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PRODUCTION OF PHENOLS FROM L.T.C. TARS

Conference in Ludwigshafen, February 5, 1943.

High Pressure Experiments
Leuna, 558

Feb. 8, 1943

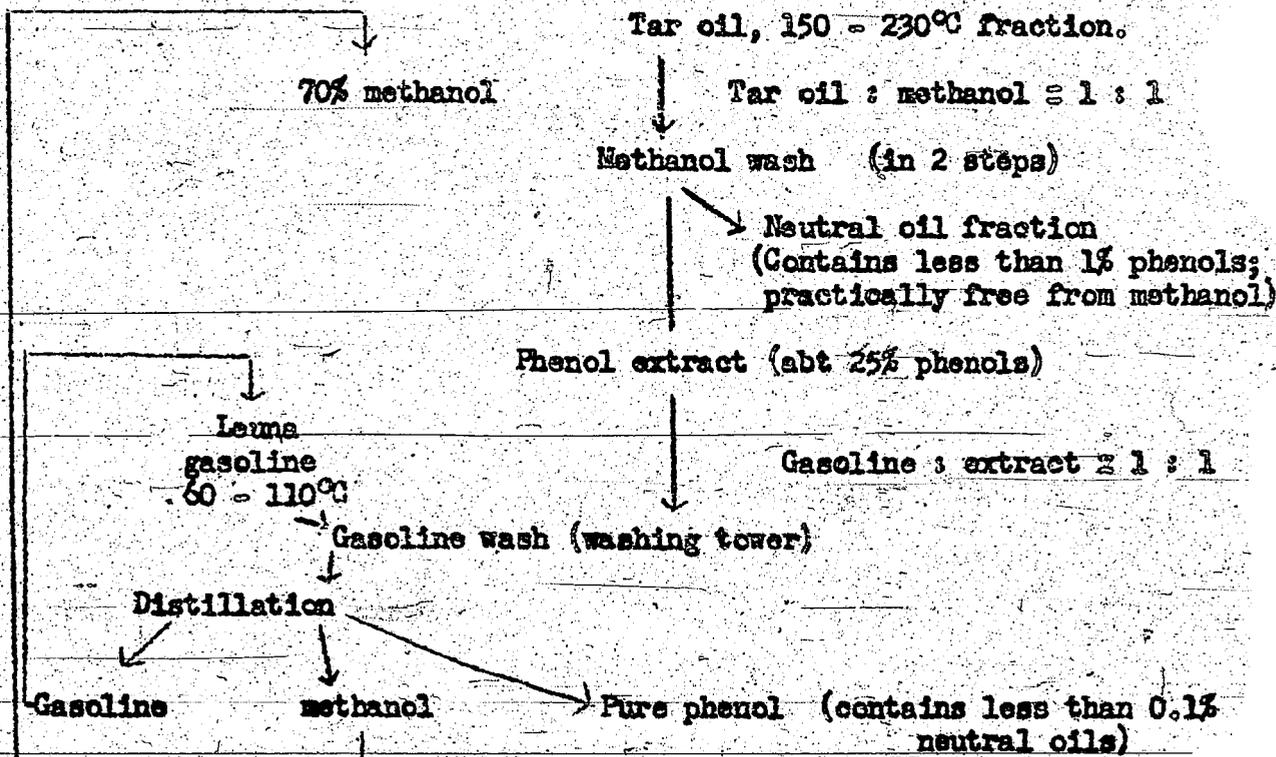
Conferees: Drs. Bayer, Dierichs, Teichmann, Pier, Bähr, Donath, Rank.

Leverkusen has developed a process of phenol recovery from the different low temperature tars.

The starting material for the recovery of phenols is the low boiling fraction of fuel oil (b.p. 150 - 230°C). The low temperature tar cut below 400°C converts the tar to a product which has a correct fire point by the removal of this fraction (11 - 12%), and after the removal of these light constituents meets the specifications for fuel oil. The proportion of phenols in this fraction is 50 - 60% and therefore very high, and this treatment offers much advantages from the economic standpoint. The economic advantages are the greater, the higher the phenols content. The cost price of the phenol oils from low temperature carbonization is 12 pfennig (compare with the coal hydrogenation middle oil with only about 26 - 28% phenol having a cost price of about 18 pfennig). The phenol oil from low temperature carbonization requires processing costs of about 6 - 7 pfg, and the finished phenols will cost about 20 pfg, which represents a favorable price situation.

No sodium hydroxide is used in the process, only distillation work is required.

