

FILM STUDY GROUP
SUBJECT INDEX AND REPORT
T.O.M. REEL NO. 84
Prepared by
SOCONY VACUUM OIL COMPANY

SOCONY-VACUUM LABORATORIES
(A Division of Socony-Vacuum Oil Co., Inc.)
Research and Development Laboratories
Paulsboro, N. J.

Review of Microfilm Reel #84
U. S. Government Technical Oil Mission

Compiled by P. D. Branton
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(Socony-Vacuum Oil Co.)
2-14-47

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Subject Index

Technical Oil Mission Microfilm Reel No. 84

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Part I Bag#3996 Target #30/3.01
DESCRIPTION OF INDIVIDUAL ITEMS (#251-255)

The yearly reports of the Rhenania-Ossag refineries are continued from T.O.M. Reel #83.

251 33p frames 6654-6686 1942

Annual Report of Reisholz Works for 1942.

252 43p frames 6687-6729 1943

Annual Report of Reisholz Works for 1943.

253 27p frames 6730-6756 1944

Quarterly Report of the Harburg Works for Jan.-
Apr., 1944.

This item gives tabular information on the operations and production of the Harburg Refinery.

254 17p frames 6757-6773 1943

Report of Grasbrook Works to June, 1943.

255 16p frames 6774-6789 1944

Report of Grasbrook Works to July, 1944.

Items 254 and 255 complete the data on the plant discussed in items 235-237 of T.O.M. Reel #83.

Kaiser-Wilhelm Institute for the Study of Coal,
Mühlheim, Ruhr

Descriptions of Individual Items (#1-3)

Part II Bag #2810 Target #30/6.01

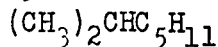
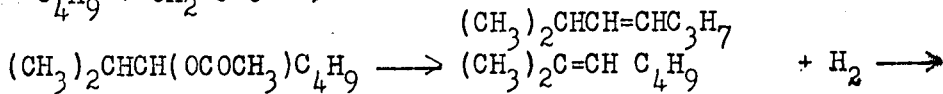
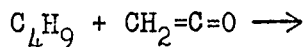
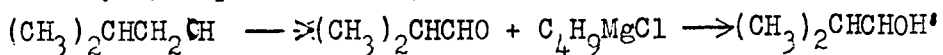
1 145p frames 3-161 7/25/41

Catalytic Aromatization of Paraffins and Olefins
Isolated from Gasoline Synthesized from Carbon
Monoxide and Hydrogen - by Dr. Coenen

The cyclization of aliphatic hydrocarbons had been previously achieved by the use of certain catalysts and reaction conditions. The aim of this work was to orient those conditions to achieve the greatest possible yields of aromatics with the least amount of cracking and secondary products.

The first part of this report describes the isolation and purification of the hydrocarbons to be tested. Normal heptane, 1-heptene, 2-heptene, normal octane, 1-octene, 2-octene, normal nonane, 1-nonene and 2-nonene were obtained from the 100-160°C fraction of 'Kogasin', the synthetic gasoline produced by the Fischer-Tropsch process. The olefins were separated in bulk from the paraffins through their addition complex with mercuric acetate. Individual compounds in each group were then separated by fractional distillation. Two types of fractionating devices are described; a normal, packed adiabatic column of 40 theoretical plates, and a rotating column of 90 theoretical plates, (HETP of 1.15 cm.).

The hydrocarbons 2-methylheptane, 2-methyl-2-heptene, and 2-methyl-3-heptene were synthesized as follows:



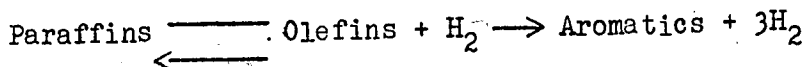
The other hydrocarbons used were available from outside sources. Physical constants are included.

The second part deals with the aromatization of these compounds. The experiments were carried out in a specially built continuous recycle apparatus coupled with a precise separatory column.

