapidly. The Baseline gas chromatograph values for H_2S, COS, and SO_2 are compared in Figures 50 to 53. The COS levels can be seen to be less than the H_2S levels. SO_2 levels generally are comparable with the detector tube values in Figures 46 to 49.

  The amount of sulfur absorbed before breakthrough was considerably greater than in the previous tests at higher space velocities. As a weight percentage of fresh sorbent, the amount is: Test 013, ~ 29; Test 015, ~ 23; and Test 017, ~ 14, indicating a decline of sorption efficiency with cycling. It is difficult to attribute a trend to this, however, because of the variation in sulfur content of gas from the different coals.

- <u>Gas Analysis for Major Species</u>

  In addition to the laboratory gas chromatographic analysis of major species in inlet and exit gas given in Tables 1 and 2, gas to and from the unit was analyzed by Bendix on-line chromatographs. The measurements, averaged by ADACS statistical analysis, are shown in Tables 4 to 16. The gas line to chromatograph 6,000 # 1 became partially blocked during the tests so that the readings from chromatograph 6,000 # 2 give a more reliable indication of the inlet gas composition.

  Some of the sulfidation exit samples in Table 1 indicate an excessive oxygen content, which should not exceed about 1 vol. percent. This may be because air was not completely displaced from the grab sample bottles.

  As before, it can be seen that the carbon monoxide shift reaction proceeds to a large extent, illustrated by Figures 54 to 56, based on Tables 1 and 2. There is an apparent small loss of total carbon oxides, presumably because of carbon formation.

- <u>Analysis of Regeneration Exit Gas</u>

  The regeneration exit gas (see Tables 1, 5, 7, and 13) in the first part of the regenerations contains substantial quantities of hydrogen, carbon oxides, and reduced sulfur compounds, predominantly hydrogen sulfide but with some carbonyl sulfide and carbon disulfide.

  This may be explained by the reaction of the regeneration gas with iron/iron carbide/carbon in the sorbent. Towards the end of the regeneration, sulfur in the regeneration exit gas is mainly in the form of sulfur dioxide with no hydrogen and carbon monoxide and little carbon dioxide present.

  ADACS plots of CO_2, H_2S, O_2, CO, and H_2 in the regeneration gas from the reactor exit condenser are shown in Figures 57 to 61 (Test No. 014) and Figures 62 to 66 (Test No. 018).

- <u>Sorbent Characterization</u>

  The sulfidized sorbent after Test No. 019 was sampled, on removal, at points along the reactor length and characterized by various techniques. The results are shown in Tables 17 and 18. As found previously (2), at similar gas conditions, the sorbent is seen to contain large amounts of cementite, particularly towards the reactor outlet. Pyrrhotite is only found near the reactor inlet where there is also some unsulfidized magnetite. The bulk of the zinc oxide at the reactor inlet has been converted to sulfide (see Table 17).

  Table 18 shows that the sorbent contains up to 25 wt. percent sulfur at the reactor inlet. Carbon content, lower at the inlet, exceeds 7 wt. percent towards the outlet. Values of surface area and pore volume at the reactor inlet are comparable to those of fresh sorbent. Values towards the reactor outlet significantly exceed those for fresh sorbent which may be accounted for by the reduction of the sorbent to cementite.

Similarly, sorbent crush strength is comparable to that for fresh sorbent at the reactor inlet, but is significantly lower at the outlet (see Table 18). About a third of the sorbent passed through a No. 12 screen as fines. This disintegration is mostly ascribed to the formation of cementite and its weakening effect.

- Particulate Analysis

During Test Nos. 013, 015, and 017, samples of inlet and outlet gas were taken to characterize the particulate content (7). In this series, both inlet and outlet samples were obtained for each test (though not at the same time) to get some idea of the change in particulate loading and size distribution for the gas in passing through the reactor. The results are summarized in Table 19. Details of particulate size distribution are given in Tables 20 to 25.

The particulate loading of the inlet gas was similar for the three tests, approximately 1 to 2 g/Nm^3. The median particle size was highest for Arkwright coal gas, ~ 26 μm (Test No. 015), somewhat less for Blacksville coal gas, ~ 21 μm (Test No. 013), and least for Kittanning coal gas, ~ 13 μm (Test No. 017). For each test, a significant proportion of the particulate appeared to be removed by the sorbent bed, reaching about 80 wt. percent in Test No. 017.

Larger particles appear to be removed preferentially, so that the median particle size at the outlet is significantly less than at the inlet, ~ 22 μm for Arkwright coal gas, ~ 14 μm for Blacksville coal gas, and ~ 5 μm for Kittanning coal gas.

There appears to be a positive correlation between the median particulate size in the inlet gas and the rate of increase of reactor pressure drop in a given sulfidation test, which was highest for Arkwright coal gas, somewhat less for Blacksville coal gas, and much less for Kittanning coal gas. Smoothed out pressure drop plots for the sulfidation tests are shown in Figure 67. The chemical analysis of the particulate samples, see Table 19, indicates that there is little difference between inlet and outlet particles, leading to the conclusion that little attrition was being experienced.

- Condensate Analysis

Reactor exit gas condensates were collected from all the tests and composite samples analyzed for minor and trace constituents (6). Values for the sulfidation tests are shown in Table 26 and for the regeneration tests in Table 27. A check analysis taken several months later for certain constituents is also included.

The analyses are similar to those obtained in previous test series. The regeneration condensate is enriched in most of the constituents but particularly in halides, ammonia, and alkali metals. Assuming that the constituents in the regeneration condensate derive form the gas flowing through the reactor in the preceding sulfidation test, it is possible to estimate the proportion removed by the sorbent based on the quantities of condensate collected (see Figures 35 to 41) and the condensate analysis.

Thus, 7 to 21 percent of NH_4, 27 to 59 percent of Cl, and 40 to 74 percent of Na are calculated to be removed from the gas by the sorbent and released into the regeneration gas.

Table 28 shows the analyses of the tar condensate collected from the sulfidation tests at the unit exit. These tars are seen to constitute up to 0.8 wt. percent of the exit gas and contain up to 3.8 wt. percent of sulfur. For comparison, analyses of tar condensates collected at the unit inlet (S4 sample point at gasifier primary cyclone exit) are shown in Table 29. The sulfur levels in these are significantly less than in the exit tars suggesting that the sulfur compounds in them are notably refractory to cracking and hydrodesulfurization. Ultimate analyses of the coals used in the test series are shown in Table 30.

Condensate was also collected in the course of taking particulate samples (7). The analyses of the aqueous parts are shown in Table 31. These have been translated into calculated gas phase compositions for sulfidation Test Nos. 013, 015, and 017 in Tables 32, 33, and 34 respectively. The outlet gas phase compositions calculated from the composite aqueous condensates in Table 26 are also included. Comparison between calculated inlet and outlet compositions generally substantiates the conclusion that a number of the gas components, particularly halides, ammonia, and alkali metals are removed by the desulfurization sorbent to some extent.

Inlet and outlet tar condensates from sulfidation Test No. 013 were analyzed by gas chromatograph/mass spectrometer for certain components as shown in Table 35. The principal sulfur compound, benzothiophene appears to be virtually unaffected. An apparently surprising result is that benzene, toluene, and xylenes are removed to a large extent; whereas, the concentrations of phenol and cresols are not changed much. This contrasts with previous test experience (8).

Conclusions

A third series of tests of the hot gas desulfurization unit in a sidestream of the METC 24 t/d fixed-bed gasifier was successfully concluded. The zinc ferrite sorbent underwent three cycles of sulfidation and regeneration, gas being provided from three different bituminous coals.

The amount of sulfur absorbed at the lower space velocity of 1,000 h^{-1} was comparable to that found at 2,000 h^{-1} in previous tests. A substantial amount of the sorbent was again converted to iron carbide, which appears to resist sulfidation and also markedly reduces the crush strength and attrition resistance of the sorbent. In spite of this, low molecular weight sulfur compounds were removed to a level of less than about 10 ppm and the sorbent recovered virtually all its absorption capacity on regeneration. A greater number of cycles will of course be necessary to demonstrate the sorbent's long-term effectiveness.

Reactor pressure drop was studied more closely in this test series and it was found that there was a positive correlation between the median particulate size in gas to the unit and the rate of pressure drop increase. Very little pressure drop increase was evident during a sulfidation test for an inlet gas median particulate size of 13 μm. Particulate gas loading was of the order of 1 g/Nm^3. Carbon deposition may also affect the pressure drop, so the correlation should be regarded with caution.

Again, it was found that minor constituents of the gas, in addition to sulfur, are absorbed, particularly halides, alkali and alkaline earth metals and ammonia.

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6. Lamey, S. C., R. R. Romanosky, and J. J. Kovach, "Analytical Data -- Producer Run No. 103," IR No. 2069, November 1984, METC.

7. Bagnell, R. D., and R. J. Anderson, "Particulate Sampling and Analysis in Support of Hot Gas Desulfurization Tests and Minimally Clean, Low-Btu Gas Combustion Tests During Gasifier Run No. 103," IR No. 1865, November 1984, METC.

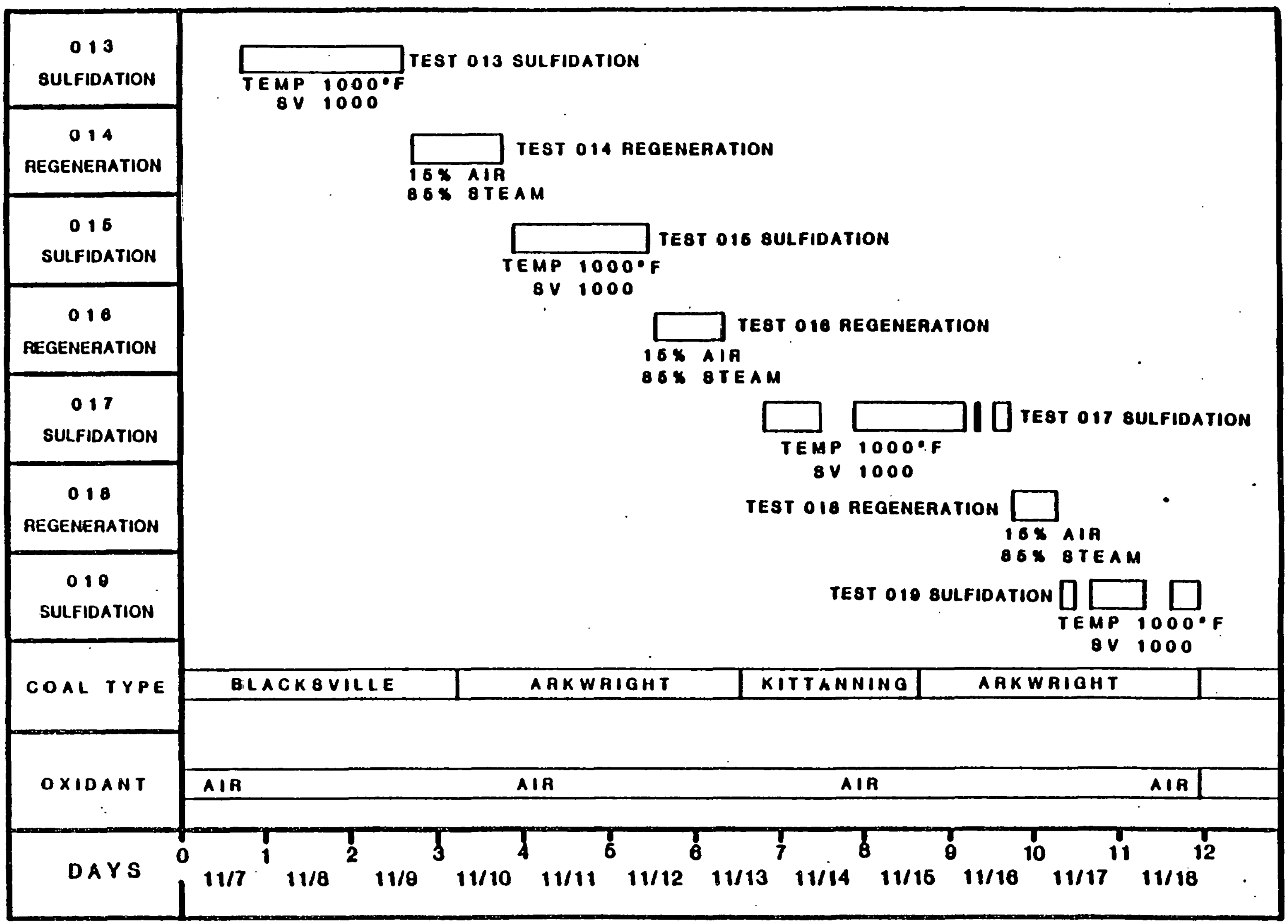
8. Green, D. A., K. E. Leese, and W. J. McMichael, "Pollution Control in Coal Gasification: Testing of Zinc Ferrite Catalyst," Research Triangle Institute for METC, Report No. DOE/MC/16441-1406 (1983).

1:910319ds.1jj

The Baseline G.C. on-line H_2S, COS, and SO_2 readings as plotted are correct only for the points: H_2S: 3 ppm; COS: 1.7 ppm; and SO_2: 5.1 ppm. Calibration curves are available for correction. Corrected plots are given in Volume 1.

Refer to letter to T. Grindley from E. E. Gorski and P. Johnson, EG&G Washington Analytical Services Center, Inc. January 30, 1986. Subject: DOE-METC/EG&G Contract No. DE-AC21-85MC21353; WBS No. 9KEX-10. HGD Project: Baseline GC/Integrator Conversion Curves.

FIGURE 1



SCHEDULE OF EVENTS

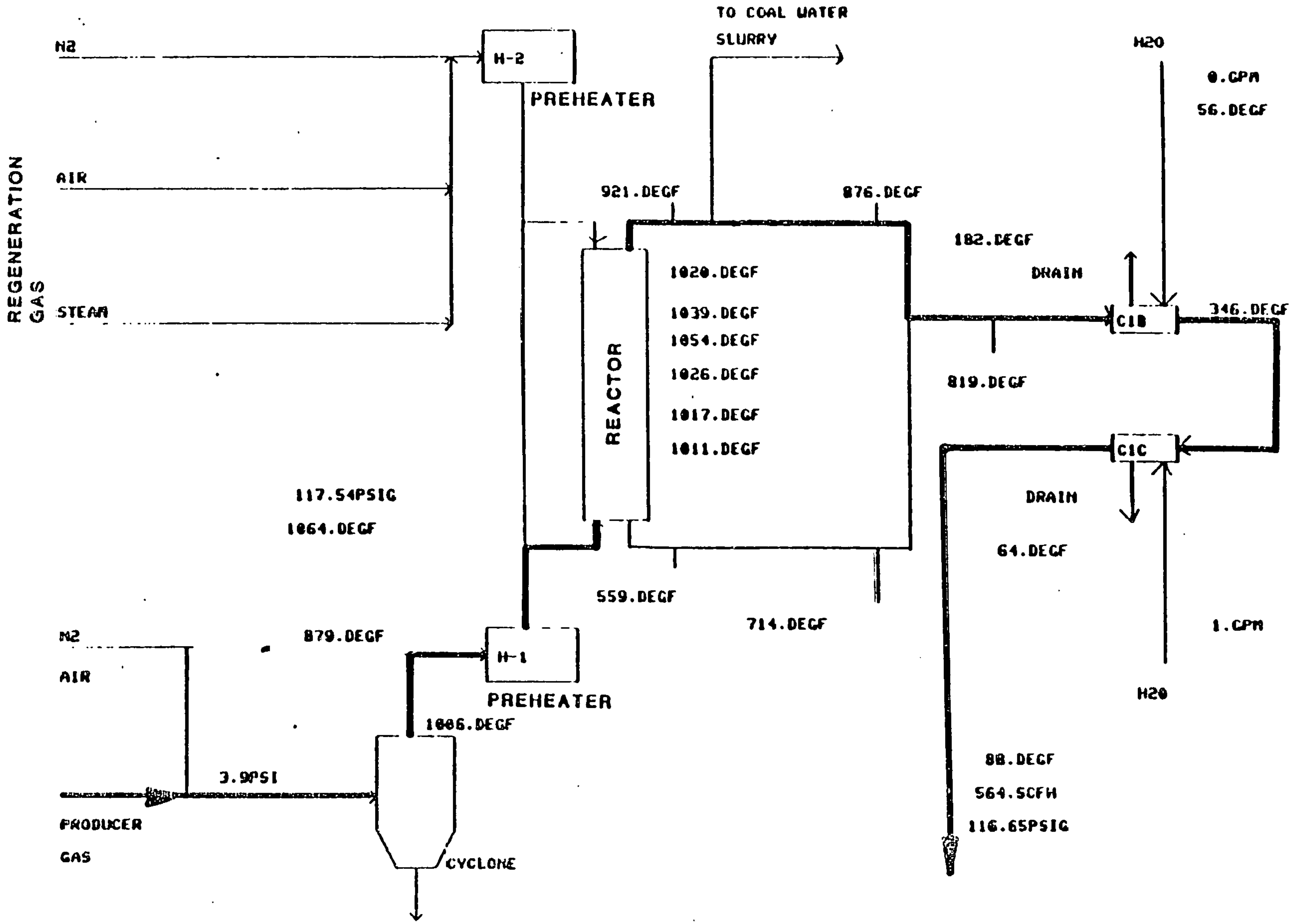
HOT GAS DESULFURIZATION SIDESTREAM TESTING 1983

GASIFIER RUN 103

FIGURE 2

TEST J

AVERAGE OPERATING PARAMETERS

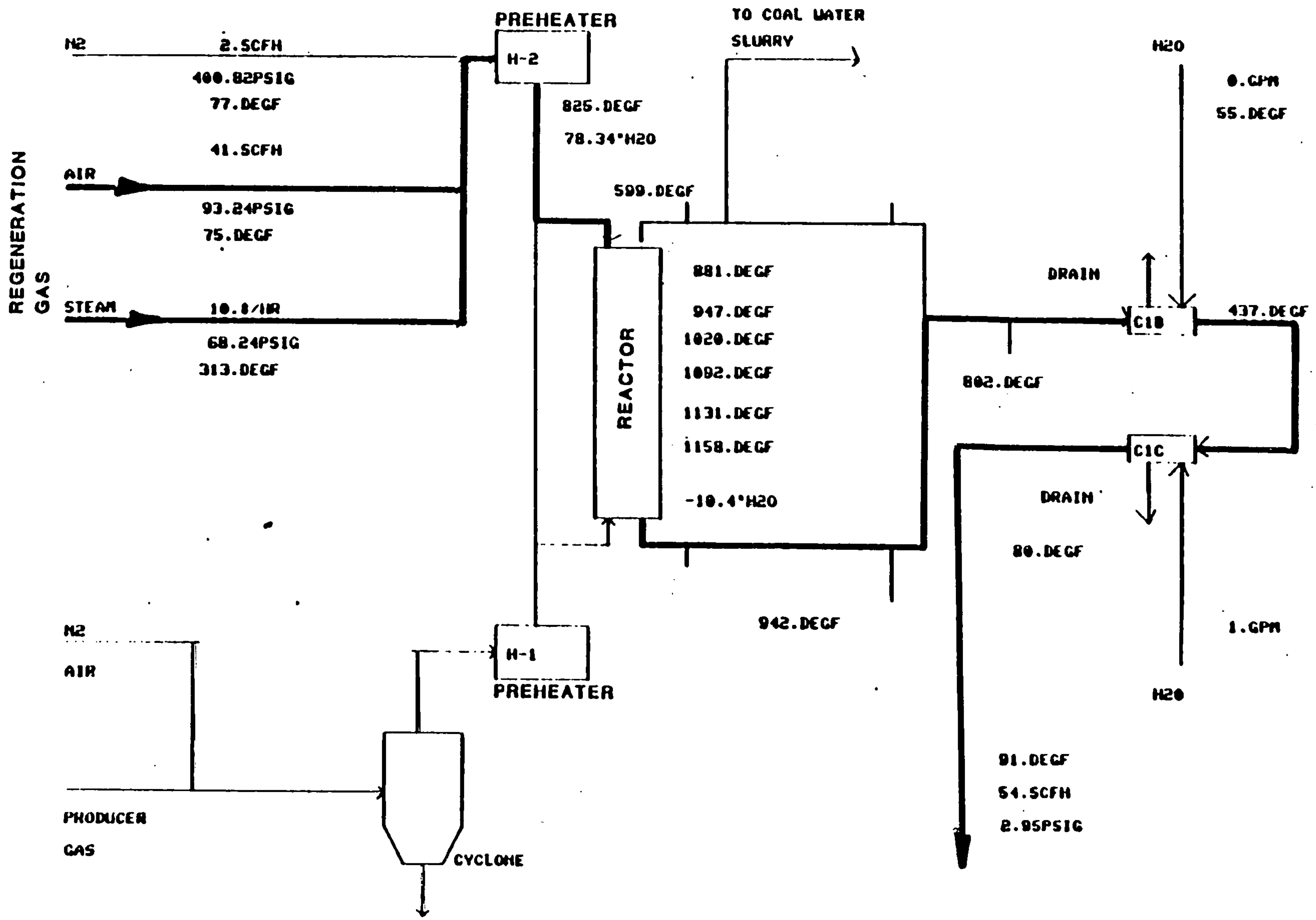


AVERAGE(S) FOR PERIOD FROM: 17:18:26 11/ 7/1983 TO 14:58:32 11/ 8/1983

FIGURE 3

TEST `14

AVERAGE OPERATING PARAMETERS

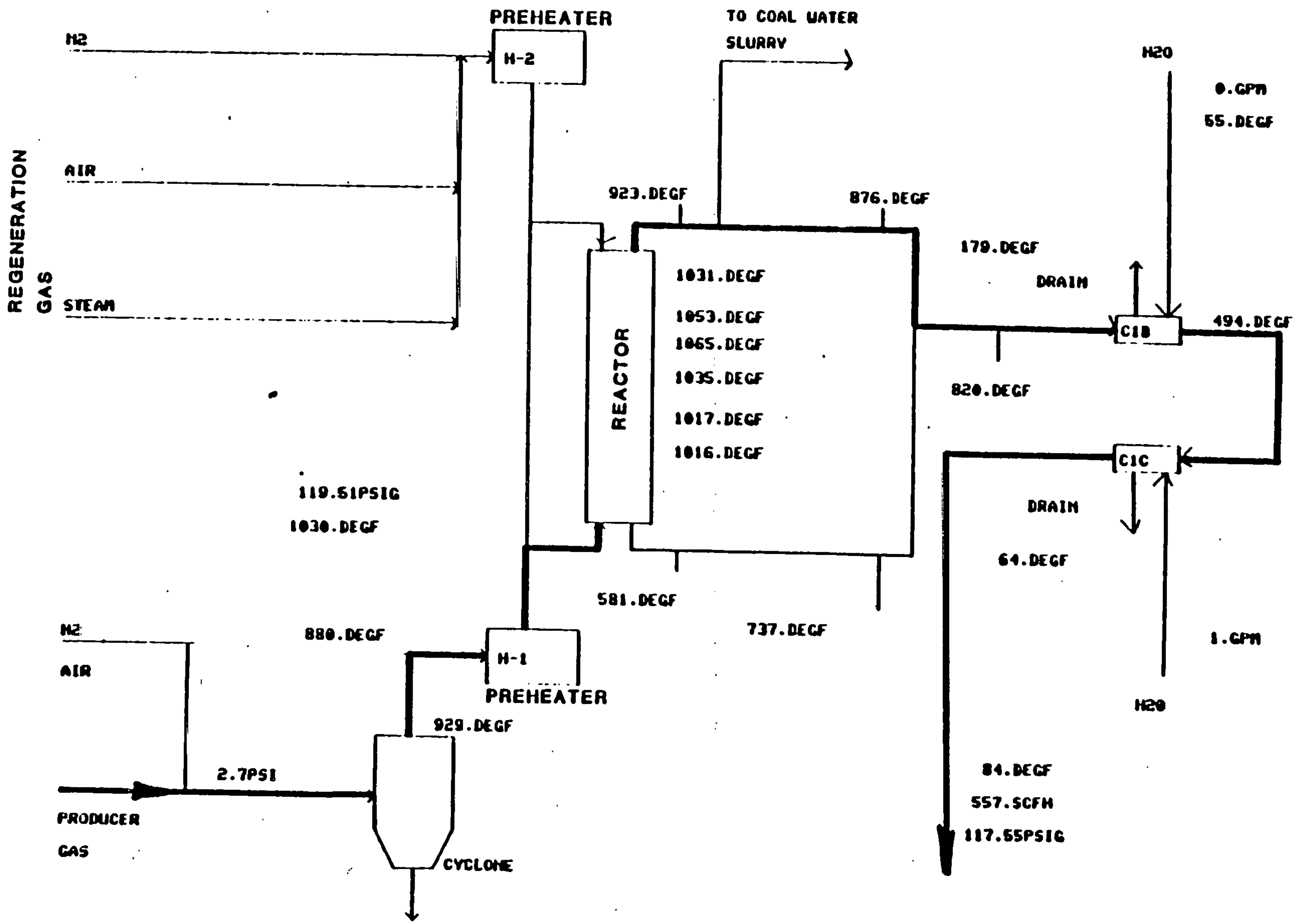


AVERAGE(9) FOR PERIOD FROM: 17:30: 2 11/ 9/1983 TO 18:39:48 11/10/1983

FIGURE 4

TEST 015

AVERAGE OPERATING PARAMETERS

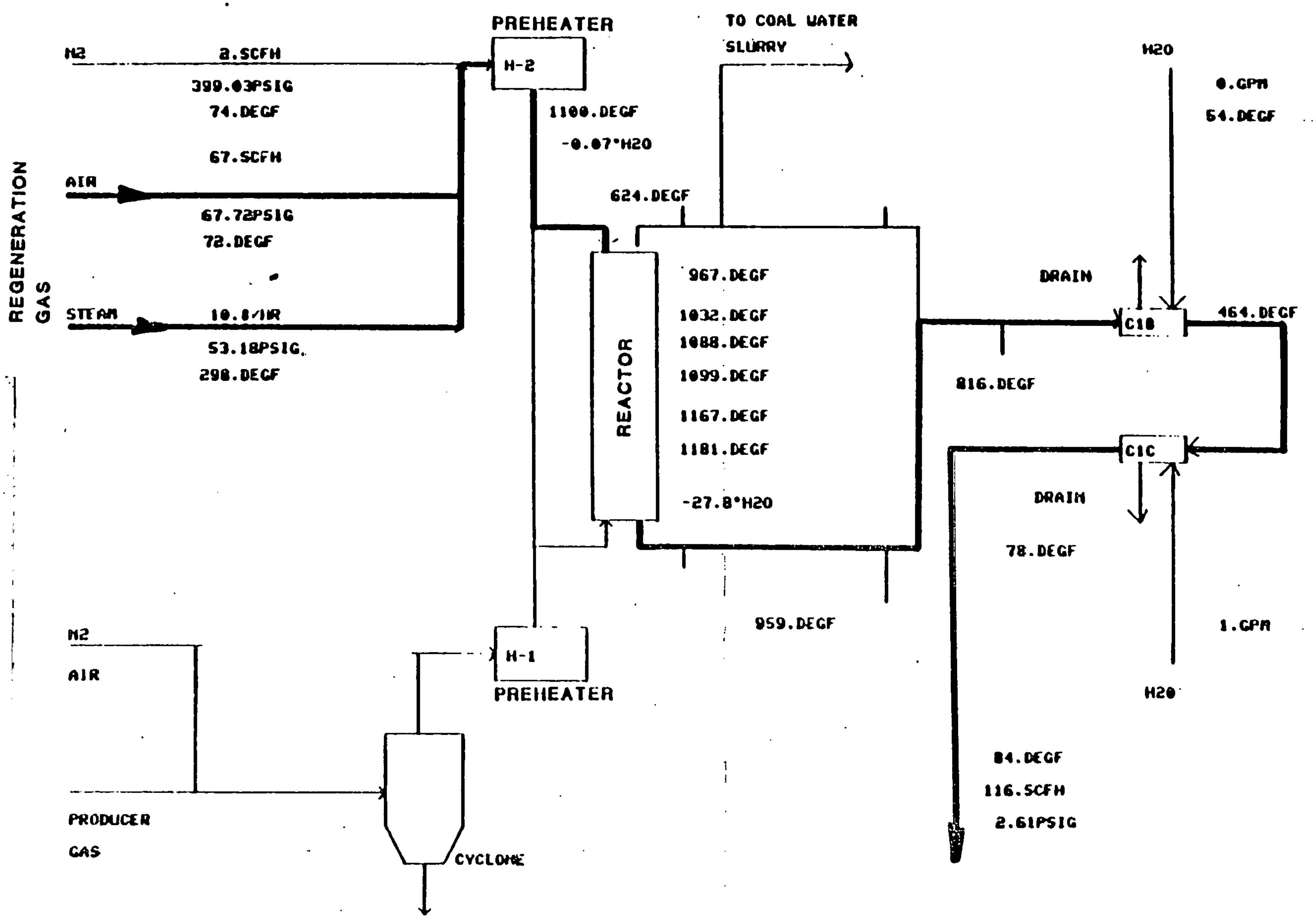


AVERAGE(6) FOR PERIOD FROM: 19: 6:17 11/10/1983 TO 11: 9:34 11/18/1983

FIGURE 5

TEST C

AVERAGE OPERATING PARAMETERS

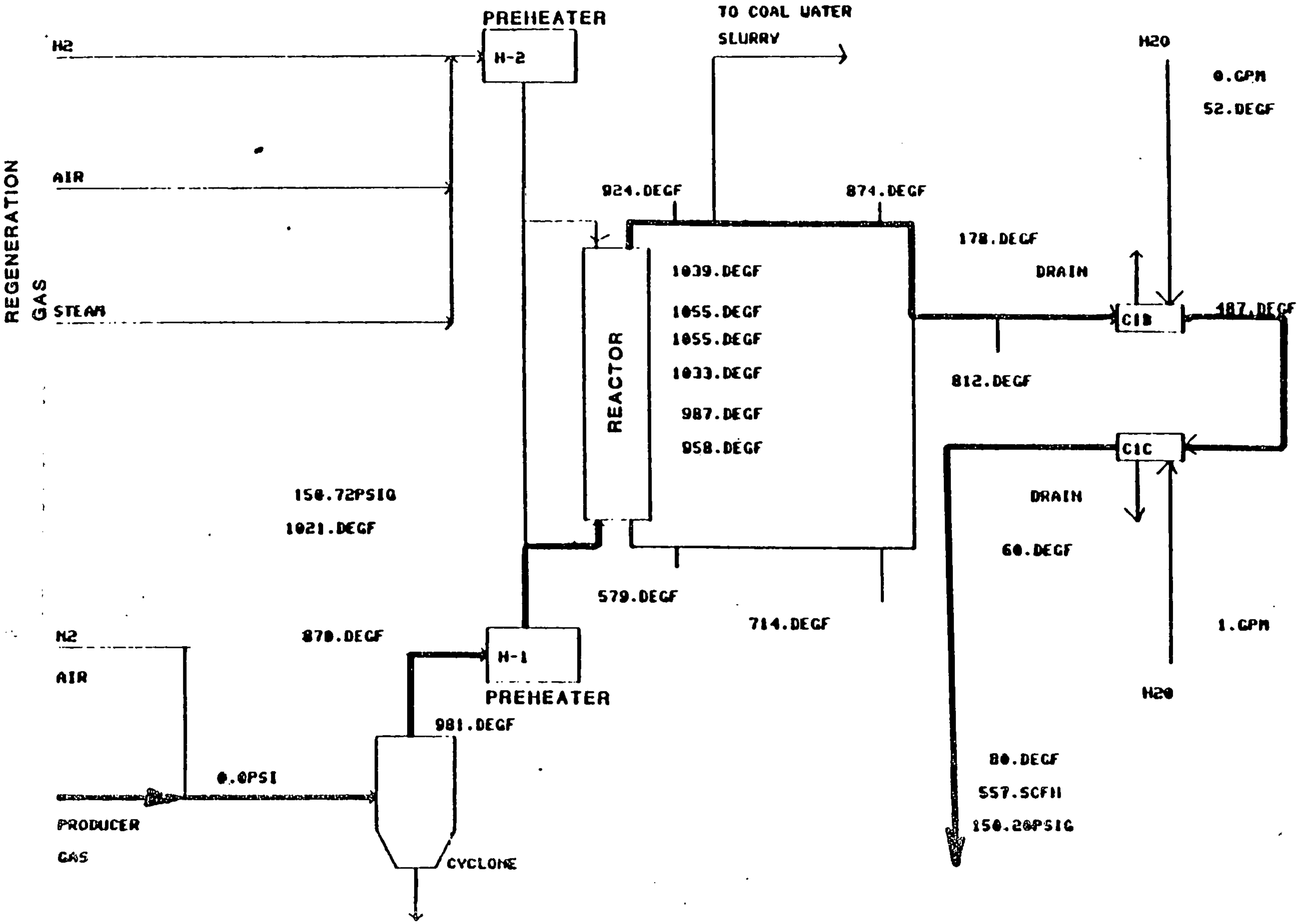


AVERAGE(9) FOR PERIOD FROM: 12:15: 0 11/18/1983 TO 8:29:38 11/13/1983

FIGURE 6

TEST 0

AVERAGE OPERATING PARAMETERS

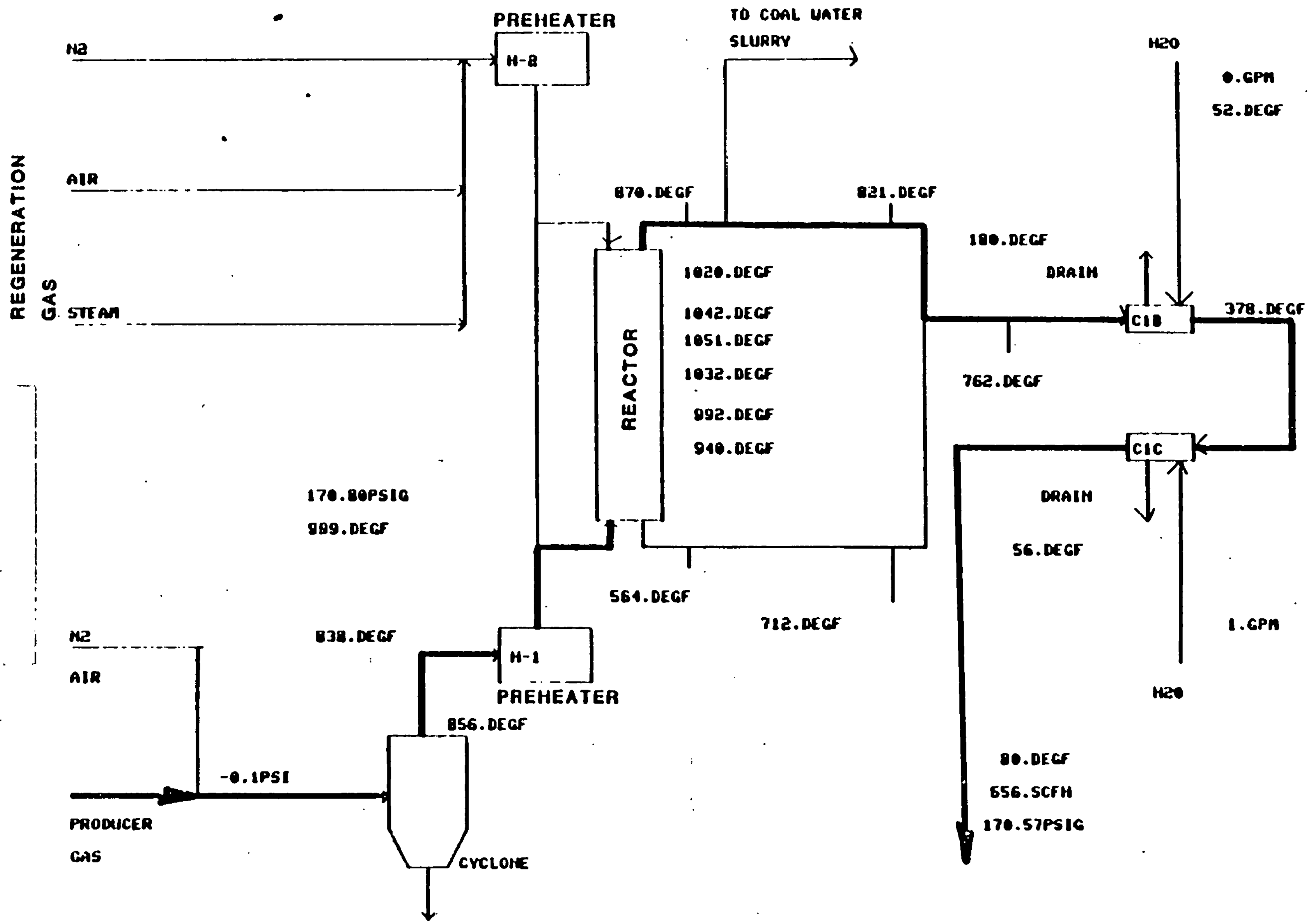


AVERAGE(S) FOR PERIOD FROM: 17:29:12 11/13/1983 TO 11:59:36 11/14/1983

FIGURE 7

TEST 0 3

AVERAGE OPERATING PARAMETERS

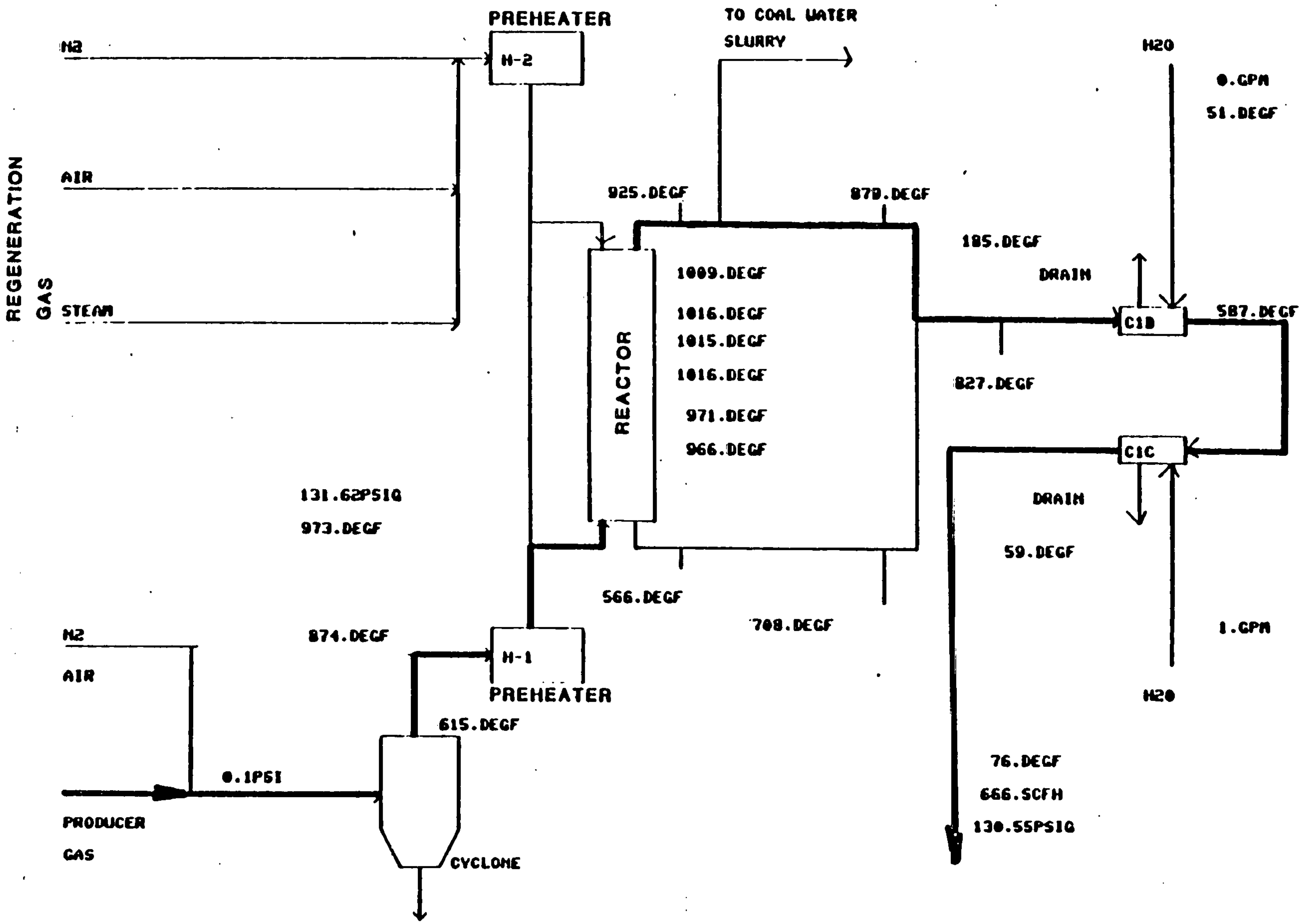


AVERAGE(S) FOR PERIOD FROM: 20: 0: 1 11/14/1983 TO 3:59:55 11/18/1983

FIGURE 8

TEST (C

AVERAGE OPERATING PARAMETERS

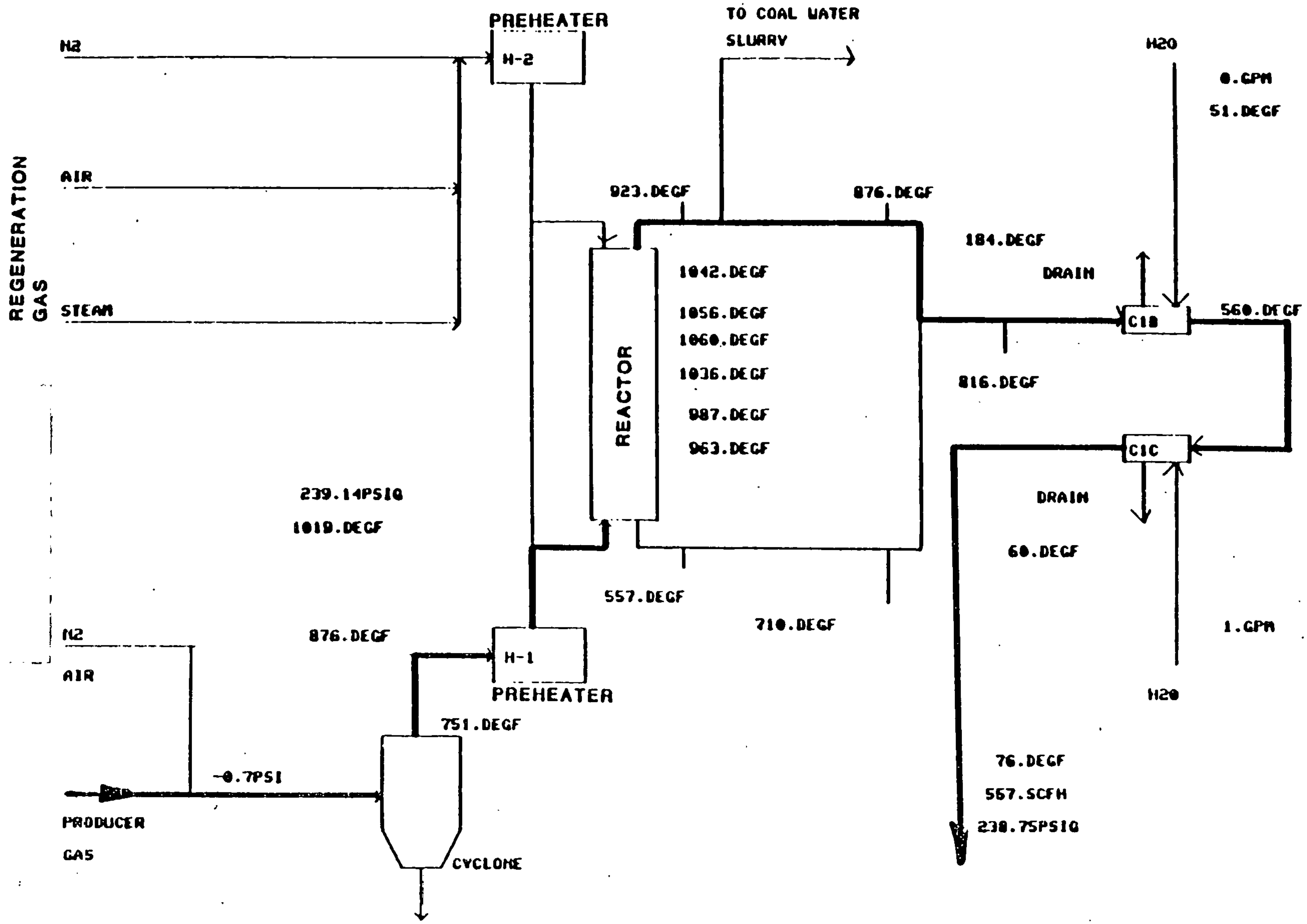


AVERAGE(8) FOR PERIOD FROM: 7: 0:18 11/10/1983 TO 8: 4:45 11/16/1983

FIGURE 9

TEST (D

AVERAGE OPERATING PARAMETERS

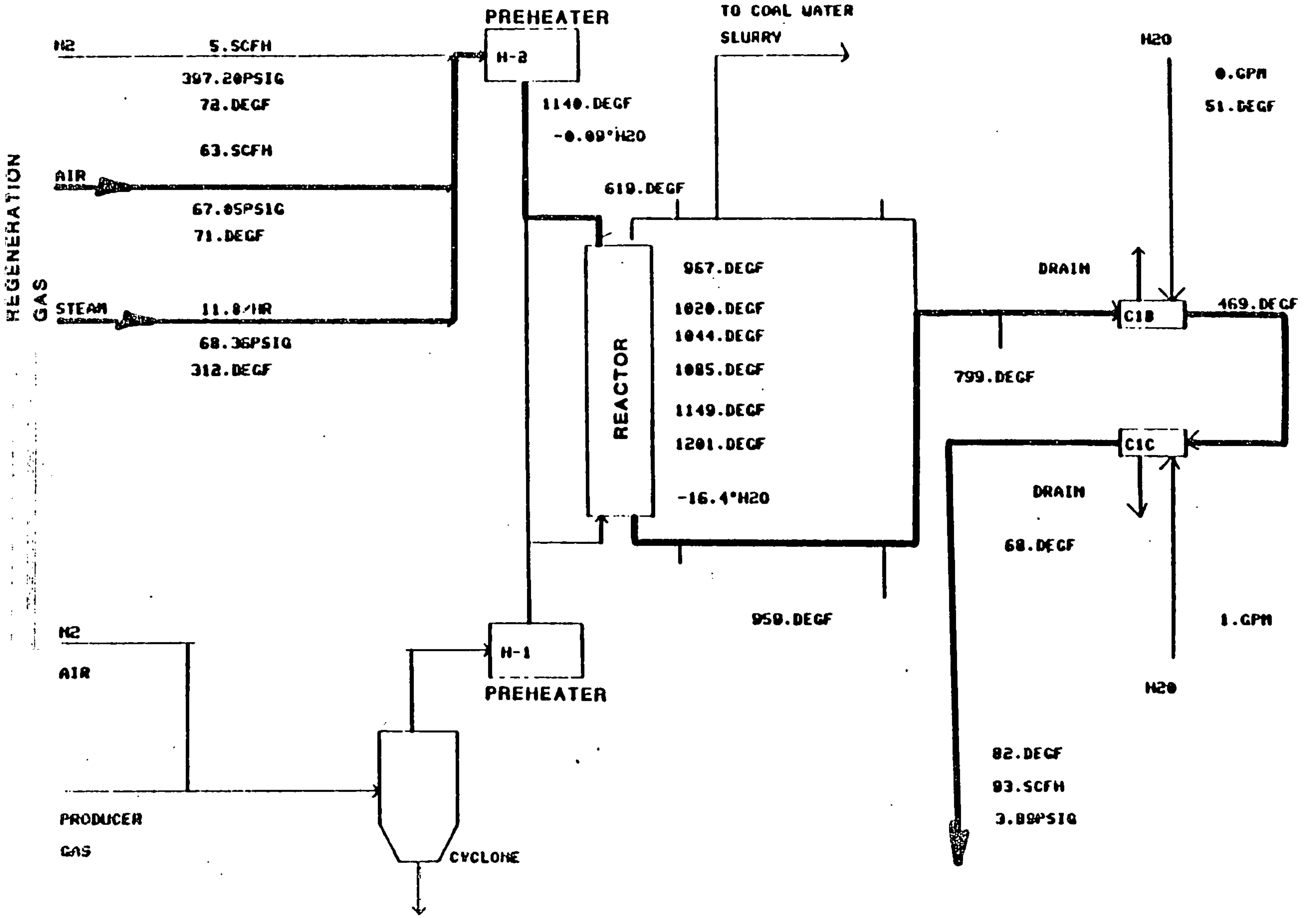


AVERAGE(6) FOR PERIOD FROM: 12:10: 4 11/16/1983 TO 18: 9:41 11/16/1983

FIGURE 10

TEST 0

AVERAGE OPERATING PARAMETERS

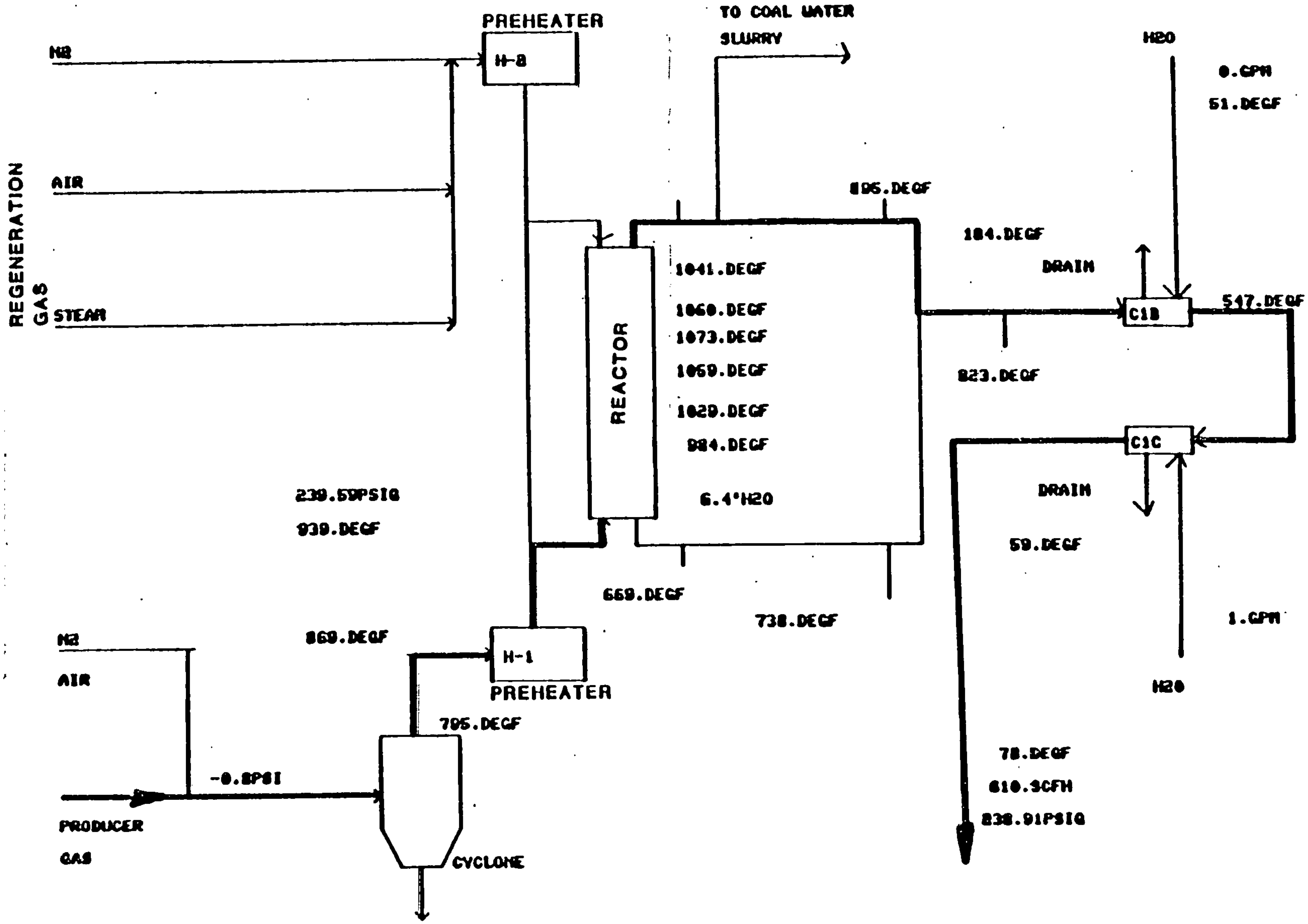


AVERAGE(S) FOR PERIOD FROM: 17:49: 7   11/10/1983 TO 5:50:36   11/17/1983

FIGURE 11

TEST 9A

AVERAGE OPERATING PARAMETERS

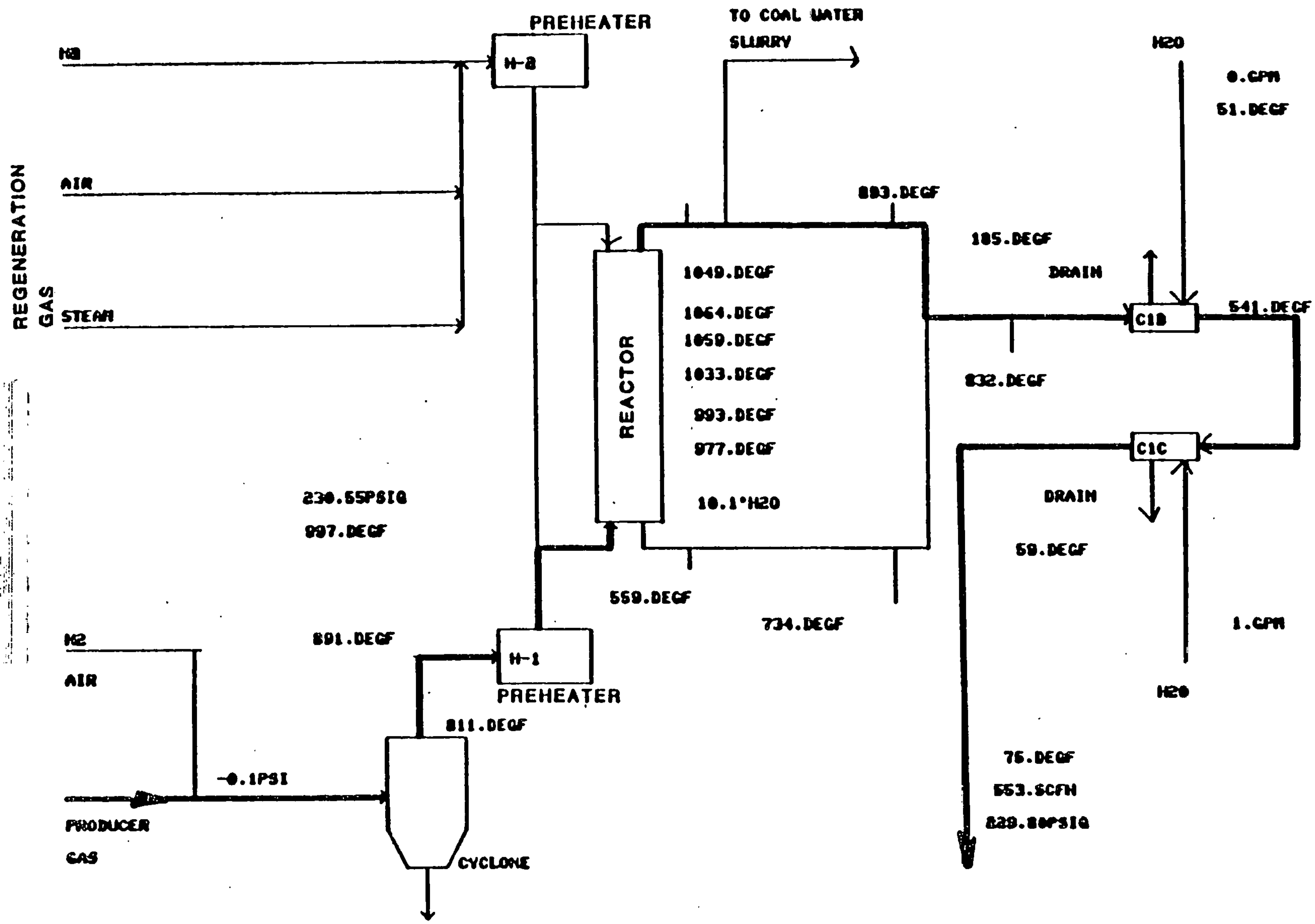


AVERAGE(S) FOR PERIOD FROM: 7:15:28 11/17/1983 TO 11:19:30 11/17/1983

FIGURE 12

TEST 0

AVERAGE OPERATING PARAMETERS

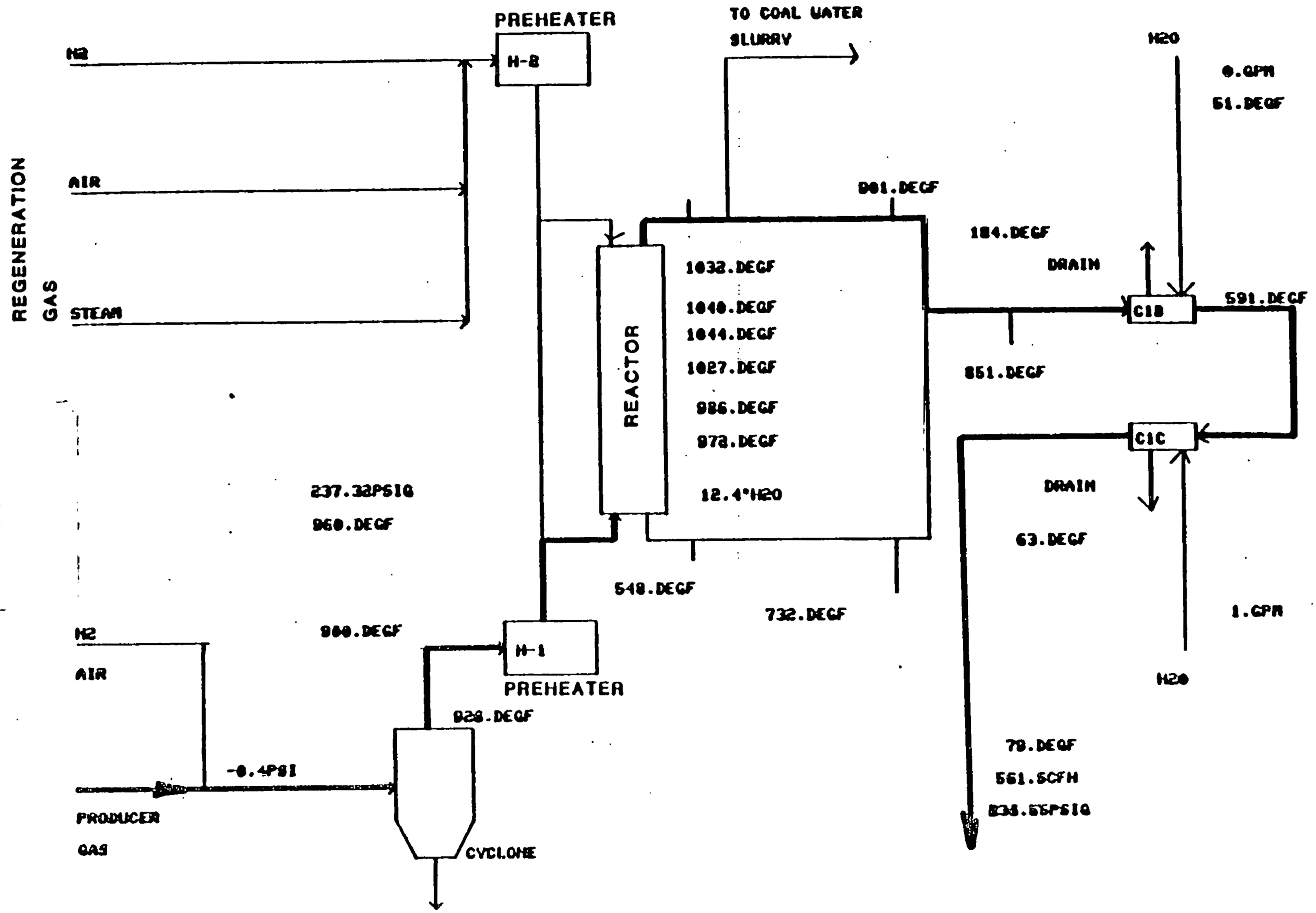


AVERAGE(S) FOR PERIOD FROM: 20:10:15 11/17/1983 TO 6:14:46 11/18/1983

FIGURE 13

TEST 019C

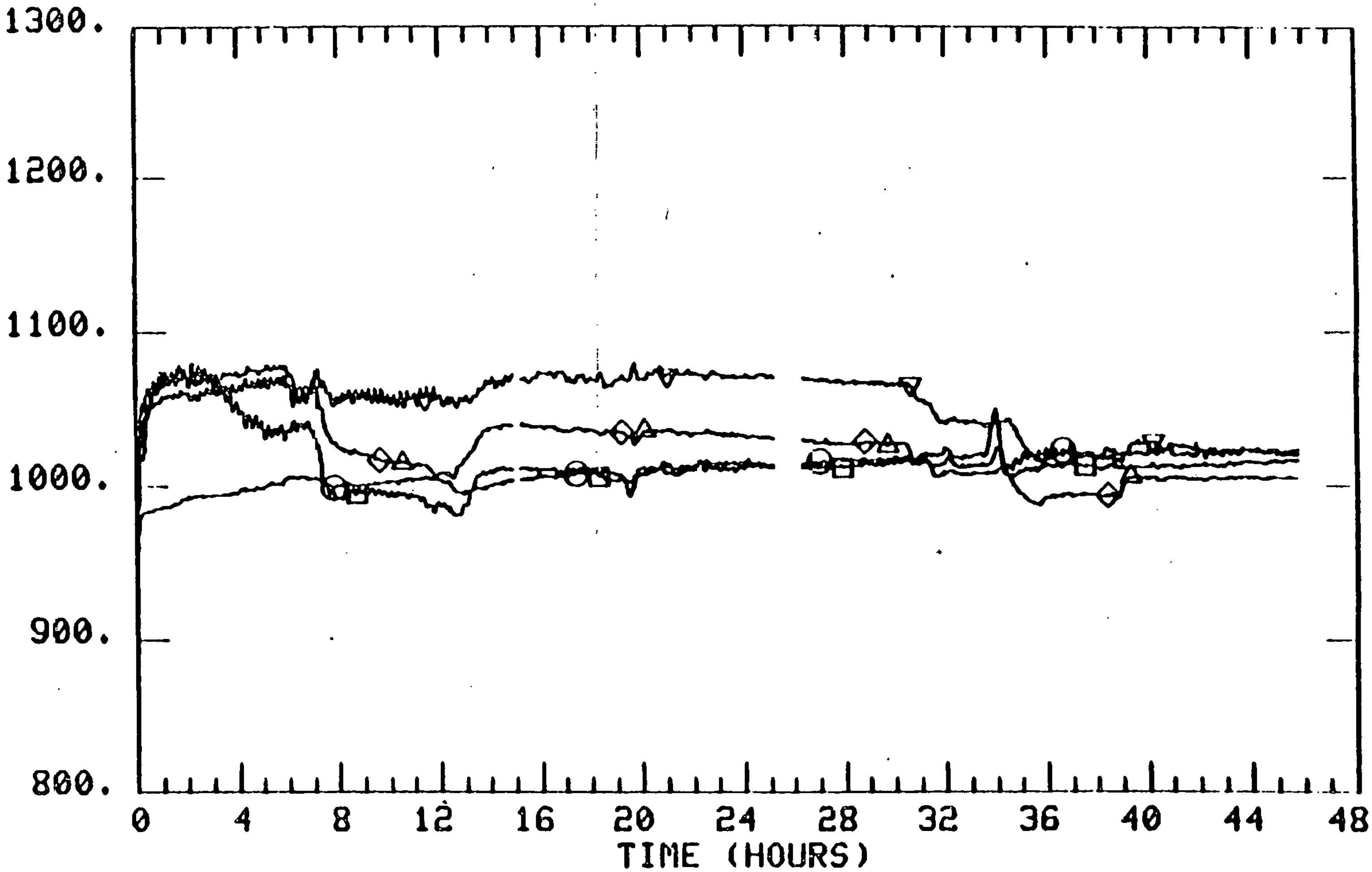
AVERAGE OPERATING PARAMETERS



AVERAGE(S) FOR PERIOD FROM: 14:10:24 11/18/1983 TO 23:20:36 11/18/1983

FIGURE 14

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	851	DEGF	ABOSABER 5° FROM BOTTOM	5322	1011.	10.90	956.8	1	1050.	3
□	852	DEGF	ABSORBER 15° FROM BOTTOM	5322	1017.	10.99	979.1	1	1081.	1
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	5322	1026.	20.91	987.8	2	1073.	5
△	853	DEGF	ABSORBER 25° FROM BOTTOM	5322	1026.	20.91	987.8	2	1073.	5
V	854	DEGF	ABSORBER 35° FROM BOTTOM	5322	1054.	20.33	1016.	8	1081.	1



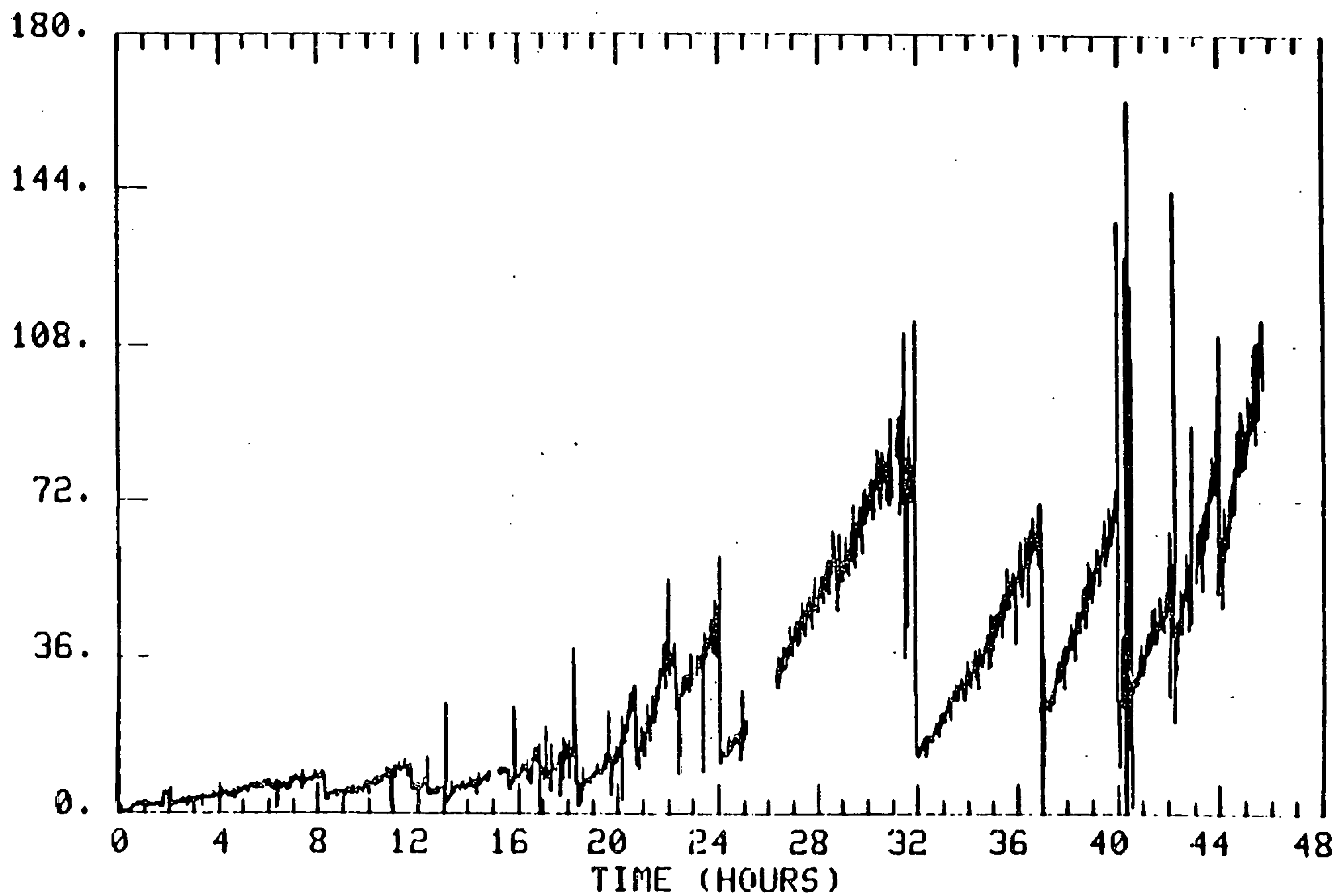
START TIME OF PLOT 17:10: 0 11/ 7/1983
STOP TIME OF PLOT 15: 0: 0 11/ 9/1983

TEST 013

REACTOR TEMPERATURES

FIGURE 15

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
294	"H2O	ABSORBER DIFFERENCE PRESSURE	5322	28.78	25.63	-8.220	1	164.9	1



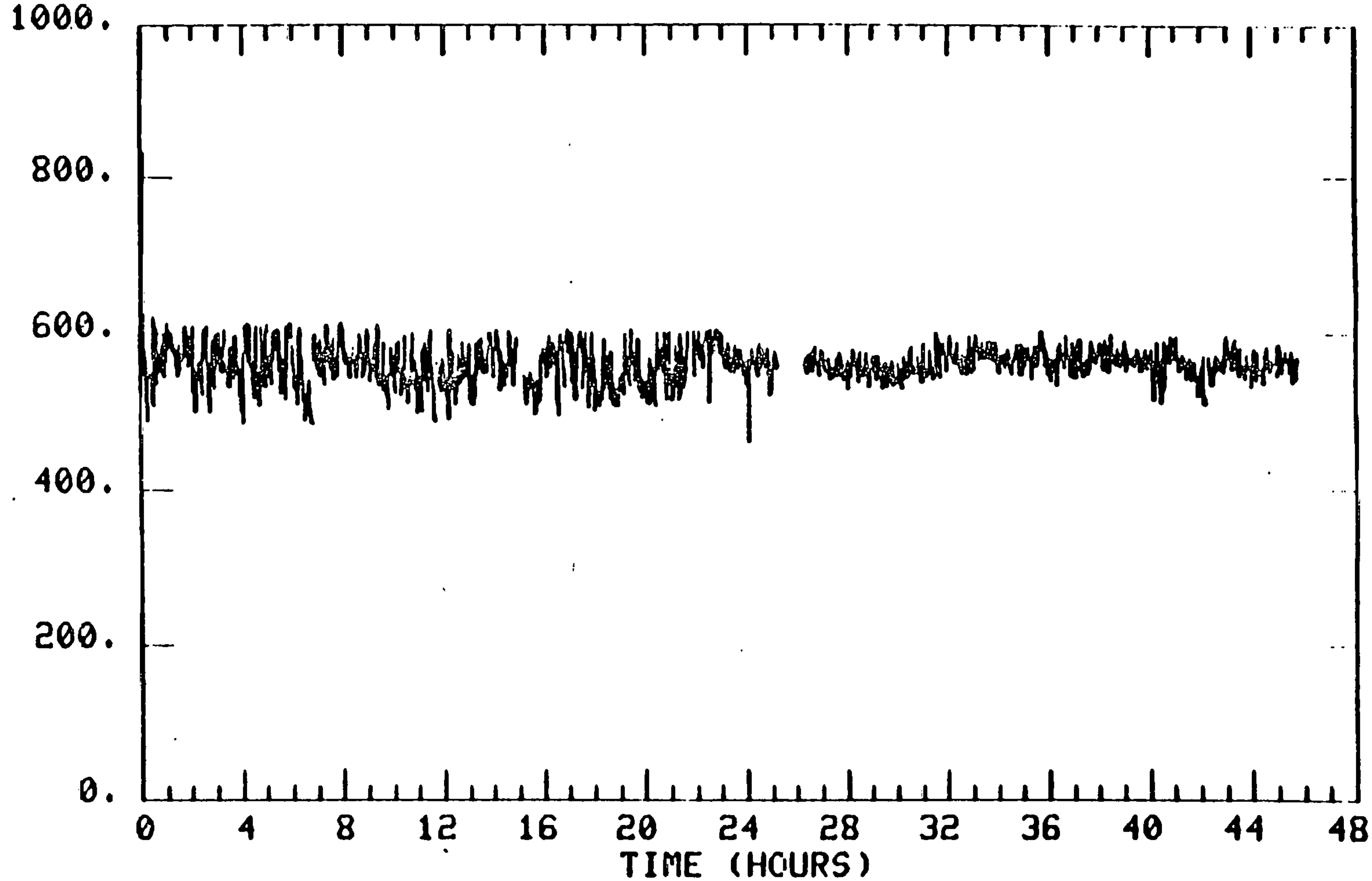
START TIME OF PLOT 17:10: 0 11/ 7/1983
STOP TIME OF PLOT 15: 0: 0 11/ 9/1983

TEST 013

REACTOR PRESSURE DROP

FIGURE 16

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
3750	SCFH	PROD. GAS OUT OF SYSTEM	5084	583.5	21.65	462.0	1	836.0	1



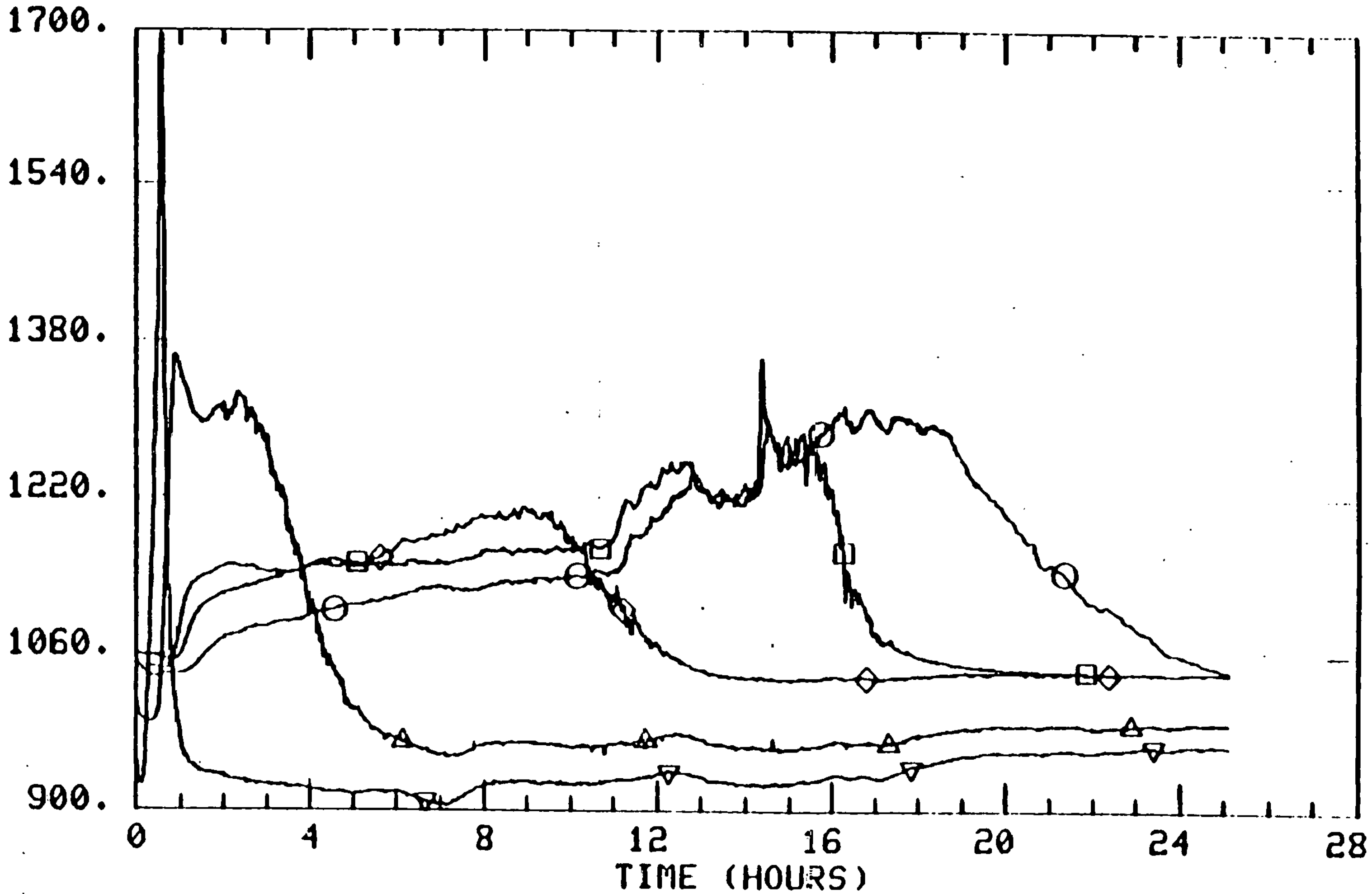
START TIME OF PLOT 17:10: 0 11/ 7/1983
STOP TIME OF PLOT 15: 0: 0 11/ 9/1983

TEST 013

EXIT FLOW

FIGURE 17

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN	FREQ	MAX	FREQ
○	851	DEGF	ABOSRBER 5° FROM BOTTOM	3022	1158.	86.25	1036.	2	1315.	1
□	852	DEGF	ABSORBER 15° FROM BOTTOM	3022	1131.	74.64	1039.	12	1363.	1
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	3022	1092.	61.96	1033.	3	1211.	4
△	854	DEGF	ABSORBER 36° FROM BOTTOM	3022	1020.	164.3	955.0	1	1367.	5
▽	856	DEGF	ABSORBER 45° FROM BOTTOM	3022	946.6	61.42	904.6	4	1699.	1



