ITEM III - ANALYTICAL METHODS

The material in this item has apparently been taken from the files of the Ludwigshafen Laboratory of the I. G. Farbenindustrie. It covers the period beginning about 1939. From the general nature of many of the included items, they apparently deal with the analytical control procedures of the many phases of operation of coal hydrogenation and hydro cracking plants, from the analyses of the feed to the evaluation of the product. There seems to be little original material in this file, most of it being taken from established sources. Interesting photographs of a portion of the routine microchemical section of this laboratory appears in Microchemie, Vol. 31, pages 196 and 198.

Sub-items 4, 14, 15, 20, much of item 34, 40 and 44 deal with the analysis of coal with which the reviewers are not familiar.

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ABSTRACTED AND TRANSLATED SUB-ITEMS

6. Total Sulfur in Gases (abstracted)

The gas is lead into a quartz tube through an orifice around which an atmosphere of oxygen is maintained. The combustion gases are led over a heated platinum star and passed into a neutral solution of H_2O_2 contained in a tower of glass beads so arranged that liquid is easily drained from the bottom. The sulfuric acid in the absorber solution is determined by precipitating as $BaSO_4$.

7. Total Sulfur in Oils (abstracted)

The sample contained in a separate chamber in a quartz tube is volatized into a stream of hydrogen which then is lead through an orifice around which an atmosphere of oxygen is maintained. The burning of the

sample vapors mixed with hydrogen requires careful manipulation. The combustion gases are lead over a heated platinum star and then through a horizontal tube filled with glass beads wetted with neutralized 3% hydrogen peroxide. The excess oxygen escapes from the end of the apparatus through a tube dipping into water in a flask. The sulfuric acid in the hydrogen peroxide and water solution is determined gravimetrically as barium sulfate.

8. Total Chlorine in Oils (abstract)

Combustion is made in the apparatus described in Sub-item 7 above except that a solution of 4% NaOH solution containing a few drops Na_2SO_3 replaces the H_2O_2 used for wetting the glass beads and the water in the flask. The chloride is determined by Volhard titration.

11. Acid and Saponification Number (abstract)

The sample is dissolved in a solution of 40% benzene, 60% alcohol containing alkali blue indicator and boiled under reflux for 1 hour. Titration of the cooled solution with 0.1 to 0.5 alcoholic KOH is made to determine the acid number. Excess alcoholic caustic is added, the solution boiled for an additional 1/2 hour. The solution is cooled and titrated with 0.1 to 0.5 N HCl to obtain the saponification number.

22. General Gas Analysis (abstract)

This covers the analysis of various gas streams from a hydrocracking plant. The analytical methods are based on the use of Hempel pipets, Bunte burets, and Podbielniak distillation with the earliest design apparatus. Gas analyses technique in the U.S.A. has progressed far beyond this point. Since the typical data given is dated 1938, it was not believed to be of any value to reproduce since more significant data should by now be available from other sources.

30. Orsat Analysis (abstract)

This describes the usual orsat analysis with one unusual feature. After removal of olefins and CO from the gas streams, butanes and propane are removed with the following reagent:

"Pipet for butane, propane: To prepare the absorption liquid, 10 g. finely powdered I_2O_5 are triturated with 125 g. of 2.5% fuming sulfuric (per 100 g., 46 g. 96% H_2SO_4, 54 g. 20% fuming H_2SO_4) in a mortar. Then the rest of the acid is added and the mixture well shaken for one hour. The prepared mixture should be placed in the pipet only shortly before the analysis and should be drained from the pipet immediately after completion of the analyses. Before each new use the mixture should be shaken anew."

The solution is contained in a Tramm pipet (not described). No directions are given in the procedure for the use of this solution but from the order of arrangement of solutions, it appears in the Orsat train, this reagent is used following the removal of CO.