Of the catalysts investigated, the most outstanding proved to be a chromium oxide preparation containing about 0.05% of iron. Other catalysts tested included copper oxide, chromium oxide, chromium sulfide, nickel metal and chromium oxide, copper and nickel metals and chromium oxide, copper metal and chromium

oxide, copper metal and chromium sulfides.

Runs using n-heptane showed that an aluminum oxide-chromium oxide catalyst at 500°C causes appreciable cracking. Therefore, the reactions were run at lower temperatures. At 400°C paraffins were found to yield, in addition to the aromatics, approximately 10% of olefins having the double bond toward the middle of the molecule. An increase in contact time led to greater yields of aromatics without changing the olefin content. A very short contact time, however, resulted in a practically complete dehydrogenation of the paraffins to the corresponding olefins. Tests carried out on olefins showed that dehydrogenation to aromatics was accompanied by hydrogenation to paraffins. These results point to the following mechanism of aromatization of paraffins.



Since the second step is by far the slower, it is the decisive one, so far as the speed of the total reaction is concerned.

It was also found that alpha or beta olefins isomerized to centrally unsaturated compounds and that whenever the ring closure could occur on different points of the paraffin molecule, it took place preferentially on secondary carbon atoms.

The use of carbon dioxide as a vehicle for the cyclization of n-heptane at 400°C over a nickel metal chromium oxide catalyst led to the formation of carbon monoxide. The yield of aromatics was lower, and the deactivation of the catalyst quicker than in similar experiments carried out with nitrogen as vehicle. It was discovered, too, that reduction of pressure had a favorable influence on the aromatization of n-heptane over a copper metal-chromium oxide catalyst. At a pressure of 10 mm. the yield was four times that obtained at atmospheric pressure.

Comparison of experiments carried out on individual hydrocarbons under the same conditions showed that within the same molecular weight group, the tendency to aromatize increases in the following order:

n-paraffins --- iso-paraffins --- n-olefins --- iso-olefins---
cycloparaffins.

Within each homologous series, that tendency increases in direct proportion to molecular weight.

References and blueprints are enclosed.

Contribution to the Knowledge of the
Catalytic Isomerization of Olefins.
Dissertation by Dr. Richter

Previous work on the catalytic migration of the double bond in aliphatic olefins and on the rearrangement of the carbon chain is described. This report deals with the action of certain catalysts such as 'kogas contact', a copper-cobalt-thoria catalyst, activated carbon, aluminum oxide, acid cadmium phosphate, and copper, on individual C₄ to C₆ olefins under various conditions. The desired hydrocarbons were obtained by careful distillation of synthetic gasoline fractions rich in olefins. In the butene series, fractionation was sufficient to yield pure products; the higher homologs had to be purified through their mercuricacetate complexes. This method of separation leaves the steric configuration of β -olefins (cis-trans-isomerism) unaltered.

Copper-cobalt-thoria catalyst at 190°C. provokes a migration of the double-bond, practically without side reactions. The reaction equilibrium for n-butenes can be reached from either the 1-butene or the 2-butene and coincides with the calculated data. At higher temperatures the same catalyst causes cracking and polymerization, as well as hydrogenation of the olefins.

Activated carbon at 325° catalyzes a bond migration with an additional hydrogen disproportionation, leading to paraffins and to compounds lower in hydrogen content (benzene from n-hexenes). Cleavage is very slight. With paraffins, this catalyst does not cause any carbon-chain isomerization.

Alumina at 190° causes only a double-bond migration, but at 400°C isomerization of the carbon chain becomes noticeable and is also accompanied by cleavage and polymerization. This cracking, however, yields olefins predominantly, although methane and ethane are just barely traceable. Higher molecular weight compounds formed under these conditions consist chiefly of olefins containing one additional CH₂-group. Pressure favors polymerization at the expense of cracking and isomerization.