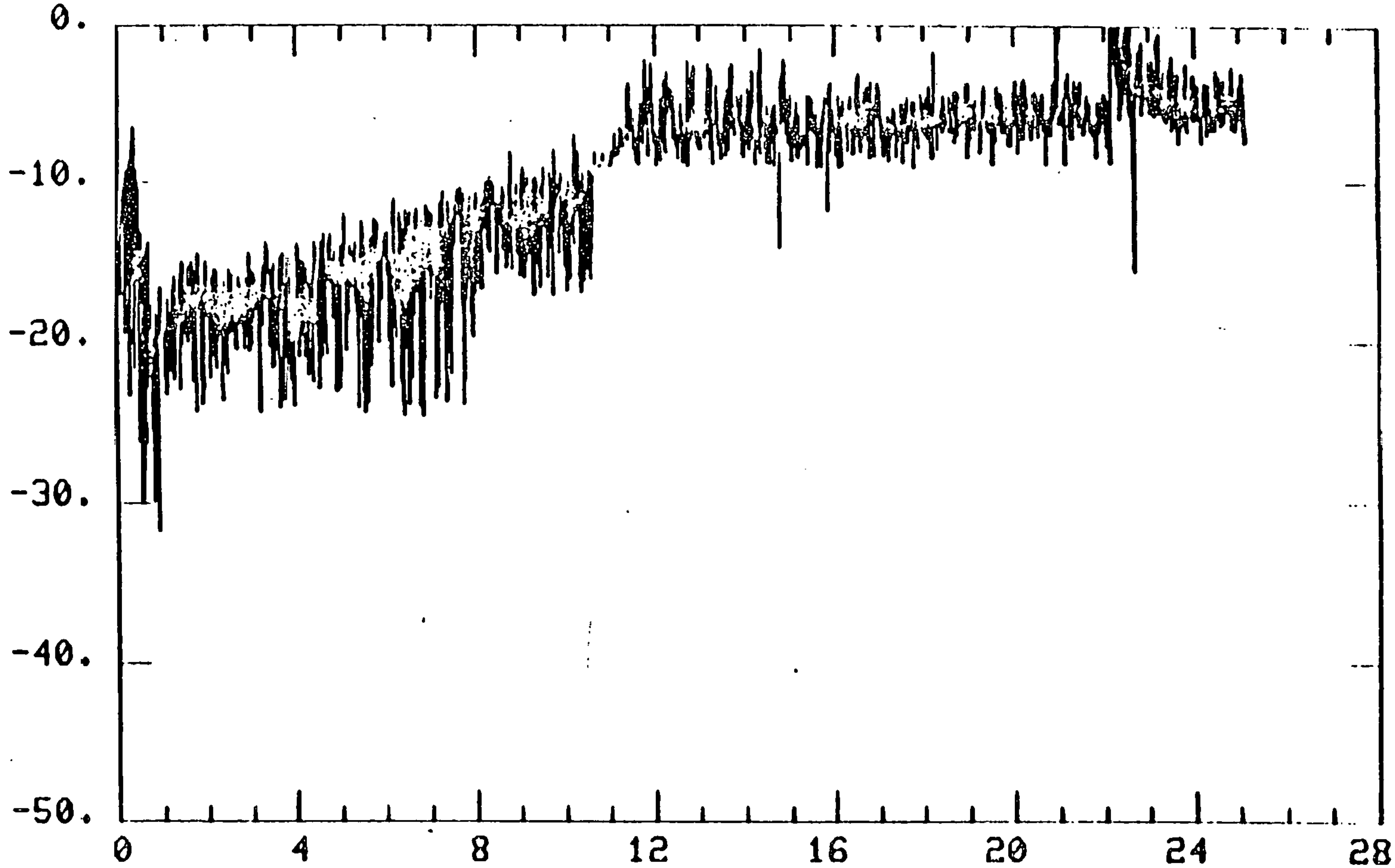
START TIME OF PLOT 17:30: 0 11/ 9/1983
STOP TIME OF PLOT 18:40: 0 11/10/1983

TEST 014

REACTOR TEMPERATURES

FIGURE 18

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
294	"H2O	ABSORBER DIFFERENCE PRESSURE	2768	-9.591	5.513	-29.86	1	4.118	1



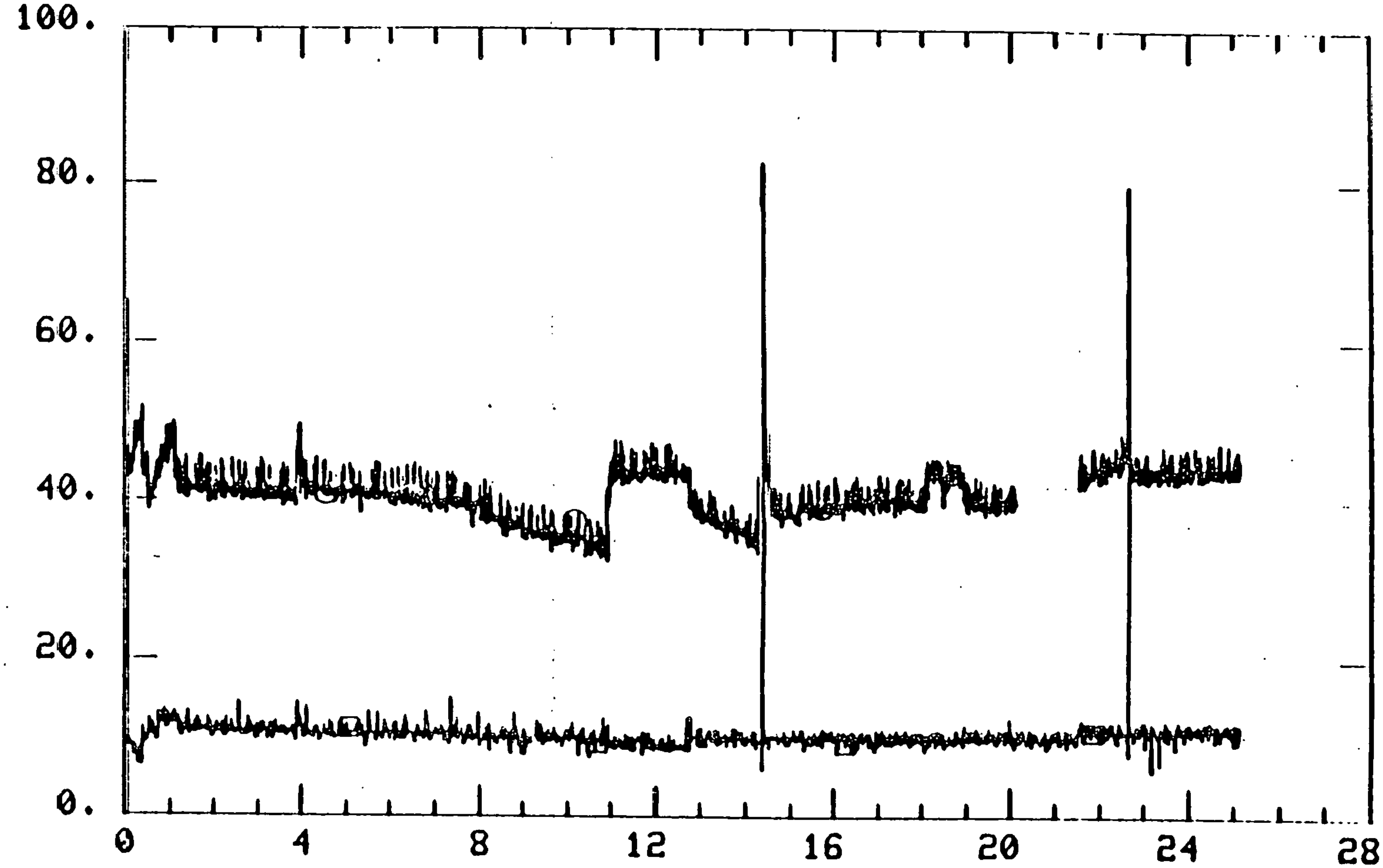
START TIME OF PLOT 17:30: 0 11/ 9/1983
STOP TIME OF PLOT 18:40: 0 11/10/1983

TEST 014

REACTOR PRESSURE DROP

FIGURE 19

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	3756	SCFH	AIR FLOW LOW RANGE	2843	41.29	4.104	0.0000E+00	3	82.82	1
□	3754	#/HR	STEAM FLOW	2948	10.56	0.8868	6.179	1	15.31	1



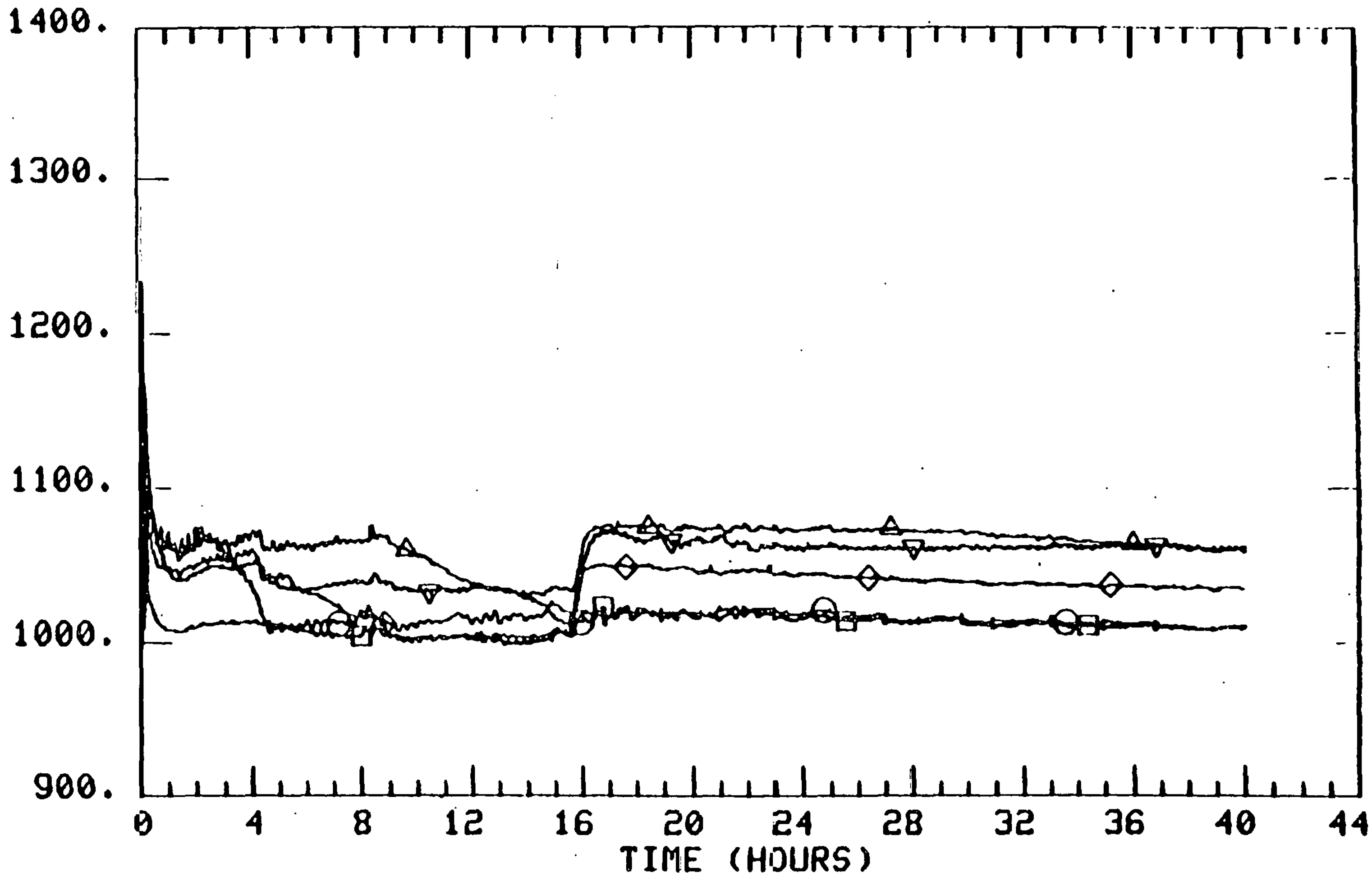
START TIME OF PLOT 17:30: 0 11/ 9/1983
STOP TIME OF PLOT 18:40: 0 11/10/1983

TEST 014

STEAM AND AIR FLOWS

FIGURE 20

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN	- FREQ	MAX	- FREQ
○	851	DEGF	ABOSRBER 5° FROM BOTTOM	4811	1015.	4.157	1007.	31	1040.	5
□	852	DEGF	ABSORBER 15° FROM BOTTOM	4811	1017.	17.59	998.8	1	1235.	1
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	4811	1035.	18.60	1003.	1	1176.	1
△	854	DEGF	ABSORBER 35° FROM BOTTOM	4811	1065.	12.95	1018.	1	1167.	1
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	4811	1053.	14.05	1001.	1	1101.	1



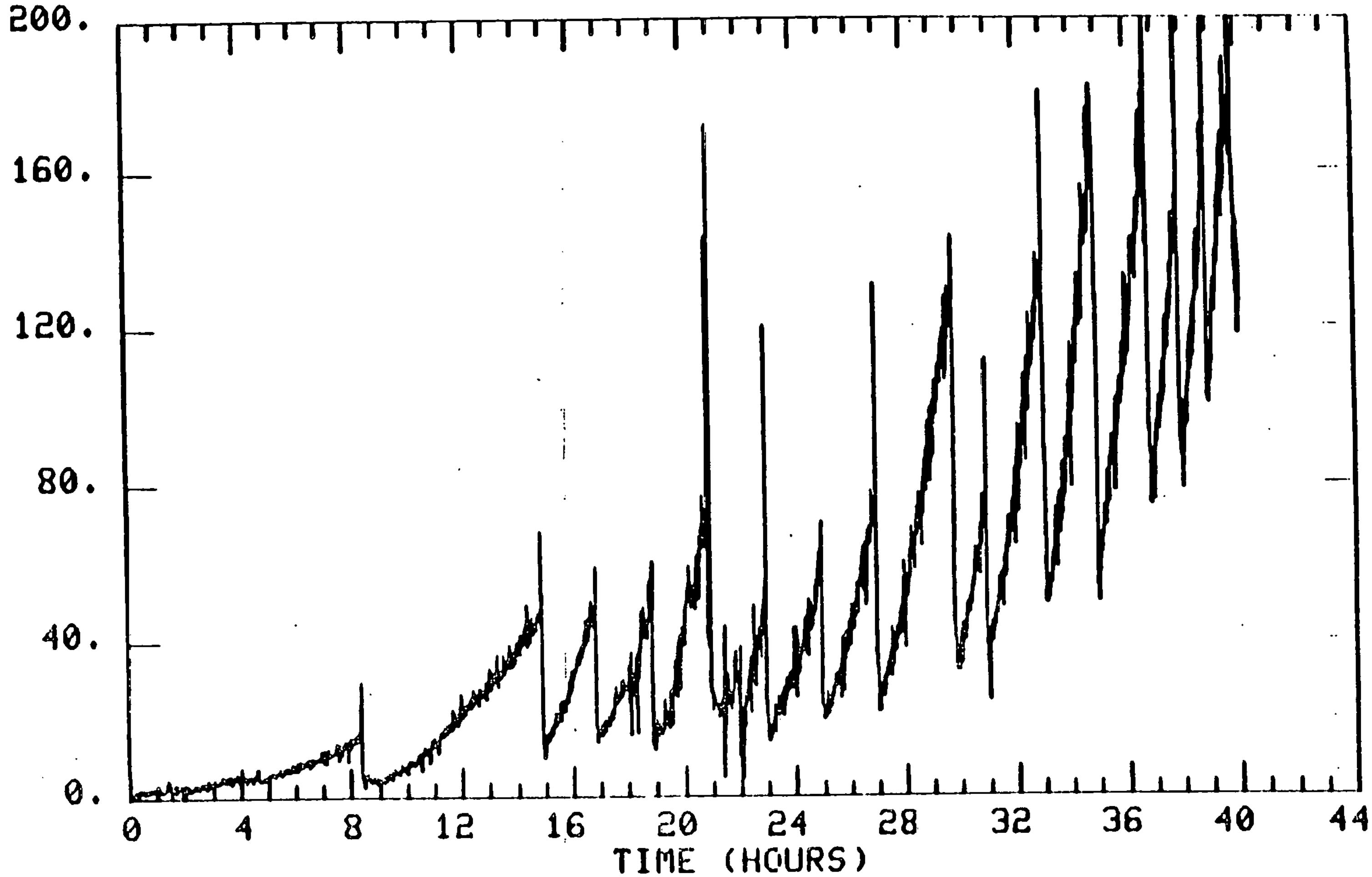
START TIME OF PLOT 19: 5: 0 11/10/1983
STOP TIME OF PLOT 11:10: 0 11/12/1983

TEST 015

REACTOR TEMPERATURES

FIGURE 21

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
B94	"H2O	ABSORBER DIFFERENCE PRESSURE	4811	46.58	44.03	-0.9899	1	226.1	1



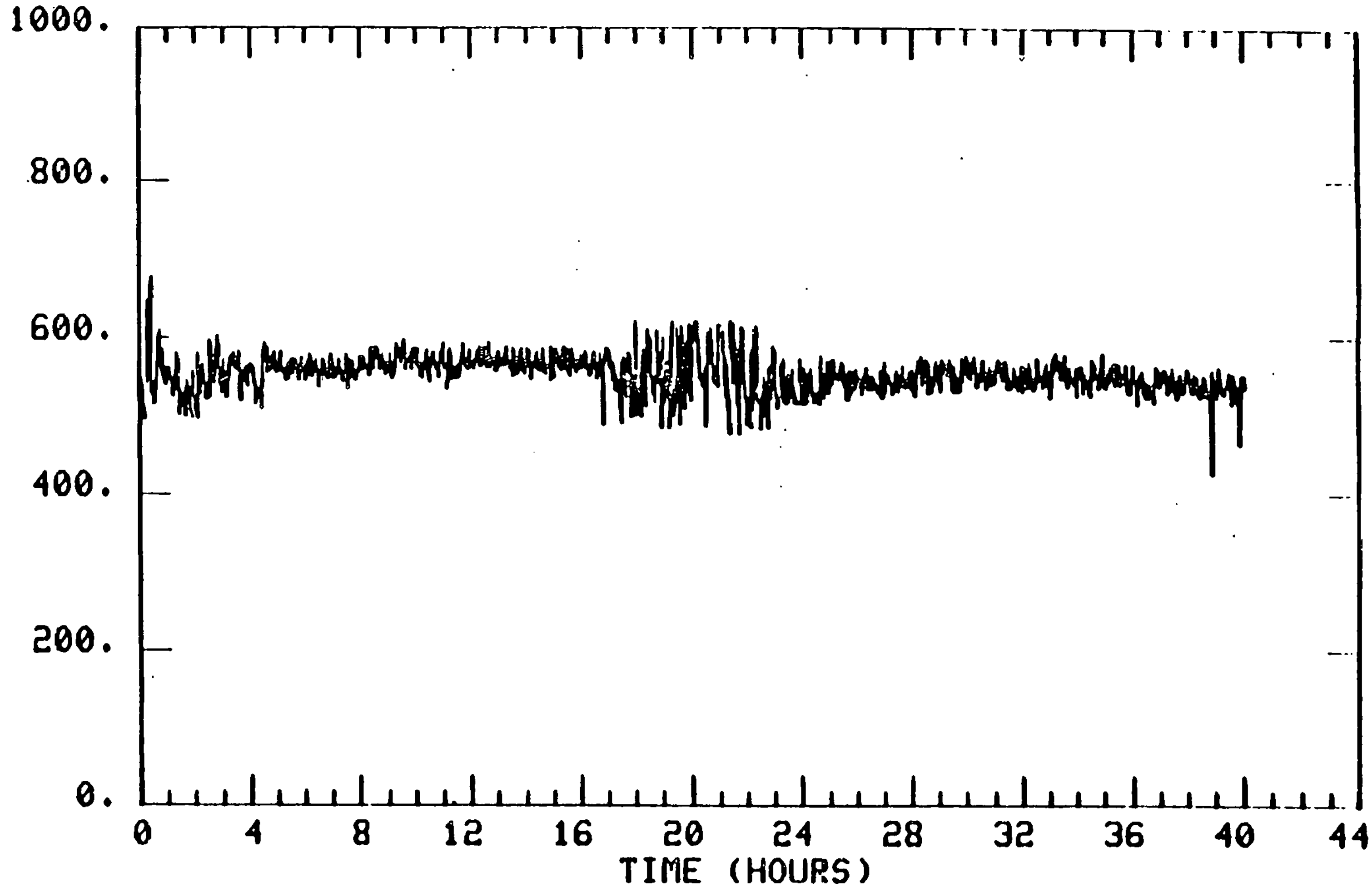
START TIME OF PLOT 19: 5: 0 11/10/1983
STOP TIME OF PLOT 11:10: 0 11/12/1983

TEST 015

REACTOR PRESSURE DROP

FIGURE 22

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
3750	SCFH	PROD. GAS OUT OF SYSTEM	4662	556.9	24.01	0.0000E+00	2	679.0	1



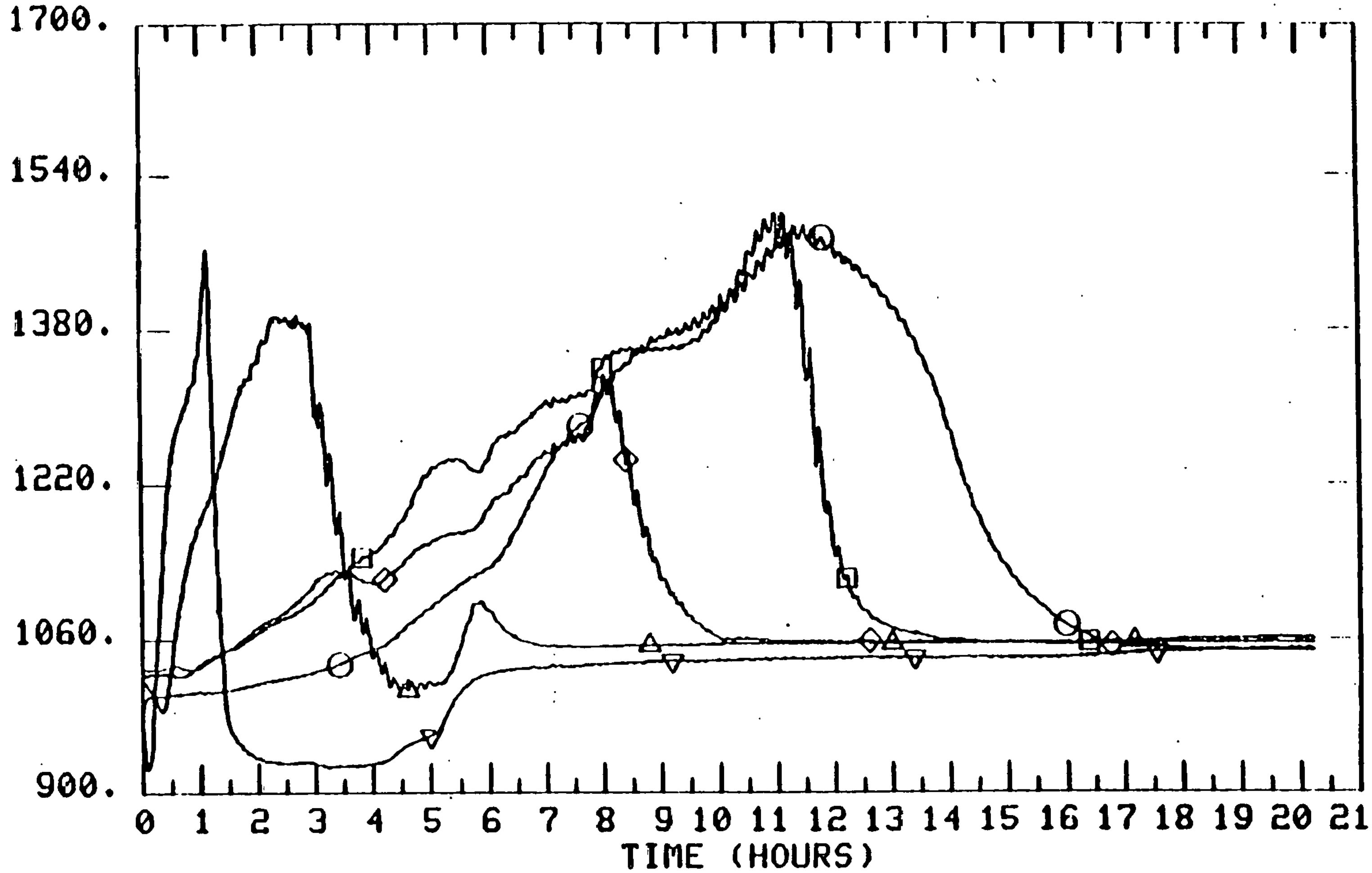
START TIME OF PLOT 19: 5: 0 11/10/1983
STOP TIME OF PLOT 11:10: 0 11/12/1983

TEST 015

EXIT FLOW

FIGURE 23

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
○	851	DEGF	ABOSRBER 5° FROM BOTTOM	2430	1181.	161.8	989.7	1	1488.	1
□	852	DEGF	ABSORBER 15° FROM BOTTOM	2430	1167.	136.5	1029.	10	1501.	3
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	2430	1099.	71.40	1022.	1	1334.	1
△	854	DEGF	ABSORBER 35° FROM BOTTOM	2430	1088.	89.32	985.6	1	1396.	1
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	2430	1032.	74.90	924.5	1	1465.	1



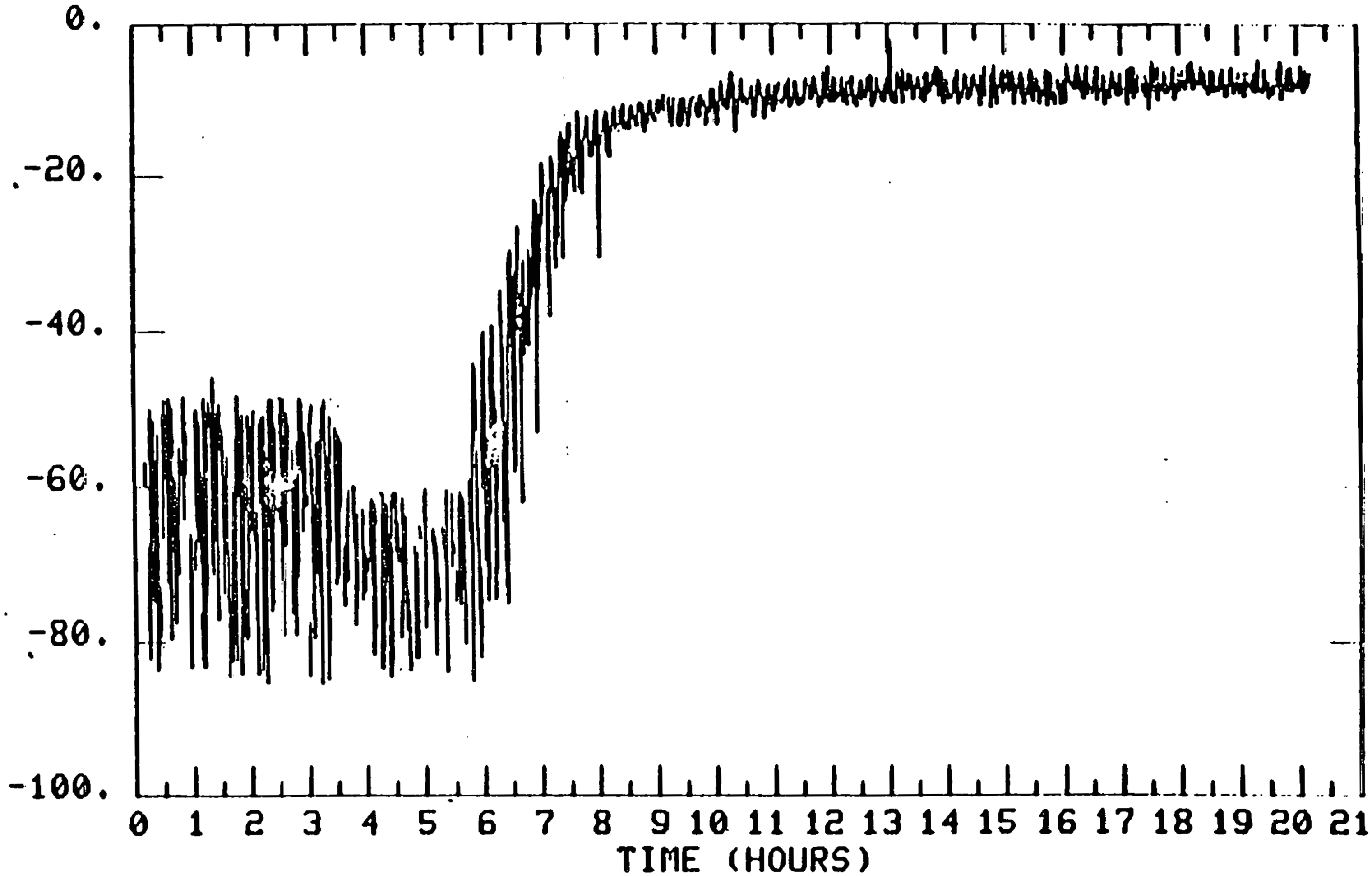
START TIME OF PLOT 12:15: 0 11/12/1983
STOP TIME OF PLOT 8:30: 0 11/13/1983

TEST 016

REACTOR TEMPERATURES

FIGURE 24

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
294	"H2O	ABSORBER DIFFERENCE PRESSURE	2098	-23.36	24.09	-85.33	1	-1.895	1



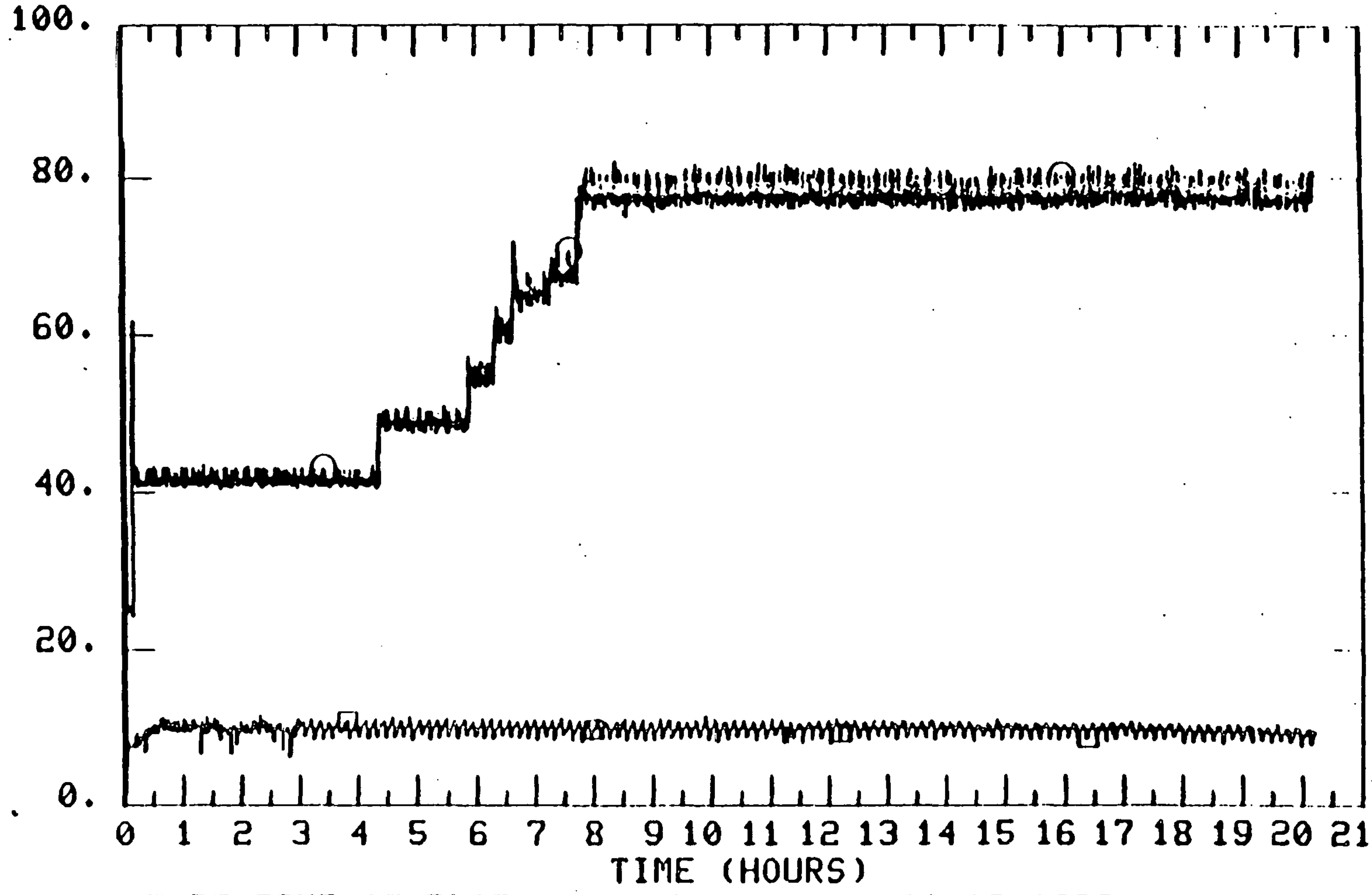
START TIME OF PLOT 12:15: 0 11/12/1983
STOP TIME OF PLOT 8:30: 0 11/13/1983

TEST 016

REACTOR PRESSURE DROP

FIGURE 25

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN	- FREQ	MAX	- FREQ
O 3756	SCFH	AIR FLOW LOW RANGE	2430	66.91	16.11	0.0000E+00	4	85.19	1
□ 3754	#/HR	STEAM FLOW	2430	9.026	0.6845	6.307	1	11.72	1



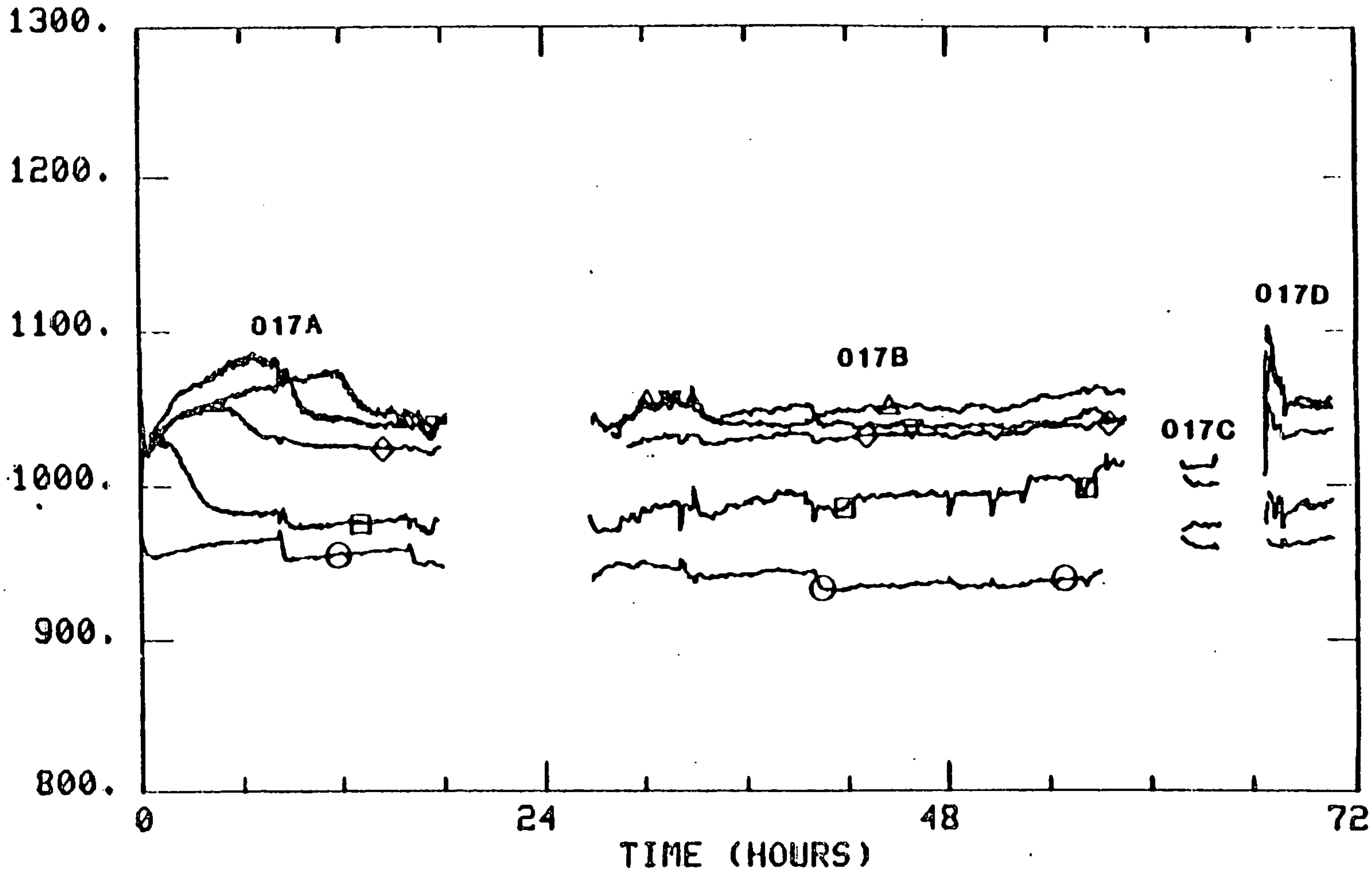
START TIME OF PLOT 12:15: 0 11/12/1983
STOP TIME OF PLOT 8:30: 0 11/13/1983

TEST 016

STEAM AND AIR FLOWS

FIGURE 26

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	851	DEGF	ABOSRBER 5° FROM BOTTOM	6761	948.6	10.80	931.2	1	971.6	1
□	858	DEGF	ABSORBER 15° FROM BOTTOM	6769	989.2	13.17	963.9	1	1037.	4
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	6524	1033.	7.510	1009.	1	1056.	1
△	854	DEGF	ABSORBER 35° FROM BOTTOM	6766	1051.	16.15	998.7	1	1105.	1
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	6758	1046.	12.53	1003.	4	1086.	3



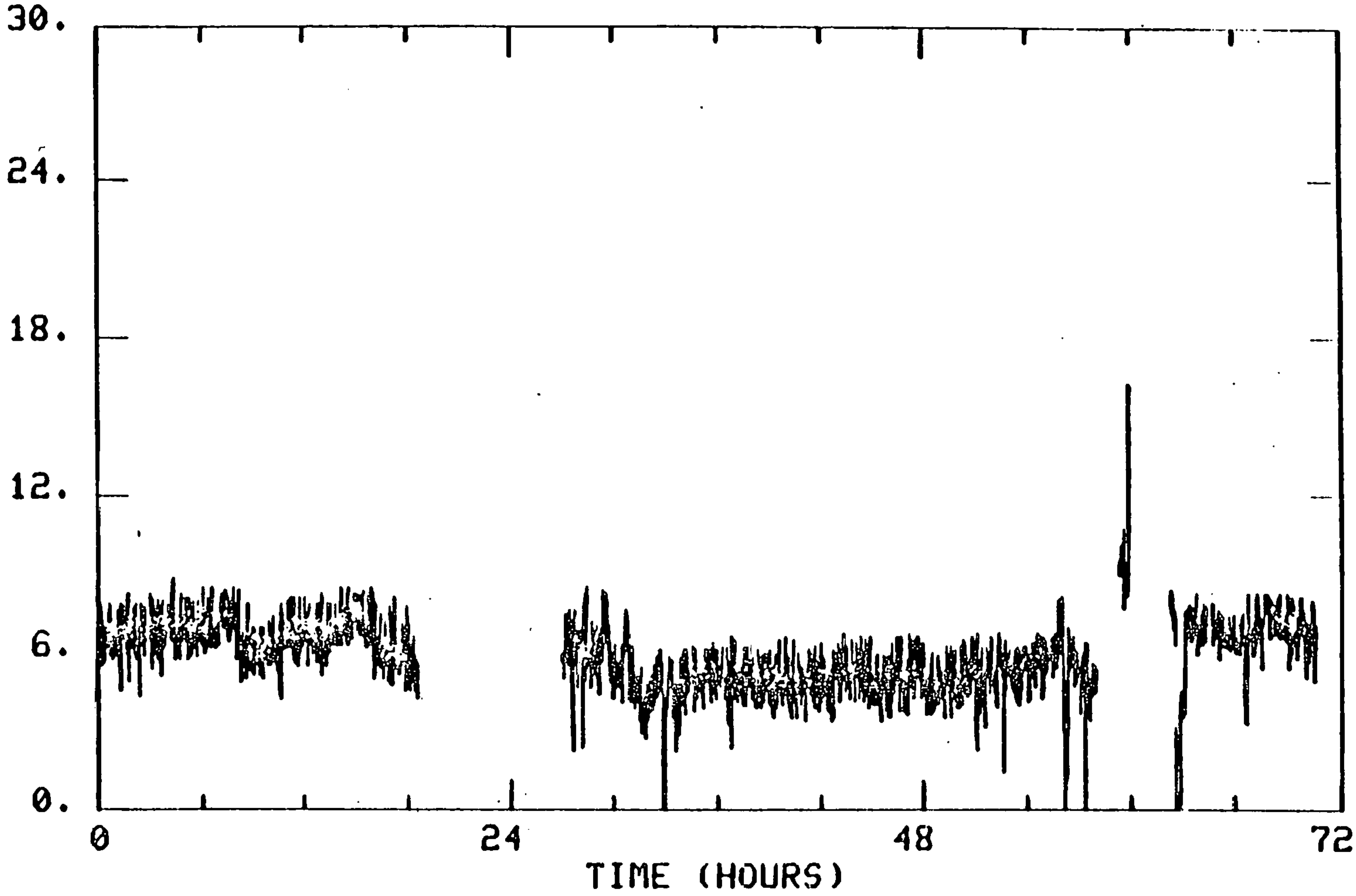
START TIME OF PLOT 17:25: 0 11/13/1983
STOP TIME OF PLOT 16:10: 0 11/16/1983

TEST 017

REACTOR TEMPERATURES

FIGURE 27

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
294	"H2O	ABSORBER DIFFERENCE PRESSURE	6940	6.004	1.298	-3.054	1	16.31	1



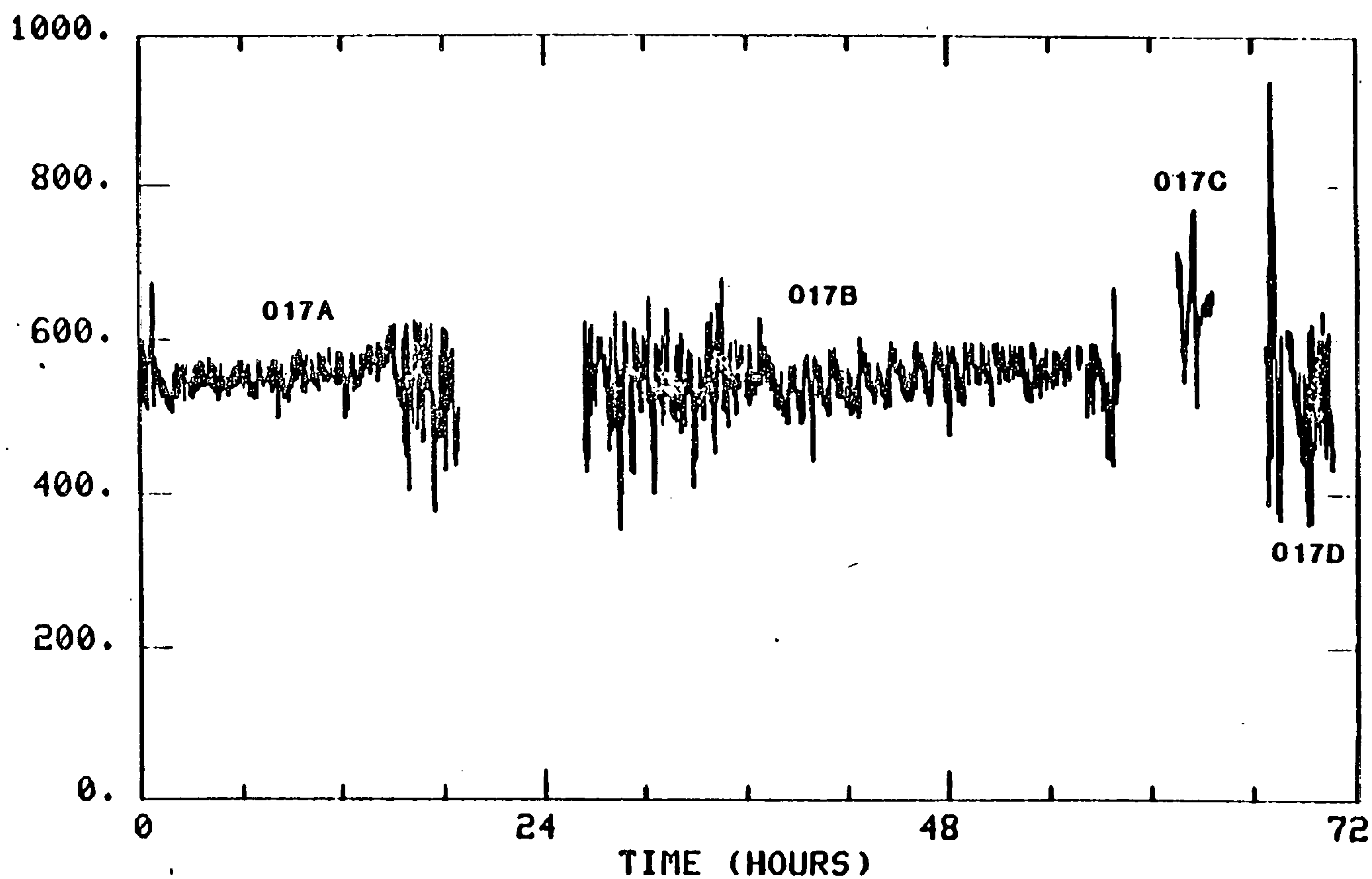
START TIME OF PLOT 17:25: 0 11/13/1983
STOP TIME OF PLOT 16:10: 0 11/16/1983

TEST 017

REACTOR PRESSURE DROP

FIGURE 28

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
3750	SCFH	PROD. GAS OUT OF SYSTEM	6418	553.9	39.23	354.3	1	943.2	1



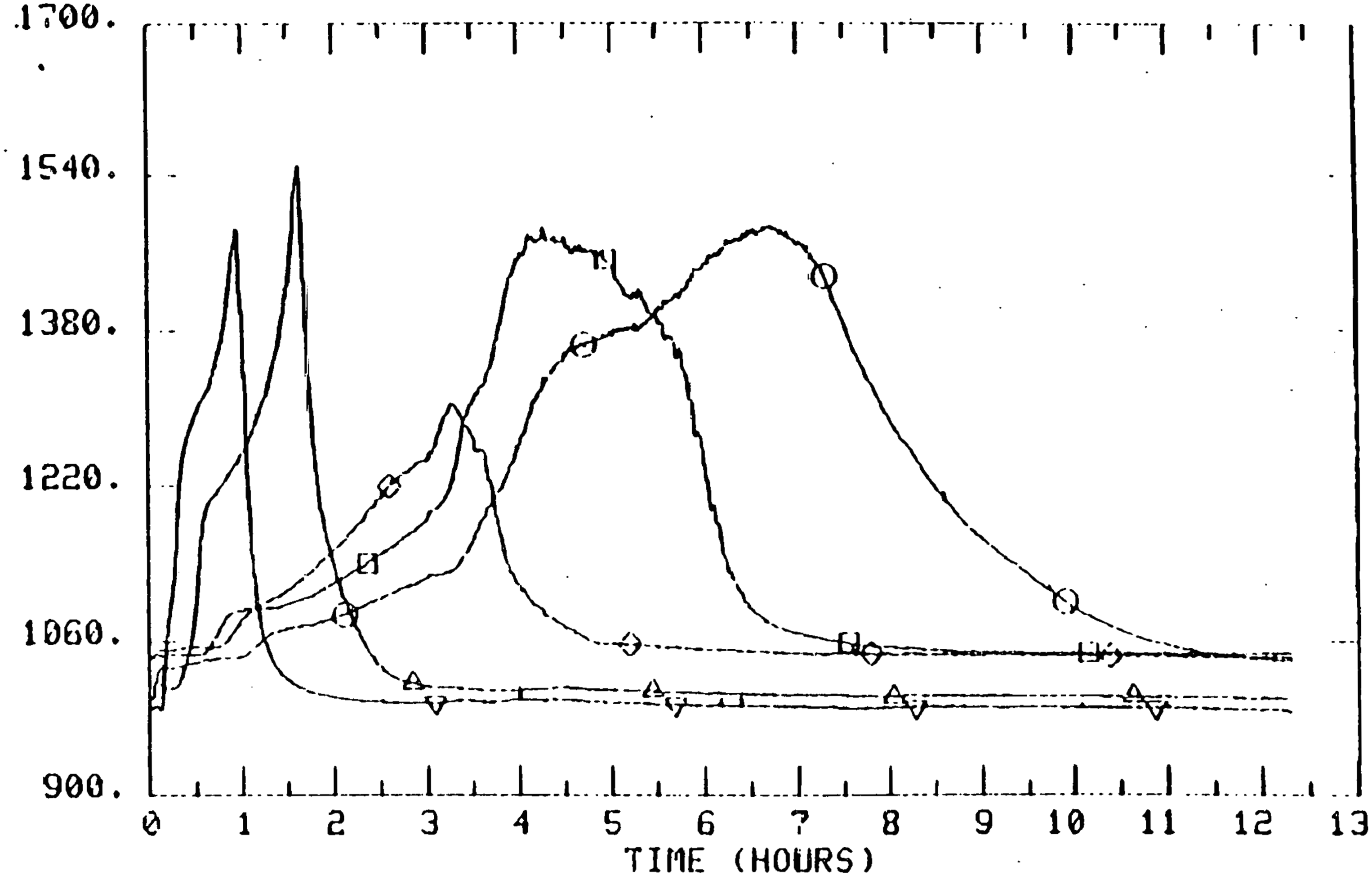
START TIME OF PLOT 17:25: 0 11/13/1983
STOP TIME OF PLOT 16:10: 0 11/16/1983

TEST 017

EXIT FLOWS

FIGURE 29

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN	FREQ	MAX	FREQ
○	851	DEGF	ABOSRBER 5° FROM BOTTOM	1481	1201.	153.1	1001.	2	1480.	1
□	852	DEGF	ABSORBER 15° FROM BOTTOM	1481	1147.	143.7	1044.	2	1488.	1
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	1481	1085.	67.32	1043.	12	1306.	1
△	854	DEGF	ABSORBER 35° FROM BOTTOM	1481	1044.	98.67	999.6	3	1551.	1
▽	855	DEGF	ABSORBER 45° FROM BOTTOM	1481	1020.	86.37	986.4	2	1486.	1



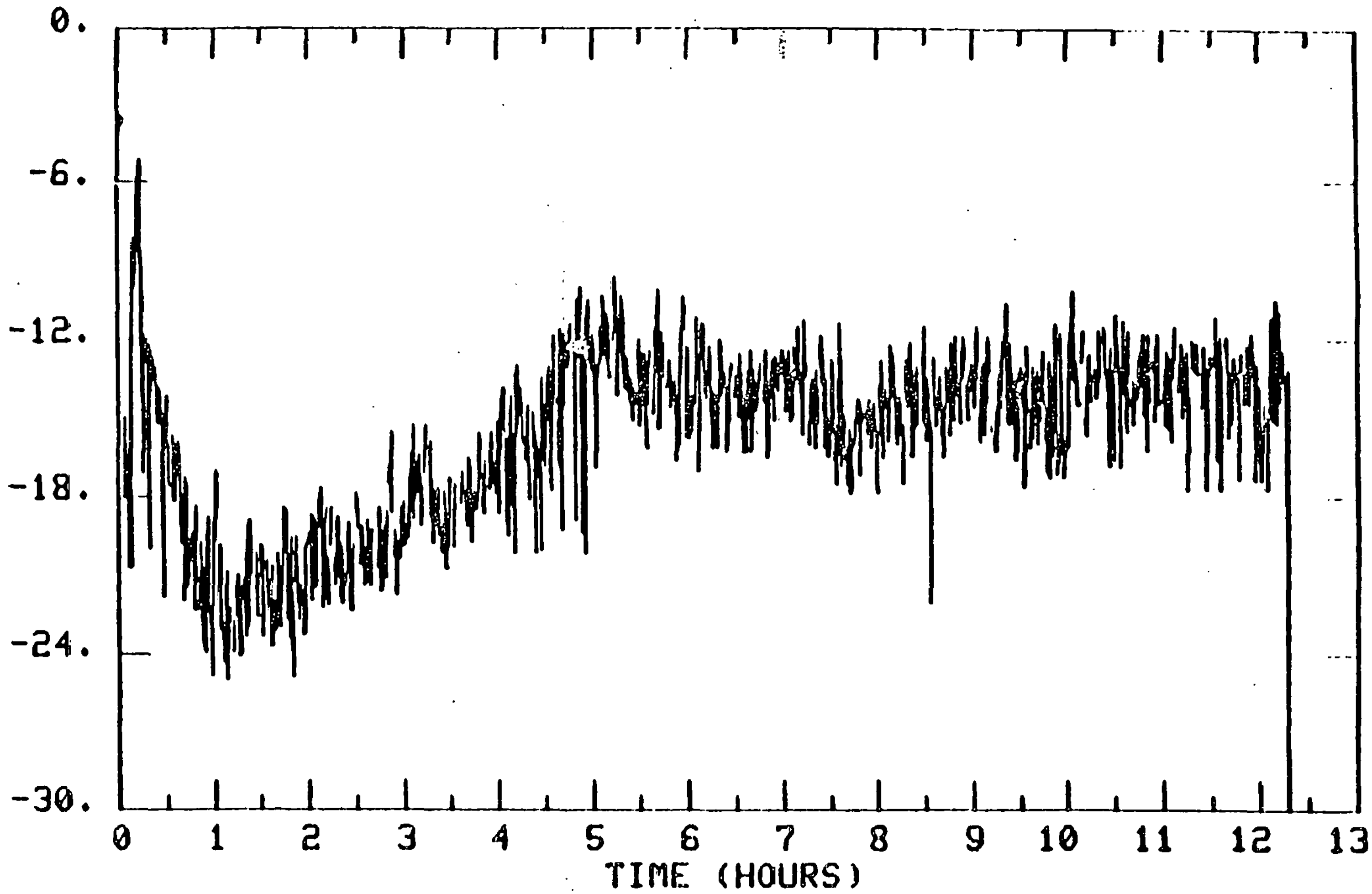
START TIME OF PLOT 17:40: 0 11/16/1983
STOP TIME OF PLOT 6: 0: 0 11/17/1983

TEST 018

REACTOR TEMPERATURES

FIGURE 30

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
294	"H2O	ABSORBER DIFFERENCE PRESSURE	1336	-15.43	3.380	-30.02	1	-3.304	2



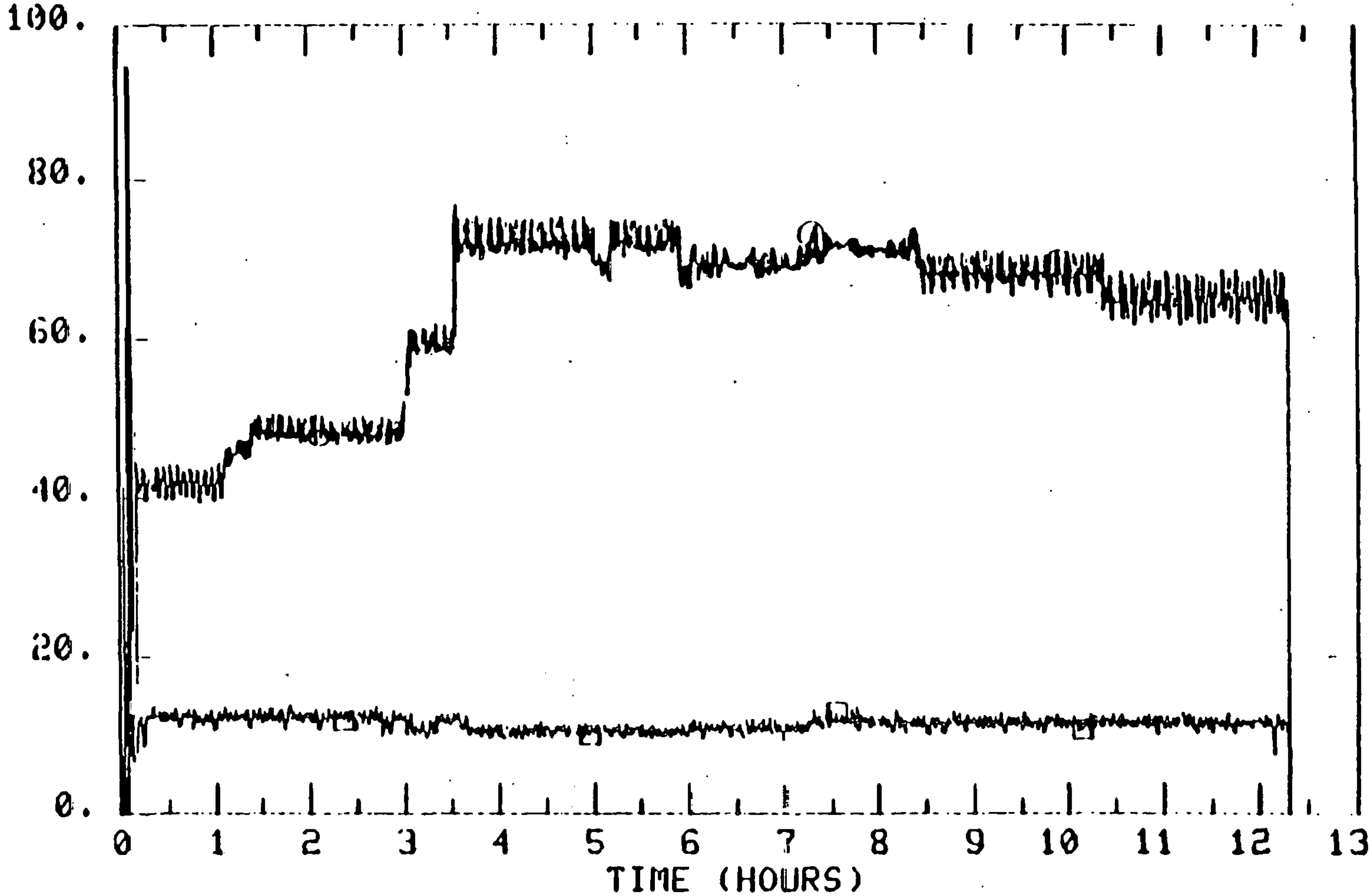
START TIME OF PLOT 17:40: 0 11/16/1983
STOP TIME OF PLOT 6: 0: 0 11/17/1983

TEST 018

REACTOR PRESSURE DROP

FIGURE 31

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN	FREQ	MAX	FREQ
○	3756	SCFH	AIR FLOW LOW RANGE	1475	63.08	12.11	0.0000E+00	9	94.84	1
□	3754	#/HR	STEAM FLOW	1430	11.50	1.410	0.0000E+00	5	42.58	1



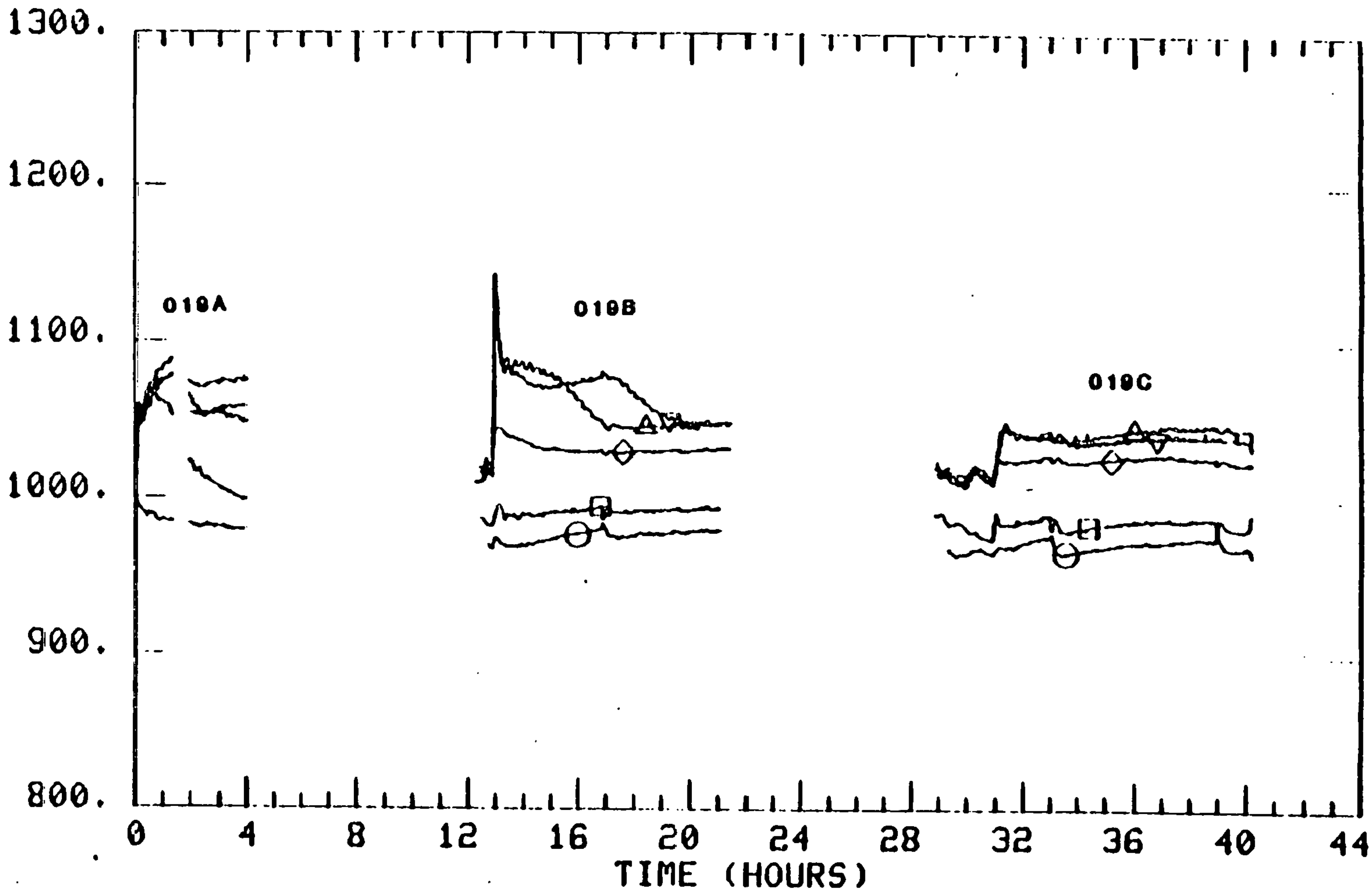
START TIME OF PLOT 17:40: 0 11/16/1983
STOP TIME OF PLOT 6: 0: 0 11/17/1983

TEST 018

STEAM AND AIR FLOWS

FIGURE 32

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	851	DEGF	ABOSRBER 5° FROM BOTTOM	2717	975.1	5.760	964.2	1	997.1	2
□	852	DEGF	ABSORBER 15° FROM BOTTOM	2795	994.0	17.99	974.9	14	1070.	3
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	2854	1033.	12.66	1010.	1	1079.	1
△	854	DEGF	ABSORBER 35° FROM BOTTOM	2836	1052.	19.42	1011.	1	1144.	1
V	855	DEGF	ABSORBER 45° FROM BOTTOM	2862	1050.	19.43	1000.	3	1112.	1



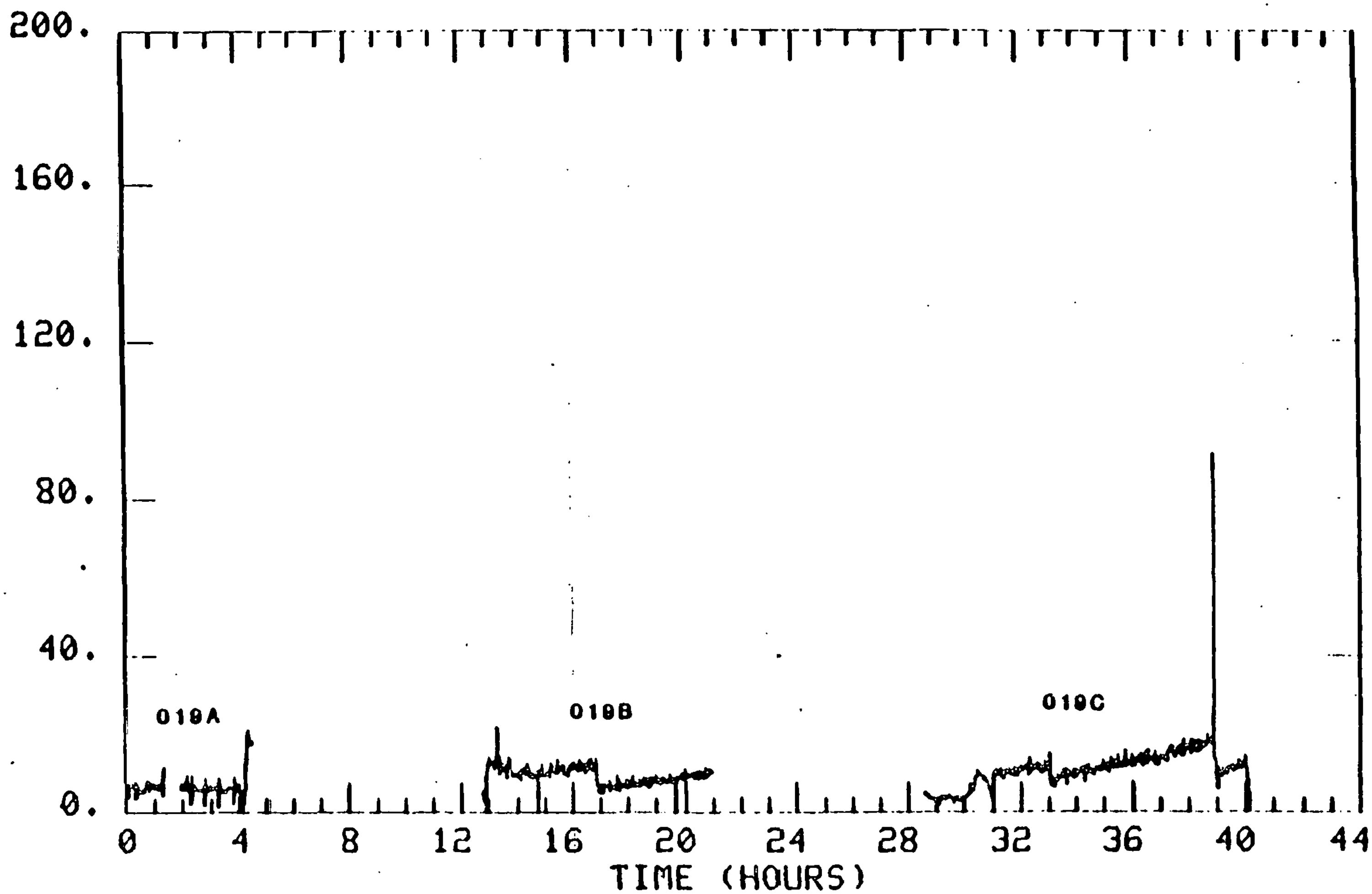
START TIME OF PLOT 7:15: 0 11/17/1983
STOP TIME OF PLOT 23:30: 0 11/18/1983

TEST 019

REACTOR TEMPERATURES

FIGURE 33

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
294	"H2O	ABSORBER DIFFERENCE PRESSURE	2854	9.875	4.107	-27.17	1	91.75	1



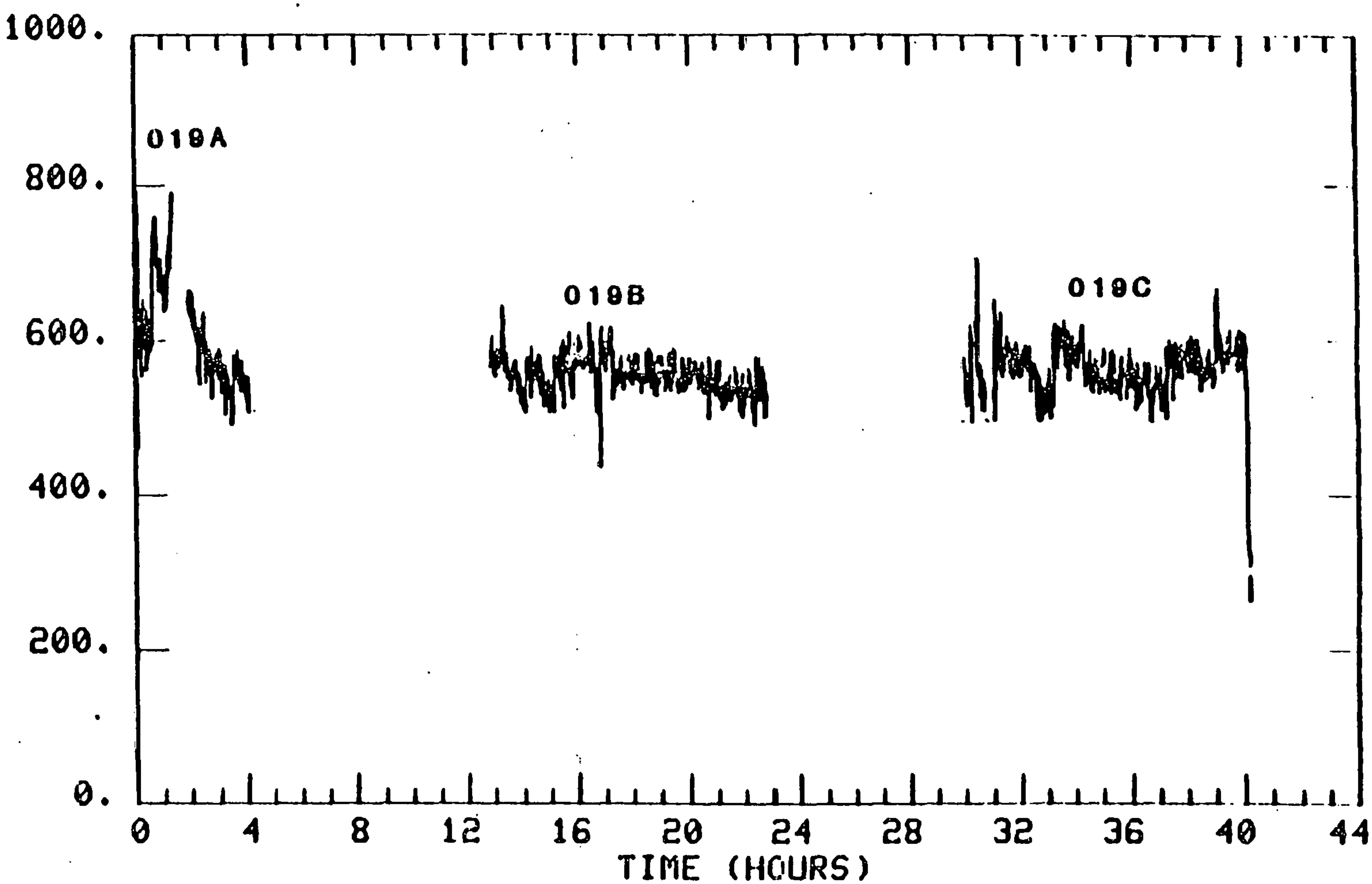
START TIME OF PLOT 7:15: 0 11/17/1983
STOP TIME OF PLOT 23:30: 0 11/18/1983

TEST 019

REACTOR PRESSURE DROP

FIGURE 34

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
3750	SCFH	PROD. GAS OUT OF SYSTEM	2775	570.0	40.05	263.6	1	795.8	1



START TIME OF PLOT 7:15: 0 11/17/1983
STOP TIME OF PLOT 23:30: 0 11/18/1983

TEST 019

EXIT FLOW

FIGURE 35

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT

TEST NO. : 013
DATE STARTED : 17:10 11/07/83
DATE ENDED : 15:00 11/09/83
TOTAL HOURS : 45.8 HOURS
TYPE : SULFIDATION
SULFUR REMOVED: 13.03 LB SB

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
COAL TYPE : BLACKSVILLE
STEAM/COAL MASS RATIO: 0.494
STEAM/AIR MASS RATIO : 0.196

HGD OPERATING CONDITIONS
TEMPERATURE: 1027 F
PRESSURE : 117 PSIG

SPACE VELOCITY: 1006 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)
H_2S	0.598			
H_2O				6.66 %
CO_2	10.52		15.61	
CO	19.38		12.33	
H_2	16.25		21.01	
CH_4	2.93		2.94	
N_2	48.58		46.30	
O_2	0.66		0.68	
C_2H_6	0.25		1.17	
FLOW:				
TOTAL LB MOLES:				
FLOW RATE:	563.9 SCFH		563.9 SCFH	604.1 SCFH

PURPOSE
SULFIDATION AT LOWER SPACE VELOCITY OF 1000 HOURLY AT TEMPERATURE OF 1000 F.

SORBENT ANALYSIS:

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

DATA ABOVE FROM T001

EXIT SULFUR DATA:
H_2S : 2.5 PPM INITIALLLY
CH_4S: 3.1 PPM
SO_2:

CONDENSATE
AQUEOUS : 87.5 LBS (ESTIMATED)
HYDROCARBON : 11.4 LBS (ESTIMATED)
TOTAL : 98.9 LBS (MEASURED)

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 7/8 INCHES
3. FLOW TO COMBUSTOR BY-PASSING HGD.
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
5. INLET DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 7000 # 4 DATA, SAMPLING AT GASIFIER CYCLONE EXIT.
6. REACTOR PRESSURE DROP INCREASED TO 108 INCHES OF WATER BY THE END OF THE RUN.
7. EXIT DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 6000 #2 DATA.

CONCLUSIONS:
1. OPERATION AT LOWER SPACE VELOCITY OF 500 HOURLY EXTENDED THE TIME TO BREAKTHROUGH BY ALMOST A FACTOR OF TWO, COMPARED TO OPERATION AT SPACE VELOCITY OF 1000 HOURLY.
2. PRESSURE DROP INCREASED MORE GRADUALLY AS EXPECTED FOR LOWER FLOW, BUT STILL INDICATES CARBON BUILDUP.
3. SULFUR LOADING INCREASES TO 28.9 % AT LOWER SPACE VELOCITY.

FIGURE 36

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 014
DATE AND TIME STARTED : 17:30 11/9/83
DATE AND TIME ENDED : 18:40 11/10/83
TOTAL HOURS : 25.16
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: 1029 F

PRESSURE : 78.34 INCHES OF WATER (AVERAGE)

SPACE VELOCITY: 468 HOURLY

		INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	41.31 SCFH	15.8 %	BENDIX GC DATA AVAILABLE AT EXIT
H_2O	10.47 #/HR	84.2 %	
N_2	0.0 SCFH		

TOTAL LB MOLES:
FLOW RATE : 261.8 SCFH

PURPOSE

FIRST REGENERATION OF SULFIDED SORBENT FROM TEST 013.
GASIFIER RUN 103.

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:

H_2S : UP TO 3 % AT MID RUN (BY BENDIX GC)

SO_2 : HIGH SO_2 (> 16 %) INITIALLY .
0.8 % SO_2 AT END OF REGENERATION.

CONDENSATE
AQUEOUS : 272.2 LBS
HYDROCARBON :
TOTAL : 272.2 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-10769 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.559 CUBIC FOOT
3. FIRST REGENERATION ON SULFIDED SORBENT
4. UP TO 3 % H_2S WAS OBSERVED AT EXIT BETWEEN 5-12 HOURS
INTO THE REGENERATION AS MEASURED BY BENDIX GC 6000 #2.
5. MAXIMUM TEMPERATURE REACHED WAS 1699 F.

CONCLUSIONS

1. REGENERATION COMPLETED.
2. CARBON APPEARS TO BE BURNED/GASIFIED OFF WITHIN THE
FIRST 11 HOURS AS OBSERVED BY CO2 CONCENTRATION AT EXIT.

FIGURE 37

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT

TEST NO. : 015
DATE STARTED : 19:05 11/10/83
DATE ENDED : 11:10 11/12/83
TOTAL HOURS : 40.08 HOURS
TYPE : SULFIDATION
SULFUR REMOVED: 10.51 LB SB

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
COAL TYPE : ARKWRIGHT

STEAM/COAL MASS RATIO: 0.491
STEAM/AIR MASS RATIO : 0.195

HGD OPERATING CONDITIONS
TEMPERATURE: 1000 F (AVERAGE)

PRESSURE : 119.5 PSIG (AVERAGE)

[illegible]E VELOCITY: 995 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)
H_2S	0.559			
H_2O				7.3 %
CO_2	8.78		14.14	
CO	20.57		13.69	
H_2	15.31		20.53	
CH_4	3.02		2.74	
N_2	45.65		43.24	
O_2	0.63		0.57	
C_2H_6	0.29		0.29	
FLOW:				
TOTAL LB MOLES:				
FLOW RATE:	557 SCFH			601 SCFH

PURPOSE
SECOND SULFIDATION AT LOWER SPACE VELOCITY OF 1000 HOURLY AND TEMPERATURE OF 1000 F.

SORBENT ANALYSIS:

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

EXIT SULFUR DATA:
H_2S : 25 PPM INITIALLLY GRADUALLY DOWN TO 3 PPM

C_4H_4S:
SO_2 : 20 PPM INITIALLY, DOWN TO UNDETECTABLE WITHIN 5 HOURS

CONDENSATE	ARKWRIGHT COAL		
AQUEOUS	: 83.9	LBS	(ESTIMATED)
HYDROCARBON	: 10.2	LBS	(ESTIMATED)
TOTAL	: 94.6	LBS	(MEASURED)

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10739 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 7/8 INCHES
3. FLOW TO COMBUSTOR BY-PASSING HGD REACTOR.
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
5. INLET DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 7900 #4 DATA, SAMPLING AT GASIFIER CYCLONE EXIT.
6. REACTOR PRESSURE DROP INCREASED GRADUALLY TO ABOVE 160 INCHES OF WATER.

7. EXIT DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 6000 #2 DATA, SAMPLING AT HGD EXIT.

CONCLUSIONS:

1. SECOND SULFIDATION AT LOWER SPACE VELOCITY RESULTED IN TIME TO BREAKTHROUGH SLIGHTLY SHORTER THAN THE FIRST SULFIDATION.
2. SULFUR REMOVED IS 19 % LOWER THAN DURING TEST 013, THE FIRST SULFIDATION.

FIGURE 38 .

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 016
DATE AND TIME STARTED : 12:15 11/12/83
DATE AND TIME ENDED : 08:30 11/13/83
TOTAL HOURS : 20.25
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

PURPOSE

SECOND REGENERATION OF SULFIDED SORBENT, GASIFIER RUN 103.

