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Report of Investigations 4891

REMOVAL OF HYDROGEN SULFIDE AND CARBON DIOXIDE FROM SYNTHESIS GAS USING DI- AND TRI-ETHANOLAMINE

BY H. W. WAINWRIGHT, G. C. EGLESON, C. M. BROCK,
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United States Department of the Interior—October 1952

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UNITED STATES DEPARTMENT OF THE INTERIOR
Oscar L. Chapman, Secretary
BUREAU OF MINES
J. J. Forbes, Director

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by

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INTRODUCTION

The Bureau of Mines at its Morgantown, W. Va., station is conducting research and development work on the problem of producing synthesis gas from raw coal for use in the Fischer-Tropsch process.[3] Synthesis gas used in this process, as well as in those for producing alcohol, ammonia, and methane, must be purified to an exceedingly high degree.[4]

In converting synthesis gas, consisting essentially of hydrogen and carbon monoxide, into any of the foregoing compounds, catalytic treatment is necessary for economical operation. As the most satisfactory and active catalysts employed in the Fischer-Tropsch synthesis are sulfur-sensitive, the purified synthesis gas must have an extremely low sulfur content. A maximum concentration of 0.1 grain of total sulfur per 100 cubic feet of purified synthesis gas has been specified for this process.

As it is known that carbon dioxide has a deleterious effect on the Fischer-Tropsch synthesis,[5] and as most of the experimental work on the synthesis has been carried out with a feed gas containing about 2 to 3 percent carbon dioxide, a part of the purification program at Morgantown has dealt with the removal of this compound from the raw synthesis gas.

The Morgantown station has constructed a pilot plant on the campus of West Virginia University to study the removal of hydrogen sulfide, organic sulfur compounds, and carbon dioxide from synthesis gas made directly from coal.

This report, the first of a series on the operation of the purification pilot plant, deals with the removal of hydrogen sulfide and carbon dioxide using aqueous solutions of di- and triethanolamine.

SUMMARY AND CONCLUSIONS

1. The purification pilot plant and the equipment, solution, and gas cycle used for removing hydrogen sulfide and carbon dioxide are described.

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[3] Strimbeck, G. R., Holden, J. H., Rockenbach, L. P., Cordiner, J. B., Jr., and Schmidt, L. D., Pilot Plant Gasification of Pulverized Coal with Oxygen and Highly Superheated Steam: Bureau of Mines Rept. of Investigations 4733, 1950, 41 pp.

[4] Sands, A. E., Wainwright, H. W., and Schmidt, L. D., Purification of Synthesis Gas Produced from Pulverized Coal: Ind. Eng. Chem., vol. 40, 1948, pp. 607-620.

[5] Elliott, M. A., Bureau of Mines, private communication, 1951.

12. Results from one pilot-plant run in which 35 percent TEA was used and the absorber was operated at atmospheric pressure rather than 300 p.s.i.g. showed that about 5.5 times as much steam for reactivation is required at the lower pressure in order to purify 1,000 cubic feet of raw gas.

13. A brief description of the analytical methods used during the pilot-plant operation is included in the report.

ACKNOWLEDGMENTS

The authors wish to express their appreciation to the members of the Synthesis Gas Branch, especially those of the Gas Treating and Testing Section, whose cooperation was most helpful.

The helpful suggestions and comments of Dr. R. G. Dressler and H. R. Batchelder of the Bureau's Coal-to-Oil Demonstration Branch are gratefully acknowledged.

Thanks are due, also, to Dr. R. E. Reitmeier and his associates of the Gas Processes Division, Girdler Corp., who freely contributed pertinent information from their extensive experience in gas purification.

PURIFICATION PROGRAM

The gas-purification research and development program at Morgantown includes the recovery of sulfur as a feature of the gas-purification system in any large Fischer-Tropsch plant. Important credits result from the sale of byproduct sulfur[6] and venting to the atmosphere of any large quantity of sulfur in one form or another is avoided. Examination of the sulfur reserves, rates of consumption, and new discoveries indicates that when large commercial synthetic liquid fuel plants were operating, production of byproduct sulfur will be of importance with respect to conservation of natural resources.

The recovery of sulfur from sour natural gas or refinery gas poses no problems in most instances as the gas is usually high in hydrogen sulfide and low in carbon dioxide. Sour natural gas from one field is known to contain 7 percent hydrogen sulfide. The sulfur-recovery plant operating on gas from this field treats an acid gas containing 56 percent hydrogen sulfide.[7]

Synthesis gas produced at the Morgantown station contains approximately 0.5 percent hydrogen sulfide and 12 to 15 percent carbon dioxide whenever a coal containing about 2.5 percent sulfur is gasified.[8] Such a ratio of carbon dioxide to hydrogen sulfide in the raw gas, 30:1, makes selective absorption of hydrogen sulfide extremely difficult. Preliminary cost estimates on

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[6] Sands, A. E., and Schmidt, L. D., Recovery of Sulfur from Synthesis Gas: Ind. Eng. Chem., vol. 42, 1950, pp. 2277-2287.

[7] Cooper, C. W. and Waddle, H., Recovery of Free Sulfur from Natural Gas: Gas, July 1945, pp. 31-33.

[8] Sands, A. E., Wainwright, H. W., and Egleson, G. C., Organic Sulfur in Synthesis Gas: Occurrence, Determination, and Removal: Bureau of Mines Rept. of Investigations 4699, 1950, 51 pp.

