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REMOVING HYDROGEN SULFIDE FROM SYNTHESIS GAS WITH IRON OXIDE AT ELEVATED PRESSURE

By G. E. Johnson, J. H. Field, W. A. Decker,
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UNITED STATES DEPARTMENT OF THE INTERIOR
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REMOVING HYDROGEN SULFIDE FROM SYNTHESIS GAS WITH IRON OXIDE AT ELEVATED PRESSURE¹

by

G. E. Johnson,² J. H. Field,³ W. A. Decker,⁴ and R. M. Jameson⁵

SUMMARY

A bench-scale study was made of removing hydrogen sulfide from synthesis gas at elevated pressure by iron oxide impregnated on wood chips. The gas contained 0.02 to 0.06 percent oxygen.

The removal of hydrogen sulfide by iron oxide impregnated on wood chips is more efficient at elevated pressure. At constant space velocity, longer periods of operation at 400 p.s.i.g. are possible before the exit gas reaches a given maximum concentration of hydrogen sulfide, compared with 200 p.s.i.g. or atmospheric pressure.

In tests at room temperature, 60° C., and 100° C., the most effective removal of hydrogen sulfide was obtained at room temperature and the least effective at 100° C.

Dry impregnated wood chips removed only about half as much hydrogen sulfide as chips wetted to a water content of 50 percent.

At constant space velocity, extending the bed depth from 6 to 24 inches while maintaining the same bed volume permitted two to three times as much hydrogen sulfide removal for the same degree of purification.

Halving the quantity of hydrogen sulfide in the feed gas enabled from 5 to 50 times as much gas to be purified to the same degree of purity.

Spent iron oxide was revived by passing air at atmospheric pressure through the bed of spent chips.

¹Work on manuscript completed October 1961.

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INTRODUCTION

The gas industry of 1961 has the inherent problem of removing hydrogen sulfide before either natural or manufactured gas can be sold commercially. In the past it had been the custom to cap off or abandon gas wells that contained high amounts of this impurity, but with modern methods of purification and with demand for natural gas approaching supply, recovering gas from sour wells has become economical. Twenty to thirty percent hydrogen sulfide in raw gas is not uncommon in the United States, and these percentages are even higher in Canada. Before it can be sold, natural gas must meet pipeline specifications of 0.25 grain of hydrogen sulfide per 100 cubic feet.

When sulfur-containing coal is gasified to produce synthesis gas, most of the sulfur is converted to hydrogen sulfide. The allowable sulfur limit for synthesis gas to be used in the Fischer-Tropsch synthesis is 0.1 grain or less per 100 cubic feet, owing to the poisoning effect of sulfur on the catalyst.

Hydrogen sulfide-bearing gases can be purified by wet processes such as ethanol amine or hot potassium carbonate or by a dry process such as iron sponge boxes.⁶ The Bureau of Mines developed the hot carbonate scrubbing process^{7 8} for removing both carbon dioxide and hydrogen sulfide. An economical method for purifying synthesis gas to Fischer-Tropsch requirements involves bulk removal of carbon dioxide and hydrogen sulfide with hot carbonate followed by final purification with iron oxide sponge to remove the residual hydrogen sulfide. Because the gas would be used at elevated pressure, it is produced at 300 to 400 p.s.i.g., and the final cleanup would be advantageously performed at this pressure.

Few data were available on removal of hydrogen sulfide by iron oxide at elevated pressure.^{9 10} Consequently, a study was begun at the Bureau of Mines Bruceton laboratories to explore variables affecting the absorption. In addition to pressures of 1 atmosphere, 200 p.s.i.g., and 400 p.s.i.g., the following effects on hydrogen sulfide removal were investigated: Operating at elevated temperatures (60° and 100° C.), moisture on the wood chips, space velocity, extending the bed depth, revivifying the wood chips, and lowering the hydrogen sulfide content in the feed. The hydrogen sulfide concentrations arbitrarily chosen for this study were 5 and 10 grains per 100 cubic feet

⁶Zapffe, Fred, Three Ways to Purify Gas: Oil Gas Jour., Sept. 8, 1958, pp. 100-103.

⁷Benson, H. E., Field, J. H., and Jameson, R. M., CO₂ Absorption Employing Hot Potassium Carbonate Solutions: Chem. Eng. Prog., vol. 50, No. 7, 1954, pp. 356-364.

⁸Field, J. H., Johnson, G. E., Benson, H. E., and Tosh, J. S., Removing Hydrogen Sulfide by Hot Potassium Carbonate Absorption: Bureau of Mines Rept. of Investigations 5660, 1960, 19 pp.

⁹Turner, C. F., Iron Oxide Versus Liquid Treating for Purifying Natural Gas: Oil and Gas Jour., vol. 42, Sept. 23, 1944, pp. 191-199.

¹⁰Wenzell, L. P. Jr., Dressler, R. G., and Batchelder, H. R., Plant Purification of Synthesis Gas: Ind. Eng. Chem., vol. 46, 1954, pp. 858-862.

because a synthesis gas made from coal, would have undergone a previous hot carbonate purification and would normally contain 1 to 10 grains of hydrogen sulfide per 100 cubic feet. Inadvertently, the gas had 0.02 to 0.06 percent free oxygen, which in some instances improved the absorption by continuously revivifying the iron oxide.

In the British Isles and in Europe, iron oxide in the form of bog ore or pellets of highly reactive oxide are mixed with sawdust and lime for use in hydrogen sulfide removal.¹¹ In the United States iron oxide impregnated on wood chips is generally employed.

DESCRIPTION OF EQUIPMENT

The experimental work was conducted in the equipment shown in figure 1. The synthesis gas-hydrogen sulfide mixture was contained in high-pressure gas cylinders fitted with stainless steel cylinder valves. The cylinders had been internally lined with an impervious phenolic resin, which is unreactive with hydrogen sulfide. Synthesis gas containing the hydrogen sulfide flowed (fig. 2) from the cylinder via a pressure regulator at a rate controlled by a throttle valve. A valve allowed gas samples to be taken in the Tutwiler apparatus for hydrogen sulfide determination. After it had passed through the flowmeters, the gas flowed downward through the iron oxide absorbent. When it had left the absorber, part of the exit gas was analyzed for hydrogen sulfide content in the methylene blue apparatus. The remainder of the gas flowed through activated carbon to remove the residual hydrogen sulfide and then through a back-pressure regulator and gasmeter before it was vented to the atmosphere.

The 2.5-inch-outside-diameter (2.067-inch-inside-diameter) absorber was constructed of type 303 stainless steel. A stainless steel retaining screen supported the 6-inch bed of wood chips. The threaded end cap equipped with an O-ring seal to prevent leakage facilitated charging and discharging. In tests at elevated temperature, temperature control was provided by an oil bath equipped with a heater and electric magnetic stirrer in which the absorber was immersed.

Except for replacement of the absorber and exclusion of the oil bath heater, the same equipment was used for the tests with the 24-inch bed of wood chips. The absorber consisted of two 18-inch sections of 1-inch-diameter stainless steel pipe (type 304) with both ends threaded and fitted with O-ring seals. Because each 18-inch section contained two 6-inch beds of wood chips whose cross-sectional area was one-fourth that of the single 6-inch bed reactor, the total volume of the absorbent was the same for both absorption units. Each 6-inch wood-chip bed was supported by a stainless steel retaining screen. Gas ports were provided so that gas leaving each section of absorbent could be analyzed. Figure 3 is a sketch of the 24-inch absorber, and figure 4 is a flow diagram of the unit.

¹¹Myhill, A. R., Gas Purification and Sulfur Recovery by the Gas Technik Process: Coke and Gas, November 1958, pp. 454-471.

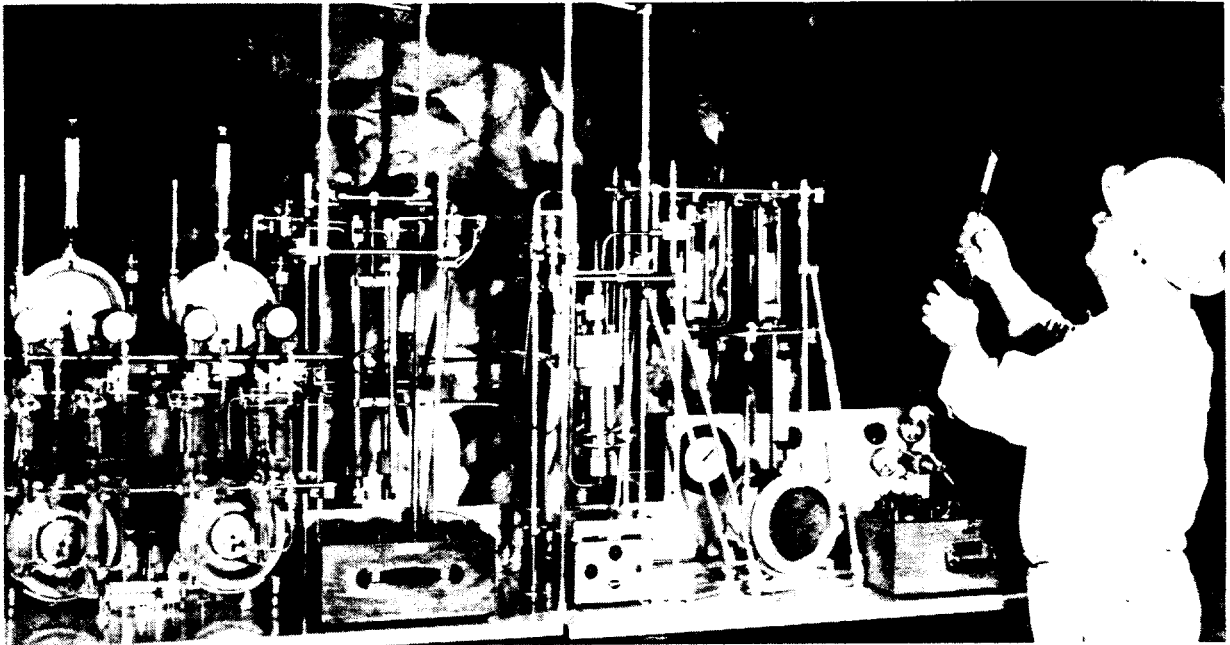


FIGURE 1. - Equipment Used in Study of H_2S Removal by Iron Oxide.

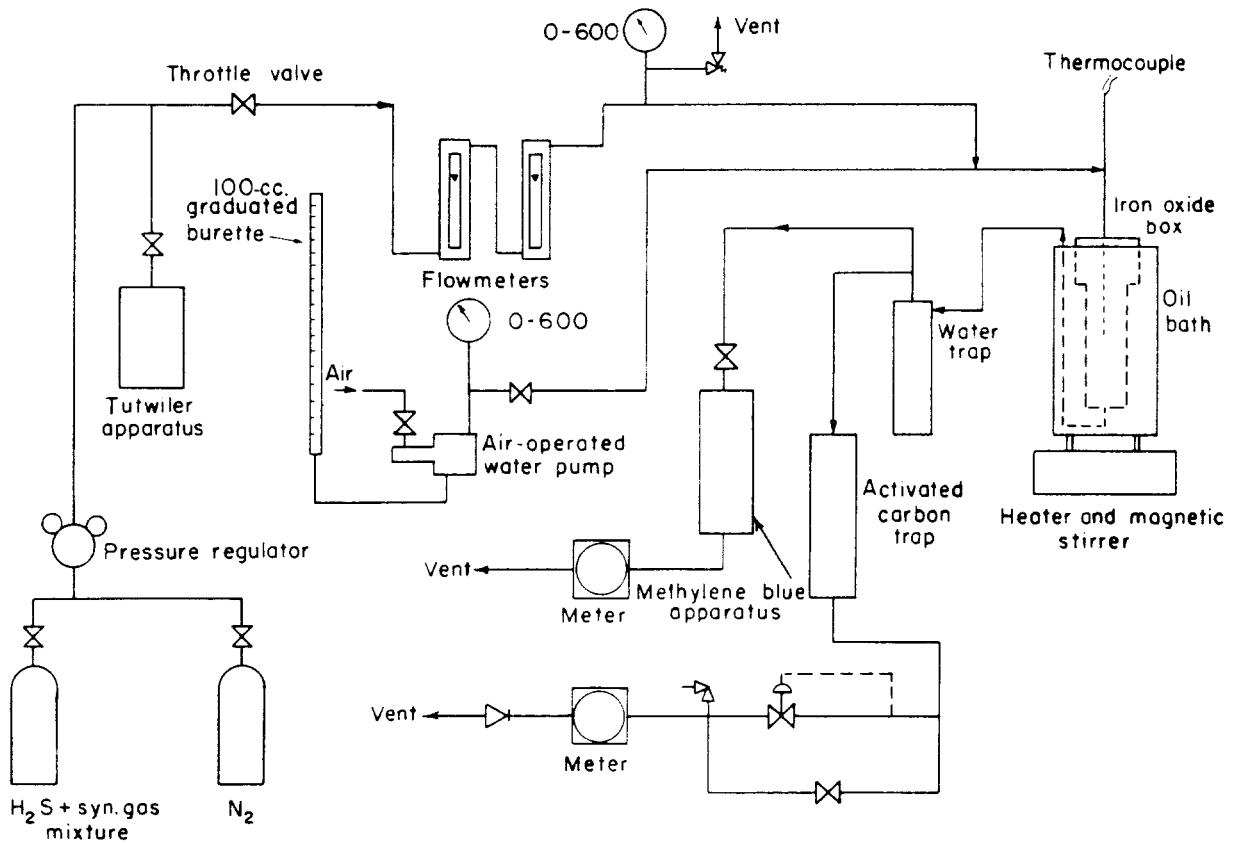


FIGURE 2. - Flow Diagram of Single-Bed Unit for H_2S Removal by Iron Oxide.