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)		

HGD OPERATING CONDITIONS

INITIAL TEMPERATURE: 1005 F (AVERAGE)

PRESSURE : 3.9 PSIG (AVERAGE AT REACTOR INLET)

SPACE VELOCITY: 494 HOURLY

		INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	66.91 SCFH	24.3 %	BENDIX GC DATA AVAILABLE AT EXIT
H_2O	9.93 #/HR	75.7 %	
N_2	0 SCFH		

TOTAL LB MOLES:
FLOW RATE : 276 SCFH

EXIT SULFUR DATA:

H_2S :

SO_2 : HIGH SO_2 (> 10 %) INITIALLY .
0.05 % SO_2 AT END OF REGENERATION.

CONDENSATE		
AQUEOUS	:	271.5 LBS (MEASURED)
HYDROCARBON	:	
TOTAL	:	271.5 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-10789 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.557 CUBIC FOOT
3. SECOND REGENERATION ON SULFIDED SORBENT

4. MAXIMUM TEMPERATURE REACHED WAS 1501 F.

CONCLUSIONS

1. REGENERATION COMPLETED.

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT

TEST NO. : 017
DATE STARTED : 17:25 11/13/83
DATE ENDED : 16:10 11/16/83 (WITH INTERUPTIONS)
TOTAL HOURS : 55.6 (42.6 HRS ON KITTANNING, 13 HRS ON ARKWRIGHT)
TYPE : SULFIDATION
SULFUR REMOVED: (2.35 LB S8 ON KITT) (3.83 LB S8 ON ARK)
6.18 LB S8 TOTAL
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

	KITTANNING	ARKWRIGHT
COAL TYPE	START TO 20 00 11/15	20 00 11/15 TO END
STEAM/COAL MASS RATIO:	0.47	0.48
STEAM/AIR MASS RATIO :	0.20	0.20

HGD OPERATING CONDITIONS
TEMPERATURE: 1017 F (WEIGHTED AVERAGE)

PRESSURE : 168 PSIG (WEIGHTED AVERAGE)

SPACE VELOCITY: 999 HOURLY

	INLET MOLE% DRY BASIS (OIL FREE) KITT	ARK	INLET MOLE% WET BASIS (OIL FREE)	EXIT MOLE% DRY BASIS (OIL FREE) KITT	ARK	EXIT MOLE% WET BASIS (OIL FREE) KITT	ARK
H_2S	0.118	0.627					
H_2O						5.7 %	
CO_2	8.83	8.33		13.10	13.53		
CO	21.92	22.06		15.56	14.89		
H_2	16.59	16.02		20.28	19.91		
CH_4	2.99	3.31		2.95	3.50		
N_2	46.08	46.52		44.81	44.22		
O_2	0.59	0.63		0.66	0.57		
C_2H_6	0.27	0.30		0.33	3.59		
FLOW:							
TOTAL LB MOLES:							
FLOW RATE:	559	SCFH				SCFH	

PURPOSE
THIRD SULFIDATION AT LOWER SPACE VELOCITY OF 1000 HOURLY AND TEMPERATURE OF 1000 F.

SORBENT ANALYSIS:

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

EXIT SULFUR DATA:
H_2S : 10 PPM INITIALLLY GRADUALLY DOWN TO 3.2 PPM

C_4H_4S:
SO_2 : 12 PPM INITIALLY, GRADUALLY DOWN TO 2 PPM

CONDENSATE		KITTANNING COAL			ARKWRIGHT COAL
AQUEOUS	:	69	LBS	(MEASURED)	
HYDROCARBON	:	4	LBS	(MEASURED)	
TOTAL	:	72.3	LBS	(MEASURED)	21.2

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES CS-346 1/2 S SR-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 7/8 INCHES
3. NO FLOW TO COMBUSTOR.
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
5. INLET DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 7000 #4 DATA, SAMPLED AT S4, GASIFIER CYCLONE EXIT.
6. REACTOR DELTA P REMAINED ESSENTIALLY CONSTANT AT 6 INCHES OF WATER.
7. EXIT DRY BASIS GAS ANALYSIS IS AVERAGE OF BENDIX GC 6000 #2 DATA.

CONCLUSIONS:
1. THIRD SULFIDATION AT LOWER SPACE VELOCITY RESULTED N 41 % LESS SULFUR REMOVED COMPARED TO SECOND SULFIDATION.
2. REACTOR PRESSURE DROP DID NOT INCREASE. THIS IS ATTRIBUTED TO SMALLER PARTICLE SIZE OF INCOMING PARTICULATES.

FIGURE 40

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 018
DATE AND TIME STARTED : 17:40 11/16/83
DATE AND TIME ENDED : 06:00 11/17/83
TOTAL HOURS : 12.33
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

HSD OPERATING CONDITIONS

INITIAL TEMPERATURE: 999.6 F

PRESSURE : INCHES OF WATER (AVERAGE)

FACE VELOCITY: 542 HOURLY

			INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	63.02	SCFH	20.8 %	
H_2O	11.40	#/HR	79.2 %	
N_2	0	SCFH		

TOTAL LB MOLES:
FLOW RATE : 303 SCFH

PURPOSE

REGENERATION OF SULFIDED SORBENT FROM T017 GASIFIIER RUN 103.

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:

H_2S :

SO_2 : >10 % INITIALLY , HIGH OF >16 % AFTER 3.8 HOURS
2000 PPM AT END OF RUN .

CONDENSATE
AQUEOUS : 148.4 LBS
HYDROCARBON :
TOTAL : 148.4 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-10789 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.559 CUBIC FOOT
3. SECOND REGENERATION ON SULFIDED SORBENT (SECOND BATCH)

4. MAXIMUM TEMPERATURE REACHED WAS 1551 F.
5. EXIT BENDIX GC WAS OPERATING AND PROVIDED O2 BREAKTHROUGH
CURVE, AND H_2,CO,CO_2,H_2S COMPOSITIONS AT EXIT.

CONCLUSIONS:
1. REGENERATION COMPLETED.

FIGURE 41

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT

TEST NO. : 019
DATE STARTED : 07:15 11/17/83
DATE ENDED : 23:30 11/18/83 (WITH INTERUPTIONS)
TOTAL HOURS : 23.49
TYPE : SULFIDATION
SULFUR REMOVED: 4.33 LB S8

PURPOSE
FOURTH SULFIDATION AT LOWER SPACE VELOCITY OF 1000 HOURLY AT TEMPERATURE OF 1000 F.

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
COAL TYPE : ARKWRIGHT

STEAM/COAL MASS RATIO: 0.474 (WEIGHTED AVERAGE)
STEAM/AIR MASS RATIO : 0.192 (WEIGHTED AVERAGE)

HGD OPERATING CONDITIONS
TEMPERATURE: [illegible] F (WEIGHTED AVERAGE)
PRESSURE : 205 PSIG (WEIGHTED AVERAGE)

SPACE VELOCITY: 1012 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)
H_2S	0.336			
H_2O				8.5
CO_2	8.71		12.44	
CO	23.81		15.21	
H_2	16.11		18.46	
CH_4	3.78		3.33	
N_2	50.49		47.48	
O_2	.68		.67	
C_2H_6	.36		.51	
FLOW:				
TOTAL LB MOLES:				
FLOW RATE:	566 SCFH			618 SCFH

SORBENT ANALYSIS:

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

EXIT SULFUR DATA:
H_2S : [illegible] PPM INITIALLY
COS :
SO_2 : 3 PPM INITIALLY

CONDENSATE		ARKWRIGHT COAL		
AQUEOUS	:	58.7	LBS	(ESTIMATED)
HYDROCARBON	:	7.5	LBS	(ESTIMATED)
TOTAL	:	66.2	LBS	(MEASURED)

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERES
2. OVERALL PACKED LENGTH 53 7/8 INCHES
3. NO FLOW TO COMBUSTOR.
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
5. GAS COMPOSITIONS AT INLET OBTAINED BY WEIGHTED AVERAGE OF BENDIX GC 7000 #4 DATA SAMPLING AT S-4 GASIFIER CYCLONE EXIT
6. GAS COMPOSITIONS AT EXIT OBTAINED BY WEIGHTED AVERAGE OF BENDIX GC 2000 AT #2 DATA, SAMPLING AT HGD EXIT.
7. REACTOR PRESSURE DROP REACHED A MAXIMUM OF 13 INCHES OF WATER.

CONCLUSIONS:
1. SULFUR REMOVED IS 30% LESS THAN PREVIOUS SULFIDATION.

FIGURE 42

REMOVAL OF HYDROGEN SULFIDE FROM COAL GAS BY ZINC FERRITE

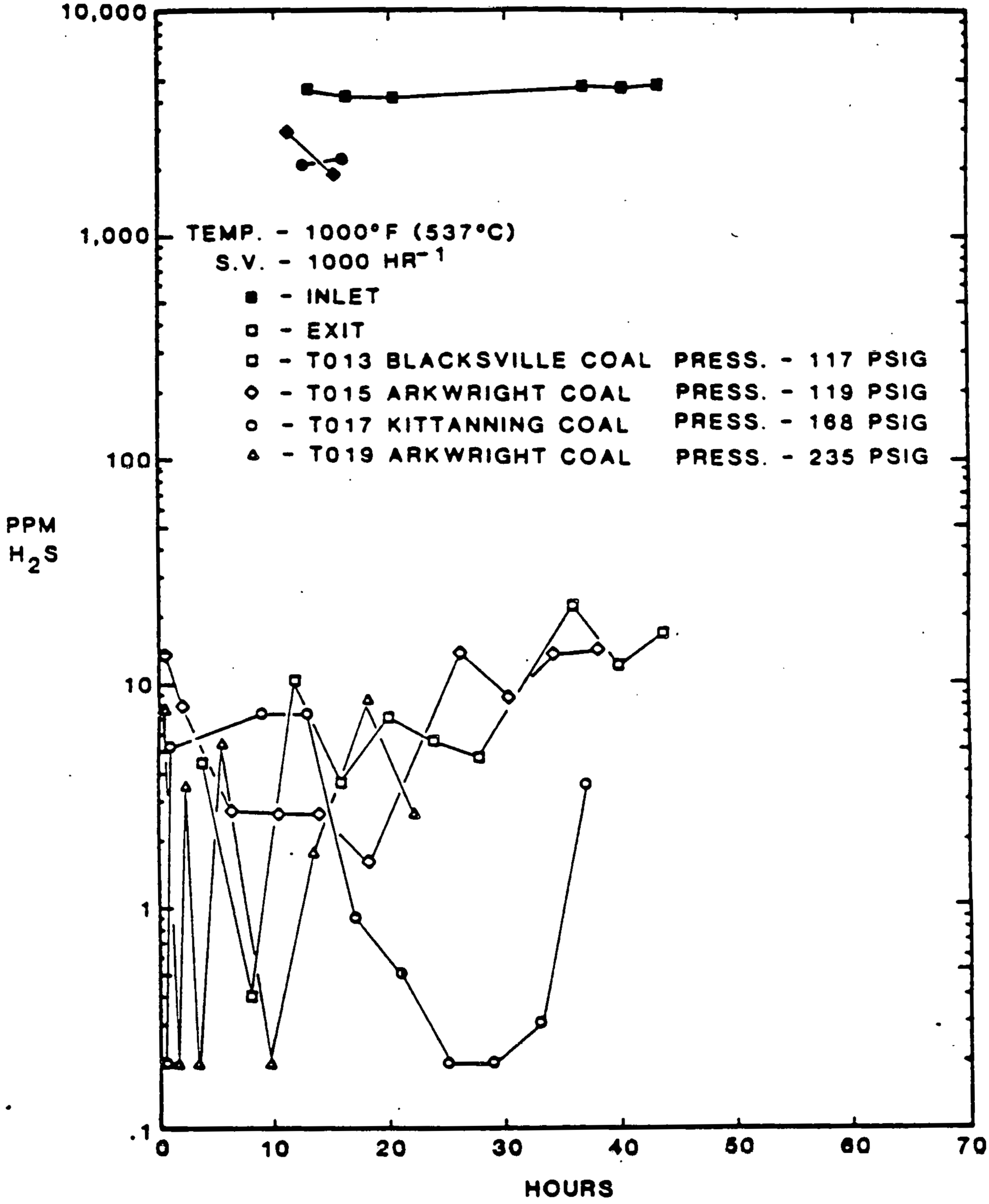
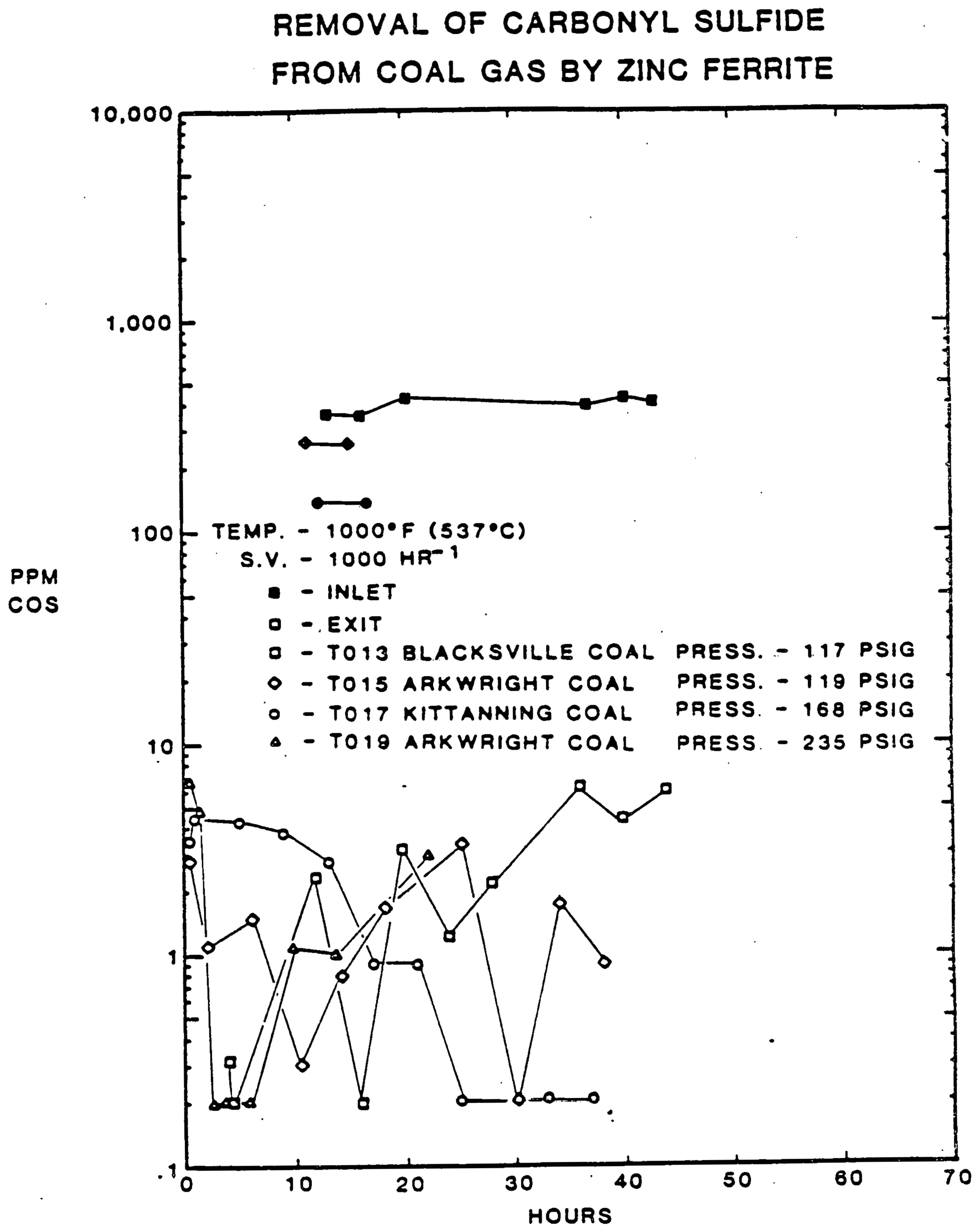


FIGURE 43



REMOVAL OF CARBONYL SULFIDE
FROM COAL GAS BY ZINC FERRITE
10,000
1,000
100
10
1
.1
PPM
COS
TEMP. - 1000°F (537°C)
S.V. - 1000 HR-1
■ - INLET
□ - EXIT
□ - T013 BLACKSVILLE COAL PRESS. - 117 PSIG
◇ - T015 ARKWRIGHT COAL PRESS. - 119 PSIG
○ - T017 KITTANNING COAL PRESS. - 168 PSIG
△ - T019 ARKWRIGHT COAL PRESS. - 235 PSIG
0
10
20
30
40
50
60
70
HOURS

FIGURE 44

REMOVAL OF CARBON DISULFIDE FROM COAL GAS BY ZINC FERRITE

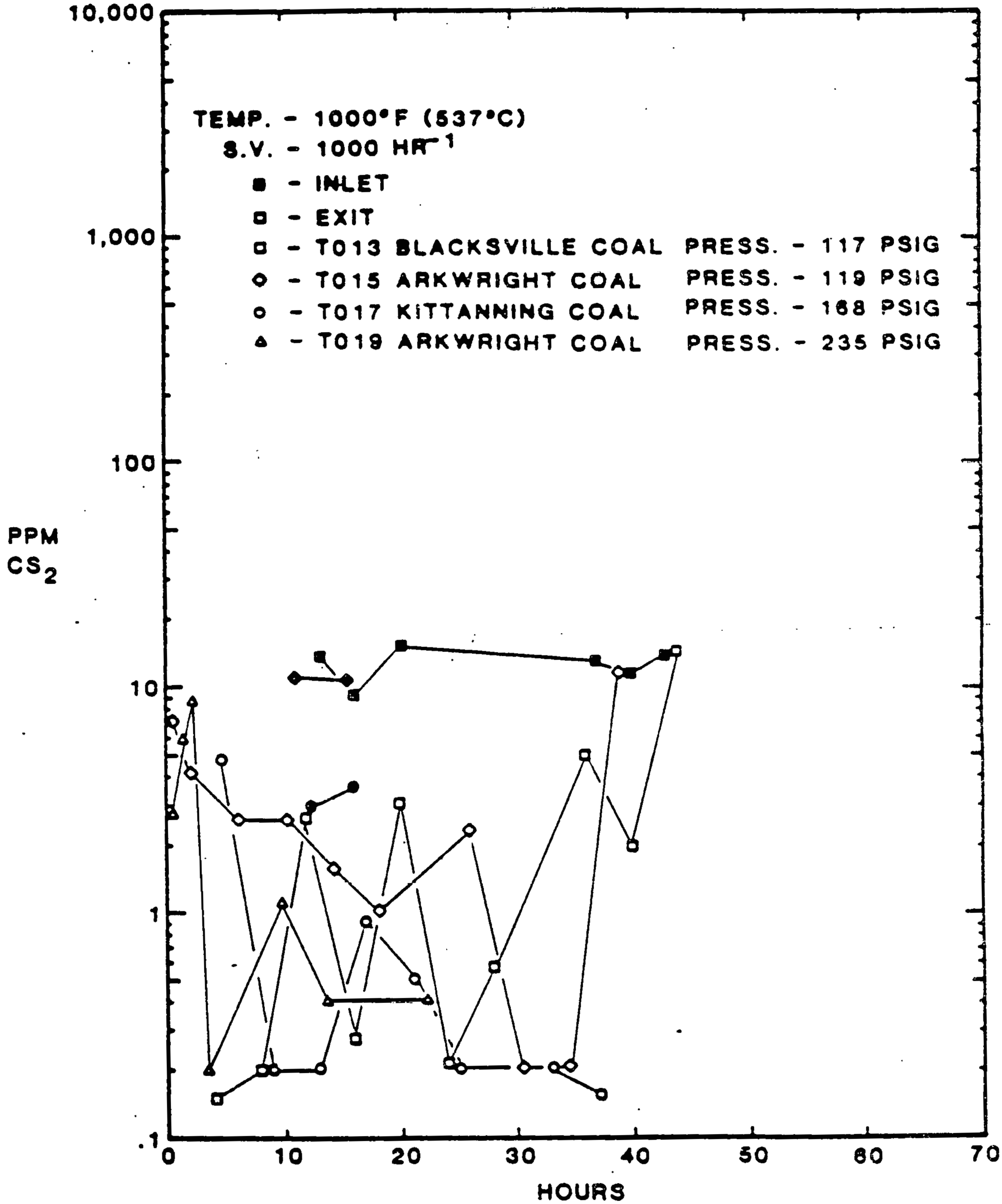


FIGURE 45

REMOVAL OF THIOPHENE FROM COAL GAS BY ZINC FERRITE

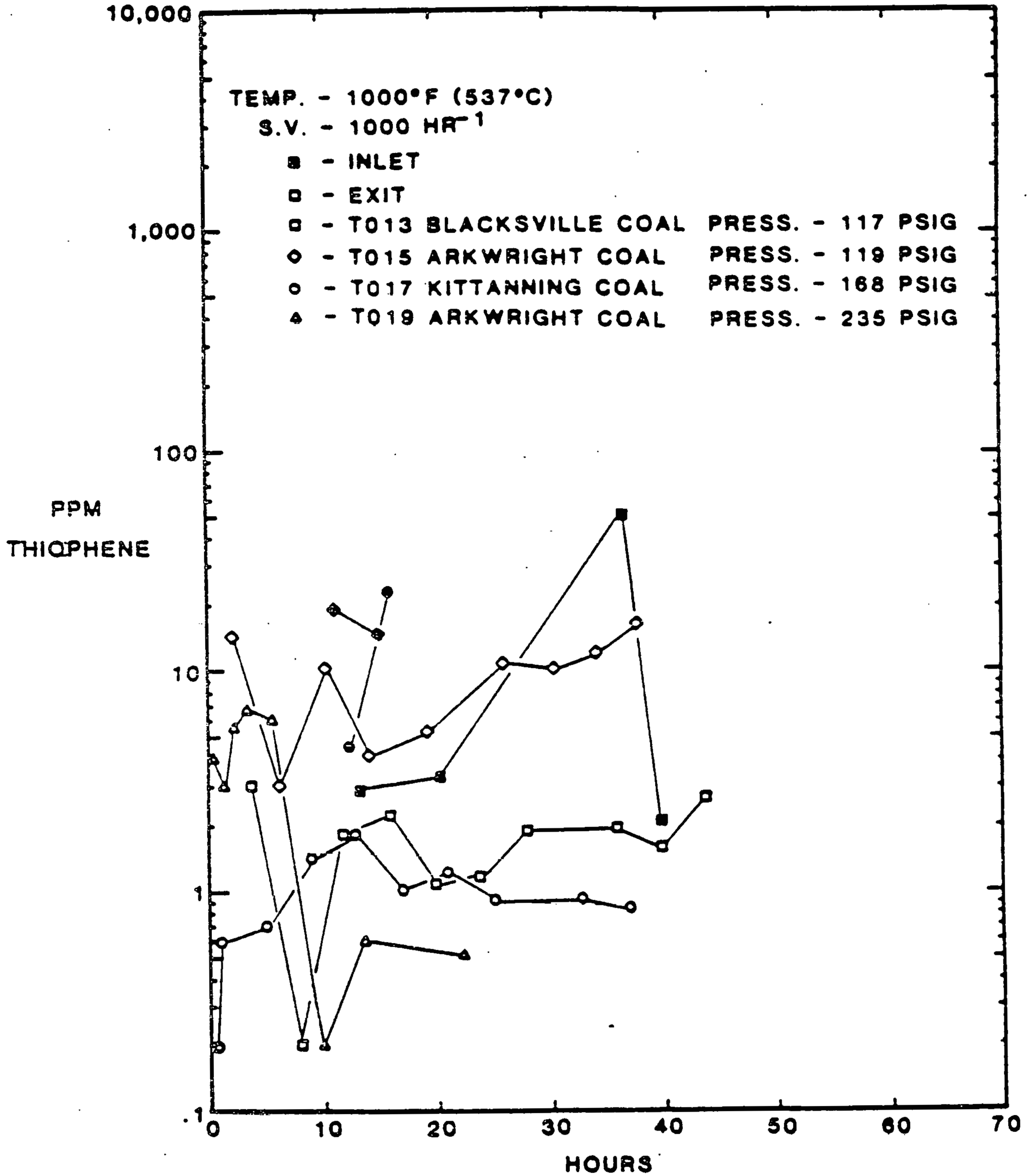


Figure 46

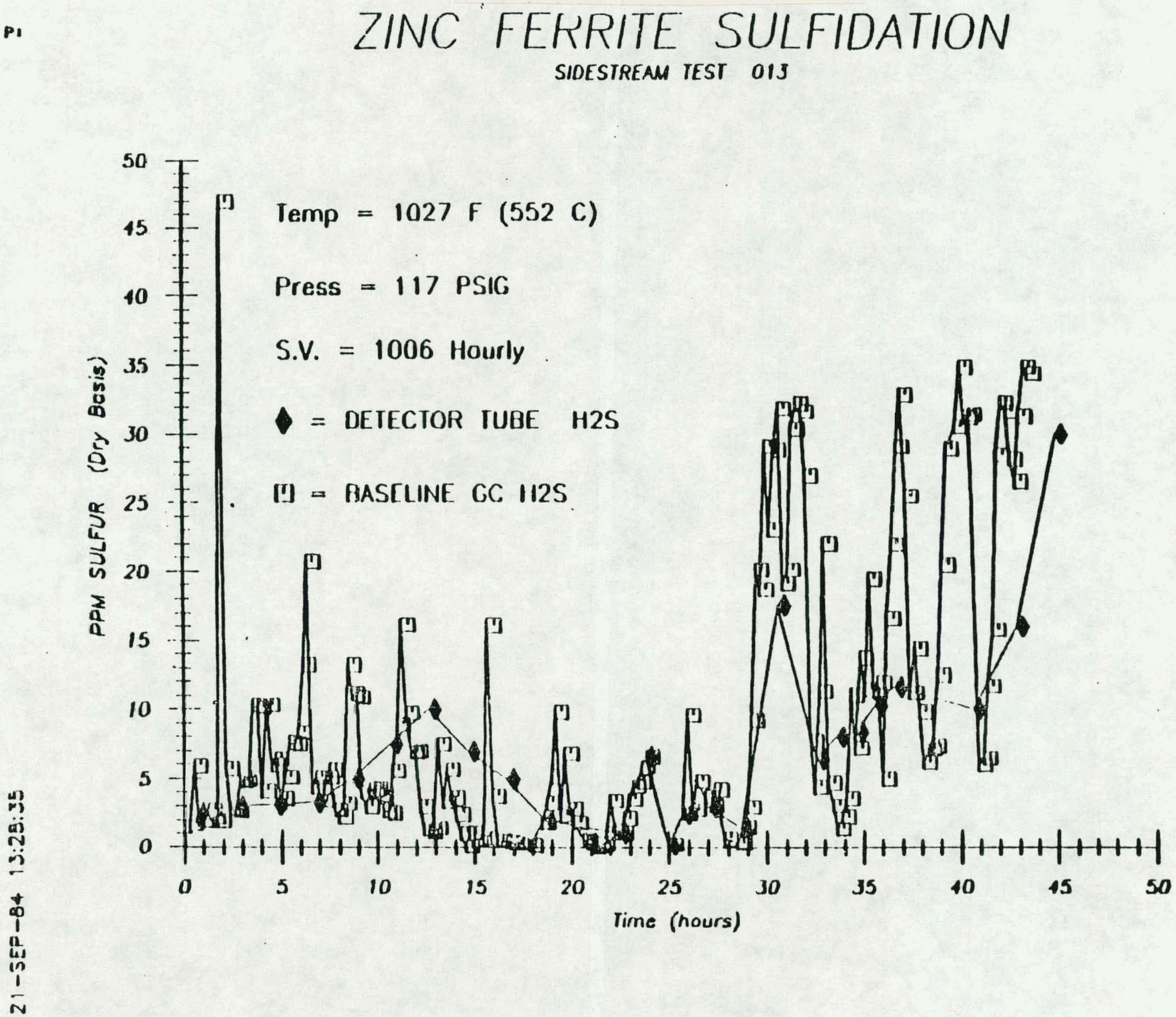

ZINC FERRITE SULFIDATION
SIDESTREAM TEST 013
Temp = 1027 F (552 C)
Press = 117 PSIG
S.V. = 1006 Hourly
= DETECTOR TUBE H2S
= BASELINE GC H2S
PPM SULFUR (Dry Basis)
Time (hours)
0 5 10 15 20 25 30 35 40 45 50
P1
21-SEP-84 13:28:35

Figure 47

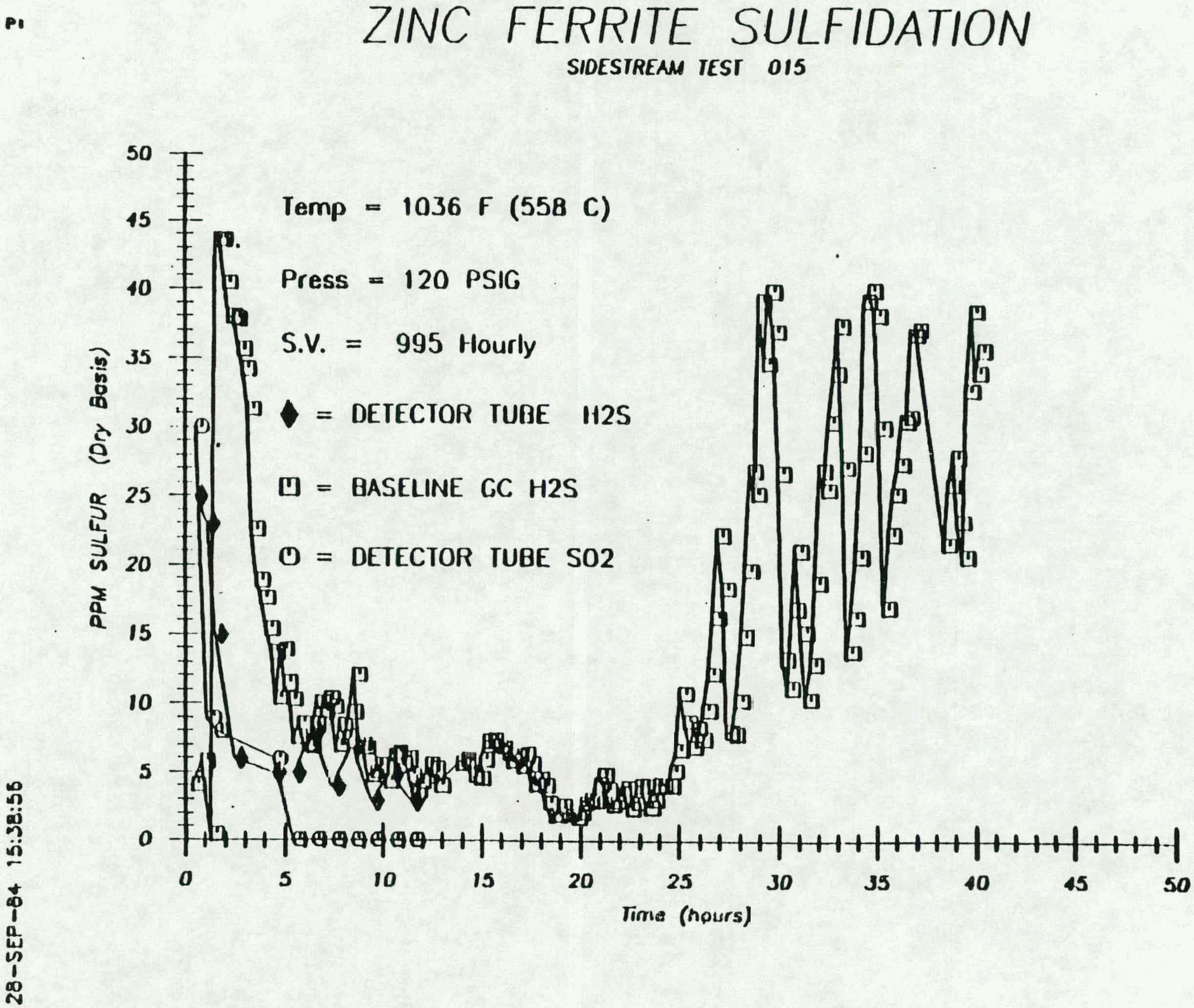
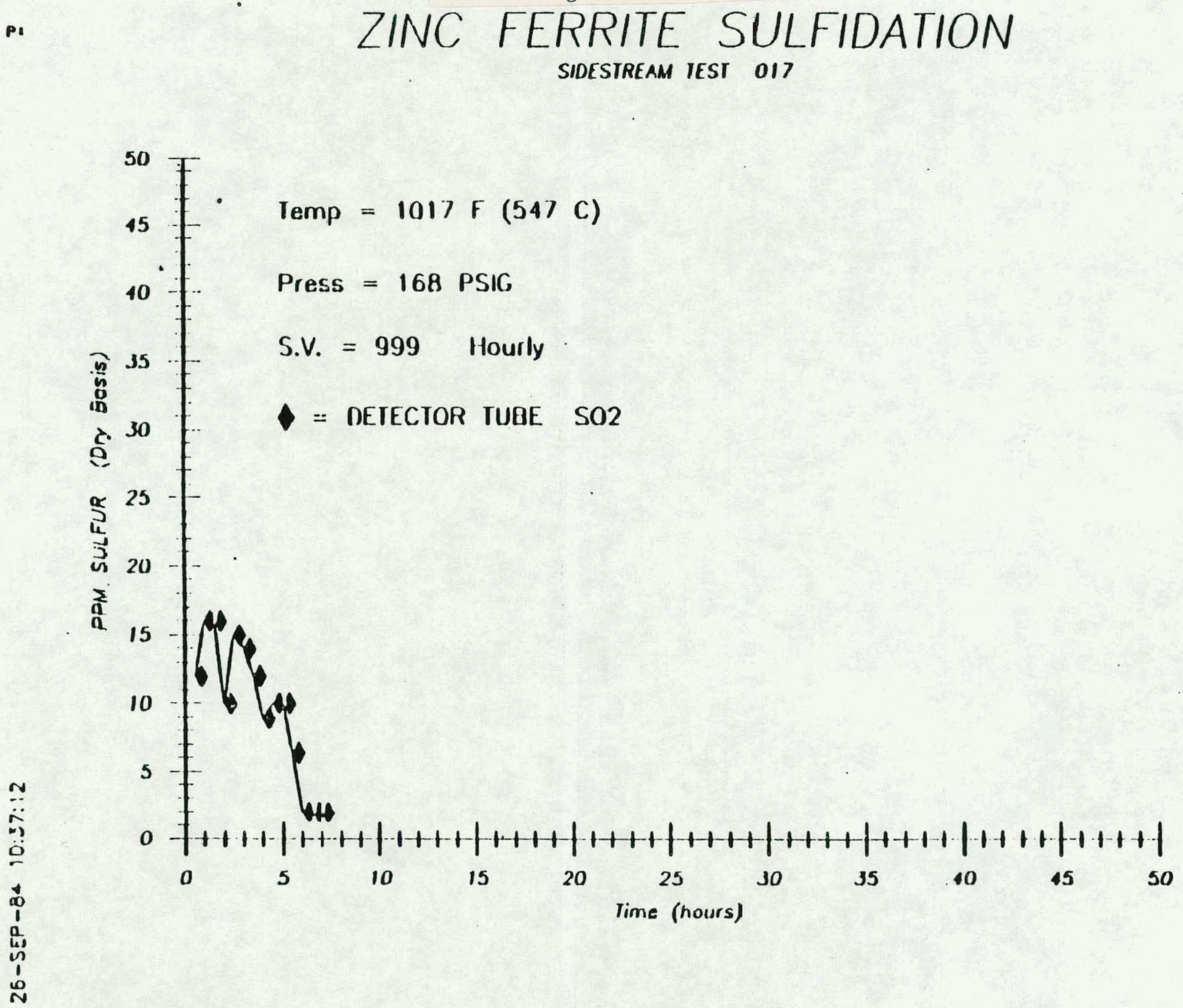
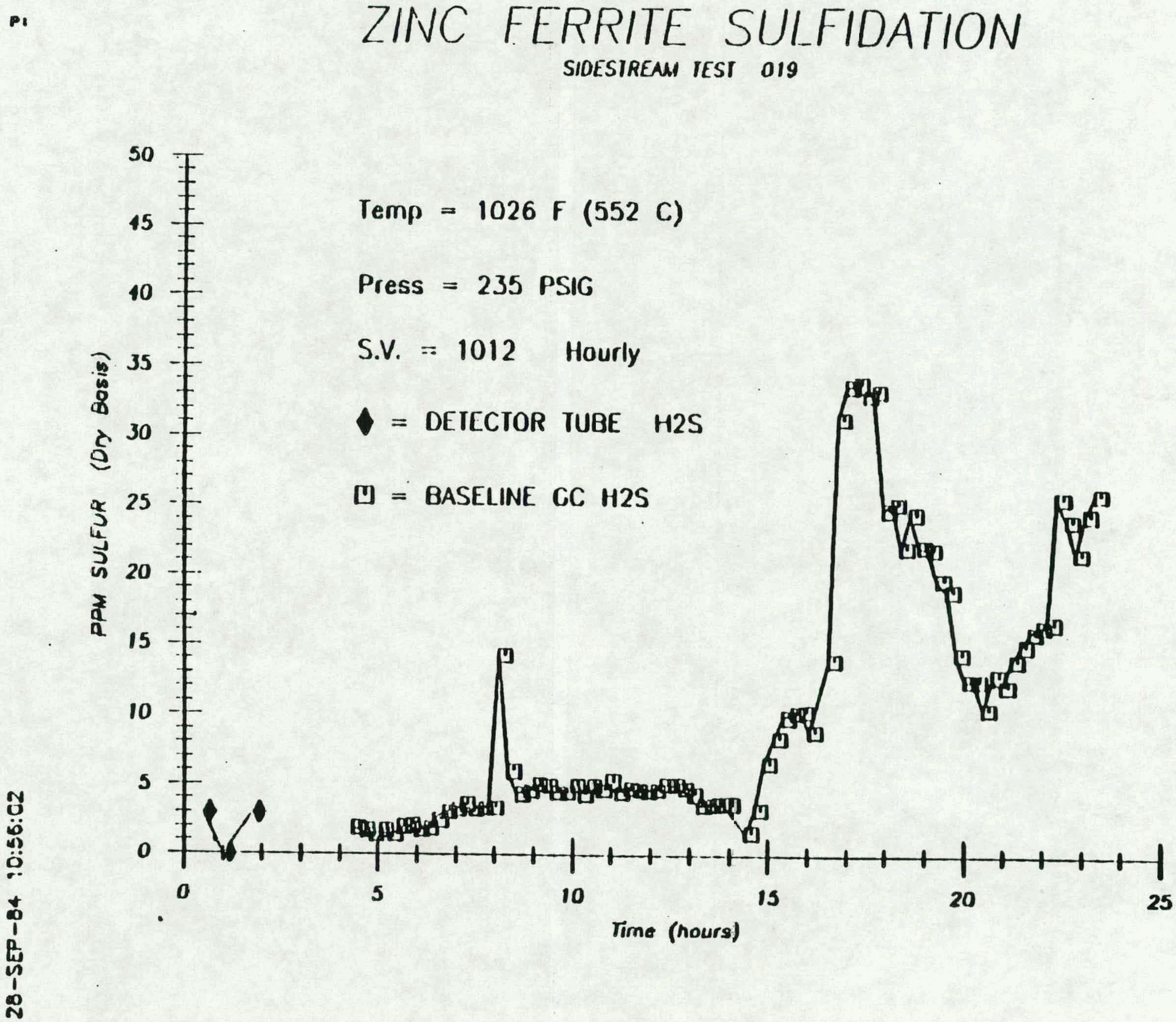

P1
ZINC FERRITE SULFIDATION
SIDESTREAM TEST 015
Temp = 1036 F (558 C)
Press = 120 PSIG
S.V. = 995 Hourly
♦ = DETECTOR TUBE H2S
□ = BASELINE GC H2S
O = DETECTOR TUBE SO2
PPM SULFUR (Dry Basis)
Time (hours)
0
5
10
15
20
25
30
35
40
45
50
28-SEP-84 15:38:56

Figure 48



ZINC FERRITE SULFIDATION
SIDESTREAM TEST 017
Temp = 1017 F (547 C)
Press = 168 PSIG
S.V. = 999 Hourly
= DETECTOR TUBE SO2
PPM SULFUR (Dry Basis)
Time (hours)
0
5
10
15
20
25
30
35
40
45
50
26-SEP-84 10:37:12

Figure 49



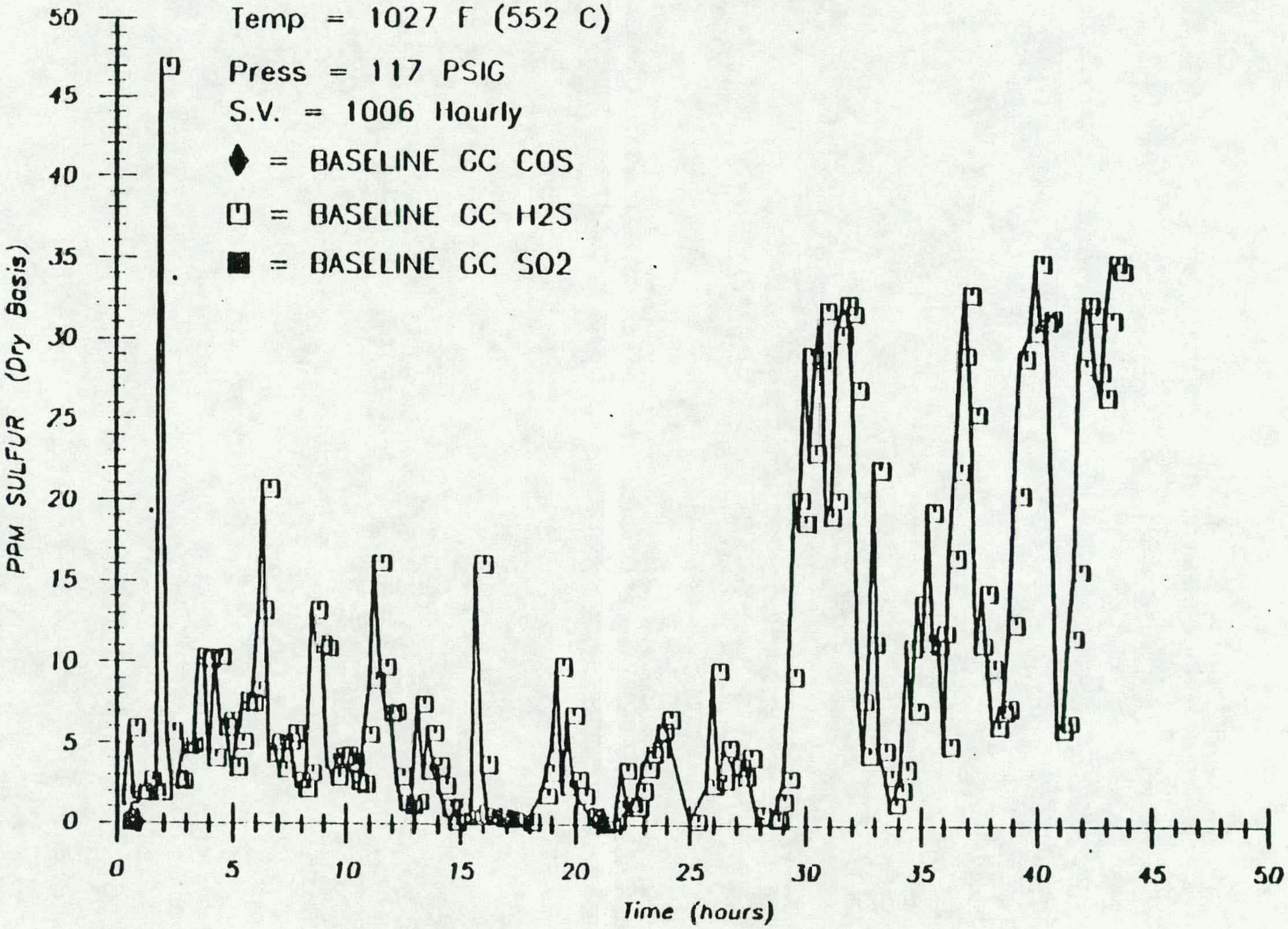
P1
ZINC FERRITE SULFIDATION
SIDESTREAM TEST 019
Temp = 1026 F (552 C)
Press = 235 PSIG
S.V. = 1012 Hourly
= DETECTOR TUBE H2S
= BASELINE GC H2S
PPM SULFUR (Dry Basis)
0
5
10
15
20
25
30
35
40
45
50
Time (hours)
28-SEP-84 10:56:02

FIGURE 50

P1

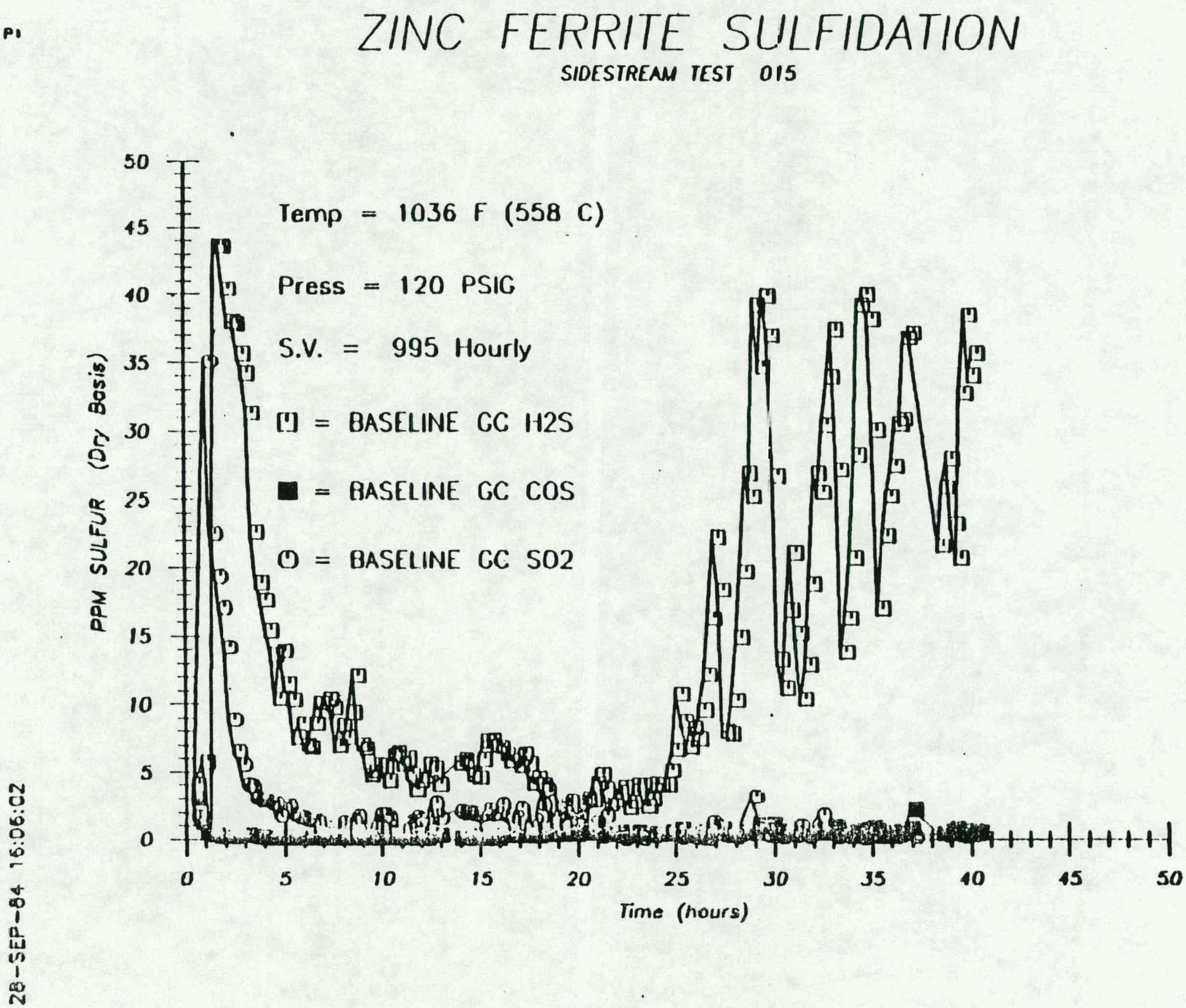
ZINC FERRITE SULFIDATION

SIDESTREAM TEST 013

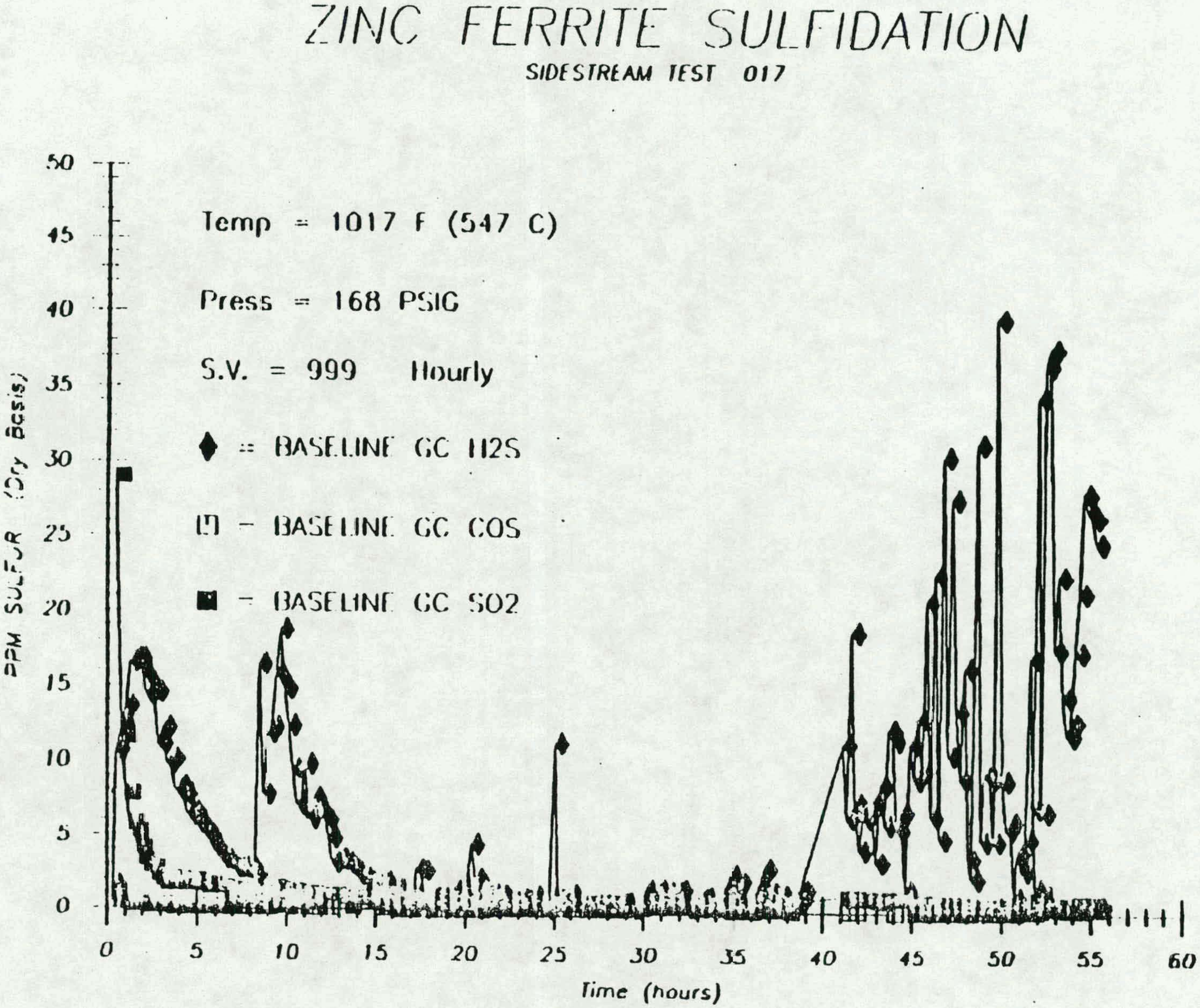


21-SEP-84 13:54:42

FIGURE 51


ZINC FERRITE SULFIDATION
SIDESTREAM TEST 015
Temp = 1036 F (558 C)
Press = 120 PSIG
S.V. = 995 Hourly
= BASELINE GC H2S
= BASELINE GC COS
= BASELINE GC SO2
PPM SULFUR (Dry Basis)
Time (hours)
0
5
10
15
20
25
30
35
40
45
50
28-SEP-84 16:06:02

FI 'E 52

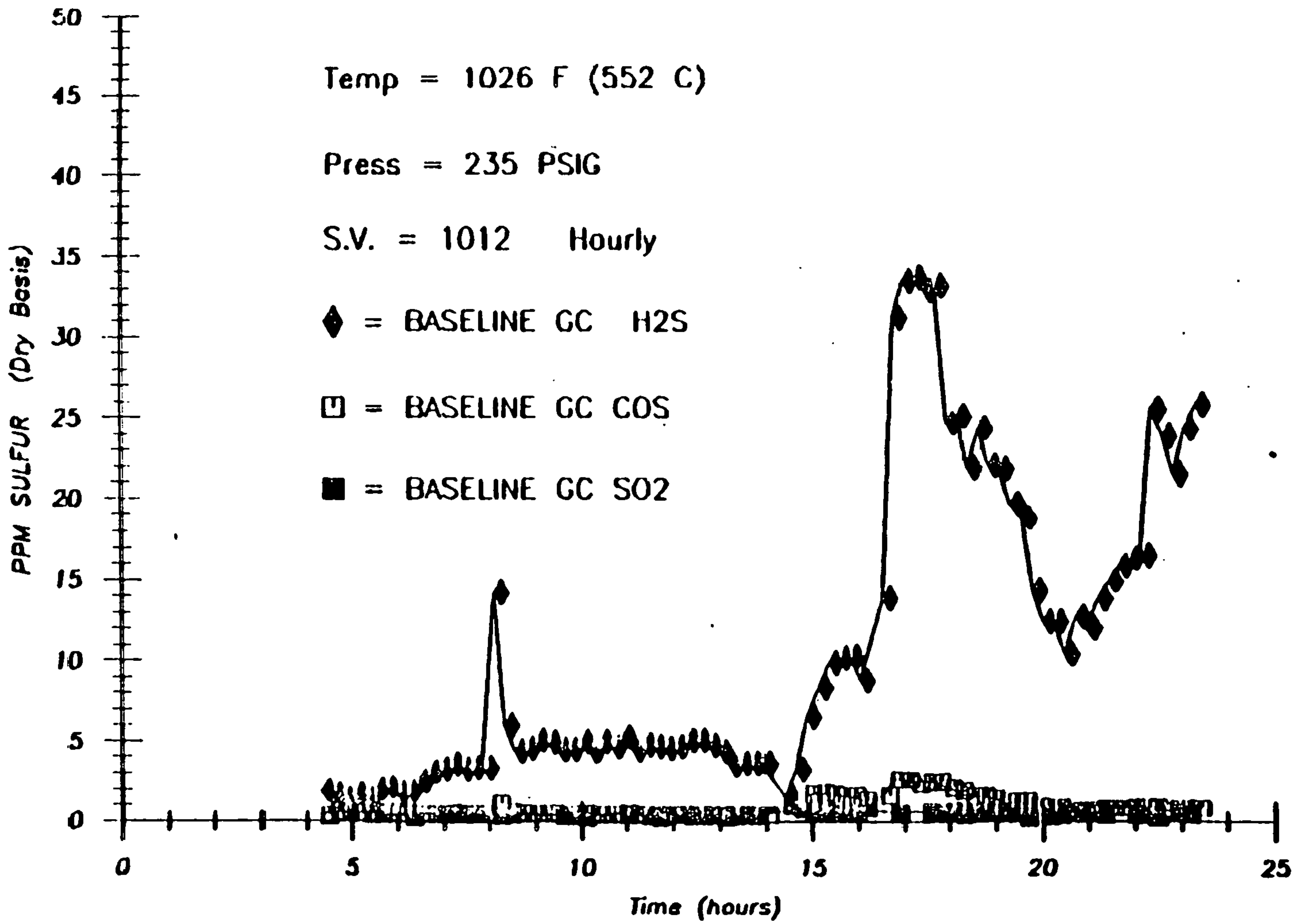


27-SEP-84 15:12:25

-625-

ZINC FERRITE SULFIDATION

SIDESTREAM TEST 019



28-SEP-84 13:46:07

FIGURE 54

SHIFT REACTION ON ZINC FERRITE SORBENT

SIDESTREAM TEST UNIT TEST 017

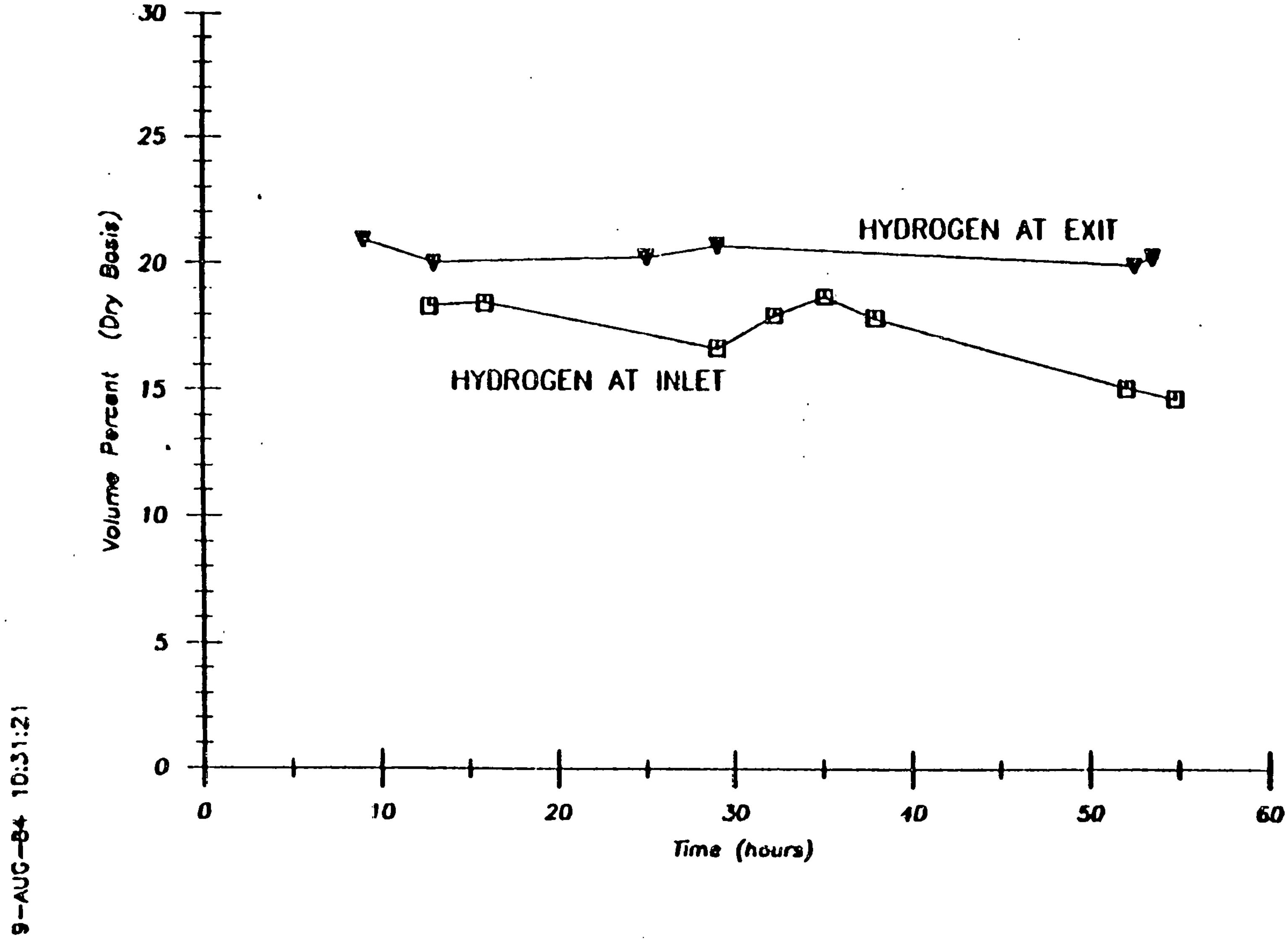


FIGURE 55

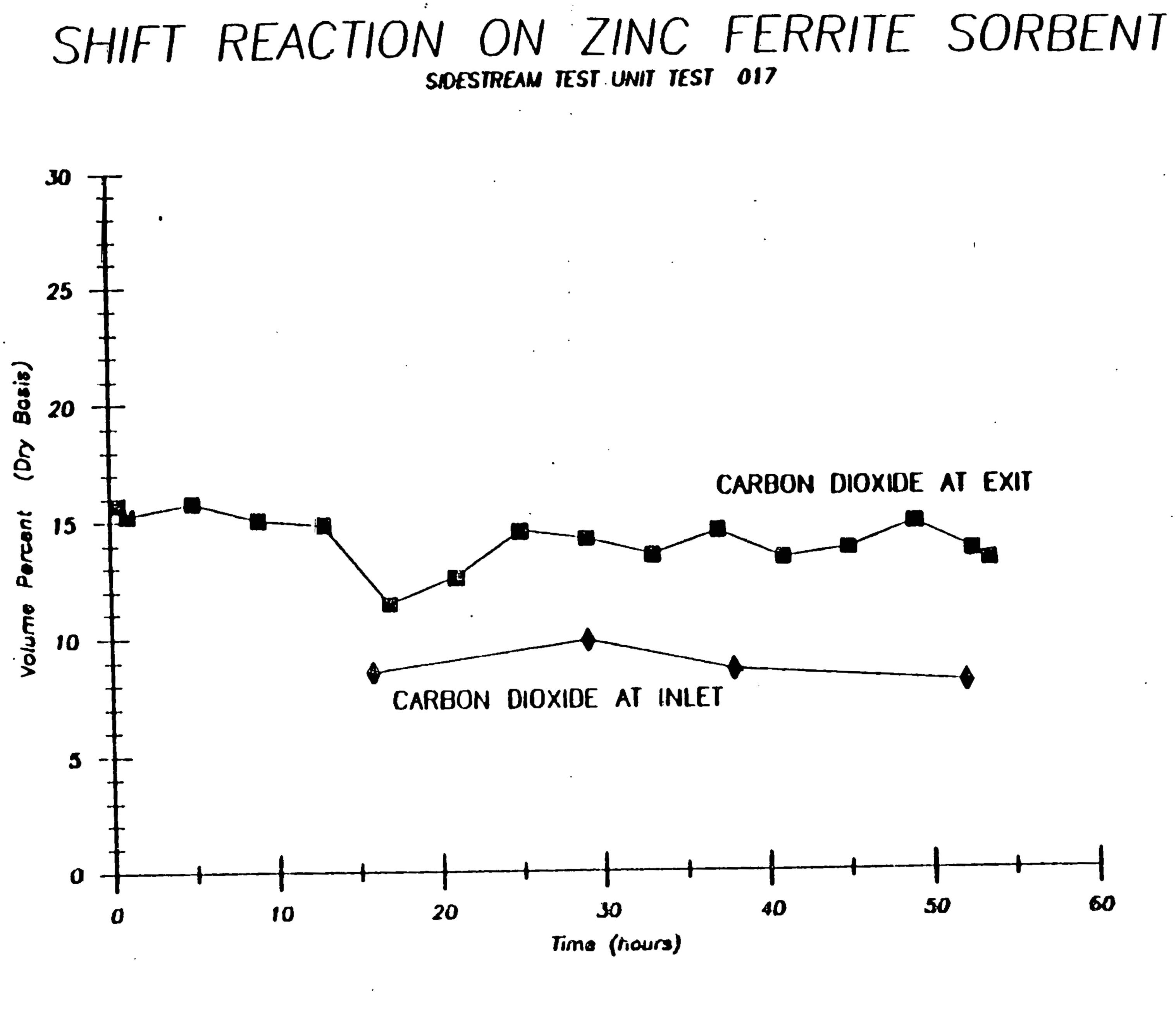

SHIFT REACTION ON ZINC FERRITE SORBENT
SIDESTREAM TEST UNIT TEST 017
Volume Percent (Dry Basis)
30
25
20
15
10
5
0
CARBON DIOXIDE AT EXIT
CARBON DIOXIDE AT INLET
0
10
20
30
40
50
60
Time (hours)
9-AUG-84 10:28:47

FIGURE 56

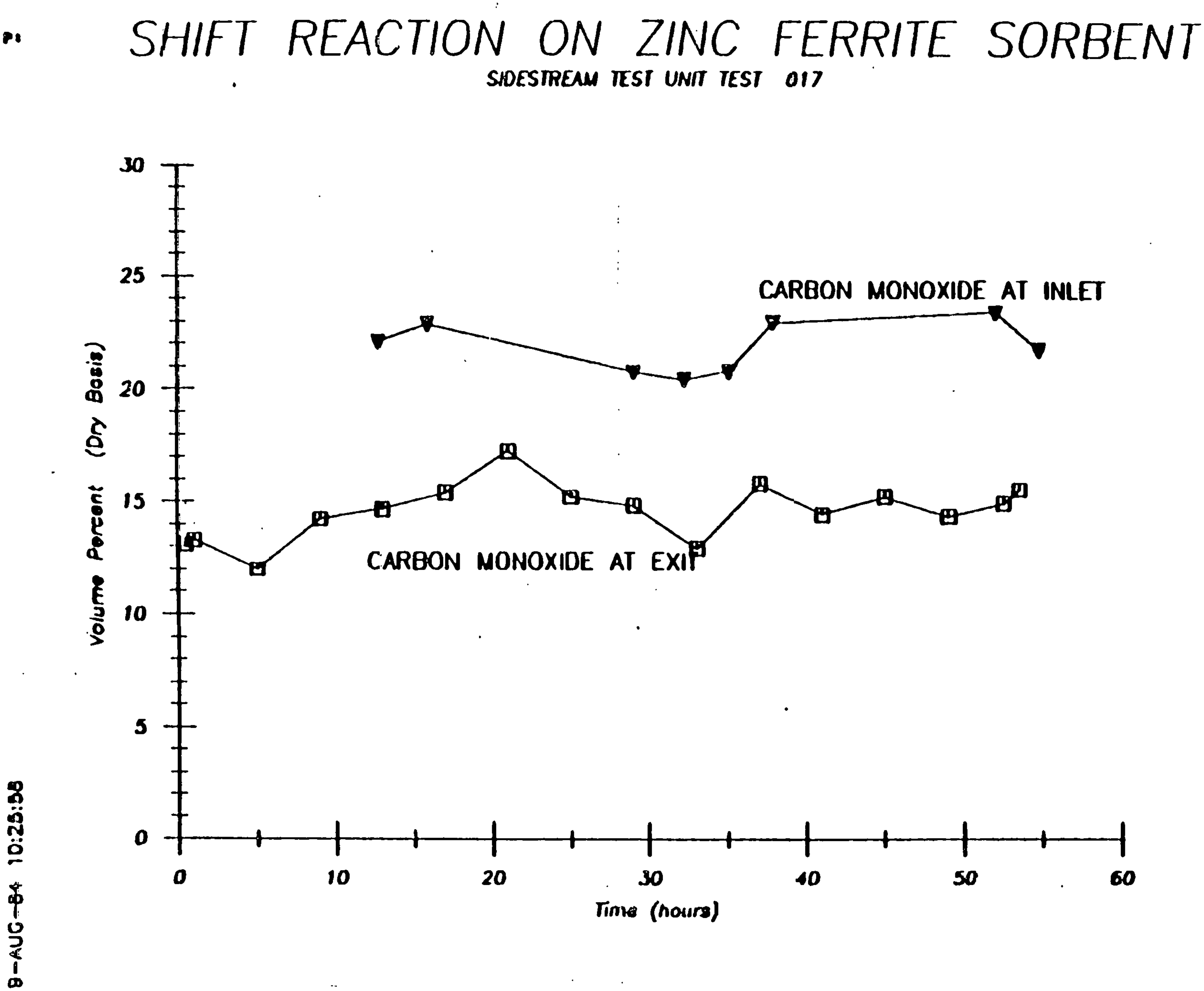

SHIFT REACTION ON ZINC FERRITE SORBENT
SIDESTREAM TEST UNIT TEST 017
CARBON MONOXIDE AT INLET
CARBON MONOXIDE AT EXIT
Volume Percent (Dry Basis)
Time (hours)
0
5
10
15
20
25
30
0
10
20
30
40
50
60
8-AUG-84 10:25:58

FIGURE 57

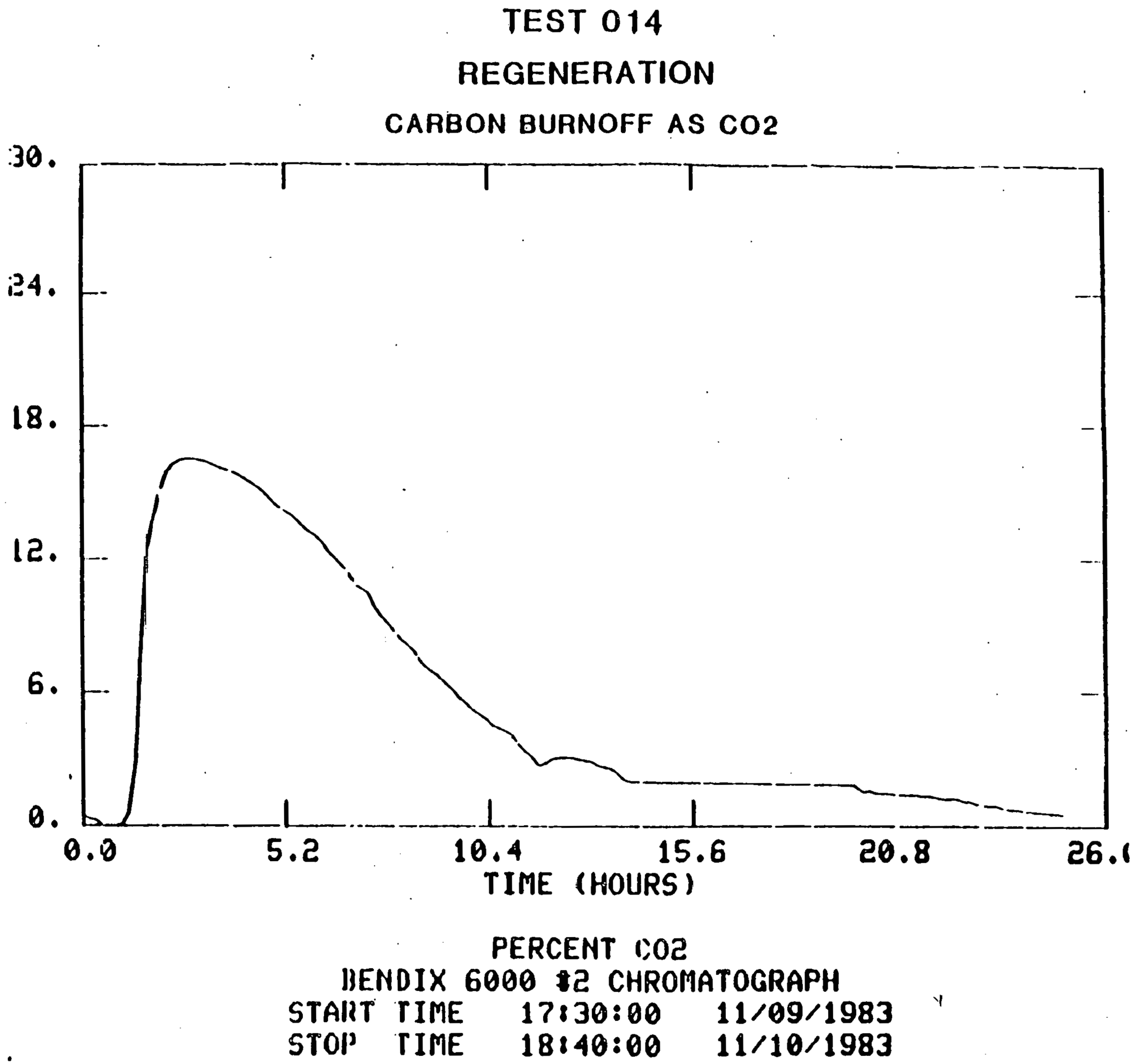
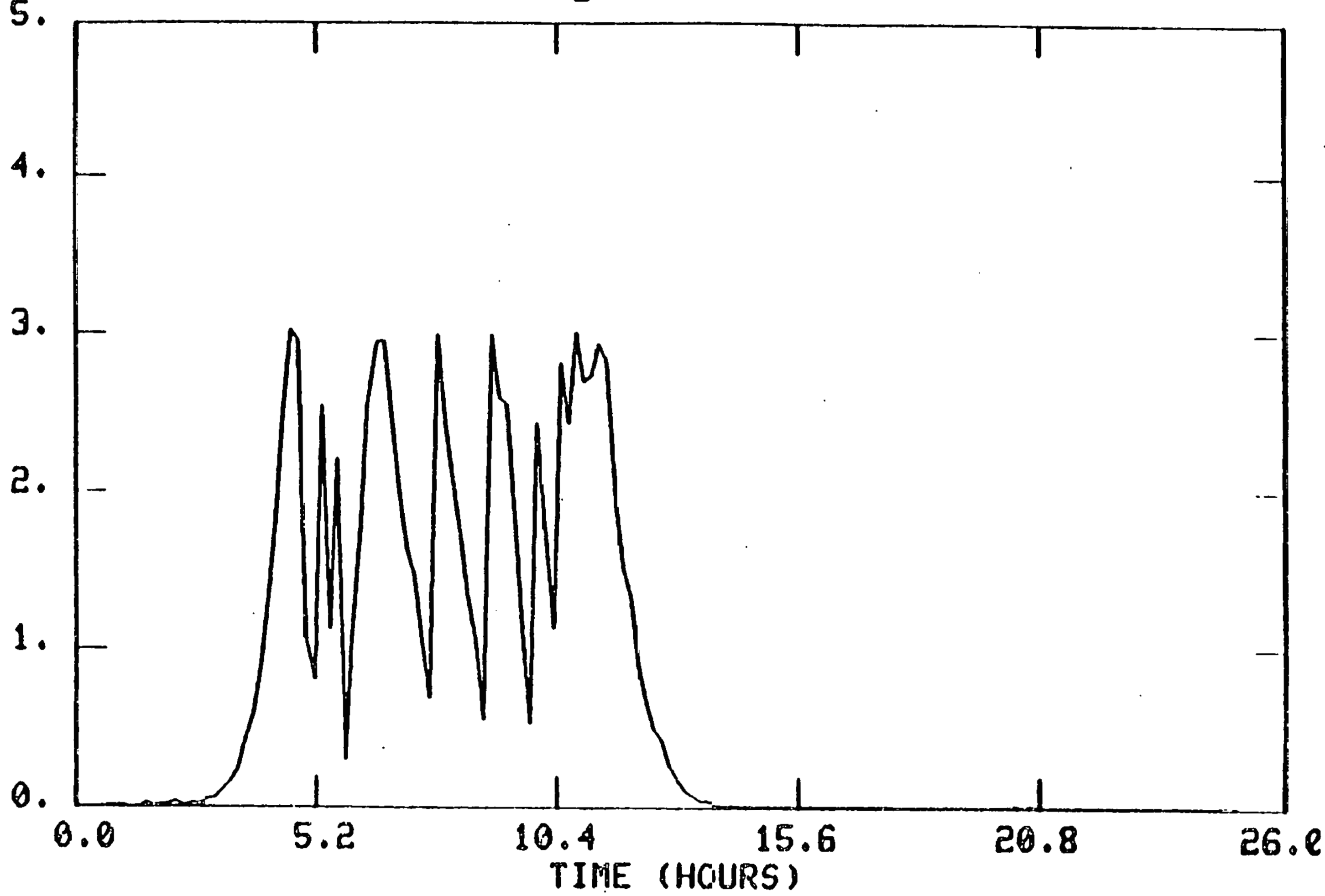

TEST 014
REGENERATION
CARBON BURNOFF AS CO2
30.
24.
18.
12.
6.
0.
0.0
5.2
10.4
15.6
20.8
26.0
TIME (HOURS)
PERCENT CO2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:30:00 11/09/1983
STOP TIME 18:40:00 11/10/1983

FIGURE 58

TEST 014

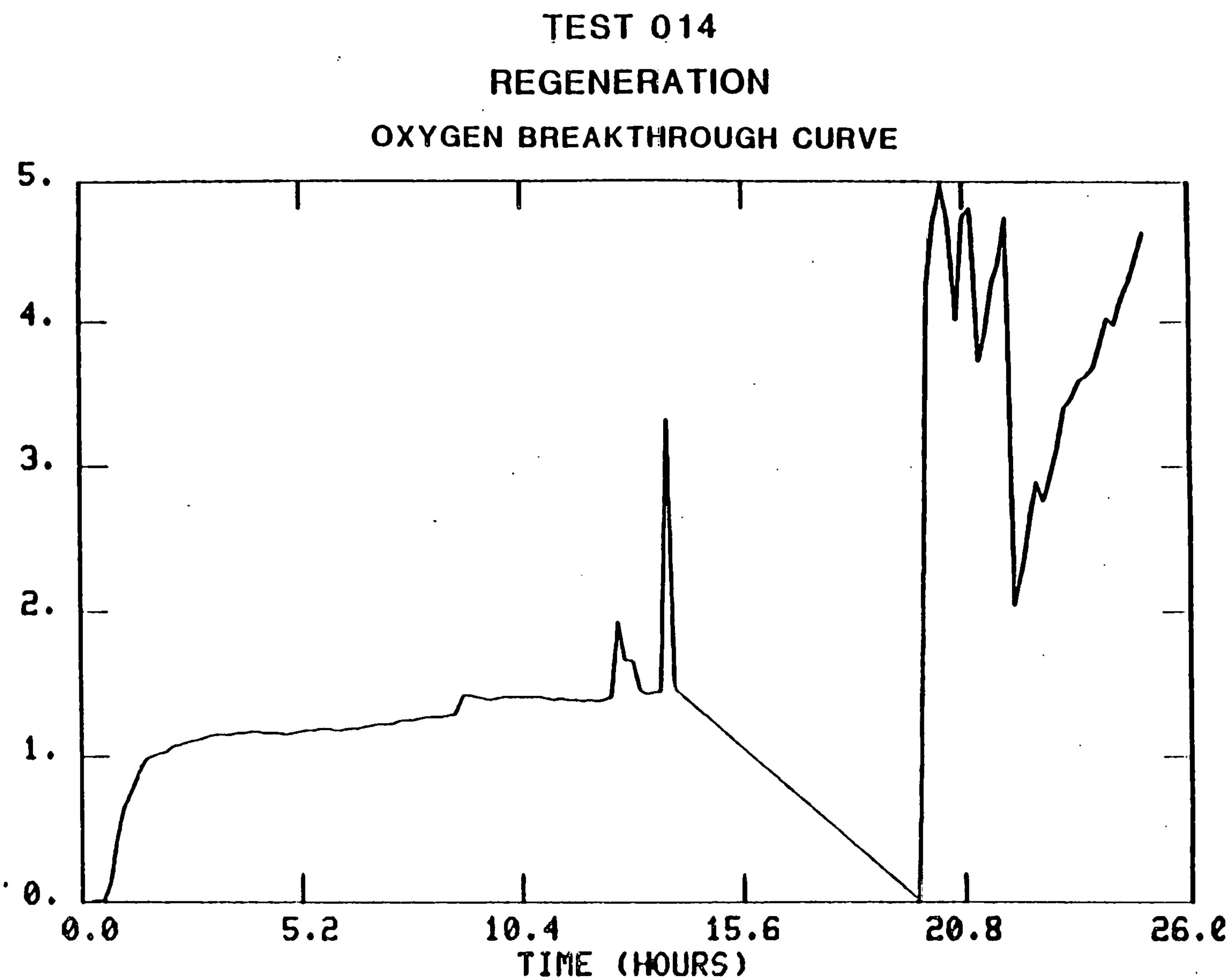
REGENERATION

H_2S EFFLUENT



PERCENT H2S
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:30:00 11/09/1983
STOP TIME 18:40:00 11/10/1983