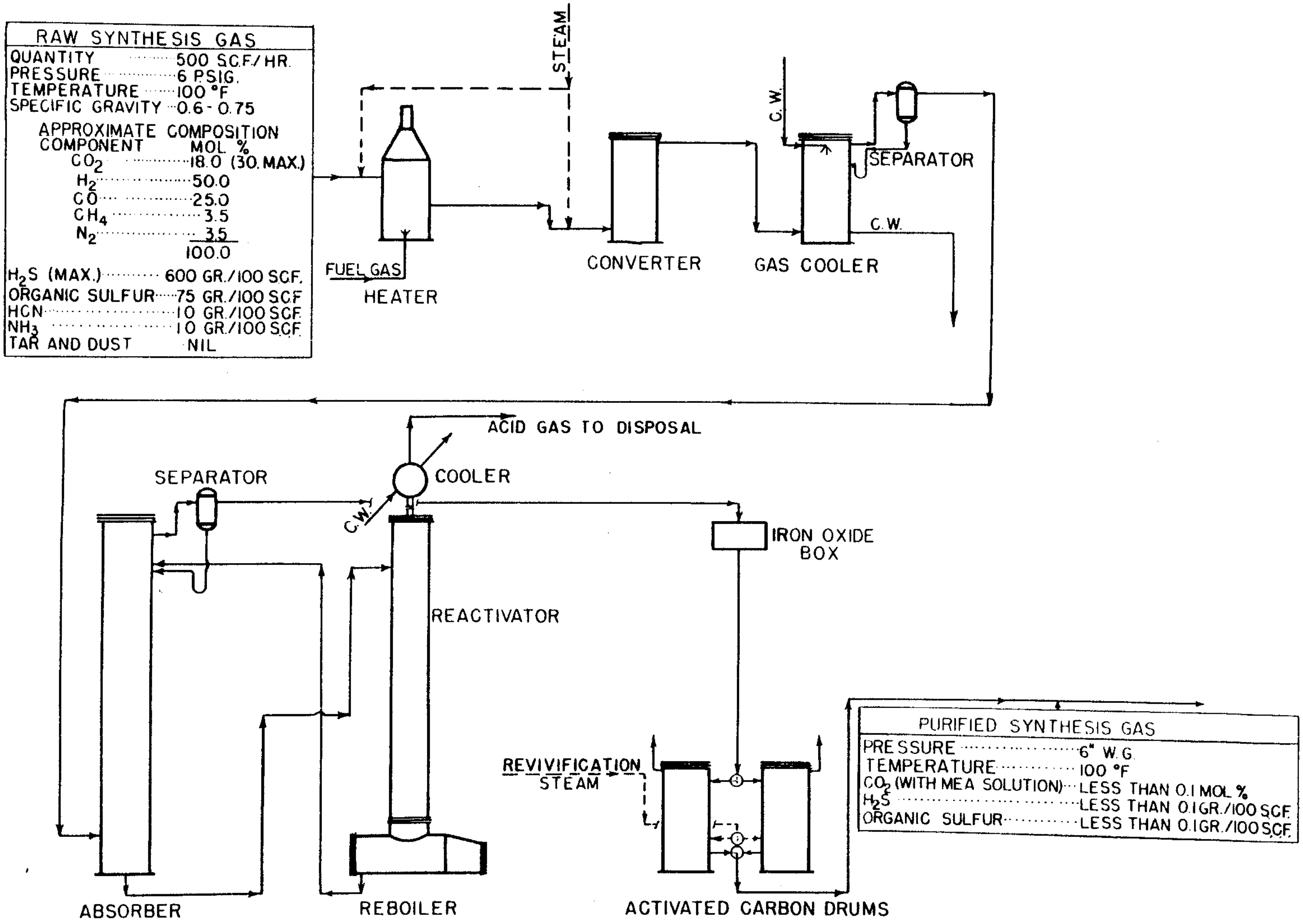

Figure 1. - Synthesis-gas-purification pilot plant.

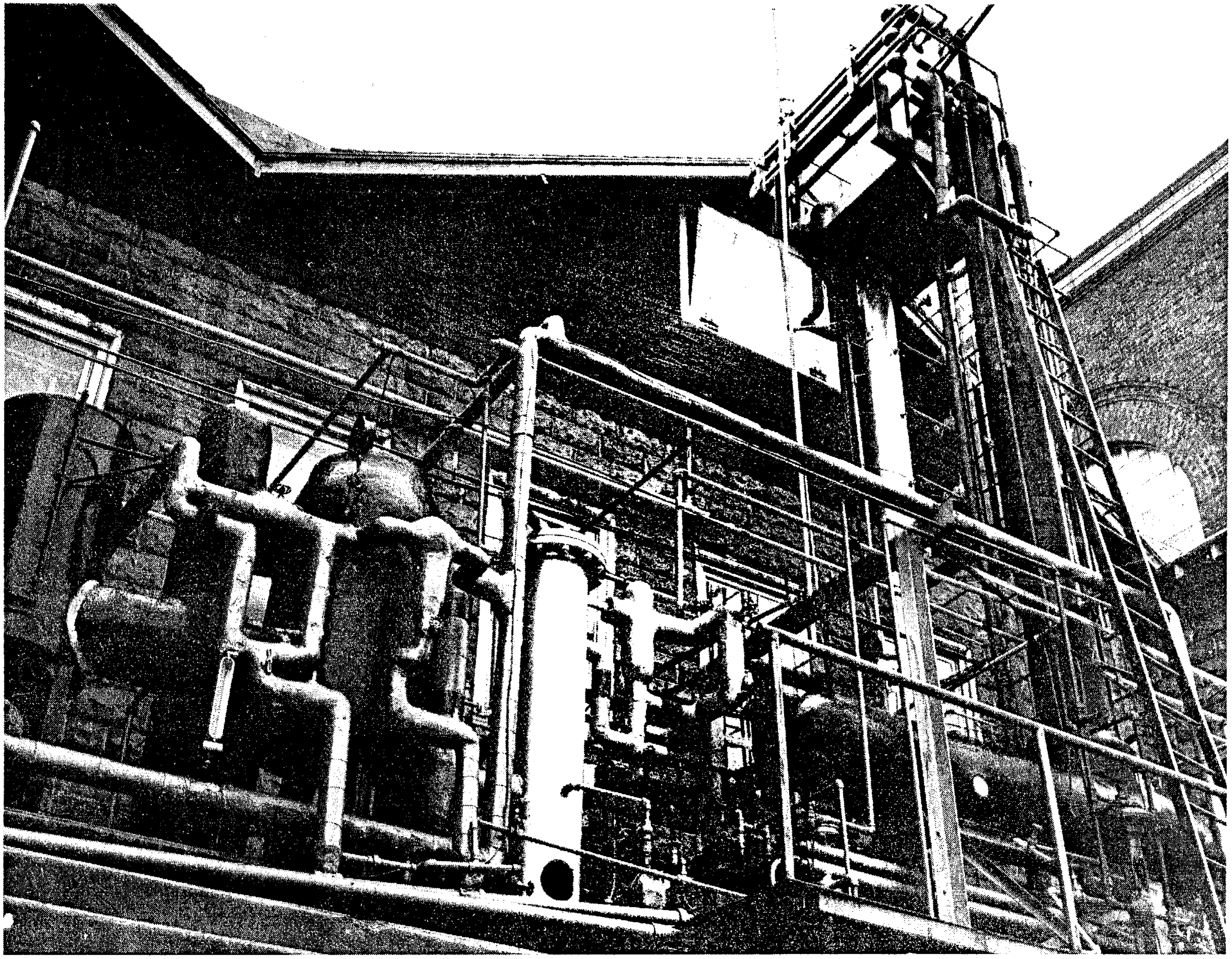

Figure 2. - View of gas-purification pilot plant.

Morgantown Station. The pilot plant is quite flexible and can be used for investigating many purification processes. The plant was designed originally to operate at a pressure of 6 p.s.i.g., but parts of it have been altered so that operation at 300 p.s.i.g. is possible.

Figure 1 is a flow diagram of the plant as it was originally designed. With operation at 6 p.s.i.g. the capacity of the pilot plant is 500 s.c.f. of raw synthesis gas per hour. The maximum flow of gas that has been used during operation at 300 p.s.i.g. has been 5,000 std.c.f. per hour, the flow of gas being limited either by foaming difficulties of the liquid scrubbing medium, the capacity of the actifier, or the limited supply of feed gas.

