

FIGURE 34. - Agitator Absorber Tested for Use in Gas Purification.

50° F. below the temperature of the main stream. Split-stream flow was particularly advantageous for reducing the carbon dioxide content of the purified gas to less than 1 percent. With more thorough regeneration of the split stream in a small secondary regenerator, the carbon dioxide content of the gas was lowered to 0.2 percent without changing the steam requirements appreciably.

could be collected after depressurizing. A 40-percent equivalent concentration of potassium carbonate was used, with 0.2 to 0.3 percent of sodium dichromate to prevent corrosion (3). In the first pilot plant, steam required for regenerating the spent carbonate solution was about one-third to one-half of that for a solution of ethanolamine. However, because the packed heights of the absorber and regenerator were only 9 and 4.75 feet, respectively, the concentration of carbon dioxide could not be lowered sufficiently.

In the second plant the heights of the packed sections were increased to about 28.5 and 25 feet, respectively. In the absorber, packing was divided into a top section of 3.5 feet and a main section of 25 feet. A small portion of the regenerated solution could be fed to the top entry, while the remainder entered above the main section of the packing (fig. 36). Equilibrium pressure above the top stream was decreased by cooling it from 15° to

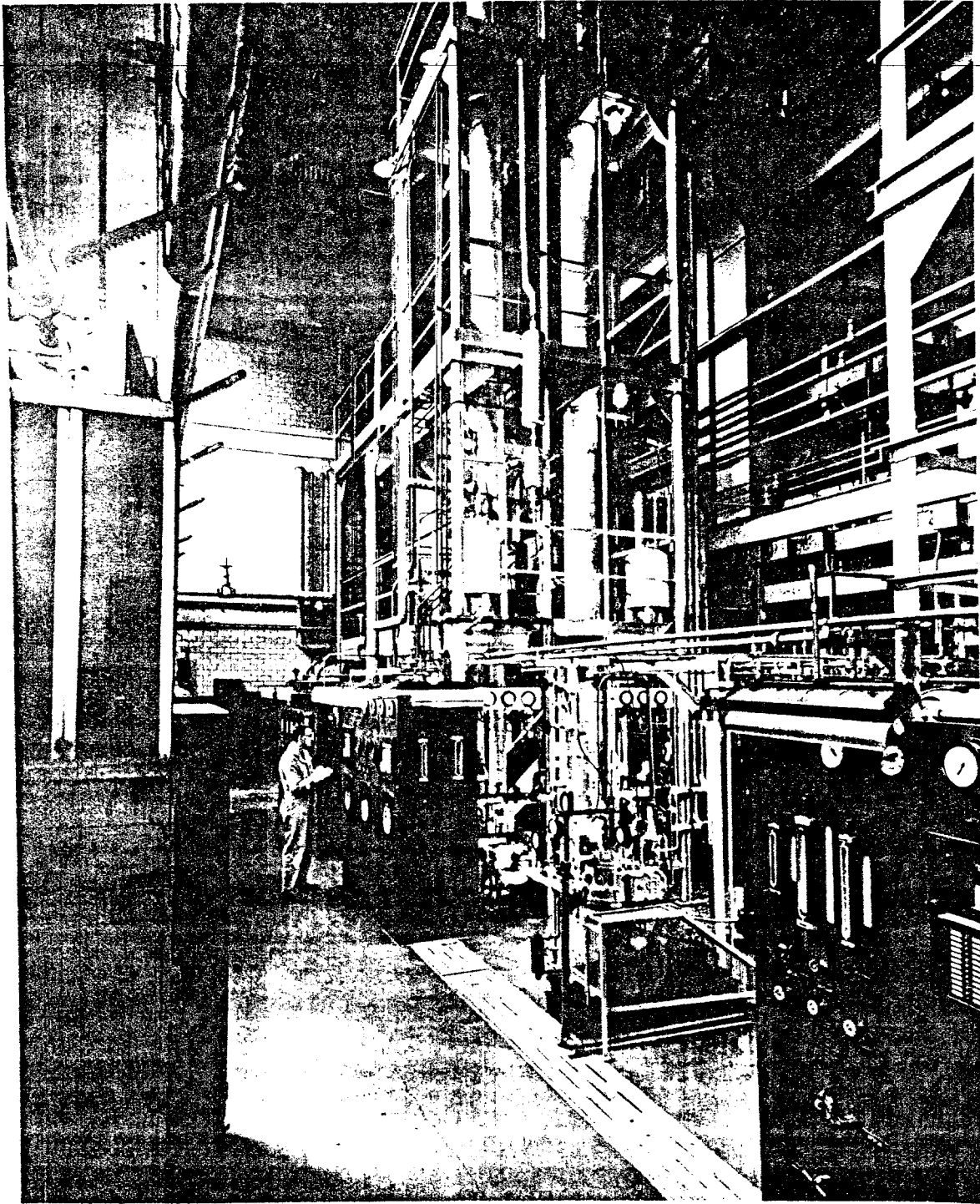


FIGURE 35. - Pilot Plant for Removing Carbon Dioxide From Synthesis Gas by Hot Carbonate Scrubbing.

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Pilot-plant tests were made with gases containing 10 to 16 percent of carbon dioxide and 0.15 to 0.8 percent of hydrogen sulfide, to investigate their simultaneous removal. About the same percentage removal was achieved for both compounds. When the carbon dioxide concentration was lowered from 10 to 0.6 percent (about 95-percent removal), the hydrogen sulfide concentration was reduced from about 0.5 percent to 0.025. Regeneration efficiency was the same as when no hydrogen sulfide was present. Lowering of the hydrogen sulfide concentration in the purified gas from 0.5 percent to 20 to 30 parts per million (0.002 to 0.003 percent) has been achieved in pilot-plant tests. Carbonyl sulfide in concentrations of 20 to 30 grains per 100 cu. ft. has been completely removed; it is hydrolyzed to carbon dioxide and hydrogen sulfide by the hot carbonate solution.

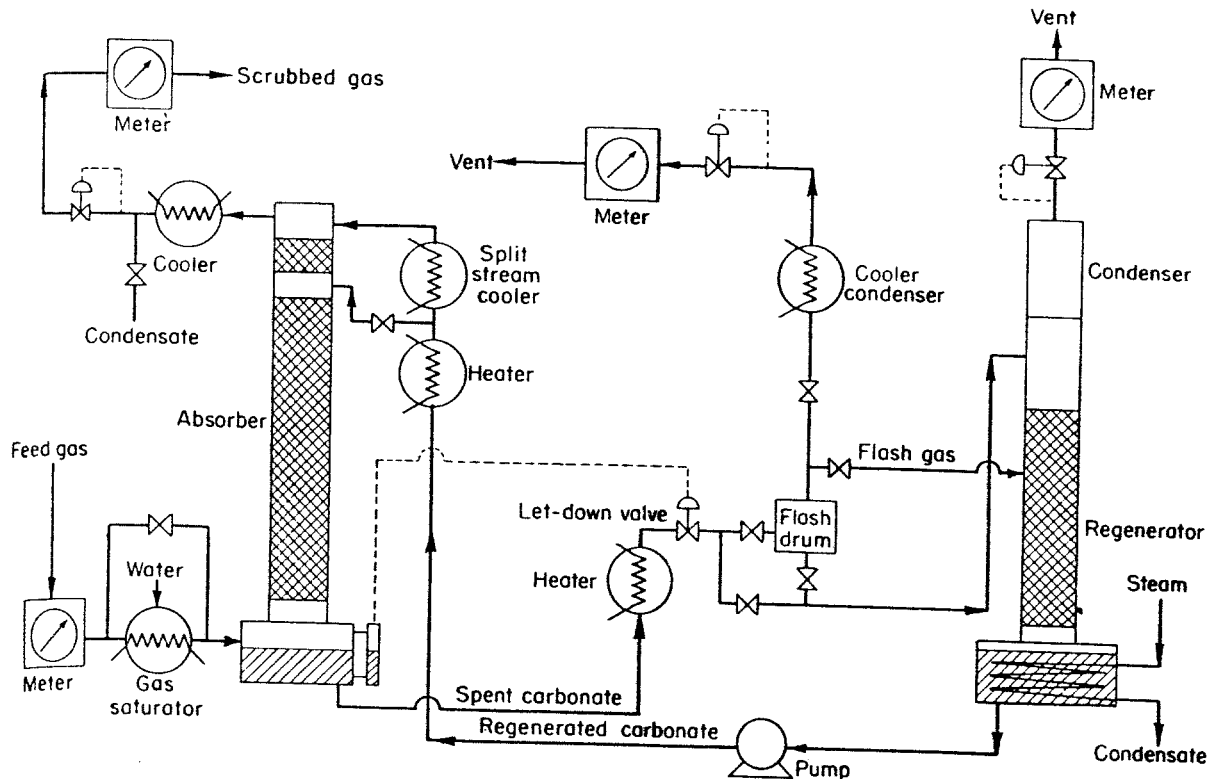


FIGURE 36. - Schematic Diagram of Pilot Plant for Hot Carbonate Scrubbing.

