FILM STUDY GROUP

REPORT ON MICROFILM REEL NO. 15

Prepared by

UNIVERSAL OIL PRODUCTS COMPANY.

GERMAN AND HUNGARIAN PETROLEUM
FRENCH REFINERIES

Pages 1467-1476

Report on the Status of Several French Oil Refineries
and Their Possible Use for German Exploitation
August 8, 1940

A Houdry plant for 50,000 tons a year was under construction at Berre near Martiques, Rhone, as well as a Kellogg hydro-forming plant at Gonfreville.

Petit Couronne

An outline is given of the capacity and lay-out of crude oil distillation plants, lubricant oil refineries, cracking plants, reforming plants, gasoline refineries, and production of C_3-C_4.

A. A short description of new plants and projects follows, one of which was a dehydrogenation plant for 30,000 tons of aviation gasoline yearly of O.N. 76 under I.G. license.

B. An alkylation plant for 28,000 tons of alkylate per year, according to the Martine process. The raw material for this process is butane of 80% isobutane - in this case the O.N is about 93. If higher olefins are used (instead of C_4 olefins) the octane number is lower. One part isobutane plus one part isobutene plus two parts sulfuric acid (96%), reaction temperature -10°C, pressure 1 atm. A large distillation column is used to remove normal butane. The alkylate is supposed to be mixed with hydrogenated gasoline for aviation fuel of O.N. 73. There are also short notes on power plants and the number of employees.

Courchelettes

Again, a short description of lay-out and capacity of the following plants:

    I. Crude Oil Distillation Plants

        A. Under Atmospheric Pressure

        B. Vacuum

   II. Dewaxing

  III. Lube Oil Refineries

        A. Edeleanu Plant

B. Sulfuric Acid Plant with Agitators

C. Clay-treating Plant

IV, Cracking and Reforming Plants

V. Refining of Cracked Gasoline

VI. Butane Plant

VII. Short Notes on Employees.

Béthune

A short description is given of a hydrogenation pilot plant for the hydrogenation of coal paste for medium oil. The director of the plant, Mr. Valette, is also the inventor of this particular process, the particulars of which have been published extensively. The coal tar oil, 180 to 290°, or petroleum medium oil is used primarily as raw material. The reaction temperature for coal oils is 500 to 510°, for petroleum oil, 400°, using a molybdenum catalyst. The gasoline from tar oil has an O.N. of 79 (Res.) and 0.84 specific gravity; 60% of the gasoline is aromatics. Gasoline from petroleum oil has an O.N. of 66-67. The capacity of the plant is 40 tons of coal daily.

Lievin Hydrogenation Plant

The Audibert process is used which is similar to the I.G. procedure and also known from the literature. A short recapitulation of the high points of this procedure follows: After an investigation of aforementioned plants only the hydrogenation plant at Lievin was found worth expanding. The Germans preferred to construct additional production capacity in Germany proper, partly with machinery from French refineries.

Page 1477

Extract from the Report on the Petroleum Economy of Hungary
November 1940

Until 1938, Hungary was completely dependent on petroleum imports. The discovery of the Lispe oil fields in that year made it possible to cover the home consumption of gasoline in 1940. Even so, there was still a large deficit on most oil fractions, especially lube oil. The crude oil quotas of the different refineries in 1939 were:

Petrolipar	24.5%
Shell	20.0
Nyirbogdan⋁	20.5
V.O.C.	15.4
Fanto	11.4
Steaua	5.3
Szoregi	2.9
	100.0

Pages 1478-1480

Report on the Refineries
Located in Budapest and Surroundings

Shell

Modern plant in Csepel. Capacity, theoretical 180,000, actual 100,000 tons per year. Ship and rail connection, petroleum harbor Kikötö. Topping Foster-Wheeler, large tube still. For secondary distillation two two-step high vacuum tubes. Still with barometric condensation, same type as Astra Romana with Shell patents. Countercurrent refinery similar to Bergedorfer with Shell patents. Modern fat plant. The Foster-Wheeler plant can be run with fuel oil; all others are run with coal. The plant makes an excellent impression.

Fanto IX Soroksar-ut 95

Theoretical capacity 50,000, actual about 35,000 per year. The plant is extremely crowded and is situated directly on a very congested street.

For topping, the factory is equipped with a horizontal battery of four stills for half and total continual operation. The residue is distilled in a high vacuum tube still, system Steinschneider-Porges of the machine factory Brünn-Konigsfeld, gasoline rectification with a Hekmann column. The fat factory, system Osterloh is obsolete. Old paraffin sweat and crystallization plant with 6 atm. filter presses. This plant is not fit to work up the Lispe oil. It is supposed to be re-equipped with 25 atm. presses. The factory is heated by coal, has a pipeline to the Danube, and a rail connection. The plant appears to be run down.

Petrolipar IX Ken-ut 3

Theoretical capacity 65,000, actual not more than 30,000. The plant is completely obsolete.

Hazai, Gyömröi-ut 140

Theoretical capacity 150,000, actual about 135,000. Plant obsolete. Company in liquidation. Taken over cooperatively by Shell and V.O.C. and is supposed to be completely modernized to work up 20,000 tons yearly of Lispe oil residue. For political reasons, the main office of the Shell Company in London postponed its decision until after the war. The plant can only be heated with coal; it has rail connections.

Lardoline XIV Kerepsi-ut 152

This plant is a small still for 4,800 tons yearly.

Pages 1480-1482

Refineries Outside Budapest

Almas-Füzitö (V.O.C.)

Theoretical capacity, 180,000, actual about 150,000. The plant has ship and rail connections. The large Foster-Wheeler plant can be used for primary and secondary distillation. There is an old but productive paraffin plant with high pressure presses. Plant for special oils equipped with solvent treatment. The plant is modern but slightly crowded. Expansion is possible. The plant is too far away from the main center of consumption.

Petfürdö near Vespren

A state-owned factory. Equipment and capacity were kept secret.

Szoreg near Szegedin

Theoretical capacity 15,000, actual capacity not more than 8,000. Old two still plant for topping. No oil refineries. No paraffin plant.

Nyirbogdany

Theoretical capacity 23,000, actual about 10,000. Old 2-still plant for topping. Low vacuum for residues. Coal heating. Rail connection. Refinery is equipped for production of transformer and white oils. Because of the way of doing business in

Hungary, this refinery occupies a special position in the
Hungarian petroleum industry which exceeds by far its actual
importance.

Munkacs

Theoretical capacity 14,000, actual about 10,000.
Modern low vacuum tube still, Brünn-Konigsfelder.

Dees

Theoretical capacity 20,000. The plant is equipped for
topping of Rumanian crude and has two vertical and one horizontal
stills. The residue is used for fuel.

Maros Vasarhely

Theoretical capacity 22,000. Three and one-half still
horizontal topping battery. Modern Brünn-Konigsfelder, Stein-
schneider-Porges high vacuum still with a capacity of 14,000 tons
residue. The refinery can process seven to eight thousand tons.
The oil capacity exceeds by far the capacity of the topping plant.
The plant used to be heated by fuel oil, but it is supposed to be
changed to coal. The plant is suited to work up Rumanian crudes,
it is owned by the Alding Company in Bucharest. The Anglo-
Rumanian bank is a minority owner. The plant has rail connections.

Legeny-Mihaly near Csap

Small, obsolete plant, 3,600 capacity.

Csap

Capacity 1,200 tons.

Szatmar Nameti

Obsolete little refinery. Out of business for years.

Mezö Teleg near Nagyvarocd

The asphalt shale beds are supposed to contain about 20%
oil, and a primitive still is supposed to be erected.

Pages 1482-1494

A Discussion of the Hungarian Petroleum Economy

The consumption of gasoline in the last year of peace,
1939, was about 100,000 tons which was practically covered by

home production. However, the gasoline sold to the consumer was not pure gasoline but one of those typical European mixtures with 10 to 20% alcohol and ketones.

A history of Hungarian petroleum prospecting follows and the discovery of the important Lispe oil fields. By use of the cracking process the Lispe oil field petroleum could satisfy the Hungarian home consumption of gasoline but it was not enough to cover the needs for diesel oils and lubricating oils. The Hungarian yearly consumption of gas oil was about 35,000 tons which used to be produced from imported Rumanian synthetic crude. Due to strained political relations with Rumania this import from Rumania practically ceased in the latter part of the '30's.