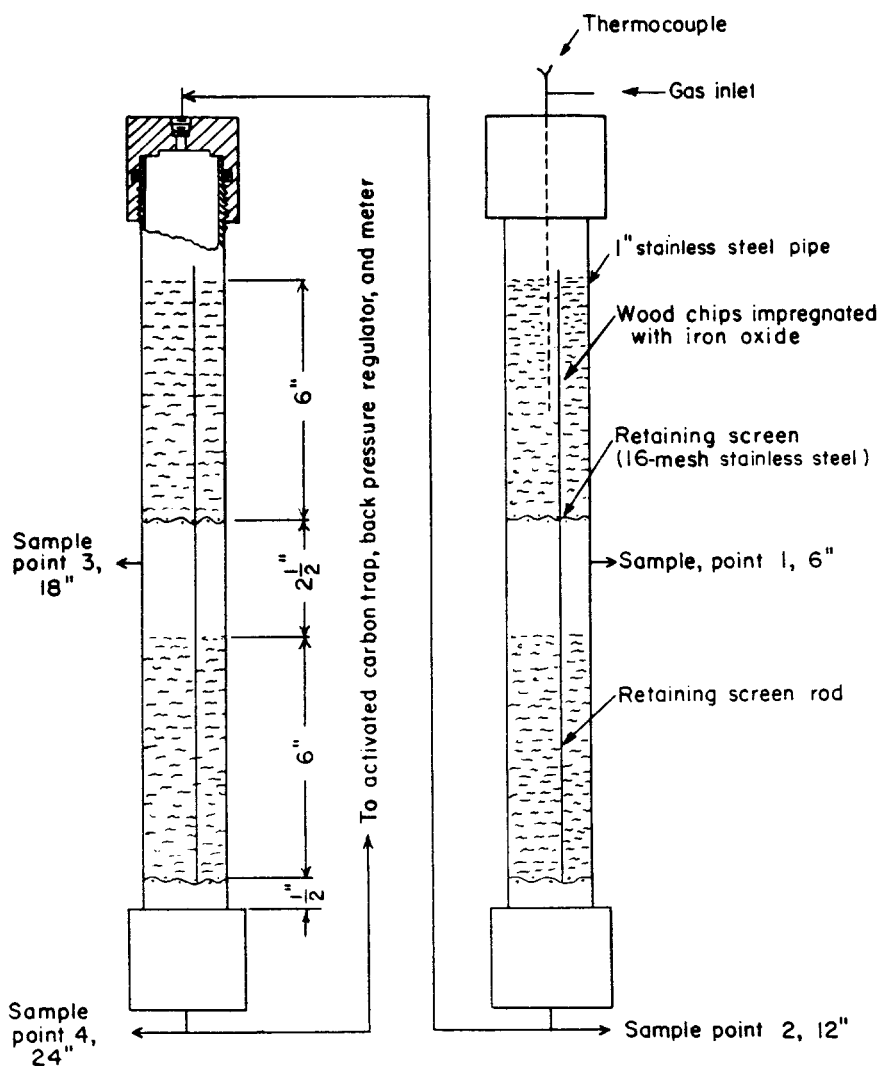


FIGURE 3. - Four-Bed Stainless Steel Absorber for H_2S Removal.

oxide bed to provide for measuring temperature. A nitrogen cylinder was provided for use in purging the system of air and for pressurizing to operating pressure before introduction of synthesis gas.

In the single-bed unit for the two tests, in which water was added to the feed gas, additional equipment was connected to the system--a visible-level trap ahead of the activated carbon trap for water condensation, and a burette-water reservoir supplying a positive-displacement microvolume pump that was designed by the Bureau of Mines.

A schematic diagram of the stainless steel bottling manifold for preparing feed-gas mixtures is shown in figure 5. All valves except cylinder valves were the stainless steel packless type to prevent gas leakage. A closed-end mercury manometer and a compound 30-inch vacuum 200 p.s.i.g. pressure gage, both with shutoff valves, allowed the system pressure to be read on the appropriate instrument. A vacuum pump was used to evacuate the manifold and

The gaslines subject to moderate or high pressures were of stainless steel, and those operated at low pressures were either aluminum or glass because the surfaces of these materials adsorb negligible quantities of hydrogen sulfide. Vinyl chloride plastic tubing was used for glassware connections. Two flowmeters were connected in series, one for indicating low and the other for indicating high feed-gas flows. Positive-displacement gasmeters filled with mineral oil were used to measure the flow of scrubbed gas and gas from the glass absorption pipets. System pressure was controlled by a back-pressure regulator.

A chromel-alumel thermocouple connected to a hand potentiometer was in the center of the initial iron

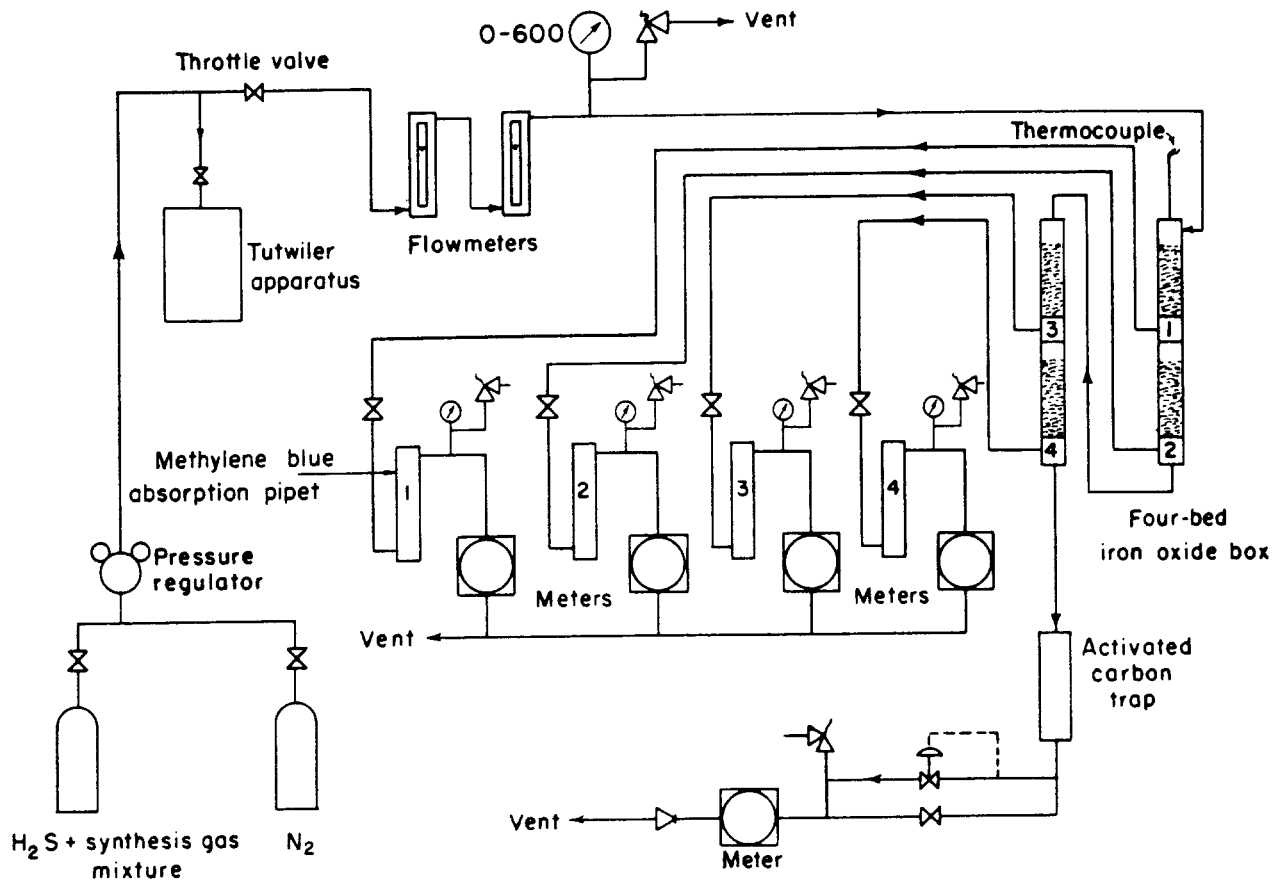


FIGURE 4. - Flow Diagram of Four-Bed Unit for H_2S Removal by Iron Oxide.

cylinders to be filled. A hydrogen sulfide cylinder regulator plus a synthesis gas cylinder and regulator equipped with an activated carbon trap for removing iron carbonyl and hydrogen sulfide complete the equipment used for preparing gas mixtures.

Absorbent

The iron oxide impregnated on wood shavings or wood chips used in this work was the commercial grade used by and obtained from a coke company in Philadelphia. One of the common methods of preparing the material is as follows: Gray cast-iron borings, such as those obtained from the automobile industry in Detroit, are treated in rotary kilns to burn off cutting oils. These iron chips are then crushed to a powder and sized to specific mesh ranges by screening. Generally the iron used for manufacturing iron oxide-purification material is of minus 40-mesh.

Weighed amounts of the metallic iron powder and water are combined with wood shavings in a mixer. Yellow pine shavings obtained from planer mills along the Atlantic seaboard are preferred because they are resilient before and after they have been impregnated with the iron.

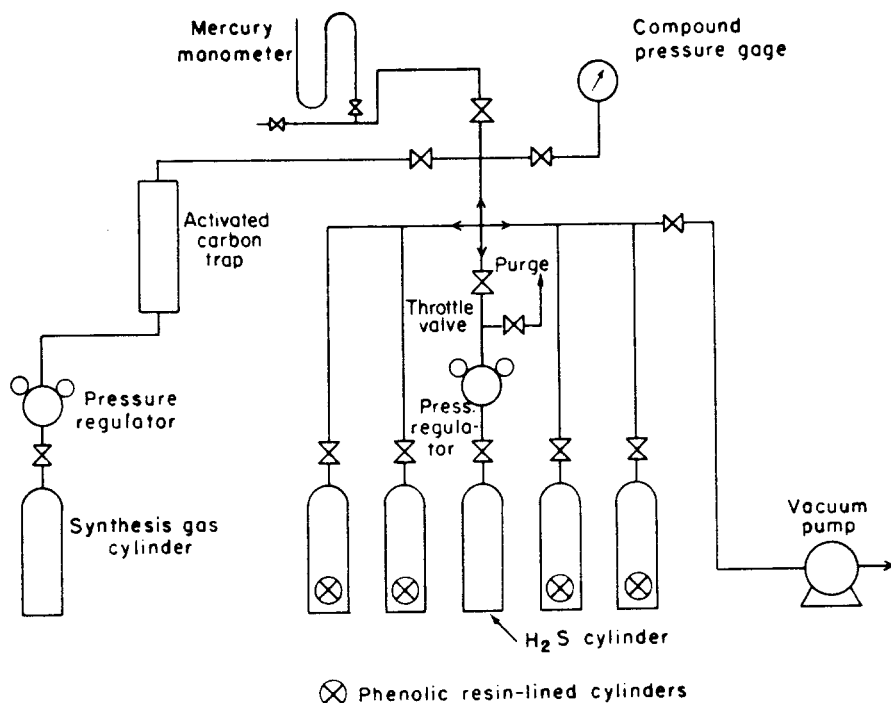


FIGURE 5. - Stainless Steel Manifold for Preparing Feed-Gas Mixtures (H_2S and Synthesis Gas).

percent, which is equivalent to about 3 pounds of Fe_2O_3 per bushel of wood chips, considerably lower than the average concentration used commercially. Generally the sponge is offered commercially in grades of 6.5, 9, 15, and 20 pounds of Fe_2O_3 per bushel.

