

FILM STUDY GROUP

REPORT

T.O.M. REEL NO. 8

Prepared by

STANDARD OIL DEVELOPMENT COMPANY

Std. Oil Dev. Co.
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Comments on Reel #8

Bag 2732

Item 2, pages 3, 6 and 7

Analysis of sump-phase catalyst #1097 for hydrogenation.
Catalyst is composed of brown coal coke and iron.

Item 2, pages 8-12

Comparison of the sump-phase operation at Pöhlitz and Belheim.
This item covers operation conditions of the two types of operation
and comments on the results obtained.

Item 2, pages 13-16 and 24-28

Discussion of the causes of increased pressure drops in sump-
phase reactors and possible remedies for this situation.

Frames 19-20

Discuss the relative merits of catalysts #11002-10927 for conversion
of asphalts. #10927 according to Leuna was not as effective as #11002.

Item 2, Frames 35-36

Discuss some difficulties at Welheim when using the Iron catalyst
#10927 instead of the molybdenum containing catalyst #11002.

Item 2, Frames 39-41

Two tables are available showing the results obtained when hydro-
genating various coal tars.

Item 3, Part 1, pages 44-50 - A translation of this item by Shell Oil Co., Inc.
follows page 4 of these comments.

Patent application on process for avoiding the deposits and coking
in the hot separators following the high pressure hydrogenation sump-
phase ovens characterized by adding large amounts of gas through
distribution devices in the lower part of the separator, the gas serving
to agitate and cool the slurry and residuum.

Item 3, Part 2, page 51

Patent application on a method for the determination of the anti-
knock properties of fuels involving the subjection of the fuel in
liquid or gas phase to a stabilized electric discharge and using the
volume of gas developed as a measure of antiknock properties.

Item 3, Part 3, pages 52-55

Patent application on hydrogenation of low temperature pitch in which the hydrogenation is conducted over a fixed catalyst. The patent describes conditions of operation that satisfy the requirements of this process.

Item 3, Part 6, pages 62-63

Patent application on the use of preoxidized alkylated benzenes as a polymerization catalyst. An example is given of the use of this catalyst for the polymerization of styrene and the copolymerization of olefins and SO₂ to give poly sulfones.

Item 3, Part 7, pages 64-66

Patent application on the preparation of poly sulfones from olefins by the reaction of olefins with SO₂ in the presence of preoxidized alkylated benzenes.

Item 3, Part 7, pages 67-72

Report on experiments to produce poly sulfones according to the previous patent application.

Item 3, Part 10, pages 78-86

Patent application on the manufacture of ash-free electrode coke.

Item 3, Part 12, pages 94-99

Patent application on the preparation of poly cyclic hydrocarbons with more than five rings, especially "coronen" (polycyclic aromatic).

Item 3, Part 14, pages 105-109

Patent application on an automatic electric single or multi-stage regulator.

Item 3, Part 15, pages 110-112

Patent application on a process in which carbonization residues from the Pott-Broche extraction processes are briquetted and gasified.

Item 3, Part 16, pages 113-127

Patent application on a method for removing gaseous impurities from recycle gas of high pressure hydrogenation plants without special washings.

Item 3, Parts 20 and 25, pages 141-144 and 161-164

Patent application on gasoline inhibitors obtained by extraction of middle oil from pitch hydrogenation (boiling range 200-250°C.). The extract is neutralized with caustic and consists of nitrogen containing compounds.

Item 3, Part 24, Pages 156-160

Patent application on improving stability of gasolines by treatment with mild alkalis such as sodium carbonate, sodium phosphate, sodium acetate, sodium formate, and sodium phenolate.

Item 3, Part 26, pages 165-166

Patent application on the extraction of coal in the absence of hydrogen in one or more stages and subsequent treatment of the extract with air.

Item 3, Part 27, pages 167-177

Miscellaneous correspondence on Wehlm applications dealing with coal extraction.

Item 3, Part 28, pages 179-193

Patent application on the recovery of high octane gasoline from hydrocarbons containing oxygenated compounds. The application describes conditions for recovering such high octane number materials.

Item 3, Part 30, pages 203-205

Patent application on high pressure hydrogenation of coal, tars, and/or mineral oils. This application relates to prevention of build up of low boiling gasoline components in the hydrogenation recycle gas.