Despite an admixture of 20% tar oil, a yearly deficit of 10,000 tons remained. Production of benzene in Hungary was only minimal, about 4,000 tons. Hungary used to import its needs of aromatics from Germany, Czechoslovakia, and Belgium. Propane and butane are not used. The Hungarian yearly need of paraffins is about 4,000 tons and is covered by the home petroleum production. There is even a slight excess which has been exported.

Hungary owns several asphalt beds which combined with the residue from the petroleum distillation, are sufficient to supply the home market. Hungary also owns several rich natural gas wells. The production of methane is estimated to be about seven million cu.m. The methane is compressed and used by the Hungarian railroads for fuel and lighting. The total consumption of lubrication oil used to be about 20,000 tons yearly in peacetime. It is distributed as follows:

Spindle Oil	3,150 tons
Machine and Motor Oil	7,000 "
Cylinder Oil	3,000 "
Vulcan Oil	2,500 "
Transformer Oil	600 "
White Oil	350 "

Pages 1495-1501

Report on the Visit to a Cracking Plant at Petfürdö
May 21, 1942

This plant belongs to the Peter Nitrogen Artificial Fertilizer Company in Budapest which is owned by the state. The little modern cracking plant of the Dubbs type was started to operate July 7, 1940.

1. Fractionating Tower

2. Furnace 30-10 atm.; 300°C entrance, 480-500°C exit

3. Reaction Chamber; 5 atm., 5-10°C lower

4. Flash Tower, can also be used as coke tower

5. See (1) above

6. Stabilization at 10 atm., 160°C

7. Absorption Column for gasoline recovery at 10 atm.

The plant uses about 90 to 100 tons crude oil daily. It is heated with fuel oil. The cracked gasoline together with straight-run gasoline is refined with 0.5% by weight 98% sulfuric acid in a simple orifice contact plant, according to known procedure. The separation is effected by the use of Laval Centrifuges. The redistillation plant for the cracked gasoline of Foster-Wheeler design consists of two towers.

1. Atmospheric

2. Vacuum

It is used with direct and indirect steam. The overhead from the stabilizer tower is used for bottled gas. This contains some gasoline and olefins. There was no knowledge of the exact composition of cracked gases and gasoline.

The following remarks on the other parts of the fertilizer plant have nothing to do with petroleum products

The ammonia synthesis produces about 42 tons daily and is supposed to be brought up to 60 tons. The state intends to erect a large new plant for this purpose near to newly found

natural gas wells in Transylvania. The alkali electrolysis produces about 2 tons of chlorine which is used for their own chlorine-containing products. The plant produces mustard gas but this particular part of the plant has been turned over to the Army. They also produce ethyl chloride, ethylene diamine, and di- and trichloroethylene. The laboratories were simple, adequate only for the most immediate analytical needs. In a little closed room high pressure equipment was found of several liters capacity for small experiments at 300 atm. This was the room Professor Varga used for his hydrogenation experiments. There is also an ethyl plant which produced all the tetraethyl lead used in Hungarian gasoline.

Pages 1502-1504

Report on the Artificial Fertilizer Plant "Pet"
of the Same Company

It is located 130 kilometers southwest of Budapest, 1 kilometer from the north end of Lake Platte, close to the highway Budapest Graz. Rail connection to the railroad Neszprem-Stuhlweissenburg near the plant Füzfö. It has its own steam power plant, two units of 6,000 H.P. each. Lignite briquettes are used for fuel. The hydrogen is produced from lignite (4,200 Kcal./kg. and 50% water) mined 10 to 15 kilometers away.

It is dried and gasified according to the Fleissner procedure. Degasification and vertical coke ovens according to Didier (procedure by Professor Szigeth), and partly gasified by addition of steam at 1,000 to 1,100°C. The coke is used for generator gas which heats the Didier oven. Composition is 82% $CO + H_2$, also some CH_4, CO_2, and N_2 remain. The gas is freed from H_2S and CO which is present up to 3%. CO_2 is removed by pressure washing in two towers. Final purification by cooling to a low temperature by means of a Linde unit, which is also used for the production of nitrogen.

Ammonia Factory

Run by the N.E.C. procedure under 300 atm. Two contact ovens, each 11 m. by 700 mm. Production is 28 tons ammonia daily. Additional chamber of two ovens under construction. Separation of ammonia by washing with water. Concentration is 16-18% ammonia by volume. Oxidation of ammonia is carried out in a Bamag plant using a rhodium-platinum catalyst. Four contact ovens - daily production of each is 3 tons. The nitrous gases are used partly for dilute HNO_3, partly for 95-98% HNO_3, partly for nitrate.

Concentrated HNO_3

Capacity: Two towers, Pauling Plant, concentration with H_2SO_4. A ferrosilicon column is used.

Nitrate Factory

The major amount of HNO_3 is used for ammonium nitrate production. Capacity: 40,000 tons yearly calcium ammonium nitrate with 17% N. The main amount of ammonium nitrate is worked up to a melt containing 2-3% water. This ammonium nitrate is mixed with powdered limestone, the mixture pressed into corrugated plates and the plates are chopped up.

Production of carbon disulfide by Zahn process with some improvements. Capacity: 6 retorts arranged in two series. Raw Materials: Lump sulfur from Reesk, charcoal. Temperature: 900°C.

Trichloroethylene

Capacity: 1 reaction tower. Raw Materials: Acetylene from bought carbide, chlorine from own electrolysis, decomposition with lime.

Alkali Electrolysis

Capacity: 46 de Nora cells (Milano), 2,000 tons yearly NaOH, 4-5 condensation apparatuses. Raw Materials: Imported rock salt.

Hydrogenation Plant

Raw Materials and Procedure: Lignite or lignite tar (Varga process). Daily Production: 10 tons.

Pages 1505-1519

Report on the Discussion of German
and Hungarian Representatives of State and Industry
Concerning Quality and Quantity of Hungarian Gasoline
Delivered to the German Aviation Gasoline
-Plant at Moosbierbaum in 1942

In this report are analytical data of Hungarian gasolines some of which follow:

Analysis of Shell-Produced
Hungarian Straight-Run Gasolines

Specific Gravity	.736-.753
O.N. (MM)	60.4-61.5
Sulfur Content	under .1%

ASTM Analyses

IBP, °C	36-47
10%	69-85
20	85-101
30	95-112
40	105-121
50	113-128
60	121-135
70	131-144
80	140-154
90	163-168
95	167-188
E.P.	177-194