FIGURE 59



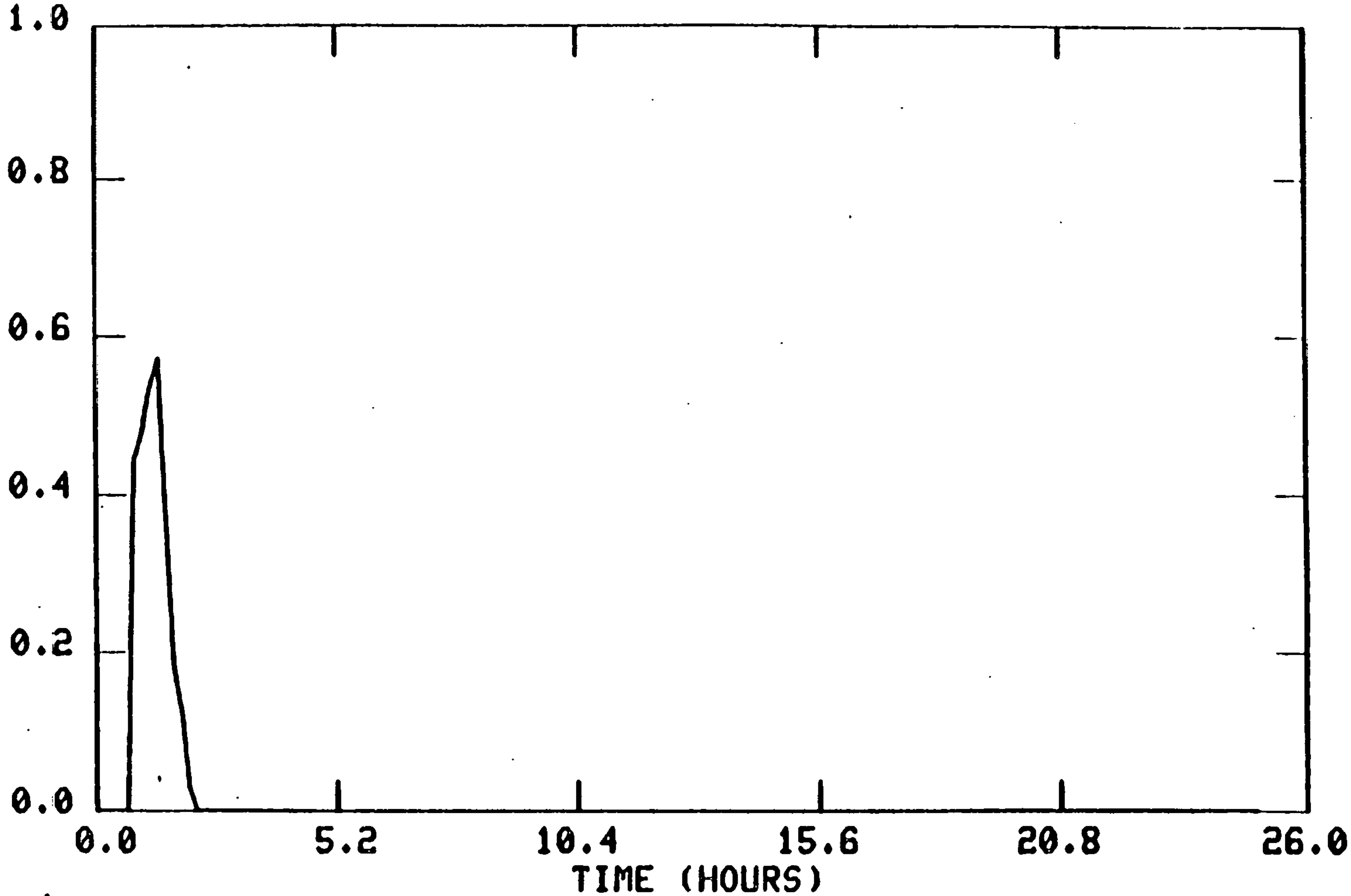
PERCENT O2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:30:00 11/09/1983
STOP TIME 18:40:00 11/10/1983

FIGURE 60

TEST 014

REGENERATION

CARBON BURNOFF AS CO



PERCENT CO
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:30:00 11/09/1983
STOP TIME 18:40:00 11/10/1983

FIGURE 61

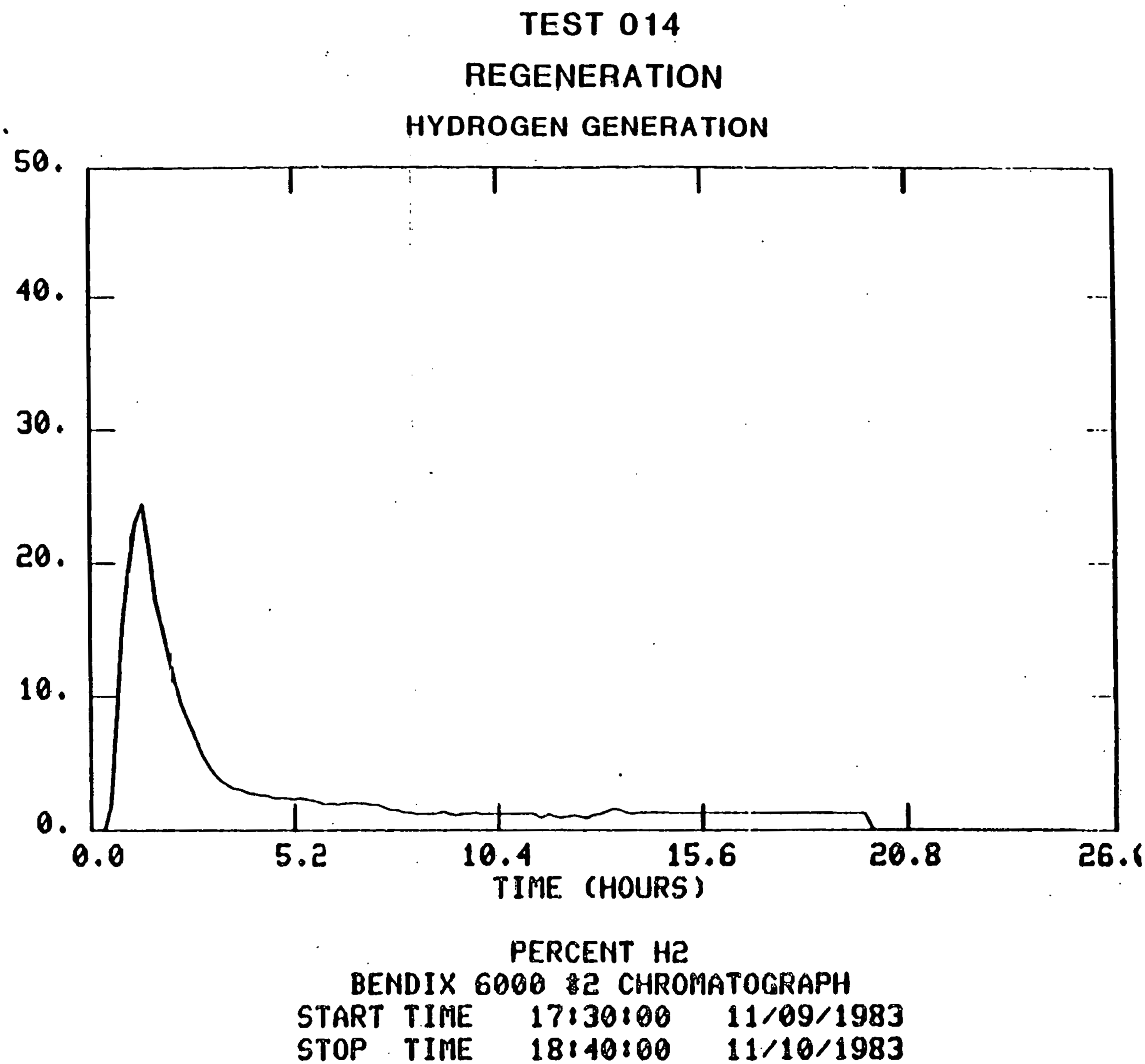
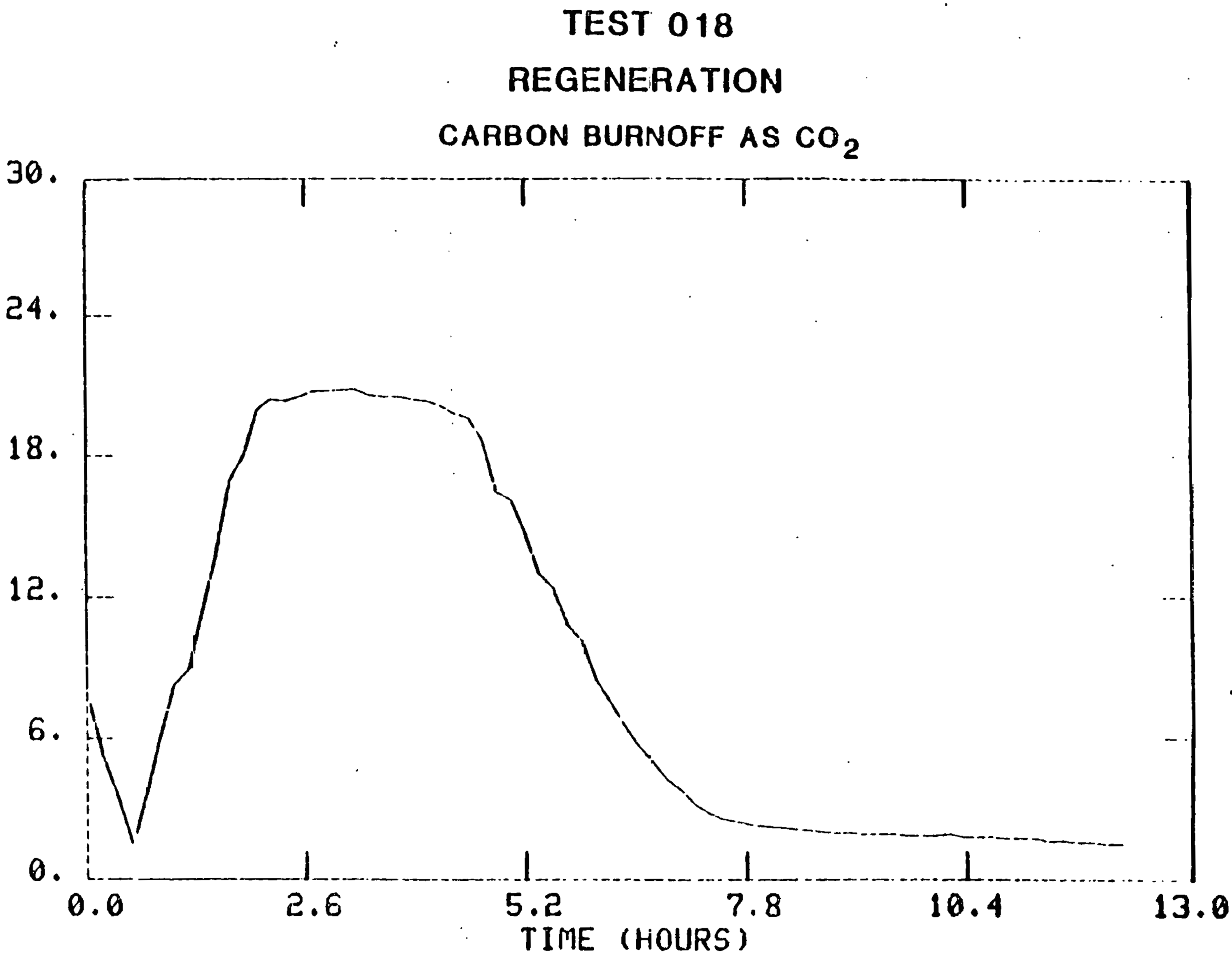
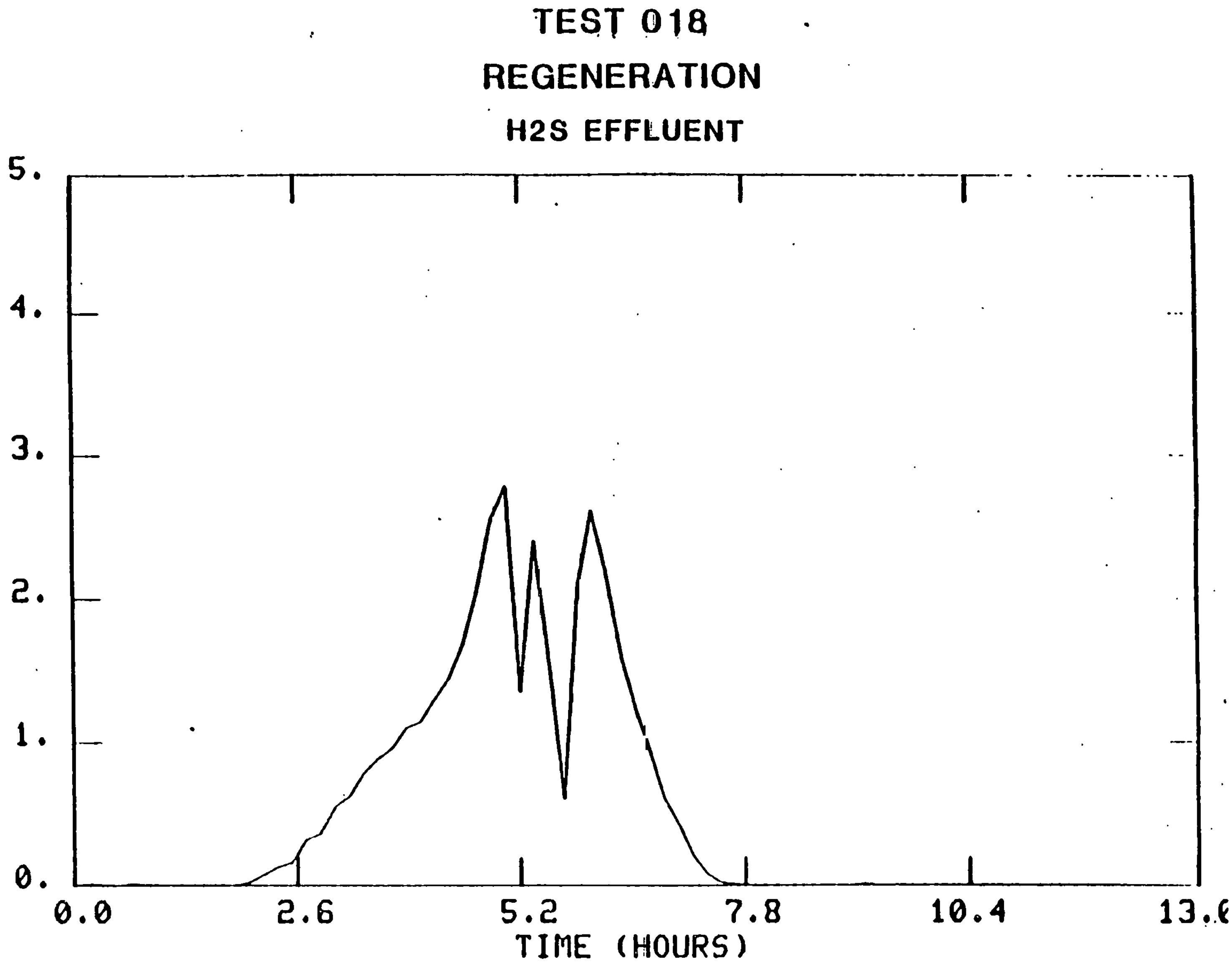

TEST 014
REGENERATION
HYDROGEN GENERATION
50.
40.
30.
20.
10.
0.
0.0
5.2
10.4
15.6
20.8
26.(
TIME (HOURS)
PERCENT H2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:30:00 11/09/1983
STOP TIME 18:40:00 11/10/1983

FIGURE 62



PERCENT CO2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:40:00 11/16/1983
STOP TIME 06:00:00 11/17/1983

FIGURE 63



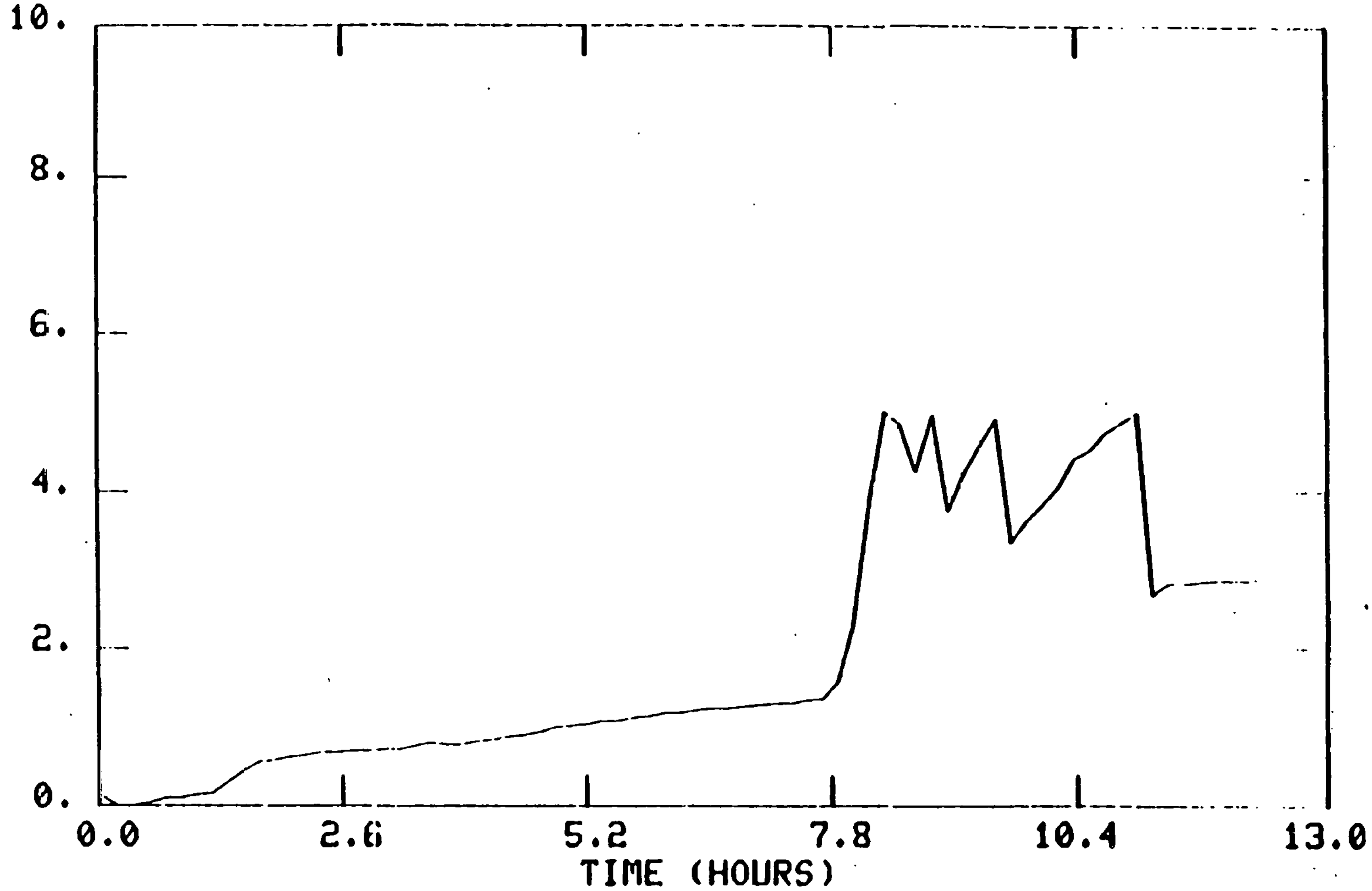
PERCENT H2S
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:40:00 11/16/1983
STOP TIME 06:00:00 11/17/1983

FIGURE 64

TEST 018

REGENERATION

OXYGEN BREAKTHROUGH CURVE



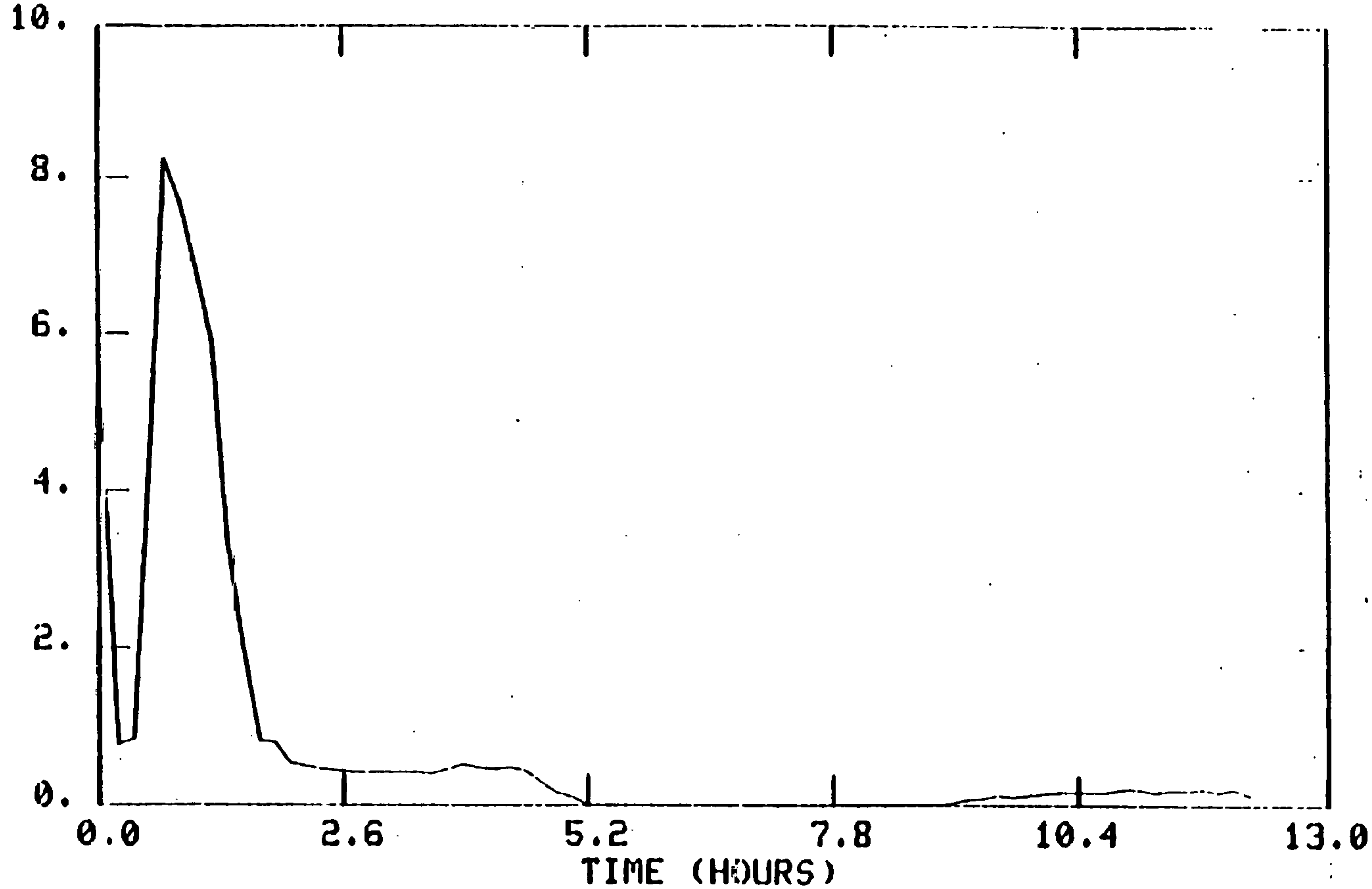
PERCENT O2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:40:00 11/16/1983
STOP TIME 06:00:00 11/17/1983

FIGURE 65

TEST 018

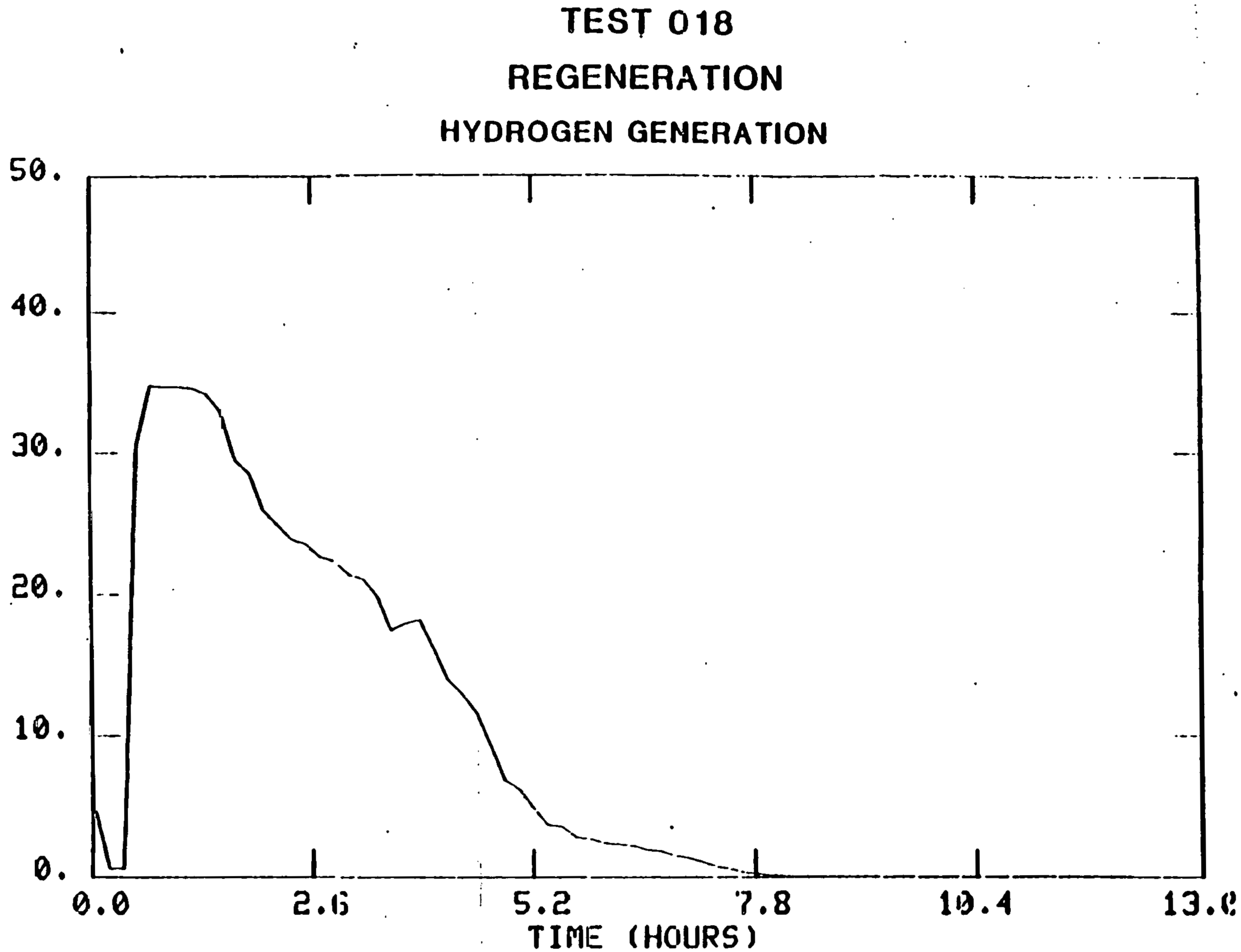
REGENERATION

CARBON BURNOFF AS CO



PERCENT CO
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:40:00 11/16/1983
STOP TIME 06:00:00 11/17/1983

FIGURE 66



PERCENT H2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 17:40:00 11/16/1983
STOP TIME 06:00:00 11/17/1983

FIGURE 67

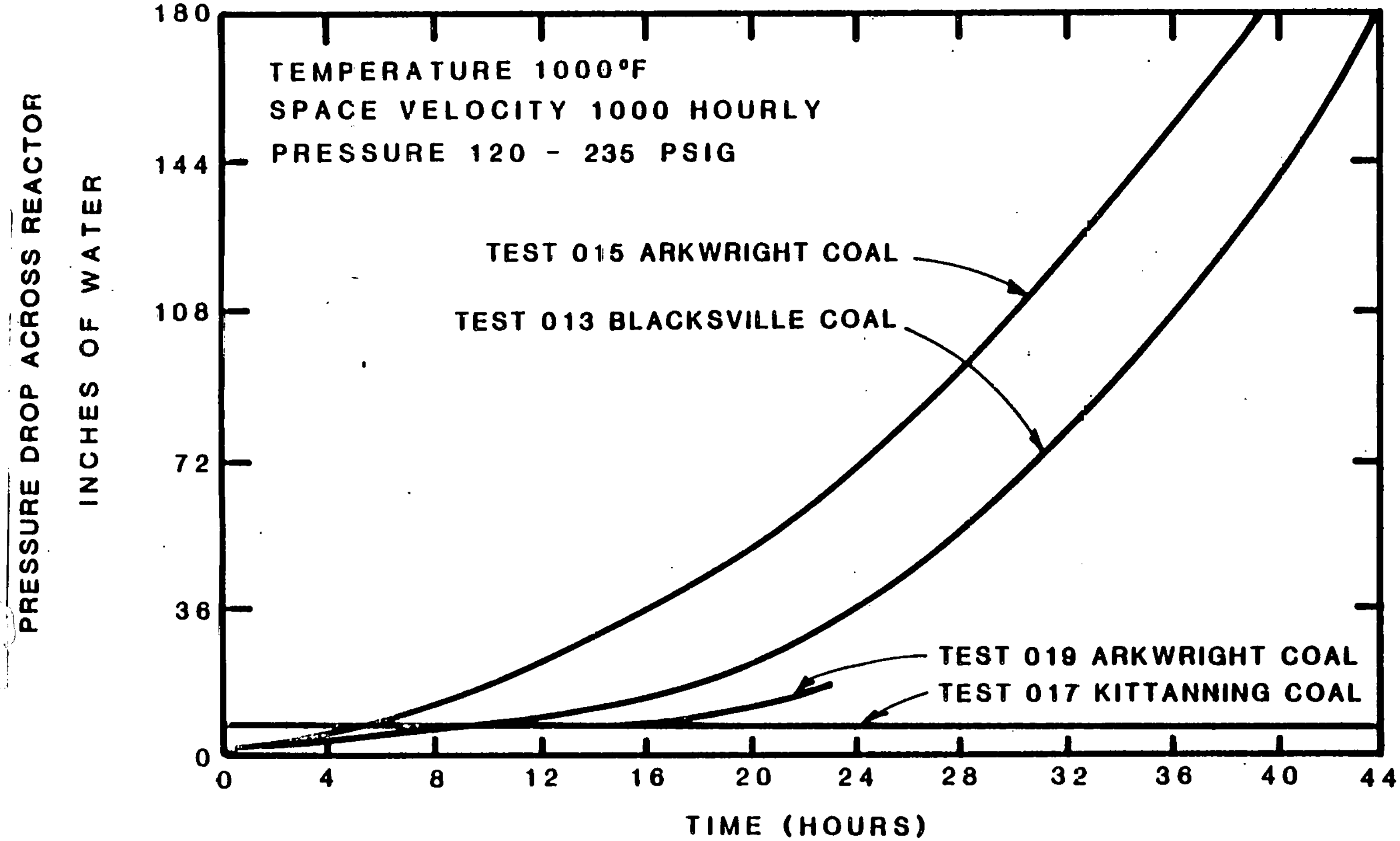

TEMPERATURE 1000°F
SPACE VELOCITY 1000 HOURLY
PRESSURE 120 - 235 PSIG
TEST 015 ARKWRIGHT COAL
TEST 013 BLACKSVILLE COAL
TEST 019 ARKWRIGHT COAL
TEST 017 KITTANNING COAL
PRESSURE DROP ACROSS REACTOR
INCHES OF WATER
TIME (HOURS)
180
144
108
72
36
0
0
4
8
12
16
20
24
28
32
36
40
44

TABLE 1

HOT GAS DESULFURIZATION

GRAB SAMPLE GAS ANALYSIS

TEST 013 to 019

	SAMPLE NO.	LOCATION	DATE TIME	H_2	O_2	N_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	i C_4	n C_4	ppm* H_2S	ppm COS	ppm CH_3SH	ppm CS_2	ppm Thio	ppm NH_3	ppm H_2O	ppm SO_2
SULFIDATION T013	HG-1	Exit	11/7/83 2110	21.63	1.33	46.86	2.67	10.74	0.19	16.00	ND	0.08	ND	ND	4.45	0.32	ND	0.15	3.10	ND	1755.8	ND
	HG-2	Exit	11/8/83 0110	20.66	1.82	47.04	2.73	11.34	0.20	15.51	0.09	0.10	ND	ND	0.41	ND	ND	ND	ND	ND	5406.8	ND
	HG-3	Exit	11/8/83 0510	22.63	0.78	44.89	2.39	11.33	0.24	16.44	0.10	0.10	ND	ND	10.24	2.38	ND	2.66	1.83	ND	1716.9	ND
	HG-4	Exit	11/8/83 0910	22.48	0.66	45.18	3.09	11.36	0.33	15.68	0.10	0.12	ND	ND	3.53	ND	ND	0.28	2.25	ND	1428.7	ND
	HG-5	Exit	11/8/83 1310	18.40	1.22	49.61	2.99	9,88	0.35	16.78	0.17	0.11	ND	ND	7.11	3.20	ND	3.11	1.07	ND	2068.2	ND
	HG-6	Exit	11/8/83 1710	20.13	0.66	48.65	2.94	8.98	0.41	17.17	0.49	0.06	ND	ND	5.49	1.20	ND	0.22	1.13	ND	1831.0	ND
	HG-7	Exit	11/8/83 2110	18.70	1.23	50.00	2.85	10.10	0.29	16.17	0.09	0.06	ND	ND	4.65	2.13	ND	0.55	1.85	ND	2682.5	ND
	HG-8	Exit					NO DATA											NO DATA				
	HG-9	Exit	11/9/83 0510	19.87	0.99	47.52	2.73	13.04	0.35	14.80	0.11	0.08	ND	ND	22.34	6.23	ND	4.85	1.87	ND	2593.0	ND
	HG-10	Exit	11/9/83 0910	20.35	0.65	46.47	2.92	13.54	0.37	14.79	0.14	0.26	ND	ND	12.11	4.35	ND	1.94	1.51	ND	2116.0	ND
	HG-11	Exit	11/9/83 1310	19.40	0.92	47.28	2.91	14.29	0.26	14.02	0.11	0.32	ND	ND	17.48	5.78	ND	13.77	2.53	ND	2364.4	883
REGENERATION T014	HG-12	Exit	11/9/83 1810	21.86	1.84	53.88	ND	0.60	ND	17.48	ND	ND	ND	ND	—	—	4.8	23.4	1.5	ND	2054.0	7.6
	HG-13	Exit	11/9/83 2010	2.75	2.21	73.17	ND	0.03	ND	12.38	ND	0.02	ND	ND						ND	4218.0	32.8
	HG-14	Exit	11/9/83 2210	0.88	1.91	73.97	ND	0.02	ND	12.19	ND	0.02	ND	ND	4.27%	444.7	ND	152.3	0.92	ND	3938.0	34.7
	HG-15	Exit	11/10/83 0010	1.04	1.30	79.97	ND	ND	ND	9.27	ND	ND	ND	ND	4.27%	444.7	ND	152.3	0.92	ND	4510	1.01%
	HG-16	Exit	11/10/83 0210	ND	1.87	82.12	ND	ND	ND	4.07	ND	2.73	ND	ND	2.75%	236.4	ND	57.2	ND	ND	4594	4.61%
	HG-17	Exit	11/10/83 0410	ND	2.05	83.15	ND	ND	ND	2.64	ND	3.24	ND	ND	2.51%	273.2	ND	50.7	ND	ND	5838	5.28%
	HG-18	Exit	11/10/83 0610	ND	1.31	84.12	ND	ND	ND	2.30	ND	4.33	ND	ND	2.43%	251.1	ND	63.9	ND	ND	5230	9.38'
	HG-19	Exit	11/10/83 0810	ND	1.99	85.00	ND	ND	ND	1.45	ND	3.87	ND	ND	1.65%	183	ND	60.3	ND	ND	4464	8.80'
	HG-20	Exit	11/10/83 1010	ND	1.30	85.11	ND	ND	ND	1.18	ND	5.75	ND	ND	9768	108	ND	67.9	ND	ND	3784	10.14*
	HG-21	Exit	11/10/83 1210																			
	HG-22	Exit																				
	HG-23	Exit	11/10/83 1510	ND	15.09	78.98	ND	ND	ND	1.06	ND	2.26	ND	ND	ND	17.6	ND	6.8	ND	ND	3024	5.09%
SULFIDATION T015	HG-24	Exit	11/10/83 2000	14.75	3.50	50.59	2.51	12.29	0.29	15.30	0.14	0.14	ND	ND	13.4	2.7	ND	7.2	ND	ND	2266	ND
	HG-25	Exit	11/10/83 2115	16.29	3.12	51.86	2.78	9.32	0.26	15.10	0.16	0.11	ND	ND	7.9	1.1	ND	4.3	14.9	ND	1618	ND
	HG-26	Exit	11/11/83 0130	22.68	0.96	45.99	2.58	11.60	0.26	15.19	0.12	0.12	ND	ND	2.7	1.5	ND	2.8	3.0	ND	1558.0	12.0
	HG-27	Exit	11/11/83 0530	23.77	0.70	42.82	2.74	14.53	0.30	14.39	0.10	0.15	ND	ND	2.6	0.3	ND	2.7	10.2	ND	1289.2	ND
	HG-28	Exit	11/11/83 0930	16.18	3.39	50.72	2.23	11.92	0.25	14.58	0.11	0.11	ND	ND	2.5	0.8	ND	1.6	4.2	ND	1314.7	ND
	HG-29	Exit	11/11/83 1330	18.86	0.81	48.09	2.69	14.44	0.32	14.04	0.13	0.14	ND	ND	1.6	1.8	ND	1.0	5.2	ND	1302.6	451
	HG-30	Exit																				
	HG-31	Exit	11/11/83 2130	13.88	6.10	55.61	1.80	11.55	0.25	10.12	0.09	0.10	ND	ND	13.6	3.3	ND	2.3	10.7	ND	1429.0	ND
	HG-32	Exit	11/12/83 0130	10.45	5.42	56.09	2.47	12.32	0.29	12.22	0.12	0.12	ND	ND	8.7	0.2	ND	ND	10.2	ND	1570.9	ND
	HG-33	Exit	11/12/83 0530	16.47	3.11	50.11	2.98	15.24	0.35	11.08	0.14	0.13	ND	ND	13.9	1.7	ND	ND	11.6	ND	1561.2	ND
	HG-34	Exit	11/12/83 0930	17.48	1.87	48.66	2.58	15.11	0.31	13.11	0.13	0.15	ND	ND	14.1	0.9	ND	11.9	16.[illegible]	ND	2245.6	101.8
REGENERATION T016	HG-35	Exit	11/12/83 1400	17.95	3.47	59.33	0.09	0.71	ND	17.94	ND	ND	ND	ND	1.29%	535.7	11.2	46.0	2.9	ND	3644	21.9
	HG-36	Exit	11/12/83 1600	4.82	3.43	67.91	ND	ND	ND	14.79	ND	ND	ND	ND	4.98%	481.1	9.5	116.5	2.0		4072.0	30.0
	HG-37	Exit	11/12/83 1800	ND	2.28	68.10	ND	0.39	ND	13.86	ND	ND	ND	ND	4.56%	504.7	2.4	122.8	1.3	-	4018.0	20.7
	HG-38	Exit	11/12/83 2000	2.15	2.23	77.80	ND	ND	ND	11.03	ND	ND	ND	ND	3.54%	128.9	ND	136.4	ND	-	3936.0	4294
	HG-39	Exit	11/12/83 2200	ND	1.21	81.87	ND	ND	ND	5.08	ND	ND	ND	ND	1.72%	336.3	ND	191.4	ND	-	4572	9.07%
	HG-40	Exit	11/13/83 0000	ND	2.33	81.43	ND	ND	ND	2.50	ND	ND	ND	ND	9835.0	164.2	ND	199.9	ND	-	4520.	12.3%
	HG-41	Exit	11/13/83 0200	ND	8.07	80.17	ND	ND	ND	1.29	ND	ND	ND	ND	1.32	39.5	ND	200.9	ND	-	3066	8.1%
	HG-42	Exit	11/13/83 0400	ND	17.2	76.52	ND	ND	ND	0.76	ND	ND	ND	ND	ND	2.9	ND	18.8	ND	-	3406	1.32%
SULFIDATION T017	HG-43	Exit	11/13/83 1755	17.23	1.74	48.18	2.99	13.10	0.41	15.74	0.09	0.13	ND	ND	ND	3.52	ND	16.2	ND	-	1652.8	25.0
	HG-44	Exit	11/13/83 1825	15.95	2.66	48.84	2.89	13.29	0.38	15.26	0.08	0.15	ND	ND	5.2	4.5	ND	2.9	0.6	-	1764.7	ND
	HG-45	Exit	11/13/83 2225	17.84	1.57	49.07	2.67	11.99	0.38	15.77	0.09	0.13	ND	ND	21.7	4.3	ND	4.9	0.7	-	1832.4	429.2
	HG-46	Exit	11/14/83 0225	20.93	0.37	44.66	3.07	14.21	0.41	15.03	0.09	0.15	ND	ND	7.2	3.8	ND	ND	1.4	-	1425.9	72.9
	HG-47	Exit	11/14/83 0625	20.01	1.26	45.27	2.85	14.65	0.39	14.81	0.10	0.15	ND	ND	7.3	2.7	ND	ND	1.8	-	1491.2	ND
	HG-48	Exit	11/14/83 1025	12.48	4.01	52.93	2.68	15.38	0.37	11.42	0.10	0.13	ND	ND	0.9	0.9	ND	0.9	1.0	ND	1490	ND
	HG-49	Exit	11/14/83 2225	17.45	1.48	47.17	3.01	17.21	0.40	12.54	0.09	0.15	ND	ND	0.5	0.9	ND	0.5	1.2	ND	1228	ND
	HG-50	Exit	11/15/83 0225	20.22	1.29	44.46	3.2	15.18	0.42	14.48	0.06	0.19	ND	ND	ND	ND	ND	0.2	0.9	ND	1268.4	ND
	HG-51	Exit	11/15/83 0625	20.67	0.95	45.42	2.89	14.8	0.43	14.15	0.06	0.13	ND	ND	ND					ND	959	ND

Table 1 (Continued)

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS
TEST 013 to 019

	SAMPLE NO.	LOCATION	DATE TIME	N_2	O_2	H_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	i C_4	n C_4	ppm* H_2S	ppm COS	ppm CH_3SH	ppm CS_2	ppm Thio	ppm NH_3	ppm H_2O	ppm SO_2
SULFIDATION T017	HG-52	Exit	11/15/83 1025	15.9	5.67	48.56	2.49	12.92	0.32	13.46	0.05	0.13	ND	ND	0.3	ND	ND	ND	0.2	ND	616.5	ND
	HG-53	Exit	11/15/83 1425	16.73	1.35	47.47	3.05	15.78	0.42	14.46	0.09	0.16	ND	ND	3.5	ND	ND	0.1	0.8	ND	1600.6	ND
	HG-54	Exit	11/15/83 1825	17.79	4.51	46.07	2.79	14.42	0.40	13.32	0.05	0.15	ND	ND	3.5					ND	1644.1	ND
	HG-55	Exit	11/15/83 2225	18.01	3.01	45.81	3.22	15.20	0.41	13.71	ND	0.13	ND	ND	3.9	1.9	ND	ND	0.9	ND	1531	ND
	HG-56	Exit	11/16/83 0225	17.52	1.05	47.09	4.05	14.35	0.49	14.81	0.03	0.12	ND	ND	13.1						1290.0	ND
	HG-57	Exit	11/16/83 1300	19.97	1.68	45.15	3.60	14.92	0.40	13.62	0.05	0.11	ND	ND	6.7	3.5	ND	3.7	2.1	ND	1457	21.6
	HG-58	Exit	11/16/83 ?	20.23	1.45	44.96	3.49	15.53	0.38	13.23	0.07	0.11	ND	ND	13.1	2.4	ND	0.2	1.8	ND	1529	ND
REGENERATION T018	HG-59	Exit	11/16/83 1940	20.08	2.72	55.49	ND	ND	ND	21.21	ND	ND	ND	ND	247.9							2.1
	HG-60	Exit	11/16/83 2140	10.47	1.39	62.45	ND	ND	ND	19.66	ND	ND	ND	ND	3.73%	ND	ND	7.5	0.4	ND	3568	ND
	HG-61	Exit	11/16/83 2340	0.95	1.71	85.58	ND	ND	ND	4.37	ND	ND	ND	ND	37.9	64.0	ND	92.2	ND	ND	3784	4.96%
	HG-62	Exit	11/17/83 0140	ND	6.28	81.44	ND	ND	ND	1.75	ND	ND	ND	ND	3.3	20.8	8.0	240.0	ND	ND	2854	16.32%
	HG-63	Exit	11/17/83 0340	ND	15.8	78.0	ND	ND	ND	1.77	ND	ND	ND	ND	ND	13.0	5.2	47.1	ND	ND	2852	8.72%
	HG-64	Exit	11/17/83 0540	ND	21.55	75.78	ND	ND	ND	1.35	ND	ND	ND	ND	0.68	ND	ND	21.0	ND	ND	4008	1.792
SULFIDATION T019	HG-65	Exit	11/17/83 0745	14.58	2.19	51.94	2.72	12.43	0.36	15.13	0.06	0.09	ND	ND	7.7	6.7	1.1	2.7	4.1	ND	3632	132.5
	HG-66	Exit	11/17/83 0845	14.79	2.40	50.83	3.63	12.03	0.41	15.22	0.08	0.10	ND	ND	ND	4.9	ND	5.8	3.0	ND	2820	60.9
	HG-67	Exit	11/17/83 0940	17.73	1.36	49.61	3.08	11.60	0.38	15.60	0.06	0.08	ND	ND	3.5	ND	ND	8.6	5.7	ND	2232	4.9
	HG-68	Exit	11/17/83 1045	17.27	1.76	43.68	3.47	12.47	0.43	15.25	0.06	0.10	ND	ND	0.2	ND	ND	ND	6.7	ND	2174	ND
	HG-69	Exit	11/17/83 2200	17.68	1.43	48.49	3.00	12.82	0.35	15.57	0.07	0.10	ND	ND	5.4	ND	ND	2.5	6.1	ND	2238	ND
	HG-70	Exit	11/18/83 0200	18.43	0.99	47.89	3.21	13.35	0.35	15.15	0.05	0.07	ND	ND	ND	1.1	ND	1.1	ND	ND	2158	ND
	HG-71	Exit	11/18/83 0600	17.73	0.77	47.39	3.51	15.85	0.41	13.17	0.07	0.10	ND	ND	1.8	1.0	ND	0.4	0.6	ND	1940	ND
	HG-72	Exit	11/18/83 1810	ND	15.04	71.01	1.16	6.39	0.15	5.70	ND	0.05	ND	ND	8.3						2114	ND
	HG-73	Exit	11/18/83 2210	ND	15.91	72.01	0.90	5.25	0.13	5.3	ND	ND	ND	ND	2.6	2.9	ND	0.4	0.5	ND	2148	ND

ND = Not Detectable

* = H_2S Detectable Limit 0.2 ppm

NOTE: All analysis other than Sulfur, Ammonia, and Water is in units of % by volume on a dry gas basis. The remaining items are as indicated in units of ppm by volume, except where noted in percent.

TABLE 2

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT INLET
GASIFIER RUN 103
MAJOR COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2	O_2	N_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	iC_4	nC_4
						VOLUME %							
SULFI-	FN 5	11/8/83 06:30-07:00	15.517	0.644	49.904	2.808	19.588	0.357	10.522	0.129	ND	ND	ND
DATION	FN 9	11/8/83 09:30-10:15	17.238	0.645	48.334	2.802	19.305	0.190	10.314	0.050	0.121	ND	ND
T013	FN 13	11/8/83 13:35-14:09	16.951	0.742	49.381	3.139	18.723	0.315	9.940	0.178	0.133	ND	ND
	FN 17	11/9/83 06:10-06:43	16.951	0.592	49.058	2.771	19.140	0.272	10.494	0.105	0.117	ND	ND
	FN 21	11/9/83 09:23-09:52	16.791	0.573	50.031	2.976	18.386	0.324	10.164	0.139	0.115	ND	ND
	FN 25	11/9/83 12:20-12:49	16.622	0.943	49.677	2.853	17.747	0.297	11.124	0.120	0.118	ND	ND
SULFI-	FN 59	11/11/83 06:15-07:16	17.54	2.64	47.38	2.90	19.91	0.29	8.06	0.13	0.16	ND	ND
DATION T015	FN 72	11/11/83 15:20-15:40	17.05	0.79	50.18	3.09	19.82	0.31	7.92	0.18	0.15	ND	ND
SULFI-	FN 100	11/14/83 06:10-06:55	18.30	0.56	46.29	2.82	22.06	0.33	8.74	0.17	0.14	ND	ND
DATION	FN 105	11/14/83 09:20-09:55	18.41	0.63	45.64	2.82	22.84	0.35	8.50	0.17	0.15	ND	ND
T017	FN 110	11/15/83 06:25-07:10	16.63	0.67	47.68	2.94	20.71	0.33	9.85	0.27	0.42	ND	ND
	FN 114	11/15/83 09:40-10:15	17.92	0.64	45.89	2.78	20.39	1.98	9.30	0.17	0.44	ND	ND
	FN 120	11/15/83 12:30-13:05	18.66	0.61	46.28	2.74	20.75	0.07	9.75	0.15	0.48	ND	ND
	FN 125	11/15/83 15:20-15:55	17.18	1.43	44.95	3.18	22.90	0.36	8.57	0.15	0.15	ND	ND
	FN 130	11/16/83 12:30-13:00	15.11	0.64	48.00	3.46	23.36	0.34	8.01	0.20	0.37	ND	ND
	FN 135	11/16/83 15:15-15:45	14.69	0.64	49.39	3.68	21.70	0.37	8.51	0.16	0.36	ND	ND
	FN 140	11/17/83 06:20	15.30	0.73	49.27	3.66	21.10	0.33	8.82	0.13	0.13	ND	ND
SULFI-	FN 145	11/17/83 09:25-10:00	15.83	0.71	49.77	3.49	20.55	0.35	8.54	0.13	0.13	ND	ND
DATION T019	FN 150	11/18/83 15:20-16:35	15.94	0.93	47.81	3.42	22.55	0.36	8.32	0.13	0.15	ND	ND

NOTES:

1. ND = Not Detectable.

TABLE 3

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT INLET
GASIFIER RUN 103
TRACE COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2S	COS	CH_3SH	CS_2 PPM	Thio	NH_3
SULFI-	FN 5	11/8/83 06:30-07:00	4528	358.9	7.9	13.1	2.9	ND
DATION	FN 9	11/8/83 09:30-10:15	4339	350.0	ND	9.2	ND	ND
T013	FN 13	11/8/83 13:35-14:09	4217	410.6	2.0	15.2	3.2	ND
	FN 17	11/9/83 06:10-06:43	4628	390.2	ND	12.7	50.6	ND
	FN 21	11/9/83 09:23-09:52	4511	413.3	ND	11.0	2.0	ND
	FN 25	11/9/83 12:20-12:49	4831	398	ND	13.6	ND	ND
SULFI-	FN 59	11/11/83 06:15-07:10	3951	259	6.1	11.0	19.0	ND
DATION T015	FN 72	11/11/83 15:20-15:40	1960	254.0	ND	10.8	14.7	ND
SULFI-	FN 100	11/14/83 06:10-06:55	2084	138.9	ND	3.0	4.7	ND
DATION	FN 105	11/14/83 09:20-09:55	2133	137.7	ND	3.6	22.7	ND
T017	FN 110	11/15/83 06:25-07:10	2161	135.7	ND	2.2	11.0	ND
	FN 114	11/15/83 09:40-10:15	2036	122.2	ND	ND	15.6	ND
	FN 120	11/15/83 12:30-13:05	1754	135.8	ND	2.4	1.9	ND
	FN 125	11/15/83 15:20-15:55	1787	124.3	ND	1.1	18.2	ND
	FN 130	11/16/83 12:30-13:00	3864	325.6	7.0	10.0	10.4	ND
	FN 135	11/16/83 15:15-15:45	3877	391.9	2.5	11.5	16.3	ND
SULFI-	FN 145	11/17/83 09:25-10:00	4898	621.5	1.7	18.0	6.9	ND
DATION 019	FN 150	11/18/83 15:20-16:35	4311	459.8	2.3	18.0	8.3	ND

NOTES:

1. ND = Not Detectable.

TABLE 4

HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT TEST 013 AVERAGE GC READINGS

DURATION

FROM	TO
17:10: 0 11/ 7/1983	15: 0: 0 11/ 9/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
174	21.014	15.608	1.168	-0.001	0.677	46.303	2.943	12.331	100.043	24.76	1.107	0.856	158.74	143.52

GASIFIER CYCLONE EXIT

BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
251	16.251	10.522	0.252	0.598	0.659	48.583	2.734	19.380	99.179	24.96	1.115	0.863	153.33	141.58

HGD INLET

BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
153	16.086	10.417	0.297	0.612	0.768	47.875	3.136	18.914	98.104	24.67	1.102	0.853	154.23	142.29

TABLE 5

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 014
AVERAGE GC READINGS

DURATION

FROM		TO	
17:30: 0	11/ 9/1983	18:40: 0	11/10/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
99	3.241	7.430	0.142	1.014	1.677	75.554	-0.014	-0.078	88.968	25.40	1.135	0.878	19.25	16.92

TABLE 6

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 015
AVERAGE GC READINGS

DURATION

FROM	TO
19: 5: 0 11/10/1983	11:10: 0 11/12/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
237	20.534	14.139	0.285	-0.001	0.572	43.239	2.735	13.687	95.190	23.29	1.041	0.805	143.62	130.19

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
238	16.309	9.786	0.286	0.559	0.625	45.650	3.016	20.574	95.304	23.71	1.059	0.819	158.54	146.66

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
238	16.542	9.020	0.327	0.571	0.752	46.621	3.268	20.995	98.096	24.30	1.096	0.840	164.02	151.70

TABLE 7

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 016
AVERAGE GC READINGS

DURATION

FROM		TO	
12:15: 0	11/12/1983	8:30: 0	11/13/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
83	14.375	14.910	0.096	0.049	0.867	81.915	0.345	2.028	94.585	25.14	1.124	0.870	58.78	51.08

TABLE 8

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 017A
AVERAGE GC READINGS

DURATION

FROM	TO
17:25: 0 11/13/1983	12: 0: 0 11/14/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
24	19.782	12.863	0.294	0.086	0.769	45.686	2.829	15.728	98.036	24.08	1.077	0.833	149.41	136.21

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
108	16.488	8.938	0.270	0.104	0.593	46.432	2.949	21.659	97.434	24.12	1.078	0.834	158.71	147.06

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
112	17.752	9.174	0.382	0.205	0.753	47.551	3.406	22.826	102.049	25.08	1.121	0.867	173.87	160.90

TABLE 9

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 017B1
AVERAGE GC READINGS

DURATION

FROM		TO	
20: 0: 0	11/14/1983	20: 0: 0	11/15/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
143	20.787	13.412	0.358	-0.003	0.551	43.949	3.069	15.325	97.517	23.72	1.061	0.820	154.61	140.61

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
141	16.696	8.721	0.279	0.133	0.592	45.736	3.035	22.184	97.376	24.01	1.073	0.830	162.30	150.43

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
140	16.986	8.723	0.383	0.163	0.730	44.910	3.313	21.758	96.966	23.79	1.063	0.822	166.74	154.27

TABLE 10

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 017B2
AVERAGE GC READINGS

DURATION

FROM	TO
20: 0: 0 11/15/1983	4: 0: 0 11/16/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
47	20.358	14.015	0.420	-0.002	0.565	43.246	3.448	14.653	96.703	23.66	1.053	0.818	155.78	141.52

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
47	16.364	8.531	0.301	0.608	0.602	45.875	3.239	22.284	97.805	24.19	1.081	0.836	167.09	154.90

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
48	16.456	8.830	0.417	0.391	0.734	44.644	3.513	21.862	96.848	23.91	1.068	0.826	169.47	156.50

TABLE 11

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 017C
AVERAGE GC READINGS

DURATION

FROM	TO
7: 0: 0 11/16/1983	8: 5: 0 11/16/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
7	5.977	3.433	0.102	-0.002	0.118	77.354	0.729	3.868	91.600	24.58	1.097	0.849	41.15	37.27

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
7	16.084	8.282	0.293	0.687	0.614	46.869	3.271	21.862	97.961	24.27	1.084	0.839	165.50	153.39

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
7	7.567	6.162	0.225	0.373	0.492	66.583	1.993	9.919	92.313	24.69	1.103	0.853	79.94	73.64

TABLE 12

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 017D
AVERAGE GC READINGS

DURATION

FROM		TO	
12:10: 0	11/16/1983	16:10: 0	11/16/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
17	19.463	13.045	0.443	0.000	0.584	45.196	3.567	15.127	97.425	23.92	1.070	0.827	156.03	142.06

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
23	15.632	8.187	0.296	0.586	0.627	47.126	3.428	22.042	97.923	24.33	1.087	0.841	165.59	153.60

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
22	15.370	7.952	0.364	0.370	0.746	47.328	3.605	21.391	97.125	24.11	1.077	0.833	154.26	152.24

TABLE 13

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 018
AVERAGE GC READINGS

DURATION

FROM	TO
17:10: 0 11/16/1983	6:0:0 11/17/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	NBTU
58	8.609	10.194	0.129	0.638	1.722	67.172	0.039	0.381	88.385	24.40	1.091	0.843	36.05	31.18

TABLE 14

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 019A

AVERAGE GC READINGS

DURATION

FROM	TO
7:15: 0 11/17/1983	11:20: 0 11/17/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
15	17.159	11.789	0.443	0.049	0.937	48.835	2.642	13.659	95.514	23.92	1.069	0.827	134.77	122.86

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
17	15.229	8.544	0.334	0.432	0.633	48.144	3.628	21.528	98.472	24.62	1.100	0.851	164.35	152.39

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
14	14.616	8.552	0.374	0.395	0.746	49.096	3.539	20.529	97.847	24.52	1.100	0.851	158.72	147.10

TABLE 15

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 019B

AVERAGE GC READINGS

DURATION

FROM		TO	
20:10: 0	11/17/1983	6:15: 0	11/18/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
59	19.385	13.720	0.529	0.000	0.618	46.790	3.536	14.622	99.200	24.56	1.098	0.849	155.38	141.36

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
61	17.058	9.325	0.382	0.370	0.734	54.126	4.072	26.101	112.168	28.05	1.253	0.969	189.97	176.61

HGD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
59	15.830	8.421	0.396	0.433	0.771	51.625	3.838	23.297	104.611	26.14	1.168	0.903	175.22	162.66

TABLE 16

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 019C

AVERAGE GC READINGS

DURATION

FROM		TO	
14:10: 0	11/18/1983	23:30: 0	11/19/1983

HSD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
55	18.068	11.383	0.530	0.002	0.602	47.663	3.426	16.560	98.235	24.27	1.085	0.839	156.26	143.00

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
54	15.493	8.147	0.338	0.397	0.641	47.610	3.549	22.354	98.518	24.51	1.095	0.847	156.34	154.34

HSD INLET — BENDIX 6000 #1 CHROMATOGRAPH

# READ	H2	CO2	C2H6	H2S	O2	N2	CH4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
36	12.770	6.439	0.333	0.313	0.923	46.824	3.233	19.319	90.654	22.78	1.018	0.787	146.06	135.78

TABLE 17

ANALYSIS OF MINERALS BY X-RAY DIFFRACTION IN SULFIDED ZINC FERRITE GASIFIER RUN 103

BED HEIGHT (INCHES)	HEMATITE Fe_2O_3	FRANKLINITE $ZnFe_2O_4$ OR MAGNETITE Fe_3O_4 (4)	ZINCITE ZnO	ALPHA ZnS	BETA ZnS	PYRROHOTITE FeS	CEMENTITE Fe_3C
		PERCENT TII (3)					
0 (1)		33.9	6.8	12.8	37.3	9.2	-
6		25.7	6.9	18.6	35.4	11.7	1.7
16		14.6	15.9	21.0	25.9	9.4	13.3
23		2.8	52.9	6.5	2.5	-	35.2
26		-	65.0	<0.5	<0.5-	-	35.0
36		-	64.8	-	0.4	-	34.8
46 (2)		-	62.7	-	1.1	-	36.2
FRESH SORBENT	4.	89.	6.0	-	-	-	-

NOTES:

(1) Bottom/Inlet.

(2) Top/Outlet.

(3) Percent TII = Percent Total Integrated Intensity; Directly proportional to the concentration of the crystalline phase. Amorphous phase is not accounted for.

(4) Franklinite and Magnetite have the same crystal structure and, therefore, cannot be readily distinguished by the diffraction pattern.

TABLE 18

SORBENT ANALYSIS OF
SULFIDED ZINC FERRITE
GASIFIER RUN 103
TEST 019

Bed Height (Inches)	Total Carbon (% Wt)	Total Sulfur (% Wt)	BET Surface Area (Sq M/G)	Skeletal Density (G/CC)[3]	Porosity % [4]	Crush Strength (Kg DWL) ± S.D. [5]	Skeletal Density (G/CC)[6]	Porosity % [7]
0[1]	1.45	23.87						
0-6			2.06	4.59	2.01	9.0 ± 1.8		
6	2.33	25.64						
6-16			2.07	4.56	1.76	4.1 ± 2.2	4.62	35.0
16	7.62	19.75						
23	6.39	3.40	4.89	5.38	5.50	1.2 ± 0.9	4.88	61.8
26	6.35	0.58						
26-36			5.72	6.69	7.67	1.1 ± 0.7		
36	6.06	0.28						
36-46			6.22	6.66	7.89	1.0 ± 0.5		
46[2]	6.12	0.61					6.21	74.6
Fresh Sorbent	0	0.13	3.13	6.16	2.59	6.3 ± 2.8	-	60.6[8]

NOTES:

1. Bottom/Inlet during sulfidation - Outlet during regeneration.
2. Top/Outlet during sulfidation - Inlet during regeneration.
3. Skeletal Density by Helium Pycnometry.
4. Porosity by Nitrogen Adsorption for pores up to 600Å diameter.
5. Average and standard deviation of 15 measurements.
6. Skeletal density by mercury porosimetry.
7. Porosity by mercury porosimetry for pores down to 120Å diameter.
8. Porosity by mercury porosimetry for pores down to 30Å diameter.

TABLE 19

PARTICULATE SAMPLING DATA
HOT GAS DESULFURIZATION TEST UNIT
GASIFIER RUN 103

TEST #	HOURS FROM START OF TEST	PARTICLE LOADING g/Nm^3		MEDIAN PARTICLE SIZE μm (microns)		CHEMICAL ANALYSIS				
						C	H	N WT %	S	ASH
		INLET	EXIT	INLET	EXIT					
013	16.6 HRS	2.12		21.3		81.1	3.8	2.4	0.1	4.4
013	21.1 HRS		1.36		14.5	67.9	4.0	3.5	NM	10.2
013	42.5 HRS		0.469		13.1	73.8	4.9	4.3	NM	3.3
015	18.3 HRS	1.52		26.0		79.1	1.4	1.7	0.3	9.5
015	15.6 HRS		0.381		21.6	72.4	4.8	2.8	NM	7.4
017	16.6 HRS	2.09		13.0		75.9	4.6	3.2	0.9	2.9
017	27.7 HRS		0.381		5.3	76.5	2.1	1.4	1.1	12.9

NOTES:
NM= Not Measured due to insufficient sample size.

DATE: 12/2/83
SAMPLE: HGDS BAGNELL GI1181 83-957
ELECTROLYTE: ISOTON LOMAR D
DISPERSANT: LOMAR D
EQUIPMENT: TAII
APERTURES: 140
OPERATOR:BAGS

TABLE 20

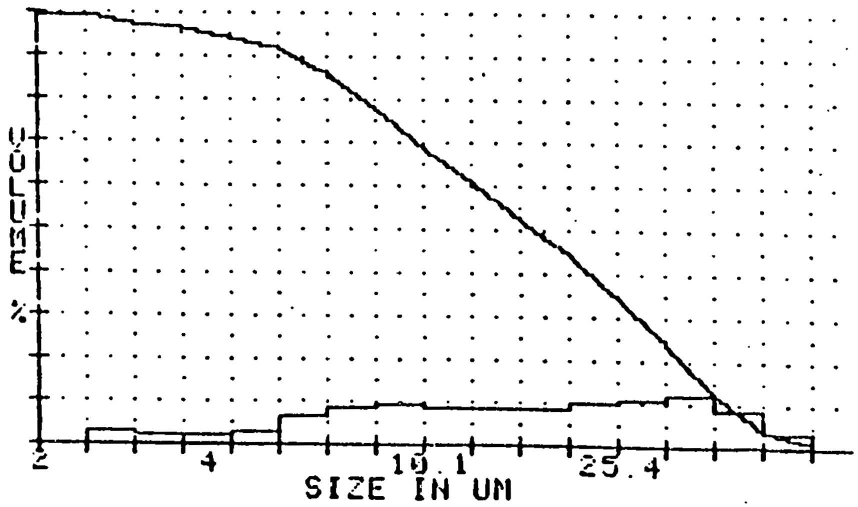
PARTICULATE SIZE DISTRIBUTION

TEST 013 INLET

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	2	0	100
2	2.52	2.6	100
3	3.17	1.6	97.4
4	4	1.6	95.8
5	5.04	2.2	94.2
6	6.35	6.1	92
7	8	8.1	85.9
8	10.08	9.1	77.8
9	12.7	8.2	68.7
10	16	8.5	60.5
11	20.16	8	52
12	25.4	9.6	44
13	32	10.5	34.4
14	40.32	11.8	23.9
15	50.8	8.6	12.1
16	64	3.5	3.5

VOLUME % STATISTICS

MEAN:	19.76 UM
MEDIAN:	21.3 UM
MODE:	43.02 UM
STANDARD DEVIATION:	2.25 UM
SKEWNESS:	.8 NEGATIVE
KURTOSIS:	2.78 PLATYKURTIC



DATE: 12/2/83
SAMPLE: HGD BAGHELL 601182 83-958
ELECTROLYTE: ISOTON
DISPERSANT: LOMARD
EQUIPMENT: TAII
APERTURES: 140
OPERATOR:BAGS

TABLE 21

PARTICULATE SIZE DISTRIBUTION

TEST 013 EXIT

21.1 HOURS FROM START OF TEST

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	2	0	100
2	2.52	4.4	100
3	3.17	3.7	95.6
4	4	4.5	91.9
5	5.04	6.5	87.4
6	6.35	9	80.9
7	8	9.6	71.9
8	10.08	8	62.3
9	12.7	7.4	54.3
10	16	10.4	46.9
11	20.16	14.7	36.5
12	25.4	9	21.8
13	32	5.5	12.8
14	40.32	2	7.3
15	50.8	5.2	5.3
16	64	0	.1

VOLUME % STATISTICS

MEAN:	13.42 UM
MEDIAN:	14.47 UM
MODE:	22.24 UM
STANDARD DEVIATION:	2.2 UM
SKEWNESS:	.93 NEGATIVE
KURTOSIS:	2.35 PLATYKURTIC

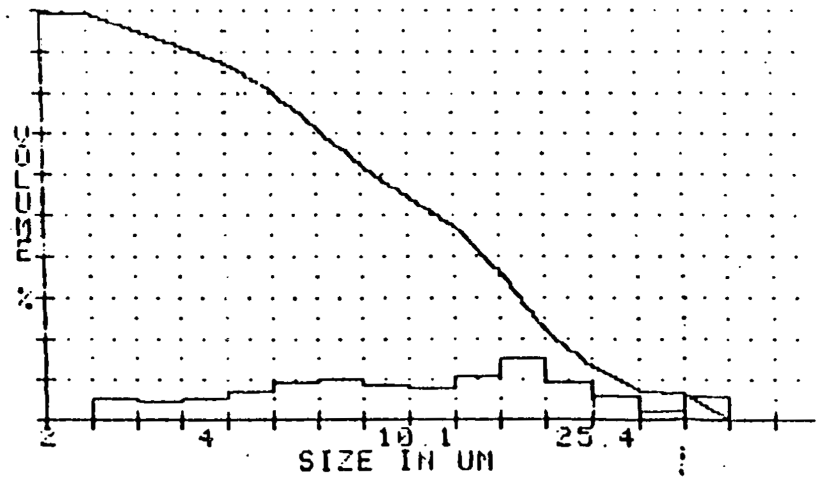


TABLE 22

DATE: 12-30-83
SAMPLE: 83-954
ELECTROLYTE: ISOTON II
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 280
OPERATOR: JK

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	4	0	100
2	5.04	1.8	100
3	6.35	2.6	98.2
4	8	4	95.6
5	10.08	5.9	91.6
6	12.7	9.1	85.7
7	16	12.4	76.6
8	20.16	12.7	64.2
9	25.4	16.7	51.5
10	32	15.3	34.8
11	40.32	9.5	19.5
12	50.8	4.7	10
13	64	2.9	5.3
14	80.63	1.3	2.4
15	101.59	1.2	1.1
16	128	0	0

PARTICULATE SIZE DISTRIBUTION

TEST 015 INLET

VOLUME % STATISTICS

MEAN:	24.67 UM
MEDIAN:	25.97 UM
MODE:	30.15 UM
STANDARD DEVIATION:	1.84 UM
SKEWNESS:	.97 NEGATIVE
KURTOSIS:	1.48 PLATYKURTIC

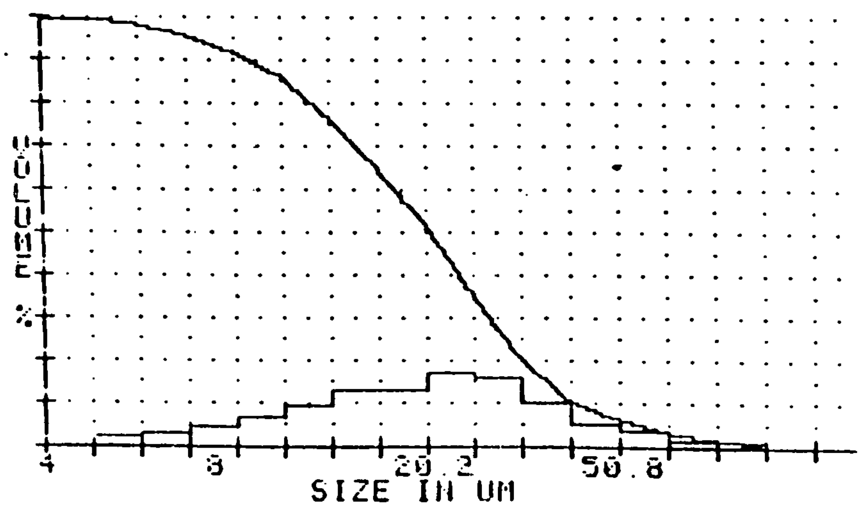


TABLE 23

DATE: 12/2/83
SAMPLE: HGDSU BAGWELL G011114 83-960
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TAII
APERTURES: 140
OPERATOR:BAGS

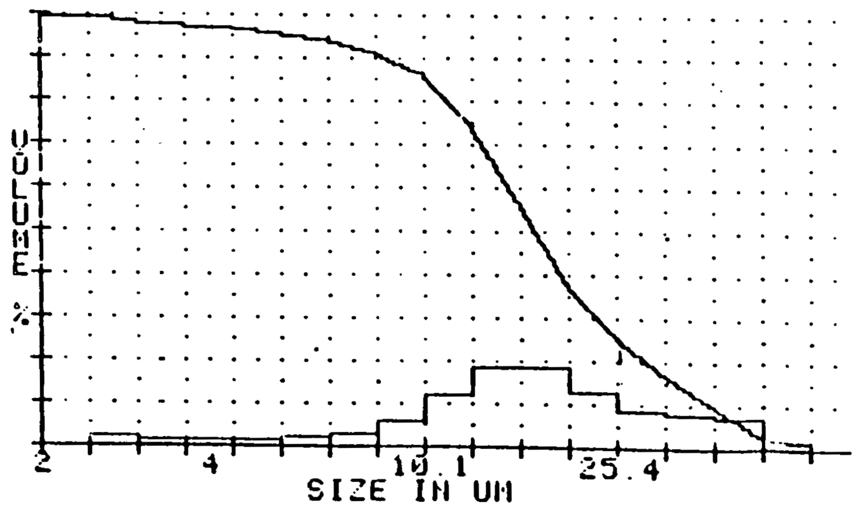
CH.#	SIZE	DIFF VOL %	CUM VOL %
1	2	0	100
2	2.52	1.5	100
3	3.17	1	98.5
4	4	1	97.5
5	5.04	1.3	96.5
6	6.35	1.5	95.2
7	8	2.4	93.7
8	10.08	5.3	91.3
9	12.7	12.2	86
10	16	18.4	73.8
11	20.16	18.3	55.4
12	25.4	12.3	37.1
13	32	8.5	24.8
14	40.32	7.4	16.3
15	50.8	7.1	8.9
16	64	1.8	1.8

PARTICULATE SIZE DISTRIBUTION

TEST 015 EXIT

VOLUME % STATISTICS

MEAN:	21.46 UM
MEDIAN:	21.6 UM
MODE:	20.12 UM
STANDARD DEVIATION:	1.87 UM
SKEWNESS:	.85 POSITIVE
KURTOSIS:	1.89 PLATYKURTIC



DATE: 12-30-83
SAMPLE: 83-556
ELECTROLYTE: ISOTONII
DISPERSANT: TYIIIA
EQUIPMENT: TAII
APERTURES: 140
OPERATOR:JK

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	2	0	100
2	2.52	.4	100
3	3.17	.6	99.6
4	4	1.2	99
5	5.04	4.2	97.8
6	6.35	9.9	93.6
7	8	17	83.7
8	10.08	16	66.7
9	12.7	9	50.7
10	16	16	41.7
11	20.16	17.8	25.7
12	25.4	7.4	7.9
13	32	.2	.5
14	40.32	.1	.3
15	50.8	.3	.2
16	64	0	0

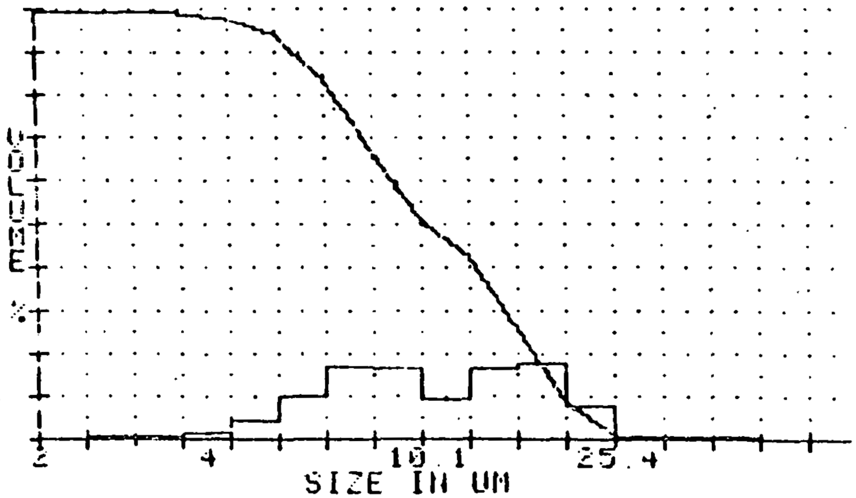
TABLE 24

PARTICULATE SIZE DISTRIBUTION

TEST 017 INLET

VOLUME % STATISTICS

MEAN:	13.24 UM
MEDIAN:	12.95 UM
MODE:	20.84 UM
STANDARD DEVIATION:	1.65 UM
SKEWNESS:	.98 NEGATIVE
KURTOSIS:	1.17 PLATYKURTIC



DATE: 12-30-83
SAMPLE: 83-955
ELECTROLYTE: ISOTONII
DISPERSANT: TYIIIA
EQUIPMENT: TAII
APERTURES: 140
OPERATOR: JK

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	2	0	100
2	2.52	8.5	100
3	3.17	15.5	91.5
4	4	23.5	76
5	5.04	13.9	52.5
6	6.35	16.8	38.6
7	8	11	21.8
8	10.08	6.8	10.8
9	12.7	2.2	4
10	16	.8	1.8
11	20.16	.3	1
12	25.4	.3	.7
13	32	.1	.4
14	40.32	.1	.3
15	50.8	.1	.2
16	64	.1	.1

TABLE 25

PARTICULATE SIZE DISTRIBUTION

TEST 017 EXIT

VOLUME % STATISTICS

MEAN:	5.65 UM
MEDIAN:	5.25 UM
MODE:	4.44 UM
STANDARD DEVIATION:	1.59 UM

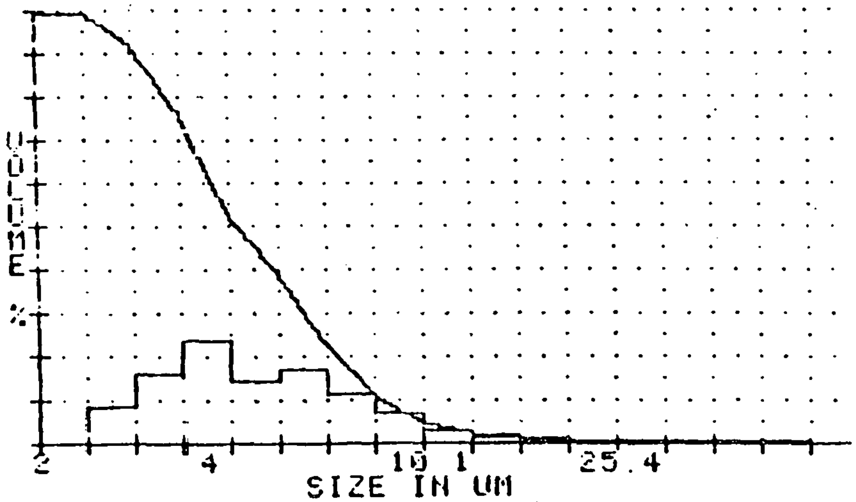


TABLE 26

TRACE COMPONENTS IN EXIT GAS CONDENSATE
DURING SULFIDATION
GASIFIER RUN 103

	AQUEOUS CONDENSATE							
	Test 013 Lab #100101		Test 015 Lab #100104		Test 017 Lab #100106		Test 019 Lab #100109	
Component	PPMW*	PPMW	PPMW*	PPMW	PPMW*	PPMW	PPMW*	PPMW
Ni	--	0.98	--	1.32	--	1.0	--	3.1
V	--	0.05	--	<0.05	--	<0.05	--	0.05
Cr	--	1.46	--	1.41	--	1.2	--	0.75
K	0.15	0.67	0.55	1.65	4.24	11.4	11.8	7.8
Na	0.57	7.4	1.63	3.06	22.9	49.6	20.6	49.6
Fe	--	1.6	--	7.04	--	15.0	--	14.1
Zn	--	0.87	--	1.66	--	3.5	--	13.0
Ca	--	3.5	--	3.15	--	13.1	--	27.2
Al	--	0.09	--	0.360	--	0.57	--	0.3
Se	--	0.614	--	0.080	--	0.046	--	0.008
As	--	2.38	--	3.58	--	0.62	--	0.32
Hg	--	<0.2	--	<0.2	--	<0.2	--	<0.2
Cu	--	<0.02	--	<0.02	--	<0.02	--	<0.02
F	93	72.4	73	49.0	56	37.9	47	43.7
Cl	1000	965	1460	2155	2040	1659	1840	2065
Br	4	6.2	6	9.7	12	6.7	24	25.6
SO_4	388	145	610	420	600	459	1180	785
PO_4	< 1	< 3	3	<3	<1	<3	<1	< 3
NO_3	< 1	< 2	3	<2	6	<2	4	<2
NH_4	3780	10130	4710	12468	4830	16000	4320	14500

NOTES:

1. Sampling location is at condenser C1C Knockout.
2. Metals determined by Atomic Absorption/Atomic Emission spectroscopy.
3. Anions and cations determined by ion chromatography.