Operating Procedure

Preparing the Feed-Gas Mixture

Four phenolic resin-lined cylinders, along with the cylinder manifold lines, were evacuated to 2 to 3 mm. of mercury, absolute, and leak-tested by letting the system stand under reduced pressure for 15 to 20 minutes. A predetermined pressure of hydrogen sulfide equivalent to a calculated hydrogen sulfide volume was added to the cylinders. When a concentration of 10 grains of hydrogen sulfide per 100 cubic feet at a cylinder pressure of 1,950 p.s.i.g. was required, hydrogen sulfide was added to give a pressure of 15.5 mm. of mercury. The cylinder pressure was initially increased to about 50 p.s.i.g. with $3H_2:1CO$ synthesis gas from a cylinder and later increased to 1,950 p.s.i.g. by compressing $3H_2:1CO$ synthesis gas from a gasholder.

Gas from each cylinder was analyzed for hydrogen sulfide content using the Tutwiler¹² method of analysis. The initial inconsistency of values obtained even from the same cylinder was eliminated when the cylinders were externally heated to a maximum wall temperature of 55° C. with an electric heater to induce uniform mixing of the gas.

¹²Altieri, V. J., Gas Analysis: Am. Gas Assoc., New York, N.Y., 1945; Mack Printing Co., Easton, Pa., pp. 338-342.

The wetted mass is placed on concrete slabs to an approximate 12-inch depth. Within an hour the metallic iron starts to oxidize. The heat of the exothermic reaction is controlled by spraying with water. In about 1 week all the iron will have been converted to Fe_2O_3 . After the Fe_2O_3 has been treated with soda ash and hydrated lime to maintain an alkaline medium in the purifying boxes, it is ready for use in gas purification.

In the Bureau tests, iron oxide content of the wood shavings averaged 23

Single-Bed Unit (6-Inch Bed Depth)

Water was added to a batch of wood chips impregnated with iron oxide to give a concentration of 50 percent by weight, and the mixture was left standing in a closed container overnight. Occasional rotation and shaking of the container insured uniform wetting. A weighed amount (approximately 150 grams) of the wetted wood chips was then packed in the absorption column to a bed height of 6 inches, giving a bed volume of 0.012 cubic foot. After the absorber was assembled, the system was purged with nitrogen and brought to operating pressure and temperature. The feed gas was adjusted to the desired flow, and the scrubbed gas was sent to the methylene blue apparatus. Samples of scrubbed gas were removed continuously after every 15 cubic feet of gas for determining hydrogen sulfide quantitatively by the methylene blue method.¹³ The feed gas was analyzed periodically by the Tutwiler method and by a modified referee sulfur method.¹⁴

Two types of tests were made: Several short tests at different rates of gas flow to determine the operating limits, and life tests to investigate the degree of hydrogen sulfide removal with time and to determine the weight of hydrogen sulfide absorbed per weight of iron. In a life test the gas flow was usually continued until the hydrogen sulfide in the exit gas exceeded 0.2 grain per 100 cubic feet.

When a test was terminated, the used wood chips were removed in the atmosphere and analyzed for water and sulfur contents. In several tests, however, the used chips were removed from the absorber and handled under an inert atmosphere to prevent oxidation of iron sulfide to free sulfur.

Operation of the four-bed (24-inch) unit was the same as the single-bed unit.

EXPERIMENTAL RESULTS AND DISCUSSION

Effects of Pressure and Residence Time

The effect of pressure on the degree of hydrogen sulfide removal was determined at atmospheric, 200 p.s.i.g., and 400 p.s.i.g. In figure 6 the hydrogen sulfide content of gas scrubbed in the 6-inch absorber is plotted as a function of the cumulative total of hydrogen sulfide removed at 200 hourly space velocity,¹⁵ room temperature,¹⁶ and an average hydrogen sulfide feed content of 10.1 grains per 100 cubic feet. Minimum scrubbed gas values plotted are 0.001 grain of hydrogen sulfide per 100 cubic feet since this

¹³Sands, A. E., Grafius, M. A., Wainwright, H. W., and Wilson, M. W., The Determination of Low Concentrations of Hydrogen Sulfide in Gas by the Methylene Blue Method: Bureau of Mines Rept. of Investigations 4547, 1947, 19 pp.

¹⁴Work cited in footnote 12, pp. 148-151.

¹⁵All hourly space velocities ($\frac{\text{gas volumes/bed volume}}{\text{hr.}}$) are based on a bed volume of 0.012 cubic foot.

¹⁶Room temperature generally was 25° to 30° C.

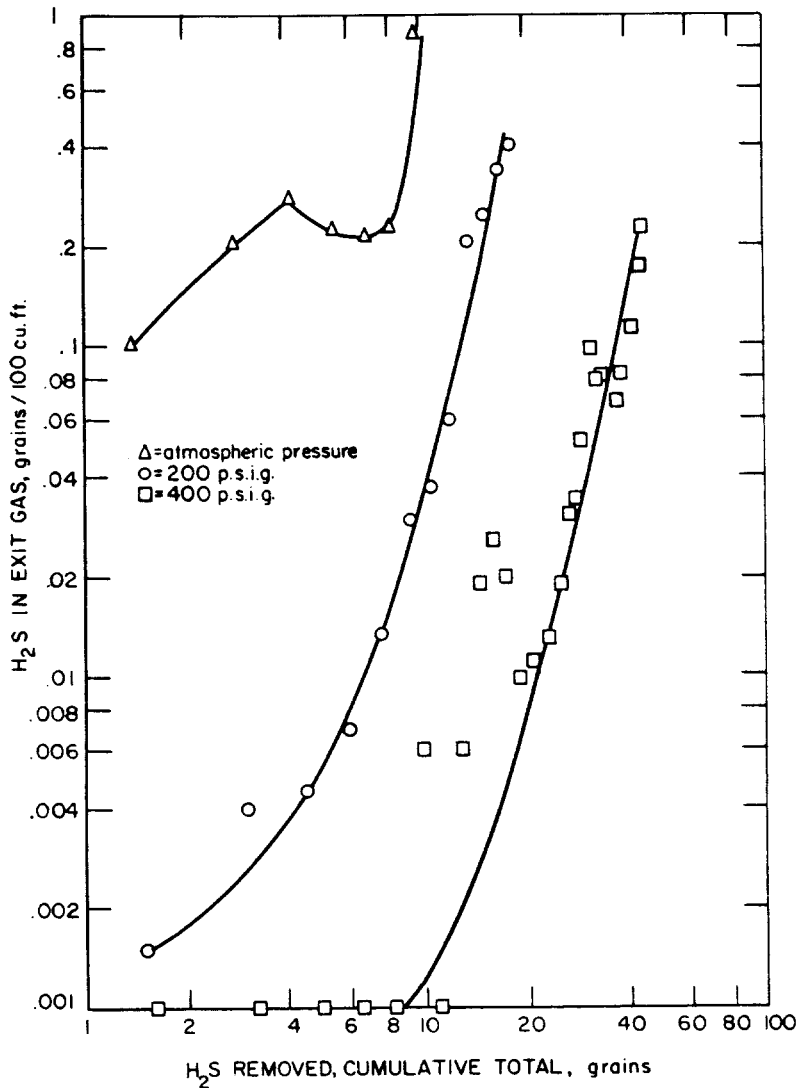


FIGURE 6. - Removal of H₂S by Iron Oxide at 200 Hourly Space Velocity. Room temperature; feed gas averaged 10.1 grains H₂S/100 cu. ft.

these results are equivalent to about 155 hours at 400 p.s.i.g. compared with 55 hours at 200 p.s.i.g. and 6 hours at atmospheric pressure.

Figure 7 shows the effect of scrubbing at 200 and 400 p.s.i.g. at 500 hourly space velocity when the average feed gas contained 9.7 grains of hydrogen sulfide per 100 cubic feet. The effect of increased operating pressure is even more pronounced at 500 than at 200 hourly space velocity. Hydrogen sulfide removal at 200 p.s.i.g. pressure was poor from the beginning; the exit hydrogen sulfide concentration was 0.02 grain per 100 cubic feet and increased sharply thereafter. However, at 400 p.s.i.g. the exit gas content remained at or below 0.001 grain of hydrogen sulfide per 100 cubic feet until 17 grains of hydrogen sulfide had been removed.

value is the sensitivity of the methylene blue method of analysis. Each point plotted is the average of two tests at atmospheric pressure, two tests at 200 p.s.i.g., and three tests at 400-p.s.i.g.

At constant space velocity a much greater degree of removal (lower exit concentration of hydrogen sulfide) was achieved at increased pressure. Thus, purification at elevated pressure is highly advantageous when a high-purity gas is required. Also, for the same degree of hydrogen sulfide removal, much more gas can be treated at the higher pressure. For example, in figure 6 when scrubbing at an hourly space velocity of 200 to an exit hydrogen sulfide concentration of 0.1 grain per 100 cubic feet, at 400 p.s.i.g. about 37 grains of hydrogen sulfide were removed by 0.012 cubic foot of iron oxide-impregnated wood chips, but at 200 p.s.i.g. only 13 grains were removed, and at atmospheric pressure the limit of purity was reached after less than 2 grains were removed. In terms of operating time at 200 hourly space velocity,

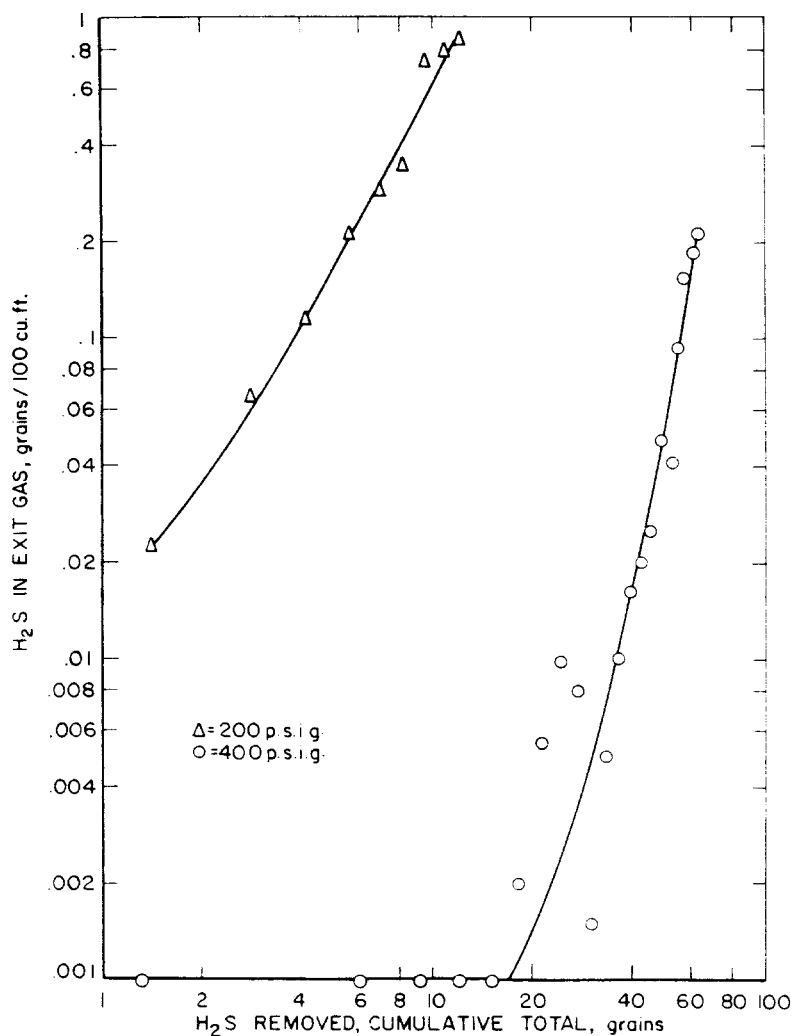


FIGURE 7. - Removal of H₂S by Iron Oxide at 500 Hourly Space Velocity. Room temperature; feed gas averaged 9.7 grains H₂S/100 cu. ft.