Acid cadmium phosphate catalyzes the branching of the olefin molecules at lower temperatures, but leads also to a more extensive polymerization. At high temperatures the polymerization decreases but the catalyst quickly loses its activity.

Bond migration in 1-hexene at 250°C is very slow in the presence of metallic copper, but the speed is increased by the addition of molecular hydrogen.

The results gained from the preceding investigation have been applied to the improvement of motor qualities of olefin-rich synthetic gasolines. Isomerization in the presence of alumina helped boost the octane rating from 52.9 to 59.2,

while the accompanying cracking yielded appreciable amounts of isobutene.

The speeds of reaction of various alpha and beta-olefins with peracetic acid and the analytical application of this reaction are discussed. It was also found that fractional precipitation with mercuric acetate yields first the addition compounds of the alpha-olefin. Since the solubility of the complexes depends less on the molecular weight of the olefin than on the position of the double bond, this precipitation can be used to determine the content of alpha-olefins in gasolines.

The behavior of various butenes in the conditions of the Fischer-Tropsch synthesis is then described. A double-bond migration can be detected as well as hydrogenation, but no isomerization of the carbon chain.

Certain predictions, which agree satisfactorily with experimental results, are made on the basis of theoretical considerations of these reactions and of the mechanism of repartition of valence electrons during the loosening of the carbon-hydrogen bond or the activation of a double bond.

3

7p

frames 366-372

no date

The Estimation of Alpha-Olefins in Synthetic Gasolines by Precipitation with Mercuric Salts
- by Drs. Koch and Richter

Treatment of an olefin mixture with mercuric acetate in glacial acetic acid as common solvent precipitates the alpha-olefin addition complex first. The comparison of the volumes of precipitates with the results obtained from standard test mixtures enables one to determine, approximately, the content of alpha-olefins in synthetic gasolines. The description of the analytical procedure and charts of results are enclosed.

Part III Bag #3499 Target #30/6.08
Lurgi, Frankfort on the Main

Description of Individual Items (#1-27)

1 lp frame 1 12/14/42

Comparison of Various Processes of Synthetic
Gas Manufacture of Coal

The following processes are compared:

- (1) Carbonization under pressure and cracking of the residual gas.
- (2) Carbonization under pressure and cracking of the crude gas (without regeneration).
- (3) Atmospheric carbonization with addition of carbon dioxide.
- (4) Carbonization under pressure with conversion.
- (5) Carbonization under pressure and cracking of the crude gas (with regeneration).
- (6) Carbonization under pressure and cracking of the residual gas.
- (7) Carbonization under pressure with addition of carbon dioxide.
- (8) Atmospheric carbonization with low temperature pre-carbonization.

Flow diagrams, operating data, and material balances are given and compared for all processes.

2 lp frame 2 5/15/44

Diagram of a Gas Recirculation Installation

Flowsheet is given which describes the neutralization and purification cycle in the synthesis of gas by the Lurgi process.

3 lp frame 3 -----

Synthesis of Hydrocarbons at Medium Pressures

A flow diagram is given of the Lurgi Recirculation Process for the synthesis of hydrocarbons from gases containing carbon monoxide and hydrogen and from conversion gases in the presence of a catalyst of the following composition: 100 parts of cobalt, 5 parts of ThO_2 , 8 parts of MgO , and 200 parts of kieselguhr. The reaction is run at 190-225°C and under 7-10 atmospheres pressure. A material balance table is included.

4 lp frame 4 -----

Synthesis of Hydrocarbons by the Lurgi Recirculation Process in the Presence of Iron Catalysts

A flow diagram is given which is similar to that in item 3. The catalyst contains 100 parts of iron, 25 parts of copper, 9 parts of alumina, 2 parts of K_2O , and 30 parts of silica. Operating conditions are 230°C and 20 atmospheres pressure.