* Second analysis taken 8 months later.

TABLE 27

TRACE COMPONENTS IN EXIT GAS CONDENSATE
DURING REGENERATION
GASIFIER RUN 103

	AQUEOUS CONDENSATE					
	Test 014 Lab #100103		Test 016 Lab #100105		Test 018 Lab #100108	
Component	PPMW*	PPMW	PPMW*	PPMW	PPMW*	PPMW
Ni	--	0.82	--	0.32	--	2.7
V	--	< 0.05	--	0.07	--	0.06
Cr	--	1.6	--	0.70	--	4.3
K	0.8	< 0.2	0.51	0.28	4.4	5.3
Na	2.6	< 0.04	1.88	2.8	20.6	17.7
Fe	--	15.2	--	3.42	--	75.2
Zn	--	0.91	--	0.60	--	28.1
Ca	--	0.81	--	0.66	--	54.4
Al	--	0.56	--	0.60	--	20.6
Se	--	0.04	--	0.016	--	
As	--	1.88	--	2.76	--	2.28
Hg	--	< 0.2	--	< 0.2	--	0.27
Cu	--	0.03	--	< 0.02	--	<0.02
F	38	28.2	35	25.7	41	40.1
Cl	440	400	610	251	1100	1148
Br	9	<4	<1	< 4	< 1	20.5
SO_4	3100	552	2030	378	1730	733
PO_4	<1	<3	<1	< 3	< 1	< 3
NO_3	2	<2	<1	< 2	< 1	< 2
NH_4	220	255	600	650	990	2052

NOTES:

1. Sampling location is at condenser C1C Knockout.
2. Metals determined by Atomic Absorption/Atomic Emission spectroscopy.
3. Anions and cations determined by ion chromatography.

* Second Analysis taken 8 months later.

TABLE 28

ULTIMATE ANALYSIS AND CONCENTRATION OF TARS LEAVING HGD TEST UNIT				
COMPONENT	WT%			
	TEST 013	TEST 015	TEST 017	TEST 019
ASH	0.02	0.09	0.07	2.34
S	2.76	3.78	2.28	3.16
H	7.14	6.89	7.44	8.14
N	0.88	1.02	1.41	1.17
TOTAL CARBON	72.20	79.77	67.28	57.94
CONCENTRATION OF TARS IN EXIT GAS OF HGD TEST UNIT	0.63	0.65	0.50	0.80

TABLE 29

TAR ULTIMATE ANALYSIS
AT HOT GAS DESULFURIZATION INLET

TEST # TYPE	SAMPLE #	DATE TIME	ASH	S	H WT. %	T.C.	N
Test 013 Sulfidation	28344	11/09/83 0500	0.25	1.15	7.46	65.63	1.14
Test 017 Sulfidation	28346	11/14/83 0500	0.40	0.94	8.07	58.76	1.33
Test 017 Sulfidation	28355*	11/14/83 2100	----	0.77	7.29	74.29	2.19
Test 019 Sulfidation	28348	11/17/83 1400	0.20	1.08	7.12	78.80	1.33
Test 019 Sulfidation	28349	11/18/83 0330	0.11	1.23	7.00	76.13	1.32
Test 019 Sulfidation	28358*	11/18/83 2100	0.14	1.38	6.37	83.79	2.04

* Limited sample available, sample ran on microsystem.

NOTES:

1. Sampling location is S-4 after gasifier cyclone
2. Sample No. 28344 obtained during operation on Air-blown Blacksville coal.
3. Sample No. 28346 and 28355 obtained during operation on Air-blown Kittanning coal.
4. Sample No. 28348, 28349 and 28358 obtained during operation on Air-blown Arkwright coal.

TABLE 30

ULTIMATE ANALYSIS OF COALS
USED DURING GASIFIER RUN 103

COMPONENT	WT % PITTSBURGH SEAM COAL ARKWRIGHT	PITTSBURGH SEAM COAL BLACKSVILLE	KITTANNING SEAM COAL KITTANNING
Moisture	0.39	1.1	0.82
Ash	7.15	10.55	6.43
S	1.82	3.20	0.98
H	5.12	5.58	5.55
N	3.05	1.30	1.61
Total Carbon	77.10	73.17	78.23
Volatiles	38.35	38.25	34.79
Btu/lb	13715	13308	14008

TABLE 31

TRACE COMPONENTS IN INLET AND EXIT GAS AQUEOUS CONDENSATE
DURING SULFIDATION GASIFIER RUN #103

TEST #	013	013	013	015	015	017
LOCATION	INLET	OUTLET	OUTLET	INLET	OUTLET	INLET
DATE	11/08/83	11/08/83	11/09/83	11/11/83	11/11/83	11/14/83
TIME	0950	1315	1140	1325	1040	1005
DURATION	160 Min	137 Min	145 Min	150 Min	120 Min	115 Min
LAB #	100303	100304	100305	100309	100308	100310
SAMPLE #	GI-11-8-1	GO-11-8-2	GO-11-9-3	GI-11-11-5	GO-11-11-4	GI-11-14-7
COMPONENT	PPMW	PPMW	PPMW	PPMW	PPMW	PPMW
Ni	0.14	3.8	0.04	0.23	1.7	0.056
V	<0.05	<0.05	<0.05	<0.05	<0.05	0.099
Cr	2.3	0.12	0.10	3.0	0.37	1.32
K	0.80	3.31	0.82	1.2	4.4	2.63
Na	6.1	12.2	3.7	0.18	8.2	11.5
Fe	14.1	2.8	2.2	5.4	4.0	20.32
Zn	<0.1	93.2	17.2	<0.1	19.8	<0.1
Ca	4.5	8.7	3.3	15.8	9.0	11.96
Al	0.19	<0.02	<0.02	0.94	0.50	0.136
Se	0.103	0.004	<0.005	0.636	0.005	0.285
As	3.22	0.30	0.07	1.66	0.25	1.66
Hg	<0.2	0.78	<0.2	<0.2	<0.2	<0.2
Cu	0.041	0.151	<0.02	<0.02	NS	NS
F	142	17	24	53	NS	NS
Cl	2058	8414	965	2012	NS	NS
Br	32	13	<4	26	NS	NS
NO3	<2	<2	<2	<2	NS	NS
SO4	465	700	495	470	NS	NS
PO4	<3	<3	<3	<3	NS	NS
NH4	7916	13196	11959	13402	NS	NS

NOTES:

1. Aqueous condensate data obtained from HGD inlet and outlet particulate sampling systems.
2. NS indicates that the component was not measured.

TABLE 32

TRACE COMPONENTS IN INLET AND EXIT GAS
DURING SULFIDATION GASIFIER RUN #103
TEST #013

LOCATION	INLET	OUTLET	OUTLET	OUTLET
LAB #	100303	100304	100305	100101
SAMPLE #	GI-11-8-1	GO-11-8-2	GO-11-9-3	----
	CALCULATED GAS PHASE COMPOSITIONS			
COMPONENT	PPMW	PPMW	PPMW	PPMW
Ni	0.014	0.410	0.004	0.048
V	<0.005	<0.005	<0.005	0.002
Cr	0.228	0.013	0.009	0.071
K	0.079	0.357	0.076	0.032
Na	0.604	1.318	0.344	0.359
Fe	1.394	0.302	0.205	0.078
Zn	<0.01	10.07	1.60	0.042
Ca	0.446	0.940	0.307	0.170
Al	0.019	<0.002	<0.002	0.004
Se	0.010	0.0004	<0.0005	0.030
As	0.319	0.032	0.007	0.115
Hg	<0.02	0.084	<0.019	<0.010
Cu	0.004	0.016	<0.002	<0.001
F	14.1	1.84	2.23	3.51
Cl	204	909	90	47
Br	3.17	1.404	1.21	0.301
NO3	<0.198	<0.216	<0.186	<0.097
SO4	46	76	46	7
PO4	<0.297	<0.324	<0.279	<0.146
NH4	784	1425	1112	491

TABLE 33

TRACE COMPONENTS IN INLET AND EXIT GAS
DURING SULFIDATION GASIFIER RUN #103
TEST #015

LOCATION	INLET	OUTLET	OUTLET
LAB #	100309	100308	100104
SAMPLE #	GI-11-11-5	GO-11-11-4	---
	CALCULATED GAS PHASE COMPOSITION		
COMPONENT	PPMW	PPMW	PPMW
Ni	0.022	0.054	0.075
V	<0.005	<0.002	<0.003
Cr	0.288	0.012	0.080
K	0.115	0.141	0.093
Na	0.017	0.262	0.173
Fe	0.518	0.128	0.398
Zn	<0.01	0.634	0.094
Ca	1.517	0.288	0.178
Al	0.090	0.016	0.020
Se	0.061	0.0002	0.005
As	0.159	0.008	0.202
Hg	<0.019	<0.006	<0.011
Cu	<0.002	NS	<0.001
F	5.1	NS	2.77
Cl	193	NS	122
Br	2.5	NS	0.548
NO3	<0.19	NS	<0.11
SO4	45	NS	24
PO4	<0.29	NS	<0.17
NH4	1287	NS	704

NS indicates that the component was not measured.

TABLE 34

TRACE COMPONENTS IN INLET AND EXIT GAS
DURING SULFIDATION GASIFIER RUN #103
TEST #017

LOCATION	INLET	OUTLET
LAB #	100310	100106
SAMPLE #	GI-11-14-7	---
	CALCULATED GAS PHASE COMPOSITION	
COMPONENT	PPMW	PPMW
Ni	0.003	0.043
V	0.005	<0.002
Cr	0.061	0.052
K	0.121	0.490
Na	0.529	2.133
Fe	0.935	0.645
Zn	<0.005	0.151
Ca	0.550	0.563
Al	0.006	0.025
Se	0.013	0.002
As	0.076	0.027
Hg	<0.009	<0.009
Cu	NS	<0.001
F	NS	1.63
Cl	NS	71
Br	NS	0.288
NO3	NS	<0.086
SO4	NS	19.7
PO4	NS	<0.129
NH4	NS	688

NS indicates that the component was not measured.

TABLE 35

ANALYSIS OF INLET AND OUTLET TARS DURING GASIFIER RUN #103
TEST #013

LOCATION	INLET	OUTLET
LAB SAMPLE #	28307	28308
PARTICULATE SAMPLE #	GI-11-8-1	GO-11-8-2
COMPONENT	WEIGHT % OF TAR SAMPLE	
BENZENE	0.486	0.025
TOLUENE	1.36	0.032
ETHYL-BENZENE	0.317	0.183
PYRIDINE	0.015	0.037
PICOLINE	0.018	0.094
M & P XYLENE	1.43	0.351
STYRENE	0.230	0.006
O-XYLENE	0.454	0.161
PHENOL	1.32	1.71
INDANE	0.379	0.418
INDENE	1.81	1.07
O-CRESOL	1.15	1.22
M & P CRESOL	2.34	2.44
NAPHTHALENE	4.49	4.59
BENZOTHIOPHENE	0.354	0.362
2-METHYL NAPHTHALENE	2.09	2.00
1-METHYL NAPHTHALENE	1.25	1.09
ACENAPHTHACENE	0.600	0.294
ACENAPHTHENE	0.606	0.794
DIBENZOFURAN	0.456	0.422
FLUORENE	0.772	0.767
PHENANTHRENE	1.21	1.54
ANTHRACENE	0.534	0.738
CARBAZOLE	0.252	0.315
FLUORANTHENE	0.621	1.32
PYRENE	0.645	1.47
MW 216	0.376	0.848
MW 228	0.310	0.804
CHRYSENE	0.269	0.731
BENZO (A) PYRENE	0.113	0.365

NOTES:

1. Only the lighter fractions of the tar were analyzed. Numbers reported are weight % of the total sample.
2. Analysis was conducted using a gas chromatagraph coupled with a mass spectrometer.
3. Analysis was performed by Weston Laboratories, Weston Way, West Chester, PA

Sidestream Testing - Gasifier Run 104

Hot Gas Desulfurization Sidestream Test Unit, Fixed-Bed Gasifier Run No. 104

Introduction

The high-temperature, sidestream desulfurization unit (1,2,3) was operated for a fourth and final series of tests during fixed-bed gasifier Run No. 104 (4,5,6) between December 8 and 13.

In these tests the main objective was to investigate the desulfurization of lignite gas.

Operation of Sidestream Test Unit

With a single batch of the same zinc ferrite sorbent used in all the previous tests, the unit was operated for two sulfidation periods with an intervening regeneration. The gasifier was operated in the air-blown mode, being fed with North Dakota lignite during the first sulfidation and Arkwright coal during the second. The operational schedule is shown in Figure 1.

The reactor was filled with the usual 20.4 kg of sorbent extrusions. Furnace temperatures were set at 1,000°F for the first sulfidation and 1,200°F for the second. In both cases the nominal space velocity was 2,000 h^{-1}. For the regeneration the furnace temperatures were set at the usual 1,000°F and nominal space velocity was 500 h^{-1}.

Average operating parameters for the tests are shown on the ADACS flow schematics in Figures 2 to 7. Test No. 020 is separated into four on-stream parts because of gas supply interruptions.

In Test No. 020, with the gasifier providing gas from lignite at 160 to 200 psig, breakthrough of hydrogen sulfide occurred after about 45 hours on-stream time. The H_2S level was generally less than 5 ppm. Reactor pressure drop increased only slightly from about 0.15 to 0.5 psi. There was an increase of reactor temperatures approaching 100°F. Reactor temperatures, pressure drop, and gas flow rate are shown in Figures 8, 9, and 10.

In the regeneration Test No. 021, the steam/air ratio, initially 85/15, was decreased stepwise during the first few hours to 80/20 in order to achieve reaction zone temperatures of about 1,500°F. The reactor pressure drop was very low throughout, about 0.15 psi. Reactor temperatures, pressure drop, and flow rates are shown in Figures 11, 12, and 13.

Test No. 022, lasting about 16 hours, was the final sulfidation of the test series. In this case, with the gasifier providing gas from Arkwright coal at about 130 psig, the aim was to study the effect of the higher temperature on sorbent behavior.

The hydrogen sulfide level in the exit gas initially exceeded 100 ppm, gradually falling to 15 ppm after 8 hours and then resuming an upward trend shortly thereafter. The reactor pressure drop behaved similarly to that in previous tests with Arkwright coal at similar conditions, exceeding bed-lifting pressure after about 12 hours on-stream. The pressure drop, initially about 0.2 psi, was about 4 psi at the termination of the test. The temperature rise was about 50°F, somewhat less than usual, and attributed to the lesser extent of the shift reaction at the higher temperature. Reactor temperatures, pressure drop, and gas flow rate are shown in Figures 14, 15, and 16.

Results

- Data Summary

  The various process conditions and data collected in Test Nos. 020, 021, and 022 are summarized in Figures 17, 18, and 19.

- Absorption of Sulfur Compounds

  Laboratory gas chromatograph analyses of grab samples of gas exiting the hot gas desulfurization unit condenser are shown in Table 1 (7). Corresponding analyses of grab samples of inlet gas from the gasifier cyclone exit are given in Table 2 during the sulfidation tests only. The concentrations of H_2S, COS, CS_2, and thiophene (C_4H_4S), respectively at both unit inlet and outlet for Test No. 020, are plotted on the semilog plots in Figures 20 to 23. The exit levels of these low molecular weight sulfur compounds are similar to those found in previous tests, the average total level being less than about 10 ppm. H_2S and COS levels during Test No. 022, in which the sorbent was at a temperature of 1,200° to 1,300°F, were anomalously high throughout their sum never falling below about 45 ppm. Possible reasons for this are discussed later.

  The hydrogen sulfide concentration in exit gas from the unit was also measured during the sulfidation tests by the Baseline Industries on-line gas chromatograph and by detector tubes. The values obtained are plotted against on-stream time in Figures 24 and 25, together with some values for sulfur dioxide measured by detector tubes during the second sulfidation. Agreement between on-line chromatograph and detector tube values can be seen to be fairly good with the former somewhat higher the further the values are above the chromatograph calibration point of 3 ppm (see Figure 24). For much higher H_2S values (see Figure 25), the order seems to be reversed.

  Carbonyl sulfide and sulfur dioxide concentrations were also measured by the Baseline chromatograph during the sulfidations. The values, together with those for hydrogen sulfide, are plotted in Figures 26 and 27. COS was not detectable in Test No. 020. In Test No. 022, however, where the H_2S concentration was much higher, the measured COS level was approximately one-third of the measured H_2S level. As found previously with regenerated sorbent, some sulfur dioxide can be seen in the early stages of the test.

  The amount of sulfur contained in the sorbent at breakthrough approached 20 wt. percent of the fresh sorbent weight in both the sulfidations.

- Gas Analysis for Major Species

  Laboratory gas chromatographic analyses, for major species of grab samples of exit and inlets gas, are shown in Tables 3 and 4 respectively.

  In contrast to Table 4, the oxygen levels in Table 3 during the sulfidations are inordinately high. This is probably caused by incomplete displacement of air from the sample bottles. Oxygen also appears to be excessively high during the regeneration. Most of the same species were also measured by Bendix on-line gas chromatographs in the exit and inlet gas streams. The measurements, averaged by ADACS statistical analysis, are shown in Tables 5 to 10. Because of the larger water content of the lignite gas, the CO shift reaction proceeded to a larger extent in Test No. 020, occurring to as much as 8 vol. percent of the gas on a dry basis. The extent of the shift reaction in Test No. 022 with Arkwright coal gas was significantly less, about 4 vol. percent. As in previous tests, there is an apparent loss of total carbon oxides between inlet and outlet gas, attributed to carbon deposition.

- Analysis of Regeneration Exit Gas

  The regeneration exit gas (see Tables 1, 3, and 9) in the early stages of regeneration, Test No. 021, contained substantial quantities of hydrogen, carbon dioxide, and reduced sulfur compounds which is attributed to the presence of iron carbide/carbon in the sorbent. More of these compounds, i.e., H_2, CO_2, H_2S, COS, and CS_2, were present than expected. The higher water content of the lignite coal gas, it was thought, should inhibit the formation of iron carbide. After the first few hours of the regeneration, the sulfur content of the exit gas was predominantly in the form of sulfur dioxide.

  ADACS plots of CO_2, H_2S, O_2, CO, and H_2 in the regeneration gas from the reactor exit condenser are shown in Figures 28 to 32.

- Sorbent Characterization

  After Test No. 022, the sulfidized sorbent was removed from the reactor and sampled for analysis and characterization at a number of points along the reactor length. The results are detailed in Tables 11 and 12. The mineral analysis (see Table 11) differs markedly from that in previous tests with Arkwright coal gas in that the sorbent contains large quantities of wustite, FeO. The amount of wustite is larger at the reactor exit where there is no pyrrhotite. At the reactor inlet a good part of the sorbent's iron content has been converted to pyrrhotite. Remarkably, there is a significant amount of unsulfidized zinc oxide at the reactor inlet, suggesting that it is somewhat inaccessible to hydrogen sulfide which may explain why the sorbent was unable to remove it to its equilibrium level. Only a small amount of magnetite/zinc ferrite remains.

  The presence of wustite is undoubtedly due to the high operating temperature of the sorbent, 1,200° to 1,300°F, and the relatively small water concentration in the coal gas undergoing desulfurization.

Table 12 shows that the sorbent contains about 22 wt. percent of sulfur at the reactor inlet, which is rather less than found in prior comparable tests. The carbon content is quite large and comparable to previous tests where iron carbide was found (none was detected in this case). Surface areas and pore volumes were similar throughout and not much different from those of fresh sorbent. Another remarkable result is that the crush strength of the sorbent significantly exceeded that for fresh sorbent at all the sample points. Less than 5 wt. percent of the removed sorbent passed through a No. 12 screen. This relatively small amount of disintegration is associated with the sorbent's preserved crush strength, in marked contrast to previous tests when the sorbent contained substantial amounts of iron carbide.

- Particulate Analysis

  In this test series, in Test No. 020, only a single sample of exit gas was taken for characterization of its particulate content (8). The results are summarized in Table 13. The particulate loading of nearly 2 g/Nm^3 is similar in magnitude to the inlet gas loadings in previous tests. The median particle size of about 6 microns is comparable to that found in Test No. 017 (3) with Kittanning coal gas; though in the latter case, the exit gas loading was a few times less. In both cases, a very low reactor pressure drop increase occurred for about 50 hours on-stream time which provides further evidence of a positive correlation between rate of increase of pressure drop and median particle size. Chemical analysis of the particulate indicates that it consists mostly of fly ash from the coal gas. The detailed size distribution for the particulate sample is shown in Table 14.

Condensate Analysis

Reactor exit gas condensates were collected from Test Nos. 020 and 021. Analyses of composite samples are shown in Tables 15 and 16, respectively. For Test No. 020, the calculated gas composition is also included. The sulfidation condensate contains significantly greater amounts of Na, K, and Ca, but less Cl and F. The regeneration condensate is also enriched in these elements and, assuming they derive from the previous sulfidation, it is estimated that about 32 percent of Na and 20 percent of Cl are absorbed by the sorbent. In contrast to previous tests with bituminous coal gas, where up to 20 percent of NH_4 was absorbed, less than 1 percent appears to be absorbed in this case. This may be the result of the lower chloride and higher water contents of the lignite gas.

Samples of inlet and outlet condensate during sulfidation Test No. 020 were also obtained in the course of obtaining particulate samples. Their analyses are shown in Table 17. Comparing this analysis of the outlet condensate with that of the composite outlet condensate (see Table 15), the concentrations of Na, K, and Ca are much less in the former. A possible reason for this could be that these constituents have leached out from the relatively large amount of particulate which is not removed before condensation occurs in this case. The significant discrepancy between the chloride concentrations in the two cases is not clear.

The tar layer amounting to about 4 wt. percent was separated from the total exit gas condensate from sulfidation Test No. 020. The ultimate analysis of a sample of this is shown in Table 18. It appears that the sample contained a large amount of water which could not be separated. Corresponding analyses of tar condensate from the unit inlet for both sulfidation tests are shown in Table 19. Regrettably, the sulfur concentrations are missing for the lignite tar. Ultimate analyses of the lignite and Arkwright coals gasified are shown in Table 20.

<u>Conclusions</u>

The fourth and final series of tests of the hot gas desulfurization unit in a sidestream of the METC 24 t/d fixed-bed gasifier was successfully concluded. Two sulfidations and an intermediate regeneration of the same zinc ferrite sorbent tested in the previous three series were carried out. North Dakota lignite and Arkwright bituminous coal, respectively, were gasified in the air-blown mode for the two sulfidation tests.

The amount of sulfur absorbed before breakthrough was similar to that in previous tests, about 20 wt. percent of fresh sorbent. In the final sulfidation test, the only one in the test series in the higher temperature range of 1,200° to 1,300°F, much of the sorbent iron content was converted to wustite, FeO. This appeared to slow down the rate at which sulfur compounds reacted with the sorbent, and the hydrogen sulfide level attained before breakthrough was significantly higher than the equilibrium level predicted.

A smaller amount of refractory sulfur compounds in the case of lignite gas than in the case of bituminous coal gas appears to be carried through the desulfurization sorbent in the vaporized tars.

The particulate loading of the desulfurization unit exit gas was rather high in the lignite test, about 2 g/Nm^3. The median particulate size, however, was low, about 5 μm and the pressure drop across the reactor remained very low throughout. This reinforces the conclusion previously reached that there is a strong positive correlation between rate of pressure drop increase and median particle size. A particulate loading of about 1 g/Nm^3 and a median particle size of about 10 μm appear to be an acceptable specification for inlet gas to the hot gas desulfurization unit.

Halides and alkali and alkaline earth metals were absorbed from the lignite gas by the sorbent and released during regeneration in the proportions of about 32 percent of sodium and 20 percent of chloride for example. Very little ammonia was absorbed in contrast to previous experience.

1:910319ds.2jj

REFERENCES

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2. Grindley, T. "Hot Gas Desulfurization Sidestream Test Unit, Fixed-Bed Gasifier Run No. 102," IR No. 1984, September 1984, METC.

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7. Lamey, S. C., R. R. Romanosky, and J. J. Kovach, "Analytical Data -- Producer Run No. 104," IR No. 2059, November 1984, METC.

8. Data provided by R. D. Bagnell, Analytical Sciences Branch, METC.

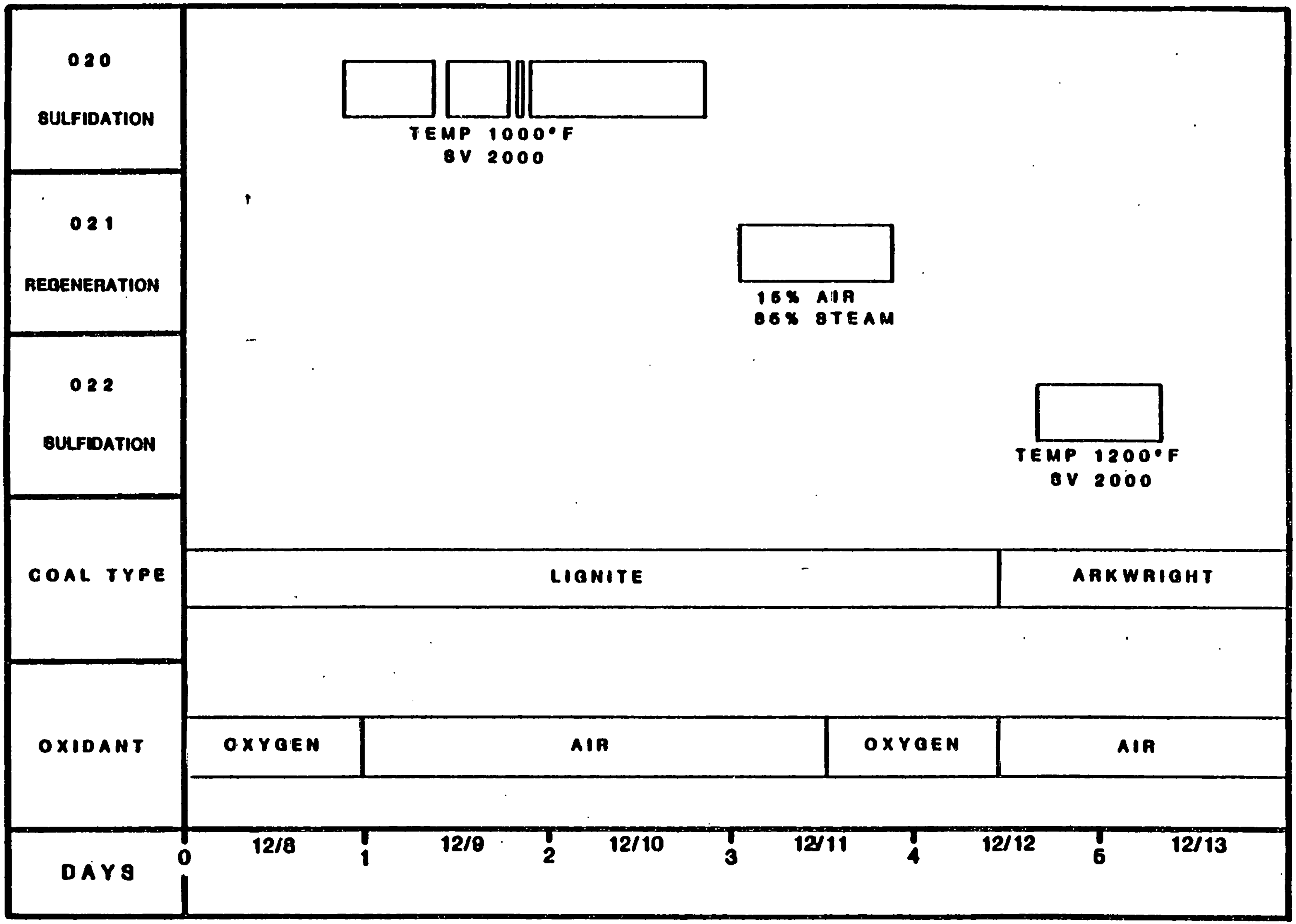
1B:11-5-84:1s:5ab

ADDENDUM TO APPENDIX H

The Baseline G.C. on-line H_2S, COS, and SO_2 readings as plotted are correct only for the points: H_2S: 3 ppm; COS: 1.7 ppm; and SO_2: 5.1 ppm. Calibration curves are available for correction. Corrected plots are given in Volume 1.

Refer to letter to T. Grindley from E. E. Gorski and P. Johnson, EG&G Washington Analytical Services Center, Inc. January 30, 1986. Subject: DOE-METC/EG&G Contract No. DE-AC21-85MC21353; WBS No. 9KEX-10. HGD Project: Baseline GC/Integrator Conversion Curves.

FIGURE 1



SCHEDULE OF EVENTS

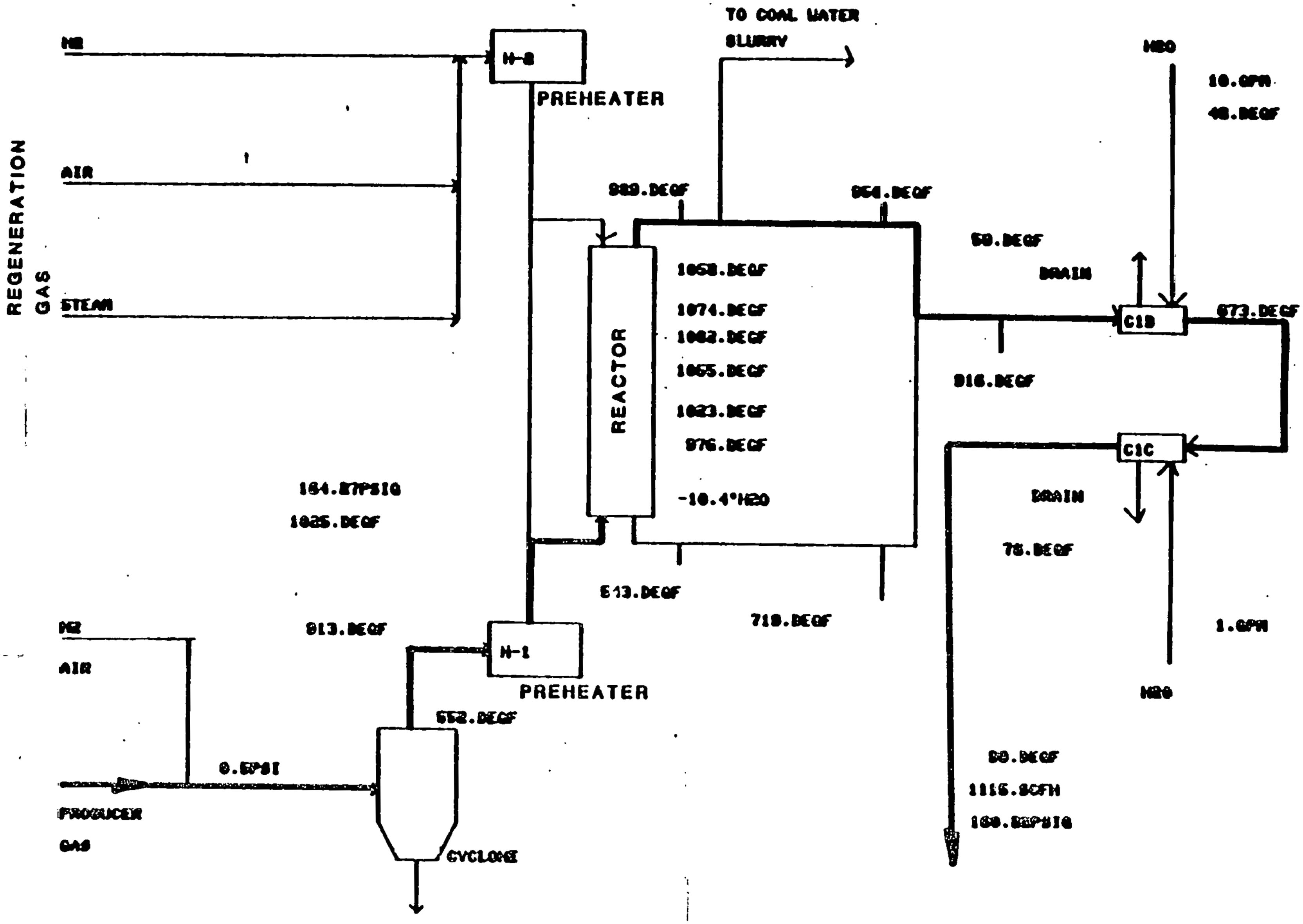
HOT GAS DESULFURIZATION SIDESTREAM TESTING 1983

GASIFIER RUN 104

FIGURE 2

TEST 020 A

AVERAGE OPERATING PARAMETERS



AVERAGE(S) FOR PERIOD FROM: 21:30:18 10/ 8/1983 TO 0:30:18 10/ 8/1983

FIGURE 3

TEST 02 B

AVERAGE OPERATING PARAMETERS

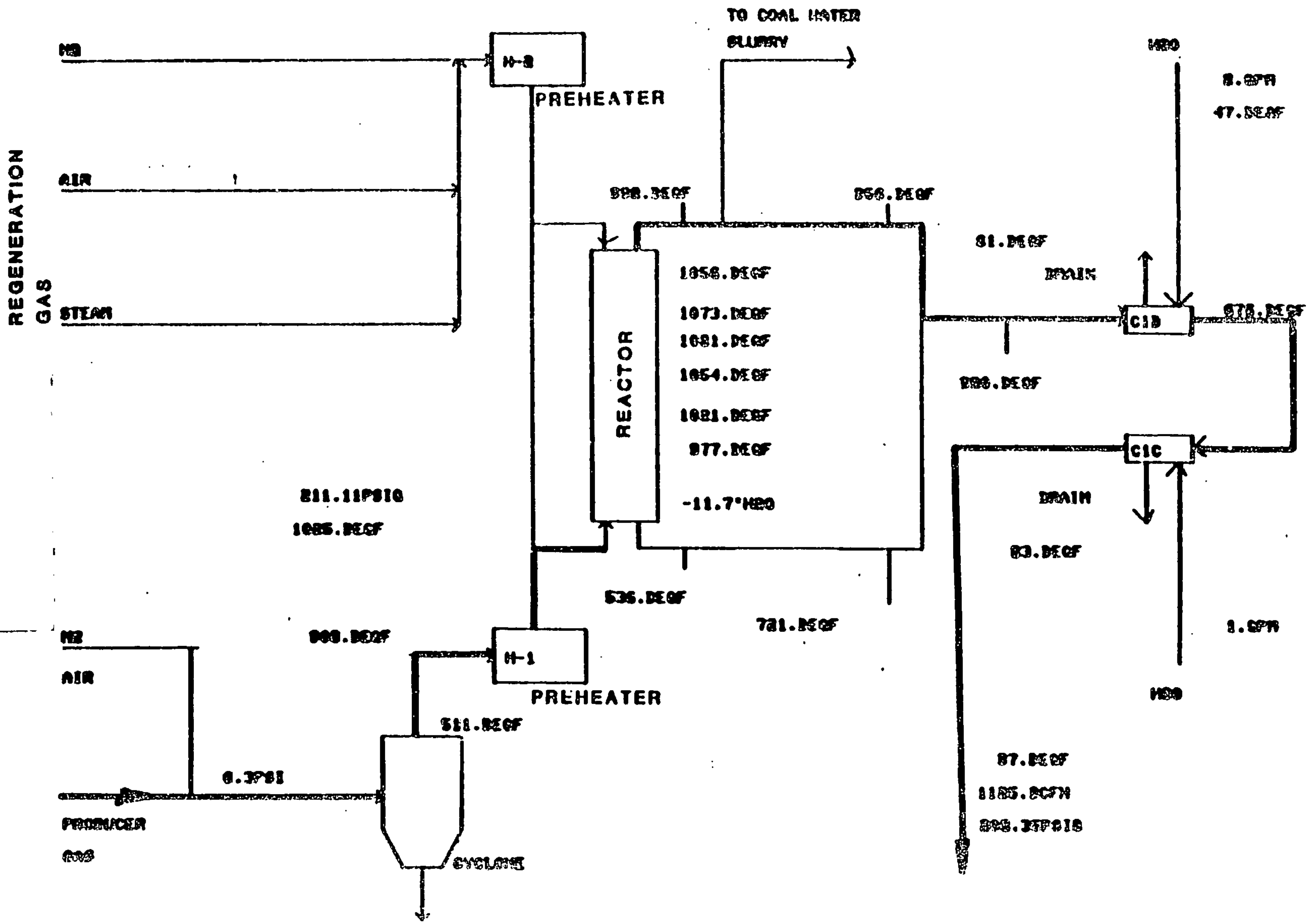


FIGURE 4

TES. 020 C

AVERAGE OPERATING PARAMETERS

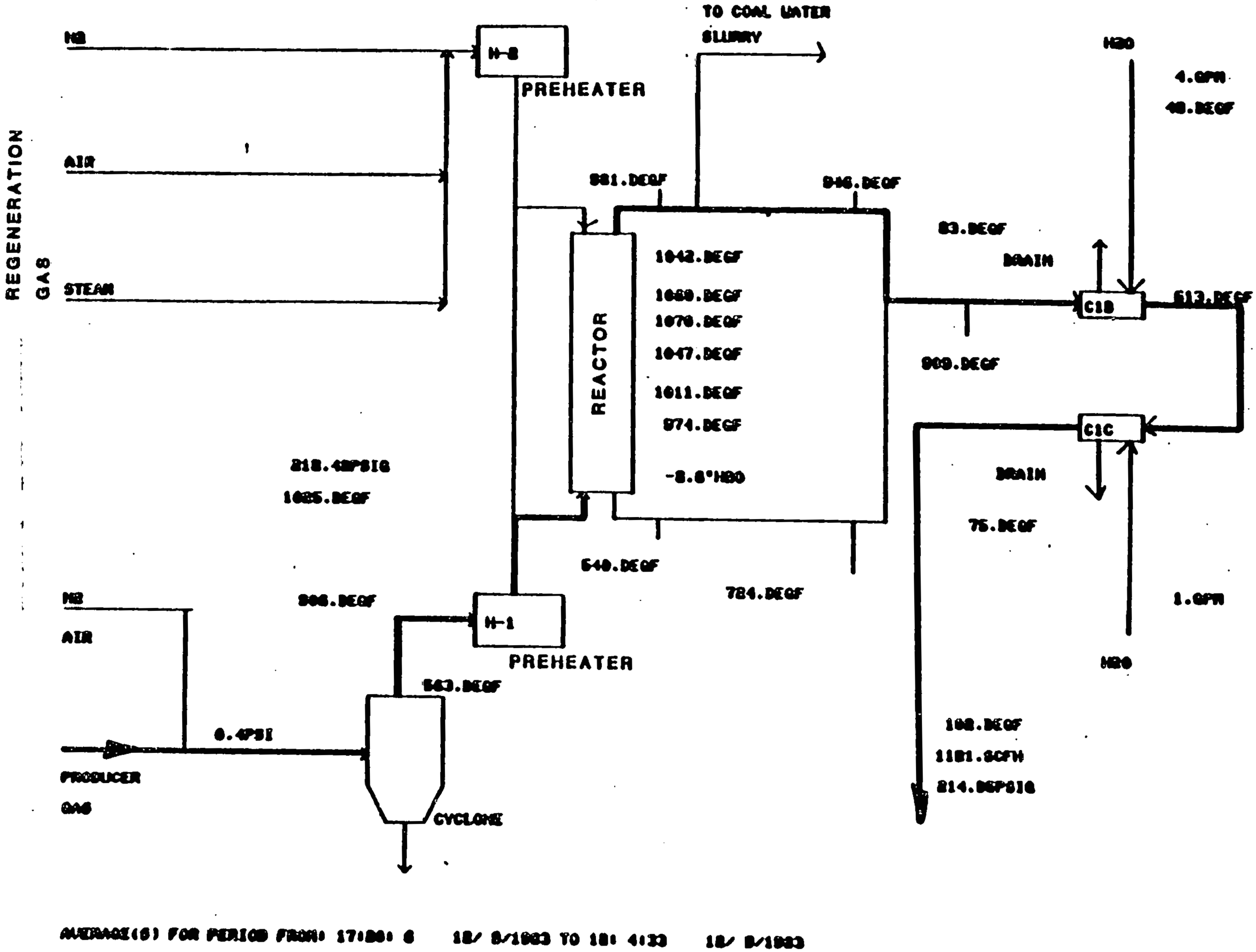


FIGURE 5

TEST. 020 D

AVERAGE OPERATING PARAMETERS

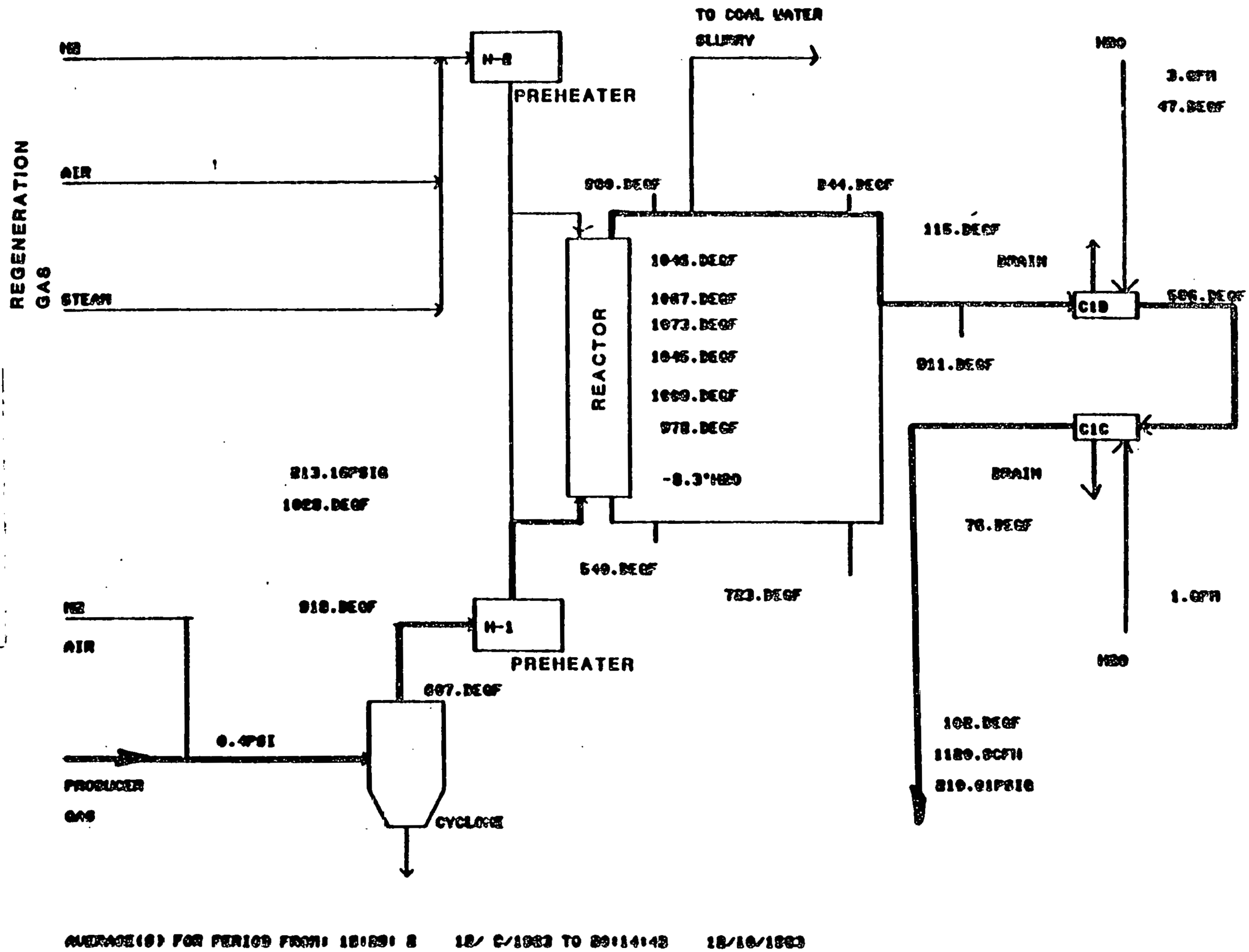
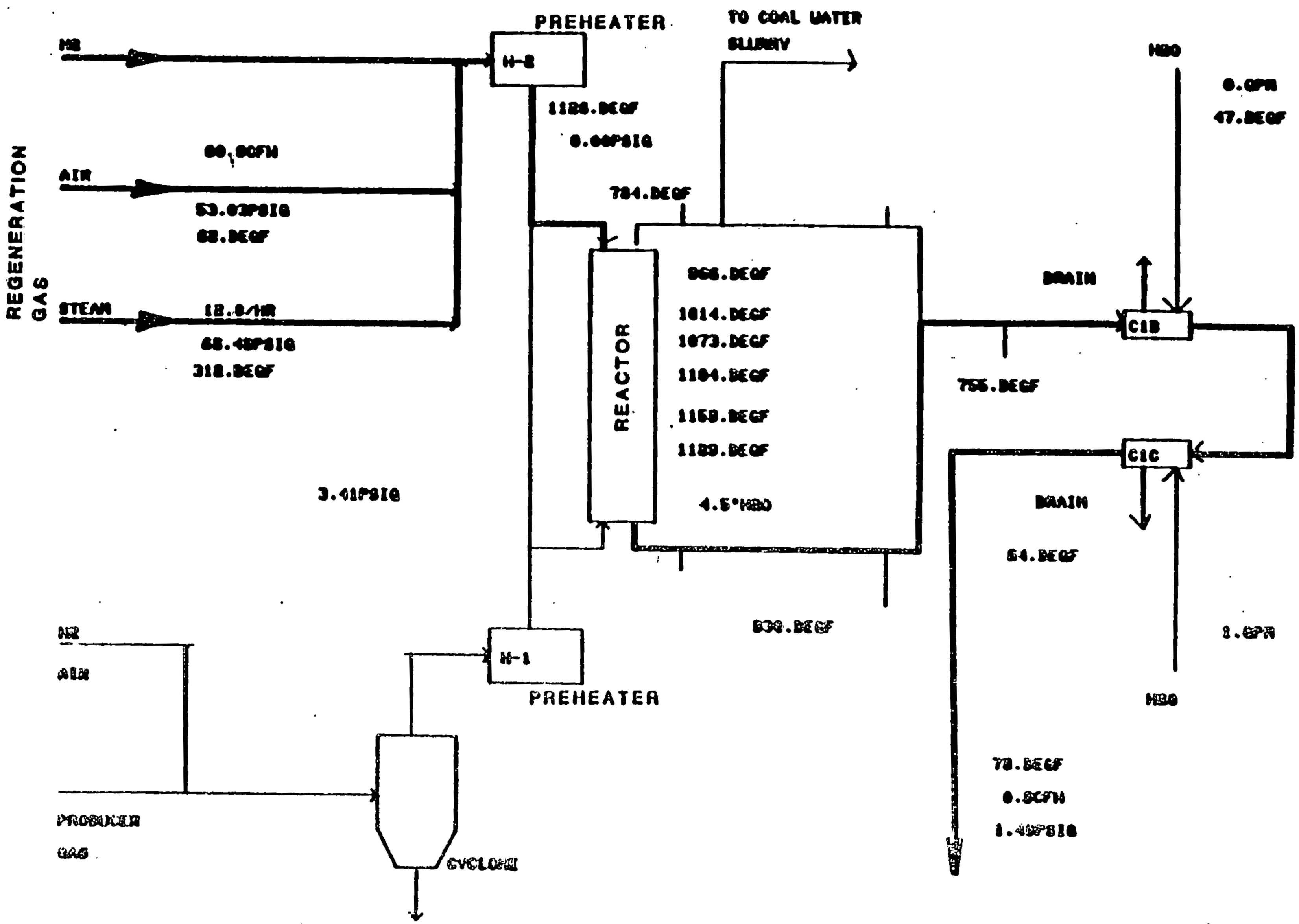


FIGURE 6

TE 021

AVERAGE OPERATING PARAMETERS

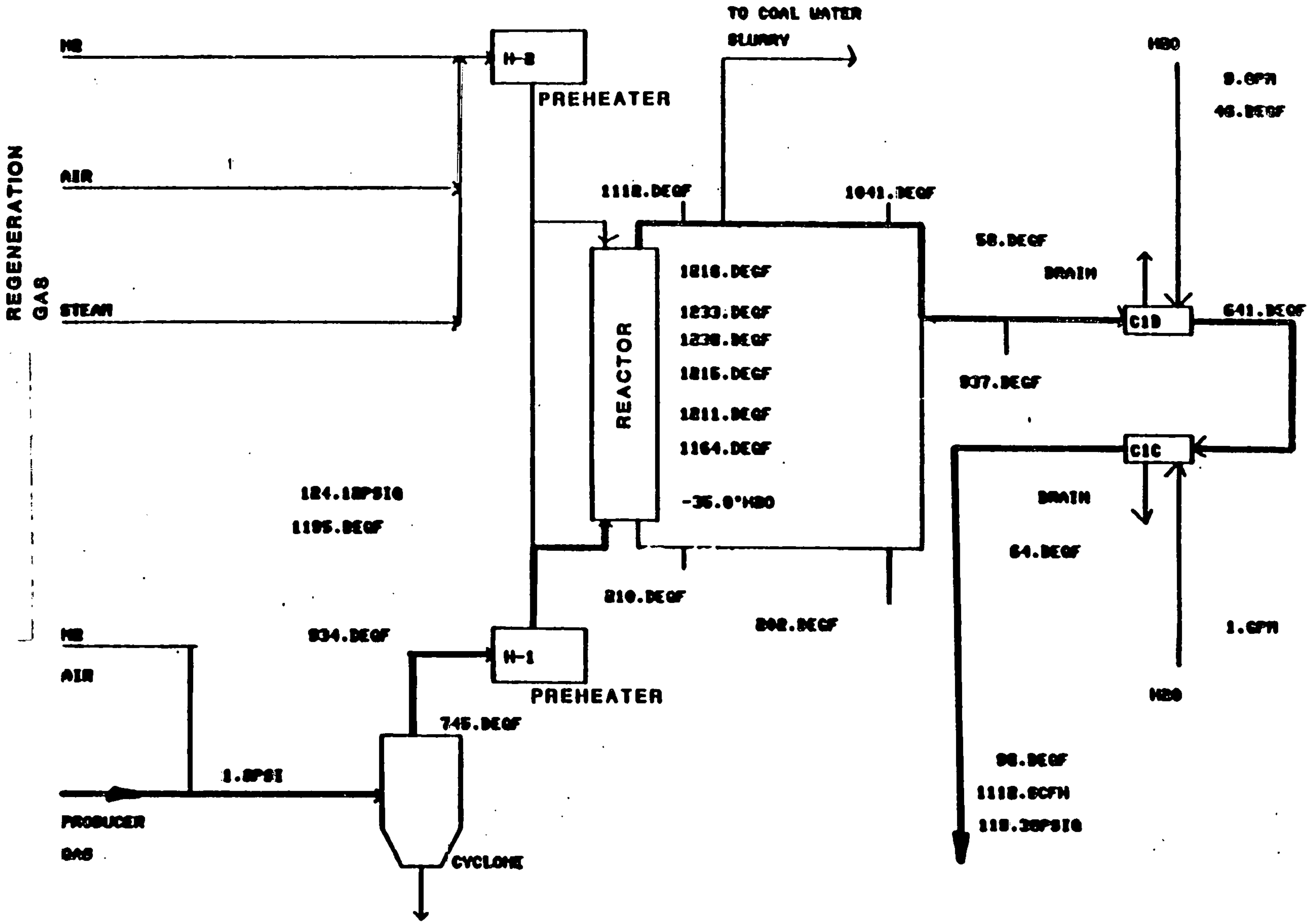


AVERAGE(S) FOR PERIOD FROM: [illegible] TO [illegible]

FIGURE 7

TEST 022

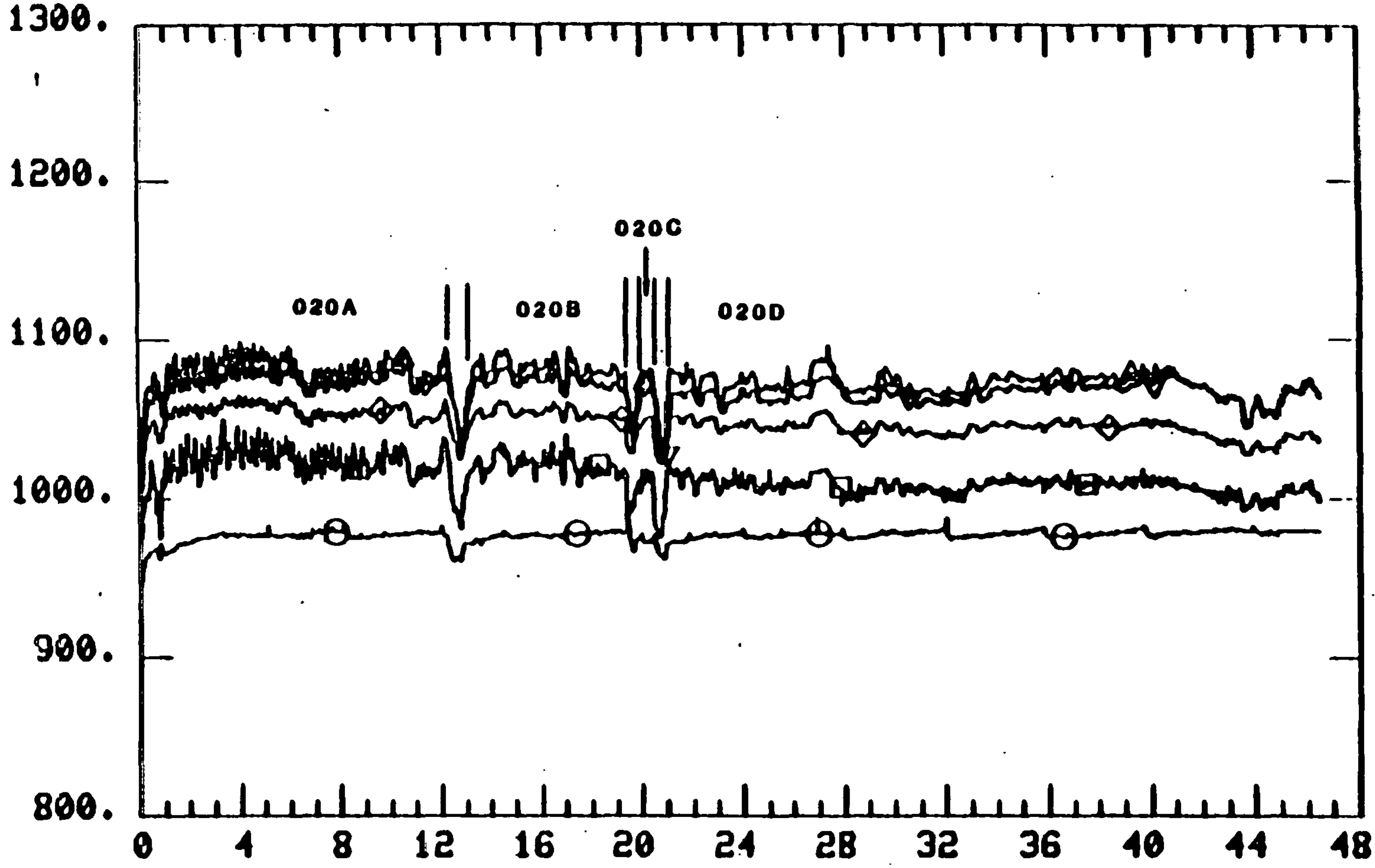
AVERAGE OPERATING PARAMETERS



AVERAGE(S) FOR PERIOD FROM: 18:40:18 12/18/1983 TO 8: 4:38 12/12/1983

FIGURE 8

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
○	851	DEGF	ABOSRBER 5° FROM BOTTOM	5587	977.8	3.605	947.8	2	989.4	1
□	852	DEGF	ABSORBER 15° FROM BOTTOM	5587	1014.	10.78	975.0	2	1061.	2
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	5587	1048.	7.836	1016.	1	1068.	1
▲	854	DEGF	ABSORBER 35° FROM BOTTOM	5587	1078.	10.95	1006.	3	1099.	2
▼	856	DEGF	ABSORBER 45° FROM BOTTOM	5587	1068.	8.988	1005.	1	1086.	6



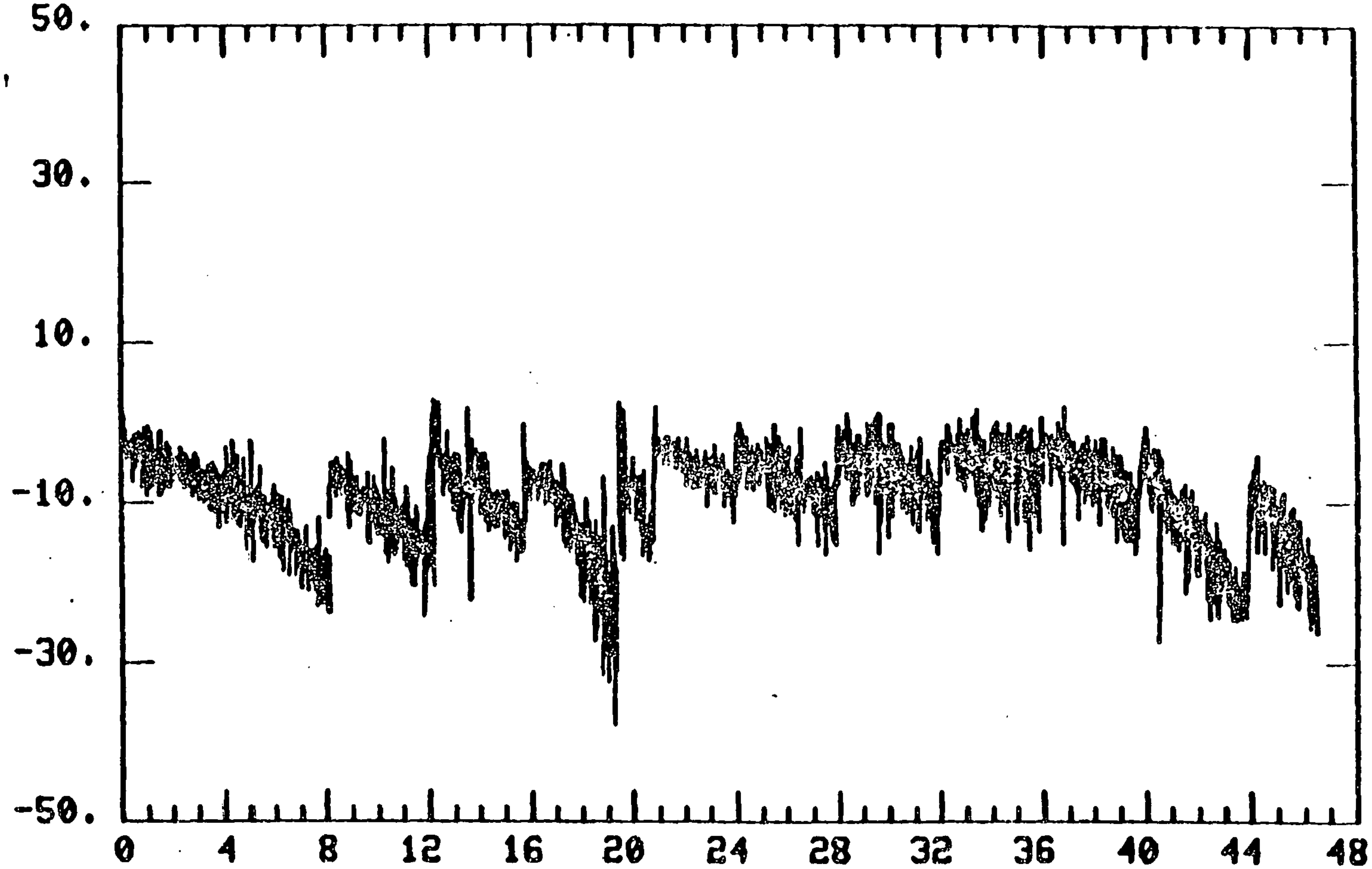
START TIME OF PLOT 21:39: 0 12/ 8/1983
STOP TIME OF PLOT 20:15: 0 12/10/1983

TEST 020

REACTOR TEMPERATURES

FIGURE 9

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
204	"H2O	ABSORBER DIFFERENCE PRESSURE	5613	-9.301	5.808	-37.81	1	2.851	1



TIME (HOURS)

START TIME OF PLOT 21:39: 0 12/ 8/1983

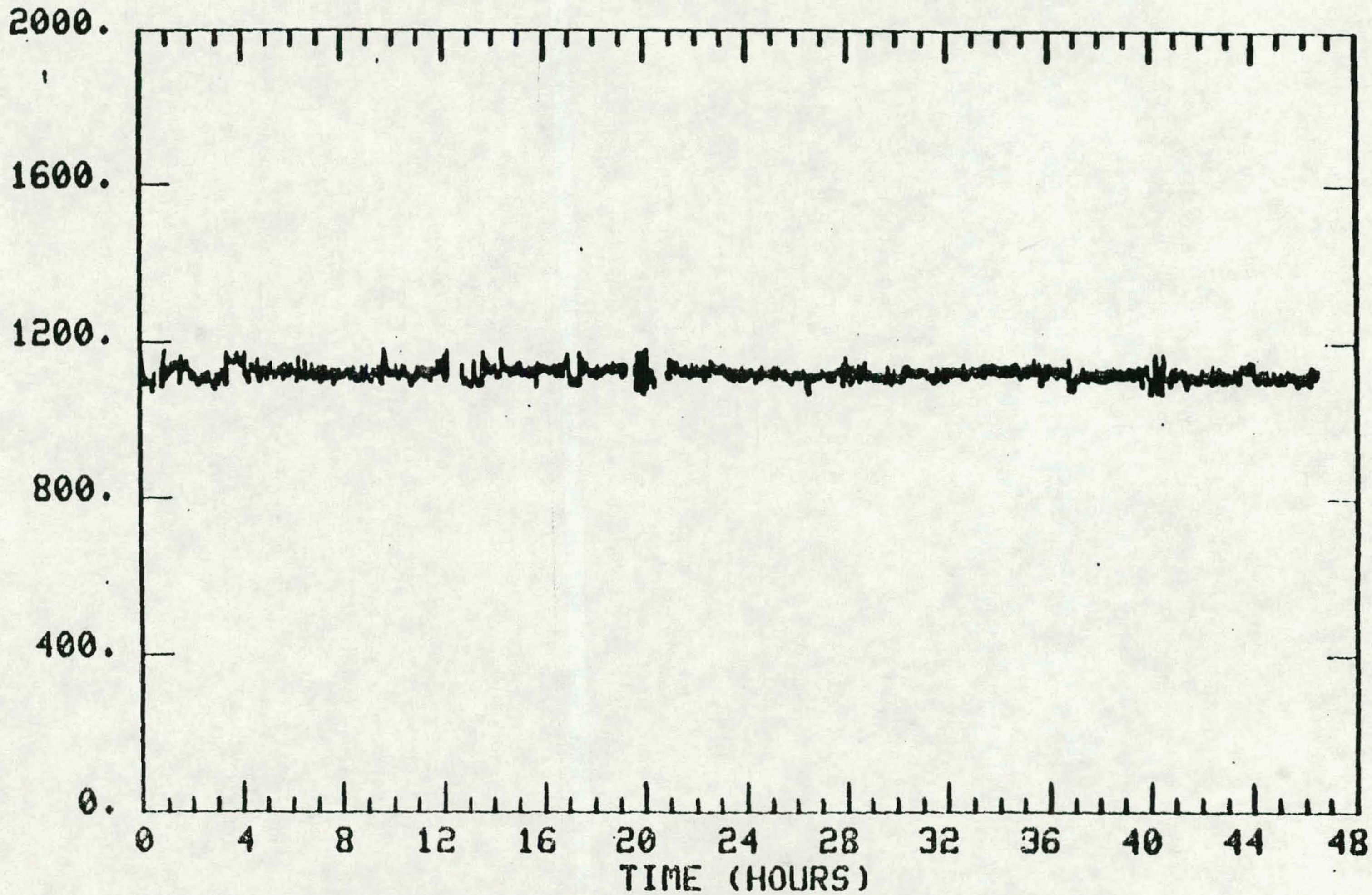
STOP TIME OF PLOT 20:15: 0 12/10/1983

TEST 020

REACTOR PRESSURE DROP

FIGURE 10

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
3750	SCFH	PROD. GAS OUT OF SYSTEM	5318	1185.	14.90	1068.	1	1187.	1



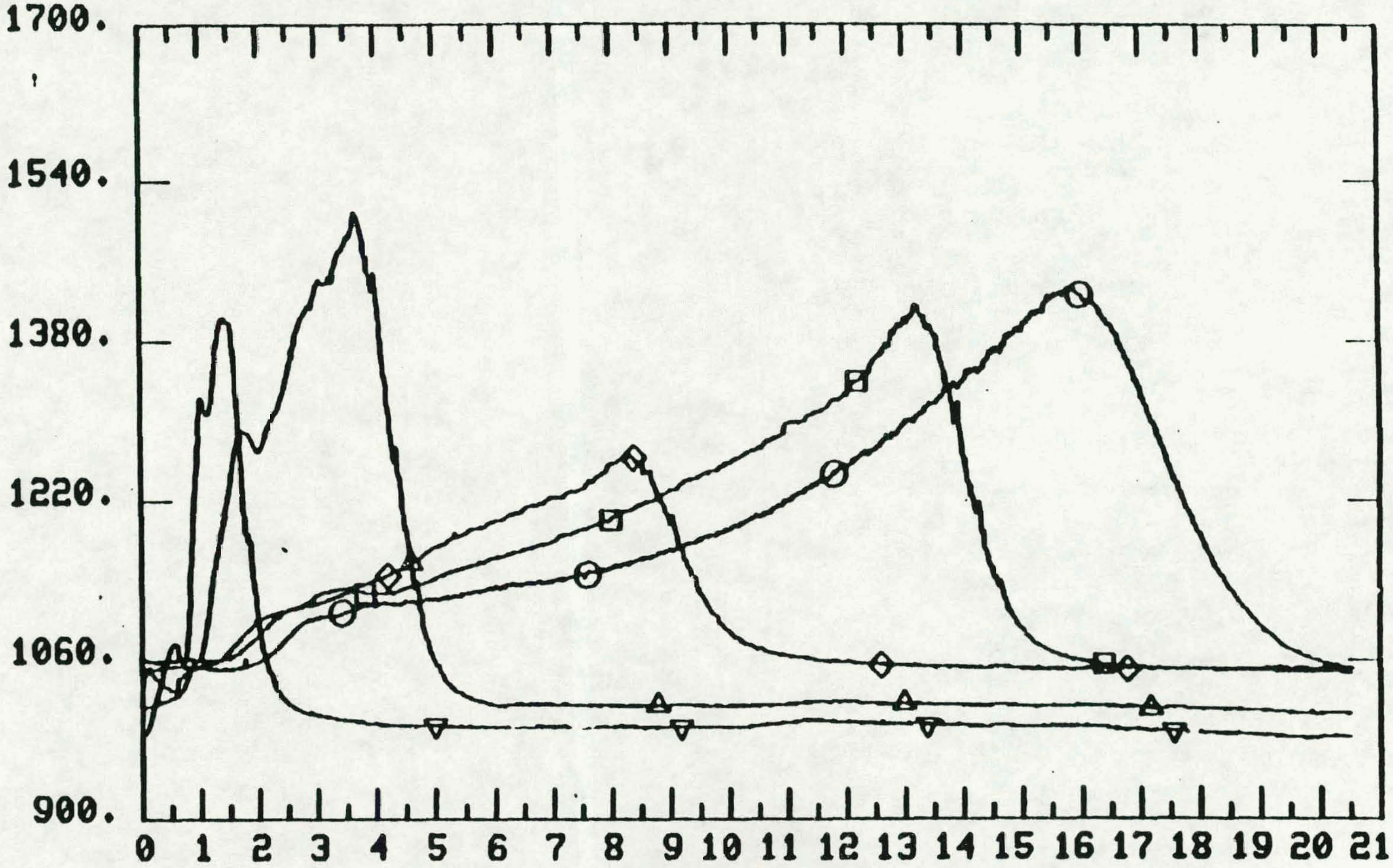
START TIME OF PLOT 21:39:99 12/ 8/1983
STOP TIME OF PLOT 20:15: 0 12/10/1983

TEST 020

EXIT FLOW

FIGURE 11

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
○	851	DEGF	ABSORBER 5° FROM BOTTOM	2489	1189.	118.4	1039.	3	1436.	3
□	852	DEGF	ABSORBER 15° FROM BOTTOM	2489	1159.	103.8	1050.	8	1418.	1
◇	853	DEGF	ABSORBER 25° FROM BOTTOM	2489	1104.	67.81	1047.	11	1267.	2
△	854	DEGF	ABSORBER 35° FROM BOTTOM	2489	1073.	129.5	1006.	1	1511.	1
▽	856	DEGF	ABSORBER 45° FROM BOTTOM	2489	1014.	72.48	981.5	1	1406.	2



TIME (HOURS)

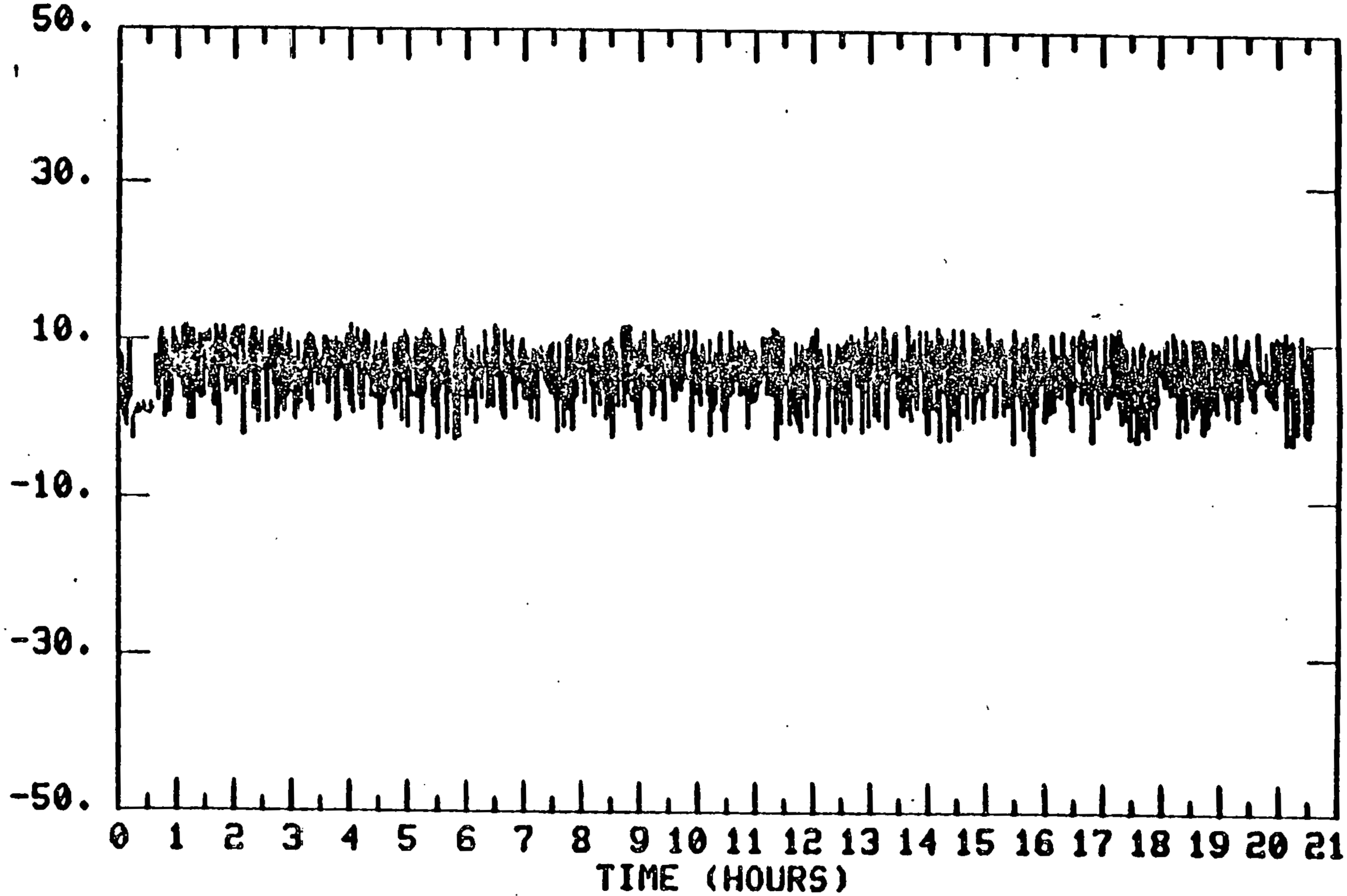
START TIME OF PLOT 0:15: 0 12/11/1983
STOP TIME OF PLOT 20:50: 0 12/11/1983

TEST 021

REACTOR TEMPERATURES

FIGURE 12

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
294	"H2O	ABSORBER DIFFERENCE PRESSURE	8117	6.486	3.017	-8.518	3	18.10	1



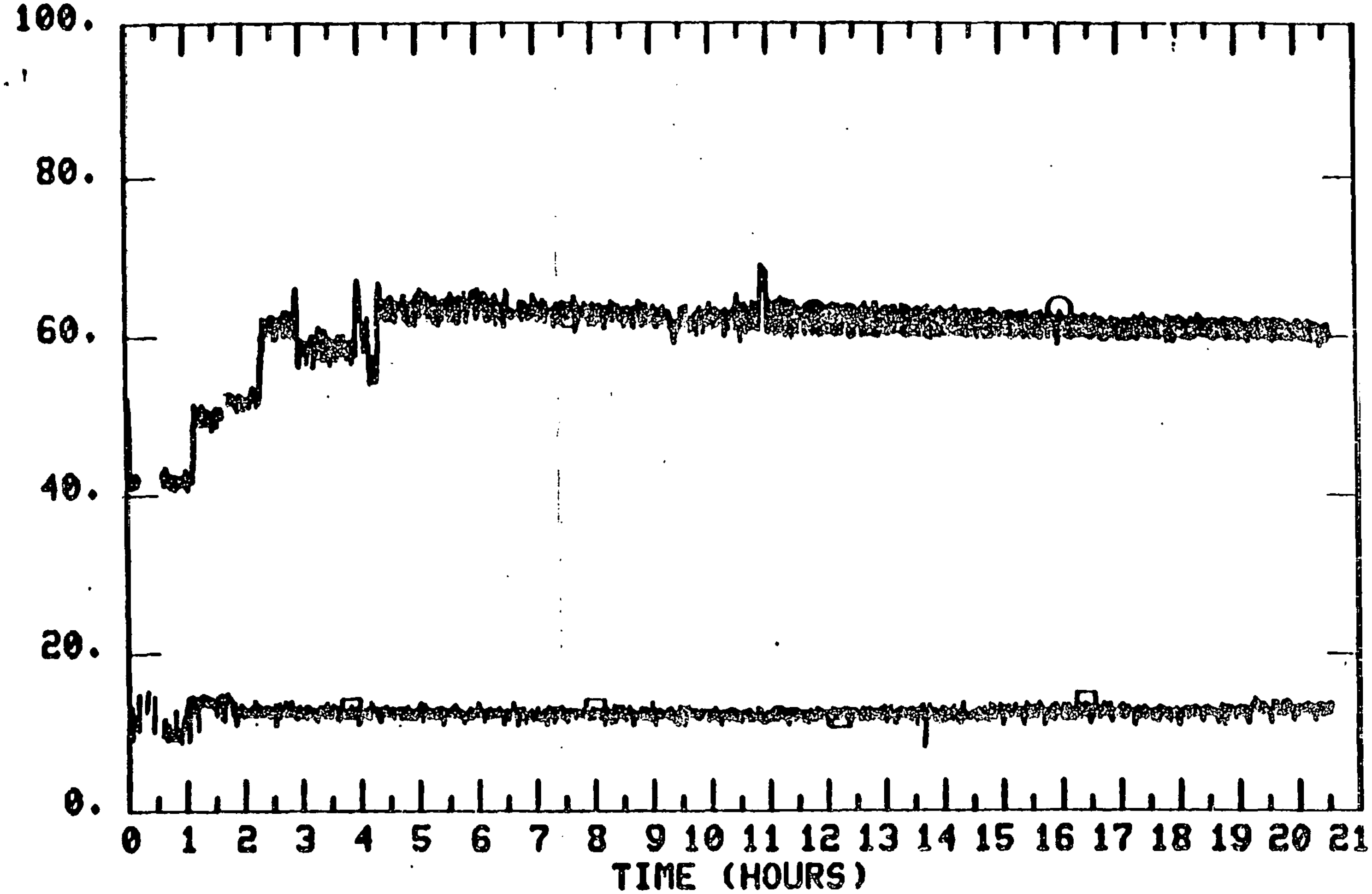
START TIME OF PLOT 0:15: 0 12/11/1983
STOP TIME OF PLOT 20:50: 0 12/11/1983