Arbitrarily established sulfur tolerances of 0.1 and 0.02 grain per 100 cubic feet for iron and nickel Fischer-Tropsch catalysts, respectively, are used as a basis for determining the scrubbing limits of the iron oxide impregnated on wood chips. Table 1 gives the results of several tests at atmospheric pressure, 200 p.s.i.g., and 400 p.s.i.g., and hourly space velocities of 100 to 1,000. Volumes of gas purified before reaching the established sulfur contents in the exit gas are shown, and the corresponding weight of hydrogen sulfide removed per weight of iron oxide is listed. All tests were conducted at room temperature with synthesis gas containing 9 to 11 grains of hydrogen sulfide per 100 cubic feet. Where more than one test was made at a given set of conditions, the results are averaged.

The advantage of purification at elevated pressure is clearly shown. At an hourly space velocity of 200 and at an exit-gas concentration limited to 0.02

grain of hydrogen sulfide per 100 cubic feet, about 3 times as much hydrogen sulfide was removed per weight of iron oxide at 400 as at 200 p.s.i.g. and at least 15 times as much as at atmospheric pressure. At an hourly space velocity of 200 and a final content of 0.1 grain of hydrogen sulfide per 100 cubic feet, about 3 times as much hydrogen sulfide was removed at 400 p.s.i.g. as at 200 p.s.i.g. and at least 20 times as much as at atmospheric pressure. The volumes of gas scrubbed during these tests were 5 to 7 times greater at 200 p.s.i.g. and 14 to 20 times greater at 400 p.s.i.g. than at atmospheric pressure.

At 500 hourly space velocity about 12 to 20 times as much hydrogen sulfide is removed per weight of iron oxide at 400 p.s.i.g. as at 200 p.s.i.g. for the same residual hydrogen sulfide content. The inconsistency of the

data at 400 p.s.i.g. and 500 hourly space velocity, whereby more hydrogen sulfide is removed and more gas is purified than at 400 p.s.i.g. and 200 hourly space velocity, is unexplained, but may have been caused by more regeneration of the iron by higher oxygen content of the gas. No comparative tests were made at 500 space velocity and atmospheric pressure because the prescribed hydrogen sulfide limits are exceeded too quickly.

TABLE 1. - Hydrogen sulfide removal by wood chips impregnated with iron oxide at various operating pressures

(Feed gas contained 9 to 11 grains H_2S /100 cubic feet; atmospheric, 200, and 400 p.s.i.g.)

Run	Operating pressure, p.s.i.g.	Space velocity, vol./vol.-hr. ¹	Residence time, at room temperature, seconds (based on bed volume)	Cumulative flow, cu.ft., until H_2S exit concentration became--		H_2S removed per gram iron oxide, gram, at exit H_2S concentration of--	
				0.02 gr./100 cu.ft. ²	0.1 gr./100 cu.ft. ³	0.02 gr./100 cu.ft.	0.1 gr./100 cu.ft.
48.....	(⁴)	100	33	<15	15	<0.004	0.004
47,49...	(⁴)	200	17	<15	15	<.005	.005
51,68...	200	200	241	88	110	.030	.041
53,54...	200	500	95	<15	35	<.005	.011
42,50,69	400	200	465	208	290	.086	.120
44,52,79	400	500	186	270	390	.099	.128
43,55,56	400	1,000	93	35	100	.011	.028

¹Based on wood chip volume of 0.012 cubic foot.

²Defined as tolerance for nickel catalyst.

³Defined as tolerance for iron catalyst.

⁴Atmospheric pressure.

By doubling the operating pressure, when doubling the residence time, about 2.5 times as much gas can be purified to the same residual hydrogen sulfide content. At constant pressure, increasing the space velocity reduced drastically the efficiency of hydrogen sulfide removal. Nevertheless the pickup hydrogen sulfide per weight of iron oxide was increased about 2.5 times (table 1) when the hourly space velocity was increased from 500 to 1,000 and the operating pressure from 200 to 400 p.s.i.g. Since the linear velocity of the gas through the bed and residence time were the same in both instances, the effect of increased pressure is again evident.

A 1959 publication states that, at given inlet and outlet concentrations of hydrogen sulfide in the gas, space velocity through iron oxide boxes can be increased proportionally to the operating pressure.¹⁷ Thus, at 20 atmospheres the space velocity can be increased 20-fold over that at atmospheric pressure, and the same quantity of hydrogen sulfide can be removed. The data in table 1 indicate a similar trend.

¹⁷Gunterman, W., and Schnürer, F., [New Knowledge and Developments in Dry Sulfur Purification, Especially in the Construction of Purifiers]: Gas-u. Wasserfach, vol. 100, No. 25, June 19, 1959, pp. 643-649.

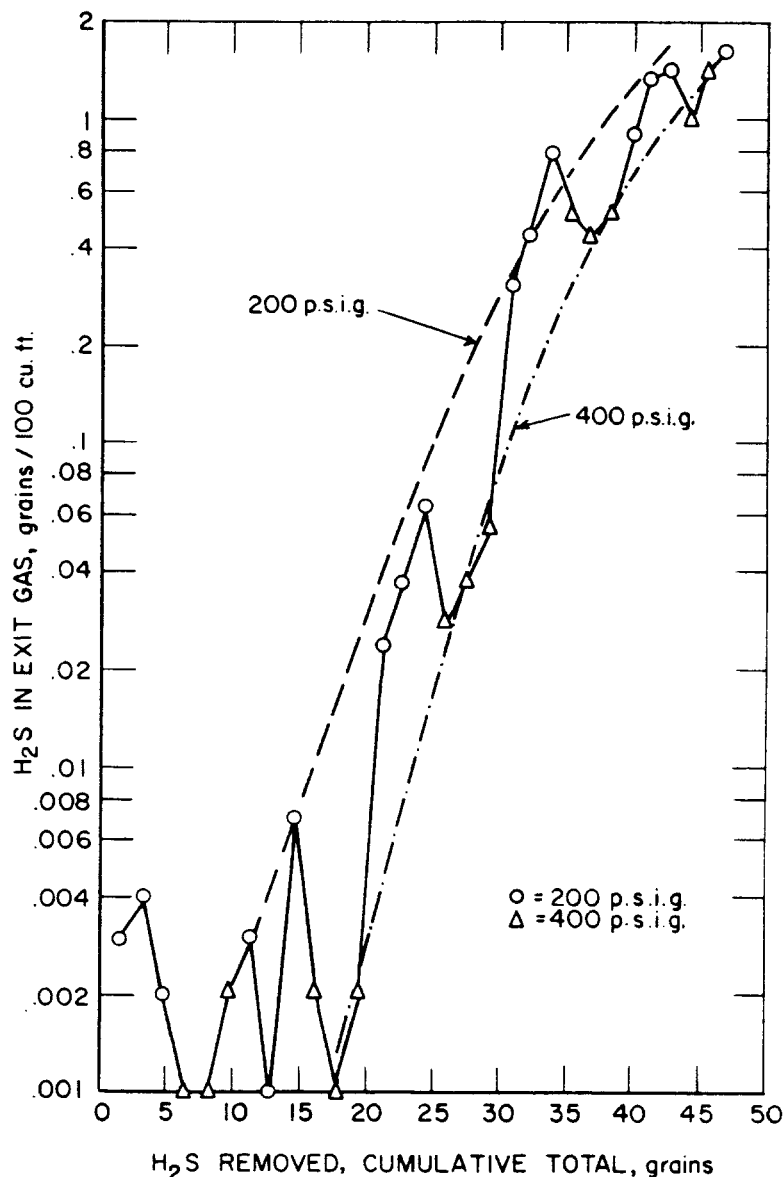


FIGURE 8. - Effect of Varying Pressures Between 200 and 400 p.s.i.g. Room temperature; 500 hourly space velocity; feed gas averaged 10.8 grains H₂S/100 cu. ft.

Figure 8 shows the variation in exit-gas concentration of hydrogen sulfide when the operating pressure was alternately changed between 200 and 400 p.s.i.g. a total of nine times at a constant hourly space velocity of 500 using the same wood chips. The purpose of this test was to prove that differences in the wood chips themselves were not causing variation in the results at different pressures. Each pressure change resulted in a change in degree of sulfur removal. Although the difference in results between operation at 200 and 400 p.s.i.g. is not as pronounced as it was between some tests at individual steady pressures, the increased effectiveness of sulfur removal as pressure was increased is evident. The most significant improvement was obtained during the first two cycles of pressure alternation when the chips were highly effective.

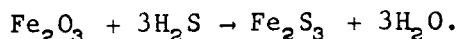
Residence or contact time in seconds is determined by dividing the bed volume expressed in cubic feet by the gas throughput in cubic feet per second under actual operating conditions.¹⁸ The residence times used in the temperature study are given in table 1.

The contact time recommended by Zapffe¹⁹ for operation at atmospheric pressure in the absence of air or oxygen is 60 seconds. It would generally be expected that, other conditions being equal, the longer the residence time the greater would be the amount of hydrogen sulfide removed until sulfur saturation of the iron oxide is reached. Somewhere between the complete and

¹⁸Zapffe, F., Practical Design Considerations for Gas Purification Processes: Refining Eng., May 1958, pp. C-19 - C-22.

¹⁹Work cited in footnote 18.

incomplete sulfur saturation points, an optimum residence time undoubtedly exists for each operating pressure, dependent on such things as economics and rate of sulfur removal, but determining these optimums was not attempted in this study. Sulfur saturation is described as the theoretical amount of sulfur that can be removed by iron oxide according to the following equation:



Thus, theoretically 160 pounds of iron oxide will remove 102 pounds of hydrogen sulfide, or 1 pound of iron oxide will remove approximately 0.64 pound of hydrogen sulfide in one fouling in the absence of air or oxygen in the gas being treated.

Effect of Elevated Temperature

To determine the effectiveness of hydrogen sulfide removal at elevated temperatures, a group of life tests was made at 60° and 100° C. at 400 p.s.i.g. operating pressure. Concentration of hydrogen sulfide in the feed gas averaged 10.1 grains per 100 cubic feet in the tests at 200 hourly space velocity, and 10.4 grains per 100 cubic feet at 500 hourly space velocity. The results of the tests at 200 hourly space velocity and at 60° and 100° C. are compared with results at room temperature in figure 9. Figure 10 shows a similar plot of tests at 500 hourly space velocity. At 200 and 500 hourly space velocity, hydrogen sulfide was removed more completely for longer periods of time at room temperature than at 60° or 100° C. When temperature increased, removal of hydrogen sulfide decreased. For example (fig. 9), after 10 grains of hydrogen sulfide had been removed, the purified gases contained 0.002, 0.017, and 0.45 grain of hydrogen sulfide per 100 cubic feet at room temperature, 60° C., and 100° C., respectively.

Although the water content of the wood chips was brought to about 50 percent at the start of each test, the moisture content of some of the discharged batches which had been operated at 60° and 100° C. was practically zero owing to the use of dry (nonhumidified) synthesis gas. This moisture loss could have been part of the reason for the poorer performance at elevated temperatures, but even at the start of the test when the moisture content of the absorbent was high, removal of hydrogen sulfide was more effective at room temperature than at the higher temperatures.