As noted in table 7, the cost of scrubbing by hot carbonate solution, provided gas is made or used under pressure, is estimated at about one-half that of an amine system (3). Decreased cost results from elimination of heat exchangers, decreased requirements for heaters and coolers, and halving of cooling-water requirements. Pumping costs were assumed to be the same for both processes, and losses of absorbent were not included.

TABLE 7. - Cost of removing 1,000 cu. ft. of carbon dioxide from synthesis gas at 300 p.s.i. absorber pressure

| | Amine | Hot carbonate |
|--|--------|---------------|
| Steam (\$0.40 per 1,000 lb.)..... | \$0.10 | \$0.04 |
| Equipment (20 percent per year)..... | .071 | .048 |
| Cooling water (\$0.01 per 1,000 gal.)..... | .02 | .01 |
| Total..... | .191 | .098 |

Gas Purification in the Demonstration Plants

The research results from Morgantown were used on a larger scale at the Bureau of Mines Demonstration Plant at Louisiana, Mo. (22, 24, 25). The three-step purification process operated here (see fig. 37) included:

1. Bulk removal of hydrogen sulfide and carbon dioxide by pumping the gas through an absorber column packed for 35 feet with Raschig rings and containing 40 percent diethanolamine solution.
2. Removal of residual hydrogen sulfide by passing the gas downward through two beds in series of iron oxide on wood chips.
3. Removal of organic sulfur compounds by passing the gas downward through two beds in series of activated carbon.

Operation was usually at the rate of 2,000,000 cu. ft. per day of gas containing initially 16 to 20 percent of carbon dioxide and 100 grains of sulfur per 100 cu. ft. This gas was purified to contain only 2 to 5 percent of carbon dioxide and less than 0.01 grain of total sulfur per 100 std. c. f. (24).

A significant difference between the pilot-plant results and results in this demonstration plant was in the activated-carbon adsorption step. In the pilot plants it had been possible to treat about 400 cu. ft. of gas per pound of carbon before regeneration of the carbon became necessary. On the plant scale, twice this amount of gas could be treated ordinarily before regeneration was needed, and in one special test 10 times this amount was treated before the effluent gas showed 0.1 grain of sulfur per 100 std. c. f. However, these plant operations were made with a gas containing 0.3 to 1.0 grain of sulfur per 100 std. c. f. and in a bed of greater depth and diameter (22).

Purification sufficient for use of the gas in synthetic fuel production was accomplished satisfactorily with synthesis gas produced from coke in a Kerpely producer and with synthesis gas from coal gasification.

Technology Yet to be Solved

Dust Removal

If gasification by means of nuclear energy becomes practical the dust problem may become most critical. Rigid purification might then be necessary to remove all radioactive dust from the gas stream.

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Large agglomerates can be removed at relatively low cost, as compared to the extreme difficulty of removing very fine particles. Accordingly, dust problems may be attacked more effectively if further information is obtained about the behavior of dusts in gas streams, particularly as to forces causing agglomeration.