TEST 021

REACTOR PRESSURE DROP

FIGURE 13

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
O	3758	SCFM	AIR FLOW LOW RANGE	8390	60.78	4.603	40.63	1	69.31	1
□	3754	#/HR	STEAM FLOW	8397	12.47	0.7569	8.850	1	15.34	1



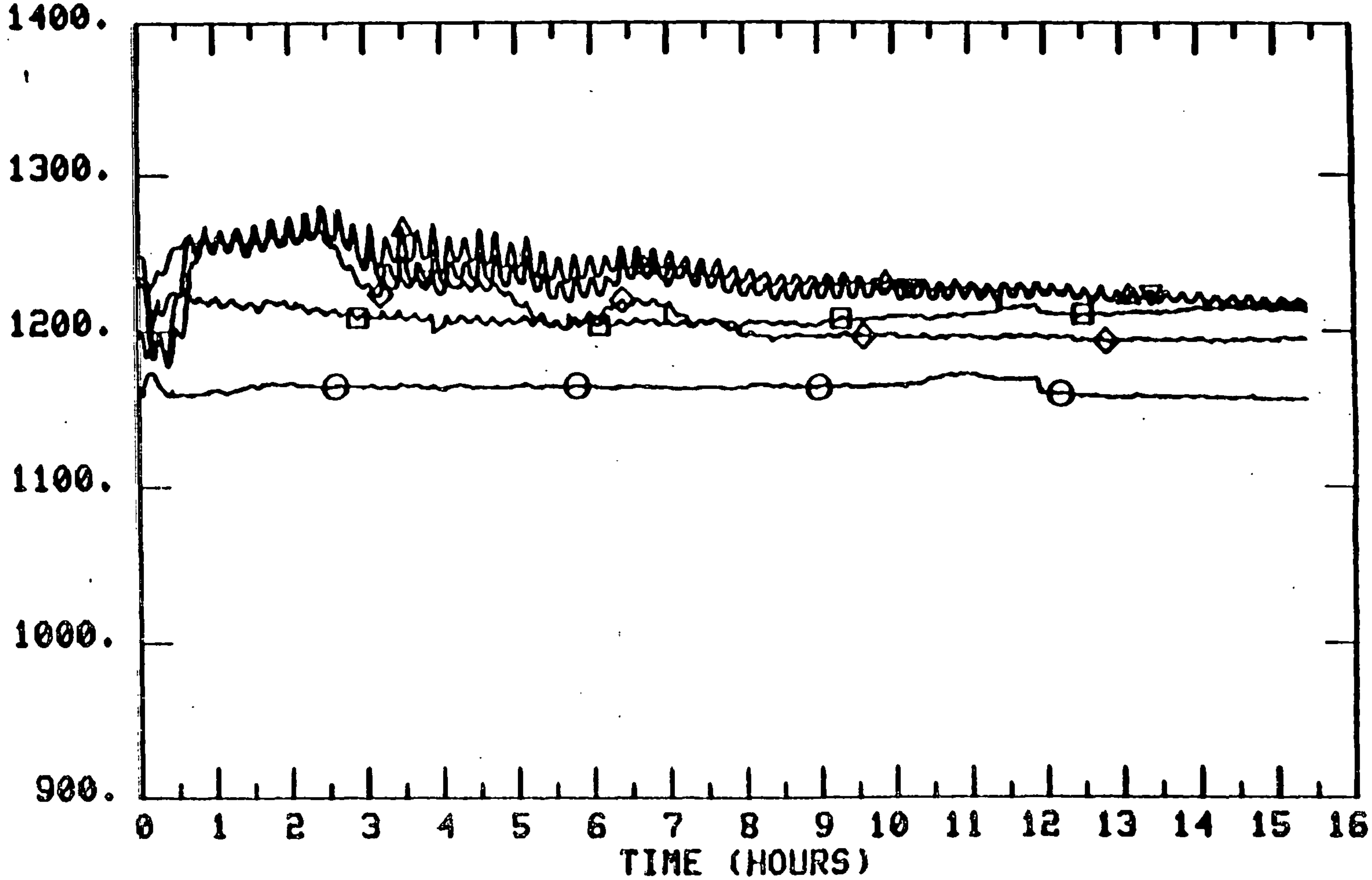
START TIME OF PLOT 0:15: 0 12/11/1983
STOP TIME OF PLOT 20:50: 0 12/11/1983

TEST 021

STEAM AND AIR FLOWS

FIGURE 14

	SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
○	BS1	DEGF	ABSORBER 5° FROM BOTTOM	1851	1164.	3.708	1157.	84	1175.	4
□	BS2	DEGF	ABSORBER 15° FROM BOTTOM	1851	1211.	5.877	1185.	2	1228.	1
◇	BS3	DEGF	ABSORBER 25° FROM BOTTOM	1851	1216.	23.04	1183.	1	1287.	1
▲	BS4	DEGF	ABSORBER 35° FROM BOTTOM	1851	1238.	16.10	1183.	4	1282.	1
▼	BS5	DEGF	ABSORBER 45° FROM BOTTOM	1851	1233.	15.30	1179.	1	1278.	2



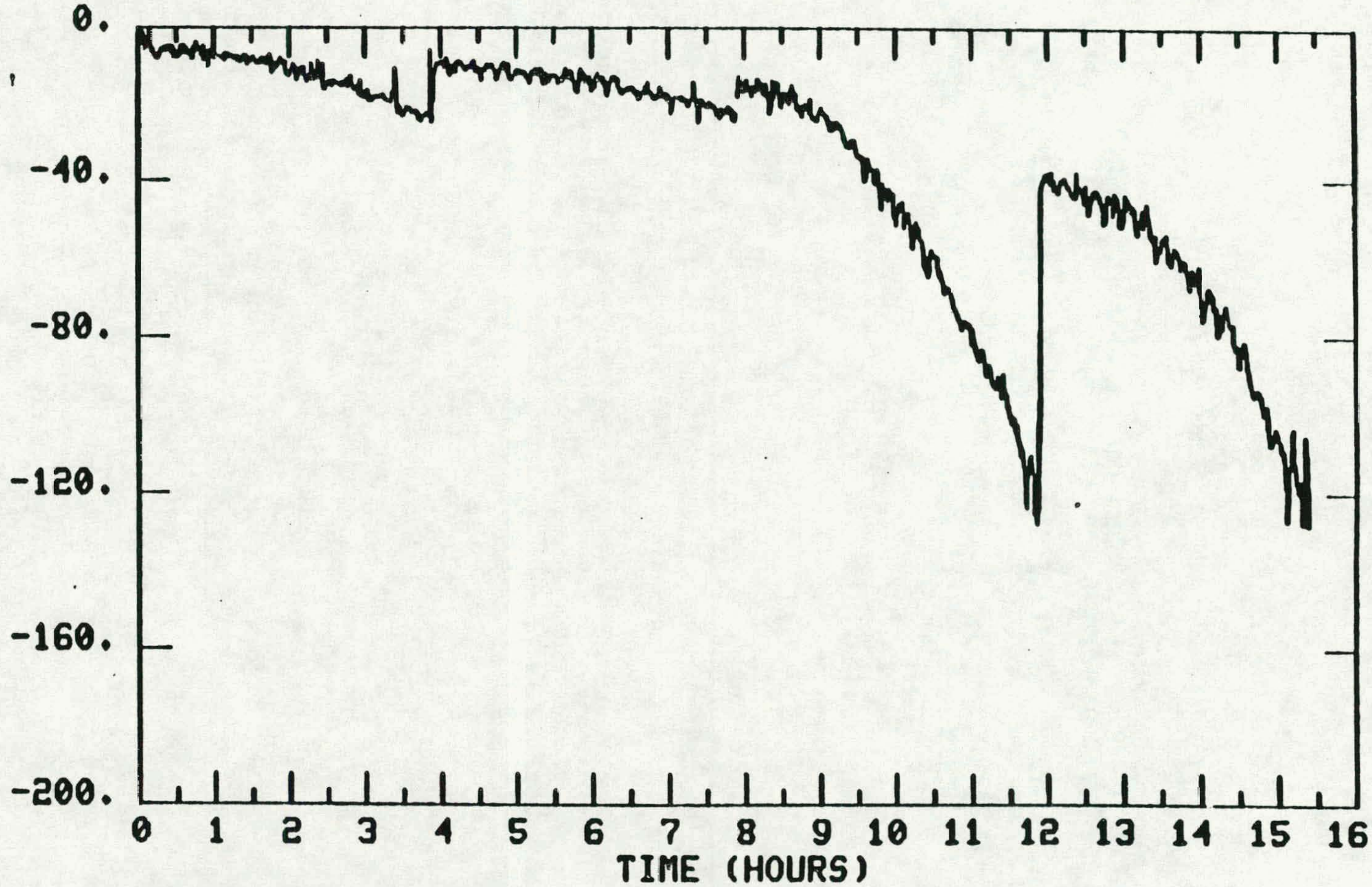
START TIME OF PLOT 16:40: 0 12/12/1983
STOP TIME OF PLOT 8: 5: 0 12/13/1983

TEST 022

REACTOR TEMPERATURES

FIGURE 15

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
804	"H2O	ABSORBER DIFFERENCE PRESSURE	1848	-34.96	30.84	-128.3	1	-0.1454	1



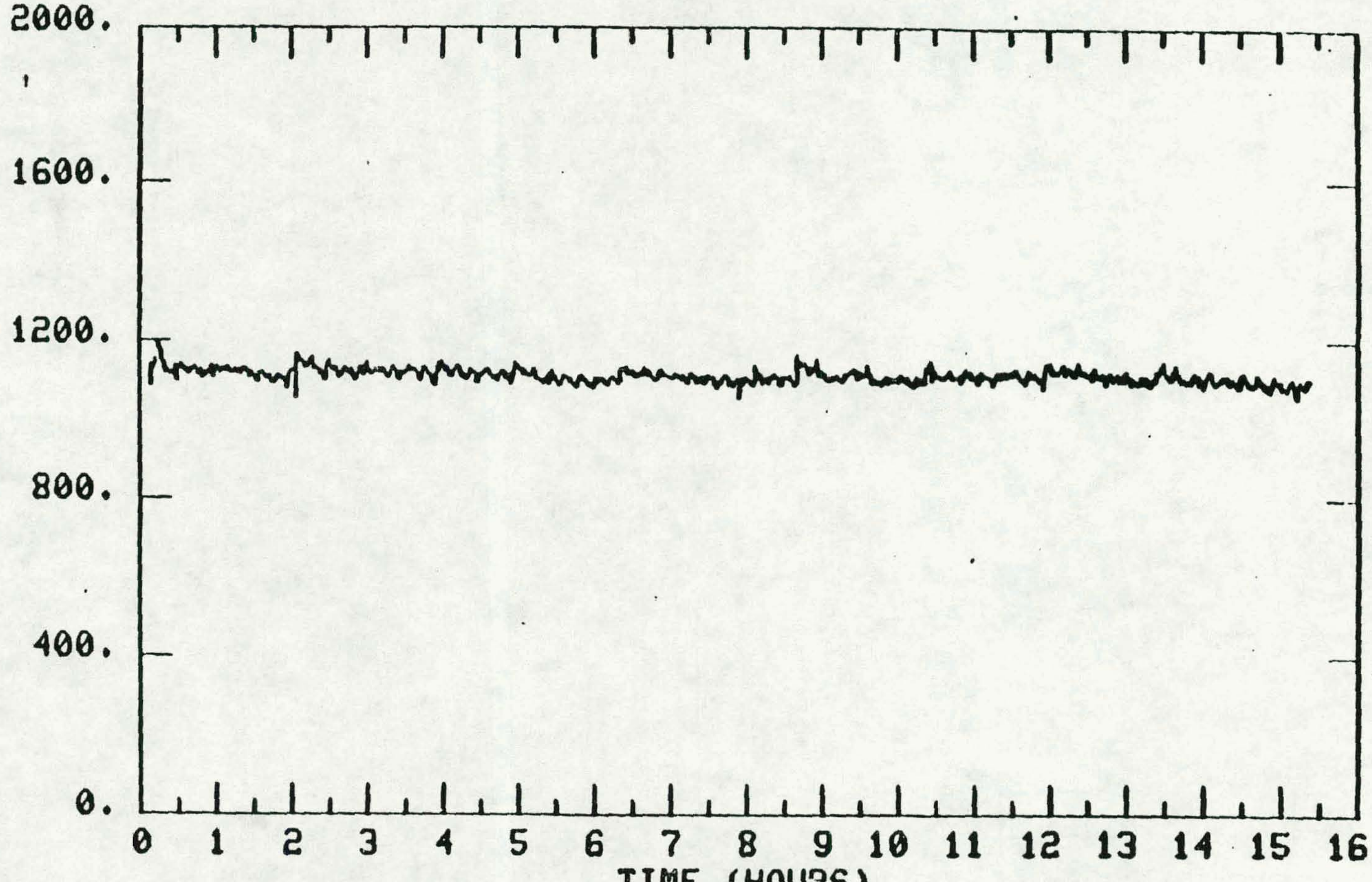
START TIME OF PLOT 16:40: 0 12/12/1983
STOP TIME OF PLOT 8: 5: 0 12/13/1983

TEST 022

REACTOR PRESSURE DROP

FIGURE 16

SLOT	UNITS	DESCRIPTION	# SAMPLES	MEAN	ST DEV	MIN - FREQ		MAX - FREQ	
3750	SCFH	PROD. GAS OUT OF SYSTEM	1827	1116.	15.40	1057.	1	1200.	1



START TIME OF PLOT 16:40: 0 12/12/1983
STOP TIME OF PLOT 8: 5: 0 12/13/1983

TEST 022

EXIT FLOW

FIGURE 17

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT

TEST NO. : 020
DATE STARTED : 21:39 12/8/83
DATE ENDED : 20:1[illegible] 12/10/83 (WITH INTERUPTIONS)
TOTAL HOURS : 45.1
TYPE : SULFIDATION
SULFUR REMOVED: 8.56 LB SB

PURPOSE
SULFIDATION ON LIGNITE DERIVED GAS.

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

SORBENT ANALYSIS:

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

GASIFIER TYPE : DOE/METC FIXED BED
COAL TYPE : LIGNITE

STEAM/COAL MASS RATIO: 0.310 (WEIGHTED AVERAGE)
STEAM/AIR MASS RATIO : 0.200 (WEIGHTED AVERAGE)

HGD OPERATING CONDITIONS

TEMPERATURE: 1040 °F (WEIGHTED AVERAGE)
PRESSURE : 200 PSIG (WEIGHTED AVERAGE)

SPACE VELOCITY: 2000 HOURLY

EXIT SULFUR DATA:

H_2S : 12 PPM INITIALLY
C_2H_4S:
SO_2 : PPM INITIALLY

	INLET MOLE% DRY BASIS (OIL FREE)	INLET MOLE% WET BASIS (OIL FREE)	EXIT MOLE% DRY BASIS (OIL FREE)	EXIT MOLE% WET BASIS (OIL FREE)
H_2S	0.2025			
H_2O				19.4
CO_2	14.29		21.51	
CO	16.55		6.45	
H_2	23.01		27.48	
CH_4	2.70		2.15	
N_2	45.67		45.88	
O_2	0.28		0.64	
C_2H_6	0.13		0.15	
FLOW:				
TOTAL LB MOLES:				
FLOW RATE:	1119 SCFH			1388 SCFH

CONDENSATE	LIGNITE		
AQUEOUS	: 576.5	LBS	(MEASURED)
HYDROCARBON	: 26.6	LBS	(MEASURED)
TOTAL	: 603.2	LBS	(MEASURED)

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHER CS-346 1/2 S SR-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 7/8 INCHES
3. NO FLOW TO COMBUSTOR.
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTE[illegible]
5. GAS COMPOSITIONS AT INLET OBTAINED BY AVERAGE OF BENDIX GC 7000 #4 DATA SAMPLING AT S-4 GASIFIER CYCL[illegible] EXIT.
6. GAS COMPOSITIONS AT EXIT OBTAINED BY AVERAGE OF BENDIX GC 6000 #2 DATA, SAMPLING AT HGD EXIT.
7. REACTOR PRESSURE DROP REACHED A MAXIMUM OF 32 INCHES OF WATER.
8. LIGNITE OPERATION RESULTS IN HIGHER MOISTURE CONTENT OF GAS DUE TO MOISTURE IN LIGNITE.

CONCLUSIONS:

1. SULFUR REMOVAL RESULTS IN 19 % AVERAGE SULFUR LOADING.
2. EXIT SULFUR HIGHER DUE TO HIGH WATER CONTENT OF GAS (19.4 %) , AND EQUILIBRIUM LIMITATIONS.

FIGURE 18

TEST SUMMARY SHEET
SIDESTREAM TEST UNIT

TEST NO. : 021
DATE AND TIME STARTED : 00 15 12/11/83
DATE AND TIME ENDED : 20 50 12/11/83
TOTAL HOURS : 20.33
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: 1024.2 F

PRESSURE : 3.4 PSIG (AVERAGE)

SPACE VELOCITY: 573 HOURLY

			INLET MOLE% WET BASIS	EXIT MOLE% DRY BASIS
AIR	59.51	SCFH	18.55 %	
H_2O	12.41	#/HR	81.45 %	
N_2	0.	SCFH		

TOTAL LB MOLES:
FLOW RATE : 321 SCFH

PURPOSE

REGENERATION OF SULFIDED SORBENT FROM T020 GASIFIIER RUN :

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:

H_2S :

SO_2 : HIGH OF 7.2 % AFTER 18.6 HOURS BY SO_2 MONITOR
0.6 % PPM AT END OF RUN .

CONDENSATE
AQUEOUS : 246 LBS
HYDROCARBON :
TOTAL : 246 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-10789 UNITED CATALYSTS
2. SORBENT PACKED VOLUME = 0.559 CUBIC FOOT
3. NO CHANGE IN PRESSURE DROP, POSSIBLE INDICATION THAT LITTLE CARBON WAS DEPOSITED DURING LIGNITE SULFIDATION.
4. MAXIMUM TEMPERATURE REACHED WAS 1511 F.
5. EXIT BENDIX GC WAS OPERATING AND PROVIDED O2 BREAKTHROUGH CURVE, AND H2,CO,CO2,H2S COMPOSITIONS AT EXIT.
6. 15 MINUTE INTERRUPTION IN REGENERATION.
7. AIR FLOW INCREASED FROM 42 TO 60 SCFH AFTER 2 HOURS TO ACCELERATE THE REGENERATION.

CONCLUSIONS:
1. REGENERATION COMPLETED.

FIGURE 19

TEST SUMMARY SHEET

HOT GAS DESULFURIZATION SIDESTREAM TEST UNIT

TEST NO. : 022
DATE STARTED : 16 40 12/12/83
DATE ENDED : 08 05 12/13/83
TOTAL HOURS : 15.41
TYPE : SULFIDATION
SULFUR REMOVED: 8.01 LB S8

PURPOSE
SULFIDATION AT 1200 F.

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

SORBENT ANALYSIS:

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

GASIFIER TYPE : DOE/METC FIXED BED
COAL TYPE : ARKWRIGHT

STEAM/COAL MASS RATIO: 0.507 (AVERAGE)
STEAM/AIR MASS RATIO : 0.210 (AVERAGE)

HGD OPERATING CONDITIONS
TEMPERATURE: 1213 F (AVERAGE)
PRESSURE : 124.1 PSIG (AVERAGE)

SPACE VELOCITY: 2000 HOURLY

EXIT SULFUR DATA:
H_2S : 260 PPM INITIALLY
C_4H_4S: 2-4.1 PPM
SO_2 : 0 PPM INITIALLY

CONDENSATE ARKWRIGHT COAL

AQUEOUS	: 63.5	LBS	(ESTIMATED)
HYDROCARBON	: 9.3	LBS	(ESTIMATED)
TOTAL	: 72.8	LBS	(MEASURED)

	INLET MOLE% DRY BASIS (OIL FREE)	INLET MOLE% WET BASIS (OIL FREE)	EXIT MOLE% DRY BASIS (OIL FREE)	EXIT MOLE% WET BASIS (OIL FREE)
H_2S	0.577			
H_2O				7.2 %
CO_2	9.95		13.62	
CO	19.55		15.06	
H_2	20.82		24.90	
CH_4	3.16		3.06	
N_2	47.10		47.34	
O_2	0.28		1.00	
C_2H_6	0.29		0.39	
FLOW:				
TOTAL LB MOLES:				
FLOW RATE:	1112 SCFH			1198 SCFH

REMARKS
1. PACKED LENGTH IN REACTOR IS 47 5/8 INCHES ZINC FERRITE
BOTTOM AND TOP 6 INCHES ARE 1/2 INCH CERAMIC SPHERE
CS-346 1/2 S SR-10789 UNITED CATALYSTS
2. OVERALL PACKED LENGTH 58 7/8 INCHES
3. NO FLOW TO COMBUSTOR.
4. WATER IN EXIT GAS WAS DETERMINED BY CONDENSATE COLLECTED
5. GAS COMPOSITIONS AT INLET OBTAINED BY AVERAGE
OF BENDIX GC 7000 #4 DATA SAMPLING AT S-4 GASIFIER CYCLO
EXIT.
6. GAS COMPOSITIONS AT EXIT OBTAINED BY AVERAGE
OF BENDIX GC 6000 #2 DATA, SAMPLING AT HGD EXIT.
7. REACTOR PRESSURE DROP REACHED A MAXIMUM OF 128 INCHES
OF WATER.

CONCLUSIONS:

1. SULFIDATION AT 1200 F RESULTED IN MUCH HIGHER H_2S LEVEL THAN EXPECTED.
2. TIME TO BREAKTHROUGH MUCH SHORTER AT 1200 F COMPARED TO 1000 F.
3. AVERAGE SULFUR LOADING IS 17.8 %. NOT MUCH LOWER THAN ON PREVIOUS SULFIDATION ON LIGNITE AT 1000 F.

FIGURE 20

REMOVAL OF HYDROGEN SULFIDE FROM COAL GAS BY ZINC FERRITE

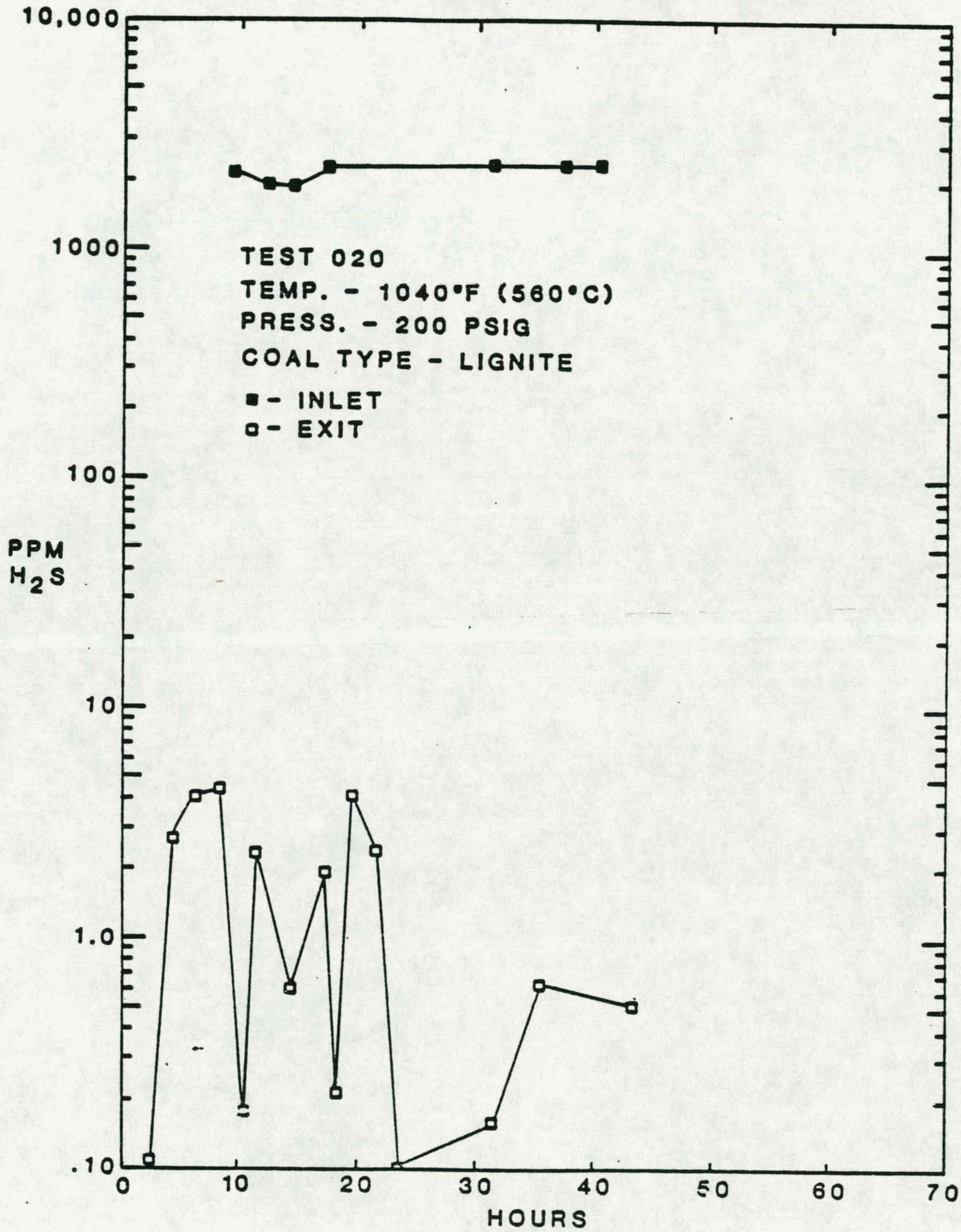


FIGURE 21

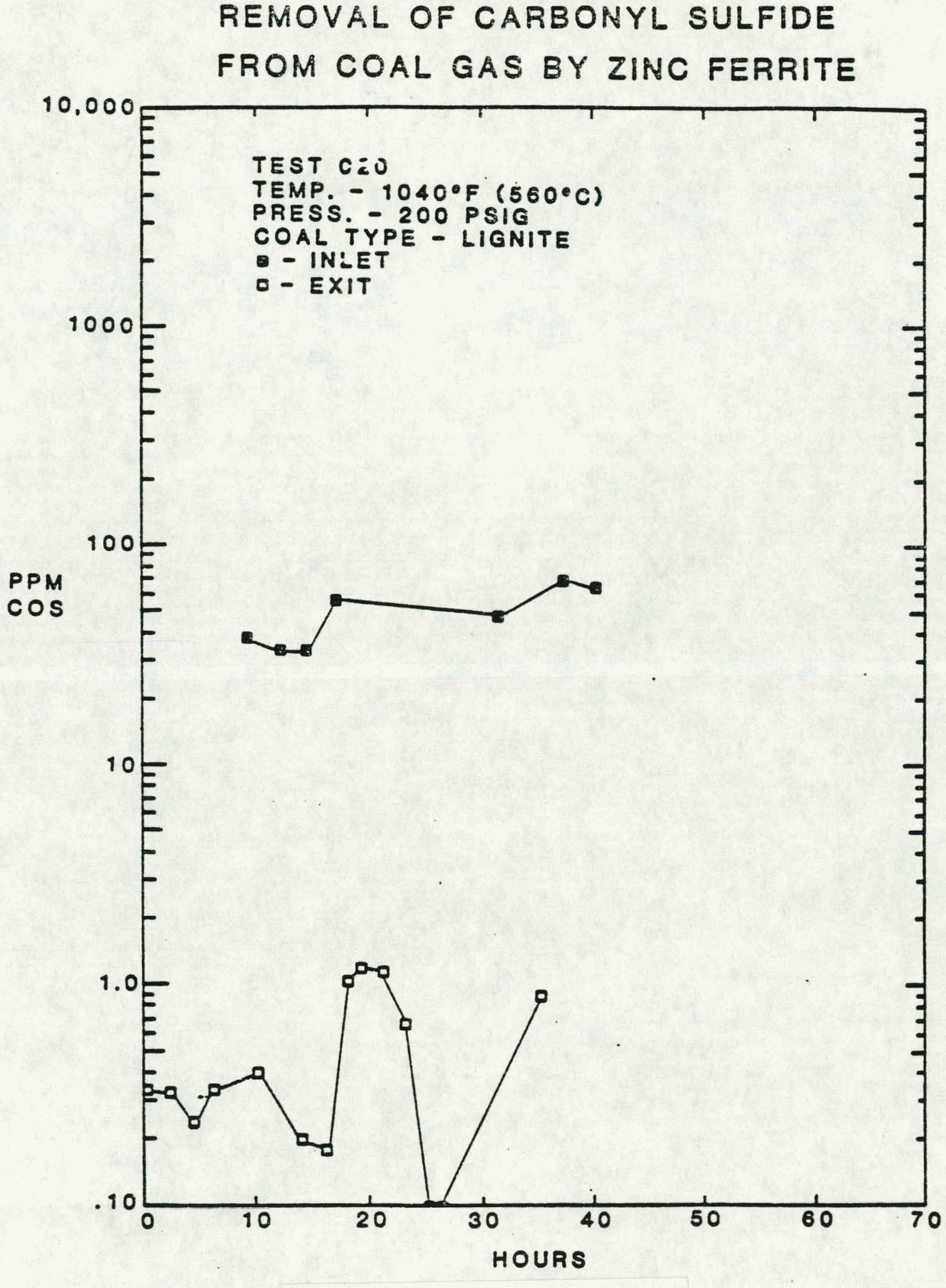

REMOVAL OF CARBONYL SULFIDE
FROM COAL GAS BY ZINC FERRITE
TEST C20
TEMP. - 1040°F (560°C)
PRESS. - 200 PSIG
COAL TYPE - LIGNITE
■ - INLET
□ - EXIT
10,000
1000
100
10
1.0
.10
PPM
COS
0
10
20
30
40
50
60
70
HOURS

FIGURE 22

REMOVAL OF CARBON DISULFIDE FROM COAL GAS BY ZINC FERRITE

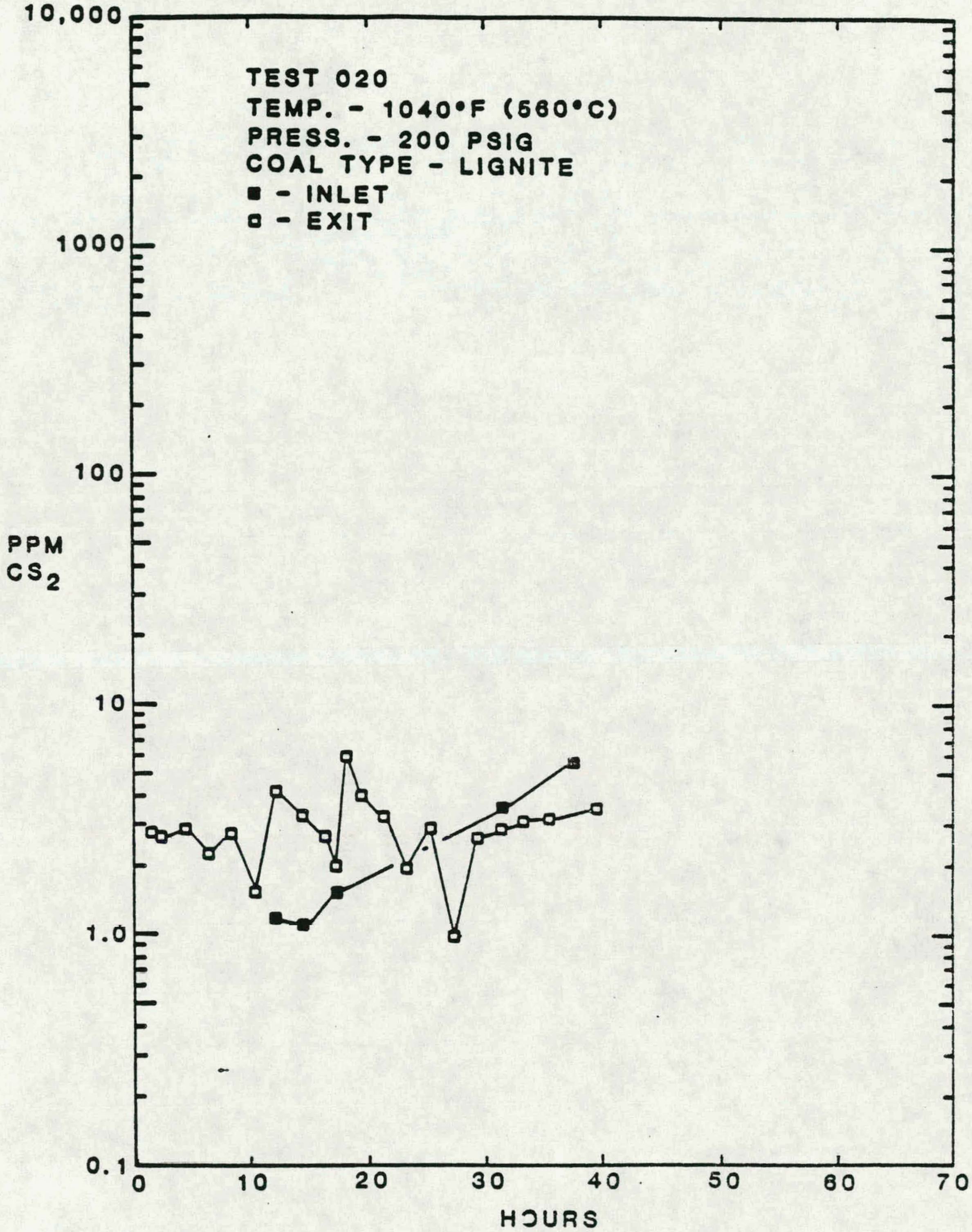


FIGURE 23

REMOVAL OF THIOPHENE FROM COAL GAS BY ZINC FERRITE

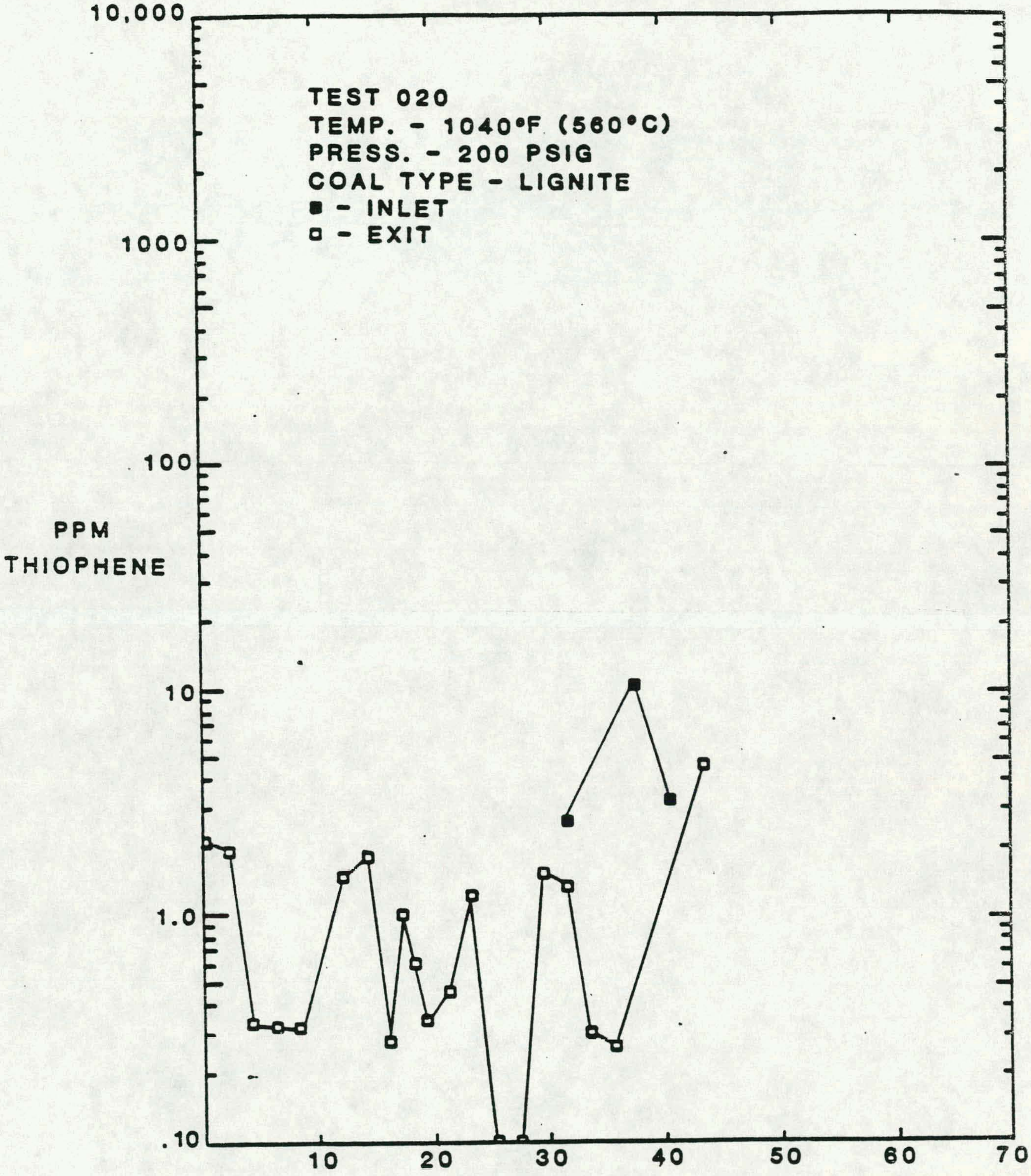
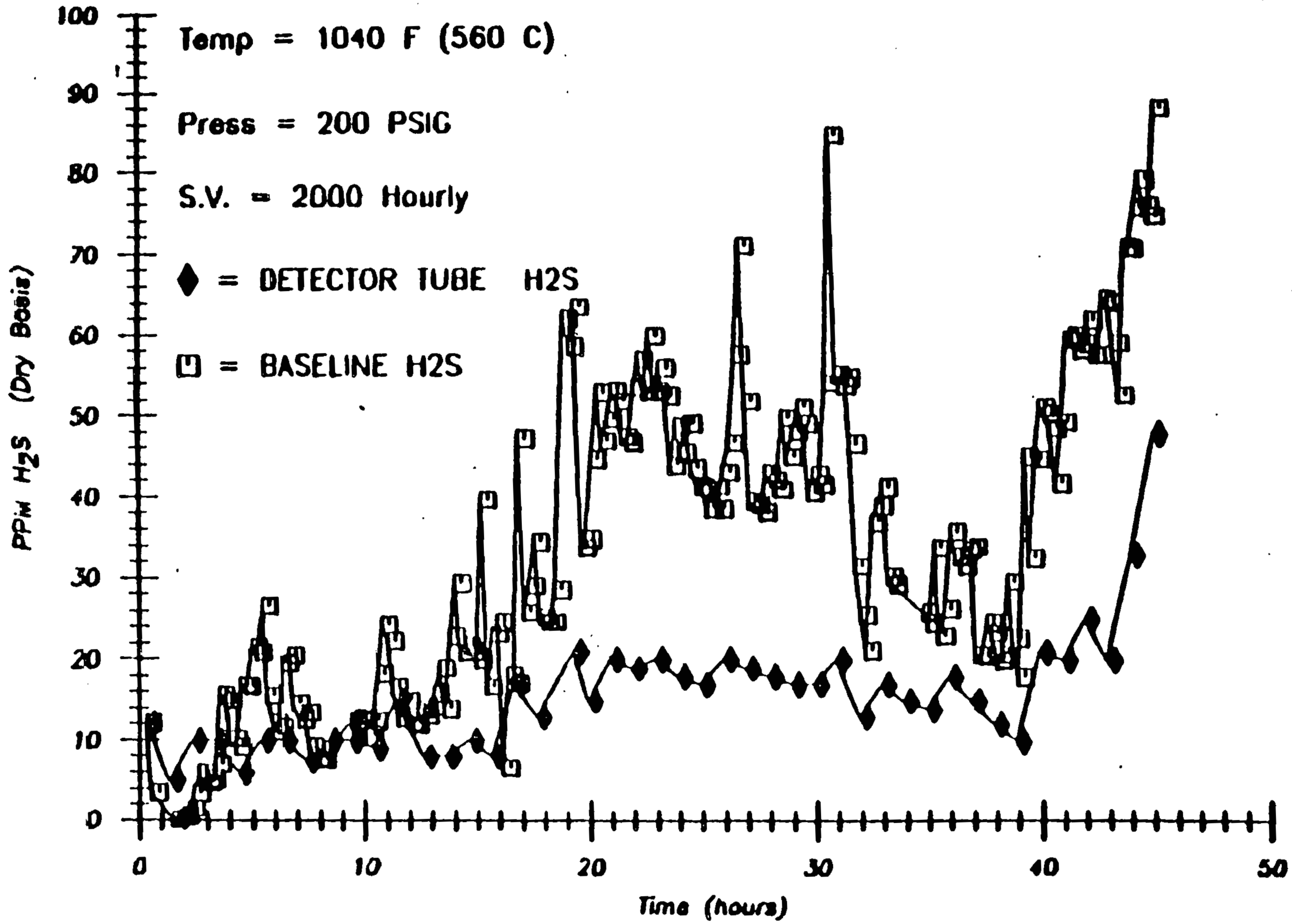


FIGURE 24

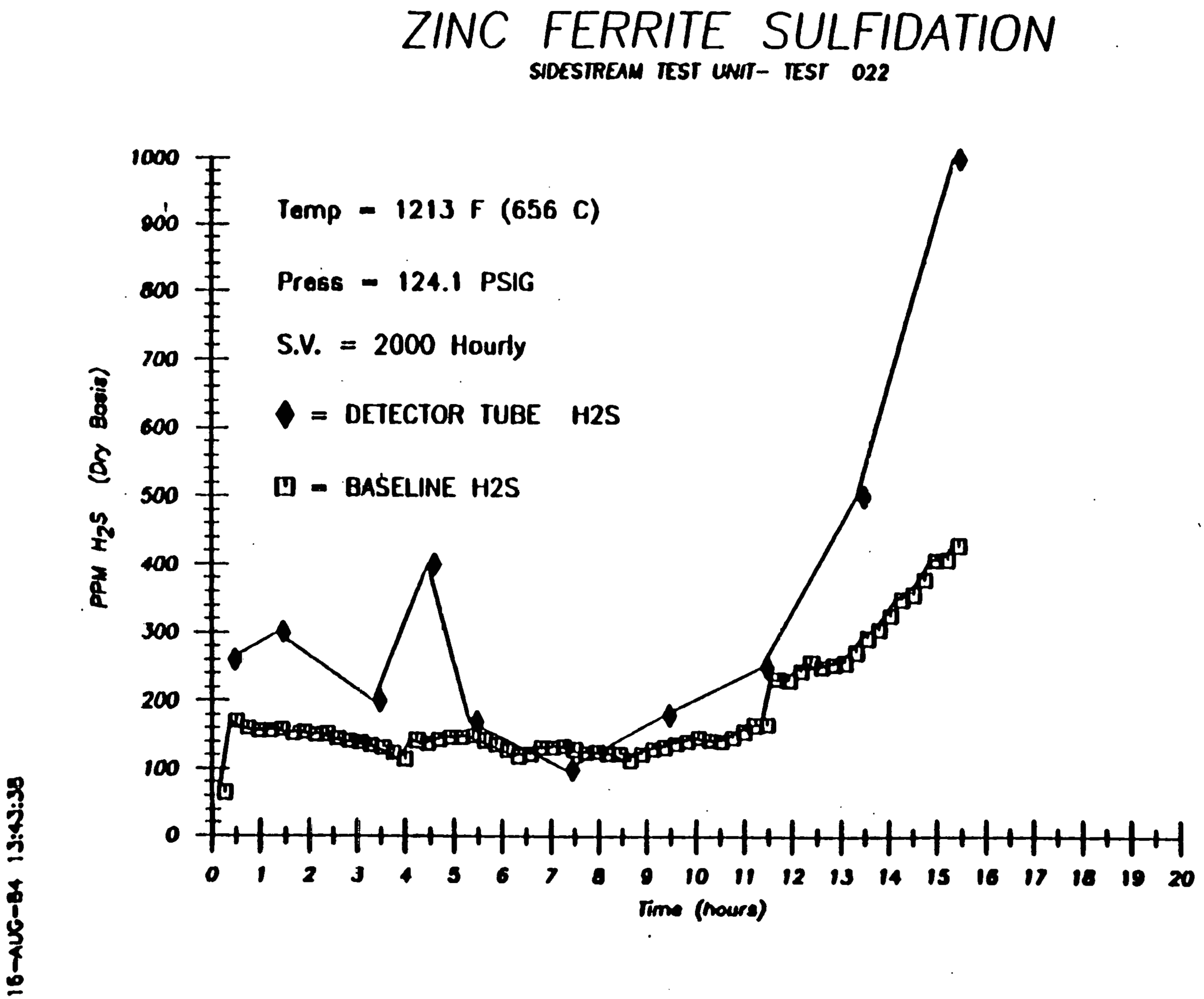
SULFIDATION ON LIGNITE DERIVED GAS

ZINC FERRITE SIDESTREAM TEST UNIT- TEST 020



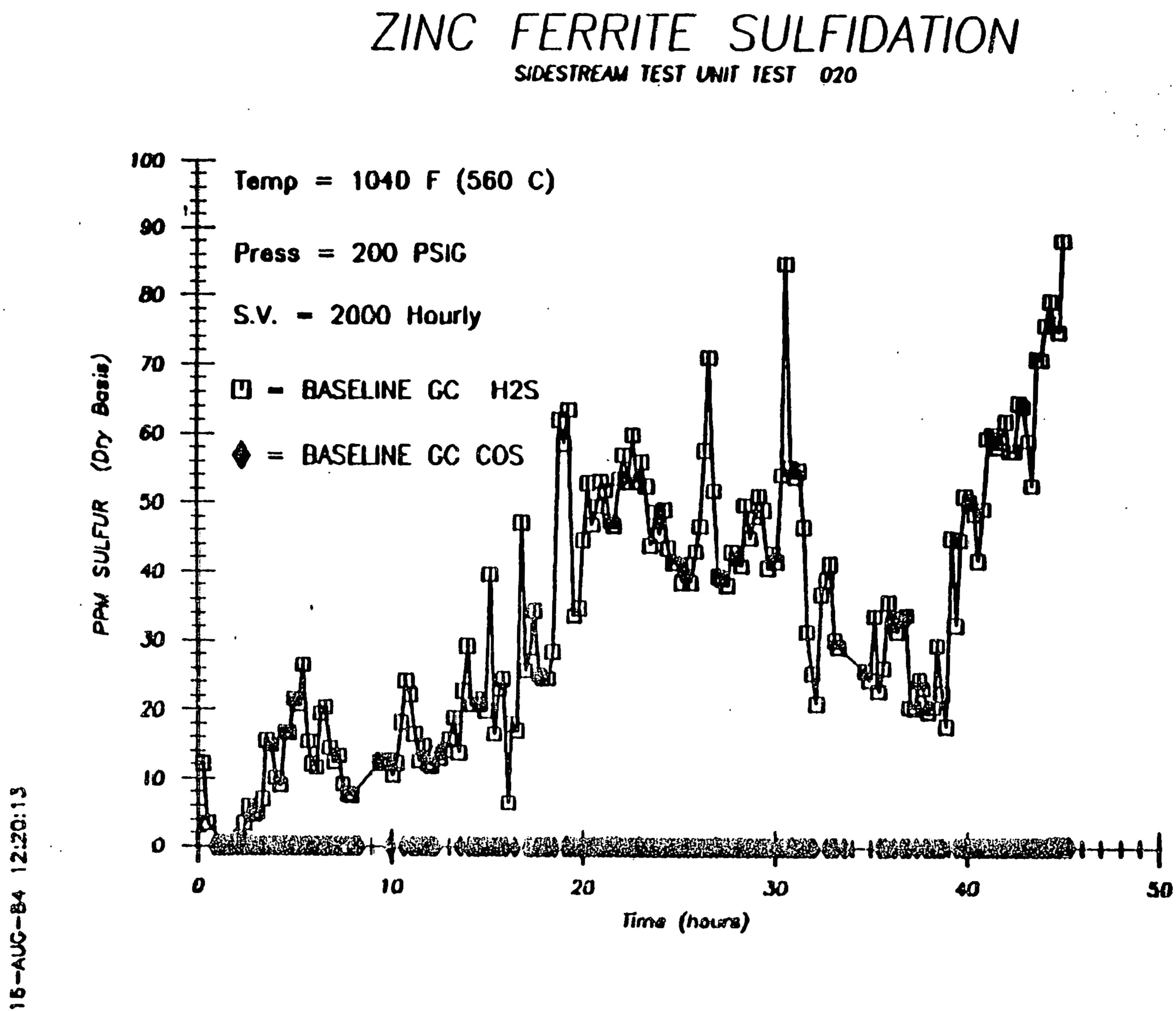
16-AUG-84 10:25:13

FIGURE 25



ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT- TEST 022
Temp = 1213 F (656 C)
Press = 124.1 PSIG
S.V. = 2000 Hourly
= DETECTOR TUBE H2S
= BASELINE H2S
PPM H2S (Dry Basis)
1000
900
800
700
600
500
400
300
200
100
0
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
Time (hours)
16-AUG-84 13:43:38

FIGURE 26



ZINC FERRITE SULFIDATION
SIDESTREAM TEST UNIT TEST 020
Temp = 1040 F (560 C)
Press = 200 PSIG
S.V. = 2000 Hourly
= BASELINE GC H2S
= BASELINE GC COS
PPM SULFUR (Dry Basis)
Time (hours)
0
10
20
30
40
50
60
70
80
90
100
15-AUG-84 12:20:13

FIGURE 27

ZINC FERRITE SULFIDATION

SIDESTREAM TEST UNIT TEST 022

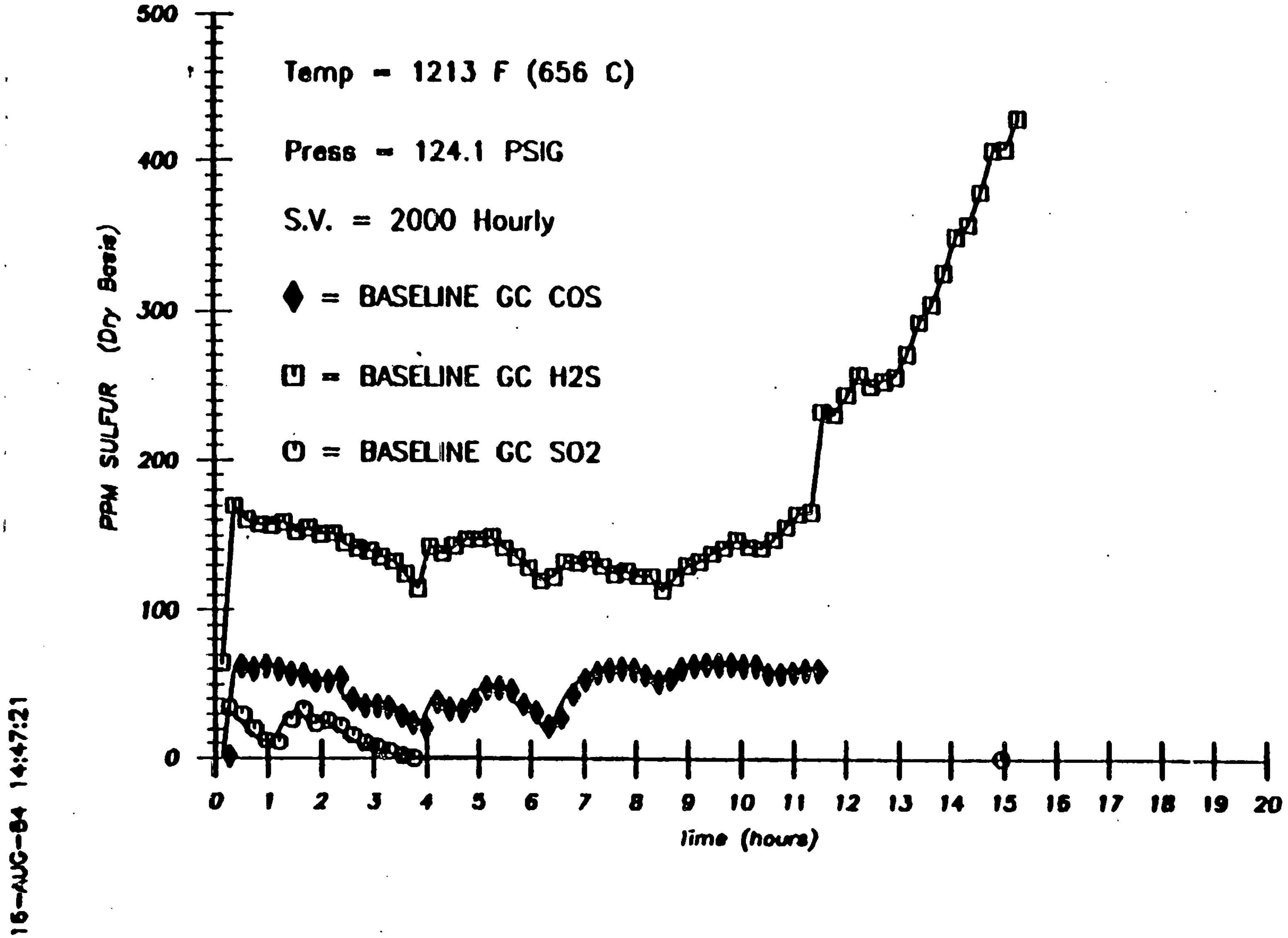
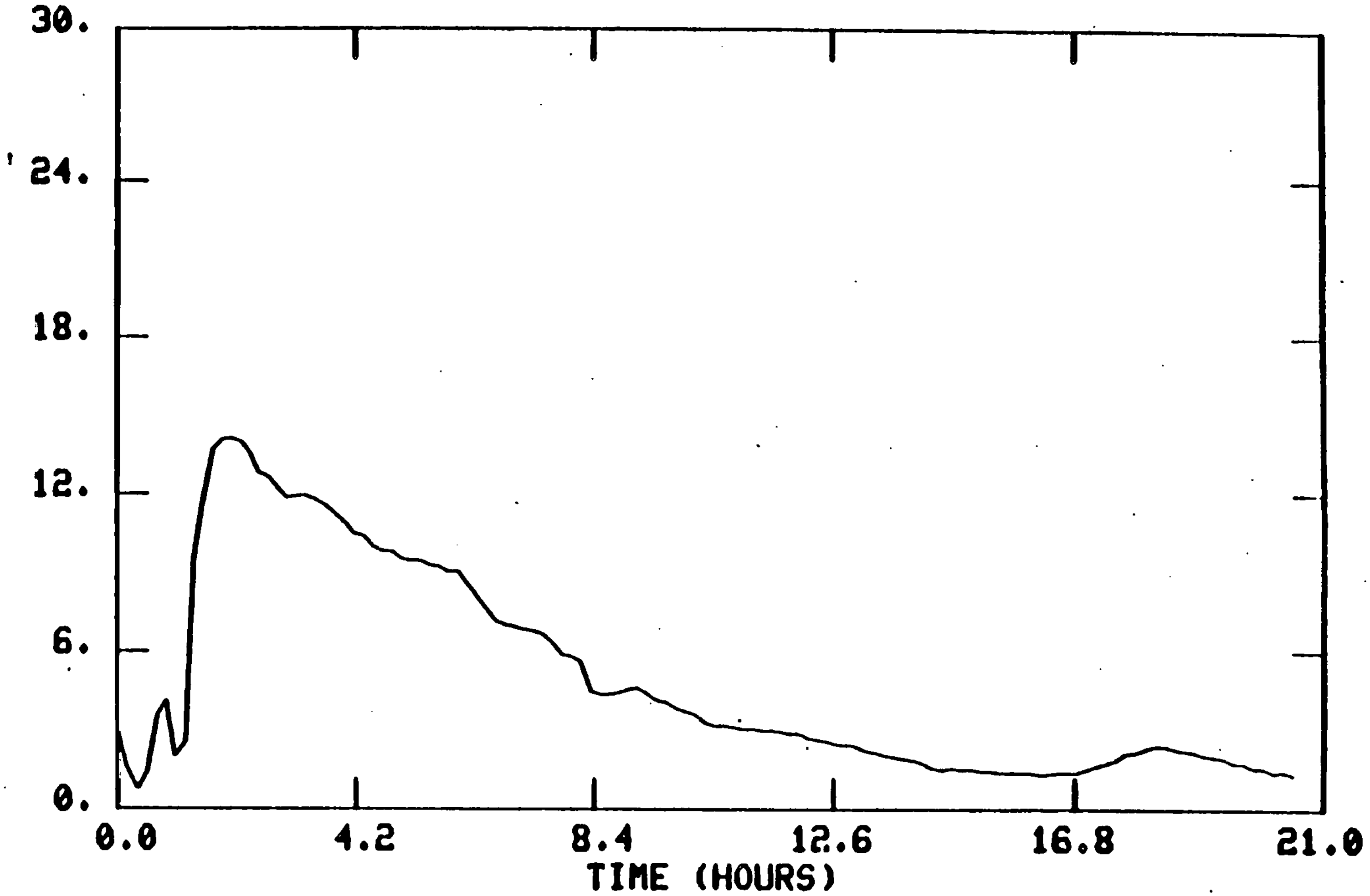


FIGURE 28



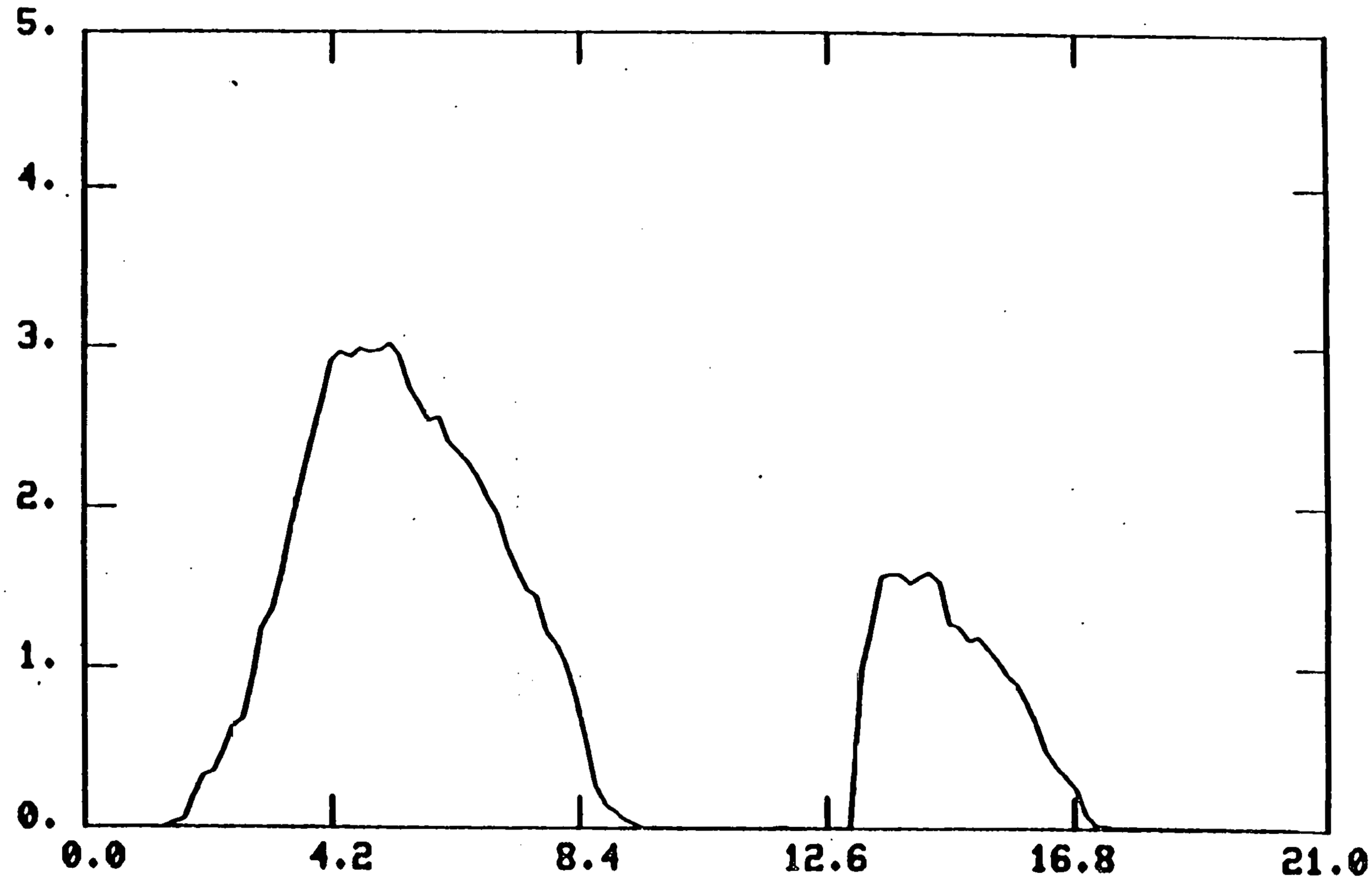
PERCENT CO2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 00:15:00 12/11/1983
STOP TIME 20:50:00 12/11/1983

TEST 021

REGENERATION

CARBON BURNOFF AS CO_2

FIGURE 29



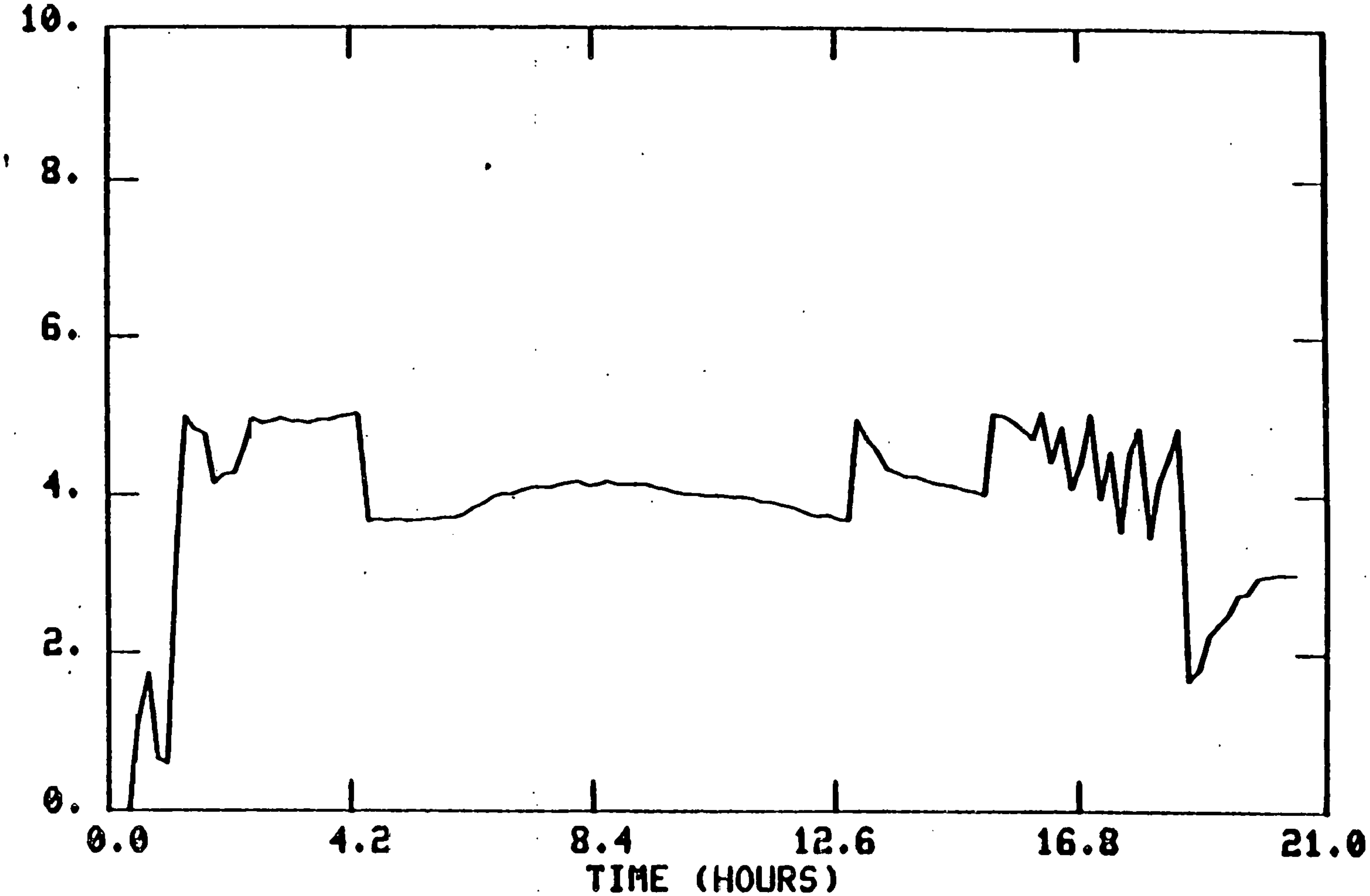
PERCENT H2S
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 00:15:00 12/11/1983
STOP TIME 20:50:00 12/11/1983

TEST 021

REGENERATION

H_2S EFFLUENT

FIGURE 30



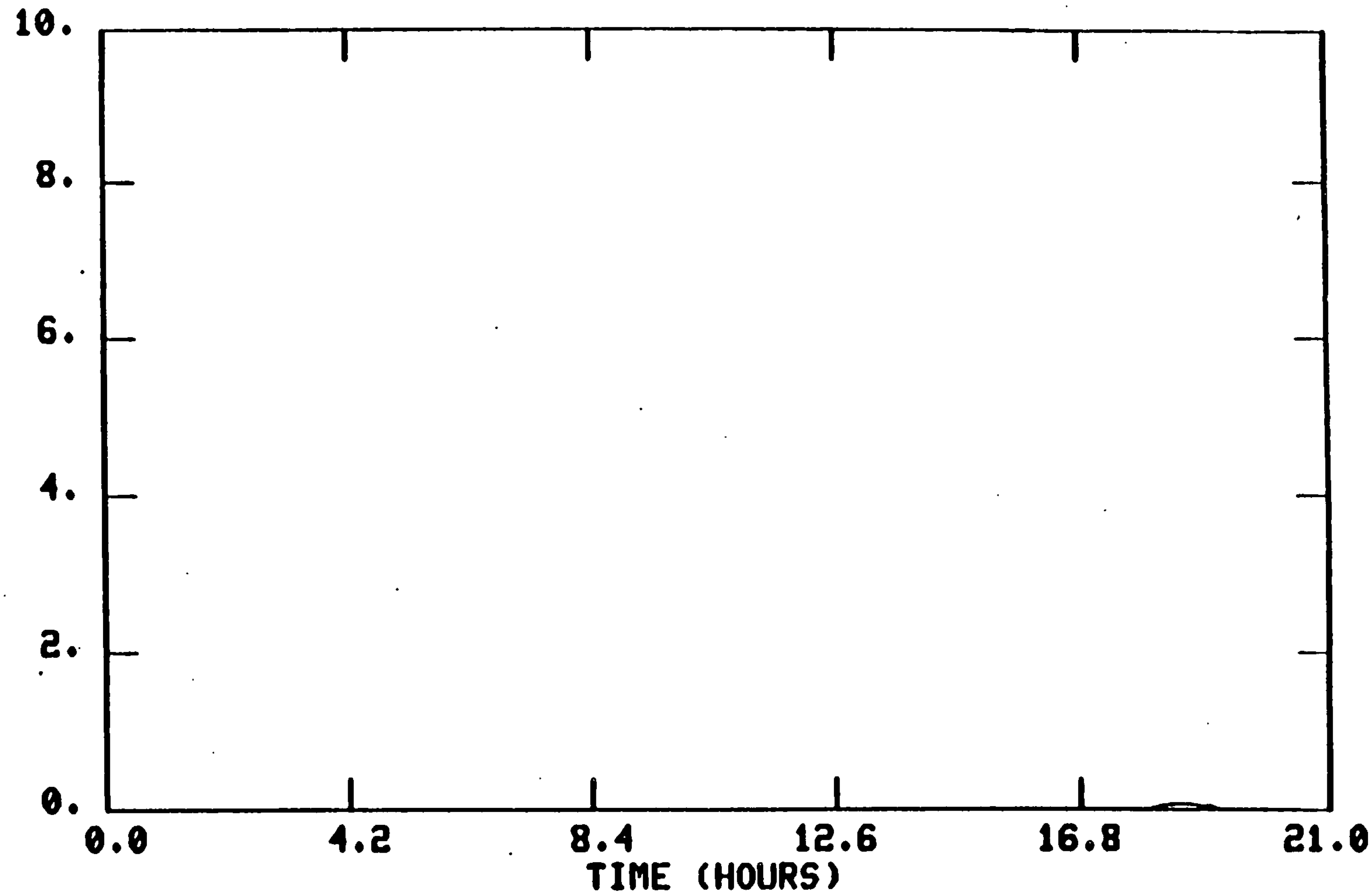
PERCENT O2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 00:15:00 12/11/1983
STOP TIME 20:50:00 12/11/1983

TEST 021

REGENERATION

OXYGEN BREAKTHROUGH CURVE

FIGURE 31



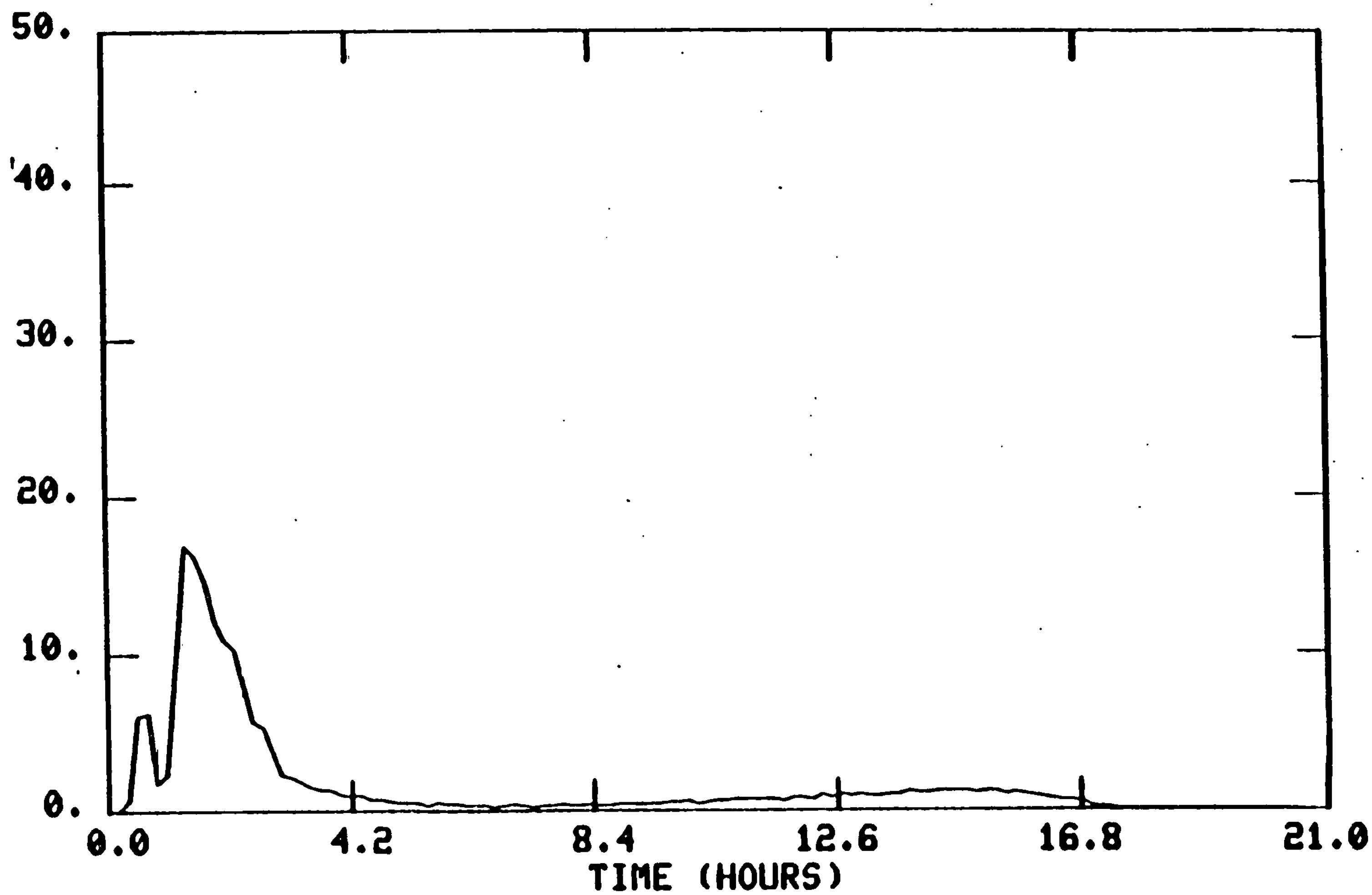
PERCENT CO
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 00:15:00 12/11/1983
STOP TIME 20:50:00 12/11/1983

TEST 021

REGENERATION

CARBON BURNOFF AS CO

FIGURE 32



PERCENT H2
BENDIX 6000 #2 CHROMATOGRAPH
START TIME 00:15:00 12/11/1983
STOP TIME 20:50:00 12/11/1983

TEST 021

REGENERATION

HYDROGEN GENERATION

TABLE 1

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT HGD EXIT
GASIFIER RUN 104
TRACE COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2S	COS	CH_3SH	CS_2 PPM	Thio	NH_3	H_2O	SO_2
SULFI-	HG 1	12/8/83 22:00	ND	0.34	ND	2.76	2.11	ND	4066	ND
DATION	HG 2	12/9/83 00:00	0.12	0.33	ND	2.72	1.98	-	3918	ND
TOZO	HG 3	12/9/83 02:00	2.70	0.24	ND	2.90	0.34	ND	3814	ND
	HG 4	12/9/83 04:00	4.12	0.34	ND	2.23	0.33	-	3848	ND
	HG 5	12/9/83 06:00	4.50	ND	ND	2.71	0.33	ND	3529	ND
	HG 6	12/9/83 08:00	0.18	0.40	ND	1.54	-	-	3638	ND
	HG 7	12/9/83 10:30	2.38	ND	ND	4.23	1.52	ND	3176	0.49
	HG 8	12/9/83 12:30	0.60	0.20	ND	3.32	1.85	-	3406	ND
	HG 9	12/9/83 14:30	ND	0.18	ND	2.69	0.28	ND	2606	ND
	HG 10	12/9/83 15:45	1.79	ND	ND	2.00	1.01	-	3330	ND
	HG 11	12/9/83 16:30	0.21	1.07	ND	6.01	0.62	ND	2916	ND
	HG 12	12/9/83 18:45	4.27	1.20	ND	4.11	0.35	-	2550	ND
	HG 13	12/9/83 20:30	2.49	1.15	ND	3.31	0.46	ND	3126	ND
	HG 14	12/9/83 22:30	0.10	0.67	ND	1.98	1.27	-	2980	6.14
	HG 15	12/10/82 00:30	ND	0.10	ND	2.97	0.09	ND	2980	ND
	HG 16	12/10/83 02:30	ND	0.10	ND	0.98	0.07	-	2272	ND
	HG 17	12/10/83 04:30	ND	ND	ND	2.69	1.54	ND	3208	ND
	HG 18	12/10/83 06:30	0.16	ND	ND	2.97	1.35	-	2422	ND

Table 1 (Continued)

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT HGD EXIT
GASIFIER RUN 104
TRACE COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2S	COS	CH_3SH	CS_2 PPM	Thio	NH_3	H_2O	SO_2
SULFI-	HG 19	12/10/83 08:30	ND	ND	ND	3.17	0.31	ND	2748	ND
DATION	HG 20	12/10/83 10:30	0.62	0.89	ND	3.22	0.28	-	2812	2.56
T020	HG 21	12/10/83 14:30	ND	ND	ND	3.51	1587	ND	-	0.87
	HG 22	12/10/83 18:30	0.54	ND	ND	ND	4.84	-	-	ND
	HG 23	12/11/83 02:20	ND	27.56	ND	18.61	3.51	ND	-	ND
REGENER	HG 24	12/11/83 06:20	2.40%	68.95	ND	16.53	ND	-	-	3.00%
ATION	HG 25	12/11/83 10:20	4118	41.86	ND	10.06	ND	ND	-	10.04
T021	HG 26	12/11/83 14:20	687	36.64	ND	7.41	ND	-	-	18.52%
	HG 27	12/11/83 18:20	ND	3.81	ND	4.43	ND	ND	-	12.31%
SULFI-	HG 28	12/12/83 17:15	116.5	40.44	0.37	2.51	1.76	-	-	ND
DATION	HG 29	12/12/83 17:40	113.1	34.98	0.26	2.19	2.34	ND	-	ND
T022	HG 30	12/12/83 21:00	69.82	23.39	ND	1.37	1.98	-	-	ND
	HG 31	12/13/83 01:00	15.12	30.30	ND	0.45	1.50	ND	-	ND
	HG 32	12/13/83 05:00	155.7	50.08	ND	1.98	3.50	-	-	ND
	HG 33	12/13/83 08:00	562.9	108.34	1.31	5.55	4.11	ND	-	ND

NOTES:
(1) ND = Not Detectables. Detectable levels for H_2S, COS, CH_3SH, CS_2, Thiophene are 0.2 ppm.
(2) - = Analysis not taken.