Additional Analyses
% over at 100	19.5-35

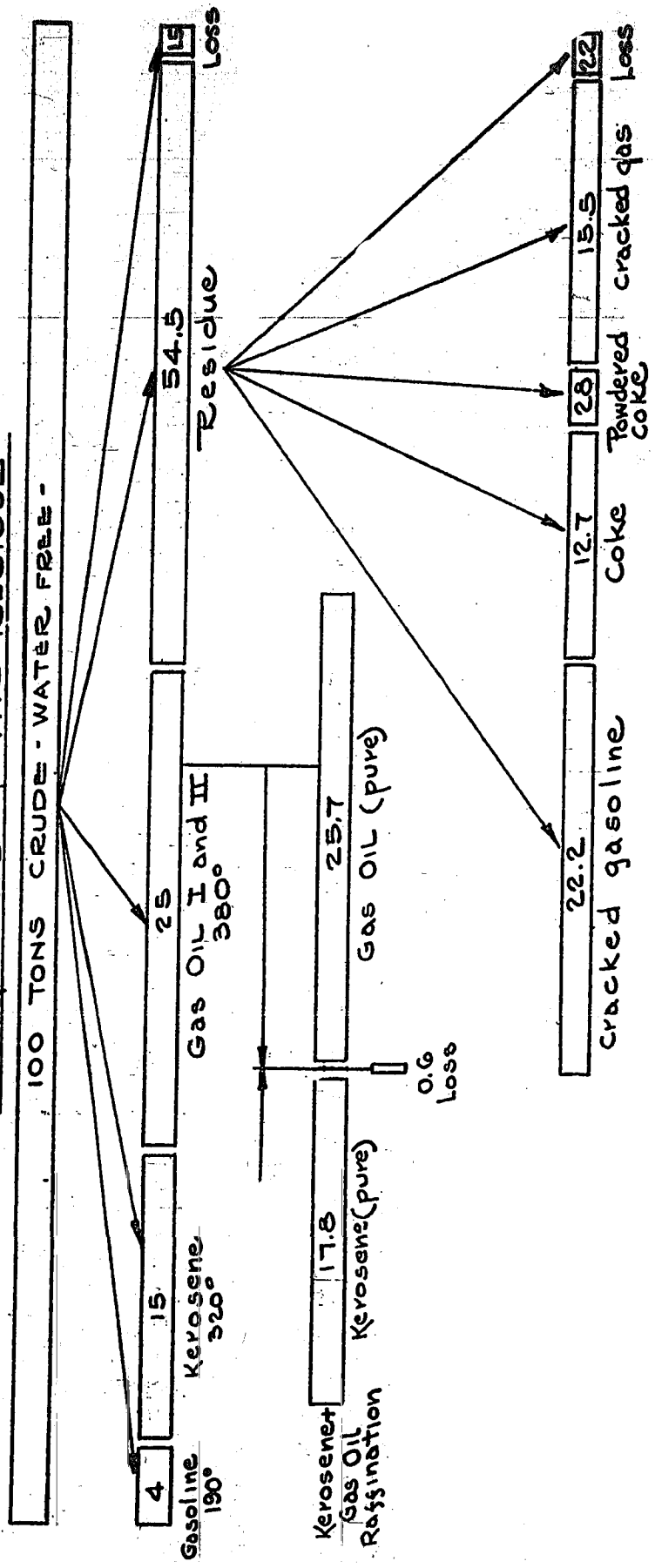Analyses by VOC

Specific Gravity	.785
O.N. (MM)	to 75° to 8%
	to 100° to 35%
E.P.	177

From pages 1520-1526 follow a number of diagrams on the working of Rumanian, Austrian, and Hungarian crudes for lube oil and paraffin. The chart which follows is an example of the processing of an Austrian crude

Fractionation of Austrian Crudes

Combined with

Cracking of the Residue

100 TONS CRUDE - WATER FREE -

4	15	25	54.5	1.5

Gasoline 190°

Kerosene 320°

Gas Oil I and II 380°

Residue

Loss

17.8

Kerosene (pure)

Kerosene + Gas Oil Raffination

25.7

Gas Oil (pure)

0.6 Loss

22.2

Cracked gasoline

12.7

Coke

2.8

Powdered Coke

15.5

cracked gas

2.2

Loss

Pages 1526-1528

Report on the Oil and Natural Gas Wells in the Hauskirchner-Muhlberg Field
October 29, 1942

Estimated yield of this field in a year from date about 100,000 tons. Disposal of the natural gas from the wells is discussed extensively.

Pages 1529-1530

Report on the Apollo Refinery
April 8, 1943

The Apollo Refinery is situated in Bratislava, erected 1895 and modernized 1936-37. The plant is equipped with crude oil distillation plant (MacKee process) and with a Dubbs cracking plant. The capacity in 1939 was 100,000 tons yearly. The I. G. took over the plant in 1939. The plant obtained in 1940-43 additional equipment to process Austrian crudes.

Crude Oil Distillation

1. Pipe still for 18,000 tons yearly of Rumanian oil; two columns, atmospheric pressure.

2. Pipe still for 120,000 tons yearly capacity for Austrian oil (1942).

Capacity of the old petroleum still 40,000 tons per year, of the Steinschneider plant 15,000 to 20,000 tons per year; filtrate oil from paraffin filter presses.

Dubbs Cracking Plant

48,000 tons yearly distilled, residue from Rumanian and Hungarian oil. The Rumanian residue is worked for coke and the Hungarian for fuel oil. When worked for coke the yields are 35-40% gasoline, 50-40% coke, 15-20% gas. When worked for fuel oil the yields are 35-40% crude gas oil, 30-35% fuel oil, 10-13% gas, and 15-20% gasoline.

Lube Oil and Paraffin Production

Old: For Rumanian production, filter presses for 20,000 tons yearly charge. Steinschneider plant for fractionation of the filtrate oil.

New: For Austrian production, a dichloroethane plant for 120,000 tons yearly charge.

In a year from now the charge is intended to be doubled, also the lube oil fraction will be extracted with phenol before the dichloroethane treatment, instead of sulfuric acid-clay treatment, for high quality aviation oils.

Approximate Production After Completion in Tons/Year

	From 80,000 Tons Rumanian and Hungarian Crudes	From 120,000 Tons of Austrian Crude
Motor Gasoline + Gasoline for Hydroforming Plant at Moosbierbaum	23,000	4,500
Kerosene	15,000	15,000
Gas Oil	15,000	31,000
Lube Oil	8,000	40,000
Paraffin	2,800	5,000
Coke & Fuel Oil	9,500	15,000

On page 1531 is a diagram of the fractionation of an Austrian crude. On page 1532 is a diagram on the Rumanian crude oil. On page 1533 begins a longhand report by a Dr. Willig on the first investigation of the Apollo Refinery in Bratislava, April 6, 1939.

On pages 1534-1539 follow the longhand reports by several other persons on the raw materials and plants present. The summary of this report is the typewritten copy on the Apollo Refinery just preceding this paragraph on pages 1529 and 1530. On pages 1541-1543 follow the diagrams on the working of Rumanian crudes of different origins for oil. On page 1545 follows several more details on the Apollo Refinery. On pages 1546-1557 is a discussion of the chemical composition and potentialities of Hungarian crudes. On pages 1558-1564 is the discussion of Austrian crudes documented with laboratory reports on their compositions.

SHALE OIL

Page 1566 reports on the yield of olefins from the distillation of shale oil in tunnel ovens. Pages 1567 to 1600 remark on the intended production of shale oil and its fractionation in Estonia complemented with charts and tables on its chemical and physical properties. The Estonian shale oil deposits are supposed to be about 5 billion tons and contain a billion tons of oil. The average yield from 100 tons of shale oil is as follows:

69 t. Heavy to Medium Oil, D =	1.040
19 t. Light Oil, D =	0.850
	IBP 70°
	5% 115°
	15% 144°
	25% 167°
	35% 180°
	70% 250°
	E.P. 360°
6.5 t. Gasoline I, D =	0.760
	80% 180°
	95% 250°
5.5 t. Gasoline II, D =	0.700
	IBP 27°
	80% 100°
	E.P. 150°

On page 1601 are given the results of analyses on shale oil from Württemberg. On page 1602 is a discussion of the possible use of Sapropelium found in Latvia. From pages 1604 to 1612 the discussion of Estonian shale oil is continued. The Germans consider the highly unsaturated shale distillates more suitable for production of alcohols and sulfonated alcohols than for their production of motor fuels. A lot of analytical data on shale oil distillates accompanies the discussion. From pages 1613 to 1615 is a discussion of some German shale oil distillates accompanied by more analytical data. On pages 1616 and 1617 are reported some preliminary experiments on Estonian shale oil on a small scale.

PATENT APPLICATIONS

Pages 2390-2392
Application by I. G. of May 5, 1942

"Procedure for the Evaporation of Foaming Solutions"

     An easily foaming solution, for example, a soap solution, is passed through a long and narrow tube which is heated to a temperature above the boiling point of the solution. The tube is arranged perpendicularly or obliquely. The solution enters the upper end; on the lower end the steam escapes whereas the molten soap flows in the receptacle below without any foaming. The speed of the solution passing through the heated tube has to be selected in such a way that there is no separation of solute from the solution in the tube. It has been found that this procedure also permits the removal of impurities from the solute by adding solvents to the solution, as for instance, hydrocarbons which exist at that temperature either in the vapor phase or having an appreciable vapor pressure.

     One diagram.

Pages 2393-2396
Patent Claim by I. G. of April 15, 1942

"Method for the Production of n-Butylene from n-Butylchloride"

     n-Butylene is produced from n-butylchloride by splitting out hydrogen chloride under pressure and high temperature in narrow tubes without catalyst. Example: n-butylchloride is passed through a tube 2 m. long and 13 mm. wide under a pressure of 10 atm. at an hourly rate of 500 g. The tube is of chrome silicon steel and is electrically heated to 550°. After leaving the tube, the gases are liquefied at -20°C. Only a small amount of cracked gases are produced. The fluid reaction product is fractionated in a column under 10 atm. pressure. The yield from 500 g. of n-butylchloride is 110 g. hydrogen chloride, 135 g. n-butylene, 230 g. butylchloride. and 20 g. of a higher boiling fraction.