Effect of Moisture

To evaluate the efficiency of hydrogen sulfide removal at 100° C., while maintaining a constant water content in the absorbent, tests were made with a wet feed gas at 500 hourly space velocity and 400 p.s.i.g. The approximate theoretical amount of water required to saturate the volume of synthesis gas fed at operating pressures and temperatures was added to the gas stream before it entered the absorber. Figure 11 illustrates two such tests when the exit-gas content of hydrogen sulfide is plotted as a function of the total hydrogen sulfide removed.

In both tests the initial water content of the chips was about 50 percent. The addition of different amounts of water in each test had no appreciable

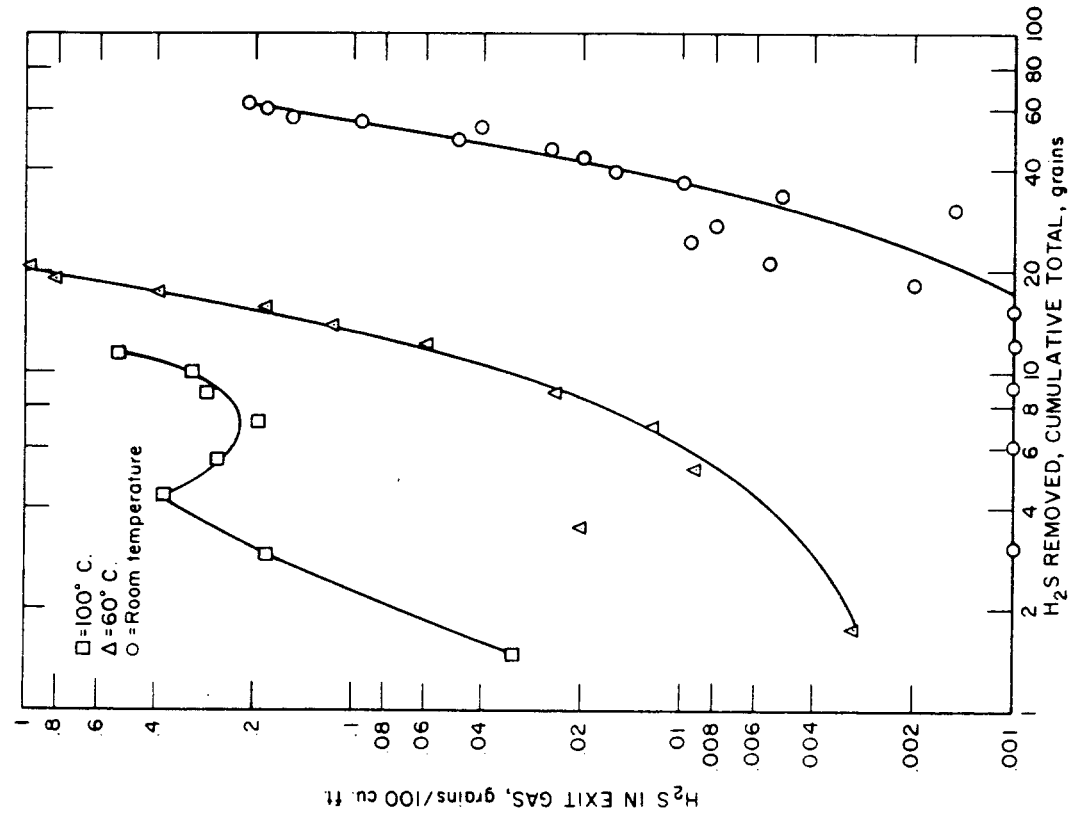


FIGURE 10. - Effect of Elevated Temperature on Removal of H₂S by Iron Oxide at 500 Hourly Space Velocity. Feed gas averaged 10.4 grains H₂S/100 cu. ft.; 400 p.s.i.g.

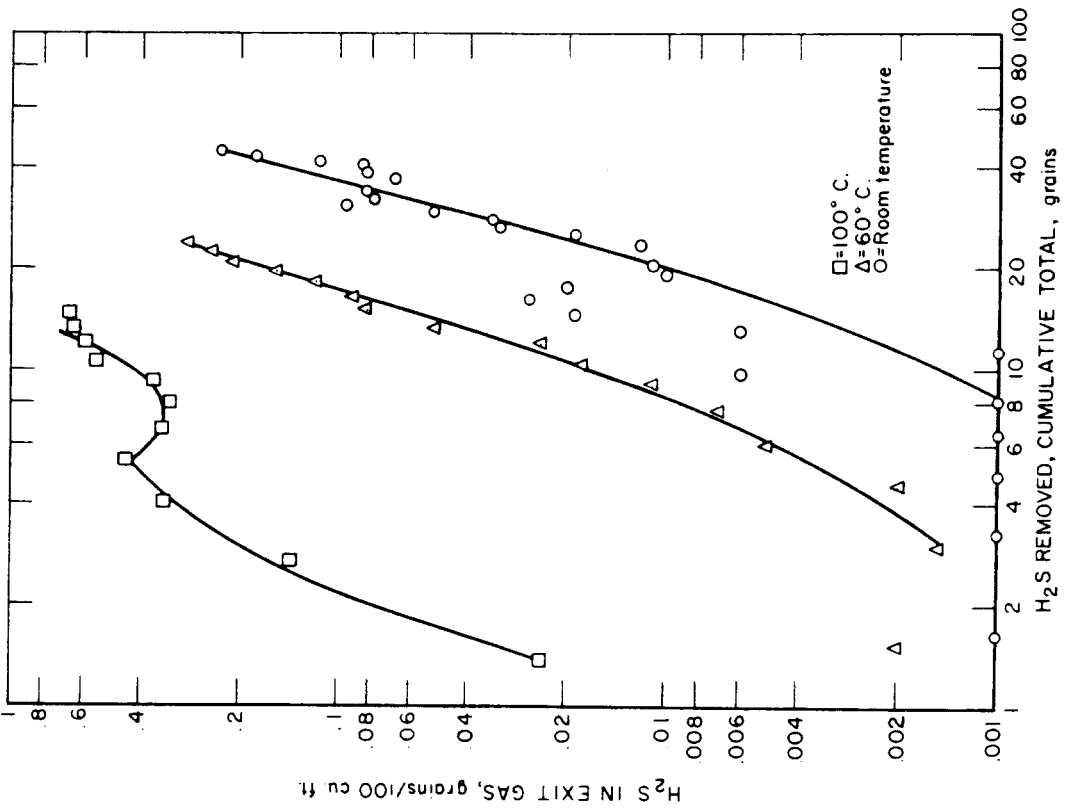


FIGURE 9. - Effect of Elevated Temperature on Removal of H₂S by Iron Oxide at 200 Hourly Space Velocity. Feed gas averaged 10.1 grains H₂S/100 cu. ft.; 400 p.s.i.g.

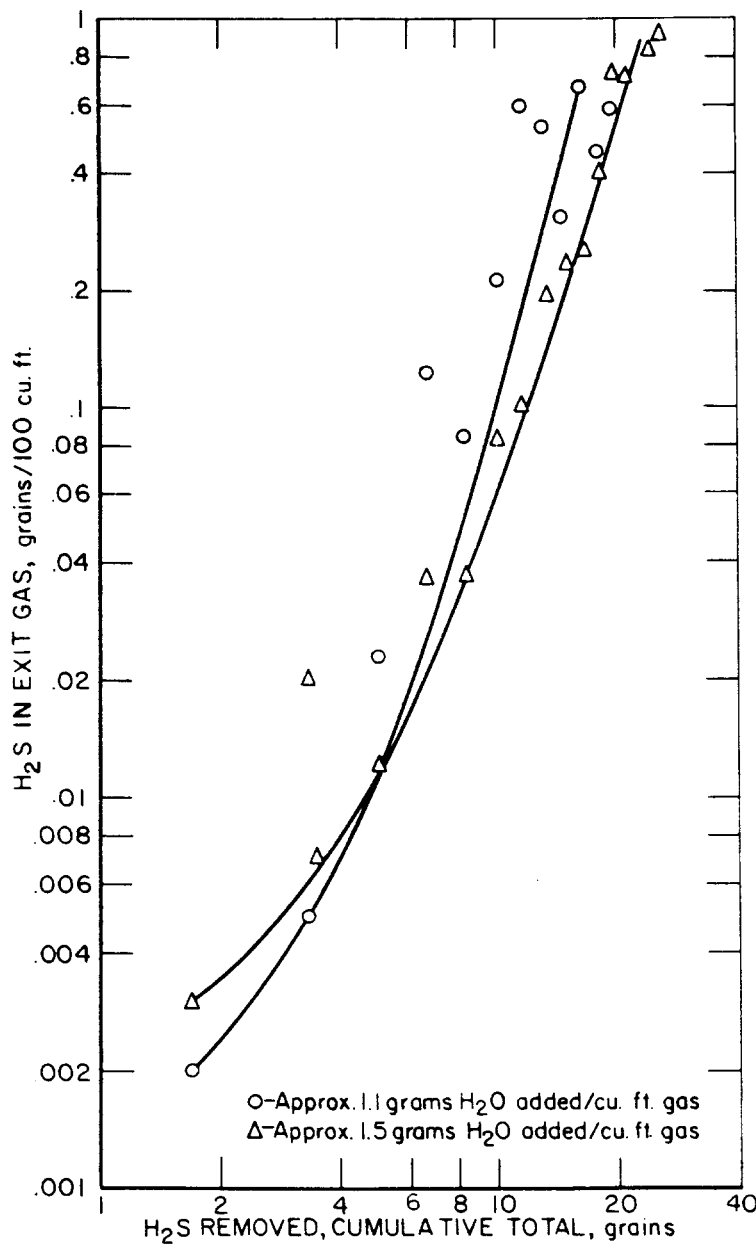


FIGURE 11. - Removal of H₂S by Iron Oxide From Wet Feed Gas. Feed gas averaged 11.2 grains H₂S/100 cu. ft.; 400 p.s.i.g.; 500 hourly space velocity; 100° C.

0.001 grain when chips of 50 percent moisture initially (400 p.s.i.g. curve, fig. 7) were used; the dry chips remained less effective throughout the tests. When the tests using dry wood chips were completed, about 2 percent moisture remained on the chips. Commercial sponge boxes generally contain between 30 and 50 percent moisture, which is maintained usually by adding steam.

effect on scrubbing capacities, judging from the similarity of results. In fact, the reproducibility shown here is better than the average throughout the tests.

When a total of 10 grains of hydrogen sulfide had been removed, the exit gas contained about 0.08 grain of hydrogen sulfide per 100 cubic feet, but without water addition (100° C. curve, fig. 10) at the same weight of hydrogen sulfide removed, the exit gas contained 0.4 grain of hydrogen sulfide per 100 cubic feet. The curves with wet gas at 100° C. match quite well the curve with dry gas at 60° C. (fig. 10). Thus, maintaining the moisture content of wood chips operated at 400 p.s.i.g., 500 hourly space velocity, and 100° C. permits greater hydrogen sulfide removal for longer periods of time than operating at similar conditions without adding moisture.