TABLE 2

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT GASIFIER CYCLONE EXIT
GASIFIER RUN 104
TRACE COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2S	COS	CH_3SH	CS_2 PPM	Thio	NH_3	H_2O	SO_2
	FN 61	12/9/83 07:00	2188	38.13	61.6	ND	ND	ND	36099	-
SULFI-	FN 66	12/9/83 09:53	1987.8	33.21	44.22	1.20	ND	ND	7840	-
DATION	FN 71	12/9/83 12:55	1900.8	33.29	36.48	1.15	ND	ND	1396	-
T020	FN 76	12/9/83 15:45	2398.4	57.24	23.2	1.57	ND	ND	734	-
	FN 81	12/10/83 07:00	2365.2	47.32	28.29	3.68	2.71	ND	1092	-
	FN 89	12/10/83 12:50	2341.0	70.60	67.76	5.69	10.98	ND	1264	-
	FN 93	12/10/83 15:35	2349.5	64.48	41.41	1.92	3.31	ND	679	-
SULFI- DATION T022	FN 125	12/13/83 07:05	4558.0	259.39	8.75	5.38	4.37	ND	692	-
	FN 128	12/13/83 10:35	4349.6	308.28	12.68	8.87	11.48	ND	780	-
	FN 131	12/13/83 13:05	4351.9	341.56	7.84	12.40	10.04	ND	959	-

TABLE 3

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT HGD EXIT
GASIFIER RUN 104
MAJOR COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2	O_2	N_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	iC_4	nC_4
						VOLUME %							
SULFI-	HG 1	12/8/83 22:00	11.26	7.51	57.68	1.32	5.27	ND	16.46	ND	ND	ND	ND
DATION	HG 2	12/9/83 00:00	15.03	4.15	52.64	1.72	6.00	0.06	19.87	0.04	ND	ND	ND
T020	HG 3	12/9/83 02:00	13.83	5.13	53.39	1.80	6.96	0.09	18.21	0.04	0.06	ND	ND
	HG 4	12/9/83 04:00	13.86	3.75	53.78	1.73	5.64	0.13	20.49	0.05	0.06	ND	ND
	HG 5	12/9/83 06:00	16.25	3.61	50.62	1.73	8.45	0.06	18.79	ND	ND	ND	ND
	HG 6	12/9/83 08:00	20.42	2.00	45.72	2.23	8.15	0.12	20.79	0.06	ND	ND	ND
	HG 7	12/9/83 10:30	11.99	3.23	56.16	1.88	5.31	0.10	20.76	ND	0.07	ND	ND
	HG 8	12/9/83 12:30	12.88	4.86	54.01	2.02	6.28	0.12	19.23	0.03	0.06	ND	ND
	HG 9	12/9/83 14:30	12.37	4.57	52.91	1.97	11.50	0.09	16.04	ND	0.06	ND	ND
	HG 10	12/9/83 15:45	ND	19.32	77.81	0.16	0.22	ND	2.00	ND	ND	ND	ND
	HG 11	12/9/83 16:30	12.22	3.68	53.83	1.87	6.90	0.10	20.84	ND	0.07	ND	ND
	HG 12	12/9/83 18:45	9.87	6.12	57.64	1.76	6.30	0.11	17.64	ND	0.06	ND	ND
	HG 13	12/9/83 20:30	9.93	5.76	58.99	1.51	4.67	0.07	18.52	ND	0.06	ND	ND
	HG 14	12/9/83 22:30	8.23	6.28	59.84	1.61	5.14	0.09	18.25	ND	0.06	ND	ND
	HG 15	12/10/83 00:30	9.69	6.29	56.94	1.98	6.07	0.15	18.24	0.05	0.08	ND	ND
SULFI-	HG 16	12/10/83 02:30	10.08	5.57	57.50	1.70	5.65	0.12	18.78	0.03	0.08	ND	ND
DATION	HG 17	12/10/83 04:30	13.47	3.30	52.66	1.98	6.32	0.14	21.53	0.04	0.07	ND	ND
T020	HG 18	12/10/83 06:30	11.13	5.04	56.57	1.96	5.42	0.10	19.20	ND	0.07	ND	ND

Table 3 (Continued)

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT HGD EXIT
GASIFIER RUN 104
MAJOR COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2	O_2	N_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	iC_4	nC_4
						VOLUME %							
	HG 19	12/10/83 08:30	7.24	8.56	60.60	1.56	4.61	0.10	16.78	ND	0.06	ND	ND
SULFI-	HG 20	12/10/82 10:30	4.84	9.87	62.67	1.64	5.89	0.10	14.44	ND	0.06	ND	ND
DATION	HG 21	12/10/82 14:30	9.52	6.91	57.89	1.71	4.84	0.14	18.35	0.07	0.07	ND	ND
T020	HG 22	12/10/82 18:30	6.07	7.87	61.32	1.30	5.73	0.08	17.08	ND	0.05	ND	ND
	HG 23	12/11/83 02:20	0.98	8.48	78.87	ND	ND	ND	9.43	ND	ND	ND	ND
REGENER-	HG 24	12/11/83 06:20	ND	8.70	83.16	ND	ND	ND	5.16	ND	ND	ND	ND
ATION	HG 25	12/11/83 10:20	ND	9.61	84.15	ND	ND	ND	2.06	ND	ND	ND	ND
T021	HG 26	12/11/83 14:20	ND	8.51	83.58	ND	ND	ND	1.16	ND	ND	ND	ND
	HG 27	12/11/83 18:20	ND	16.00	78.81	ND	ND	ND	1.61	ND	ND	ND	ND
	HG 28	12/12/83 17:15	10.62	4.29	56.33	2.85	11.83	0.35	12.96	0.12	0.14	ND	ND
SULFI-	HG 29	12/12/83 17:40	11.87	3.73	55.54	2.81	12.24	0.34	12.70	0.12	0.14	ND	ND
DATION	HG 30	12/12/83 21:00	12.75	3.76	55.76	2.58	12.04	0.31	12.06	0.10	0.13	ND	ND
T022	HG 31	12/13/83 01:00	5.86	6.95	60.59	2.12	13.52	0.31	9.89	0.09	0.17	ND	ND
	HG 32	12/13/83 05:00	6.77	5.74	60.33	2.37	11.69	0.32	12.00	0.11	0.17	ND	ND
	HG 33	12/13/83 08:00	10.17	4.00	55.30	2.72	15.50	0.37	11.14	0.12	0.18	ND	ND

NOTES:
(1) ND = Not Detectable. Detectable levels for H_2S, COS, CH_3SH, CS_2, Thiophene are 0.2 ppm.
(2) - = Analysis not taken.

TABLE 4

HOT GAS DESULFURIZATION
GRAB SAMPLE GAS ANALYSIS AT GASIFIER CYCLONE EXIT
GASIFIER RUN 104
MAJOR COMPONENTS

TEST # TYPE	SAMPLE NO.	DATE TIME	H_2	O_2	N_2	CH_4	CO	C_2H_6	CO_2	C_2H_4	C_3H_8	iC_4	nC_4
						VOLUME %							
	FN 61	12/9/83 07:00	18.33	0.78	47.48	2.12	16.02	0.13	14.47	0.11	0.06	ND	ND
SULFI-	FN 66	12/9/83 09:53	14.62	0.75	49.65	2.48	18.90	0.14	12.51	0.08	0.36	ND	ND
DATION	FN 71	12/9/83 12:55	17.30	0.98	47.61	2.35	16.52	0.49	13.57	0.38	0.31	ND	ND
T020	FN 76	12/9/83 15:45	16.50	1.05	50.22	2.16	14.43	0.13	14.89	0.06	0.06	ND	ND
	FN 81	12/10/83 07:00	18.19	0.89	47.59	2.33	14.34	0.40	15.03	0.36	0.37	ND	ND
	FN 89	12/10/83 12:50	18.78	0.71	46.25	2.36	16.41	0.12	14.38	0.13	0.36	ND	ND
	FN 93	12/10/83 15:35	19.42	0.74	45.19	2.69	16.34	0.17	14.78	0.09	0.08	ND	ND
SULFI-DATION 022	FN 123	12/13/83 07:05	16.20	0.78	50.50	2.82	18.53	0.03	10.06	0.08	0.50	ND	ND
	FN 128	12/13/83 10:35	15.78	0.34	50.20	2.79	18.96	0.23	10.13	0.11	0.47	ND	ND
	FN 131	12/13/83 13:05	15.28	0.90	51.31	3.31	16.99	0.25	10.79	0.18	0.49	ND	ND

TABLE 5

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 020A
AVERAGE GC READINGS

DURATION

FROM	TO
21:39: 0 12/ 8/1983	9:40: 0 12/ 9/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
12	29.287	22.318	0.140	-0.002	0.652	45.879	2.126	6.222	106.620	25.60	1.145	0.886	139.24	122.26

GASIFIER CYCLONE EXIT

BENDIX 7000 #4 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
69	22.634	13.887	0.110	0.184	0.279	46.055	2.569	17.140	102.858	24.87	1.112	0.860	157.93	143.78

TABLE 6

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 020B
AVERAGE GC READINGS

DURATION

FROM	TO
10:25: 0 12/ 9/1983	17: 0: 0 12/ 9/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
38	26.261	20.451	0.144	-0.001	0.615	46.794	2.076	7.001	103.341	23.17	1.126	0.871	131.50	116.07

GASIFIER CYCLONE EXIT

BENDIX 7000 #4 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
38	22.464	13.549	0.122	0.163	0.278	45.626	2.733	17.623	102.559	24.75	1.107	0.856	160.68	146.45

TABLE 7

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 020C
AVERAGE GC READINGS

DURATION

FROM		TO	
17:20: 0	12/ 9/1983	18: 5: 0	12/ 9/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
5	24.010	19.041	0.144	-0.001	0.595	51.070	1.950	5.755	102.564	25.33	1.133	0.876	118.90	104.72

GASIFIER CYCLONE EXIT

BENDIX 7000 #4 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
5	22.130	14.230	0.124	0.213	0.278	46.450	2.661	16.579	102.664	24.99	1.117	0.864	155.87	141.85

TABLE 8

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 020D
AVERAGE GC READINGS

DURATION

FROM	TO
19:29: 0 12/ 9/1983	20:15: 0 12/10/1983

HGD EXIT — BENDIX 6000 #2 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
154	27.063	21.481	0.161	-0.001	0.650	45.503	2.179	6.441	103.477	25.16	1.126	0.871	133.65	117.7[illegible]

GASIFIER CYCLONE EXIT — BENDIX 7000 #4 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
154	23.365	14.672	0.136	0.221	0.278	45.478	2.758	16.014	102.921	24.80	1.109	0.858	159.30	144.54

TABLE 9

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 021
AVERAGE GC READINGS

DURATION

FROM		TO	
0:15: 0	12/11/1983	20:50: 0	12/11/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
119	1.617	5.079	0.045	0.852	4.048	78.234	-0.015	-0.109	89.753	25.75	1.150	0.890	11.07	9.78

TABLE 10

HOT GAS DESULFURIZATION
SIDESTREAM TEST UNIT
TEST 022
AVERAGE GC READINGS

DURATION

FROM	TO
16:40: 0 12/12/1983	8: 5: 0 12/13/1983

HGD EXIT

BENDIX 6000 #2 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
92	24.904	13.622	0.385	0.004	1.002	47.342	3.057	15.061	105.377	24.91	1.113	0.861	167.33	151.23

GASIFIER CYCLONE EXIT

BENDIX 7000 #4 CHROMATOGRAPH

# READ	H_2	CO_2	C_2H_6	H_2S	O_2	N_2	CH_4	CO	TOTAL	AVG MW	AVG DTY	AVG SG	G BTU	N BTU
92	20.815	9.949	0.287	0.577	0.280	47.100	3.162	19.554	101.724	24.35	1.088	0.842	171.53	157.23

TABLE 11

ANALYSIS OF MINERALS BY X-RAY DIFFRACTION IN SULFIDED ZINC FERRITE GASIFIER RUN 104

BED HEIGHT (INCHES)	HEMATITE Fe_2O_3	FRANKLINITE $ZnFe_2O_4$ OR MAGNETITE Fe_3O_4 (4)	ZINCITE ZnO	WUSTITE FeO	ALPHA ZnS	BETA ZnS	PYRRHOTITE FeS
			PERCENT TII (3)				
0 (1)		2.8	5.9	23.4	19.6	33.0	13.3
6		1.0	7.2	30.1	18.7	29.5	13.5
16		1.4	7.1	29.2	16.2	32.3	13.9
23		1.6	6.8	30.4	16.7	31.1	13.3
26		3.2	9.8	36.2	13.3	27.5	9.9
36		5.3	15.8	50.7	7.0	18.4	2.8
46 (2)		4.9	22.9	58.6	2.8	10.8	-
FRESH SORBENT	4	89	6				

NOTES:

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

TABLE 12

SORBENT ANALYSIS OF
SULFIDED ZINC FERRITE
GASIFIER RUN 104
TEST 022

Bed Height (Inches)	Total Carbon (% Wt)	Total Sulfur (% Wt)	BET Surface Area (SQ M/G)	Skeletal Density (G/CC)(3)	Porosity %(4)	Crush Strength (Kg DWL) ± S.D.(5)	Skeletal Density (G/CC)(6)	Porosity %(7)
0(1)	4.92	21.55	2.28	4.17	1.91	10.1 ± 1.8	3.99	36.5
6	6.89	19.11	1.89	3.87	1.49	9.6 ± 2.4		
16	7.51	18.85	1.64	4.18	1.02	10.6 ± 1.4		
23	7.66	18.75	1.89	3.99	1.54	9.3 ± 2.5	3.49	30.5
26	8.40	15.76	1.65	4.31	0.88	9.8 ± 2.1		
36	7.73	5.98	3.64	6.56	3.79	10.0 ± 2.3		
46(2)	7.95	4.14	2.60	7.55	2.86	9.4 ± 2.3	4.41	43.9
Fresh Sorbent	0	0.13	3.13	6.16	2.59	6.3 ± 2.8	-	60.6(8)

NOTES:

1. Bottom/Inlet during sulfidation - Outlet during regeneration.
2. Top/Outlet during sulfidation - Inlet during regeneration.
3. Skeletal Density by Helium Pycnometry.
4. Porosity by Nitrogen adsorption for pores up to 600Å diameter.
5. Average and standard deviation of 15 measurements.
6. Skeletal density by mercury porosimetry.
7. Porosity by mercury porosimetry for pores down to 120Å diameter.
8. Porosity by mercury porosimetry for pores down to 30Å diameter.

TABLE 13

PARTICULATE SAMPLING DATA
HOT GAS DESULFURIZATION TEST UNIT
GASIFIER RUN 104

TEST #	HOURS FROM START OF TEST	PARTICLE LOADING g/Nm^3 INLET	PARTICLE LOADING g/Nm^3 EXIT	MEDIAN PARTICLE SIZE µm (microns) INLET	MEDIAN PARTICLE SIZE µm (microns) EXIT	CHEMICAL ANALYSIS WT % C	H	N	S	ASH
020	13.9		1.89		5.8	78.0	2.7	2.1	-	15.7

TABLE 14

DATE: 5/14/84
SAMPLE: HGDSU BAGNELL G01291 84-113
ELECTROLYTE: ISOTON
DISPERSANT: LOMAR D
EQUIPMENT: TA II
APERTURES: 70
OPERATOR:ARG

CH.#	SIZE	DIFF VOL %	CUM VOL %
1	.79	0	100
2	1	0	100
3	1.26	3.6	100
4	1.59	5.7	96.4
5	2	7.8	90.7
6	2.52	7.9	82.9
7	3.18	9.7	75
8	4	9.6	65.3
9	5.04	11.3	55.7
10	6.35	7.9	44.4
11	8	8	36.5
12	10.08	10.2	28.5
13	12.7	7.9	18.3
14	16.01	5.3	10.4
15	20.17	.7	5.1
16	25.41	4.4	4.4

* NOTE THAT PARTICLE SIZE DISTRIBUTION IS very BROAD AND ~ 4.4% of the PARTICLES ARE LARGER THAN 25μm

PARTICULATE SIZE DISTRIBUTION

TEST 020 EXIT

VOLUME % STATISTICS

MEAN:	5.84 UM
MEDIAN:	5.67 UM
MODE:	5.45 UM
STANDARD DEVIATION:	2.18 UM
SKEWNESS:	1.05 POSITIVE
KURTOSIS:	2.24 PLATYKURTIC

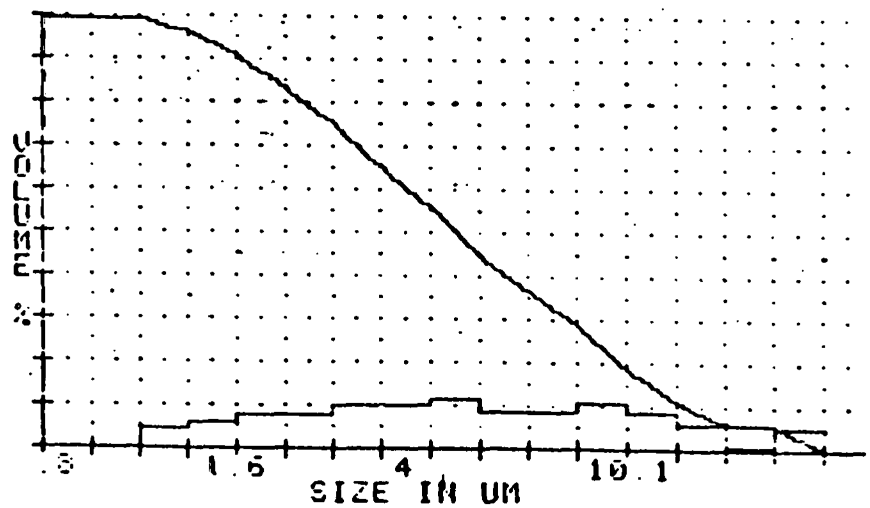


TABLE 15

TRACE COMPONENTS IN EXIT GAS
DURING SULFIDATION
GASIFIER RUN 104

COMPONENT	TEST 020 Aqueous Condensate Analysis PPMW	Calculated Gas Phase Composition PPMW
Ni	0.33	0.046
V	0.18	0.025
Cr	0.25	0.035
K	36.55	5.102
Na	102.5	14.31
Fe	18.7	2.61
Zn	0.247	0.034
Ca	20.59	2.87
Al	1.20	0.17
Se	0.215	0.030
As	0.18	0.025
Hg	<0.2	<0.028
Cu	0.16	0.022
F	10	1.40
Cl	119	16.61
Br^-	<4.	0.558
SO_4	215	30.01
PO_4	<3	0.42
NO_3	<2	0.28
NH_4	7898	1102.

NOTES:

1. Aqueous condensate analysis from sample # 1000290
2. Sample location is at condenser CIC Knockout.
3. Metals determined by Atomic Absorption/Atomic Emission spectroscopy.
4. Anions and cations determined by ion chromatography.

TABLE 16

TRACE COMPONENTS IN EXIT GAS AQUEOUS CONDENSATE DURING REGENERATION GASIFIER RUN 104

COMPONENT	TEST 021 Aqueous Condensate Analysis PPMW
Ni	0.30
V	0.61
Cr	0.44
K	52.71
Na	113.6
Fe	149.2
Zn	15.6
Ca	115.2
Al	83.2
Se	0.044
As	1.14
Hg	0.383
Cu	<0.02
F	<1
Cl	66
Br	296
SO_4	590
PO_4	<3
NO_3	<2
NH_4	79

NOTES:

1. Aqueous condensate analysis from samples # 1000292.
2. Sample location is at condenser CIC Knockout.
3. Metals determined by atomic absorption/atomic emission spectroscopy.
4. Anions and cations determined by ion chromatography.

TABLE 17

TRACE COMPONENTS IN INLET AND EXIT GAS AQUEOUS CONDENSATE
DURING SULFIDATION GASIFIER RUN #104
TEST #020

LOCATION	INLET	OUTLET
DATE	12-09-83	12-09-83
TIME		
LAB #	100307	100306
SAMPLE #	GI-12-9-2	GO-12-9-1
COMPONENT	PPMW	PPMW
Ni	0.11	0.20
V	<0.05	<0.05
Cr	4.9	0.22
K	2.6	0.52
Na	16.3	2.6
Fe	11.7	1.5
Zn	0.24	2.5
Ca	15.6	3.4
Al	0.18	<0.02
Se	0.496	<0.005
As	2.29	0.04
Hg	<0.2	<0.2
Cu	0.103	<0.02
F	NS	34
Cl	NS	1041
Br	NS	<4
NO3	NS	<2
SO4	NS	32
PO4	NS	<3
NH4	NS	9281

NOTES:

1. Aqueous condensate data obtained from HGD inlet and outlet particulate sampling systems.
2. NS indicates that the component was not measured.

TABLE 18

ULTIMATE ANALYSIS AND CONCENTRATION
OF TARS LEAVING HGD TEST UNIT
GASIFIER RUN 104

COMPONENT	TEST 020 WT %
ASH	0.40
S	0.44
H	10.53
N	0.8
TOTAL CARBON	6.11
CONCENTRATION OF TARS IN EXIT GAS OF HGD TEST UNIT	0.68

NOTES:

1. Sample number 28250
2. Sample location is at Condenser CIC Knockout
3. Sample contained water, Resulting in low Carbon value.

TABLE 19

ULTIMATE ANALYSIS OF TARS LEAVING GASIFIER CYCLONE GASIFIER RUN 104

HGD TEST #	TEST 020	TEST 020	TEST 020	TEST 022	TEST 022
DATE	12/9/83	12/9/83	12/9/83	12/12/83	12/13/83
TIME	06:00 HRS	10:00 HRS	22:00 HRS	21:00 HRS	03:00 HRS
LAB #	28362	28363	28364	28368	28370
COMPONENT			WT %		
S	NM	NM	NM	1.04	1.14
H	8.03	7.94	8.88	7.29	7.20
N	1.86	1.59	1.58	1.81	1.92
TOTAL CARBON	82.75	82.95	56.45	77.69	80.72

NOTES:

1. Analysis Obtained from limited samples which were run on microsystem.
2. Samples collected from S-4 precipitator.
3. NM = Not Measured.

TABLE 2[illegible]

ULTIMATE ANALYSIS OF COALS
USED DURING GASIFIER RUN 104

COMPONENT	WT%	
	PITTSBURGH SEAM COAL AKRWRIGHT	NORTH DAKOTA LIGNITE INDIAN HEAD
Moisture	0.39	0.52
Ash	7.15	10.28
S	1.82	1.66
H	5.12	4.76
N	3.05	1.27
Total Carbon	77.10	67.54
Volatiles	38.35	40.33
Btu/lb	13715	11956

APPENDIX I

Laboratory-Scale Testing - Simulated KRW Gas

Performance of Zinc Ferrite Sorbent in Simulated Hot Coal Gas From the KRW Process Development Unit

Introduction

In connection with the conceptual design of a zinc ferrite hot gas desulfurization unit by Westinghouse Electric Corporation Synthetic Fuels Division (now KRW Energy Systems, Inc.) as part of the Waltz Mill Coal Gasification Program (1), a program of experiments was designed for the METC, newly revamped, laboratory-scale, high-temperature desulfurization unit to investigate the performance of zinc ferrite sorbent in simulated coal gas corresponding to that anticipated from the KRW process development unit. The experiments were carried out from March to May 1984.

Laboratory-Scale Unit

Details of the laboratory-scale unit are given in contractor reports (2,3). The flow schematic is shown in Figure 1. The unit is a redesigned version of that employed in previous test series (4).

The new reactor component, which is 60 inches long by 2 inches diameter, is also made from 316 stainless steel but contains an integral preheater and after cooler and is aluminized internally to minimize hydrogen sulfide reaction with the reactor insides. The reactor is located within a 3-zone clamshell electrically heated furnace. A 12-inch section at the middle of the reactor length is available for the desulfurization sorbent. Temperatures at six points along the center line are measured by means of thermocouples situated in a stainless steel thermowell. The system is maintained at a positive pressure of 25 psig primarily to ensure flow through the on-line gas chromatograph, which measures H_2O, COS, and SO_2 concentrations in the exit gas. Process conditions can be recorded by an automatic data acquisition and control system (ADACS)(5).

Gas Conditions

It is planned to operate the KRW process development unit in both oxygen-blown and air-blown modes. Estimated compositions of the water-quenched gases entering the zinc ferrite unit are shown in Table 1 (6). Gas compositions approximating these were employed at the laboratory scale in a total of eight sulfidation tests: four oxygen blown and four air blown. Carbonyl sulfide and ammonia were excluded as being of minor consequence, and the hydrogen sulfide concentration was elevated so that breakthrough could occur within a day's operation at the space velocity of 2,000 h^{-1} employed-corresponding to a linear velocity of 15 cm/s through the reactor. The tests were conducted at 4 reactor furnace temperature settings: 1,000, 1,100, 1,200, and 1,300°F.

Operation of Laboratory-Scale Unit

The operating conditions for the series of tests are summarized in Figures 2 to 13. The first two tests, Nos. 001 and 002, were designed to verify that hydrogen sulfide in simulated coal gas reacts to a negligible extent with the inside of the aluminized reactor. In Test No. 001, simulated Lurgi air-blown gas containing 0.267 vol. percent H_2S was passed through the empty reactor for about 3 hours. Figure 14, which is a plot of H_2S concentration in the reactor condenser exit gas, measured by detector tube, shows that the H_2S level rapidly reached a plateau corresponding to the inlet H_2S concentration. When H_2S into the reactor inlet gas was shut off, during a further hour, the H_2S level in exit gas quickly fell to a few ppm indicating that very little sulfidation of the reactor had taken place. This conclusion was confirmed in Test No. 002, in which a typical regeneration gas consisting of 50 vol. percent each of air and steam was passed through the reactor for 2 hours. Only a few ppm of SO_2, measured by detector tube, was detected in the reactor condenser exit gas. For a further short period, simulated Lurgi air-blown coal gas was passed through the reactor. H_2S in the exit gas amounted to only a few ppm.

Test Nos. 003, 004, 005, and 006 were carried out with simulated oxygen-blown gas, containing 1 vol. percent H_2S, at furnace temperature settings of 1,000, 1,100, 1,200, and 1,300°F, respectively. The reactor contained 715 g of fresh United Catalysts L-1504 3/16-inch zinc ferrite extrusions in each test except for Test No. 003, where the amount was 593 g. Figures 15, 16, 17, and 18 show plots of the H_2S concentration in reactor condenser exit gas against time on stream for the four tests. Discontinuities in the plots are caused by overnight breaks in the tests. The plots are superimposed in Figure 19. The H_2S levels before breakthrough are a few ppm as predicted by thermodynamic chemical equilibrium computations. However, the prediction that the level should increase with temperature is not borne out for the 1,200 and 1,300°F tests. In both of these, the H_2S level is lower than in the 1,100°F test and comparable to the 1,000°F test.

The amounts of sulfur absorbed by the sorbent, computed from the total gas flow through the reactor and the inlet H_2S concentration in the four tests, are shown in Figure 20, expressed as weight percent of the fresh sorbent. The amount is greatest at 1,200°F but falls off at higher temperatures in accordance with previous experience with the sorbent.

Test No. 003 was repeated in Test No. 003R with 715 g of sorbent instead of 593 g to make it more comparable with the other tests. However, because of a malfunction related to water and hydrogen sulfide admission, the results are not fully valid.

Test Nos. 007, 008, 009, and 010 were carried out with simulated air-blown gas, containing 1 vol. percent H_2S, at furnace settings of 1,000, 1,100, 1,200, and 1,300°F, respectively. The reactor contained 715 g of fresh United Catalysts L-1504 3/16-inch zinc ferrite extrusions in each test. Test No. 010 was repeated in Test No. 010R because of poor temperature control in the former test. Figures 21, 22, 23, and 24 show plots of the H_2S concentration in reactor condenser exit gas against time on stream for the four temperatures. The plots are superimposed in Figure 25. The H_2S concentration levels attained are remarkably similar to those previously obtained for the oxygen-blown gas tests. Again, the H_2S levels above 1,100°F were unaccountably lower than expected.

The amounts of sulfur absorbed by the sorbent, computed from the total gas flow through the reactor and the inlet H_2S concentration in the four tests, are shown in Figure 20 together with the corresponding values for the oxygen-blown gas tests for comparison. The fall-off in sulfur absorbed above 1,200°F appears to be somewhat sharper for the air-blown gas case.

Reactor Pressure Drop

The differential pressure across the combined reactor and integral quartz wool packed preheater was measured throughout the desulfurization tests. This pressure drop was initially about 2 inches of water gauge in all cases. In the course of a test, this measured pressure drop increased somewhat, which is attributed to the formation of a water leg at the bottom of the reactor, resulting from unanticipated condensation of water from the reactor inlet gas stream. There is strong evidence, supported by a later test, that the actual reactor pressure drop remained virtually constant in each test.

Reactor Temperatures

Tables 2 and 3 show the average temperatures at 2-inch intervals along the reactor axis for Test Nos. 003, 004, 005 and 006 and 007, 008, 009, and 010R, respectively. These temperatures generally do not differ from the furnace settings of 1,000, 1,100, 1,200, and 1,300°F by more than about 25°F.

Sorbent Analysis

After breakthrough of H_2S in each test, the sorbent was cooled under nitrogen and removed from the reactor. Samples were taken from the top (gas outlet) and bottom (gas inlet) of the sorbent bed and analyzed for total sulfur, total carbon, surface area, pore volume, "helium" density, and mineral content. The mineral content, determined by X-ray diffraction, is shown in Tables 4 and 5 for Test Nos. 003, 004, 005, and 006 and 007, 008, 009, and 010R, respectively. The other analyses are given in Figures 4 to 13.

The total sulfur measured in the sorbent at the reactor inlet is generally in accord with that calculated from the flow of H_2S through the sorbent (see Figure 20). In Test Nos. 003, 004, 005, and 006, the values are 25.5, 27.2, 27.8, and 21.0 wt. percent, respectively, and in Tests Nos. 007, 008, 009, and 010R, they are 21.1, 30.1, 28.6, and 18.4 wt. percent, respectively. Total sulfur at the reactor outlet is usually about 1 wt. percent.

The total carbon measured in the sorbent at both reactor inlet and outlet is less than 0.1 wt. percent, being rather more at the outlet.

The sorbent surface areas at both reactor inlet and outlet are lower at the higher operating temperatures, falling from about 2 to 3 m^2/g at 1,000°F to 0.5 to 1.5 m^2/g at 1,300°F. Pore volumes are also correspondingly lower. Surface areas at the reactor inlet, where the sorbent is predominantly in the sulfide form, are generally somewhat lower, up to about two times, than at the outlet, where only a small amount of the sorbent is sulfidized.

The mineral analyses (see Tables 4 and 5) for all the tests indicate that most of the sorbent at the reactor inlet is converted to zinc and iron sulfide whereas at the reactor outlet, the sorbent is predominantly in the form of franklinite (and/or magnetite) and zincite. At 1,200 and 1,300°F, a substantial amount of wustite is present, generally more at the outlet than at the inlet. Cementite is only found in one case, that of air-blown gas at 1,300°F. However, this is not corroborated by the total carbon content, which is less than 0.1 wt. percent.

Crush Strength

Fifteen (15) extrusions from fresh sorbent and from each sample, approximately 1 cm long, were crushed between parallel flat surfaces and the crushing force was measured. The results are given in Table 6 (fresh sorbent) and Tables 7 to 14 (Test Nos. 003, 004, 005, 006, 007, 008, 009, and 010R).

Compared with the fresh sorbent, which has an average crush strength of about 8 kg/cm, the crush strength of the sorbent from the reactor inlet is generally rather higher, up to about two times, though this falls off with temperature and, at 1,300°F, the crush strength is about the same as that of fresh sorbent. The crush strength of sorbent from the reactor outlet tends to be slightly less than that of fresh sorbent at all temperatures but, because of the size of the standard deviation, this is not very significant.

Gas Analysis

During all the tests, grab samples of gas exiting the reactor outlet condenser were taken every 2 hours and analyzed for H_2, O_2, N_2, CO, CO_2, and CH_4 for comparison with the metered gas constituents fed to the reactor.

The analyses for the oxygen-blown case, i.e., Test Nos. 003 to 006, are shown in Table 15 and those for the air-blown case, i.e., Test Nos. 007, 008, 009, and 010R, in Table 16.

It is evident that the shift reaction, $CO + H_2O \rightarrow CO_2 + H_2$, has proceeded to a large extent. This is more so at the lower temperature of 1,000°F than at 1,300°F as expected by thermodynamic equilibrium calculation.

In Table 15, some of the samples can be seen to contain amounts of oxygen and nitrogen which should be absent. This is attributed to incomplete displacement of air from the grab sample bottles.

A small amount of water condensed from reactor inlet gases so that the actual composition entering the sorbent bed differs somewhat from the compositions given in Figures 1 to 12. The corrected compositions are given in Table 17.

Condensate Analysis

The water condensing out from the gases exiting the reactor was sampled and analyzed for zinc, iron, and sulfate. The results for Test Nos. 003 to 010 are given in Table 18. The concentration of zinc is generally less than 5 ppmw which, translated to gas concentration, is less than 1 ppm. Though not conclusive (see Test No. 6), there appears to be more zinc in the condensate at higher temperatures. The amount of sulfate in the condensate, when translated to gas concentration, is of the order of only about 1 ppmw of sulfur. Iron in the condensate is very low, suggesting very little carry over of sorbent into the condenser.

Summary and Conclusions

The series of tests carried out, in which simulated oxygen- and air-blown coal gas from the KRW process development unit was passed through zinc ferrite sorbent in a laboratory-scale unit, has demonstrated that the sorbent can function in the regime of gas composition and temperature planned for pilot-scale tests, lowering H_2S concentration to a few ppmw.

There is evidence that at 1,200°F and above, wustite is formed in both oxygen- and air-blown cases, resulting in a lower sorbent conversion to sulfide before hydrogen sulfide breakthrough. Thus, operation above 1,200°F and at lower steam concentrations in the gases than those employed in these tests is not recommended.

The amount of zinc carry over, less than 1 ppm in the gas, is thought to be acceptable. This level would be an order of magnitude smaller (based on zinc vapor equilibrium) at the higher pressure of operation of the process development unit.

There is a relatively small drop in sorbent surface area at the temperatures investigated but, in the absence of long-term cumulative effects, this will probably be tolerable. Also, sorbent crush strength is not altered markedly by the sulfidation process. In fact, sulfidized sorbent generally has a higher crush strength than fresh sorbent.

Virtually no carbon deposition occurred during the tests and the reactor differential pressure remained constant at a low level.

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TABLE 1

Compositions of Gases to be Treated by the Zinc Ferrite Process Development Unit

Component	Oxygen-Blown Gas (Vol.%)	Air-Blown Gas (Vol.%)
CH_4	1.2	0.63
CO	29.43	12.47
CO_2	26.12	10.96
H_2	11.23	13.76
H_2O	30.99	20.0
H_2S	0.59	0.301
COS	--	0.016
N_2	0.44	41.74
NH_3	--	0.11

TABLE 2

Average Reactor Temperature Profile
Laboratory-Scale Test Nos. 003 to 006

Test No.	Height Above Bottom	Duration of Test Hours	Average Temperature °F	Standard Deviation °F
003	2 inch	8.0	1,009	20
003	4 inch	8.0	1,035	19
003	6 inch	8.0	1,042	22
003	8 inch	8.0	1,034	17
004	2 inch	9.0	1,102	17
004	4 inch	9.0	1,118	18
004	6 inch	9.0	1,123	19
004	8 inch	9.0	1,122	15
005	2 inch	12.3	1,182	6
005	4 inch	12.3	1,175	16
005	6 inch	12.3	1,238	17
005	8 inch	12.3	1,253	20
006	2 inch	10.0	1,203	13
006	4 inch	10.0	1,273	4
006	6 inch	10.0	1,290	2
006	8 inch	10.0	1,301	2

Notes:

1. Height above bottom is the height above the reactor bottom/inlet.

2. Temperatures were recorded every 1/2 hour. The average temperature and standard deviation were determined from these recorded temperatures.

TABLE 3

Average Reactor Temperature Profile
Laboratory-Scale Test Nos. 007 to 010R

Test No.	Height Above Bottom	Duration of Test Hours	Average Temperature °F	Standard Deviation °F
007	2 inch	9.5	816	5
007	4 inch	9.5	966	2
007	6 inch	9.5	557*	5
007	8 inch	9.5	1,040	13
008	2 inch	12.0	908	9
008	4 inch	12.0	1,090	9
008	6 inch	12.0	659*	19
008	8 inch	12.0	1,119	9
009	2 inch	12.5	1,005	7
009	4 inch	12.5	1,199	17
009	6 inch	12.5	679*	28
009	8 inch	12.5	1,236	18
010R	2 inch	5.0	1,314	13
010R	4 inch	5.0	1,327	13
010R	6 inch	5.0	1,337	13
010R	8 inch	5.0	1,345	13

* Thermocouple functioned intermittently during Test Nos. 007, 008, and 009. A break in the thermocouple lead wire was discovered and repaired prior to Test No. 010R.

Notes:

1. Height above bottom is the height above the reactor bottom/inlet.

2. Temperatures were recorded every 1/2 hour. The average temperature and standard deviation were determined from these recorded temperatures.

TABLE 4

Analysis of Minerals by X-Ray Diffraction
in Sulfidized Zinc Ferrite Laboratory-Scale
Test Nos. 003 to 006

Test No. Sample Point	003 Top	003 Bottom	004 Top	004 Bottom
Franklinite	77.8	34.2	78.9	27.7
Zincite, ZnO	21.3	3.4	19.1	3.7
alpha-ZnS	0.0	13.7	0.8	13.7
beta-ZnS	0.9	36.8	1.2	40.5
Wustite, FeO	0.0	0.0	0.0	0.0
Pyrrhotite, FeS	0.0	11.8	0.0	12.0
Cementite, Fe_3C	0.0	0.0	0.0	0.0
Total % TII	100.0	99.9	100.0	97.6

Test No. Sample Point	005 Top	005 Bottom	006 Top	006 Bottom
Franklinite	66.2	14.9	72.1	13.6
Zincite, ZnO	24.2	2.1	17.8	7.3
alpha-ZnS	0.9	2.3	3.1	2.6
beta-ZnS	3.0	70.1	2.7	60.5
Wustite, FeO	5.6	0.8	2.5	11.8
Pyrrhotite, FeS	0.0	9.8	1.7	4.3
Cementite, Fe_3C	0.0	0.0	0.0	0.0
Total % TII	99.9	100.0	99.9	100.1

Notes:

1. Simulated oxygen-blown gas was used for Test Nos. 003 to 006.

2. Bottom is equivalent to absorber inlet.

3. Top is equivalent to absorber outlet.

4. % TII = Percent Total Integrated Intensity; directly proportional to the concentration of the crystalline phase. Amorphous phase is not accounted for.

5. Franklinite ($ZnFe_2O_4$) and Magnetite (Fe_3O_4) have the same crystal structure and cannot be readily distinguished by diffraction pattern. % TII reported for Franklinite could be Franklinite and/or Magnetite.

TABLE 5

Analysis of Minerals by X-Ray Diffraction
in Sulfidized Zinc Ferrite Laboratory-Scale
Test Nos. 007 to 010R

Test No. Sample Point	007 Top	007 Bottom	008 Top	008 Bottom
Franklinite	76.4	46.5	76.7	20.9
Zincite, ZnO	21.2	11.8	19.7	3.0
alpha-ZnS	0.8	12.2	1.1	13.3
beta-ZnS	1.6	16.2	2.5	44.3
Wustite, FeO	0.0	0.0	0.0	0.5
Pyrrhotite, FeS	0.0	13.2	0.0	18.0
Cementite, Fe_3C	0.0	0.0	0.0	0.0
Total % TII	100.0	99.9	100.0	100.0

Test No. Sample Point	009 Top	009 Bottom	010R Top	010R Bottom
Franklinite	41.9	3.9	25.8	33.7
Zincite, ZnO	25.9	2.4	24.9	8.5
alpha-ZnS	0.9	3.8	0.7	4.7
beta-ZnS	6.7	72.4	9.4	40.7
Wustite, FeO	19.3	3.0	30.4	4.6
Pyrrhotite, FeS	5.2	14.5	0.0	7.7
Cementite, Fe_3C	0.0	0.0	8.9	0.0
Total % TII	99.9	100.0	100.1	99.9

Notes:

1. Simulated air-blown gas was used for Test Nos. 007 to 010R.

2. Bottom is equivalent to absorber inlet.

3. Top is equivalent to absorber outlet.

4. % TII = Percent Total Integrated Intensity; directly proportional to the concentration of the crystalline phase. Amorphous phase is not accounted for.

5. Franklinite ($ZnFe_2O_4$) and Magnetite (Fe_3O_4) have the same crystal structure and cannot be readily distinguished by diffraction pattern. % TII reported for Franklinite could be Franklinite and/or Magnetite.

TABLE 6

Fresh Zinc Ferrite Sorbent Crush Strength

Sorbent Crush Strength Test No.	Fresh Sorbent Extrusion Length (cm)	Fresh Sorbent Normalized Force (kgf/cm)
1	1.41	15
2	0.59	5
3	1.13	4
4	0.91	13
5	1.04	10
6	1.09	4
7	0.90	5
8	0.87	4
9	0.67	10
10	0.68	4
11	1.18	8
12	0.80	5
13	1.22	10
14	1.02	7
15	1.00	5
Average	0.97	8
Standard Deviation	0.23	3.50

TABLE 7

Sulfidized Zinc Ferrite Sorbent Crush Strength After Test No. 003

Sorbent Crush Strength Test No.	T 003 Bottom Extrusion Length (cm)	T 003 Bottom Normalized Force (kgf/cm)	T 003 Top Extrusion Length (cm)	T 003 Top Normalized Force (kgf/cm)
1	0.82	18	1.23	8
2	0.73	12	0.96	9
3	1.10	17	0.80	5
4	1.06	14	1.19	12
5	1.37	11	0.96	4
6	0.70	8	1.02	2
7	0.85	19	0.73	2
8	0.91	17	0.88	16
9	0.95	14	0.81	19
10	1.00	11	1.17	5
11	0.91	20	0.80	2
12	0.73	9	0.69	4
13	1.24	8	0.77	11
14	1.03	20	0.95	2
15	1.00	7	0.86	8
Average	0.96	14	0.92	7
Standard Deviation	0.19	5	0.17	5

Notes:

1. Bottom is equivalent to absorber inlet.

2. Top is equivalent to absorber outlet.

TABLE 8

Sulfidized Zinc Ferrite Sorbent Crush Strength After Test No. 004

Sorbent Crush Strength Test No.	T 004 Bottom Extrusion Length (cm)	T 004 Bottom Normalized Force (kgf/cm)	T 004 Top Extrusion Length (cm)	T 004 Top Normalized Force (kgf/cm)
1	1.19	11	0.91	4
2	1.04	15	0.82	1
3	0.78	12	1.04	2
4	0.99	14	1.12	11
5	0.98	19	1.18	2
6	1.07	6	0.68	2
7	0.68	6	1.13	4
8	1.01	17	1.20	2
9	1.18	9	1.11	4
10	0.72	9	1.01	3
11	0.87	19	0.73	4
12	0.85	19	1.20	5
13	1.18	7	1.15	1
14	1.25	14	0.73	2
15	0.73	10	0.88	1
Average	0.97	12	0.99	3
Standard Deviation	0.19	5	0.19	3

Notes:

1. Bottom is equivalent to absorber inlet.

2. Top is equivalent to absorber outlet.

TABLE 9

Sulfidized Zinc Ferrite Sorbent Crush Strength After Test No. 005

Sorbent Crush Strength Test No.	T 005 Bottom Extrusion Length (cm)	T 005 Bottom Normalized Force (kgf/cm)	T 005 Top Extrusion Length (cm)	T 005 Top Normalized Force (kgf/cm)
1	0.90	9	1.16	3
2	1.13	7	0.91	3
3	0.93	10	0.87	4
4	0.71	12	0.96	8
5	0.96	7	1.20	2
6	1.09	10	0.71	2
7	0.93	16	0.99	6
8	0.72	12	0.55	1
9	0.71	13	0.94	2
10	0.81	10	0.91	1
11	0.91	11	0.82	3
12	0.80	11	0.92	3
13	1.21	12	0.88	2
14	1.15	15	1.08	2
15	0.65	6	0.85	2
Average	0.91	11	0.92	3
Standard Deviation	0.18	3	0.16	2

Notes:

1. Bottom is equivalent to absorber inlet.

2. Top is equivalent to absorber outlet.

TABLE 10

Sulfidized Zinc Ferrite Sorbent Crush Strength After Test No. 006

Sorbent Crush Strength Test No.	T 006 Bottom Extrusion Length (cm)	T 006 Bottom Normalized Force (kgf/cm)	T 006 Top Extrusion Length (cm)	T 006 Top Normalized Force (kgf/cm)
1	0.91	6	1.12	10
2	0.73	19	1.31	9
3	1.17	11	0.55	3
4	1.35	4	0.98	3
5	1.18	4	0.82	2
6	1.01	8	0.97	6
7	0.82	7	1.14	5
8	0.99	17	1.00	2
9	0.72	11	0.77	3
10	0.69	7	1.02	6
11	0.76	6	1.10	5
12	1.19	5	1.05	3
13	1.21	6	1.42	7
14	1.10	9	0.91	2
15	0.98	6	0.80	1
Average	0.99	8	1.00	5
Standard Deviation	0.21	4	0.22	3

Notes:

1. Bottom is equivalent to absorber inlet.

2. Top is equivalent to absorber outlet.

TABLE 11

Sulfidized Zinc Ferrite Sorbent Crush Strength After Test No. 007

Sorbent Crush Strength Test No.	T 007 Bottom Extrusion Length (cm)	T 007 Bottom Normalized Force (kgf/cm)	T 007 Top Extrusion Length (cm)	T 007 Top Normalized Force (kgf/cm)
1	0.95	5	1.27	7
2	1.31	5	1.36	4
3	0.67	11	0.82	8
4	1.11	16	1.25	2
5	1.00	9	1.01	7
6	1.08	4	1.04	4
7	0.96	5	0.91	17
8	0.72	8	0.81	7
9	1.10	16	1.19	10
10	0.85	10	1.20	18
11	0.73	7	1.05	3
12	0.89	8	0.73	10
13	1.07	5	1.05	10
14	0.85	5	0.90	3
15	0.68	22	0.92	5
Average	0.93	9	1.03	8
Standard Deviation	0.19	5	0.19	5

Notes:

1. Bottom is equivalent to absorber inlet.

2. Top is equivalent to absorber outlet.

TABLE 12

Sulfidized Zinc Ferrite Sorbent Crush Strength After Test No. 008

Sorbent Crush Strength Test No.	T 008 Bottom Extrusion Length (cm)	T 008 Bottom Normalized Force (kgf/cm)	T 008 Top Extrusion Length (cm)	T 008 Top Normalized Force (kgf/cm)
1	1.30	11	0.85	2
2	1.04	14	0.74	2
3	0.67	9	1.00	6
4	1.03	13	0.99	5
5	1.09	9	1.03	3
6	0.75	10	1.07	2
7	0.86	17	0.88	3
8	0.96	10	0.68	3
9	1.01	19	0.91	2
10	1.21	18	0.93	2
11	1.11	12	0.92	22
12	1.01	14	0.87	6
13	1.09	18	1.44	6
14	1.05	12	0.82	4
15	1.40	10	0.67	4
Average	1.04	13	0.92	5
Standard Deviation	0.19	4	0.19	5

Notes:

1. Bottom is equivalent to absorber inlet.

2. Top is equivalent to absorber outlet.

TABLE 13

Sulfidized Zinc Ferrite Sorbent Crush Strength After Test No. 009

Sorbent Crush Strength Test No.	T 009 Bottom Extrusion Length (cm)	T 009 Bottom Normalized Force (kgf/cm)	T 009 Top Extrusion Length (cm)	T 009 Top Normalized Force (kgf/cm)
1	1.23	6	1.00	5
2	0.81	5	1.28	4
3	1.31	5	1.11	7
4	0.94	5	1.00	9
5	1.09	8	0.81	5
6	0.99	4	1.17	8
7	1.06	5	0.66	3
8	0.86	13	0.70	17
9	1.11	18	0.97	13
10	1.40	6	1.03	10
11	0.89	7	0.95	8
12	0.97	8	0.52	3
13	1.20	14	1.32	4
14	0.97	7	0.88	4
15	1.02	12	0.74	10
Average	1.06	8	0.94	7
Standard Deviation	0.17	4	0.23	4

Notes:

1. Bottom is equivalent to absorber inlet.

2. Top is equivalent to absorber outlet.

TABLE 14

Sulfidized Zinc Ferrite Sorbent Crush Strength After Test No. 010R

Sorbent Crush Strength Test No.	T 010R Bottom Extrusion Length (cm)	T 010R Bottom Normalized Force (kgf/cm)	T 010R Top Extrusion Length (cm)	T 010R Top Normalized Force (kgf/cm)
1	1.05	5	0.72	4
2	1.66	2	1.00	8
3	0.97	3	1.13	2
4	0.73	3	1.03	4
5	0.85	6	1.06	4
6	1.24	4	1.34	9
7	0.81	12	0.82	9
8	0.89	3	0.93	16
9	1.03	4	0.81	4
10	1.17	2	1.07	3
11	0.93	4	1.05	3
12	1.19	4	1.10	3
13	1.20	16	1.95	9
14	1.02	10	0.93	4
15	0.98	19	0.90	2
Average	1.05	7	0.99	6
Standard Deviation	0.23	5	0.15	4

Notes:

1. Bottom is equivalent to absorber inlet.

2. Top is equivalent to absorber outlet.

TABLE 15

Grab Sample Gas Analysis for Major Components at Condenser Exit Laboratory-Scale Test Nos. 003 to 006

Sample No.	Lab No.	Sample Date	Time	H_2 %	O_2 %	N_2 %	CO %	CO_2 %	CH_4 %	Total %
Test No. 003										
HG-003-1	HG No. 1	04/04/84	1100	26.34	0.13	1.30	26.16	44.33	1.23	99.49
HG-003-2	HG No. 2	04/04/84	1300	32.65	0.07	0.96	23.15	41.46	1.21	99.50
HG-003-3	HG No. 3	04/04/84	1500	20.05	0.94	7.89	24.03	45.32	1.27	99.50
HG-003-4	HG No. 4	04/05/84	1100	25.60	0.05	1.13	25.64	45.76	1.33	99.51
Test No. 004										
HG-004-1	HG No. 5	04/10/84	1100	18.24	1.33	10.34	26.90	41.52	1.17	99.50
HG-004-2	HG No. 6	04/10/84	1300	15.34	2.21	16.68	24.46	39.64	1.16	99.49
HG-004-3	HG No. 7	04/10/84	1500	12.28	3.26	23.94	20.62	38.40	1.01	99.51
HG-004-4	HG No. 8	04/11/84	0930	20.20	1.07	8.53	25.74	42.71	1.25	99.50
Test No. 005										
HG-005-1	HG No. 9	04/13/84	1000	4.05	8.40	32.88	23.57	29.65	0.94	99.49
HG-005-2	HG No. 10	04/13/84	1200	2.06	10.16	44.14	18.09	24.33	0.72	99.50
HG-005-3	HG No. 11	04/13/84	1400	5.56	7.10	28.28	24.63	33.02	0.98	99.57
HG-005-4	HG No. 12	04/16/84	0900	8.18	4.65	18.88	30.07	36.63	1.09	99.50
HG-005-5	HG No. 13	04/16/84	1100	25.06	3.72	8.46	23.61	37.07	1.58	99.50
HG-005-6	HG No. 14	04/16/84	1515	23.10	0.07	0.65	30.99	43.34	1.34	99.49
Test No. 006										
HG-006-1	HG No. 15	04/18/84	1030	18.04	0.33	1.45	33.40	44.64	1.65	99.51
HG-006-2	HG No. 16	04/18/84	1230	19.35	0.29	1.37	35.32	41.74	1.42	99.49
HG-006-3	HG No. 17	04/18/84	1430	26.55	0.13	1.09	30.68	39.75	1.30	99.50
HG-006-4	HG No. 18	04/18/84	1630	24.57	0.69	5.19	26.41	41.32	1.32	99.50
HG-006-5	HG No. 19	04/18/84	1820	19.98	1.51	11.28	20.22	44.51	2.00	99.50

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TABLE 16

Grab Sample Gas Analysis for Major Components at Condenser Exit Laboratory-Scale Test Nos. 007 to 010R

Sample No.	Lab No.	Sample Date	Time	H_2 %	O_2 %	N_2 %	CO %	CO_2 %	CH_4 %	Total %
Test No. 007										
HG-007-1	HG No. 20	04/25/84	1000	23.64	0.08	47.67	9.86	17.20	1.04	99.49
HG-007-2	HG No. 21	04/25/84	1200	16.72	2.66	54.69	8.64	15.70	1.09	99.50
HG-007-3	HG No. 22	04/25/84	1400	19.21	0.85	51.86	9.66	16.74	1.20	99.52
HG-007-4	HG No. 23	04/25/84	1600	22.51	0.11	49.74	9.10	16.83	1.16	99.45
Test No. 008										
HG-008-1	HG No. 24	05/02/84	1030	26.24	0.45	46.98	10.56	14.09	1.17	99.49
HG-008-2	HG No. 25	05/02/84	1230	19.00	1.20	52.02	11.07	15.06	1.15	99.50
HG-008-3	HG No. 26	05/02/84	1430	17.40	1.62	53.13	10.96	15.20	1.19	99.50
HG-008-4	HG No. 27	05/02/84	1630	21.32	0.23	49.47	11.02	16.25	1.20	99.49
HG-008-5	HG No. 28	05/02/84	1830	16.36	1.87	54.36	10.48	15.22	1.21	99.50
HG-008-6	HG No. 29	05/02/84	2025	18.47	1.03	52.27	10.94	15.50	1.29	99.50
Test No. 009										
HG-009-1	HG No. 30	05/09/84	1000	13.47	2.12	56.69	14.62	11.29	1.30	99.49
HG-009-2	HG No. 31	05/09/84	1200	20.08	0.30	49.28	13.54	15.11	1.18	99.49
HG-009-3	HG No. 32	05/09/84	1400	21.20	0.25	49.01	13.26	14.44	1.33	99.49
HG-009-4	HG No. 33	05/09/84	1600	13.96	2.34	56.13	11.39	14.41	1.27	99.50
HG-009-5	HG No. 34	05/09/84	1800	19.99	0.10	49.80	12.63	15.82	1.17	99.51
HG-009-6	HG No. 35	05/09/84	2000	16.18	1.08	53.81	11.81	15.48	1.13	99.49
Test No. 010R										
HG-010R-1	HG No. 38	06/12/84	1045	17.00	0.10	53.64	12.06	15.52	1.16	99.48
HG-010R-2	HG No. 39	06/12/84	1245	16.47	0.23	54.48	11.41	15.69	1.23	99.51
HG-010R-3	HG No. 40	06/12/84	1405	16.31	0.28	61.77	7.03	12.85	1.26	99.50

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TABLE 17

Calculated Compositions of Gas Entering the Zinc Ferrite Sorbent Bed
Lab Scale Tests No. 001 through 010R

Test	Volume %								
Number	H_2S	H_2O	CO_2	CO	H_2	CH_4	N_2	Air	Total
001	0.3	28.8	9.6	11.5	16.8	2.8	30.2		100.0
002		50.0						50.0	100.0
003	1.0	32.0	25.0	30.0	11.0	1.0			100.0
003R	1.0	30.7	25.5	30.6	11.2	1.0			100.0
004	1.0	32.0	25.0	30.0	11.0	1.0			100.0
005	1.0	29.8	25.8	31.0	11.3	1.0			100.0
006	1.0	28.8	26.2	31.4	11.5	1.0			100.0
007	1.0	16.9	11.3	12.9	14.4	0.7	42.8		100.0
008	1.0	17.2	11.3	12.9	14.3	0.7	42.6		100.0
009	1.0	17.4	11.3	12.8	14.3	0.7	42.5		100.0
010	1.0	19.9	10.9	12.4	13.8	0.6	41.2		100.0
010R	1.0	19.9	10.9	12.4	13.8	0.6	41.2		100.0

TABLE 18

Analysis of Exit Gas Knockout Condensate
Laboratory-Scale Test Nos. 003 to 010

Test No.	Sample No.	Lab No.	Zn ppm	Fe ppm	Sulfate ppm
003	HG-003	100315	3.98	0.342	47.9
004	HG-004	100316	1.40	0.293	32.4
005	HG-005	100317	4.65	0.067	8.2
006	HG-006	100318	1.50	0.067	8.1
007	HG-007	100319	1.71	< 0.050	8.9
008	HG-008	100320	1.31	0.104	10.9
009	HG-009	100321	4.86	0.151	4.5
010	HG-010	100322	18.75	2.020	45.8

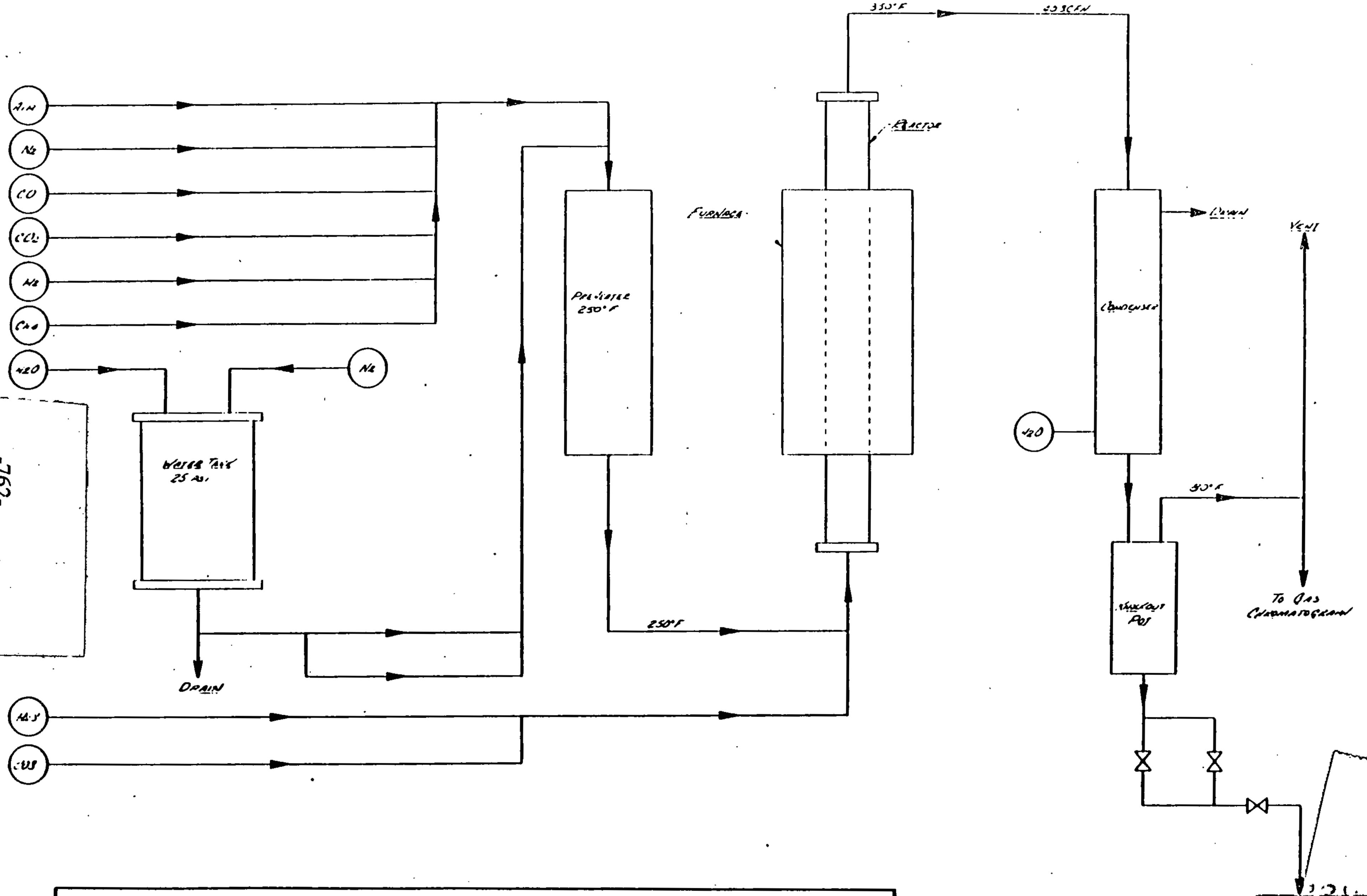
Notes:

1. Sample date was May 29, 1984.

2. Simulated oxygen-blown gas was used in Test Nos. 003 to 006.

3. Simulated air-blown gas was used in Test Nos. 007 to 010.

FIGURE 1



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

Design Specifications
Design Pressure
Design Temperature
Operating Pressure

FIGURE 2

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. : 001
DATE STARTED : 11:00 3/27/84
DATE ENDED : 14:40 3/27/84
TOTAL HOURS : 3.6 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: NONE
SORBENT NO. :

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : 30 PSIG

SPACE VELOCITY:

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.100 SCFH	0.267 %	0.375 %
H2O (10.77 SCFH)	3.872CC/MIN	28.76%	
AIR			
CO2	3.6 SCFH	9.61 %	13.5 %
CO	4.3 SCFH	11.48 %	16.12 %
H2	6.3 SCFH	16.82 %	23.62 %
CH4	1.07 SCFH	2.85 %	4.01 %
N2	11.3 SCFH	30.17 %	42.36 %
TOTAL FLOW :	37.44 SCFH		

PURPOSE

SULFIDE EMPTY ALONIZED METAL REACTOR
SHAKEDOWN NEWLY BUILT QUARTZ/METAL TEST UNIT

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S: 2000 - 8000 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. SULFUR GAS SHUT OFF AT 1400 HOURS.
2. EXIT SULFUR DOES NOT MATCH INLET SULFUR AS CALCULATED.

CONCLUSIONS

1. SULFIDING OF REACTOR APPEARS MINIMAL.

FIGURE 3

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. : 002
DATE STARTED : 11:00 3/28/84
DATE ENDED : 13:00 3/28/84
TOTAL HOURS : 2 HRS
TYPE : REGENERATION

SORBENT TYPE/WEIGHT: NONE
SORBENT NO. :
SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR REMOVED :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : AMBIENT

SPACE VELOCITY:

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

PURPOSE

REGENERATION OF EMPTY REACTOR TEST.

ANALYSIS:

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

DATA: (DETECTOR TUBE)

EXIT H2S:

EXIT SO2: 2.5 - 7.5 PPM

EXIT S2:

EXIT H2:

REMARKS

1. AT 13:00 HOURS SWITCHED TO SULFIDATION GASES WITHOUT H2S.

2. 2 - 15 PPM H2S MEASURED ON SULFIDATION GASES MINUS H2S.

CONCLUSIONS

1. SULFUR DETECTED DURING REGENERATION WAS MINIMAL, INDICATING AGAIN THAT SULFUR PICKUP BY ALONIZED METAL WALLS IS VERY SMALL.

FIGURE 4

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO.	:	803
DATE STARTED	:	09:00 4/04/84
DATE ENDED	:	11:30 4/5/84 WITH INTERRUPTION
TOTAL HOURS	:	8.5 HRS
TYPE	:	SULFIDATION

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE OXYGEN BLOWN GAS COMPOSITION

SORBENT TYPE/WEIGHT: ZINC FERRITE/593 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	AFTER EXIT
TOTAL SULFUR (%W/W):	0.13	25.5	1.08
SURFACE AREA (M2/G):	3.13	2.419	1.985
SCELETAL DENSITY (G/CC) :	6.16	4.648	6.467
PORE VOLUME (CC/G):	.00432	.004395	.003237
POROSITY, % ~~MINERAL-ANALYSIS:~~	2.6	2.0	2.1
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.05	0.06

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

DATA: (DETECTOR TUBE)

EXIT H2S: 3-5 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.5 %
H2O (9.94 SCFH)	3.57 CC/MIN	32.0 %	
AIR			
CO2	7.77 SCFH	25.0 %	36.8 %
CO	9.32 SCFH	30.0 %	44.1 %
H2	3.4 SCFH	11.0 %	16.1 %
CH4	0.31 SCFH	1.0 %	1.5 %
N2			
TOTAL FLOW :	31.07 SCFH		

REMARKS

1. H2 WAS SHIFTED FROM 16.1% TO 26.16% AVG.
2. CO WAS SHIFTED FROM 44.1% TO 24.70% AVG
3. CO2 WAS SHIFTED FROM 36.8% TO 44.21% AVG
4. REACTOR WAS ON N2 HOT HOLD OVERNIGHT.
5. CONDENSATE COLLECTED IS 419 CC , ONLY 23 % OF H2O FED AT INLET.

CONCLUSIONS

1. GOOD PERFORMANCE OBTAINED WITH THE HIGH H2O CONTENT OF OXYGEN BLOWN GAS.

FIGURE 5

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. : 003R
DATE STARTED : 08:45 6/19/84
DATE ENDED : 17:00 6/20/84
TOTAL HOURS : 21 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: ZINC FERRITE/715 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.5 %
H2O (9.94 SCFH)	3.57 CC/MIN	32.0 %	
AIR			
CO2	7.77 SCFH	25.0 %	36.8 %
CO	9.32 SCFH	30.0 %	44.1 %
H2	3.4 SCFH	11.0 %	16.1 %
CH4	0.31 SCFH	1.0 %	1.5 %
N2			
TOTAL FLOW:	31.07 SCFH		

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE OXYGEN BLOWN GAS COMPOSITION. REPEAT OF T003 WITH 715 GM SORBENT LOADING INSTEAD OF 593 GM.

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.13	33.36	4.26
SURFACE AREA (M2/G):	3.13	2.9168	1.8811
DENSITY (G/CC) :	6.16	3.911	5.086
PORE VOLUME (CC/G):	.00432	.005017	.003243
POROSITY % MINERAL ANALYSIS:	2.6	1.9	1.6
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.06	0.06

DATA: (DETECTOR TUBE)

EXIT H2S: 1 PPM FOR 6.75 HOURS, GRADUAL RISE THEREAFTER

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. CONDENSATE DRAINED FROM KNOCKOUT POT @ TEST END: 1800 CC H2O
2. CONDENSATE DRAINED FROM REACTOR AT END OF TEST: 270 CC H2O. THEREFORE, ACTUAL COMPOSITION OF GAS ENTERING REACTOR SORBENT BED WAS DIFFERENT THAN COMPOSITION SHOWN AT LEFT. ACTUAL H2O FLOW RATE ENTERING SORBENT BED WAS 3.36 CC/MIN.
3. ONE HOUR INTERRUPTION 1430-1530 HOURS 6/20/84 DUE TO CHANGING OF CO CYLINDER AND GETTING WATER OUT OF H2S ROTAMETER.
4. WATER PRESSURE APPARENTLY OVERCOMES H2S PRESSURE AND CAUSES WATER TO BACK UP INTO H2S FLOWMETER.
5. REACTOR PRESSURE DROP INCREASES FROM 3 - 12 INCHES OF WATER.
6. TEST INTERRUPTED FOR N2 HOT HOLD OVERNIGHT.

CONCLUSIONS:

1. GOOD PERFORMANCE MEASURED, BUT WATER IN H2S ROTAMETER TOWARDS LATTER PART OF TEST RAISES QUESTION AS TO ACCURACY TO DATA.

FIGURE 6

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. : 004
DATE STARTED : 09:00 4/10/84
DATE ENDED : 10:45 4/11/84
TOTAL HOURS : 8.75 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE OXYGEN BLOWN GAS COMPOSITION

SORBENT TYPE/WEIGHT: ZINC FERRITE/715 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

	BEFORE	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.13	2.82	
SURFACE AREA (M2/G):	3.13	.2019	2.299
SKELETAL DENSITY (G/CC) :	6.16	4.26	5.49
PORE VOLUME (CC/G):	.00432	.001595	.003192
POROSITY % MINERAL ANALYSIS:	2.6	0.7	1.7
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.07	

OPERATING CONDITIONS

TEMPERATURE: 1100 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

DATA: (DETECTOR TUBE)

EXIT H2S: 3 - 6 PPM

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.5 %
H2O (9.94 SCFH)	3.57 CC/MIN	32.0 %	
AIR			
CO2	7.77 SCFH	25.0 %	36.8 %
CO	9.32 SCFH	30.0 %	44.1 %
H2	3.4 SCFH	11.0 %	16.1 %
CH4	0.31 SCFH	1.0 %	1.5 %
N2			
TOTAL FLOW :	31.07 SCFH		

REMARKS

1. CONDENSATE COLLECTED: 836 CC, 45% OF H2O FED AT INLET.
2. GRAB SAMPLES FOR MAJORS CONTAINED 8-23% N2, INDICATING THAT SAMPLE BOTTLES WERE NOT PURGED LONG ENOUGH.
3. TEST WAS INTERRUPTED WITH N2 HOT HOLD OVERNIGHT AND AND CONTINUED THE NEXT DAY.
4. REACTOR PRESSURE DROP INCREASED FROM 2.3-10 INCHES OF WATER, AND DROPPED SLIGHTLY AFTER N2 HOT HOLD.

CONCLUSIONS

1. REASONABLY GOOD PERFORMANCE WITH HIGH H2O CONTENT ON OXYGEN BLOWN GAS.

FIGURE 7

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. : 005
DATE STARTED : 08:00 4/13/84
DATE ENDED : 16:00 4/16/84 WITH INTERRUPTION
TOTAL HOURS : 12.25 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE OXYGEN BLOWN GAS COMPOSITION AT 1200 F.

SORBENT TYPE/WEIGHT: ZINC FERRITE/715 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.13	27.8	1.76
SURFACE AREA (M2/G):	3.13	0.548	1.018
DENSITY (G/CC) :	6.16	4.15	6.50
PORE VOLUME (CC/G):	.00432	.000370	.001324
MINERAL ANALYSIS:	2.6	0.2	0.9
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.03	0.08

Skeletal Porosity %

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.5 %
H2O (9.94 SCFH)	3.57 CC/MIN	32.0 %	
AIR			
CO2	7.77 SCFH	25.0 %	36.8 %
CO	9.32 SCFH	30.0 %	44.1 %
H2	3.4 SCFH	11.0 %	16.1 %
CH4	0.31 SCFH	1.0 %	1.5 %
N2			
TOTAL FLOW :	31.07 SCFH		

DATA: (DETECTOR TUBE)

EXIT H2S: 1 - 6 PPM FOR 12 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. CONDENSATE DRAINED FROM KNOCKOUT POT @ TEST END: 980 CC H2O.
2. CONDENSATE DRAINED FROM REACTOR AT END OF TEST: 260 CC H2O. THEREFORE, ACTUAL COMPOSITION OF GAS ENTERING REACTOR SORBENT BED WAS DIFFEREDNT THAN COMPOSITION SHOWN AT LEFT. ACTUAL H2O FLOW RATE ENTERING SORBENT BED WAS 3.22 CC/MIN.
3. REACTOR BED HEIGHT IS 8 INCHES.
4. GRAB SAMPLES FOR MAJOR COMPONENTS CONTAINED UP TO 44 % NITROGEN, INDICATING THAT SAMPLE BOTTLES WERE NOT PURGED LONG ENOUGH DURING SAMPLING.
5. UP TO 0.4 PPM COS DETECTED AT BEGINNING OF RUN.
6. TEST INTERRUPTED ON N2 HOT HOLD OVER THE WEEK END.
7. REACTOR PRESSURE DROP INCREASED FROM 1.8 - 9.4 INCHES BUT DROPPED DOWN TO 2.2 AFTER HOT HOLD.

CONCLUSIONS

1. GOOD PERFORMANCE AT 1200 F AND HIGH H2O CONTENT OF OXYGEN BLOWN COMPOSITION.

FIGURE 8

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. : 006
DATE STARTED : 08:30 4/18/84
DATE ENDED : 18:30 4/18/84 (NO INTERRUTIONS)
TOTAL HOURS : 10 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: ZINC FERRITE/715 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION: :

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1300 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.5 %
H2O (9.94 SCFH)	3.57 CC/MIN	32.0 %	
AIR			
CO2	7.77 SCFH	25.0 %	36.8 %
CO	9.32 SCFH	30.0 %	44.1 %
H2	3.4 SCFH	11.0 %	16.1 %
CH4	0.31 SCFH	1.0 %	1.5 %
N2			

TOTAL FLOW : 31.07 SCFH

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE OXYGEN BLOWN GAS COMPOSITION AT 1300 F.

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.13	21.0	5.43
SURFACE AREA (M2/G):	3.13	0.57	1.56
SKELETAL DENSITY (G/CC) :	6.16	4.50	5.27
PORE VOLUME (CC/G):	.00432	.000436	.002071
POROSITY % MINERAL ANALYSIS:	2.6	0.2	1.1
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.05	0.05

DATA: (DETECTOR TUBE)

EXIT H2S: 1 - 3 PPM FOR 7 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. CONDENSATE DRAINED FROM KNOCKOUT POT @ TEST END: 550 CC H2O.
2. CONDENSATE DRAINED FROM REACTOR AT END OF TEST: 300 CC H2O. THEREFORE, ACTUAL COMPOSITION OF GAS ENTERING REACTOR SORBENT BED WAS DIFFERENT THAN COMPOSITION SHOWN AT LEFT. ACTUAL H2O FLOW RATE ENTERING SORBENT BED WAS 3.07 CC/MIN.
3. REACTOR BED HEIGHT IS 8 INCHES.
4. REACTOR PRESSURE DROP INCREASED FROM 3.4 TO 11.4 INCHES OF WATER.
5. H2 SHIFT FROM 16.1% TO 21.3% AVG.
   CO SHIFT FROM 44.1% TO 33.1 AVG.
   CO2 SHIFT FROM 36.8% TO 42.04% AVG

CONCLUSIONS:
1. GOOD PERFORMANCE AT 1300 F ON HIGH H2O CONTENT OXYGEN BLOWN GAS COMPOSITION.

FIGURE 9

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. : 007
DATE STARTED : 08:00 4/25/84
DATE ENDED : 17:30 4/25/84
TOTAL HOURS : 9.5 HRS
TYPE : SULFIDATION

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE AIR BLOWN GAS COMPOSITION

SORBENT TYPE/WEIGHT: ZINC FERRITE/715 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.13	21.09	1.79
SURFACE AREA (M2/G):	3.13	1.38	2.13
DENSITY (G/CC) :	6.16	4.25	5.65
PORE VOLUME (CC/G):	.00432	.002007	.003018
MINERAL ANALYSIS:	2.6	0.8	1.7
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.08	0.08

OPERATING CONDITIONS

TEMPERATURE: 1000 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

DATA: (DETECTOR TUBE)

EXIT H2S: 1 - 4 PPM FOR 8 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.2 %
H2O (6.2 SCFH)	2.22 CC/MIN	20.0 %	
AIR			
CO2	3.4 SCFH	11.0 %	13.7 %
CO	3.87 SCFH	12.5 %	15.5 %
H2	4.3 SCFH	13.8 %	17.2
CH4	0.20 SCFH	0.63 %	0.78 %
N2	12.82 SCFH	41.7 %	51.6 %
TOTAL FLOW :	31.07 SCFH		

REMARKS

1. CONDENSATE DRAINED FROM KNOCKOUT POT @ TEST END: 490 CC H2O.
2. CONDENSATE DRAINED FROM REACTOR AT END OF TEST: 230 CC H2O. THEREFORE, ACTUAL COMPOSITION OF GAS ENTERING REACTOR SORBENT BED WAS DIFFERENT THAN COMPOSTION SHOWN AT LEFT. ACTUAL H2O FLOW RATE ENTERING SORBENT BED WAS 1.82 CC/MIN.
3. REACTOR BED HEIGHT IS 8 INCHES.
4. H2 WAS SHIFTED FROM 17.2 % TO 20.52 % AVG.
   CO WAS SHIFTED FROM 15.5 % TO 9.3 % AVG.
   CO2 WAS SHIFTED FROM 13.7 % TO 16.6 % AVG.

CONCLUSIONS

1. GOOD PERFORMANCE ON AIR BLOWN GAS COMPOSITION AT 1000 F

FIGURE 10

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. :	008
DATE STARTED :	08:30 5/2/84
DATE ENDED :	20:30 5/2/84
TOTAL HOURS :	12 HRS
TYPE :	SULFIDATION

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE AIR BLOWN GAS COMPOSITION

SORBENT TYPE/WEIGHT:	ZINC FERRITE/715 GRAMS
SORBENT NO. :	L-1504
SORBENT COMPOSITION:	
SORBENT PELLET SIZE:	
PRETREATMENT :	
SULFUR LOADING :	

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.13	30.14	2.90
SURFACE AREA (M2/G):	3.13	1.24	1.72
SKELETAL DENSITY (G/CC) :	6.16	4.22	5.27
PORE VOLUME (CC/G):	.00432	.001918	.002435
POROSITY, % MINERAL ANALYSIS:	[illegible]	0.8	1.3
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.03	0.05

OPERATING CONDITIONS

TEMPERATURE: 1100 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.2 %
H2O (6.2 SCFH)	2.22 CC/MIN	20.0 %	
AIR			
CO2	3.4 SCFH	11.0 %	13.7 %
CO	3.87 SCFH	12.5 %	15.5 %
H2	4.3 SCFH	13.8 %	17.2
CH4	0.20 SCFH	0.63 %	0.78 %
N2	12.82 SCFH	41.7 %	51.6 %
TOTAL FLOW :	31.07 SCFH		

DATA: (DETECTOR TUBE)

EXIT H2S: 5 PPM FOR 11.5 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. CONDENSATE DRAINED FROM KNOCKOUT POT @ TEST END: 680 CC H2O.
2. CONDENSATE DRAINED FROM REACTOR AT END OF TEST: 260 CC H2O. THEREFORE, ACTUAL COMPOSITION OF GAS ENTERING REACTOR SORBENT BED WAS DIFFERENT THAN COMPOSTION SHOWN AT LEFT. ACTUAL H2O FLOW RATE ENTERING SORBENT BED WAS 1.86 CC/MIN.
3. REACTOR BED HEIGHT 8 INCHES.
4. H2 SHIFTED FROM 17.2 % TO 19.8 % AVG. CO SHIFTED FROM 15.5 % TO 10.8 % AVG. CO2 SHIFTED FROM 13.7 % TO 15.2 % AVG.
5. REACTOR PRESSURE DROP INCREASED FROM 3.1 TO 11.0 INCHES

CONCLUSIONS

1. GOOD PERFORMANCE ON AIR BLOWN GAS COMPOSITION AT 1100 F.
2. H2S AT EXIT HIGHER THAN AT 1000 F, AS EXPECTED BY EQUILIBRIUM CONSIDERATIONS.