Pages 2397-2404
Patent Claim by I. G. of June 23, 1943

"Method for Purifying the Reaction Products of Carbon Monoxide and Hydrogen"

     This claim relates to the purification of the dark residue, b.p. 400°C or higher, produced by the treatment of

water gas at 25 atm. and 150°C in the presence of sintered iron
catalyst. This residue is diluted with solvents which consist
mainly of mixtures of saturated aliphatic or cyclic hydrocarbons.
The solution of the residue can be treated with differenct kinds
of adsorbents; clay, Fuller's earth, Floridin, charcoal, and
aluminum hydroxide. It is advantageous to stir while treating
the residue solution with the adsorbents. After this paraffinic
residue has been clarified by treatment with different kinds of
adsorbents, it can be further decolorized by hydrogenation with
suitable catalyst; for example, copper chromite, copper barium
chromite, cobalt, nickel, molybdenum sulfide, tungsten sulfide,
zinc sulfide, iron sulfide and mixtures thereof and with
added activators as alkali and alkalisulfides. The materials
used for adsorption can be regenerated by treating them with
organic oxygen-containing or halogen-containing or cyclic hydro-
carbon solvents. The adsorbers are then reactivated by treatment
with acids.

Pages 2405-2413
Patent Claim by I. G. of July 29, 1942

"Method of Reacting Gases by Circulating the Catalyst from
 Reactor to Regenerator and Back"

        This patent claim is supposed to be an additional improve-
ment to the main patent 715066. The previous patent is changed by
using a regenerator above a reacting zone so that the flue gases
are more easily used to preheat the catalyst entering the regen-
eration zone. The previous patent employed a reaction zone above
the regeneration zone. Such a procedure does not add anything
new to our current knowledge in the field of moving bed catalytic
cracking. Somewhat better yields and lower carbon formation is
claimed by the changed operating procedure (see p. 17 for sketch).

Pages 2414-2418
Patent Claim by I. G. of September 22, 1942

"Method for the Improvement of Carbon Monoxide Reduction
 Products"

        During the reduction of carbon monoxide with hydrogen
at pressures of 12-15 atm. and at 180-230°C in the presence of an
iron catalyst, a large amount of oxygen-containing compounds are
obtained along with hydrocarbons. The value of the hydrocarbons
as a motor fuel is greatly reduced by the presence of these
oxygen-containing compounds because of their bad odor and their
tendency to gum formation. These oxygen-containing compounds
can be readily removed by washing with a 20-30% alkali or alkaline
earth bisulfite solution. This bisulfite washing also improves
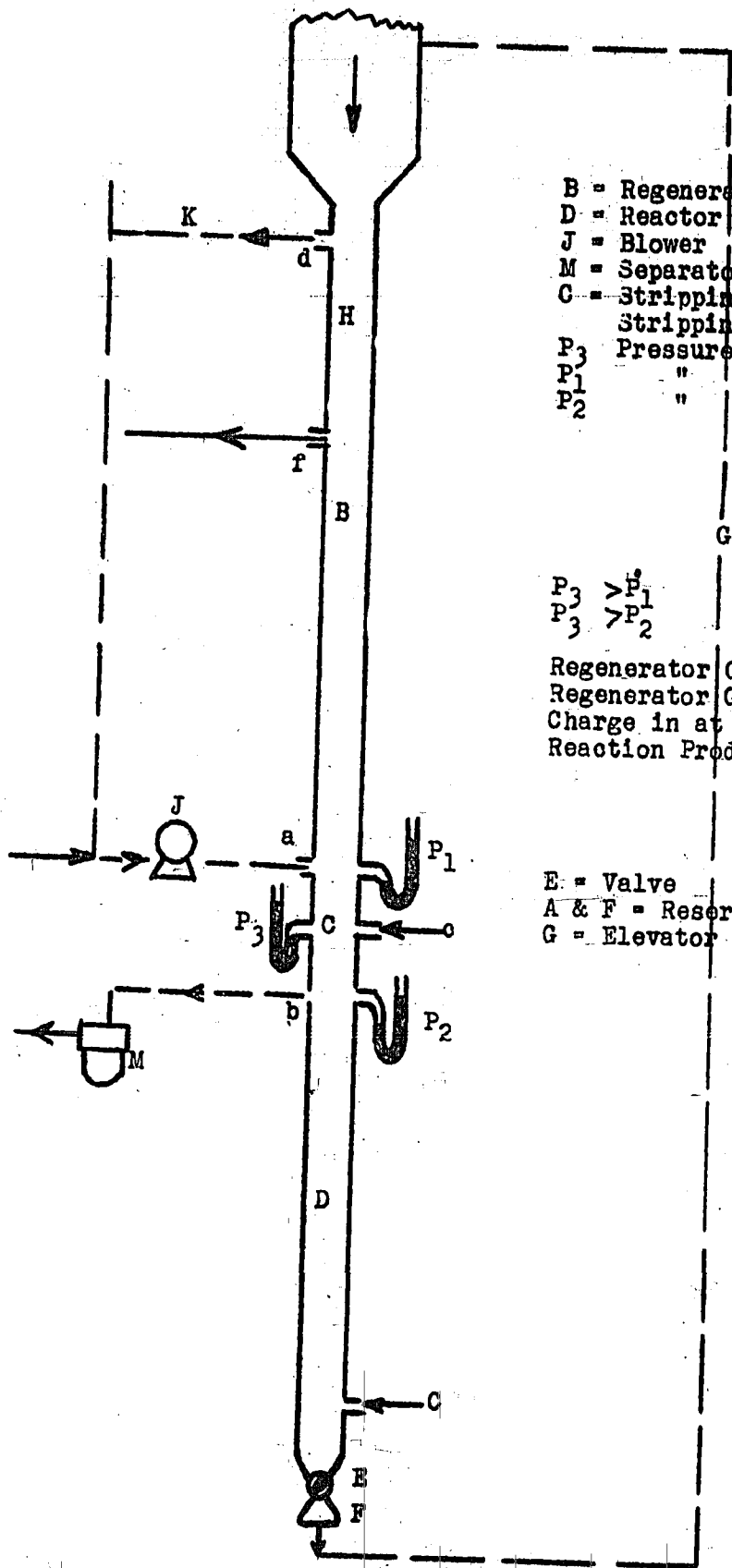
B = Regenerator
D = Reactor
J = Blower
M = Separator
C = Stripping Zone
    Stripping Gas in at c
P_3 Pressure at c
P_1 "   a
P_2 "   b

$P_3 > P_1$
$P_3 > P_2$

Regenerator Gas in at a
Regenerator Gas out at d & f
Charge in at c
Reaction Product out at b

E = Valve
A & F = Reservoir
G = Elevator

Method of Reacting Gases by Circulating the Catalyst from Reactor
to Regenerator and Back - July 29, 1942   Pages 2405-2413

the life of the catalyst used later for the isomerization of the hydrocarbons freed from the oxygen-containing compounds.

Pages 2419-2422
Patent Claim by I. G. of January 13, 1943

"Method for tne Production of Motor Fuels"

During the catalytic reduction of carbon monoxide with hydrogen, higher boiling alcohols are obtained which have a limited use. Together with crude gasolines from the distillation of coal, these alcohols can be transformed into motor fuel of O.N. 70 in the presence of hydrogen or hydrogen-containing or hydrogen-donating gases under pressure and with hydrogenating catalyst. The pressure is about 40-70 atm. The catalysts are mixtures of sulfides or oxides of the metals of the 6th and 8th groups of the Periodic System. They can also be mixed with the oxides of the metals of the 2nd to the 5th groups, for instance, one of the catalysts used consisted of 2 parts tungsten sulfide and 1 part nickel sulfide.