A group of tests was made in which the iron oxide-impregnated wood chips were not moistened to 50-percent water content but were used as stored (about 5 percent moisture). The average results of these tests are plotted in figure 12. The dry wood chips were less effective from the beginning; the exit gas contained 0.025 grain of hydrogen sulfide per 100 cubic feet compared with less than

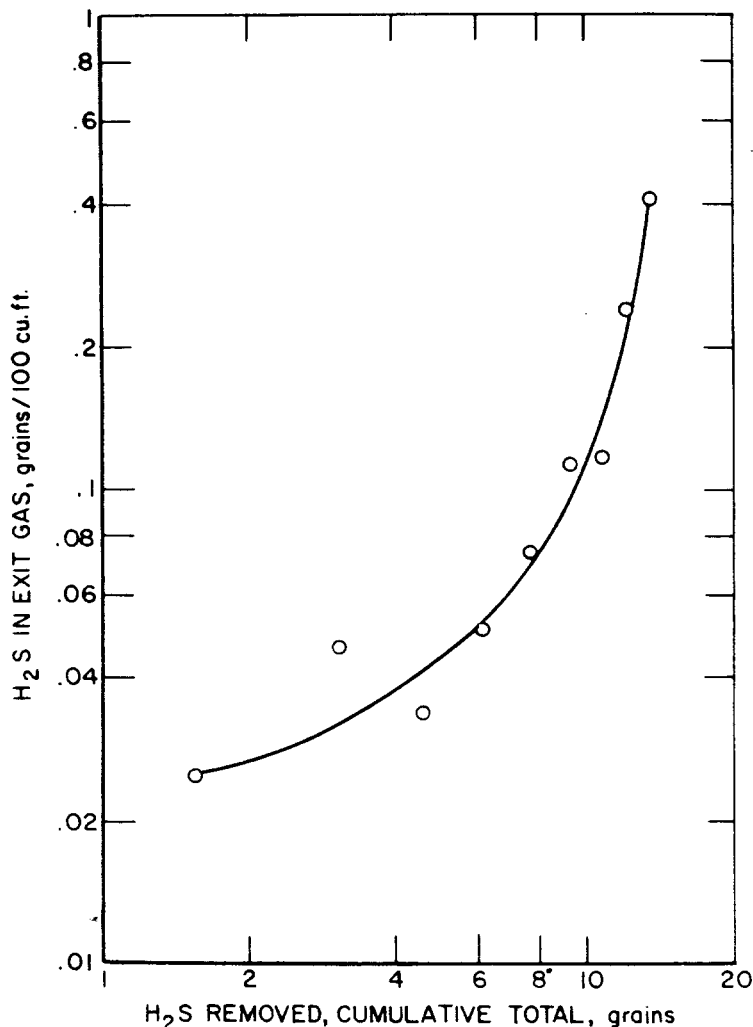


FIGURE 12.- Removal of H₂S by Dry Iron Oxide at Room Temperature. Feed gas averaged 10.3 grains H₂S/100 cu. ft.; 500 hourly space velocity; 400 p.s.i.g.

Each oxide has a best range of water content, and any increase or decrease from this range affects the activity.²⁰ The importance of humidity control in maintaining optimum moisture contents has been shown.²¹

The function of the water in the box is to act as a differential solvent for the hydrogen sulfide and to hold the sulfide long enough for the oxide to react with it.²² A further function of the water is to furnish drips enough to dissolve and remove the soluble salts formed during purification from the box. Salts that are not removed crystallize on the surface of the iron oxide, thus preventing contact with the hydrogen sulfide in the gas and therefore retarding or stopping entirely further purification. The moisture also lessens a potential fire hazard in the reaction between iron sulfide and air or oxygen. The revivification reaction is highly exothermic; the heat evolved initially is enough to raise the bed temperature to the kindling temperature of the wood chips.

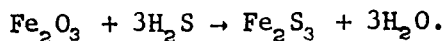
Effect of Revivifying Oxide

In the process of removing hydrogen sulfide by iron oxide, iron sulfide is formed:

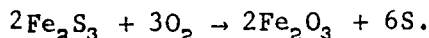
²⁰Dunkley, W. A., and Leitch, R. D., Conditions Affecting the Activity of Iron Oxides in Removing Hydrogen Sulfide From City Gas: Bureau of Mines Tech. Paper 332, 1924, 33 pp.

²¹Milbourne, C. G., and Huff, W. J., Humidity Effects in the Iron Oxide Process for the Removal of Hydrogen Sulfide From Gas: Ind. Eng. Chem., vol. 22, No. 11, November 1930, pp. 1213-1224.

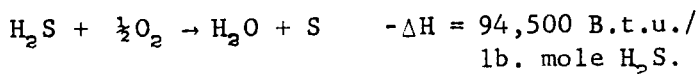
²²Seil, G. E., Dry Box Purification of Gas: Am. Gas Assoc., Inc., New York, N.Y., 1943, 289 pp.



As the iron oxide becomes depleted by the reaction, its scrubbing capacity decreases. This capacity can be restored by oxidizing the iron sulfide back to iron oxide.



The overall reaction in simplified form is shown in the following equation:



The heat of reaction is distributed so that approximately 10 percent is generated in the sulfide reaction and 90 percent in the revivification.²³

As air is generally used for regeneration of fouled iron oxide boxes in commercial operation, several tests were made to determine the effect of air

regeneration on iron oxide chips used for purification at atmospheric and elevated pressures. Revivification at room temperature and atmospheric pressure was accompanied by an increase in bed temperature owing to the heat of reaction.

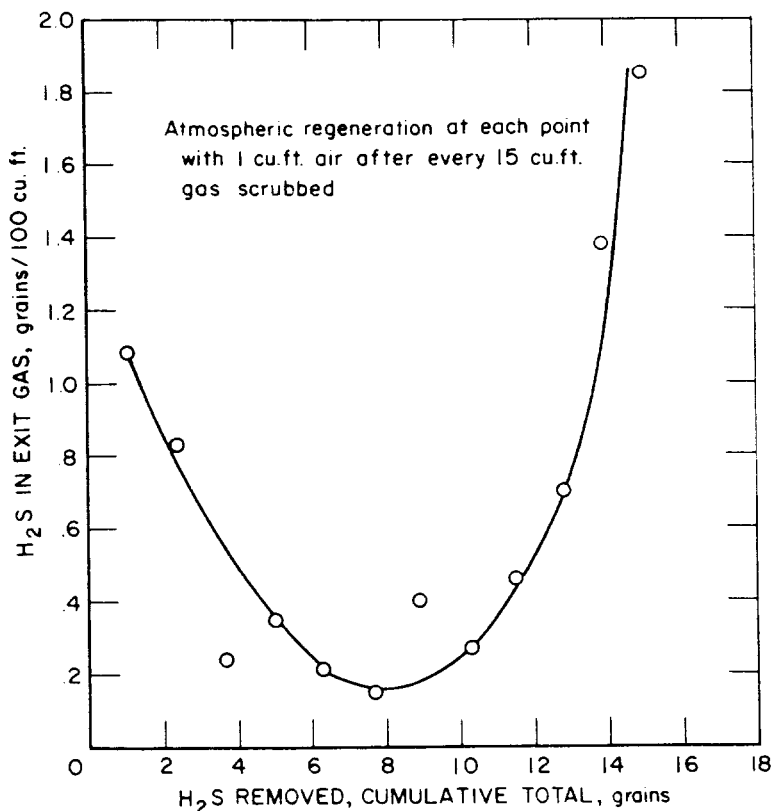


FIGURE 13. - Effect of Regeneration of Iron Oxide on H₂S Removal at Atmospheric Pressure. Feed gas averaged 9.0 grains H₂S/100 cu. ft.; 500 hourly space velocity; room temperature.

²³ Kohl, A. L., and Riesenfeld, F. C., Gas Purification: McGraw-Hill Book Co., Inc., New York, N.Y., 1960, pp. 240-280.

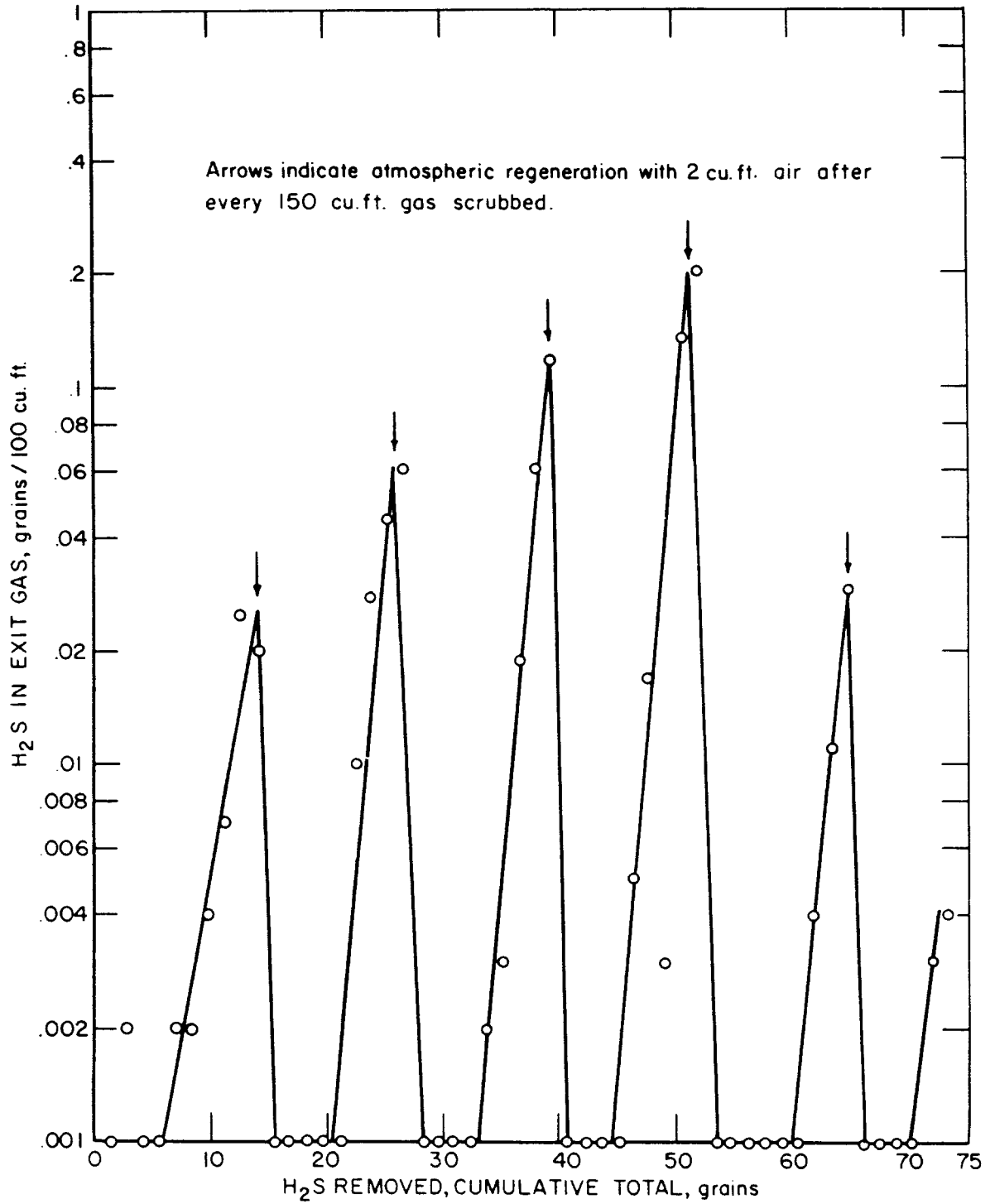


FIGURE 14. - Effect of Atmospheric Regeneration of Iron Oxide on H₂S Removal. Feed gas averaged 9.4 grains H₂S/100 cu. ft.; 400 p.s.i.g.; 500 hourly space velocity; room temperature.

regeneration. The first 15 cubic feet of gas was purified from 9 to about 1.1 grains of hydrogen sulfide per 100 cubic feet. After regeneration with 1 cubic foot of air, the next 15 cubic feet of gas was purified to about 0.8 grain of hydrogen sulfide per 100 cubic feet. After six successive periods of scrubbing followed by revivification, the effluent gas reached a low of about 0.15 grain of hydrogen sulfide per 100 cubic feet. Thereafter, revivification had little effect, and the hydrogen sulfide content of the effluent gas continued to increase with gas flow.

Results of revivifying iron oxide that pertain in the absorption at 400 p.s.i.g. are shown in figure 14. In this instance with fresh oxide the exit-gas content of hydrogen sulfide increased to about 0.02 grain per 100 cubic feet after about 150 cubic feet of gas was purified. Then the chips were regenerated at atmospheric pressure with 2 cubic feet of air. When purification was resumed at 400 p.s.i.g., the regenerated chips initially removed the hydrogen sulfide to the same limit as achieved with the fresh chips (0.001 grain per 100 cubic feet or lower) and continued to do so while treating an equal volume of gas. After another 150 cubic feet of gas had been purified, the chips were again regenerated. After five such periods of scrubbing followed by regeneration, the initial hydrogen sulfide value of the scrubbed gas was duplicated. Thus, air regeneration at atmospheric pressure can be used to revivify iron oxide for use in purification at atmospheric or elevated pressures.