Flow sheet of the process



10% of the oils enter the circuit. The process operates at atmospheric pressure and at normal temperatures. The process is similar to the alcoholic washing process of Risbeck. The advantages consist in the two step washing and the combination with gasoline (the Byk - Gulden process uses gasoline). The patent situation is not entirely clear. The Leuna gasoline used was a 110° C cut, because the 200° C cut does not separate from methanol. The process is less successful with the higher phenols. Up to now, only phenols b.p. -230°C are further treated chemically, but there is hope that the higher phenols will also find use at some later date.

In addition to phenols, a poor diesel oil is produced in the Leverkusen process with a cetane number of 25 with radiating * surface tars, and 36 when made with the circulation * gas tars, and can therefore only be considered as an admixture to good diesel oils.

Upper Silesia. Composition of tars, according to Dr. Dierichs:

- 1.8% phenol
- 4.8% cresols
- 12.5% xylenols

For comparison, in the coal liquefaction:

- 1.5% phenol
- 3.5% cresols
- 2.2% xylenols
- 7.2% higher phenols.

While discussing the general aspects of the low temperature carbonization program, the Leverkusen representatives mentioned the general tendency of the Government to increase the value of the product by chemical after-treatment and give a more secure economic foundation to the low temperature carbonization process. The radiation surface retorts are at present preferred because of the higher quality of the tar (occasionally in combination with hydrogenation). Leverkusen was interested in learning the Ludwigshafen opinion on the development of the low temperature carbonization, since the chemical development must be based on the best assured raw material. Dr. Pier and Dr. Bähr then expressed the opinion, that the deciding factor in carbonization is the quality of the coke, since it is the main product. The recovery of valuable chemicals from the tar cannot become a deciding factor for the task as a whole, in the selection of the process. A combination of low temperature carbonization and hydrogenation is correct, with a preference of that method of carbonization which produces the greatest amounts of tar at the lowest price. (circulation gas tars; briquetting). The flexibility of such a process insures a working up of the tar and the production of high quality fuel oils. The recovery of valuable products may be equally good, or even better and simpler (asphalt-free oils) from the hydrogenation products. A combination with the prehydrogenation of coal enables in addition the production of a L.T.C. coke suitable for blast furnace operations. These problems are of particular importance for Lotharingia and Upper Silesia. The production of motor fuels occupies but a secondary role.

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* The radiating surface low temperature carbonization tars are obtained in retorts heated indirectly by radiation; in circulation retorts the heating is done by returning the hot gases through the retorts. Dr. E. E. Donath

The radiation surface processes are considerably more expensive than the gas circulation processes, and the difference permits the use of hydrogenation, with the production of a combination process developed under consideration of all the factors, while the circulation gas process is less flexible with respect to raw materials, tar yield and quality.

In this connection Dr. Bähr presented his scheme for a combination of low temperature carbonization with hydrogenation (90% of coal in the L.T.C., 10% in hydrogenation).

The Leverkusen representatives then showed their interest in such hydrogenation products as the purest toluene, pure xylene (o., m., p.) carbazole and the 6-ring naphthenes. Carbazol finds application as a pest control, (nirosan), there is a great shortage in it. At present anthracene residues are used as raw material, with the anthracene worked up in the manufacture of carbon black. The yearly requirements in carbazol amount to 10,000 tonnes. (The cost of carbazol could reach 70 pfg). In coal hydrogenation, about 0.1% gasoline is produced in the distillation of L.T.C. oils.

The coal raw material for the DHD process contains in its whole boiling point range about 50 - 60% naphthenes, and in the 80°C fraction there still remain 40 - 50% naphthenes. Dr. Teichmann asks to have about 150 li. sent to him of the 70 - 100° and the 100 - 170° fractions of the Scholven gasoline. (The gasoline need not be cut into fractions.)

Dr. Dierichs has established that in the dephenolization of the off-water by the phenolsolvan process in Gelsenberg the results were poorer than in Politz and Leuna (Scholven water has not been investigated). The Gelsenberg water yields poor phenol (contaminated with 12 - 14% neutral oils). The trouble must be occasioned by some compound (abt. 0.5 g/li) which is water-soluble, is extracted with sodium hydroxide, boils in the boiling point range of phenols and possibly is polymerized during isolation (diolephine?). Identification of this compound will be attempted in Ludwigshafen and its formation in Gelsenberg accounted for.

There is 4.5 g phenol in the Gelsenberg off-water, more in Politz (6 g.). The gas producer water is added here. The economic advantages of the phenolsolvan process have a lower limit at about 4.5 g/li of off-water.

/s/ Rank.

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