Pages 2423-2425
Patent Claim by I. G. of June 18, 1943

"Method for the Production of Low Boiling Aromatics"

Aromatic hydrocarbons boiling over 200°C and freed from oxygen and hydrogen compounds are treated together with hydrogen under pressures from 200-700 atm. and 390-440°C with a synthetic aluminum or magnesium silicate catalyst which is covered with 0.2-10% molybdenum oxide. 0.5% carbon bisulfide is added to the oil charge. The gasoline obtained by this process contains 43% boiling up to 170°. The higher boiling fraction is put back into the process. The fraction boiling up to 170° contains 40% boiling up to 100°C and contains 60% aromatics.

Pages 2426-2429
Patent Claim by I. G. of June 21, 1943

"Method for the Production of Diolefins"

1,3-Dioxanes are dehydrated in the vapor phase with catalysts which consist of mixtures of phosphoric acid with infusorial earth heated to 500°C.

Pages 2430-2432
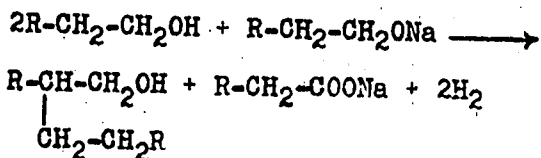Patent Claim by I. G. of September 29, 1943

"The Production of High Molecular Alcohols"

The number of carbon atoms in an alcohol can be doubled by heating them at 260° with sodium alcoholate in amounts less/ than 1 mole alcoholate to 2 moles alcohol and in the presence of a water-binding material, for example, calcium oxide or calcium carbide.
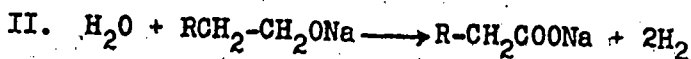
Pages 2433-2435

Some more examples for the doubling of the carbon chain of alcohols described in the preceding chapter are given. There is special emphasis placed on the need to reduce the amount of sodium used in the reaction; for instance, 864 parts of straight-chain C_9 alcohols are mixed with 3.8 parts sodium and 85 parts calcium oxide. The reaction mixture is heated while stirring for 48 hours at 240°. A final pressure of 2.4 atm. was reached.

According to Guerbet the following reactions take place:

$$2R\text{-}CH_2\text{-}CH_2OH + R\text{-}CH_2\text{-}CH_2ONa \longrightarrow$$

$$R\text{-}CH\text{-}CH_2OH + R\text{-}CH_2\text{-}COONa + 2H_2$$
$$\quad | $$
$$\quad CH_2\text{-}CH_2R$$

probably 2 steps

I. $2R\text{-}CH_2\text{-}CH_2OH \longrightarrow R\text{-}CH - CH_2OH + H_2O$
$$\qquad\qquad\qquad\qquad\qquad | $$
$$\qquad\qquad\qquad\qquad\quad CH_2\text{-}CH_2R$$

II. $H_2O + RCH_2\text{-}CH_2ONa \longrightarrow R\text{-}CH_2COONa + 2H_2$

The alcohols are α-substituted. They are C_n-alkanol-σC_n-alkyl.

Pages 2436-2440
Patent Claim by I. G. of November 2, 1943

"Method for Dehydrogenation of C_3-C_4"

C_3-C_4 mixtures are dehydrogenated to the corresponding olefin using temperatures from 450-600 in narrow iron tubes which contain metal oxides as catalyst. The catalysts are regenerated by heating with air. It has been found that it is not necessary

to burn off all the carbon deposited on the catalyst for regeneration, about 1/3 can be left.

Pages 2441-2443
Patent Claim by I. G. of June 17, 1944
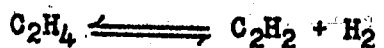
"Method for the Conversion of Hydrocarbons"

Gasolines of low octane number or high boiling gasolines fractions are converted into hydrocarbons of a high octane number by passing the starting material over a catalyst which becomes progressively smaller in size.

Pages 2445-2504

"Calculation of Organic Gas Equilibria on the Basis of Spectroscopic Data" by Hans Sachse - November 28, 1935

For an accurate calculation of chemical equilibria knowledge of the specific heat of the reaction partners and, of course, their change with the temperature is important. The specific heat of a molecule is composed of translation, rotation, and vibration energy. The translation and rotation energy is not dependent on the temperature; for each 1/2 R per degree of freedom. Only the vibration part of the internal energy is temperature dependent. The contribution of the vibration energy to the specific heat can be calculated on the basis of the Planck-Einstein theory, if one knows the number of vibrational degrees of freedom of the molecule and their frequencies. A molecule of n atoms has 3 n degrees of freedom of which 6 belong to translation and rotation energy (in the case of linear molecules, 5). There remain 3n-6, or for linear molecules, 3n-5 vibrational degrees of freedom. These correspond to the same number of normal frequencies which can be determined from the infrared and Raman spectra. However, not only the normal frequencies occur in these spectra but also the overtones. Because of this complexity the choice of the normal frequency is often ambiguous. Before further application of these optical data. their reliability has to be tested from time to time.

To each frequency is attributed an additive value and these values can be found as functions of temperature and frequency in Landolt-Börnstein. The concentration of the reactants is given by the equilibrium constant:

$$C_2H_4 \rightleftharpoons C_2H_2 + H_2$$

$$K = \frac{P_{C_2H_4}}{P_{C_2H_2} \cdot P_{H_2}}$$

$$P_{C_2H_4} = {}^oC_2H_4 \cdot P \qquad (P = \text{Total Pressure})$$

$$K = \frac{{}^oC_2H_4}{{}^oC_2H_2 \cdot {}^oH_2} \cdot \frac{1}{P}$$

The mole fractions of the reaction partners can be expressed:

$${}^oC_2H_2 = {}^oH_2; \quad {}^oC_2H_4 = 1 - {}^oH_2$$

1.) $$K = \frac{1 - {}^oH_2}{c^2_{H_2}} \cdot \frac{1}{P}$$

To obtain the equilibrium constant Equation 1 has to be solved for c. Since it would be a cumbersome procedure in the case of more complicated equilibria, it is much simpler to solve the equation graphically. To get a greater range log K is plotted against c. The higher the change in the number of molecules in the reaction, the greater the concentration change with the total pressure. All gas equilibria are written in such a way as to indicate disintegration, therefore, all reactions are endothermal and the equilibria constant changes with the temperature to the right side. Since it is desireable to gain an insight into these equilibria from one graph, the change of free energy is also plotted.

2.) $$\Delta F = -RT \ln K$$

In particular, the free energy of formation for 1 carbon atom is plotted; for instance, in the case of diacetylene 1/4 of the actual free energy is given, in the case of benzene, 1/6. From the free energies of formation log K is obtained after dividing by 2.3 RT but all of the data of thermodynamics can not foretell how fast the equilibrium is reached; for this purpose kinetic

data have to be applied. A discussion of the work by Belchetz, Rideal, Kassel, and Storch follows which can be found in the American literature. The actual task of this paper is the calculation of the free energy of formation on the basis of spectroscopic data.

$$F = \int_0^T c_p dT - T \int_0^T \frac{c_p^o}{T} dT$$

The evaluation of this integral is simple if the specific heat is independent of the temperature as is the case with the translation and rotation part of the specific heat. Contracting the temperature constant part of this specific heat into the c_p^o it follows that

$$\int_0^T c_p^o dT = c_p^o \, T$$

$$\int_0^T \frac{c_p^o}{T} dT = c_p^o \ln T$$

These formulas need a correction since they are calculated as if the gas has a constant specific heat c_p^o down to the absolute zero. Actually the gas condenses far above the absolute zero, and the resulting fluid has a specific heat which is temperature dependent and decreases with falling temperature. This error can be compensated for, with a constant characteristic for each gas. In this way the free energy for translation and rotation is obtained.

5). $\quad F_{tr,rot} = c_p^o T - T c_p^o \ln T - T (2.3RC + c_p^o)$

The last parenthesis multiplied with T corresponds to afore-mentioned correction constant and is called the entropy constant and is connected with the chemical constant (c) of the gas.

