

In the lower-boiling fractions, phenol and o-cresol were isolated as pure compounds and m- and p-cresol as m- and p-methyl-phenoxyacetic acids. Four isomeric xylenols were found. 3,5-Xylenol and 2,5-xylenol were isolated as pure compounds. 2,4-Xylenol was identified through its N-(2-fluorenyl)-carbamate; 3,4-xylenol was not detected by chemical methods but was shown by infrared spectroscopy to be present in small quantities. Of the ethylphenols, only one, m-ethylphenol, was identified by chemical means; o- and p-ethylphenols were shown to be present by infrared spectroscopy.

Analysis of the higher-boiling fractions is considerably more difficult because of the greater complexity of the tar-acid mixtures and because no infrared reference spectra are available. However, application of the countercurrent distribution technique has resulted in the isolation and identification of 4- and 5-indanol and p-phenylphenol. 3-Methyl-5-ethylphenol and o-phenylphenol were also isolated and identified. The countercurrent distribution technique, when applied to a synthetic mixture of 3,5-xylenol, 2,5-xylenol, and 2,4-xylenol or a mixture of o-, m-, and p-ethylphenols, shows that 3,5-xylenol and o-ethylphenol can be readily separated from their respective mixtures. The shapes of the distribution curves show that each compound distributes itself essentially independently of the others, in accordance with the distribution law. This technique permits measurement of partition coefficients of the components of a mixture without isolation of the individual compounds. These coefficients should be considered in the same category as the boiling point, melting point, and refractive index of a compound.

The presence of relatively large amounts of the indanols suggests that their unsaturated analogues - the hydroxy indenenes - may constitute a significant fraction of the "reactive fragments" formed in the primary hydrogenation of coal.

Gasification of Coal

Vortex Combustion Studies for Powdered-Coal Gasification

An experimental unit of the vortex type (see Figs. 48 and 49) was operated in a series of tests in which coal was gasified with oxygen and steam. Material and heat balances were calculated from the resulting data and indicate that the following correlations may be drawn:

1. The percentage of carbon gasified is almost directly proportional to the pounds of oxygen supplied per pound of coal in the feed; it is independent of the throughput, which appears to be a result of the hydrodynamics of the unit. At higher throughputs, despite the shorter residence time of the gas, higher tangential velocities occur in the vortex, thus keeping smaller coal particles in the vortex longer than in the case of lower throughputs.
2. The water-gas equilibrium is reached in the exit gases.
3. The reduction of carbon dioxide by carbon proceeds so slowly that the ratio $\frac{P_{CO}}{P_{CO_2}}$ is about 1/100,000 of the theoretical value at exit gas temperature.
4. The volume of carbon monoxide and hydrogen produced per pound of coal gasified is a function of the heat lost from the unit. On extrapolation of the data to zero heat loss, a production of 30 to 40 cubic feet of carbon monoxide and hydrogen per pound of coal gasified was predicted. However, the actual heat losses for this unit varied from 20 to 40 percent, and this extrapolated value must be used cautiously. Examination of the order in which the reactions occur in the vortex unit

indicate that the gasification efficiency of the unit depends largely upon the rate of the steam-carbon reaction.

Oxygen Production by Multistage Fractional Permeation

An important factor in the cost of coal gasification is the cost of oxygen used in the process. At present, oxygen is produced commercially almost exclusively by the Linde process. The fact that many organic membranes are appreciably more permeable to oxygen than they are to nitrogen is the basis for experimental work on a possible process for oxygen production.

Major improvements in the economy of a permeation process could be made by (1) increasing the magnitude of the film permeabilities or (2) increasing the selectivity of the films toward oxygen. Improvements in (1) would permit a decrease in the area of the film required to achieve a given throughput; this might be achieved by finding more permeable films or by making thinner films. Improvements in (2) would not only decrease the power requirements necessary for a given oxygen production but would also decrease the size of the plant required. Such improvements might be achieved by finding films more selective than rubber latex or by incorporating into films specific oxygen carriers (possibly cobalt chelate complexes).