Effect of Lowered Hydrogen Sulfide Feed Content and Oxygen

Several tests were made using a lower concentration of hydrogen sulfide in the feed, 4.8 grains per 100 cubic feet. Tests at 500 hourly space velocity and room temperature were made with a 6-inch bed of wood shavings at pressures of atmospheric, 200 p.s.i.g., and 400 p.s.i.g. The results of these tests are shown in table 2 and figure 15. The results of the two tests at 200 p.s.i.g. vary so widely that they could not justifiably be averaged; thus, they are both plotted. The better results obtained in one test might be partly because the impregnated wood shavings used were handpicked and thus smaller and more uniform in size than the general run of shavings used. The curve representing atmospheric pressure is the average of two tests. The single test at 400 p.s.i.g. was ended after absorbing 95 grains of hydrogen sulfide; the exit gas never contained more than 0.01 grain of hydrogen sulfide per 100 cubic feet.

At the lower hydrogen sulfide content in the feed stream, as compared with 10-grain feed gas (table 1), from 10 to 50 times more gas at 200 p.s.i.g. was purified to an exit gas that contained 0.1 grain of hydrogen sulfide per 100 cubic feet, and more than 5 times as much was purified at 400 p.s.i.g. When purifying gas to a residual hydrogen sulfide content of 0.02 grain per 100 cubic feet, at 200 p.s.i.g. 4 to 60 times more hydrogen sulfide was removed per weight of iron oxide when using a 5-grain feed gas than when feed gas containing 10 grains of hydrogen sulfide per 100 cubic feet of gas was used. At 400 p.s.i.g., about 40 times more hydrogen sulfide was removed when the feed gas contained the 5-grain concentration.

TABLE 2. - Removal of hydrogen sulfide by wood chips impregnated with iron oxide; feed gas contained 4.8 grains H₂S/100 cubic feet

(500 hourly space velocity; room temperature)

Run	Operating pressure, p.s.i.g.	Cumulative gas flow, cu.ft., until H ₂ S concentration became--		H ₂ S removed per gram iron oxide, gram, at exit-gas H ₂ S concentration of--		Sulfur as free sulfur on discharged wood chips, percent
		0.02 gr./100 cu.ft.	0.1 gr./100 cu.ft.	0.02 gr./100 cu.ft.	0.1 gr./100 cu.ft.	
73	(¹)	-	210	-	0.045	26
71	200	150	355	0.023	.054	52
74	200	1,710	1,890	.310	.343	69
72	400	² 1,995		.435	>.435	83

¹ Atmospheric pressure.

² Purified; exit H₂S concentration not above 0.01 gr./100 cu. ft. at any time.

The relatively high absorption capacity of the wood shavings in purifying gas containing 5 grains of hydrogen sulfide per 100 cubic feet suggested that traces of oxygen in the feed gas were simultaneously regenerating the impregnated chips during purification.

Oxygen determinations were made with a Haldane apparatus sampling the feed gas directly from the cylinders, and oxygen contents ranging from 0.02 to 0.06 percent were found in all the cylinders tested. This concentration of oxygen was at least double the concentration of hydrogen sulfide in the feed gas (about 0.01 percent). The oxygen content of the gas must be over 0.4 percent before oxidation of the fouled oxide (or direct oxidation of hydrogen sulfide, whichever occurs) takes place.²⁴ However, the relatively high absorption capacity obtained and the comparatively large amount of free sulfur on the wood shavings discharged from the unit indicated that at elevated pressures and low concentrations of hydrogen sulfide in the feed gas (5 grains per 100 cubic feet) simultaneous revivification of the iron oxide or direct oxidation of hydrogen sulfide was occurring with 0.02 to 0.06 percent oxygen present.

Analyses of gas from the synthesis gasholder line gave the same oxygen contents (0.02 to 0.06 percent) as found in the gas mixture in cylinders. This range of oxygen was probably present in the gas used in all Bureau tests. If so, however, the revivification effect when using 10-grain gas was not detected. Oxygen present in the gas being purified at elevated pressures would be beneficial for the overall process since longer absorption periods would be possible before revivification would be required.

²⁴ Work cited in footnote 22, p. 16.

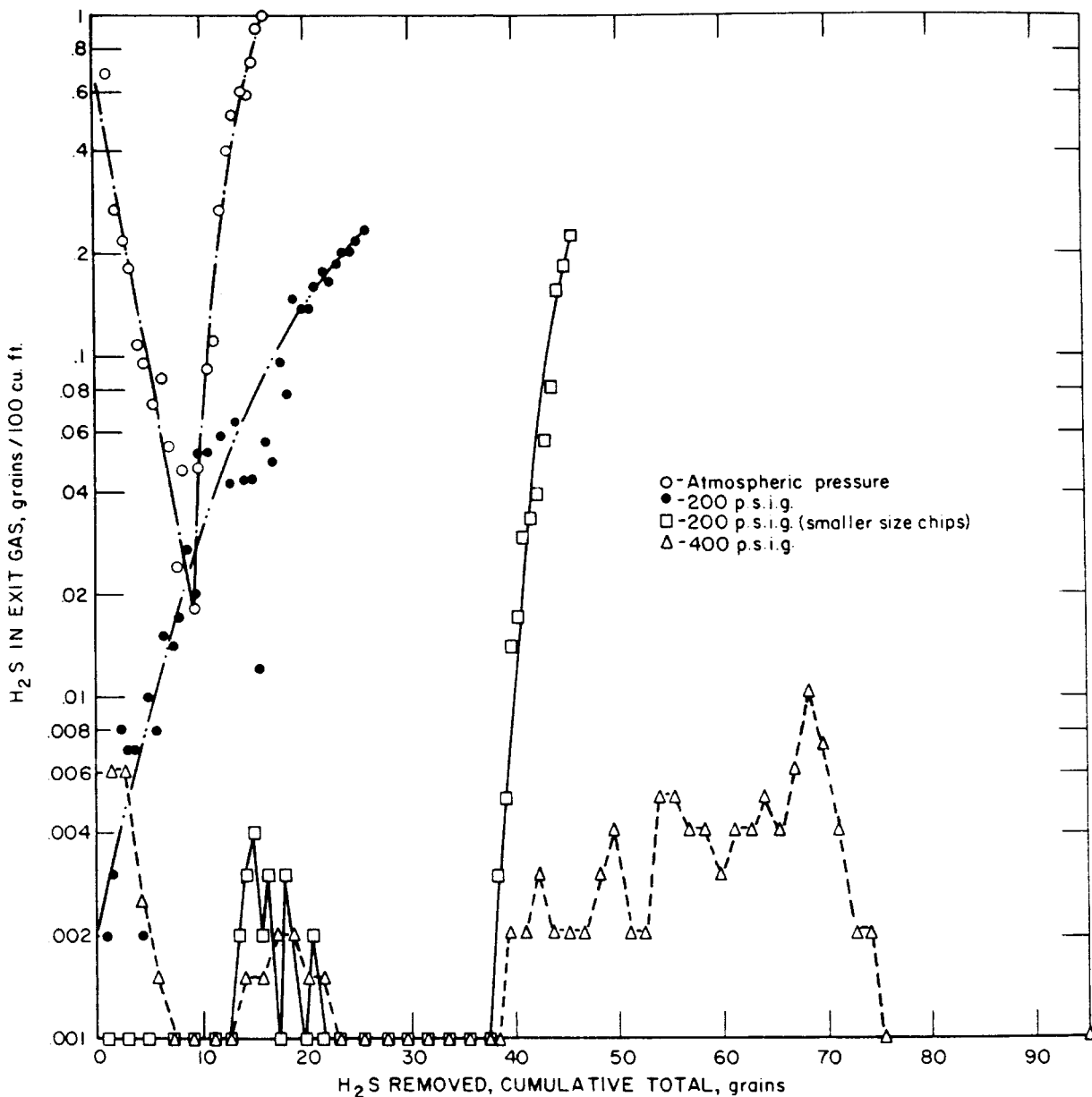


FIGURE 15. - Removal of H_2S by Iron Oxide at 500 Hourly Space Velocity. Room temperature; feed gas averaged 4.8 grains $H_2S/100$ cu. ft.

Effect of Extended Bed Depth

In commercial operation at atmospheric pressure, iron oxide-wood shavings beds are usually at least 10 feet deep.²⁵ In all Bureau tests discussed, a 6-inch bed was used in a 2-inch-diameter tube. Later, tests using a 24-inch bed of wood chips were made to determine the effect of increased bed depth while the same total bed volume as contained in the 2-inch-diameter, 6-inch bed was maintained. The chips were packed in two 1-inch-diameter stainless steel absorbers, each containing two beds 6 inches in depth. Operating

²⁵Work cited in footnote 19, p. 12.

conditions were 500 hourly space velocity based on total bed volume, 400-p.s.i.g., and room temperature. Proportionated exit-gas samples were taken from each 6-inch bed of wood chips. The gas analyses from one such test listed in table 3 were made on the gas samples proportionated during each incremental passage of 30 cubic feet of gas through the beds. Hydrogen sulfide loading of the shavings proceeds progressively from the section of initial contact downward through the bed.

TABLE 3. - Removal of hydrogen sulfide using a 24-inch bed of wood shavings impregnated with iron oxide

(At 400 p.s.i.g., 500 hourly space velocity,
and room temperature; feed gas containing
11.1 grains H_2S /100 cubic feet
bed volume = 0.012 cu. ft.)

Total gas scrubbed, std. c.f.	H_2S content of effluent gas, gr./100 cu.ft.			
	6-inch bed depth	12-inch bed depth	18-inch bed depth	24-inch bed depth
30.....	0.007	0.030	0.004	0.000
60.....	.000	.001	.000	.000
90.....	.027	.001	.001	.000
120.....	.074	.002	.000	.000
150.....	.499	.001	.000	.000
180.....	1.29	.287	.001	.000
240.....	-	.546	.000	.000
270.....	-	1.10	.000	.000
300.....	-	1.17	.000	.000
330.....	-	.947	.000	.000
360.....	-	1.22	.000	.000
390.....	1.09	1.34	.005	.001
420.....	.932	2.38	.000	.006
450.....	-	-	.027	.008
480.....	-	-	.001	.004
510.....	-	-	.012	.008
540.....	-	-	.006	.011
570.....	-	-	.003	.014
600.....	-	-	.008	.013
630.....	-	-	.002	.018
660.....	-	-	.029	.028
690.....	-	-	.035	.025
720.....	-	-	.030	.030
750.....	-	-	.059	.038
780.....	-	-	.091	.046
810.....	.994	.031	.180	.055
840.....	1.84	.675	.210	.067
870.....	-	-	.241	.092
900.....	-	-	.553	.159
930.....	-	-	.467	.126
960.....	-	-	.597	.172
990.....	-	-	1.03	.360
1,020.....	-	-	1.07	.370
1,050.....	-	-	.862	.352
1,080.....	-	-	.324	.325
1,110.....	-	-	-	.554
1,140.....	-	-	-	.717
1,170.....	-	-	-	1.05