Transforming 5

5') $\quad F = -T c_p^o \ln T - 2.3 \, RT.C$

The chemical constant of a gas can be calculated according to Schottky, Sackur, Tetrode and Stern for linear molecules

$$C = 36.815 + 1.5 \log M + \log J + \log \frac{k}{\sigma}$$

for non-linear molecules,

$$C = 56.265 + 1.5 \log M - 1.5 \log J + \log \frac{g}{\sigma}$$

J is the moment of inertia

σ is the symmetry number

For the vibration each single normal frequency contributes an additive amount to the free energy. These contributions are tabulated in Landolt-Börnstein. The free energies are given as functions of

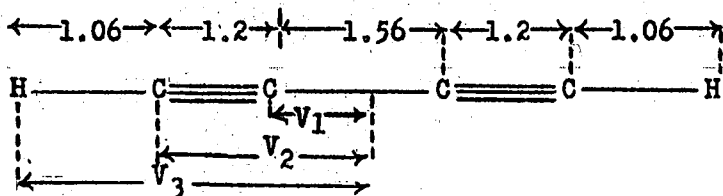$$\frac{\Theta}{T} \quad \text{where} \quad \Theta = \frac{hv}{k} = 1.435v \ cm^{-1}$$

In the case of well investigated spectra, the normal frequencies are given with great accuracy (CH_4, C_2H_2, C_2H_4). In other cases (diacetylene) the accuracy is still satisfactory for the calculation. In the case of more complicated molecules the exact evaluation of the normal frequency is not possible any more; one has to use analogies to similar molecules (butane, butylene, butadiene, vinylacetylene). Experimentally investigated according to this method were hydrogen, graphite, acetylene, diacetylene, vinylacetylene, butadiene, i-butane, i-butylene, ethylene, ethane, methane, and benzene.

The case of diacetylene will be given as an example. The diacetylene spectrum has been investigated in the infrared by Bartholomé and the Raman effect by Timm and Mecke. Very probably, the molecule is linear because the infrared spectrum would otherwise be much more complicated. The following 13 normal frequencies ($3 \times 6 - 5 = 13$) are given according to Timm and Mecke.

3350	Infrared	Single
2085	"	"
3330	Calc.	"
2183	Raman	"
644	"	"
730	Infrared	Double
231	Raman	"
720	Calc.	"
488	Raman	"

The greatest uncertainty is in the calculated frequency of 720 cm^{-1} which may be too low by 150 cm^{-1}. If the upper limit of 900 cm^{-1} is used for the calculation in the equilibrium with acetylene at 1500°C, the result would be 23% C_4H_2 instead of 21%. The moment of inertia of diacetylene has not been measured directly, therefore, it has to be calculated from the molecular form; a knowledge

of the atomic distances is necessary for that. They also have
not been measured, but the atomic distances of acetylene and
ethane can be used with an approximate certainty.

$$\overset{\longleftarrow 1.06 \longrightarrow}{H} \quad \overset{\longleftarrow 1.2 \longrightarrow}{C} \equiv \overset{\longleftarrow 1.56 \longrightarrow}{C} \quad \overset{\longleftarrow 1.2 \longrightarrow}{C} \equiv \overset{\longleftarrow 1.06 \longrightarrow}{C} \quad H$$

$$\longleftarrow V_1 \longrightarrow$$
$$\longleftarrow V_2 \longrightarrow$$
$$\longleftarrow V_3 \longrightarrow$$

For the moment of inertia:

$$J = \sum m V^2 = 2 m_H V_3{}^2 + 2 m_c V_2{}^2 + 2 m_c V_1{}^2 = 211 \cdot 10^{-40}$$

Symmetry Number = 2, statistical weight = 1.
Analogous to acetylene for the chemical constant
    C = 1.385
Entropy at room temperature
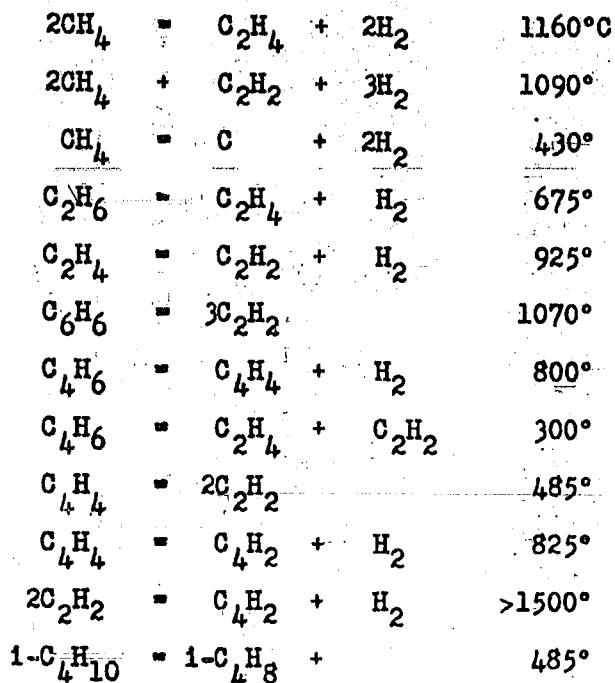   Entropy constant = C.4.56 = c_p^o =   13.28
    Vibration Part   6.62
    Translation and Rotation Part   39.6
                                          ‾‾‾‾‾
                                           59.50

The greatest uncertainty in the calculation of the free energy of
formation is the heat of combustion of diacetylene. It can be
calculated from the heat of atom linkage. On the basis of this
calculation, the heat of formation of diacetylene from the elements
is +117 Kcal. On the basis of the interpolation of the formula
by Kharasch, the heat of combustion is 367.1 Kcal. per mole and,
therefore, the heat of formation is 128.1 Kcal. at room temper-
ature. Finally the heat of combustion has been determined by
Neukirch and Doebke to 353.3 Kcal. per mole which corresponds to
heat of formation of 107 Kcal. It is very difficult to determine
the heat of combustion experimentally because, during the com-
bustion, soot is deposited only too readily; therefore, most
values are too low. Actually the experimentally determined heat
of formation of diacetylene is surprisingly small and leads to
the conclusion that the diacetylene formation from acetylene is
slightly exothermic which seems quite incredible.

$$2 C_2 H_2 = C_4 H_2 + H_2 + 1,300 \text{ cal.}$$
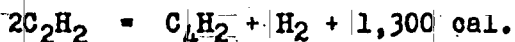
The values calculated according to Kharasch were used in the
actual calculations and also for the plotting of the graphs.
The equilibria of all the investigated gases were characterized
by the temperature at which 50% of the gas represented on the
left side of the equation is present. With increasing temper-
ature the equilibrium is shifted to the right side. A large
number of tables and graphs is included in the article.

$$2CH_4 \;=\; C_2H_4 \;+\; 2H_2 \qquad 1160°C$$

$$2CH_4 \;+\; C_2H_2 \;+\; 3H_2 \qquad 1090°$$

$$CH_4 \;=\; C \;+\; 2H_2 \qquad 430°$$

$$C_2H_6 \;=\; C_2H_4 \;+\; H_2 \qquad 675°$$

$$C_2H_4 \;=\; C_2H_2 \;+\; H_2 \qquad 925°$$

$$C_6H_6 \;=\; 3C_2H_2 \qquad 1070°$$

$$C_4H_6 \;=\; C_4H_4 \;+\; H_2 \qquad 800°$$

$$C_4H_6 \;=\; C_2H_4 \;+\; C_2H_2 \qquad 300°$$

$$C_4H_4 \;=\; 2C_2H_2 \qquad 485°$$

$$C_4H_4 \;=\; C_4H_2 \;+\; H_2 \qquad 825°$$

$$2C_2H_2 \;=\; C_4H_2 \;+\; H_2 \qquad >1500°$$

$$1\text{-}C_4H_{10} \;=\; 1\text{-}C_4H_8 \;+\; \qquad 485°$$

Pages 2505-2520

An Investigation of the Gasoline
Synthesis According to Franz Fischer
by K. Meisenhelmer

The first investigations with a pure cobalt catalyst without any addition, yielded 135 cc. of liquid and solid products from 1 cu.m. of synthesis gas (H_2:CO = 2:1). Fischer obtained only 150 cc. with his cobalt-thorium oxide-infusorial earth catalyst. This proved that cobalt is the actual catalyst for the gasoline synthesis, but the lifetime of this pure cobalt catalyst is extremely short. Magnesium oxide was used as the stabilizer. A great number of catalysts which contain, besides cobalt, different amounts of magnesium, yielded under normal process conditions (4 liters of gas/hr., and 4 g. cobalt as metal) 100 cc. liquid and solid products/cu. m. gas. By increasing the amount of catalyst up to 32 g. cobalt as metal, the yield was also increased to 133 cc. liquid and solid products. The lifetime of the catalyst was about 100 days. Aluminum oxide, kaolin, and especially infusorial earth are good carriers for the cobalt-magnesium oxide catalyst. The synthesis gas was purified by passing it over Cu at 300°C. This procedure kept the S content to 0.5 mg./cbm. The catalyst was prepared from $Co(NO_3)_2$ and

$Mg(NO_3)_2$ either by roasting them together or precipitating them with KOH, in which case hydrazine was added. The reduction temperatures for the catalyst ranged from 225-700°, but it was found that at too high temperatures the reduced cobalt starts to sinter, and thereby loses its activity.