5 lp frame 5 -----

Synthesis of Hydrocarbons by the Lurgi Recirculation Process with a Special Iron Catalyst for High Gasoline Yields

The installation is similar to that in items 3 and 4. The catalyst is a fused mass impregnated with 3% of copper. The temperature is 275°C, the pressure 20 atm.

6 lp frame 6 5/20/41

Diagram of the Experimental Hydrocarbon Synthesis Plant (Iron Catalyst)

Flow diagram almost illegible.

7 lp frame 7 2/5/41

Catalytic Reactor

Blueprint and construction details of a 500 mm. diameter, high-pressure catalyst chamber for the Lurgi process.

8 lp frame 8 5/3/41

Reduction and Oxidation Furnace with Cooling Zone

Diagram of furnace for the reduction of catalyst by hydrogen, using carbon dioxide for cooling.

9 lp frame 9 1/7/41

Catalytic Reactor

Blueprint of a 300 mm. diameter, high-pressure catalyst chamber for operation under 50-80 atm.

10 lp frame 10 6/27/41

Experimental Reactor

Diagram of a lamellar type vessel.

11 lp frame 11 1/14/39

Diagram of an Experimental High-Pressure Synthesis Plant of the Ruhrchemie A.G.

Flow diagram and details of unit.

12 lp frame 12 2/24/39

Experimental High-Pressure Reactor

Drawing of a 140 liter tubular vessel, operating under 100 atm.

13 lp frame 13 1/20/41

Flow Diagram of a Semi-Technical Experimental Installation

This item describes an installation for synthesis and purification of hydrocarbons by the Lurgi Recirculation Process.

14 lp frame 14 1/27/39

Experimental Vessel

Blueprint of a converter for synthesis under 70 atm. water pressure.

15 88p frames 15-102 2/14/40 -
9/28/42

Lurgi-Hoesch Negotiations on the Recirculation Process

This item includes a collection of letters, memos, meeting reports and cost analyses pertaining to the negotiations for the expansion of the Lurgi Recirculation Process installation at the Hoesch-Benzin Plant in Dortmund.

The process itself is described in a flow sheet. (frame 36) Water-gas or a similar gas mixture containing hydrogen and carbon monoxide in a ratio of 1.45-1.6:1.0 is used as raw material. Converter gas is also recycled. The installation plans include catalytic reactors, heat exchangers, coolers and condensers, gas recycling units, activated carbon absorbers - the so-called AK towers - neutralization and washing units and separators. An iron or cobalt base catalyst is used under atmospheric pressure and at 200°C. Neutralization is achieved efficiently with carbonate solutions. Liquid gasoline fractions, gas oil, diesel oil, and paraffin gatsch are recovered. The octane number of gasoline can be improved by treatment with bleaching earth.

The bulk of the item deals with material balances and quotations for installation, and economic considerations for possible modifications of the process. Corrosion resistant linings for coolers, heat exchangers, and settling tanks are discussed; in the absence of copper, the best material is "Remanit" containing about 18% of chromium.

and separation of high boiling paraffin gatsch, are sent to the neutralization tower where oil fractions are also recovered. The gasoline and Gasol are then separated by scrubbing the gases under pressure with oil, from which they are later recovered by distillation. The gasoline-free gas is recycled through a special compressor into the catalytic unit.

The principal advantages of this process are higher yields of products per m^3 of gas converted, and a greater rate of dilution of the synthesis gas in the reactors. The latter allows a good elimination of heat and prevents local overheating, excessive formation of methane, carbon deposition, and clogging of the catalyst. This in turn helps prolong the life of the catalyst. The gasoline formed is continuously eliminated from the reactor gases so that subsequent hydrogenation cannot occur. This increases the olefin content and thus the octane number of the gasoline.

The gross yeild is about 160g of liquid products per normal m^3 of gas and consists of about 65% of a 200°C endpoint gasoline of 52% olefin content and 63 octane number; 25% of oil boiling from 200-320°C, and 10% of paraffin gatsch. The activity of the catalyst remains practically unimpaired after 7 months of use.