An apparatus was assembled to investigate the selective permeability of organic films. Of the different commercial materials studied, natural rubber is outstanding for its high permeability and its selectivity; it is, however, subject to oxidation. Ethyl cellulose and polyethylene are promising. Additional work with ethyl cellulose films done in the Princeton laboratories and with pure gases showed that these films had an $O_2:N_2$ permeability ratio of about 3. Argon is enriched to almost the same proportions as oxygen, while carbon dioxide becomes enriched to a much greater extent. For any commercial application, it will probably prove desirable to remove carbon dioxide before or just after the first permeation stage. The removal of argon will be impractical, and it will be enriched along with the oxygen.

For appropriate gas mixtures, the power requirements of a permeation process are of the same order of magnitude as those for low-temperature separations. Because of low film permeabilities, relatively large plants will generally be required. The total film area (that is, plant size) needed, however, is approximately inversely proportional to the pressure on the high-pressure side. Within limits, therefore, doubling the pressure decreases the plant size roughly by a factor of 2; this is accomplished, of course, by simultaneously increasing the compression power requirements. Determination of the economically optimum pressure would depend on a detailed balancing of the cost of large-scale film packs and the cost of compression.

On the basis of work done thus far, it appears that the production of pure oxygen by a permeation process does not seem to be competitive with the Linde process because of high power requirements and very large plant size. The production of slightly enriched air by a single-stage permeation process is much closer to having practical interest. Helium separation from natural gas appears very promising from the viewpoint of power consumption; the relatively small total production of helium means that the plant need not be inordinately large. The enrichment of hydrogen-containing gases is also interesting because of the large separation factors which can be obtained with polystyrene films.

Any large-scale application of permeation processes for gas separation must be based on the development of cheap film cases. The films must, of course, be supported in order to withstand super-atmospheric pressure, and the engineering problems associated with an economical solution of this problem have not yet been worked out.



Figure 50. - Foreign documents section - print files.



Figure 51. - Foreign documents section - card index.

All of the work carried out thus far has been with commercially available packaging films. In view of the fact that there seems to be no correlation between the chemical nature of a film and its separative properties, it is almost certain that the optimum film has not been found for any given gas separation. Major improvements may thus be found in film selectivities and permeabilities which would vitally affect the feasibility of certain gas separations.

Technical Reports and Foreign Documents

Synthetic Liquid Fuels Abstracts

The current literature has been thoroughly covered and abstracted; German patent references in the foreign document files have been searched and those of interest abstracted. About 1,000 copies of each issue of the Abstracts have been distributed. Indexes of all abstracts issued since October 1944 have been made, thus opening up for easy inspection all of the synthetic fuels literature since that date.

Bibliographies

The review and compilation of the literature on the pressure hydrogenation of liquid and solid carbonaceous materials have been completed. This bibliography covers the literature to January 1, 1949, and the patents to May 1, 1949, and contains more than 6,000 abstracts. Comprehensive subject and patent number indexes are nearing completion.

The Fischer-Tropsch bibliography is still in the formative stage; the card index is being completed, and it is anticipated that this bibliography will contain about 5,000 abstracts.

A bibliography of Bureau of Mines investigations on the production of synthetic liquid fuels has also been completed.

Foreign Documents

Work has been continued in collecting, arranging, classifying, and indexing the foreign documents from German sources containing information of interest on synthetic liquid fuels (see figs. 50 and 51). Individual subject indexes of 279 TOM reels are being consolidated into a single index. Enlargement prints selected from the TOM reels and printed CIOS, BIOS, and FIAT reports are available as well as translations of documents from various sources. Documents other than TOM reels are being indexed in considerable detail, and an attempt will be made to correlate this index with the TOM-reel index. The document index now covers more than 1,500 separate documents. It is anticipated that, upon completion, this index will contain a reference to every document or report from German sources containing specific information of interest in connection with synthetic liquid fuels.