Pages 2522-2551

Extractive Hydrogenation
of Coal According to Uhde-Pfirrman
October 1937-March 1938

The main procedure consists of extracting pulverized coal with aromatic phenol-containing medium oils in the presence of hydrogen. After this treatment the ashes and unreacted coal have to be separated by filtration. The filtrate is distilled to regain the medium oil for reuse in the process. The residue from this distillation which is called the primary bitumen is a partially hydrogenated product. The procedure was investigated under large laboratory and semi-pilot plant scales. The procedure is compared with the coal extraction process of Pott-Broche. The filtration of the crude extract from ash and un-reacted coal proved very difficult. The reaction temperature is about 410°, the reaction time 60 min., the pressure 350 atm., and 7-8% hydrogen.

The report is accompanied by 8 charts and drawings. This report was very difficult to read because the carbon copies were very poor.

Pages 2552-2579

"Separation of Gaseous Hydrocarbons from the Gas
Mixture by Washing with Liquids"   December 1938

The solubility of methane, ethane, propane, n-butane, i-butane, ethylene, propylene, n-butylene, i-butylene, butadiene, acetylene, and vinylacetylene were determined with a great number of organic solvents at 20°C; for instance, one of the solvents which is especially well adaped for the isolation of olefinic gases is tetralin. It is possible to separate gaseous olefins as a whole from other hydrocarbons, but not to separate them from each other. Another procedure was the separation of olefins from gases, obtained by the distillation of lignite, by washing with a cuprous salt solution. Carbon dioxide, hydrogen sulfide, and sulfur dioxide will also dissolve in organic solvents, therefore, they have to be removed from the gaseous mixture. It is possible to separate gaseous hydrocarbons by washing with oil. In the lube oil plant at Leuna, the olefins,

especially propylene and butylene, were separated by washing
with oil on a large scale; daily charge was 3,000 cu. m. crude
gas. The crude gases rich in olefins were obtained by thermal
cracking of lignite distillates. The gaseous olefins together
with an oil distillate from the coal hydrogenation were
polymerized to lube oils with aluminum chloride as catalyst.

Seven tables follow. Parts of this report were unread-
able because of poor carbon copies.

Pages 2582-2607

Alkylbenzenes, Their Production, Properties and Use as Antiknock Agents and Safety Fuel
March 24, 1939

The alkylation of benzene and some of its homologues
was effected with olefins in the presence of sulfuric acid under
atmospheric pressure. Polymethyl and polyethyl benzenes were
synthesized using chloroalkyls according to Friedel-Crafts.

The lower limit of sulfuric acid concentration for
alkylation depends on the rate of absorption of a particular
olefin by the sulfuric acid; for instance, isobutylene is ab-
sorbed faster than n-butylene and propylene whereas ethylene
does not react at all with sulfuric acid as an alkylating agent
regardless of concentration. Systematic experiments with benzene
and propylene show that at least in this case it is more advan-
tageous to use 92% sulfuric acid. At higher acid concentrations
the yield of alkylbenzene decreases while at lower acid concen-
trations the side reactions are favored; as for instance,
polymerization of the olefins. The best reaction temperatures
were found to be 10°C and lower. The amount of acid employed
has also some effects on the reaction. In the case where the
production of mono-alkylbenzenes is intended, a greater amount
of acid is an advantage.

After the reaction has been completed, the separated
oil phase can not be distilled immediately. It has to be freed
first from the alkylsulfates and sulfoacids. This is done by
washing the oil phase with alkali or boiling it with an alkali
under reflux. The acid-free oil is then fractionated.

The content of each fraction on olefins and aromatics
was determined. In most cases, mixtures containing unchanged
hydrocarbons, mono- and di- and higher alkylated benzenes; di-
products prevail. The preferred formation of the one or the
other reaction product was quite different under equal conditions
with different olefins and benzenes. For example, in the reaction
of benzene + propylene and benzene + n-butylene, the yield of

monoalkyl derivatives exceeded substantially that of dialkyl
derivatives. In the reaction benzene + i-butylene, dimerisation
occurs first. The dimers break up and react with benzene to
give the dialkyl product. The reactions, toluene + i-butylene
and i-propylbenzene + i-butylene prefer mono-alkylation. Xylene
gives predominantly monoalkyl derivatives with propylene,
butylene, and i-butylene. Also the alkylation of ethylbenzene
yields mainly mono-substitution products, whereas in the case
of diethylbenzene monoalkyl derivatives are only produced in
the case of propylene and n-butylene. With i-butylene only
polymerization occurs and almost no mono- or dialkyl products
are obtained. 35% of the products of this reaction are high
boiling polymers without any defined boiling point.

     Yields in the case of benzene are 90% and higher under
optimum conditions. The alkylation of benzene homologues give
lower yields especially in the case of xylene. The loss of
olefins by side reactions is especially great in this case.
Since large amounts of sulfuric acid are needed for the alkyl-
ation, it has to be regenerated. Olefins diluted with alkanes
could also be used for alkylation of benzene. The use of diluted
40% propylene, obtained by chlorinating and dechlorinating of
propane, gave similar results.

     The use of these alkylbenzenes as a safety aviation
fuel is proposed because of its high boiling point (over 200°C)
and its high octane value. They have a low pour point, a high
flash point, and a high heat value (10,000-11,000 Kcal./kg.)
with a higher hydrogen content (10-12%) than benzene (7.7%).
They have a lower heat fading than benzene, toluene, or xylene.
They also have a higher lead susceptibility than benzene,
toluene, or xylene; for instance, 20% alkylbenzene-containing
mixtures have the same lead susceptibility as 50% mixtures of
benzene, toluene, and xylene.

     The article is accompanied by numberous tables and
graphs.

Insert between pages 2038-2039
Report by Dr. Walter Krönig to Dr. Holroyd of May 29, 1945

The Production of Aviation
and Heating Fuel from Upper Silesian Coal

     The flow sheet is made up for the working of coal only,
small amounts of tar from the coal distillation are omitted.
Quantitative data are calculated on an hourly basis; one year
totals 8,000 hours. The analyses of products with number
indices are tabulated in Appendix A. Letter indices refer to
explanations in Appendix B.

APPENDIX A

Flow Sheet of the Hydrogenation Plant at Blechhammer

Analysis of the Products

No.	Oil-Free Material of Sump Phase — Kind	% H$_2$O in Crude Product	% Ash in Dry Substance	% C in Pure Substance
1	Crude Coal	10	5	81.0
2	Bayermass	15	-	-

No.	Oil-Containing Material of Sump Phase	% Solid	Distillation Analysis of Oils in %, °C			
			− 160	160-210	210-325	>325
3	Grinding Oil	10.0	0	0	40.7	59.3
4	Thick Paste	51.0	-	-	-	-
5	Heavy Oil Let-Down	27.0	0	0	33.0	67.0
6	Thin Paste	43.0	-	-	-	-
7	Catch Pot Heavy Oil	0	0	0	0	100.0
8	Catch Pot	0	5.50	4.06	44.75	45.69
9	Dilution Oil	0	0	0	42.8	57.2
10	Centrifuge Mixture	16.0	0	0	37.8	62.2
11	Operational Loss during Centrifuging	16.0	0	0	37.8	62.2
12	Centrifuge Oil	11.2	0	0	37.8	62.2
13	Centrifuge Residue	38.0	0	0	37.8	62.2
14	Operational Losses during Distillation	38.0	0	0	37.8	62.2
15	Distillate from Flash Chamber	0	0	0	84	15 1
16	Concentrated Centrifuge Residue	53.0	0	0	5.0	95.0
17	Distillate	0	0	0	15.0	85.0
18	Distillation Residue	95.0	0	0	10.0	90.0
19	Operational Loss from A-Distillation	0	5.50	4.06	44.75	45.69
20	Fuel Oil for Sale	0	0	0	34.05	65.95
21	Gas Phase Products	0	23.1	17.1	59.8	