A series of graphs and flow diagrams gives the material balance of the process and describes the functioning of the installation.

18

8p

frames 48-55 2/27/41

Installation for Purification of
Synthesis Gas and Gasoline Recovery

The synthesis gas, free of hydrogen sulfide, is purified under atmospheric pressure at 200°C by activated carbon. One kg. of carbon can purify up to 10,000 normal m^3 of gas by removing all aromatic hydrocarbons, tar forming components, and high-boiling organic sulfur compounds, particularly thiophene, to a degree of less than 0.1 g. of impurities per 100 m^3 of gas. The absorbed impurities can be removed by blowing with steam.

This process is claimed to be better than the one operating under pressure.

19

5p

frames 56-60

7/18/41

High Temperature Recirculation Process
with a Cobalt Catalyst

This item pertains to the functioning of catalytic reactors in the recirculation installation erected at the Schaffgotsch-Benzin Co. It is recommended, when working with a cobalt catalyst, not to exceed gas conversion of 80%. The critical temperature is 185°C., above which failure of the reactors and leakage of the paraffin can be expected. A series of material balances for ten-day runs at 186°C., and 10 atm. are given, showing an optimum yield of 120 gms. of liquid products per m³ of ideal gas.

20

54p

frames 61-114

9/12/39 -
2/12/40

Installation for the Production of
550,000 Nm³/day of Synthesis Gas
by the Lurgi Process

This item describes several modifications of the process for preparation of a carbon monoxide-hydrogen mixture by carbonization of lignite under pressure, followed by oxidative cracking of the crude gas. Cost and production estimates pertaining to the installation of this process in the Falkenau plant are also given.

Dry coal or lignite is submitted to carbonization under a pressure of 17-21 atm. in the presence of oxygen and superheated steam. After it is cooled and gasoline and tar fractions are removed, the crude gas is washed under pressure and purified to remove organic sulfur compounds. Carbon dioxide, partially removed in the pressure washing unit, is recycled to the gas generator and cracking unit. A synthesis gas containing carbon monoxide and hydrogen in ratios ranging from 0.5-1.5 can be obtained by proper regulation of reaction conditions. A certain amount of residual gas containing mostly carbon dioxide, nitrogen and methane is cracked and added to the synthesis gas.

Several flowsheets, most of which are illegible, describe the various steps of this synthesis and are accompanied by material data, production and cost estimates.

Note: A part of this correspondence, (61-70) refers to a project for a complete Lurgi hydrocarbon synthesis installation in Japan and is discussed in item #23.

24

4p

frames 129-132

8/28/39

Two-Stage Hydrocarbon Synthesis from
Water Gas with Iron and Cobalt Catalysts

Residual gas from the medium pressure hydrocarbon synthesis over a cobalt catalyst is converted over an iron catalyst (K314) at 254°C and 20 atm. pressure. Gasoline of 56% olefin content is obtained. The conversion of the original gas is increased from 71.2% - 94.6%. The catalyst is still good after 57 days of operation.

25

19p

frames 133-151

8/7/39 -
5/28/40

Application of Iron Catalyst in the
Synthesis of Hydrocarbons Under Pressure

The Lurgi Recirculation synthesis process applied to iron catalyst requires reaction conditions of 230-280°C and 20 atm. pressure as compared with the 170-220°C and 7-10 atm. sufficient for a cobalt catalyst. The CO to H₂ ratio is about 2.0 for iron catalysts and about 0.5 for cobalt catalyst. The latter ratio is difficult to obtain with the actual method of preparation of synthesis gas, and it is unfavorably affected by the dilution of the gas by the inert components introduced through recirculation of the residual gas. The severer conditions required for an iron catalyst also yield more methane and less paraffins.

The heat generated during the reaction is carried away by the vaporized cooling water. The construction of an adequate lamellar type catalytic reactor is described and a diagram, not enclosed, is referred to. In all probability it is of the type described in items 10, 11, and 13 of this same bag.

