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CENTRAL INTELLIGENCE AGENCY

INFORMATION REPORT

REPORT

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East Germany

DATE DISTR.

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SUBJECT

VEB Synthesewerk Schwarzheide Research
and Development:
Refining of Olefines

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1. Among the 1954 research and development projects carried out by the Research and Development Department of VEB Synthesewerk Schwarzheide was a project on "Experiments for the Processing of Olefines from the CO_2H_2 Synthesis". The short title of the project was "Refining of Primary Products". The project was carried out under the supervision of Dr. Will (fnu) and Eng. Chem. Menzel (fnu).

2. The following indications on the project were given in the 1954 research and development report of the Schwarzheide enterprise:

- a. The project aimed at the further processing of olefines contained in the liquid gases of the synthesis.

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- b. Alcohols from olefines.

- (1) Isopropyl alcohol. Technical propane contains about 12.5% propylene. The propylene was absorbed with the aid of sulfuric acid and the isopropyl sulfuric acid was saponified to alcohol through hydrolysis. The experiments were carried out in a glass tube filled with sulfuric acid. Technical propane was introduced from below through a frit. The gas charge, the gas distribution, the sulfuric acid concentration, and the temperature were varied. The most favorable results were obtained with 85% sulfuric acid, at room temperature, and the finest distribution of the gas. After hydrolysis and fine fractionization, 160 grams of $\text{C}_3\text{H}_7\text{OH}/\text{H}_2\text{O}$ -azeotrop and 41 grams of polymerizate were obtained from 1 kilogram of technical propane.
- (2) Secondary butyl alcohol. The experiments were first carried out in the same way as with technical propane. The technical butane contained about 36% butylene. The most favorable sulfuric acid concentration was 77%; thus, the polymerization of the butylene was at a minimum. For purposes of better olefine exploitation, the experiments were carried out in stages. An installation for

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the periodic separation of the polymerizates brought good results. The butyl sulfuric acid was processed by hydrolysis and distillation. Dehydration of the azeotrop was carried out by way of azeotropic distillation with caustic sodium and with hexane. The yield was 152 grams of secondary butyl alcohol and 81 grams polymerizate per kilogram of technical butane. Better results were obtained through reaction in the liquid phase under pressure. Butane and sulfuric acid were intensely mixed in an agitator at room temperature and 2.5 atu. The reaction product was processed as above. The yield was increased to 220 grams of secondary butyl alcohol and 113 grams of polymerizate per kilogram of technical butane. In subsequent experiments, the alcohol is to be processed to butanon. The polymerizate can be separated into benzine and Diesel oil. The benzine has the O-number 82; the C-number of the Diesel oil is less than 20. The solidification point of minus 70, however, is remarkable.

c. Preparation of butyl acetate

Secondary butyl acetate was directly prepared from butylene and acetic acid in the presence of sulfuric acid. Normal pressure experiments were unsatisfactory. Good yields were obtained under pressure. The best results were obtained with 83% acetic acid. The most favorable relation of acetic acid to sulfuric acid was 3 to 1. Two phases were obtained. The crude acetic acid was processed by washing and water-steam distillation. The distillate contained 20% secondary butyl alcohol. From there 96% secondary butyl acetate was obtained through redistillation.

d. Addition (Anlagerung) of H_2S to Olefines.

Gaseous benzine containing olefines was mixed with H_2S under normal pressure and led over the following contacts.

- (1) Charcoal - H_3PO_4 contact (23% P_2O_5)
- (2) Al_2O_3 - Fe_2O_3 contact (9 to 1 parts).

The reaction was weak. The reaction products contained only 0.3 to 1.4% sulfur. These experiments were discontinued. Experiments under pressure could not be carried out due to material difficulties (lack of suitable corrosion-proof pressure installation).

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