The hydrogen sulfide and carbon dioxide removal equipment (absorber, reactivator, and iron oxide boxes), as well as the activated carbon drums for organic sulfur removal, have been modified to permit operation at 300 p.s.i.g. The converter and its accessories, also designed for low pressure operation, are being removed at present and are to be replaced with high-pressure equipment. The converter will be used to investigate the simultaneous removal of hydrogen sulfide and organic sulfur compounds at elevated temperatures and pressures.

Figure 2 gives a view of the pilot plant. In this figure, the absorber and reactivator are to the right and the heater, converter, and cooler to the left. The high-pressure iron oxide boxes (not shown) are to the right of the reactivator, and the activated carbon drums are behind the insulated converter. A description of the equipment used for removing the hydrogen sulfide and carbon dioxide from the raw gas follows:

Absorber

The absorber consists of a 21-foot length of 10-inch schedule 40 steel pipe, and is equipped with a drain, pressure relief valve, seven gas sample connections, and seven liquid sample connections. There is a liquid separator in the exit gas line at the top of the absorber where small amounts of entrained solution are removed from the gas stream and returned to the column. The pressure drop across the absorber is indicated by a water-filled manometer.

The absorber was packed originally with 15 feet of half-inch porcelain Raschig rings. The half-inch rings were replaced later with 1-inch rings in an attempt to reduce difficulties caused by the foaming of the amine solutions.

Reactivator

The reactivator consists of a 17-foot length of 10-inch schedule 40 steel pipe that is connected directly to the reboiler by 10-inch flanges. The reboiler is about 10 feet in length and 16 inches in diameter. It is fitted with a pressure gage, a liquid-level gage glass, a dial thermometer located near the end of the tube bundle, and connections for adding make-up condensate and draining the solution.

The steam used for reactification is controlled by a pressure controller and is measured either by a steam rotameter or by weighing the condensate.

hydrogen sulfide is removed and then to activated carbon drums where the organic sulfur is removed.

The fouled amine solution leaving the bottom of the absorber through a liquid level control valve flows by absorber pressure through a heat exchanger, where it is heated by hot lean amine solution, and enters into the upper section of the reactivator.

The fouled amine solution passes down through the reactivator to the reboiler where sufficient indirect heat is supplied with low-pressure steam to boil the amine solution and expel the acid gas. The hydrogen sulfide, carbon dioxide, and steam leave the top of the reactivator and pass through an acid gas cooler, and the steam is condensed and returned to the column. Whenever operation is such that the purified gas leaving the absorber (or activated carbon drums) is returned to the gas holder for recycling, the acid gas leaving the acid-gas cooler likewise is returned.

Reactivated amine solution leaves the reactivator, flows through the heat exchanger where it gives up a portion of its heat to the fouled solution leaving the absorber, and is pumped through a water-cooled solution cooler back to the top of the absorber.

ANALYTICAL METHODS

Whether the pilot plant was operating on TEA or DEA, the following analytical tests were necessary:

1. Hydrogen sulfide content of gas entering absorber.
2. Hydrogen sulfide content of gas entering and leaving the iron oxide boxes.
3. Hydrogen sulfide content of the acid gas.
4. Hydrogen sulfide content of the fouled amine solution.
5. Hydrogen sulfide content of the lean amine solution.
6. Thiosulfate content of the lean amine solution.
7. Carbon dioxide content of gas entering and leaving the absorber.
8. Carbon dioxide content of the lean amine solution.
9. Organic sulfur content of gas entering and leaving the absorber.
10. Total sulfur content of the lean amine solution.
11. Determination of free amine in solution.
12. Determination of total amine in solution.

sulfide and sodium thiosulfate concentrations, was considerably less than that obtained by titrating an iodine-amine solution which had stood for 24 hours. Inasmuch as it is possible to determine the concentrations of the various sulfur compounds, this phenomenon of excessive titration was not studied, but it is believed to result from the unsaturated hydrocarbons, which probably are absorbed by the amine solutions.

The sulfide sulfur and total sulfur contents of the amine solutions are determined as follows:

The hydrogen sulfide content is determined by one of two methods. In the first method, concentrated hydrochloric acid is added to the solution (contained in a closed flask) and the liberated hydrogen sulfide is swept from the flask with nitrogen into a Shaw flask containing 10 percent cadmium chloride. The excess of iodine then is titrated with standard thiosulfate solution. In the second method (recommended by Girdler), the sulfide is precipitated, as nickel sulfide using a 5 percent aqueous solution of nickel sulfate. The nickel sulfide is filtered from the solution and added (along with the filter paper) to an acidified iodine solution. The excess iodine is titrated with standard thiosulfate solution.

Because certain organic sulfur compounds do react with the amine solutions, it was advisable to know the total sulfur content of the lean solution. This was determined by pipetting a sample of the amine solution into a beaker containing an excess of bromine covered with dilute hydrochloric acid. The mixture is evaporated to dryness on a steam bath and the residue taken up with triple distilled water. The solution then is boiled, and the sulfur is precipitated as barium sulfate.

SELECTIVE REMOVAL OF HYDROGEN SULFIDE

As mentioned previously, it is feasible to obtain an acid gas high enough in hydrogen sulfide content to allow economic recovery of sulfur from gases containing low amounts of carbon dioxide. As the carbon dioxide content of a gas increases, selective removal of hydrogen sulfide becomes increasingly difficult and the cost of hydrogen sulfide removal rises. Using triethanolamine as the scrubbing agent, pilot-plant runs were made to study the effectiveness of this compound in removing hydrogen sulfide selectively from a gas containing relatively high amounts of carbon dioxide.

It also was desirable to obtain sufficient data so that cost estimates could be made on the purification of gases having various compositions. An extensive program was carried out, which included the study of various solution concentrations, the effect of varying concentrations of carbon dioxide and hydrogen sulfide in the raw gas on solution circulating rates, the effect of organic sulfur compounds on the purifying solution, the effect of scrubbing rates on residual hydrogen sulfide in the purified gas, and the foaming characteristics of amine solutions.

