Item 78 - Determination of Acetylene in Synthesis Gas.

Sulfur was detectable in the copper-acetylene precipitate from acetylene analyses. It was shown that organic sulfur might combine with the Illovsay reagent. It was found, for example, that carbon disulfide of known concentration gave a copper precipitate while thiophene did not. By calculation of the sulfur contained in the copper precipitate it was possible to find from the total C₂H₂ and CS₂ a figure which indicates the difference between the amount of acetylene in the gas and other similar unsaturated compounds. No analytical technique was described.

Items 212-247 (41 pages)

Deals with experimental unit of the butane-tanol type to be built at Heydebreck, Upper Silesia. The process received a mixture of propane and butanes. Normal butane was isomerized, the isobutane was dehydrogenated to isobutylene which was polymerized to di-isobutylene and then hydrogenated to isoctane (FT 110). The dehydrogenation step was accomplished by chlorine, and the byproduct HCl was reconverted to chlorine by oxygen. Pages 66-72 (back of sheets) are summary specifications including principal process equipment, estimate of construction costs, labor and material requirements, operating utilities, chemical and catalyst protection.

The bulk of the remainder of this section is correspondence on policy matters, financing and general discussion of a non-technical nature. The translation of these items 212-247 appears unnecessary. Process details are not given, and the material is considered of historical interest only, or might be reviewed if details are later uncovered on the catalytic steps. It is revealed that I.G. hoped to develop a working process for the production of 2,2,3 trimethyl butane.

Item (not numbered) - Production of Butadiene from n-butane - January 5, 1943.

Technical details of the production of butadiene from N-butane by dehydrogenation to butene, reaction with HCl to form monochlorobutane, chlorination to dichlorobutane and decomposition to HCl and butadiene. Gas compositions, yields, heats of reaction, contact times and cost estimates for a plant producing 2700 kg butadiene per hour are given.

Production of butadiene from n-butane - December 4, 1942.

Preliminary design calculations for a 20,000 ton/year butadiene plant. Detailed data are given for material balance of plant, temperature and pressure conditions, utilities, raw materials and cost in two cases. Case 1: Using n-butane with 5% butenes. Case 2: Using n-butane with 40% butenes. A detailed flow diagram of the proposed plant is given.

Other reports and letters

Five flow sheets, showing little detail, am given for the production of butadiene and synthetic rubber from alcohol via acetaldehyde.

Section 6 - CHD

Activated Clay catalyst plant Hoydebreck 669 - December 12, 1942.

Detailed technical plans for the large scale production of three types of catalysts.

- 1. DHD catalyst, which is 5-10% molybdic acid on cubes of activated clay.
- Gas-phase hydrogenation catalyst, made in pellet form, consisting of 75% activated clay and 25% tungsten and nickel sulfide.
- 3. Dehydrogenation catalyst for the production of isobutene and n-butene which is made in form of spheres, consisting of 90% Al₂O₃, 8% Cr₂O₃ and 2% K₂O.

The machines necessary for producing catalysts in the form of cubes, pellets and spheres are described. A detailed description is made of the technical production of activated clay. A table is given showing in summary form 16 steps in the manufacturing process of activated clay. Another table shows the various steps in the technical production of 3 types of catalysts made in the form of apheres.

Activated Clay catalvat plant Heydebreck - May 8, 1943.

Questions of technical organization discussed. No interest.

Section 7 - Oxo Process

Preparation of Oxo-alcohols (Keto- and Aldo-alcohols) from a synthetic olefin - December 30, 1941.

A mixture of olefins prepared catalytically from carbon-monoxide and hydrogen was distilled into 13 fractions for the purpose of analysis. Analytical data for each fraction are given. The olefin mixture was then oxidized to Keto- and Aldo-alcohols. After fractionation of this mixture it was found that the hydrocarbons present were concentrated in the lighter fractions. The separation of the alcohols from hydrocarbons in each fraction was accomplished by esterification with boric acid and fractional distillation of the hydrocarbons from the high-boiling esters. The analytical data show that the mixture contained branched alcohols from C, to C17. Normal alcohols are present in only small amounts.

Section 8 - Synthetic Lube 011

Propane dewaxing - March 5, 1942

The underlying reasons for discrepancies in the filtration rate in the propose dewaxing of various heavy lube oils in a large scale plant and in a pilot plant were investigated. Although it was at first believed that a non-crystalline precipitate was responsible for the low filtration rates in large-scale rotary filters, it was finally established by a series of tests that this is not the case. Further tests established reasonably good evidence that the low filtration rates were caused by unintentionally rapid cooling in one section of the plant. Medium lube oils refined with phenol were found difficult to dewax.

Manufacture of synthetic lube oils' - February 16, 1944.

By dehydrogenation of ethane to ethylene and sufficient purification of the ethylene synthetic oils could be produced which were satisfactory in most respects (viscosity index 92-115 according to degree of gas purification). The best results were obtained when the small amounts of CO contained in the ethylene were removed by washing with "nickel-caustic". Without this treatment oils of good quality could also be obtained especially in the presence of aluminum powder during polymerization. A table is given showing the properties of synthetic oils as a function of various methods of gas purification and catalyst composition.