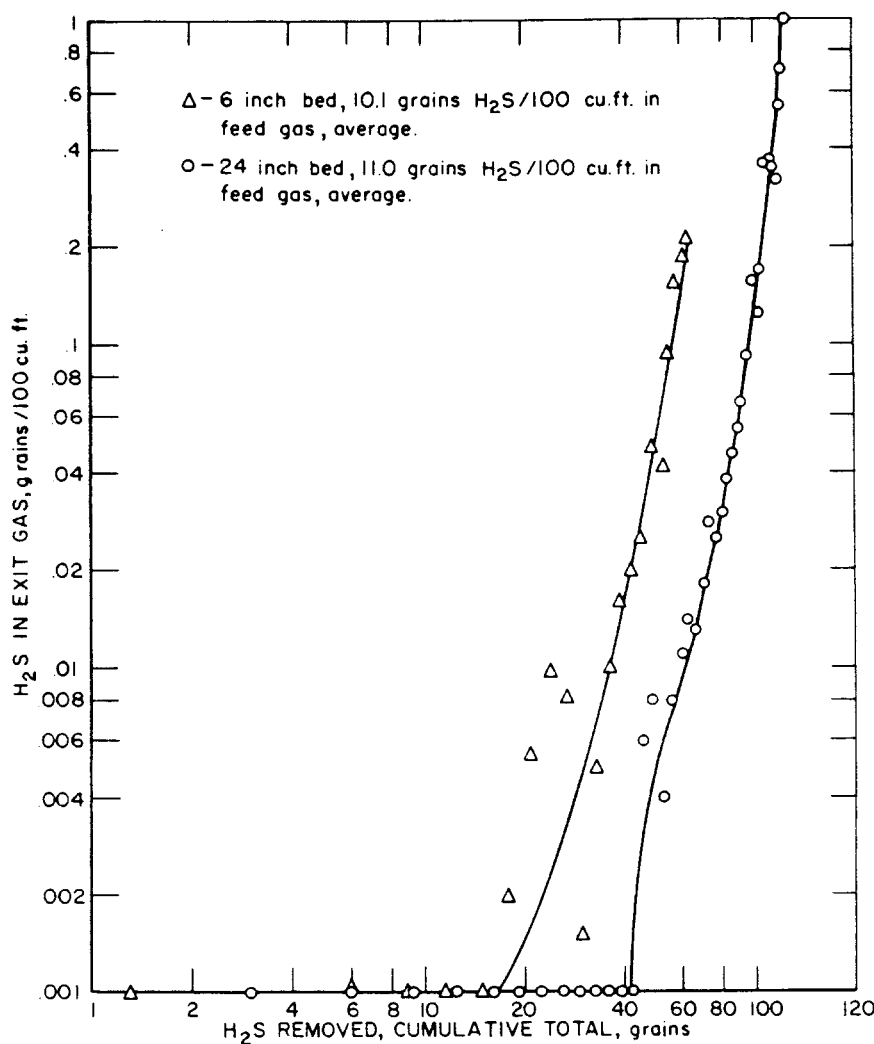


FIGURE 16. - Removal of H_2S by 24- and 6-Inch Beds of Iron Oxide. Room temperature, 500 hourly space velocity.

In figure 16 hydrogen sulfide exit gas concentrations from this same test are plotted as a function of the total hydrogen sulfide removed and are compared with results on a 6-inch bed. More complete removal of hydrogen sulfide was obtained for longer periods of time with the 24-inch bed than with the 6-inch bed. For example, after 40 grains of hydrogen sulfide had been removed, the purified gas from the 24-inch bed contained 0.001 grain of residual hydrogen sulfide per 100 cubic feet, compared with 0.15 grain per 100 cubic feet from the 6-inch bed.

In table 4 the average results of two tests in the 24-inch bed are compared with average results of three tests in the 6-inch bed at similar operating conditions.

Volumes of gas purified before reaching the arbitrarily established sulfur tolerances in the exit gas are given, along with the corresponding weight of hydrogen sulfide removed per weight of iron oxide.

As shown in table 4, the 24-inch bed purified about three times as much gas as the same volume of wood shavings in a 6-inch bed, when scrubbing to the same residual hydrogen sulfide. Expressed in terms of weight of hydrogen sulfide removed per weight of iron oxide on the shavings, the 24-inch bed removed about three times as much as the 6-inch bed when purifying to the same residual hydrogen sulfide content, for example, 0.356 to 0.128 gram of hydrogen sulfide per gram of Fe_2O_3 when the exit gas contained 0.1 grain of hydrogen sulfide per 100 cubic feet.

TABLE 4. - Hydrogen sulfide removal by iron oxide
impregnated on wood shavings

(Feed gas contained 9 to 11 grains of hydrogen sulfide per 100 cubic feet; 400 p.s.i.g.; 500 hourly space velocity,¹ room temperature)

Run	Wood chip bed, depth, inches	Cumulative flow, cu.ft., until H ₂ S exit concentration became--		H ₂ S removed per gram iron oxide, gram, at exit H ₂ S concentration of--	
		0.02 gr./100 cu.ft. ²	0.1 gr./100 cu.ft. ³	0.02 gr./100 cu.ft.	0.1 gr./100 cu.ft.
44, 52, 79.....	6	270	390	0.099	0.128
1-4, 2-4.....	24	820	950	.318	.356

¹Vol./vol.-hr. based on bed volume of 0.012 cubic foot.

²Defined as tolerance for nickel catalyst.

³Defined as tolerance for iron catalyst.

An explanation for the larger capacity for hydrogen sulfide obtained with the deeper bed is that the first two 6-inch sections continued absorbing after their effluent gas exceeded the hydrogen sulfide specification. Apparently the oxygen in the gas revived the iron oxide to the extent that in these sections the absorption was 90 percent of the total absorption as shown by the data in table 5. In these two sections the free sulfur was 86 and 77 percent of the total sulfur, and the first section absorbed 1.13 pounds of sulfur per pound of iron oxide. Further extension of the bed to the 10-foot depth used commercially may even increase this capacity. However, it may be impossible to achieve the theoretical capacity of 0.64 pound of hydrogen sulfide per pound of Fe₂O₃ on one fouling of the oxide chips without exceeding the arbitrary limits of hydrogen sulfide in the exit stream.

Analyses of Impregnated Wood Chips

Table 5 lists typical analyses of wood chips used in tests at the conditions stated. An average sulfur value of 2.38 percent is given for the fresh wood shavings. The percentage of free sulfur on the used chips correlates with pressure (experiments 69, 68, and 47), indicating that pressure is desirable for continuous revivification. Probably revivification is related to the oxygen partial pressure.

TABLE 5. - Analyses of wood chips impregnated with iron oxide

Experiment.....	1-4 (4-bed unit) ¹				79	69	54	68	47	61	72
	1st bed	2d bed	3d bed	4th bed							
Space velocity.....vol./vol.-hr.	2,000	2,000	2,000	2,000	503	201	503	201	201	201	500
Bed temperature.....°C.	26	26	26	26	26	27	27	28	24	100	26
Bed pressure.....p.s.i.g.	400	400	400	400	400	400	200	200	(²)	400	400
Analyses before test:											
Weight of charge.....grams	33.60	40.30	33.80	50.60	158.30	134.25	134.70	140.05	140.00	160.63	141.55
Moisture.....percent	52	52	52	52	52	45	44	53	48	57	54
Ash (moisture free).....do..	49.6	49.6	49.6	49.6	49.6	56.1	60.8	58.6	47.6	46.1	51.6
H ₂ S concentration in feed gas, average.....gr./100 cu.ft.	11.0	4.1	1.0	0.4	11.0	10.5	10.0	10.3	8.8	9.4	4.6
H ₂ S concentration in exit gas at end of test.....do..	-	-	0.002	0.003	0.003	0.815	0.820	0.027	0.293	0.66	0.001
Sulfur in fresh chips (moisture free), average.....percent	2.38	2.38	2.38	2.38	2.38	2.38	2.38	2.38	2.38	2.38	2.38
Analyses after test:											
Weight of discharge.....grams	21.00	37.30	32.80	45.75	136.85	125.80	119.03	132.75	84.96	67.79	103.90
Weight loss.....do..	12.60	3.00	1.00	4.85	21.45	8.45	15.67	7.30	55.04	92.84	37.65
Moisture.....percent	3.9	42.1	51.1	51.0	42.8	48.3	53.3	51.7	15.6	6.8	21.0
Total iron.....grams	2.62	2.94	2.65	3.65	11.86	15.32	9.51	11.22	11.47	9.48	12.23
Sulfur:											
Combined.....do..	0.65	0.56	0.47	0.62	2.30	2.15	2.06	2.12	2.22	1.87	2.30
Combined.....percent	14	23	49	79	26	44	77	77	84	71	17
Free.....grams	4.06	1.88	0.48	0.16	6.58	2.34	0.61	0.61	0.43	0.76	11.16
Free.....percent	86	77	51	21	74	52	23	23	16	29	83
Total.....grams	4.71	2.44	0.95	0.78	8.88	4.49	2.67	2.76	2.65	2.63	13.46
Pickup.....do..	4.23	1.93	0.57	0.25	6.98	2.94	1.35	1.23	0.94	0.56	11.51
Feed sulfur removed.....percent	62.6	76.4	95.7	1000.0	103.2	90	184	163	166	59	205
Total sulfur removed.....do..	62.4	28.5	8.4	3.7	103	0.20	0.10	0.08	0.06	0.04	0.66
Sulfur removed per pound of Fe ₂ O ₃pounds	1.13	0.46	0.15	0.05	0.41	0.20	0.10	0.08	0.06	0.04	0.66
Total flow of gas.....cubic feet	1,005	1,005	1,005	1,005	1,005	510	120	120	105	165	1,995
Total hours.....	170	170	170	170	170	212	20	37	66	69	328

¹Used wood chips discharged and handled in inert atmosphere for this test; all others discharged in air.

²Atmospheric pressure.

The inconsistency of the "feed sulfur removed, percent" figures of the single-unit tests is explained by the sampling technique used. Only part of each discharged batch of shavings was collected as a representative sample and analyzed; in the multiple-bed tests the complete beds were analyzed. Hydrogen sulfide balances for two of the tests with the 24-inch beds are given below:

	<u>Run 1-4</u>	<u>Run 2-4</u>
Total H ₂ S fed.....grains	110.88	130.85
Total H ₂ S remaining in exit gas.....do..	.02	4.65
H ₂ S picked up, (total in minus total out).....do..	110.86	126.20
H ₂ S on discharged chips minus H ₂ S equivalent of fresh chips...do..	114.02	116.55
H ₂ S weight balance (H ₂ S on discharged chips minus H ₂ S equivalent of fresh shavings per H ₂ S in minus H ₂ S out)....percent	102.9	92.4

CONCLUSIONS

Bench-scale studies showed that purification of synthesis gas using wood chips impregnated with iron oxide can reduce the hydrogen sulfide content below the arbitrarily established limits of 0.02 and 0.10 grain per 100 cubic feet for nickel methanation and Fischer-Tropsch catalysts, respectively. Operating pressures and temperatures, bed depth, and moisture content of the impregnated wood shavings are important factors in the design and operation of iron oxide units for removing hydrogen sulfide from synthesis gas.

The allowable gas throughput for a given sulfur removal increases linearly with pressure. As the amount of hydrogen sulfide absorbed per weight of iron oxide is limited by the stoichiometry of the reaction and rate of revivification, the iron oxide for a given volume of absorbent becomes spent in a shorter time at higher gas throughputs. An average of 0.356 gram of hydrogen sulfide was removed per gram of Fe₂O₃ on the shavings in the bench-scale tests using a 2-foot bed depth, compared with a stoichiometric value of 0.64 gram of hydrogen sulfide per gram of Fe₂O₃. However, as the efficiency of removal was improved by increasing the depth of the bed of wood chips from 6 inches to 24 inches, a value closer to the theoretical can probably be achieved in commercial operation using beds about 10 feet in depth.

The small amounts of oxygen in the feed gas probably contributed to the removal of hydrogen sulfide. Synthesis gases produced by gasification of coal with steam and oxygen might contain small quantities of oxygen. At elevated pressure a small amount of oxygen in the synthesis gas appears to revivify the iron oxide in situ.

The effectiveness of hydrogen sulfide removal was decreased at elevated temperature. At 100° C. the concentration of hydrogen sulfide in the effluent gas from the absorbent exceeded 0.1 grain per 100 cubic feet in a relatively short time. Even at 60° C., for a given exit concentration of hydrogen sulfide, less than half as much hydrogen sulfide was removed as at room temperature (25° to 30° C.). Decreased effectiveness of removal at elevated temperature was caused partly by loss of moisture from the chips. Relatively dry absorbent gave considerably poorer removal of hydrogen sulfide at room temperature than chips containing 50 percent moisture.

Regeneration of Fe_2S_3 to obtain Fe_2O_3 by oxidation with air is effective for renewing the activity of the spent absorbent. No loss of activity was indicated through five cycles of absorption and regeneration.

Tests made with a feed gas containing a lower concentration of hydrogen sulfide (5 grains per 100 cubic feet compared with 10 grains) gave considerably greater removal before a sulfur breakthrough occurred in the exit gas. However, the small quantity of oxygen in the feed gas may have increased the efficiency.

This study yielded specific data that are obviously incomplete; the individual effect of the operating variables, particularly at elevated pressure, will be more thoroughly investigated and reported in a future publication.