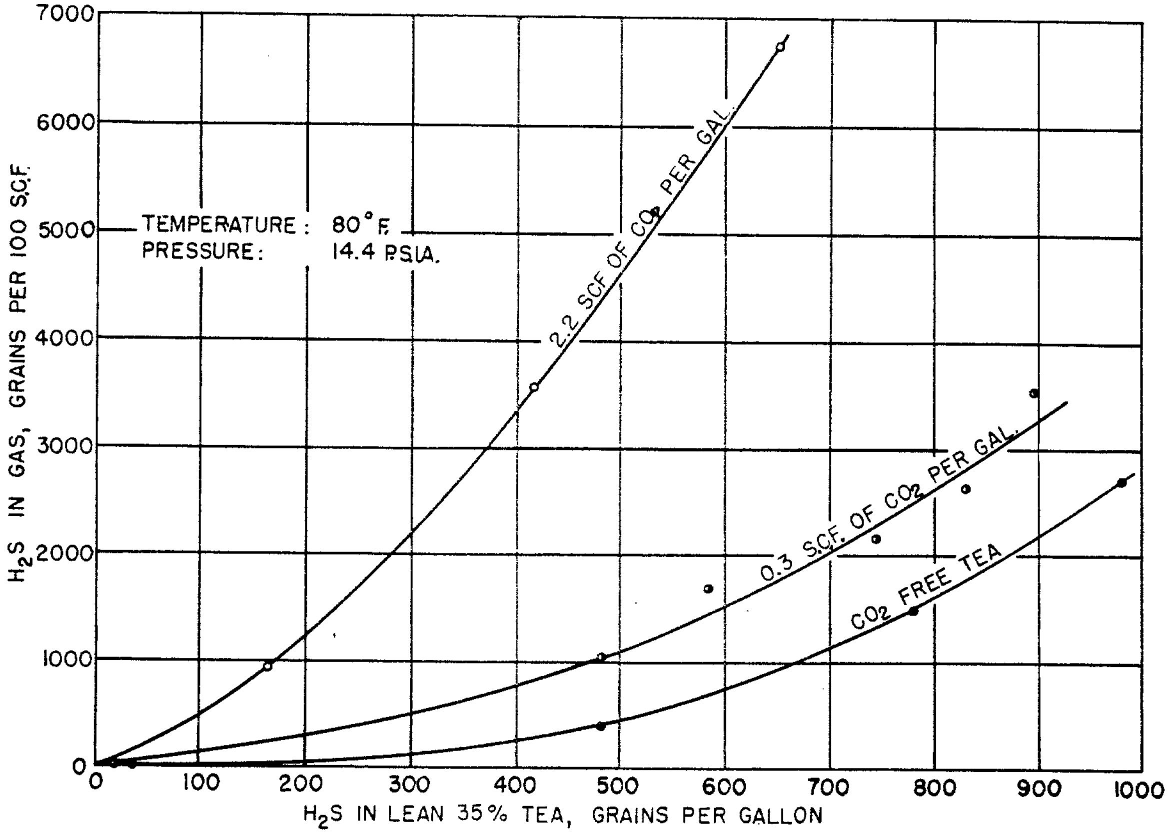

Figure 3. - Effect of CO_2 on the vapor pressure of H_2S over 35-percent TEA at 80° F. and 14.4 p.s.i.a.

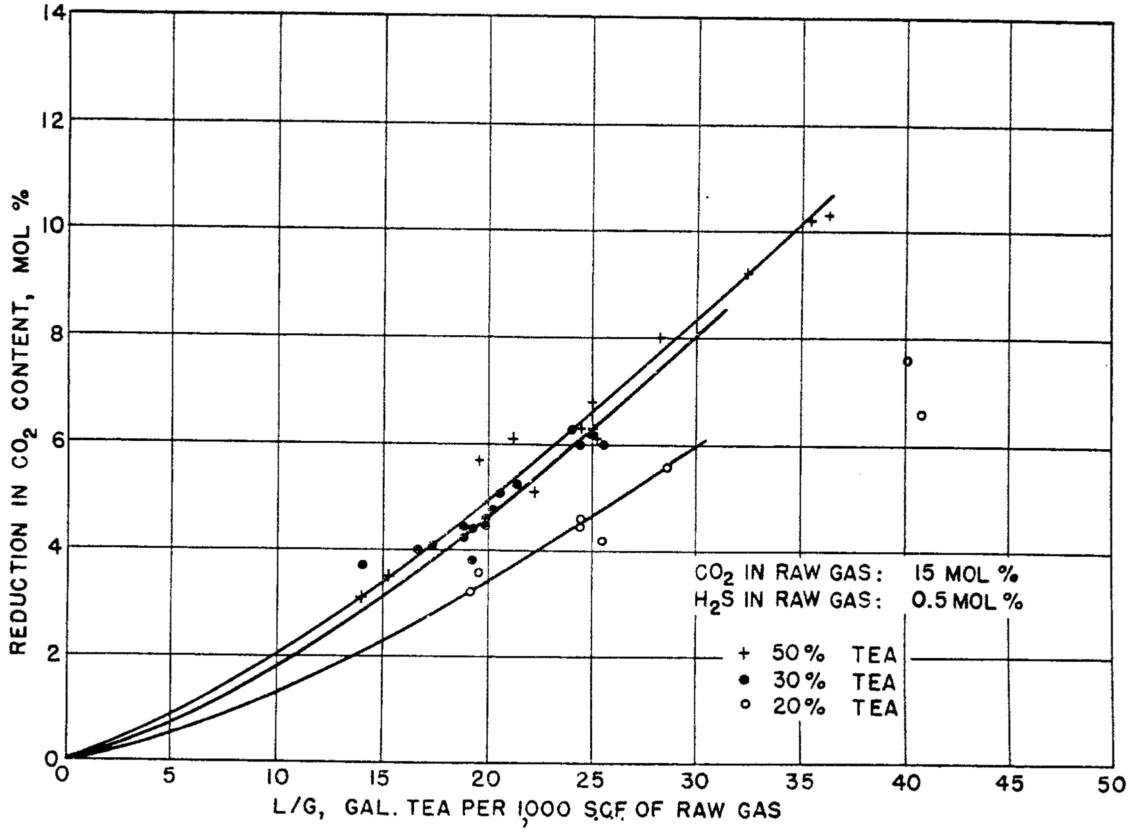

Figure 4. - Effect of TEA scrubbing rates on CO_2 removal.