Item 3, Part 32, pages 217-222

Patent application on a device for preventing excessive pressure changes in measuring instruments.

Item 3, Part 35, pages 237-239 and 240-242

Patent application on high pressure hydrogenation of extracts and oil fractions obtained from coal. Material deposited in hot separator is continuously removed and worked up in the presence of coal tar.

Item 3, Part 37, pages 249-254

Patent applications on improving powdered fuels by treatment with nitric acid at high temperatures below the ignition point.

Item 3, Part 40, pages 268-278

Patent application on the removal of gaseous impurities from recycle gas in hydrogenation process.

Item 3, Part 41, pages 279-282

Patent application on the manufacture of hydrocarbons from solid coals; the high boiling oils from the hydrogenation of the extract are used as the solvent.

Item 3, Part 42, pages 283-286

Patent application on the separation of solids from liquids produced in coal extraction by means of filters.

Item 4, pages 293-355

Miscellaneous correspondence and memoranda on the Welheim 700 atmos. tar hydrogenation plant. This section gives a considerable amount of information on separation, costs, catalysts used, and other information.

Item 5, pages 357-440

Analyses of feed, coal, products and code letters for the various streams at the Welheim 700 atmos. hydrogenation plant and extraction plant.

Bag #2733

Item 2, Part 2, pages 500-505 - 504-509

Short memorandum discussing the use at Welheim of lecithin preparations as gasoline inhibitors.

Item 3, Part 1-3, pages 511-529

Description of the I.G. - Lurgi phenosolvan method of dephenolizing water which contains phenols. A flow sheet of this process is included. This section also contains results on tests and distribution of phenols in the water layers.

Item 4, pages 232-560

Report on cooperative tests to compare the performance of I.G. and CFR motor for octane numbers.

Item 6, Part 3, pages 593-596

A report which gives a summary of the effectiveness of phenolic inhibitors for gasoline. The best of these was d:ortho methyl phenol.

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New York 20, N. Y.
July 2, 1946

TECHNICAL OIL MISSION MICROFILM

REEL 8

BAG 2733 ITEM 3 SECTION 1

TRANSLATION

Description of the I.G. - Lurgi - Phenosolvan Process
for Removal of Phenols from Aqueous Effluents of Low
Temperature Carbonization, Hydrogenation, etc.

The Phenosolvan-Process was developed jointly by the I. G. Farbenindustrie and the Lurgi Company for Heat Technique. It consists of an extraction treatment of the phenolic-water effluent with a special solvent "Phenosolvan" and a separation of the absorbed phenols from the extracting medium by distillation.

Phenosolvan is an almost insoluble-in-water organic solvent. It consists of a mixture of esters of lower or secondary aliphatic alcohols. It boils between 110 - 130°C and has an exceptionally strong specific dissolving power for phenols. Whereas an application factor of 100 - 200% is necessary with other solvents, related to water, the use of Phenosolvan requires a treatment with only 10% solvent in order to attain dephenolation down to about 200 mg./l.

The extraction is accomplished in three countercurrent stages. The wash stage consists only of a system of pumps and separators. Emulsions between the solvent and phenolic water do not occur since the Phenosolvan possesses an emulsion destroying action similar to ordinary ether.

The release of phenols from Phenosolvan is accomplished by distillation. The greatest advantage of this solvent which exists to-date is that it boils lower than the phenols. A clear solvent is obtained on distillation and over a long period of operation in this plant no tarry impurities of the phenolic effluent have been able to concentrate in the solvent; the latter is characteristic of solvents which have a higher boiling point than the phenols.

The dephenolated waste water from the three stage extraction plant contains about 1% by volume of dissolved Phenosolvan, which is driven off and recovered by application of live steam in a column. The recovered Phenosolvan is returned for continued treatment of the water to constitute a cycle.