26

1p

frames 152

6/22/38

Diagram of an Installation for the High
Pressure Synthesis of Gasoline for Fushin,
Japan

The flowsheet is similar to that of the Rumoi-installation, (item 23), except that the tar from the gas synthesis and the oil from the main synthesis are also sent to the cracking unit instead of being hydrogenated.

27

1p

frames 153

2/26/40

Alterations in the Large-Scale Experimental
Unit for Iron Catalysts at the Falkenau
Plant of the Hoesch-Benzin Co.

The actual unit, operating under 10 atm. pressure,
must be rebuilt to allow work under 20 atm., as required for
the synthesis using an iron catalyst.

Part IV Bag #3979 Target #30/4.02

I. G. Farben Industrie, in Leuna

Description of Individual Items (#1-6)

1 27p frames 3-29 1/23/44

Automatic Regulation of the Liquid and
Gas Phase Hydrogenation Vessels

This report by Dr. Weis describes the self-regulating mechanism operating the valves, piping, etc., which control and adjust the flow of gases and liquids and the temperature of the various units of the high-pressure hydrogenation installation. The auto-regulation is achieved by the ingenious use of various pneumatic, electromagnetic and photoelectric devices and allows the entire installation to be run without supervision. A general flowsheet and several diagrams and photographs explain the functioning and the control of regulators for individual units, such as reactors, high and low temperature separators, settling tanks, cold strippers, etc.

2 2p frames 30-31 2/10/45

Damage to High Pressure Reactors

Brief report on the damage incurred by the high pressure apparatus in the Leuna factory on January 9, 1945 and on the possibilities of its repair. No mention is made of the cause of the damage which may have been an air raid.

3 7p Frames 32-38 4/4/44

Investigation on Hydrogen Penetration of Steels

Several samples of steels used in the building of high-pressure hydrogenation equipment were studied with respect to their resistance to hydrogen. Tests conducted for 1000 hours, at pressures ranging from 200-700 atm. and temperatures of 560°C show the influence of composition and of pretreatment of steels on that resistance. Chromium greatly increases the pressure threshold of the attack. The thermal treatment of steels (N_0 or N_{10}) also plays an important role. Most resistant are steels annealed at low or very high temperatures; least resistant are those treated at 700-750°C. Steels tempered at 1050°C., and

water-quenched are more resistant, while those overheated to 1150°C. or oil-quenched fail quicker. This phenomenon is explained by the different rate of diffusion of hydrogen through the granular and crystalline phase of the alloy and consequently, the different speed of attack of the carbon or carbide particles. The continuous hydrogenation of those compounds in the granular phase and the diffusion of carbon or carbide particles from the crystalline to the granular phase, bring about a disappearance of these compounds from the alloy and simultaneously the failure of the mechanical resistance. The speed of this break-down is thus conditioned by the crystalline structure which in turn is dependent on the heat treatment of the alloy.

4 lp Frame 39 12/9/44

Chromium in High Pressure Units

Use of alloys of the following composition is recommended for high pressure equipment.

For wall-thickness up to 200 mm.: 1.4-1.7% Cr, 0.1-0.15% V
" " " over 200 mm.: 1.7-2.0% Cr, 0.1-0.15% V

The thicknesses are those of rough-machined plates prior to heat treatment.

5 3p Frames 40-42 5/19/45

Building Materials for a 70,000 tons/year
Hydrogenation Plant

Gives a statistic and cost estimate for construction materials and masonry work, relative to the erection of a large-scale hydrogenation installation.

6 262p Frames 43-121 Varied dates

Specifications for High-Pressure Equipment

A list is given of standards for various metal parts, such as pipes, tubes, beams, flanges, screws, bolts, joints, also wiring diagrams, used in the building of equipment operating under a pressure of 325 atmospheres. Blueprints and wiring diagrams are included. This item is continued in Reel #85.