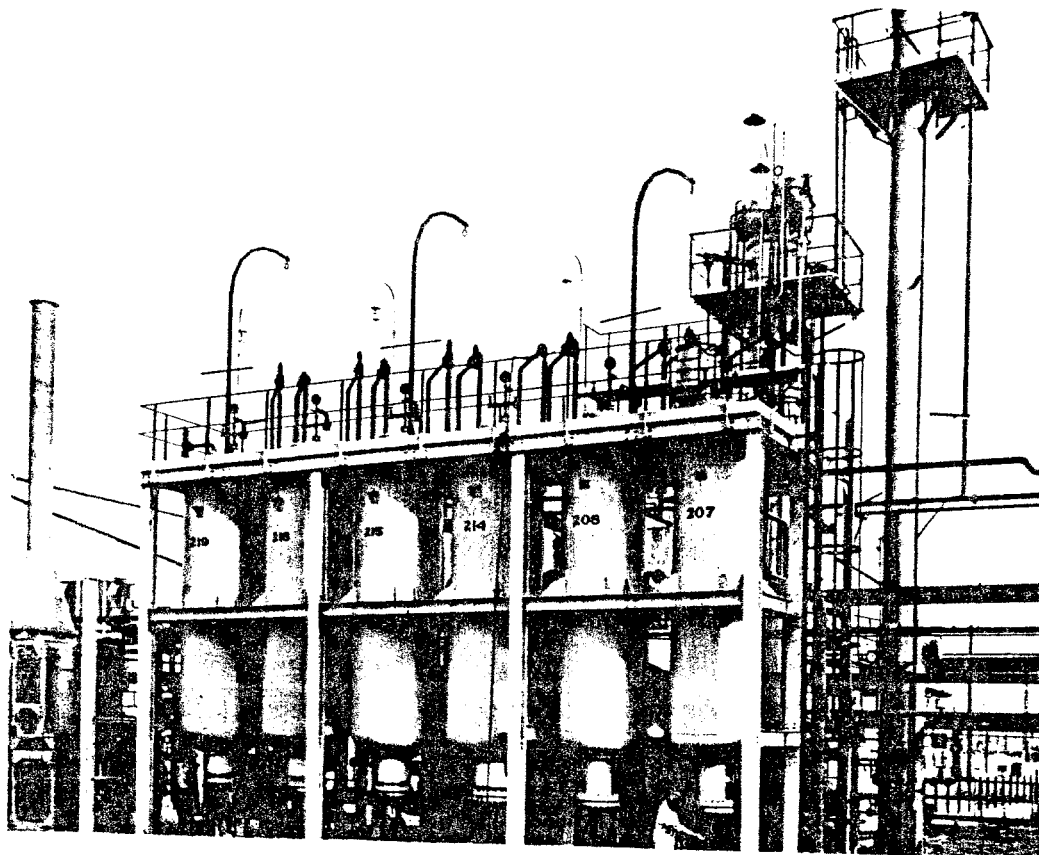


FIGURE 37. - Equipment for Removing Sulfur From Synthesis Gas in Bureau of Mines Demonstration Plant.

Evaluation of various dust-removing equipment used in pilot-plant gasification runs should be continued. Such results will be of greater value by use of a method, developed by the Bureau's laboratories at Morgantown, for determining the particle or agglomerate size distribution of dust as it actually exists in the gas stream.

The moving-bed filter, a promising device for fine purification, even of hot gases under pressure, deserves further improvement. The high-pressure scrubber also gave good results in the pilot plant. A smaller version (see fig. 38) is now installed for tests in the laboratory. For laboratory tests of dust-removing equipment, a suitable feeding device for adding dust to gas streams will be advantageous and is being developed.

There is need for more work on dust cleanup of synthesis gas at pressures of 30 atmospheres and temperatures up to 1,000° F. Successful dust removal from hot synthesis gas would save heat, and heat-transfer equipment.