TABLE 1. - Data from pilot-plant runs using TEA

Run No.	Solution strength, wt. percent	Type of gas	Solution rate, g.p.m.	Gas rate, std.c.f.h.	L/G, gal. sol./Ms.c.f. of gas	H_2S, gr./100 std.c.f.		CO_2, mol percent		H_2S in Acid gas, mol percent	H_2S in lean sol., gr./gal.
						in	out	in	out		
1-T	50	Syn.	1.0	1,850	32.4	350	25	22.4	12.5	4.4	20
2-T	50	do.	1.0	1,700	35.3	350	20	22.1	11.2	4.1	20
3-T	50	do.	1.0	2,050	29.2	350	30	21.8	13.1	4.8	-
4-T	50	do.	1.0	3,050	19.7	350	80	21.2	14.9	5.6	-
5-T	50	Inert	1.0	2,400	25.0	350	25	14.4	7.7	6.7	-
6-T	50	do.	1.0	4,150	14.5	220	75	13.8	10.8	6.7	75
7-T	50	do.	1.0	1,650	36.3	450	5	12.9	2.8	6.3	20
8-T	50	do.	1.8	4,300	25.1	375	25	12.7	6.8	8.0	20
9-T	50	do.	1.0	2,450	24.5	350	25	12.3	6.3	7.4	20
10-T	50	do.	1.0	2,400	25.0	200	10	11.3	5.4	4.6	30
11-T	50	do.	1.0	2,700	22.2	300	15	9.2	4.6	8.6	40
12-T	50	do.	1.0	2,800	21.4	320	25	9.1	3.6	7.7	40
13-T	50	Syn.	1.0	3,950	15.2	350	25	7.0	4.3	15.8	-
14-T	35	Inert	1.0	2,500	24.0	650	25	14.0	7.8	12.6	20
15-T	35	do.	1.0	2,450	24.5	650	25	13.8	7.9	13.1	20
16-T	35	do.	1.0	2,400	25.0	600	10	13.3	7.3	12.5	30
17-T	35	do.	1.0	3,100	19.3	380	65	11.4	8.0	11.9	65
18-T	35	do.	1.0	3,100	19.3	380	20	11.4	7.4	11.6	20
19-T	35	do.	1.0	3,000	20.0	300	60	10.3	6.3	8.1	120
20-T	35	do.	1.3	4,150	18.8	270	60	10.1	6.3	7.4	115
21-T	30	do.	1.4	4,150	20.2	300	40	13.9	9.2	7.4	20
22-T	30	do.	1.0	2,800	21.4	350	20	13.9	8.7	8.4	-
23-T	30	do.	1.0	2,350	25.6	350	5	13.8	7.9	7.9	-
24-T	30	do.	1.0	3,000	20.0	320	15	10.9	6.8	10.0	20
25-T	30	do.	1.0	2,900	20.7	280	10	10.4	5.7	7.6	20
26-T	30	do.	1.0	3,200	18.8	285	15	10.4	6.4	9.1	20
27-T	30	do.	1.0	3,450	17.4	310	20	10.3	6.6	10.4	20
28-T	30	do.	1.0	3,600	16.7	350	20	10.2	6.7	12.5	20
29-T	30	Syn.	.7	3,000	14.0	230	25	7.5	4.5	9.2	-
30-T	20	Inert	1.4	4,300	19.5	230	55	12.8	9.4	6.8	65
31-T	20	do.	1.7	4,250	24.0	330	60	12.7	8.3	8.3	85
32-T	20	do.	1.8	1,200	90.0	350	<5	11.8	.4	4.7	20
33-T	20	do.	1.0	2,500	24.0	350	20	11.0	6.9	10.7	20
34-T	20	do.	1.0	2,100	28.6	350	10	11.0	5.8	8.8	20
35-T	20	do.	1.0	1,500	40.0	350	<5	11.0	3.8	6.9	20
36-T	20	do.	1.4	4,400	19.1	310	35	5.4	3.2	16.4	95
37-T	20	do.	1.7	4,000	25.5	345	15	10.8	7.0	11.5	20

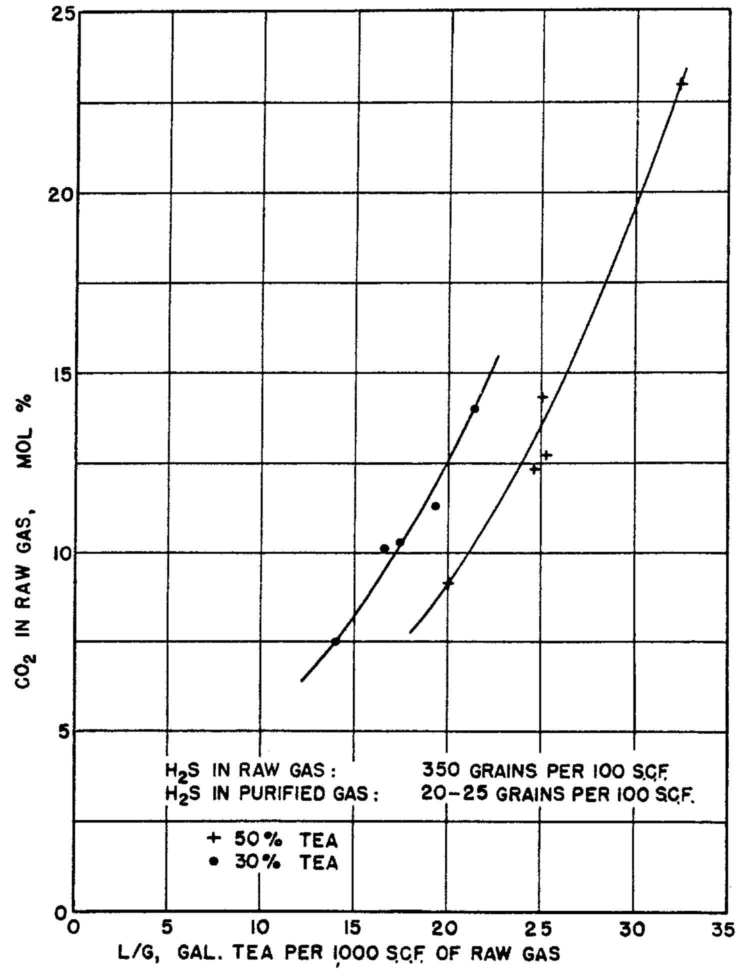

Figure 5. - Effect of CO_2 in raw gas on TEA scrubbing rates.

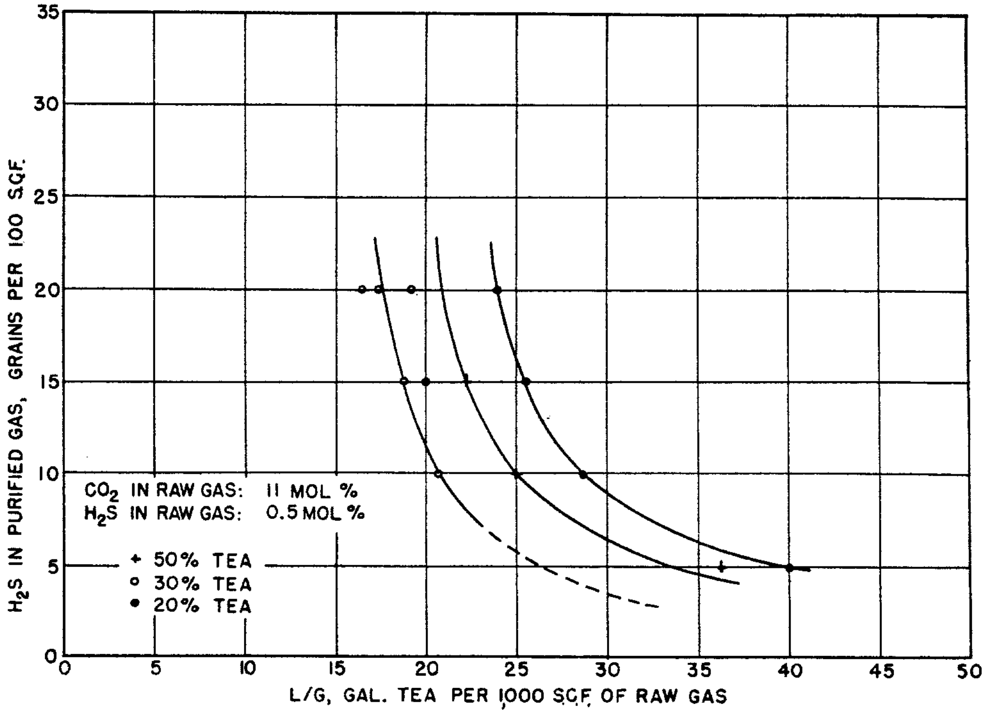

Figure 6. - Effect of TEA scrubbing rates on residual H_2S in purified gas.

