

Attachment VI

Description of the Rheinpreussen Lubricating Oil Plant

(Lurgi Company - May 31, 1945)

Description of the Rheinpreussen Lubricating Oil Plant.

The flow diagram of the synthetic lubricating oil plant is given in Drawing No. AS 1263 (not included here).

The first step of the process is the chlorination of the crude distillate from the Fischer-Tropsch synthesis boiling between 200° and 350°C approximately. Operation is intermittent. During an interval of several hours, about one cubic meter of crude distillate is pumped to the chlorination tower, first brought to a temperature of 80°-100°C by passage through a heat exchanger, and then later kept at this temperature by cooling to remove the heat of reaction. The chlorine is taken from a supply tank (which stays at the saturation pressure of liquid chlorine, namely, 10 to 15 atmospheres), is expanded through a throttle valve, evaporated, and conducted to the lower part of the chlorination tower. From the upper end of this tower, HCl gas is evolved which is cooled in a cooling tower and then led from the plant to a gas holder. The cooling tower is cooled indirectly by means of benzene as cooling liquid, which is circulated and kept at low temperature in a cooler. The chlorinated intermediate is collected in an accumulator and from this conducted to the second step.

The second stage is the actual synthesis. The chlorinated crude distillate is mixed batchwise with benzene and naphthalene in agitators. The amounts taken are measured in measuring vessels before charging. The measuring vessel for the crude distillate has a capacity of 600 liters, the one for benzene and for naphthalene, 1400 liters. Each of the six agitators has a volume of 3 cubic meters. In starting the synthesis, the contents of the agitators are kept at about 120°C by means of a heating jacket, and aluminum chips or shavings are added. The reaction takes place at atmospheric pressure, and the HCl gas evolved is cooled in the same cooling tower in which the HCl gas from the chlorination was cooled, as mentioned above. The undesired tarry by-products formed in the synthesis are precipitated in four settling tanks, and reextracted with benzene in an agitator. The remaining tarry residue is withdrawn. The agitators for the mixing of tar and benzene have a capacity of 4 cubic meters.

The intermediate product formed in the synthesis is led from the settling tanks to two parallel-connected agitators where it is contacted with clay and lime at 150°C. The contacting with lime serves to neutralize the product. The used clay and lime are removed by means of a filter press.

The third step of the process is distillation, whereby the intermediate products formed in the synthesis are fractionated. First the admixed low-boiling materials, benzene and naphthalene, are distilled off in a distillation column. The distillation takes place at an absolute pressure of 20-300 mm. Hg. The distillation kettle is heated with steam of 100-150 atmospheres pressure. Water is heated in a boiler and circulated between this and the distillation kettle. The fractionating column operates continuously and is designed to handle 1800 kg. per hour maximum input and 1100 kg. per hour of distillate. The distillate from the fractionating column is separated into naphthalene and benzene in a low-temperature cooler connected in series to the outlet. This low-temperature cooler consists of two chiller apparatus which are cooled to about -20°C with liquid ammonia, and a centrifuge which throws down the naphthalene.

The bottoms from the fractionating column consists of oils which have been synthesized. These are separated into four fractions in a high-vacuum distillation apparatus, namely, Distillate I, Distillate II, Distillate III, and Distillate IV. Distillate IV is the desired lubricating oil whereas Distillates I, II, and III cover the lower-boiling range.

The last step of the process is the dewaxing and clay contacting of Distillates I, II, and III. Both these parts of the plant were not needed and therefore were not put in operation. It was intended to carry out the dewaxing with acetone as solvent. The mixture of the distillate and acetone is cooled to low temperature in the chiller apparatus and conducted to a drum filter of about 1.5 square meter surface, which can accommodate a maximum of 500 kg. of liquid (acetone and oil). The filtrate is withdrawn by means of a vacuum pump. The acetone is distilled off from the dewaxed oil. In order to avoid the danger of explosion, these apparatus are filled with inert gas. Finally, the dewaxed oil is contacted with clay in an agitator which has a capacity of about 5 cubic meters. The used clay is separated in a filter press

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