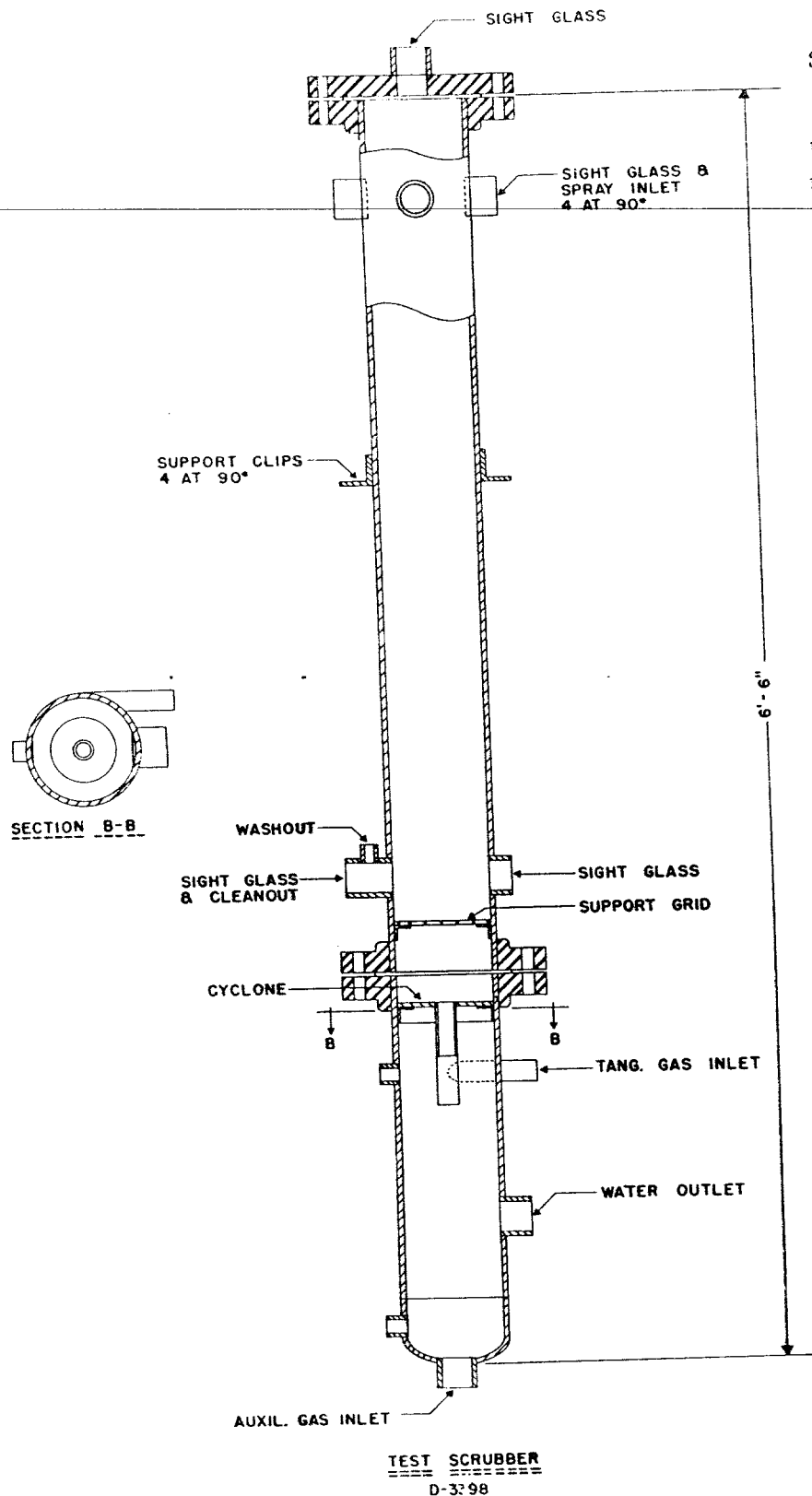


FIGURE 38. - Scrubber for Laboratory Tests on Removal of Dust From Gas.

Sulfur and CO₂ Removal

Research is needed both as to basic information and its application in practical gas purification. In view of the results at the demonstration plant, experiments should be continued on the effect of low concentrations of organic sulfur, low linear velocities, and greater depth of bed on activated carbon efficiency. Studies should be pursued on simultaneous removal of organic sulfur and relatively large quantities (1/2 to 1 percent) of hydrogen sulfide with activated carbon, using the fluidized-bed technique. Complete removal of large quantities of hydrogen sulfide by iron oxide under pressure should be studied in both fixed and fluidized beds.

Although the aluminosilicates ("molecular sieves") available at present have not been promising for removing sulfur compounds from synthesis gas, some further research may be desirable in testing new selective sieves, with particular attention to simultaneous removal of hydrogen sulfide and organic sulfur.