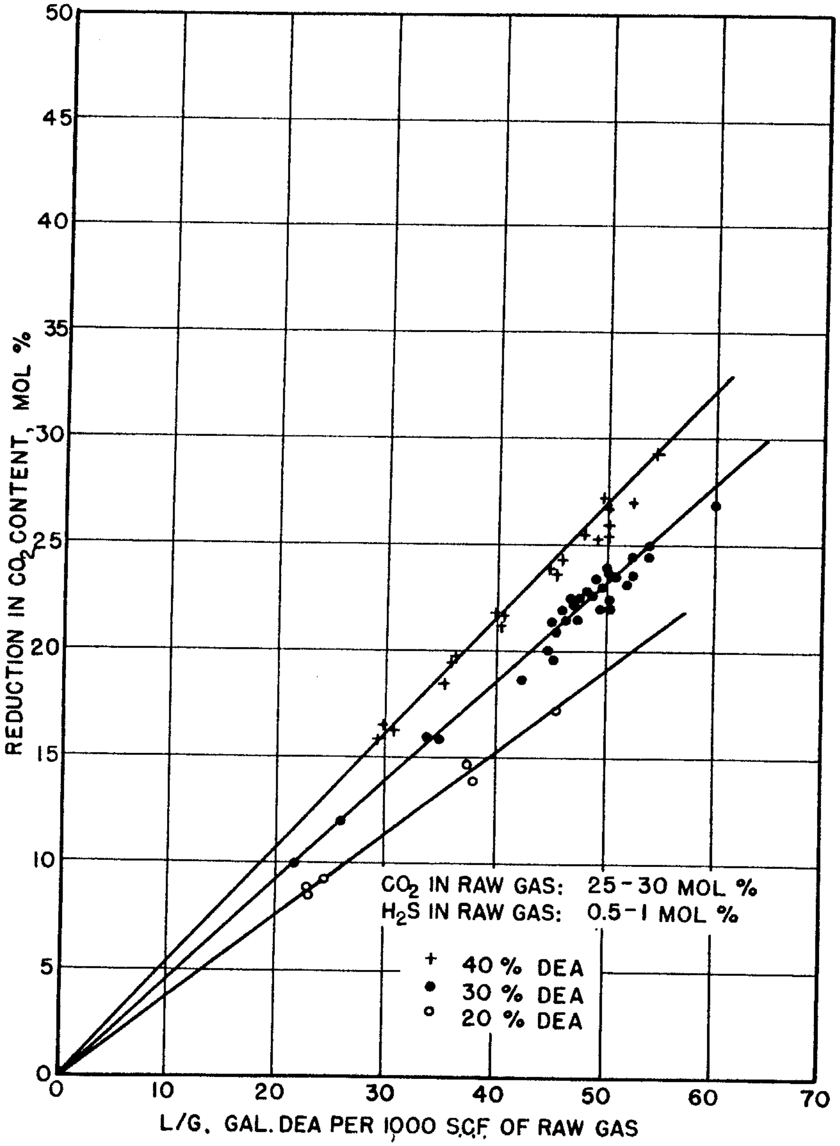

Figure 7. - Effect of DEA scrubbing rates on CO_2 removal.

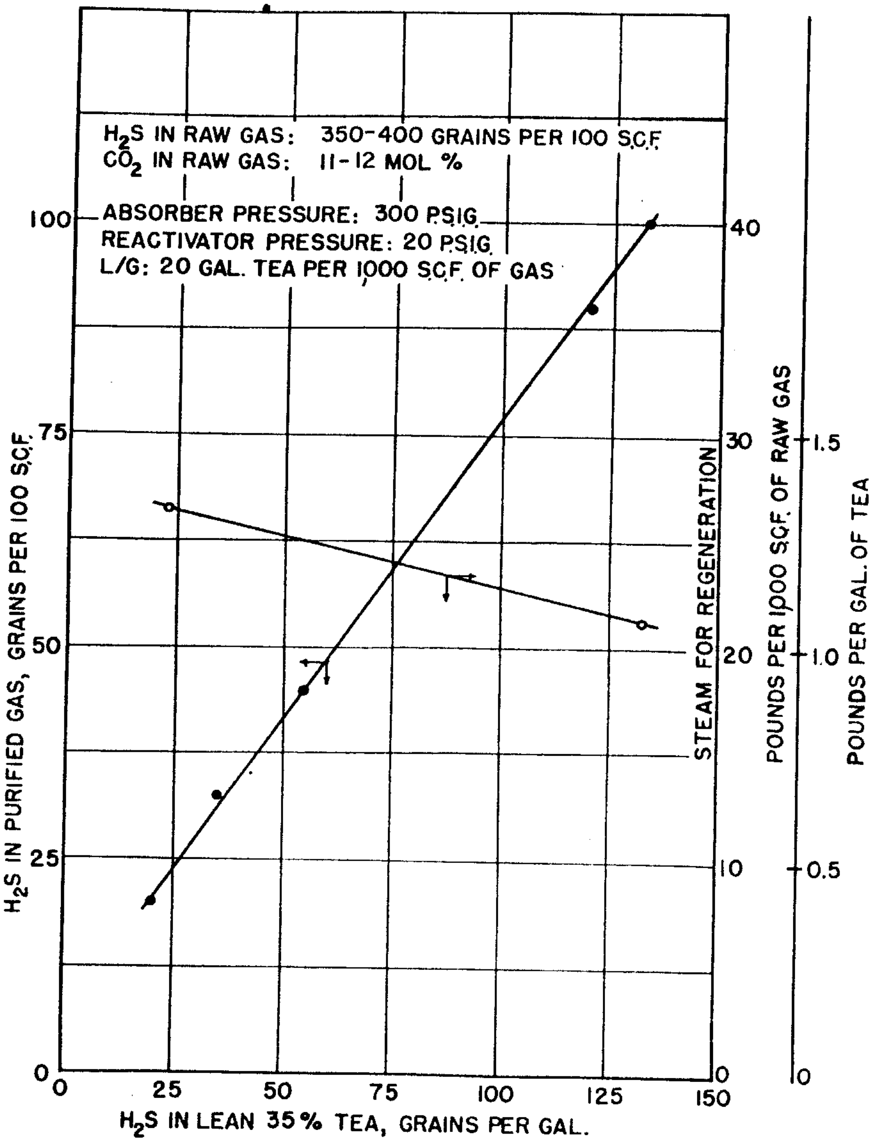

Figure 8. - Effect of H_2S concentration in lean 35-percent TEA on H_2S concentration in purified gas and on steam consumption at 300 p.s.i.g.

	Inlet gas	Outlet gas	Acid gas
CO_2	12.0 percent	7.7 percent	90 percent
H_2S	350 grains	25 grains	10 Do.

In order to obtain varying degrees of purity, estimates may be made by examining figure 6 and adjusting the L/G value accordingly.