No.	Material of Gas Phase	% Boiling			E.P.	V.P.	% N No Pb	With 0.09 Pb
		−100°	-155°	>155°				
22	Catch Pot from Pre-hydrogenation	-	35.0	65.0	-	-	-	-
23	Loss from B-Distillation	-	35.0	65.0	-	-	-	-
24	Gasoline Recovery, Catch Pot	-	49.6	50.4	-	-	-	-
25	Loss from C-Distillation	-	49.6	50.4	-	-	-	-
26	Aviation Fuel	55	98.0	-	157	0.5	74	88

I. SUMP PHASE

1. Preparation of the Coal

112.5 t. crude coal ① = 96.20 t. purified coal

1.29 t. $FeSO_4.7$ aq. | a | addition Cat. I

b | drying

104.0 t. moisture-free coal + Cat. I

1.69 t. "Bayermasse" addition Cat. II

②

0.34 t. Na_2S → | o | Addition Cat. III

③

105.69 t. moisture-free coal + Cat. I, II, III

124.6 t. grinding oil → | d | grinding

228.37 t. total paste

④

e

e

138.30 t. paste

oil
27.50 t. grinding

f | dilution

90.07 t. paste

152.10 t.

15.78 t.
heavy-oil-let-down
⑤

181.58 t.
thin paste
⑥

2. High Pressure

g | heat exchangers

5.26 t.
grinding oil
(lube oil)

1

h | hairpin
preheater

15.78 t.
Recycle heavy oil-let-down

i | converter
oven

31.56 t.
heavy-oil-let-down

5.26 t. Catch Pot
Heavy Oil = Wash Oil ⑦

31.56 t.
Heavy-Oil-
Let-Down

| 1 | Converter Ovens |

132.60 t.
Heavy-Oil
Let-Down

| k | Separator |

| m | Catch Pot |

144.85 t. Catch Pot ⑧

101.04 t.
Heavy-Oil-
Let-Down

3. Working of Residue

157.36 t.
Grinding
Oil

171.31 t.
Centrifuge Mixture ⑩

0.39 T. Mechanical
Loss ⑪

| n | Centrifugation |

⑫

140.31 t.
Centrifuge Oil

30.10 t.
Centrifuge Residue ⑬

70.26 t.
Dilution Oil ⑨

7.55 t.
Distillate

| o | Flash Chamber |

⑭

0.15 t.
Mechanical
Loss

⑮

22.05 t.
conc. Centrifuge
Residue ⑯

6.47 t.
Distillate ⑰

| p | Spherical Oven |

13.20 t.
Distillation
Residue ⑱

4. A-Distillation

70.26 t.
Dilution Oil

144.85 t.
Catch Pot

5.25 t.
Catch pot
Heavy-Oil

7.93 t.   160°C

q
A-
Distillation

5.85 t.
160-210°C

30.10 t   64.26 t.
210-325°C   210-325°C

0.72 t. Mechanical Loss   (19)

34.16 t.
210-325°C

66.09 t.
>325°C

r   Liquor
Enrichment

5.26 t. >325°C
Catch Pot Heavy-Oil

13.78 t. 210°C

40.16 t.
>325°C

60.83 t.
>325°C

20.67 t.
>325°C

31.30 t.
Fuel Oil
(20)

10.63 t.
210-325°C

20.50 t.
210-325°C

II.   GAS PHASE

1.   Prehydrogenation

34.28 t. gas phase products   (21)

s   High Pressure

33.30 t. Catch Pot   (22)

33.30 t.
Catoh Pot   ㉒

15.58 t. 155°C | t | B- Distillation | 0.17 t loss   ㉓

17.55 t.
>155°C

29.84 t.
aviation fuel

2. Gasoline Production

| u | High Pressure | 14.40 t. >155°
recirculation

28.80 t. catch pot
㉔

14.26 t.
155°C | t | C- Distillation | 0.14 t. loss   ㉕

14.40 t.   >155°C

Total Hydrogen Consumption

     128,000 N ou.m. (97.0%)

Total Yield of Gaseous Hydrocarbons:

CH_4 = 5.50 t.
C_2H_6 = 4.38 t.
C_3H_8 = 6.50 t.
C_4H_{10} = 6.75 t.

APPENDIX B

Explanations

a ═ The salt in water solution is sprayed on the moving coal (about 40°C crystallization temperature).

b ▾ Partial neutralization of the alkali in the coal takes place during heating by the SO_4 ions of the catalyst.

c ═ Because of its sensitivity the salt is added just before the introduction of coal into the oil.

d ═ The entire coal is ground to a thick paste; during this procedure the remaining water evaporates. The thick paste contains 103.77 tons moisture-free coal plus catalysts I, II, and III, also moisture-free.

e ⤳ The thick paste is so proportioned that a large part (66.9%) of the total paste (271.65 tons) goes through the heat exchangers.

f ═ The thick paste is diluted in order to be able to heat it in the heat exchangers. The solid content of the paste should not exceed 43%. Heavy-oil-let-down is also used for dilution to improve the carrying capacity of the not very viscous grinding oil by addition of oils rich in asphalt.

g ═ There are four equally equipped chambers for the sump phase hydrogenation. The reaction conditions are 700 atm. total pressure, 585 atm. H_2 partial pressure in the entrance chamber, 3.5 N cu. m. total gas/kg. coal. The yields are 96% of C in coal decomposed and 21% of the C gasified. Calculations were performed with a C percentage of 86.0 in the produced oil. There are three heat exchangers in each chamber. The temperature at the exit of the heat exchangers is 310°C.

h ═ For each chamber one hairpin preheater with about 30 hairpins of a capacity of 12×10^6 Kcal. The temperature at the exit is 415, maximum 425.

i ═ Each chamber has four ovens. The temperature is kept at 485. All four chambers have a combined reaction volume of 148.0 cu. m.

k ═ For each chamber one separator.

l ═ Since it suffices to have available only half of the recycle heavy-oil-let-down at higher temperatures, only that amount of the recycle heavy-oil-let-down is recycled hot to conserve energy.

m ■ Each chamber has one cold catch pot.

n ■ Contains de Laval centrifuges.

o ■ The centrifuge residue is heated to 450 under 50 atm. pressure, first, in the heat exchanger then in the hairpin preheater to keep it from drying out because of it high medium oil content. To increase the flow velocity, small amounts (maximum 0.1 N cu. m./kg. centrifuge residue) of compressed distillation gas are added. Expansion takes place in the flash chamber and a large part of the oil is evaporated and topped.

p ■ The concentrated centrifuge residue flows from the flash chamber under its own gravity into a spherical oven. The distillation residue is thrown away.

q ■ Gas heated distillation at N pressure gives the following fractions: To 160°, 160-210°, 210-325°, >325°.

r ■ The fraction, 160-210, can be led through the liquor enrichment off the "Phenosolvan Plant," to enrich the liquor (8-10 g. phenol/l.) or the already dephenolized water (.2 g./ l.) up to 18 g./l. Simultaneously, the more valuable phenols can be exchanged against the less valuable phenols. Material balance changes due to this procedure have not been considered in this flow diagram.

s ■ Two chambers with three ovens each of 8 cu. m. catalyst volume; two ovens, catalyst 8376, one oven, catalyst 5058. Conditions: 300 atm. total pressure, 410 temperature and 0.8 kg./l./hr. maximum throughput. The reaction product has an IBP of 50°C.

t ■ Steam heat distillation at atm. pressure. Stabilization and washing of the gasoline are omitted in this scheme because everything was calculated with gas-free products.

u ■ Two chambers with two ovens each of 8 cu. m. catalyst volume, catalyst 6434. Conditions: 300 atm. total pressure, 390 temp., and 0.45 kg.-155°/l. catalyst volume per hour.