No particular value need be placed on the preliminary purification of the water to be treated. Oil and tarry impurities from low temperature carbonization effluents pass automatically into the crude phenol and ordinarily cause little trouble since they have a boiling point which lies above 230°. As a preliminary purification of the above effluent, a clean, mechanically operated separating vessel is generally satisfactory. The water may contain any amount of effluent from low temperature carbonization, ammonia and carbonic acids. It is desirable that some ammonia be bound with the carbonic acids. In certain waters from hydrogenation processes, large quantities of free ammonia are present and a gasification of the untreated water with gas containing carbonic acids is desirable. Water from low temperature carbonization usually contains carbonic acids in excess and require no such preliminary treatment. Organic or inorganic salts create no disturbance as long as they do not produce any strong alkaline reactions or lead to any corrosion.

The Phenosolvan Process is not only a dephenolation but is also a water purification process. The permanganate number of the purified water is less than that achieved by any other dephenolation process, such that any additional aftertreatment, as for example, the biological purification with special low costs, is quite practical.

The crude phenol production is 30 - 50% higher than the amount of phenols held in the water as determined by analysis. In spite of this, the crude phenol is in no manner of inferior quality, since it is almost free of neutral oil accumulation. It contains practically no fatty acids which are usually bound with ammonia in these waters and have not been extracted. The non-phenolic part is preponderantly of a higher boiling nature and remains in the residue of the distillation operation. This residue represents an additional substance of some value produced in the Phenosolvan Process and in many cases covers the cost of the solvent used in the process.

The Phenosolvan consumption is on the average less than 200 gr./cu.m. treated water. This includes the losses due to unavoidable leaks in the system.

The recovery of approximately 4 grams of phenols per liter plus the current prices of phenols covers the operating costs of the plant including amortization. A higher phenol content in the effluent will give the plant an additional profit. If, as is the case in low temperature carbonization plants, the middle oil fraction containing phenols is present, then the most valuable fractions of these phenols are recovered without any increase in the consumption of Phenosolvan. The middle oil is vigorously mixed with the untreated water and treated under fixed conditions such that the water is charged with the phenols from the middle oil. In this manner, the phenol yield of the plant as well as the overall economy is considerably increased.

CONSTRUCTION OF A PHENOSOLVAN PLANT ACCORDING TO DESIGN EP. 1007

The untreated waters and any other effluents pass to a preheater (105) and then are brought into contact with middle oil in agitators (101 and 103). This takes place countercurrently, the water flows into agitator (101) and the oil enters agitator (103). The water and oil which has already passed through agitator (103) is mixed further in agitator (101). The oil-water mixture flows continuously from agitator (101) to the separator (102) in which the oil and water separate from each other by settling. The oil is conveyed from here for its normally intended use, while the water is returned for mixing with the fresh incoming oil in agitator (103) and then passes to separator (104).

From the latter vessel, the oil passes to agitator (101); the water, however, is pumped through a cooler (1) to the gasification tower (2). The waste gases from the plant pass to this tower for recovery, by scrubbing, of the solvent vapors. Then the water is treated countercurrently with Phenosolvan in the multistage extraction unit (3). The dephenolated water still contains a certain amount of Phenosolvan and passes to an intermediate vessel (4) and from there through the heat exchanger (5) and the preheater (6) to the distillation column (11). In the lower section of the distillation column (10) heat is supplied by injection of live steam; the mixture of water and Phenosolvan from the condenser (9) flows through the gas refiner (8) in the separatory vessel (7) and they are separated according to their specific gravity. The upper layer of Phenosolvan is lead off to the Phenosolvan receivers (12-13); the lower water layer is returned to the intermediate vessel (4). The waste gases from the gas refiner (8) are scrubbed with untreated water in absorber (9).

The water discharged from the lower section (10) of the distillation column passes through heat exchanger (5) to the waste water sewer.

The phenol-enriched Phenosolvan from the multistage extraction unit (3) is resolved in distillation column (15). This is equipped with the distilling vessel, a condenser (16), a reflux condenser (17) and a gas separator (18). The Phenosolvan is recovered as distillate and passes to the receivers (12-13) and is returned from here to the extraction cycle. The crude phenol product is drawn off from the lower section of the column, cooled and collected in a storage vessel (20).

TRANSLATOR'S NOTE: In the report describing operations at the Wesseling Hydrogenation Plant near Cologne, Germany, the additional information elicited was that the Phenosolvan was chiefly isobutyl acetate in a mixture of esters that boil lower than the extracted phenols.