Estimates of liquid:gas ratios for varying amounts of hydrogen sulfide in the raw gas may be made by referring to table 1, where a comparison of runs 14-T and 15-T with runs 21-T and 22-T shows that an increase from 350 to 650 grains of hydrogen sulfide per 100 std.c.f. requires an increase from 21 to 24 gal. per 1,000 std.c.f. in scrubbing rate.

SIMULTANEOUS REMOVAL OF HYDROGEN SULFIDE AND CARBON DIOXIDE

In accordance with the Bureau's agreement with the Southern Natural Gas Company, pilot-plant runs were made to study the efficiency of diethanolamine in removing both hydrogen sulfide and carbon dioxide from raw gas.

Fouled inert gas was used for the majority of the runs but synthesis gas, having the following compositions, was used in runs 1-D, 7-D, 8-D, 9-D, and 15-D.

TABLE 3. - Composition of synthesis gas for DEA runs

Run No.	CO_2	O_2	Ill.	H_2	CO	CH_4	N_2
1-D	31.7	0.6	0.9	27.9	34.3	2.1	2.8
7-D, 8-D	24.3	1.0	1.0	36.9	29.8	2.5	4.5
9-D	21.1	.4	.8	38.9	33.0	2.7	3.1
15-D	18.6	.9	1.2	37.2	33.4	4.7	4.0

DEA aqueous solutions of 20, 30, and 40 percent were tested with gases having carbon dioxide contents ranging from 13 to 30 percent and hydrogen sulfide concentrations from 175 to 600 grains per 100 std.c.f. With the exception of run 12-D, in which the absorber was operated at 200 p.s.i.g., all runs were made with the absorber at 300 p.s.i.g. Data from 17 runs are presented in table 4. Most of these data, plus some additional data, have been plotted (fig. 7) to show the effect of solution rate on the amount of carbon dioxide removed from a raw gas containing from 25 to 30 percent carbon dioxide.

A comparison of the data from the run at 200 p.s.i.g., run 12-D, with those from the runs made at 300 p.s.i.g. shows that the efficiency for removing acid gas is about the same at both pressures. On the basis of the results from this one run, it appears that the only real advantage of the higher pressure is the additional volume of gas that can be treated without increasing the size of the equipment.

Runs 13-D and 14-D were made using 30 percent DEA and one-inch Raschig rings in the absorber. Comparison of these data with those from runs in which the absorber was packed with half-inch rings shows that greater efficiency was obtained with the smaller rings.

The purpose of runs 10-D and 11-D was to find the minimum solution rate at which purification to 25 grains of hydrogen sulfide per 100 std.c.f. could be obtained. It was found that L/G ratios comparable to those used with 30 percent TEA were necessary, the difference being in the amount of carbon dioxide removed. Whereas the TEA removed only 36 percent of the carbon dioxide in the raw gas, the DEA removed about 63 percent.

STEAM CONSUMPTION FOR REACTIVATION

Table 5 summarizes the steam consumption for several runs using TEA and DEA. Steam consumption, of course, increased as the reactivator pressure was increased, about 1.0 to 1.1 pounds being used per gallon of circulating solution whenever the reactivator pressure was 5 p.s.i.g. As the reactivator was small and partly exposed to the weather, climatic conditions affected the steam consumption somewhat. A commercial plant operating under similar conditions would undoubtedly use less steam per gallon of solution. The steam figures shown in the table were obtained by weighing the condensate.

TABLE 5. - Steam consumption for reactivation

Run No.	Solution and strength, percent	Reboiler pressure, p.s.i.g.	Steam consumed, lb. per gal.
28-T....	30 TEA	20	1.33
8-D....	30 DEA	10	1.18
33-T....	20 TEA	10	1.13
15-T....	35 do.	6.5	1.15
14-T....	35 do.	5	1.07
5-T....	50 do.	5	1.06
7-D....	30 DEA	5	1.06
9-T....	50 TEA	5	1.04
5-D....	40 DEA	5	1.01
34-T....	20 TEA	5	.96

Unlike operation with monoethanolamine, in which the reactivator must be operated at elevated pressures (higher temperatures) in order to substantially remove carbon dioxide from the fouled solution completely, virtually complete reactivation of DEA and TEA can be effected at atmospheric pressure. Reed and Wood[17] attribute this to the greater alkalinity of MEA as compared to DEA and TEA. As would be expected, the operation at the higher reactivator pressures removed carbon dioxide from the DEA and TEA more completely, and the hydrogen sulfide less completely.

The concentration of hydrogen sulfide in the gas leaving the absorber depends upon the amount of hydrogen sulfide remaining in the lean solution, which, in turn, is controlled by the degree of reactivation. As the steam usage constitutes the greatest part of hydrogen sulfide removal costs, data were obtained on the amount of steam required to give various concentrations

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[17] Reed, R. M., and Wood, W. R., Recent Design Developments in Amine Gas Purification Plants: Trans. Am. Inst. Chem. Eng., vol. 37, 1941, pp. 363-383.

TABLE 6. - Removal of organic sulfur with TEA and DEA (Cont.)

Solution strength, wt., percent	L/G, gallon solution per 1,000 std.c.f. gas	Organic S, grains/100 std.c.f.		Organic sulfur removed, percent
		Before absorber	After absorber	
		TEA		
50	30.0	40.0	32.0	20.0
50	32.2	21.7	12.6	41.9
50	19.2	31.3	21.5	31.3
50	27.7	17.4	12.1	30.5
50	34.6	11.7	6.7	42.7
50	35.2	5.5	5.1	7.3
50	31.2	14.2	5.8	59.1
50	35.2	11.1	4.6	58.6
50	33.8	7.9	3.2	59.4
50	26.1	36.0	23.4	35.0
50	26.2	37.0	22.6	38.9
50	15.7	34.8	24.1	44.3
30	40.8	28.8	15.8	45.2
30	18.4	38.0	14.8	61.1
30	17.6	19.8	11.1	43.8

Laboratory analyses of the lean amine solutions definitely established a gradual build-up of sulfur compounds in the lean solutions. The distribution of sulfur compounds in a lean TEA and DEA solution is shown by the following analyses:

	Grains per gallon	
	TEA	DEA
Total apparent H_2S (excess I_2 titrated immediately)	68	170
Total apparent H_2S (excess I_2 titrated after 2 days)	139	723
H_2S content determined from NiS precipitate	39	-
H_2S content determined by liberation with acid	42	34
Thiosulfate content of lean solution	6	98
Sulfate content of lean solution	0	0
Total sulfur content (by precipitating as $BaSO_4$)	174	495

It can be observed that the total sulfur content of the lean solutions greatly exceeded the combined hydrogen sulfide and thiosulfate concentrations. The effect of time on the total apparent hydrogen sulfide content of the solutions can also be noted. As stated previously, this great difference is probably due to the presence of unsaturated compounds. No such difference in values occurred whenever the amine solutions were used with inert gas only.

These results indicate that the compounds formed by the amines and organic sulfur do not break down under the ordinary operating conditions of our reactivator.