FIGURE 11

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. :	009
DATE STARTED :	08:00 5/09/84
DATE ENDED :	20:40 5/9/84
TOTAL HOURS :	12.5 HRS
TYPE :	SULFIDATION

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE AIR BLOWN GAS COMPOSITION

SORBENT TYPE/WEIGHT: ZINC FERRITE/715 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.13	28.63	1.84
SURFACE AREA (M2/G):	3.13	0.74	1.17
[illegible] DENSITY (G/CC) :	6.16	4.18	5.33
PORE VOLUME (CC/G):	.00432	.000748	.001714
[illegible] MINERAL ANALYSIS:	2.6	0.3	0.7
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.03	0.04

OPERATING CONDITIONS

TEMPERATURE: 1200 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.2 %
H2O (6.2 SCFH)	2.22 CC/MIN	20.0 %	
AIR			
CO2	3.4 SCFH	11.0 %	13.7 %
CO	3.87 SCFH	12.5 %	15.5 %
H2	4.3 SCFH	13.8 %	17.2 %
CH4	0.20 SCFH	0.63 %	0.78 %
N2	12.82 SCFH	41.7 %	51.6 %
TOTAL FLOW :	31.07 SCFH		

DATA: (DETECTOR TUBE)

EXIT H2S: 1 PPM FOR 11.5 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. CONDENSATE DRAINED FROM KNOCKOUT POT @ TEST END: 670 CC H2O.
2. CONDENSATE DRAINED FROM REACTOR AT END OF TEST: 250 CC H2O. THEREFORE, ACTUAL COMPOSITION OF GAS ENTERING REACTOR SORBENT BED WAS DIFFERENT THAN COMPOSITION SHOWN AT LEFT. ACTUAL H2O FLOW RATE ENTERING SORBENT BED WAS 1.89 CC/MIN.
3. REACTOR BED HEIGHT 8 INCHES.
4. H2 SHIFTED FROM 17.2 % TO 17.5 % AVG.
   CO SHIFTED FROM 15.5 % TO 12.9 % AVG.
   CO2 SHIFTED FROM 13.7 % T 14.42 % AVG.
5. REACTOR PRESSURE DROP INCREASED FROM 2.6 TO 9.2 INCHES.

CONCLUSIONS

1. GOOD PERFORMANCE ON AIR BLOWN GAS COMPOSITION AT 1200 F.
2. NOT MUCH SHIFT CONVERSION AT THIS TEMPERATURE.

FIGURE 12

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. : 010
DATE STARTED : 08:00 5/15/84
DATE ENDED : 10:40 5/15/84
TOTAL HOURS : 2.67 HRS
TYPE : SULFIDATION

SORBENT TYPE/WEIGHT: ZINC FERRITE/715 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1300 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.2 %
H2O (6.2 SCFH)	2.22 CC/MIN	20.0 %	
AIR			
CO2	3.4 SCFH	11.0 %	13.7 %
CO	3.87 SCFH	12.5 %	15.5 %
H2	4.3 SCFH	13.8 %	17.2 %
CH4	0.20 SCFH	0.63 %	0.78 %
N2	12.82 SCFH	41.7 %	51.6 %
TOTAL FLOW :	31.07 SCFH		

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE AIR BLOWN GAS COMPOSITION

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.13	8.44	1.40
SURFACE AREA (M2/G):	3.13	1.16	1.35
SPECIFIC DENSITY (G/CC) :	6.16	4.91	5.25
PORE VOLUME (CC/G):	.00432	.00157	.001762
POROSITY % MINERAL ANALYSIS: —	2.6	0.8	0.7
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.04	0.04

DATA: (DETECTOR TUBE)

EXIT H2S: 1 PPM FOR 1.5 HOURS

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. REACTOR BED HEIGHT 8 INCHES.
2. PROBLEM WITH T/C AT 6 INCHES FROM INLET READING TOO LOW
3. CONDENSATE COLLECTED: 220 CC, 62% OF H2O FED AT INLET.

CONCLUSIONS

1. PROBLEMS WITH TEMPERATURE CONTROL.
2. TEST TO BE REPEATED.

FIGURE 13

TEST SUMMARY SHEET
HOT GAS DESULFURIZATION LAB SCALE TEST UNIT

TEST NO. :	010R
DATE STARTED :	08:45 6/12/84
DATE ENDED :	14:05 6/12/84
TOTAL HOURS :	5.33 HRS
TYPE :	SULFIDATION

SORBENT TYPE/WEIGHT: ZINC FERRITE/715 GRAMS
SORBENT NO. : L-1504

SORBENT COMPOSITION:

SORBENT PELLET SIZE:
PRETREATMENT :
SULFUR LOADING :

OPERATING CONDITIONS

TEMPERATURE: 1300 F

PRESSURE : 25 PSIG

SPACE VELOCITY: 2000

	FLOW RATE	MOLE% WET BASIS	MOLE% DRY BASIS
H2S	0.31 SCFH	1.0 %	1.2 %
H2O (6.2 SCFH)	2.22 CC/MIN	20.0 %	
AIR			
CO2	3.4 SCFH	11.0 %	13.7 %
CO	3.87 SCFH	12.5 %	15.5 %
H2	4.3 SCFH	13.8 %	17.2
CH4	0.20 SCFH	0.63 %	0.78 %
N2	12.82 SCFH	41.7 %	51.6 %
TOTAL FLOW :	31.07 SCFH		

PURPOSE

SULFIDE ZINC FERRITE (L1504) USING WESTINGHOUSE AIR BLOWN GAS COMPOSITION. THIS IS A REPEAT OF TEST 010 DUE TO THERMOCOUPLE PROBLEMS IN THAT TEST.

ANALYSIS:

	BEFORE (FRESH)	AFTER INLET	EXIT
TOTAL SULFUR (%W/W):	0.31 73	18.42	1.00
SURFACE AREA (M2/G):	3.13	0.9079	1.3030
DENSITY (G/CC) :	6.16	4.603	5.384
PORE VOLUME (CC/G):	0.00432	0.001355	0.001891
MINERAL ANALYSIS:	2.6	0.6	1.0
ELEMENTAL ANALYSIS:			
TOTAL CARBON (%W/W):	0.00	0.03	0.04

DATA: (DETECTOR TUBE)

EXIT H2S: 2 - 3 PPM FOR 5 HOURS.

EXIT SO2:

EXIT S2:

EXIT H2:

REMARKS

1. REACTOR BED HEIGHT 8 INCHES.
2. REACTOR PRESSURE DROP INCREASED FROM 3.5 TO 5.9 INCHES.
3. H2 SHIFTED FROM 17.2 % TO 16.6 % AVG.
   CO SHIFTED FROM 15.5 % TO 10.2 % AVG.
   CO2 SHIFTED FROM 13.7 % TO 14.7 % AVG.
4. CONDENSATE COLLECTED: 485 CC, 69% OF H2O FED TO REACTOR.

CONCLUSIONS

1. TIME TO BREAKTHROUGH IS SHORTER , AS EXPECTED.
2. NOT MUCH SHIFT CONVERSION.

FIGURE 14

ZINC FERRITE SULFIDATION

LAB SCALE TEST UNIT TEST 001

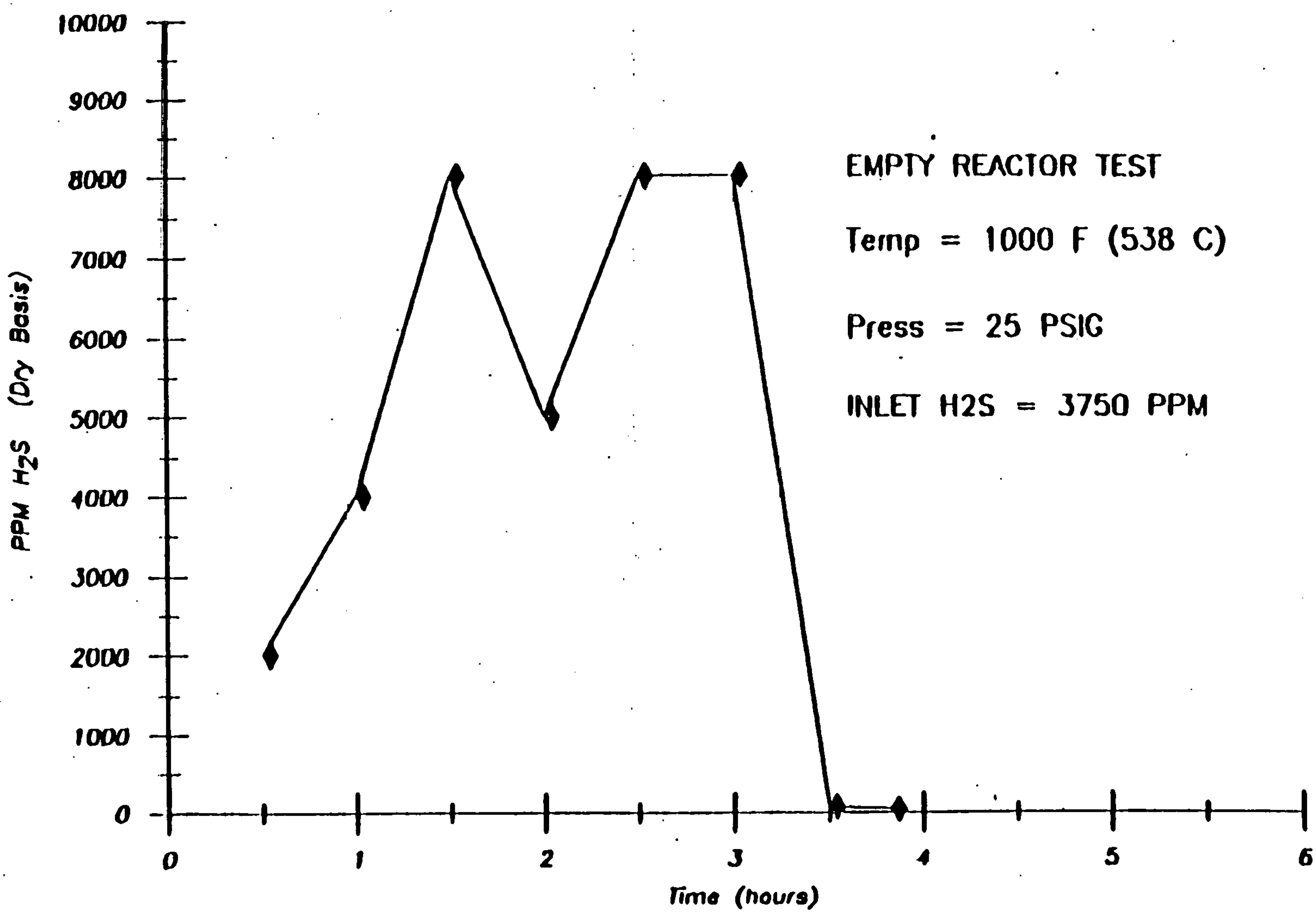


FIGURE 15

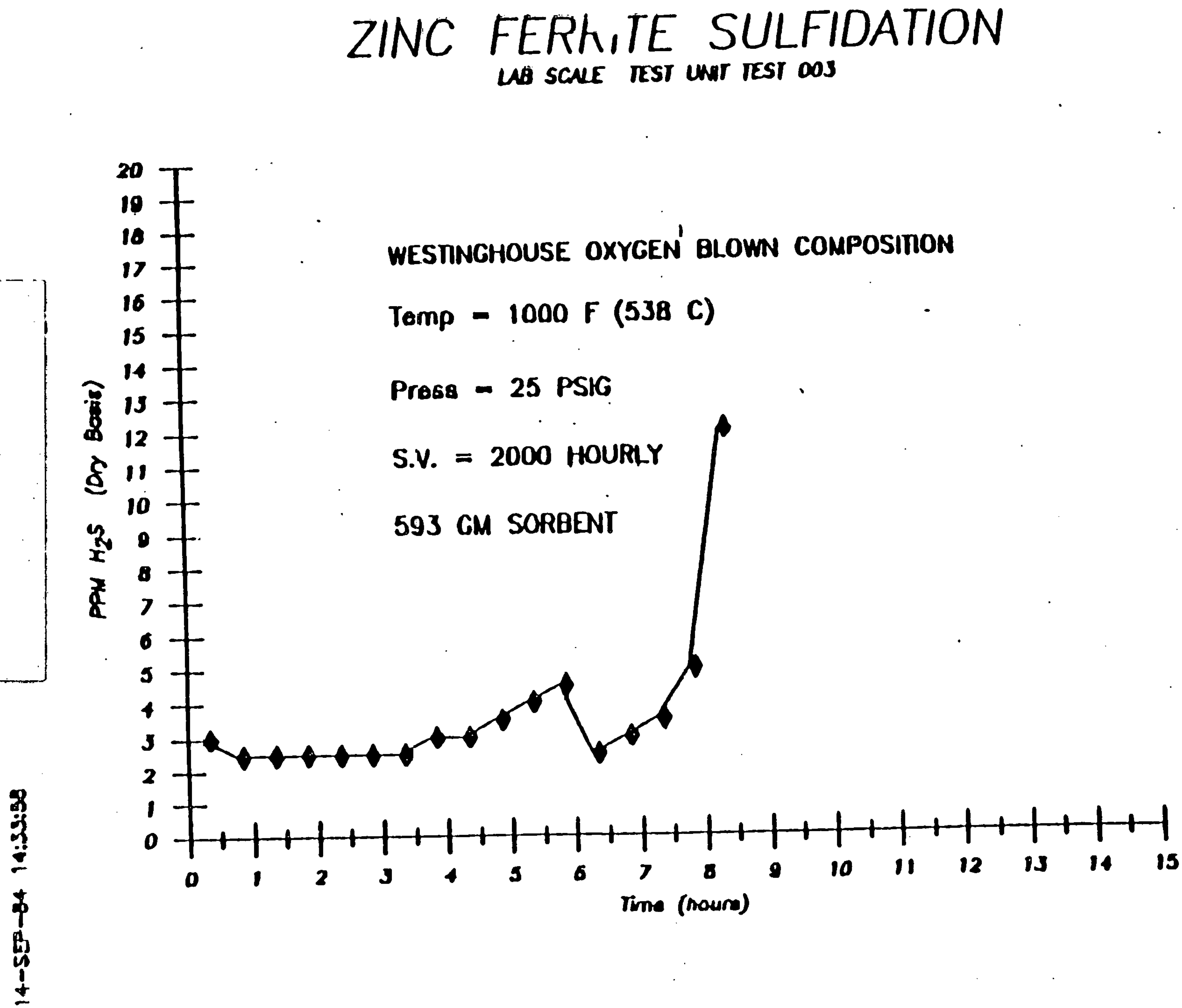


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FIGURE 16

ZINC FERRITE SULFIDATION

LAB SCALE TEST UNIT TEST 004

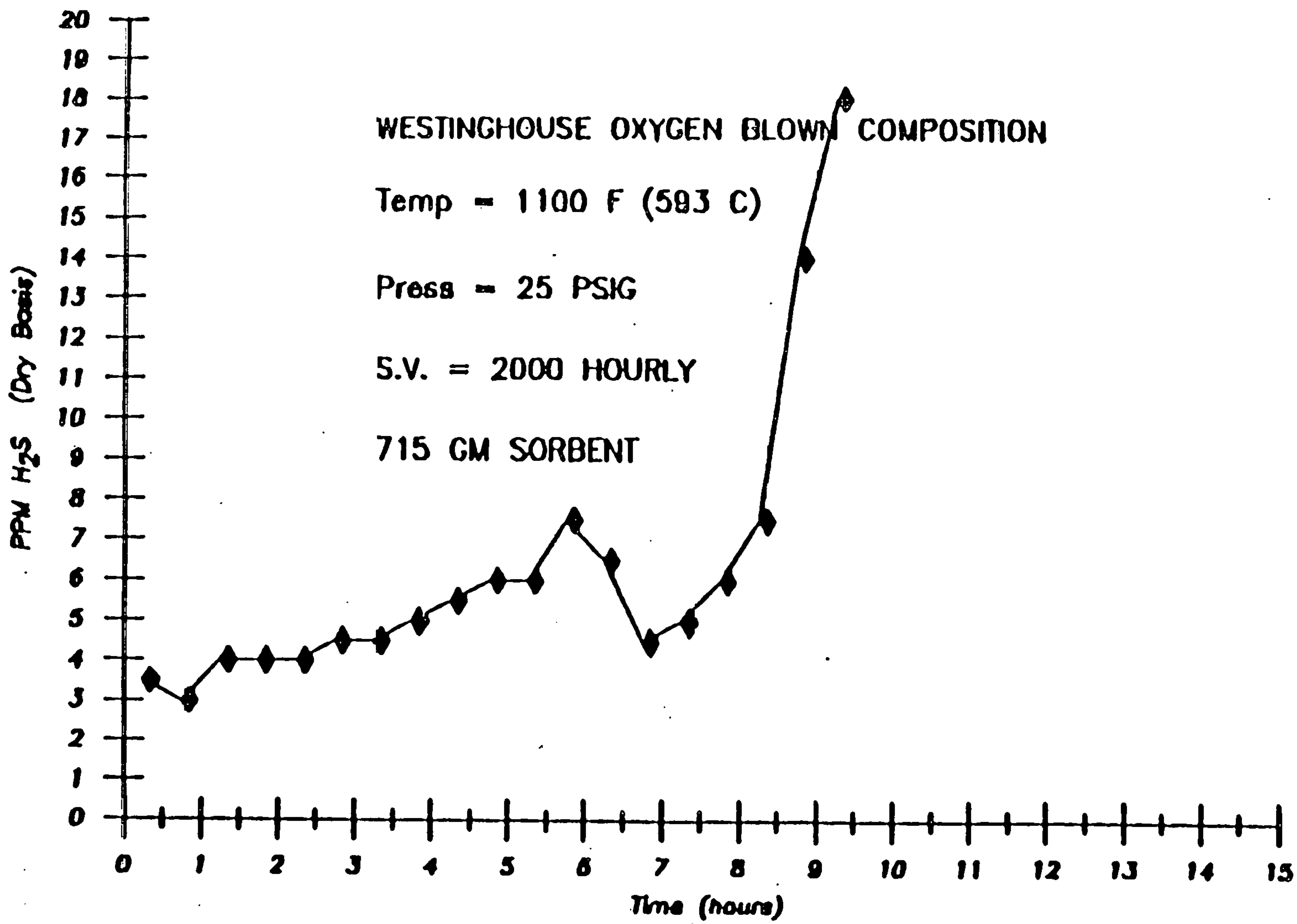
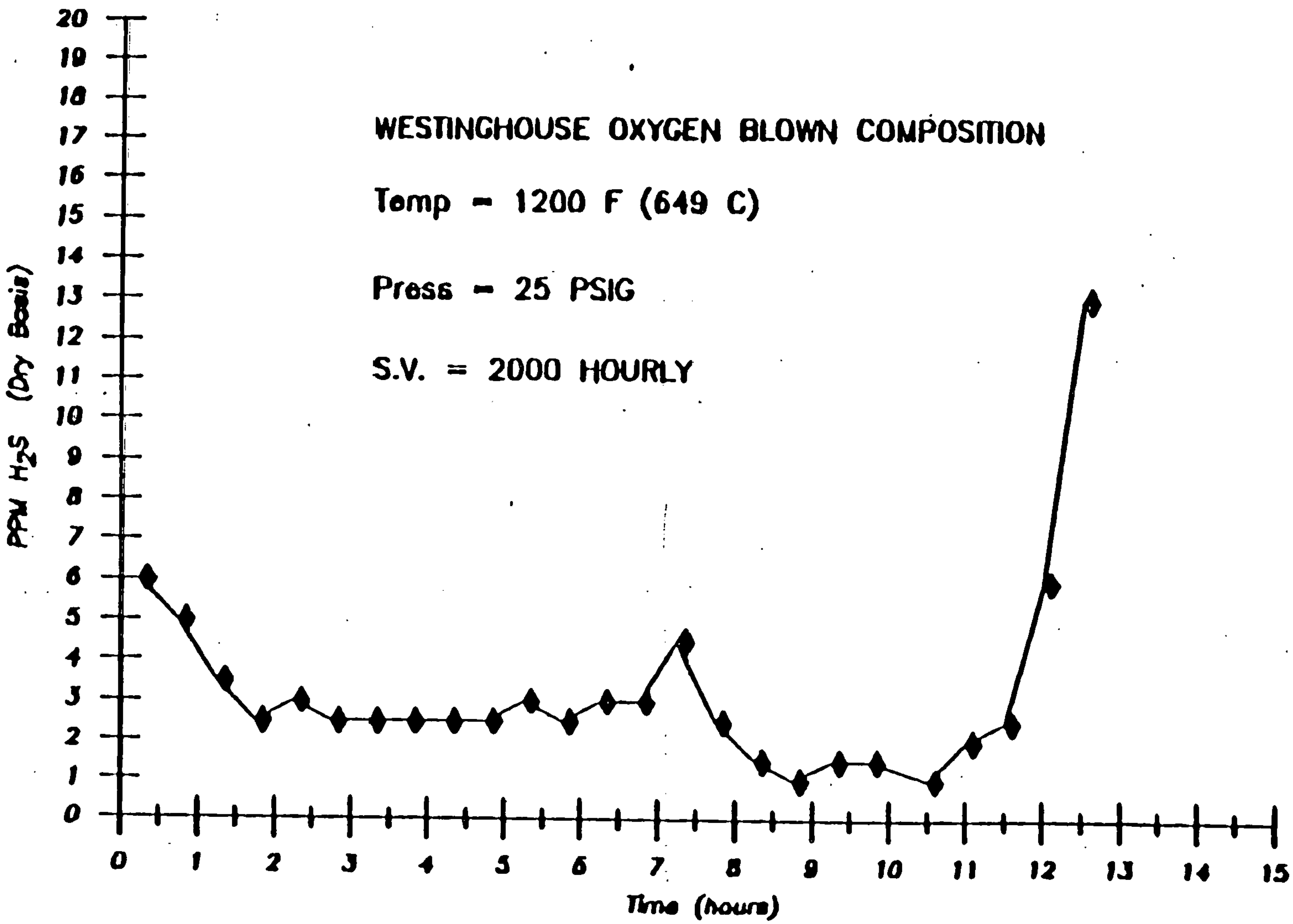


FIGURE 17

ZINC FERRITE SULFIDATION

LAB SCALE TEST UNIT TEST 005

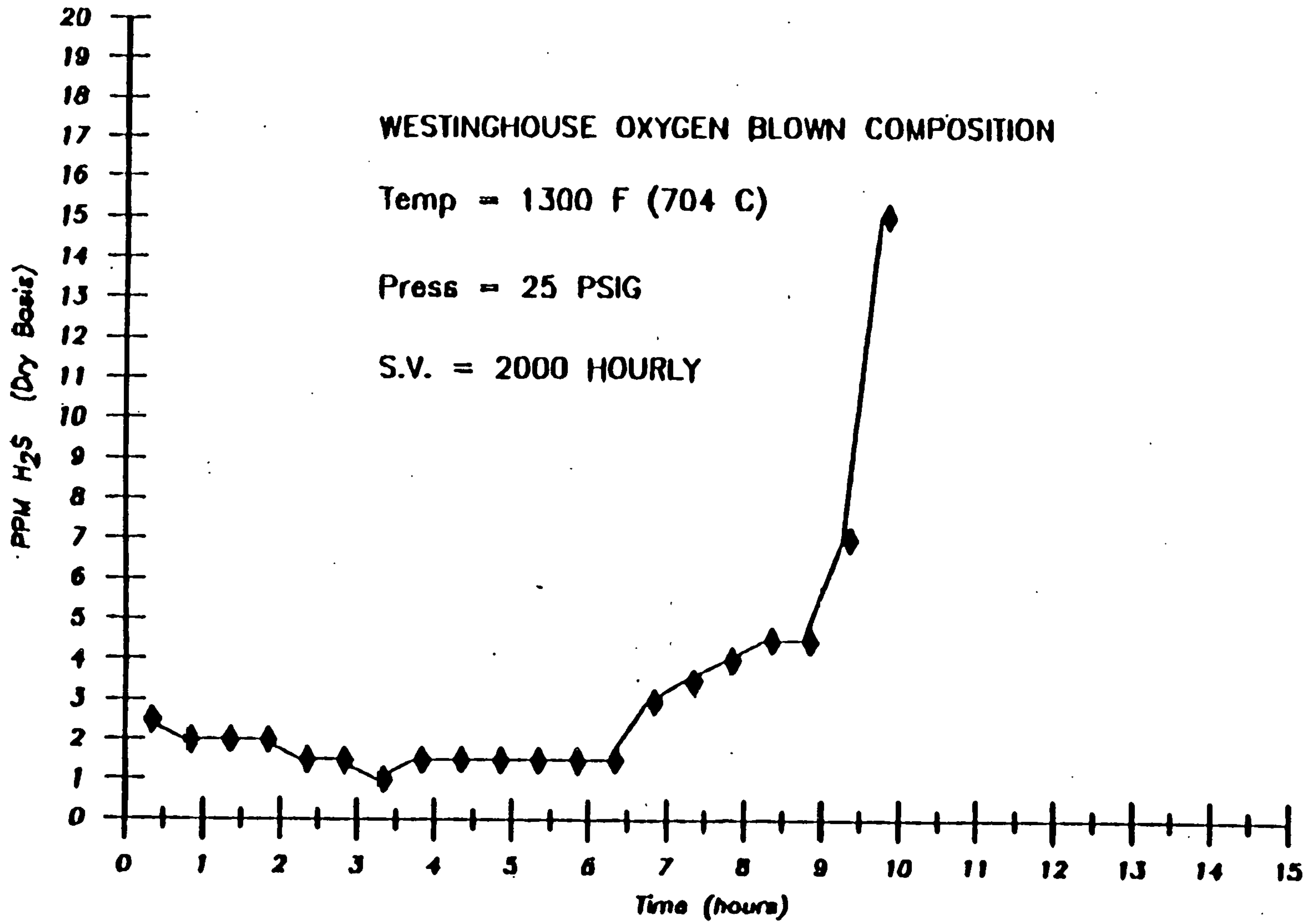


14-SEP-84 14:56:04

FIGURE 18

ZINC FERRITE SULFIDATION

LAB SCALE TEST UNIT TEST 006

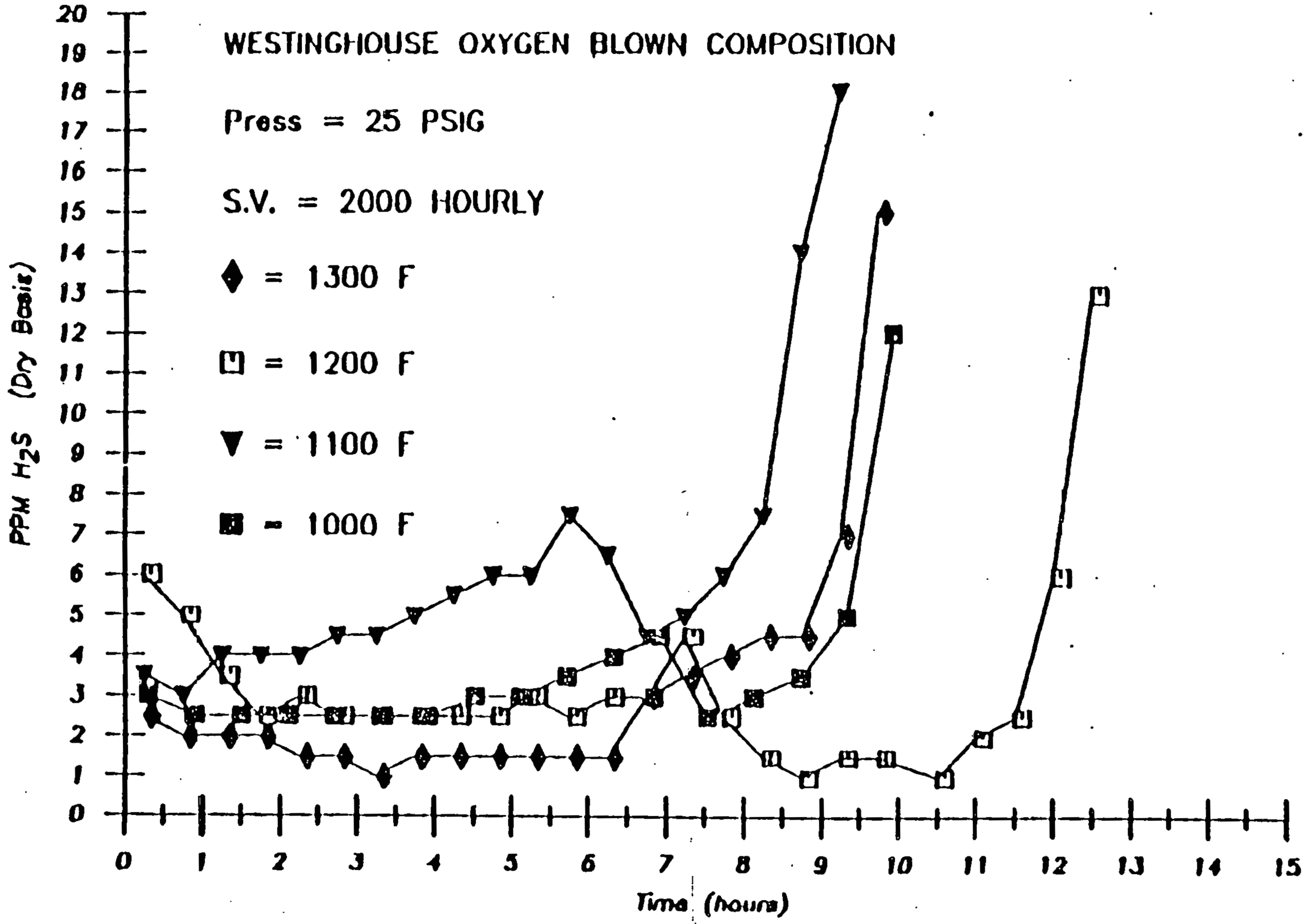


13-SEP-84 11:56:40

FIGURE 19

ZINC FE RITE SULFIDATION

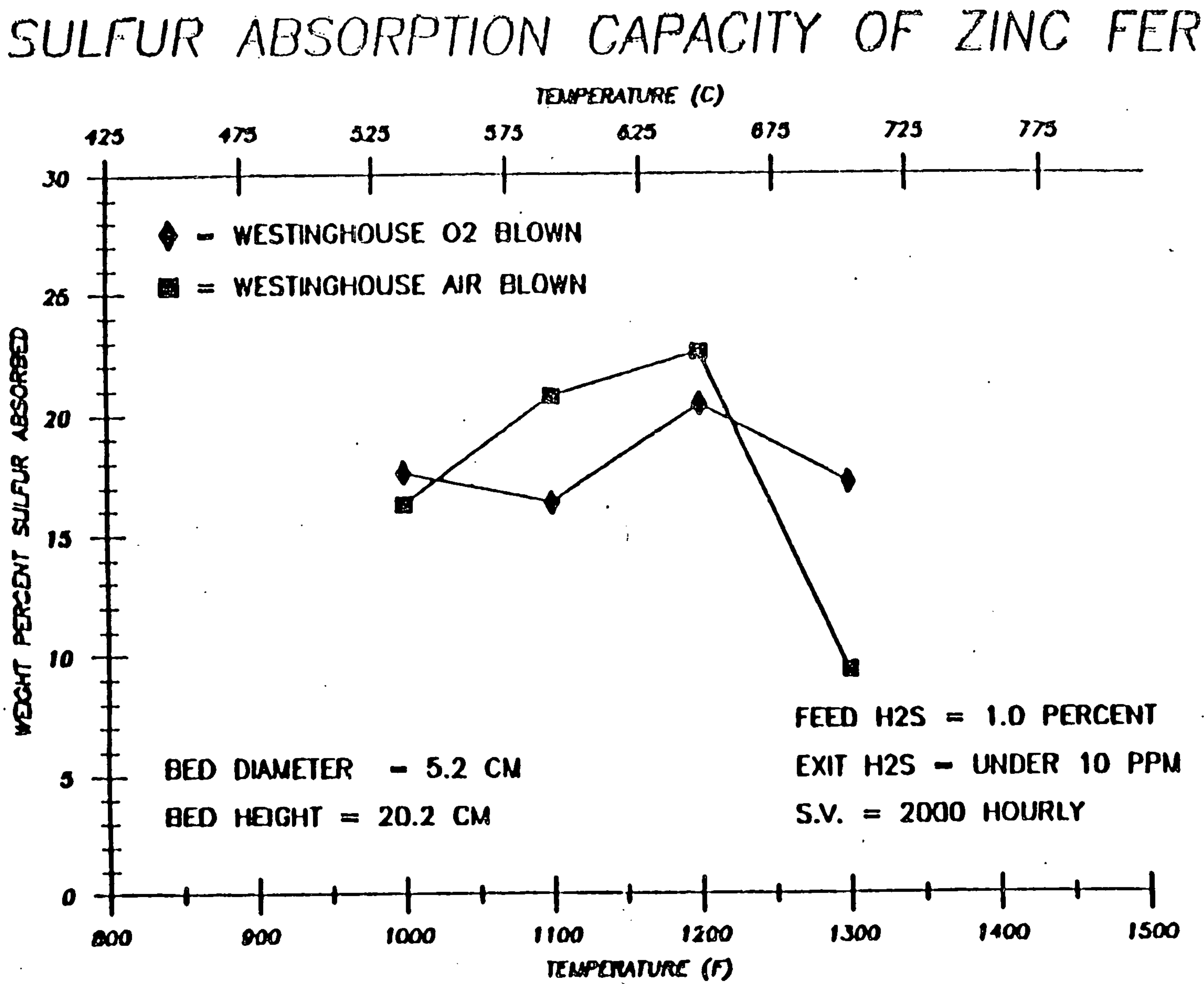
LAB SCALE TEST UNIT TESTS 003, 004, 005, 006



-780-

13-SEP-84 11:58:43

FIGURE 20



14-SEP-84 15:30:34

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FIGURE 21

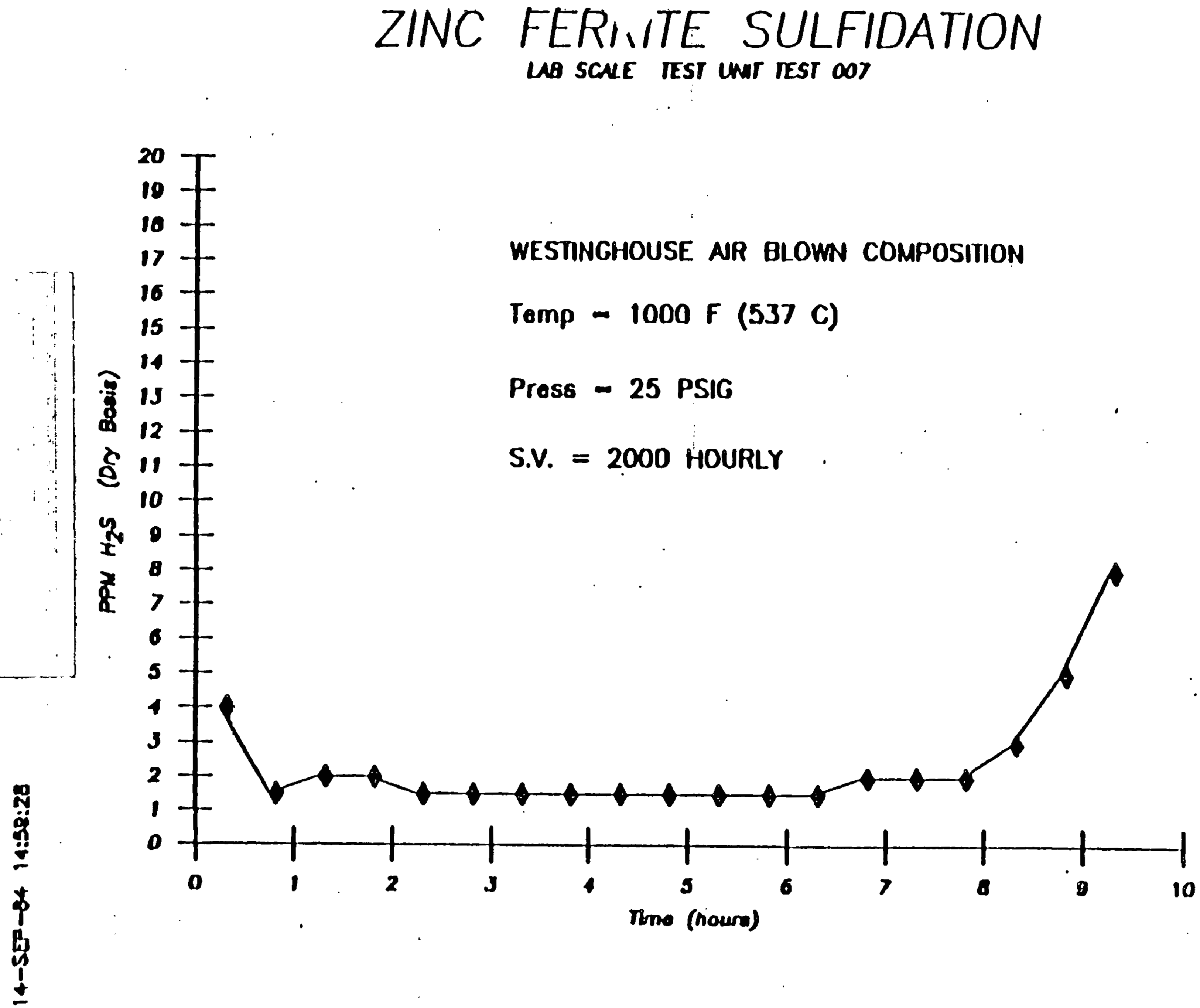

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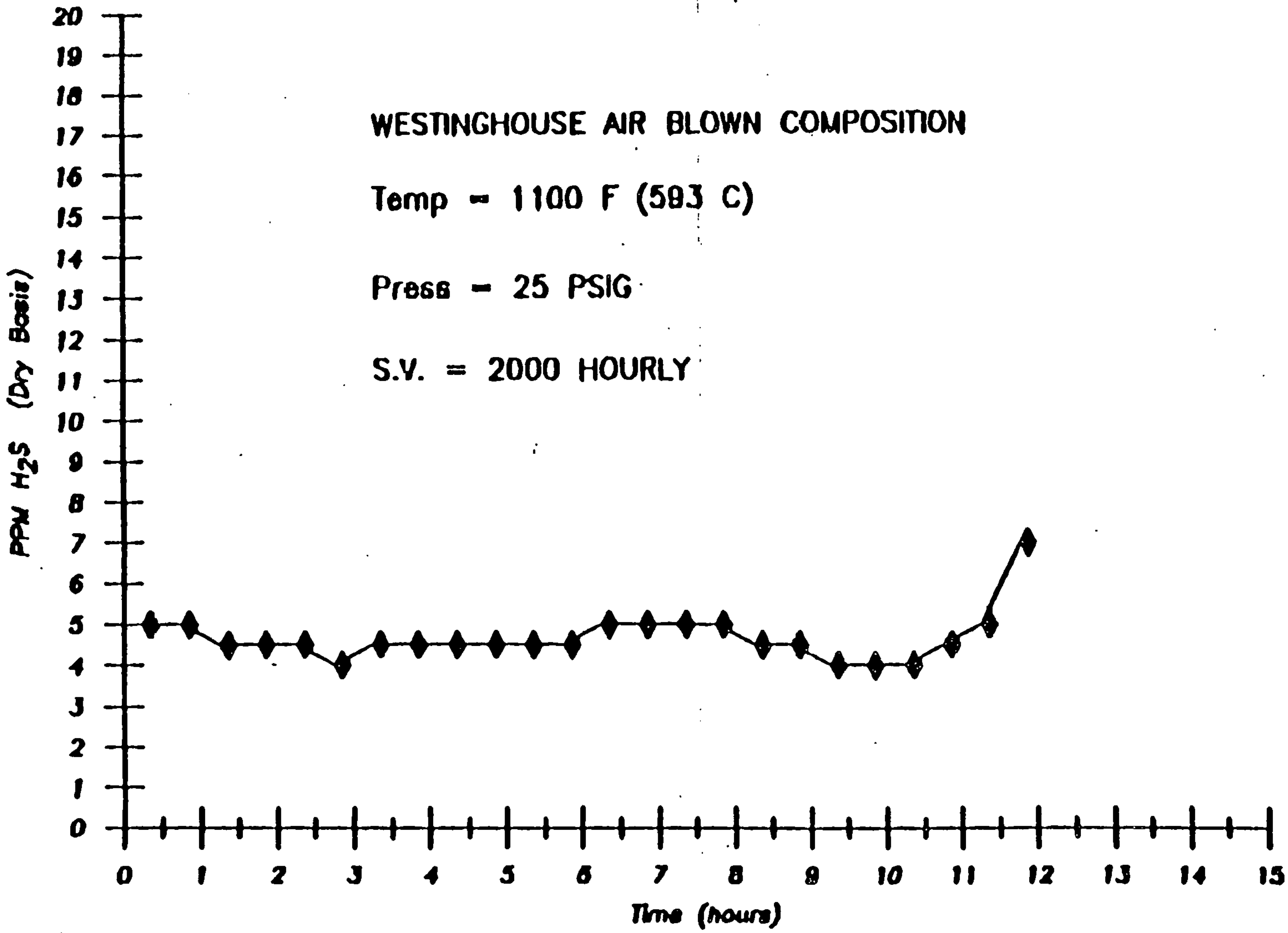
FIGURE 22

ZINC FERRITE SULFIDATION

LAB SCALE TEST UNIT TEST 008



14-SEP-84 08:31:13

FIGURE 23

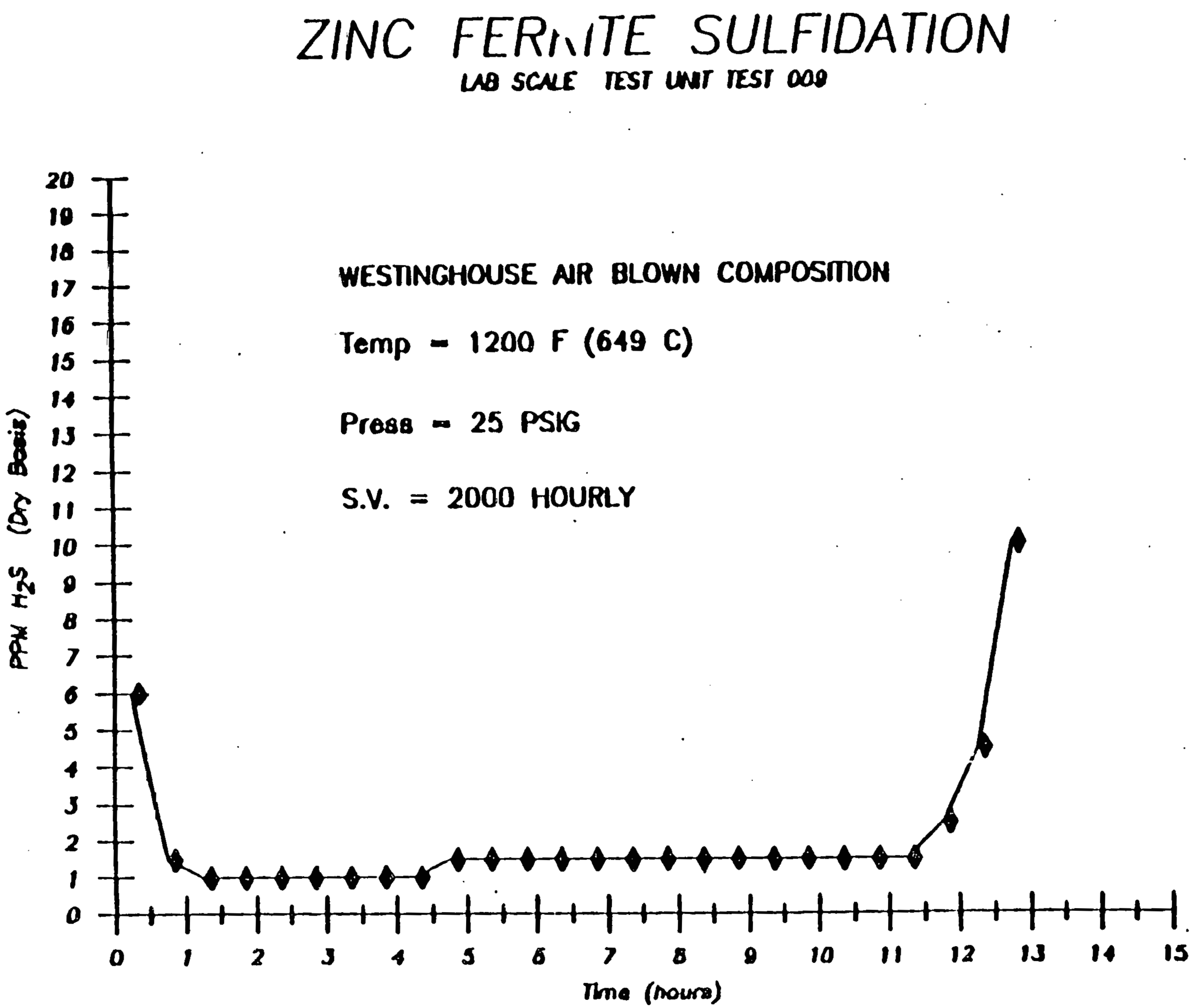


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FIGURE 24

ZINC FERRITE SULFIDATION

LAB SCALE TEST UNIT TEST 010R

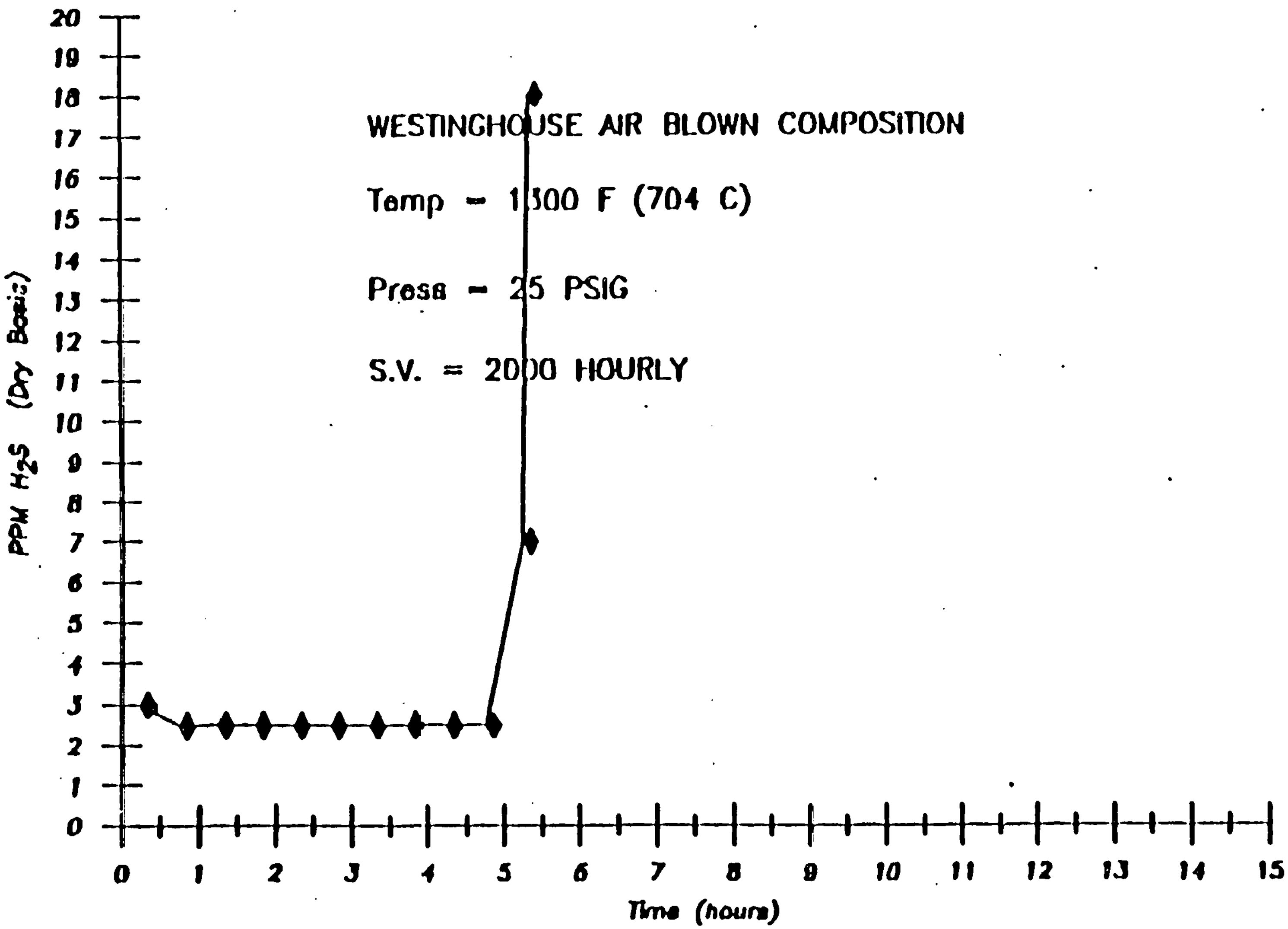

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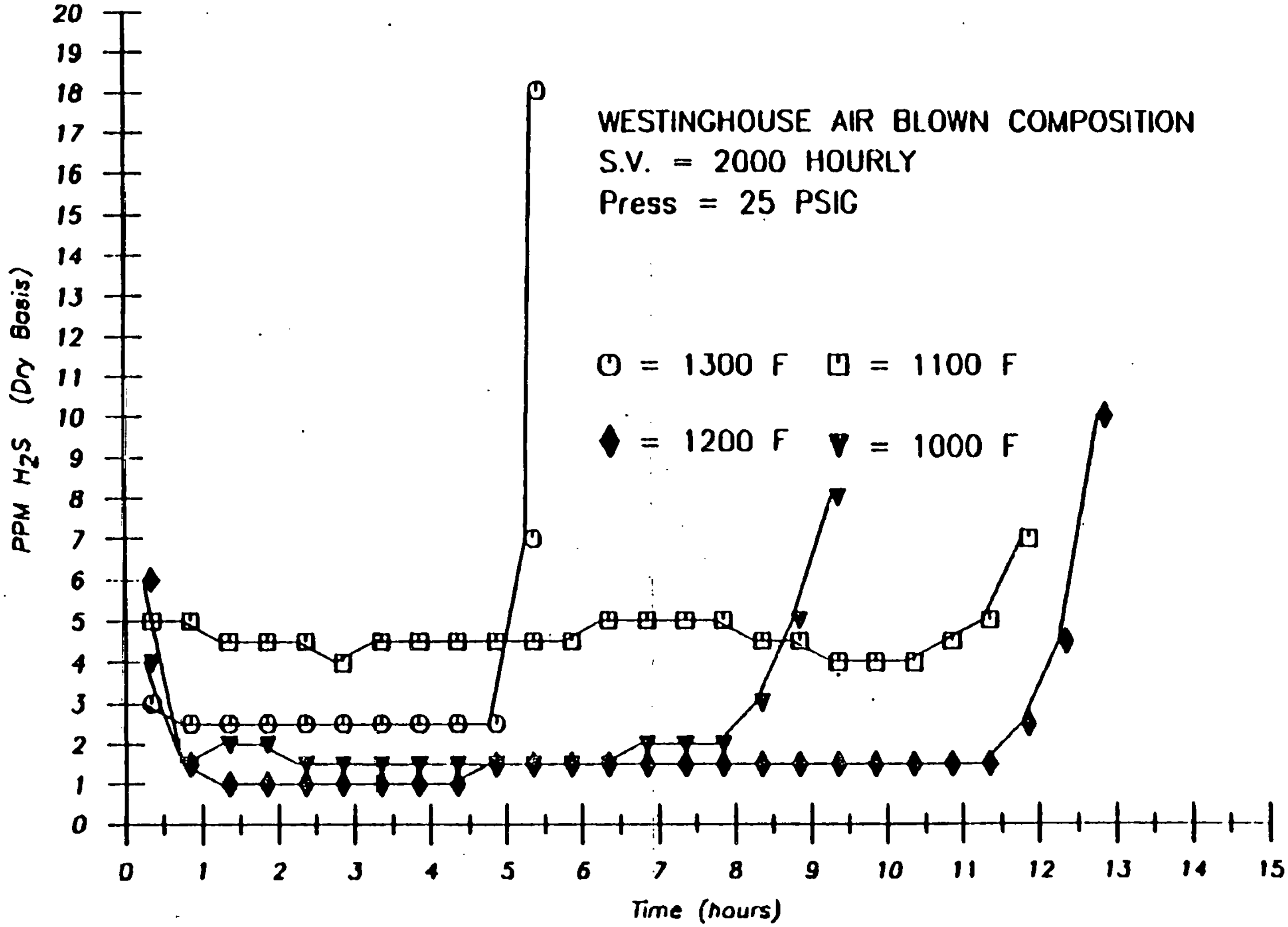
FIGURE 25

ZINC FERRITE SULFIDATION

LAB SCALE TESTS 007, 008, 009, 010R



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Feasible Gasifiers Gas Stream Compositions for Use With METC's Zinc Ferrite Sulfur Removal Process in a Minimally Cleaned Gas, Combined-Cycle Plant Configuration

Objective/Summary

This report summarizes available information on the above topic, specifically with regard to the effect of fuel gas composition and temperature on the composition of the sorbent. The major species content of the fuel gas, i.e., carbon monoxide (CO), carbon dioxide, and water (H_2O), affects sorbent composition and this fact may have significant implications on the operability of the zinc ferrite ($ZnFe_2O_4$) hot-gas desulfurization process in the configuration used in the METC research program. However, there are uncertainties in much of the data leading to these conclusions. Design options exist which can address the operability concerns and which, as far as is known, do not lead to significant changes in the overall performance or cost of a minimally cleaned gas power generation plant. Both the system effects and the design options require further study.

This report (a) describes METC's research which led to identification of gas composition as a potential concern in zinc ferrite system operation; (b) examines the fuel gas from commercial or near-commercial gasifiers and gives an estimate of the degree of concern for these gasifiers; (c) describes potential changes in either the sorbent system design or the gasifier/fuel gas system to address the concern; and (d) enumerates the remaining uncertainties.

Problem Definition

In the zinc ferrite hot-gas desulfurization process, both the fuel gas composition and the process operating temperature affect sorbent composition. On the basis of results from the zinc ferrite research program currently underway at METC, i.e., a program conducted using 3/16-inch extruded pellets in a packed-bed configuration, the zinc ferrite process should operate at fuel gas stream conditions where magnetite (Fe_3O_4) is the stable iron species. This stability region is shown in Figure 1, which is an iron/carbon/oxygen equilibrium diagram computed for atmospheric pressure. The figure is plotted in terms of temperature and the ratio of CO to CO_2 expressed as the equilibrium concentration of $CO/(CO + CO_2)$. At 1,000°F, a $CO/(CO + CO_2)$ ratio of less than about 0.5 is required for the sorbent to be in the magnetite stable region of the equilibrium diagram. If the zinc ferrite system operates outside this region, species other than magnetite will be present in the absorption bed. These "other" iron species, i.e., iron/iron carbides and wustite (FeO), have deleterious effects on the performance of the zinc ferrite process as tested in the METC configuration, i.e., an extruded sorbent in a packed bed.

Two possible options are available to overcome these problems. One option is to operate the gasification/fuel gas system in a way which will circumvent the formation of iron/iron carbides or wustite in the sorbent bed. A second option is to develop an alternate sorbent or sorbent system to negate the deleterious effects. A process development activity is needed to choose the best approach. METC will sponsor work with a FY 84 procurement now "on the street" to address the research needed to better define the problem and provide a basis for further process development. Definitive phase equilibrium diagrams are one aspect of this research effort.

The unquenched fuel gas from an oxygen-blown dry ash Lurgi gasifier has a $CO/(CO + CO_2)$ ratio such that the zinc ferrite system will operate in the magnetite region of the equilibrium diagram. The unquenched fuel gas projected for the oxygen-blown commercial version of the Kellogg/Rust Westinghouse (KRW) gasifier is such that a zinc ferrite system which operates at 1,000°F and uses the METC process configuration will be on the borderline of the magnetite stable region. However, when treating unquenched fuel gas from either the BGC/Slagger or the oxygen-blown Texaco gasifier in a zinc ferrite process with the METC configuration, it will be necessary to increase the steam content of the fuel gas stream in order to move the system into the magnetite stable region. Increasing the steam content of the fuel gas reduces the amount of CO because of the CO shift conversion reaction,

$$H_2O + CO \rightarrow H_2 + CO_2.$$

The shift reaction is catalyzed by zinc ferrite so that equilibrium, or near equilibrium, should be achieved in the bed.

The following are possible options for increasing the steam content of the fuel gas:

1. Water quenching a high-temperature fuel gas stream (e.g., fuel gas from a KRW or Texaco gasifier).
2. Adding high-temperature steam to a lower temperature fuel gas stream (e.g., fuel gas from a BGC/Slagger).
3. Operating the gasifier at a higher steam-to-air ratio.

Effect of Increasing the Moisture Content of the Fuel Gas on System Performance

Although sulfur absorption is affected by the moisture content in the fuel gas, increasing the moisture content to the desired level would result in only a very slight decrease in the sulfur removal levels achievable in the zinc ferrite process. Sulfur concentrations as low as 5 ppm could be obtained in a fuel gas stream with the moisture content contemplated.

It was estimated that adding moisture to the fuel gas of a minimally cleaned gas system would have only a very small effect on overall cycle efficiency or system cost. However, the mass and energy balances for a process design in which moisture was added to the fuel gas would be different from those of a nonmoisturized design.

The addition of steam to the fuel gas would have some effect on the performance of the turbine combustor in a gasification combined-cycle plant. The additional steam would tend to reduce the thermal NO_x formation in the gas turbine combustor but probably will have no effect on fuel-bound nitrogen NO_x production (which apparently is the greater concern in minimally cleaned gas systems). The additional steam would also affect the flammability characteristics of the gas. The steam reduces the heating value of the gas on a Btu/scf wet basis. This may be a concern with gasification systems which already produce a relatively low-heating value gas, e.g., air-blown fluidized-bed or entrained flow gasifiers. The CO shift reaction further reduces the lower heating value of the fuel gas, but the reaction also increases the hydrogen content of the gas. The General Electric water-cooled test program showed that the flammability characteristics of low-Btu fuel gas are sensitive to the hydrogen content of the gas, being improved by a higher hydrogen content.

Phase Equilibrium Diagram Data

Iron/carbon/oxygen phase equilibrium diagrams are widely available due to their connection with the steel making industry. Equilibrium diagrams are also used in connection with iron catalysts which are used in various chemical processes. Most of the diagrams for iron chemistry have been generated for atmospheric pressure, but similar effects are expected for zinc ferrite systems operating at pressure.

It is noted that the phase equilibrium diagram shown in Figure 1 does not take into account the presence of sulfur nor does it include the effect of reaction kinetics. The relative importance of omitting sulfur is unknown. However, one opinion is that it is minimal since, under gas compositions favoring iron/iron carbides formation, the sulfur would be absorbed in the reaction zone with the remainder of the bed converted to iron/iron carbides before the reaction zone approaches it.

Therefore, while there is some certainty as to the general trends shown in Figure 1, the application of the diagram should not be considered definitive in this early stage of the process design. There may be uncertainty as to the absolute location of specific phase separation boundaries, particularly when operating at pressure. More definitive phase equilibrium diagrams for the zinc ferrite system at the operating pressures of gasifiers/gas stream cleanup systems should be available within approximately one year. Responses to an RFP on this topic are currently being assessed at METC.

Validation of Phase Diagram Data at METC

Two data points on the phase diagram were confirmed for pressure operation during the zinc ferrite research program conducted using a side stream of METC's 42-inch fixed-bed gasifier. The first data point confirms that, for temperatures in the 1,000 to 1,100°F range, the METC zinc ferrite process typically operated in the iron/iron carbides stable region of the phase diagram. The presence of iron/iron carbides in the sorbent bed was first suspected during gasifier Run 102 (September 1983), and this was confirmed in post-test mineral analysis of the sorbent material. It is noted that even though the METC process was operating in the iron/iron carbides region, gas-phase sulfur levels on the order of 5 ppm were routinely achieved. The problem encountered was physical sorbent disintegration (discussed below).

An additional data point on the phase diagram was confirmed following gasifier Run 104 (December 1983). The zinc ferrite bed operated in the 1,200 to 1,300°F temperature range during this run, and wustite was found in the sorbent material following the test. The phase diagram predicts wustite formation at this operating temperature.

The sorbent phase equilibrium results from the METC gasifier are not applicable to dry ash Lurgi gasifiers. The METC gasifier was operated at low steam-to-air ratios (approximately 0.2 wt./wt.), a large fraction of the heat liberated during the gasification process being removed from the gasifier by a water wall. A dry ash Lurgi has a significantly lower water wall surface-to-bed volume ratio. Thus the oxygen-blown Lurgi, which typically operates at a steam-to-air ratio of 0.5 wt./wt., produces a fuel gas with a higher steam content. With this fuel gas composition, the zinc ferrite process would operate in the magnetite stable region of the phase diagram.

Process Chemistry Specifics

When the zinc ferrite process operates in the magnetite region of the iron-carbon-oxygen equilibrium diagram, the chemical reactions taking place during sulfidation and regeneration may be represented as follows.

Sulfidation

$$3ZnFe_2O_4 + H_2 \rightarrow 3ZnO + 2Fe_3O_4 + H_2O$$

$$Fe_3O_4 + 3H_2S + H_2 \rightarrow 3FeS + 4H_2O$$

$$ZnO + H_2S \rightarrow ZnS + H_2O$$

Regeneration

$$2FeS + 7/2O_2 \rightarrow Fe_2O_3 + 2SO_2$$

$$ZnS + 3/2O_2 \rightarrow ZnO + SO_2$$

$$ZnO + Fe_2O_4 \rightarrow ZnFe_2O_4$$

In the iron/iron carbides region of the phase diagram, the zinc chemistry is thought to be identical to that shown above. The overall iron chemistry during the sulfidation process may be represented as follows.

$$Fe_3O_4 + 4H_2 \rightarrow 3Fe + 4H_2O$$

$$3Fe + C \rightarrow Fe_3C$$

$$3Fe + H_2S \rightarrow FeS + H_2$$

In the wustite stable region, the following iron reactions are thought to occur during the sulfidation process.

$$Fe_3O_4 + H_2 \rightarrow 3FeO + H_2O$$

$$FeO + H_2S \rightarrow FeS + H_2O$$

Concerns Over Operating Outside the Magnetite Stable Region

The data collected on the METC packed-bed zinc ferrite reactor indicate that the iron/iron carbides and the wustite stable regions should be avoided for the following reasons:

1. Reducing the iron oxide to elemental iron results in a porous sorbent pellet which is more prone to physical disintegration.

Based on experience with high-temperature shift catalysts (which are iron oxides and, therefore, similar to the zinc ferrite sorbent material), a life in excess of one year could reasonably be projected for the zinc ferrite sorbent. Circumstantial evidence during METC's zinc ferrite test program indicates that physical disintegration of the sorbent could lead to a significantly shorter sorbent lifetime under the adverse circumstances of excessive sorbent reduction and coke formation discussed in Item No. 2, below.

The following data gives some feel for the degree of sorbent disintegration experienced at METC. Following two separate zinc ferrite tests conducted during METC gasifier Run 102, the sorbent material was removed from the bed with a vacuum hose and screened. (These processes obviously involve the potential for generating additional fines.) The sorbent material from one test was subjected to two complete absorption and regeneration cycles prior to vacuuming/screening and was found to contain 2 percent fines. The sorbent from another test was subjected to one and one-half cycles (i.e., absorption, regeneration, and absorption). This second sorbent, which was vacuumed/screened in the sulfidized form, was found to contain 30 percent fines. Since the bed was operating in the iron/iron carbides stable region of the phase diagram in both these tests, it may be inferred that the presence of iron/iron carbides diminished the physical strength of the sorbent, leading to increased sorbent attrition. A substantially smaller amount of fines (about 10 percent) was generated in a third test during gasifier Run 101. In this case, the sorbent was in a sulfidized form and had been operating in the magnetite stable region immediately prior to removal from the reactor. Sorbent durability data will be obtained as a result of an RFP currently being assessed at METC. The aim of this project is to identify techniques to enhance zinc oxide based sorbent durability and to demonstrate sorbent durability for 100 absorption/regeneration cycles.

Problems associated with physical disintegration might be circumvented by using an inert solid material as a support for the active sorbent. The Institute Francais du Petrole has carried out extensive research along these lines for zinc oxide based sorbents. During the earlier iron oxide research, METC found supported sorbents to be as effective as pure iron oxide when normalized for iron content, and to have durability advantages over nonsupported sorbents. For a packed-bed zinc ferrite process, the use of a supported sorbent would be a tradeoff between the benefits of a more durable sorbent and the penalties associated with an increased reactor volume. (Sorbent utilization would be expected to be fairly high in a zinc ferrite process which operated in the magnetite stable region. In laboratory scale tests conducted at METC using simulated fuel gas containing sufficient steam to be in the magnetite stable region, the zinc ferrite bed absorbed on the order of 30 percent sulfur by weight or, very roughly, 90 percent of the theoretical amount of sulfur.) For a continuous zinc ferrite process design which used moving or fluidized-bed reactors, it may be that supported sorbents are a practical and/or necessary solution to the problems associated with sorbent disintegration. However, it is too early in the zinc ferrite testing program to make definitive statements about either the optimum reactor configuration or sorbent form.

2. The presence of iron catalyzes the formation of coke both through the cracking of any tar that might be present in the fuel gas stream and through the Boudouard reaction:

$$2\ CO \rightarrow C + CO_2.$$

Coke formation is undesirable because it reduces the heating value of the fuel gas stream and can contribute to sorbent disintegration.

Coke buildup may also contribute to increased pressure drops through a packed-bed absorber which could result in lifting of the sorbent in an upwards-flow reactor. (A fluidized-bed reactor design would obviate the pressure buildup problem.)

3. The presence of iron also catalyzes the very exothermic methanation reaction,

$$CO + 3\ H_2 \rightarrow CH_4 + H_2O,$$

which could cause excessive temperatures in the sorbent bed.

In the METC tests, temperature excursions in the absorption bed were not a problem. The system operated with a gas composition which contained sufficient steam that significant methane formation would not be expected, even if the methanation reaction went to equilibrium. Also, the small scale of the reactor offered the opportunity for heat dissipation through the reactor walls.

4. Elemental iron further reacts to form iron carbides which are less reactive than magnetite. This limits the absorptive capabilities of the sorbent bed and H_2S breakthrough occurs earlier than it would if the bed were operating in the magnetite stable region. As a specific example, examination of sulfidized sorbent following one test conducted during METC gasifier Run 102 indicated that, at the inlet to the bed, approximately 1/2 of the iron was in the form of iron carbides. Proceeding into the bed, greater amounts of iron had been converted to iron carbides. From the middle of the bed to the outlet, virtually all of the iron was in the form of iron carbides. This indicates that, once the iron is converted to iron carbides, there is a significant fall in the rate of sulfur pickup. The overall sulfur loading of the sorbent was approximately 10 percent during this test.

During regeneration, the iron carbides participate in the steam-iron reaction with the consequent formation of hydrogen. This reaction results in a regeneration tail gas containing a variety of species: H_2, H_2S, SO_2, elemental sulfur, etc. Such a tail gas would need to be incinerated when using a sulfur recovery process which is designed to remove oxidized sulfur compounds. In the absence of iron/iron carbides, the predominant species in the regeneration tail gas is expected to be sulfur dioxide (SO_2) with minor amounts of sulfur trioxide (SO_3).

A possible option is to treat the mixed composition tail gas stream by incinerating it and removing its sulfur content in a fluidized bed containing a throwaway, calcium-based sorbent material.

5. The wustite stable region of the phase diagram should also be avoided if very high sulfur removal is required. The absorptive powers of wustite are significantly lower than those of magnetite. For example, in a zinc ferrite test conducted during METC gasifier Run 104, H_2S levels in excess of roughly 100 ppm were observed almost immediately following the initiation of the absorption cycle. This test was conducted with bed temperatures of 1,200 to 1,300°F, and, therefore, the process was operating in the wustite stable region of the phase diagram. It is possible that the presence of wustite in the sorbent structure makes the zinc oxide less accessible.

<u>System Implications for the BGC/Slagger/Zinc Ferrite System</u>

The BGC/Slagger exit gas composition is well into the iron/iron carbides stable region of the phase diagram. Therefore, some modification to the fuel gas is required when operating with the METC zinc ferrite configuration. It was calculated that adding 20 vol. percent (based on the total gas flow) of steam to the fuel gas would move the phase equilibrium to the boundary of the magnetite stable region. Reference 1 was used as a source for the raw fuel gas composition for these calculations. The $CO/(CO + CO_2)$ ratio was determined at shift equilibrium. The shift equilibrium was recalculated following the injection of the additional steam. Adding an additional 5 vol. percent of steam would increase the margin of safety and move the system away from the uncertain boundary area.

Steam to moisturize the fuel gas could be obtained from the heat recovery steam generator. Adding steam to the gasifier exit gas (typically at temperatures in the 800 to 850°F range) would raise the gas stream temperature through the heat released in the exothermic CO shift conversion reaction. Each 1 percent of CO converted would raise the gas stream temperature roughly 18°F. Therefore, adding 20 mole percent of 640°F steam to the fuel gas could be used to increase the gas stream temperature to approximately 1,000°F. This temperature increase for the fuel gas stream is desirable because it diminishes the possibility of tar condensation in the cleanup system.

The presence of a catalyst is apparently required for the shift reaction to proceed at temperatures below 1,200°F. While detailed homogeneous kinetic rate data for the shift reaction have not been located, Reference 2 indicates that the extent of conversion is not significant without catalysis at temperatures below 1,200°F. The CO shift conversion reaction is expected to go to virtual equilibrium in the relatively low-temperature BGC/Slagger fuel gas stream because of catalysis by the sorbent.

It appears more feasible to add steam to the fuel gas at the gasifier exit than to perturb the gasifier operation. Increasing the steam-to-air ratio in the gasifier could be used to increase the moisture level in the fuel gas but it would have significant effects on the operation of the gasifier. Specifically, it might cause the gasifier to operate in a dry ash mode. Increasing the steam feed to the gasifier would also increase the superficial velocity through the gasifier. Higher velocities may not be acceptable from an operational standpoint since they could lead to channeling in the bed and/or greater fine carryover. The additional steam may also negate the greater throughput advantage of the BGC/Slagger over the dry ash Lurgi. These comments are speculations on which opinions should be sought from industrial sources.

System Implications for the KRW Gasifier/Zinc Ferrite System

The fuel gas produced in the KRW process demonstration unit (PDU) is such that the equilibrium is well into the magnetite region of the phase diagram. (In the PDU, KRW partially quenches the gas to avoid deposition problems in the cyclones.) With the fuel gas composition projected for a commercial oxygen-blown version of the KRW gasifier (Reference 1), the CO/CO_2 ratio is such that the system would be on the boundary of the magnetite stable region at 1,000°F if the gas were cooled from gasifier operating temperatures without the addition of water. Thus, a conservative design would inject a small amount, 5 vol. percent of total gas flow, of water into the fuel gas stream.

Adding a small amount of water to the approximately 1,800 to 1,900°F fuel gas stream should not present any major design problems. However, there may be some difficulty in controlling the water quench flow rate, such that the desired fuel gas temperature is obtained. In a commercial design, either of two methods could be used to cool and add the required moisture to the fuel gas. In one method, all the high-temperature heat exchangers would be eliminated, and the fuel-gas stream temperature would be lowered from the 1,800 to 1,900°F range to the operating temperature of the zinc ferrite system, by using a water quench. (This approach would significantly increase the steam content of the fuel gas above the requirements of the zinc ferrite process.) Alternatively, a partial quench to achieve the desired fuel-gas moisture content could be employed at some point in the temperature reduction process. Radiant and/or convective heat exchangers would be used to complete the temperature reduction.

System Implications for a Texaco Gasifier/Zinc Ferrite System

The fuel gas from a Texaco gasifier (Reference 1) would require injection of approximately 10 vol. percent of water for the system to reach the boundary of the magnetite stable region on the phase diagram at 1,000°F. A 15 percent water injection rate would be a conservative design. The considerations in adding water to the Texaco gasifier are similar to those discussed above for the KRW gasifier.

Inlet Particulate Limitations for the Zinc Ferrite Process

The METC zinc ferrite reactor operates with acceptable reactor pressure drops, with an inlet particulate loading of 1 g/Nm^3, and a median particle size of approximately 13 microns. Zinc ferrite systems which use packed-bed reactors to treat fuel gases which have particulate loadings with parameters exceeding to treat fuel gases which have particulate loadings with parameters exceeding these values may require the use of some particulate removal device prior to the absorption bed.

Zinc ferrite pellets larger than the 3/16-inch diameter extrusion size presently used in the METC test program may be helpful in avoiding bed blinding problems in packed-bed reactors by providing larger interstitial spaces. Granular-bed filter theory predicts that larger spaces would increase the allowable particle size in the inlet stream to the bed. A pellet diameter of 5/16 inch may be the maximum size that would still allow adequate sulfur diffusion into the sorbent.

Other reactor designs, e.g., a fluidized-bed reactor, could bypass any problems of blinding by particulates. Alternatively, sulfur capture and particulate control could be combined in a granular-bed filter arrangement. Improvements in sorbent strength beyond that of the METC extrudate would be needed to go these routes. Development of sorbents to address such reactor design considerations has not been part of the research program at METC.

Conclusion

On the basis of experimental results and economic assessments completed to date, the METC configuration of the zinc ferrite process appears to be an attractive process for hot gas cleanup. Fuel gas composition requirements do not seem to present roadblocks to its systems applications. However, the process is in its early stages of development. Other hot-gas desulfurization sorbents, pellet forms, and/or reactor configurations may also be viable options for the desulfurization of fuel gas in minimally cleaned gas, combined-cycle plants.

Acknowledgements

L. C. Headley, J. S. Halow, V. P. Kothari, J. M. Thames, J. E. Notestein, and L. E. Graham participated in discussions, contributed information, and provided reviews of draft versions of this report. Their contributions are gratefully acknowledged.

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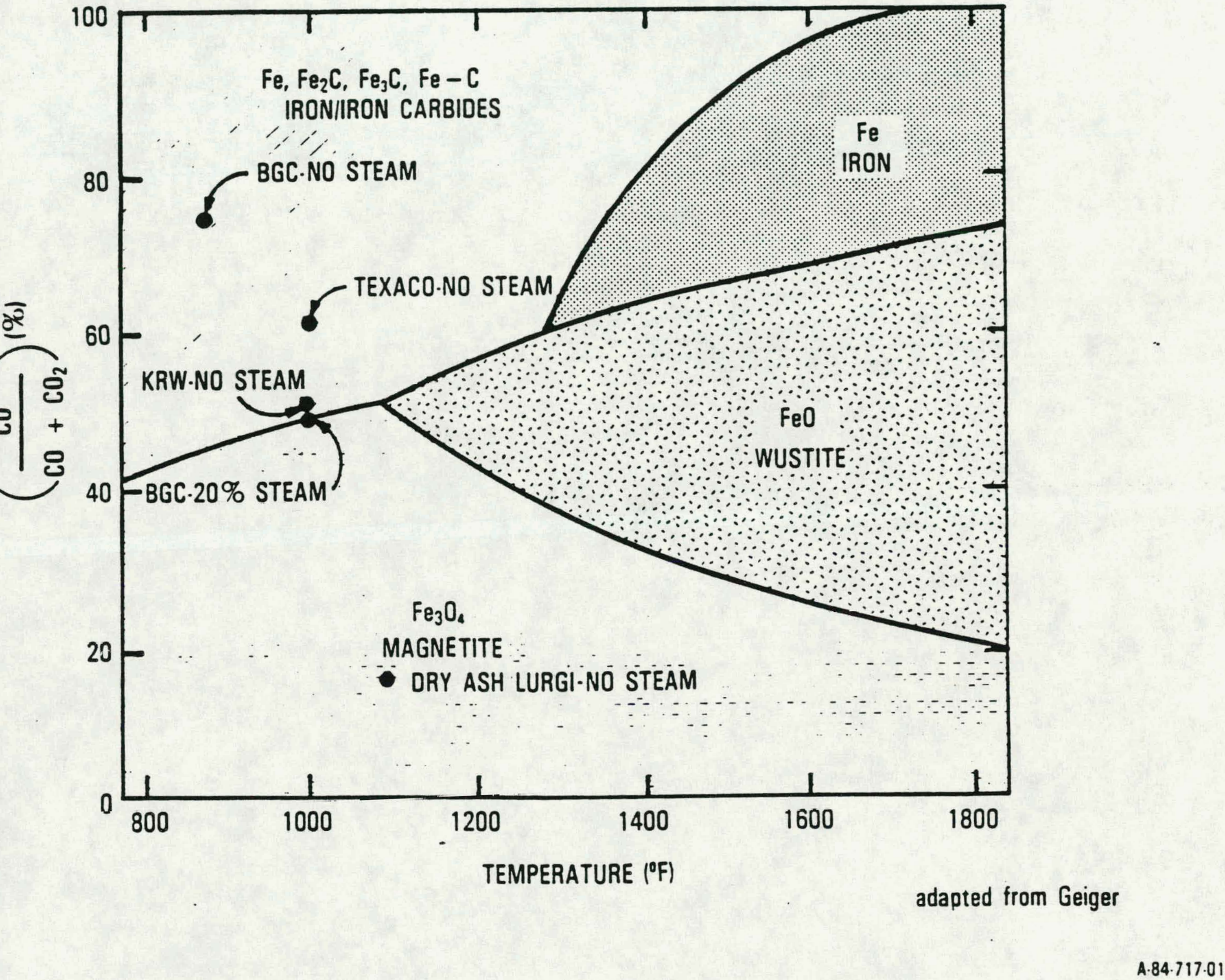

FIGURE 1. Iron-Carbon-Oxygen Equilibrium Diagram at Atmospheric Pressure