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Literature Cited

1. BENSON, H. E., AND FIELD, J. H. Perfectionnements relatifs au procédé de séparation de l'anhydride carbonique et de sulfure d'hydrogène d'avec des mélanges gazeux. French Patent 1,076,348. Granted Apr. 21, 1954, pub. Oct. 26, 1954.
2. _____. Separation of Carbon Dioxide and Hydrogen Sulfide From Gas Mixtures. British Patent 725,000, Mar. 2, 1955.
3. BENSON, H. E., FIELD, J. H., AND HAYNES, W. P. Improved Process for CO₂ Absorption Uses Hot Carbonate Solutions. Chem. Eng. Progress, vol. 52, No. 10, October 1956, pp. 433-438.
4. BENSON, H. E., FIELD, J. H., AND JIMESON, R. M. CO₂ Absorption Employing Hot Potassium Carbonate Solutions. Chem. Eng. Progress, vol. 50, No. 7, July 1954, pp. 356-364.
5. BUREAU OF MINES. Synthetic Liquid Fuels - Annual Report of the Secretary of the Interior for 1952. Part I. - Oil From Coal. Rept. of Investigations 4942, 1953, 85 pp.
6. _____. Synthetic Liquid Fuels - Annual Report of the Secretary of the Interior for 1954. Part I.- Oil From Coal. Rept. of Investigations 5118, 1955, 73 pp.
7. _____. Synthetic Liquid Fuels - Annual Report of the Secretary of the Interior for 1955. Part I. - Oil From Coal. Rept. of Investigations 5236, 1956, 42 pp.
8. EGGLESON, G. C., SIMONS, H. P., KANE, L. J., AND SANDS, A. E. The Moving-Bed Coke Filter: A Practical Method for Removing Dust From Gas Streams. Bureau of Mines Rept. of Investigations 5033, 1954, 8 pp.
9. _____. Moving Coke-Bed Gas Filter for Dust Removal. Ind. Eng. Chem., vol. 46, No. 6, June 1954, pp. 1157-1162.
10. KANE, L. J., WAINWRIGHT, H. W., SHALE, C. C., AND SANDS, A. E. Determination of Solid and Liquid Impurities in Synthesis Gas. Bureau of Mines Rept. of Investigations 5045, 1954, 23 pp.
11. MINISTRY OF FUEL AND POWER. Report on the Petroleum and Synthetic Oil Industry of Germany. London, H. M. Stationery Office, 1947, 134 pp.
12. PURSGLOVE, L. A., AND WAINWRIGHT, H. W. Colorimetric Determination of Carbonyl Sulfide in Synthesis Gas. Anal. Chem., vol. 26, No. 11, November 1954, pp. 1835-1839.
13. SANDS, 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, 1949, 19 pp.

14. SANDS, A. E., AND SCHMIDT, L. D. Recovery of Sulfur From Synthesis Gas. Ind. Eng. Chem., vol. 42, No. 11, Nov. 1950, pp. 2277-2287.
15. 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.
16. SANDS, A. E., WAINWRIGHT, H. W., AND SCHMIDT, L. D. Purification of Synthesis Gas Produced From Pulverized Coal. Ind. Eng. Chem., vol. 40, No. 4, April 1948, pp. 607-620.
17. SEBASTIAN, J. J. S. Powdered-Coal Gasification - Effect of Variables. Ind. Eng. Chem., vol. 44, No. 5, May 1952, pp. 1175-1184.
18. STONE, D. E., KANE, L. J., CORRIGAN, T. E., WAINWRIGHT, H. W., AND SEIBERT, C. B. Investigation of a Photoelectric Device for the Determination of Low Concentrations of Dust. Bureau of Mines Rept. of Investigations 4782, 1951, 6 pp.
19. WAINWRIGHT, H. W., EGLESON, G. C., AND BROCK, C. M. Laboratory-Scale Investigation of Catalytic Conversion of Synthesis Gas to Methane. Bureau of Mines Rept. of Investigations 5046, 1954, 10 pp.
20. WAINWRIGHT, H. W., EGLESON, G. C., BROCK, C. M., FISHER, J., AND SANDS, A. E. Removal of Hydrogen Sulfide and Carbon Dioxide From Synthesis Gas Using Di- and Tri-ethanolamine. Bureau of Mines Rept. of Investigations 4891, 1952, 19 pp.
21. WAINWRIGHT, H. W., EGLESON, G. C., BROCK, C. M., FISHER, J., AND SANDS, A. E. Selective Absorption of Hydrogen Sulfide From Synthesis Gas. Ind. Eng. Chem., vol. 45, No. 6, June 1953, pp. 1378-84.
22. WAINWRIGHT, H. W., KANE, L. J., WILSON, M. W., SHALE, C. C., AND RATWAY, J. Purification of Synthesis Gas. Removal of Dust, Carbon Dioxide, and Sulfur Compounds. Ind. Eng. Chem., vol. 48, No. 7, July 1956, pp. 1123-1133.
23. WAINWRIGHT, H. W., AND LAMBERT, G. I. Colorimetric Method for the Determination of Thiophene in Synthesis Gas. Bureau of Mines Rept. of Investigations 4753, 1950, 11 pp.
24. WENZELL, L. P., JR., DRESSLER, R. G., AND BATCHELDER, H. R. Plant Purification of Synthesis Gas. Ind. Eng. Chem., vol. 46, No. 5, May 1954, pp. 858-862.
25. WILLMOTT, L. F., BATCHELDER, H. R., WENZELL, L. P., JR., AND HIRST, L. L. Performance of a Girbotol Purification Plant at Louisiana, Mo. Bureau of Mines Rept. of Investigations 5196, 1956, 18 pp.