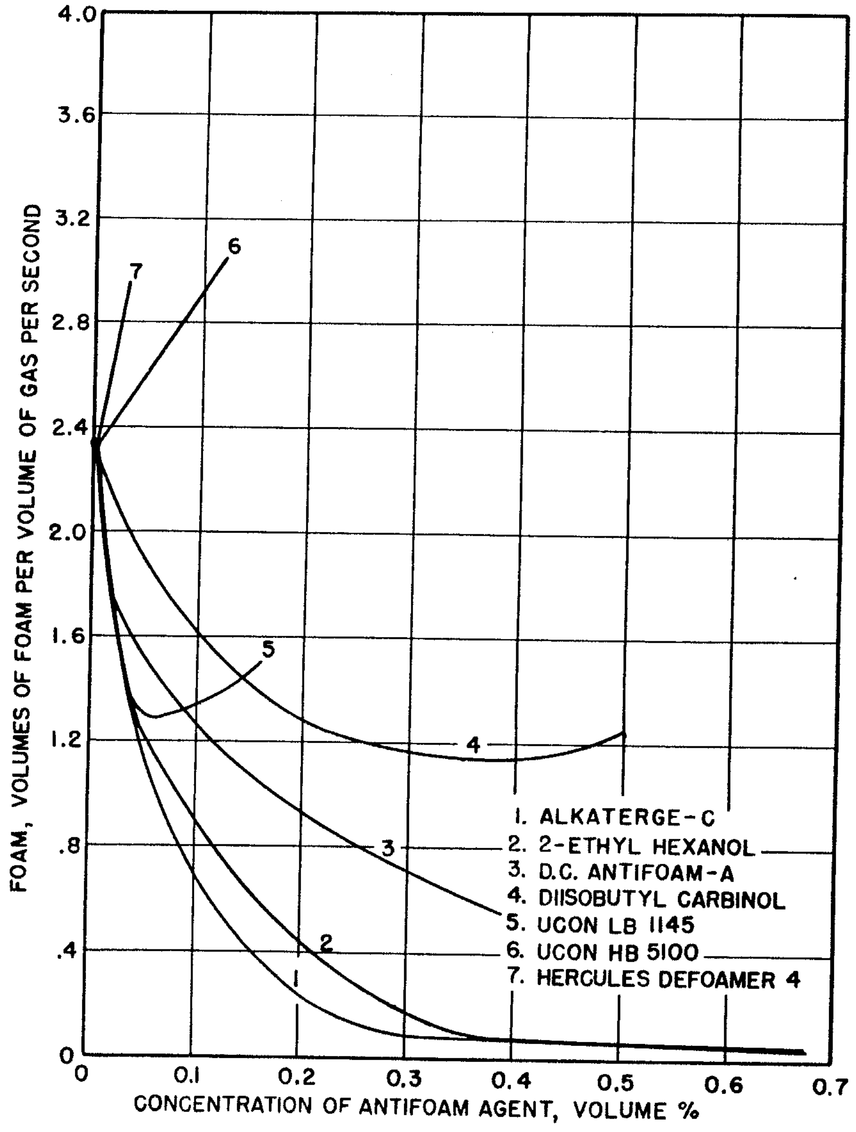

Figure 9. - Characteristics of antifoam agents with 35-percent TEA.

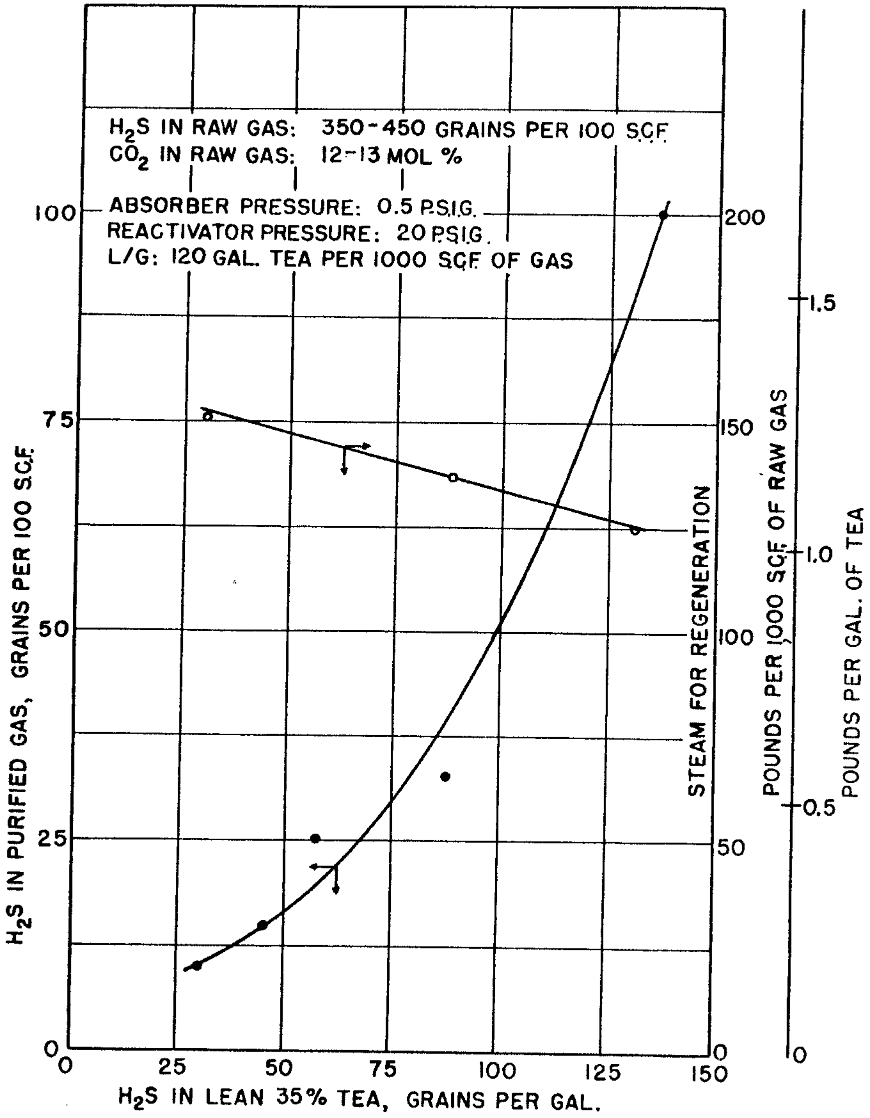

Figure 10. - Effect of H_2S concentration in lean 35-percent TEA on H_2S concentration in purified gas and on steam consumption at 0.5 p.s.i.g.

those from the pressure run in figure 8. It can be noted that at atmospheric pressure a scrubbing rate of 120 gallons per 1,000 std.c.f. of gas is needed to reduce the hydrogen sulfide content from 400 to 25 grains per 100 std.c.f. This rate is about 6 times the circulation rate needed at 300 p.s.i.g. The steam consumption for the low-pressure operation appears to be about 10 percent less on the basis of pound of steam per gallon of solution and about 5.5 times as much for each 1,000 cubic feet of gas purified. Results from this one run indicated that the selective absorption of hydrogen sulfide is improved somewhat at elevated pressures.