

FILM STUDY GROUP

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T. O. M. REEL NO. 148

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UNITED STATES GOVERNMENT TECHNICAL OIL MISSION

MICROFILM REEL 148

SUMMARY

Reel 148 of the microfilms obtained by the United States Government Technical Oil Mission was examined in response to a request from A. E. Miller, Chairman of the API-TOM Study Group.

The attached report contains a table of contents and summaries of all the reports included in Reel No. 148.

5/21/47

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50 ATMOSPHERE DEHYDROGENATION WITH A
CATALYST THAT CAN BE REGENERATED. RE-
PORT ON RESULTS IN THE 100 CGM. FURNACE.

Ref. No.:	N-15	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Guenther
Date:	8/17/40	Contents:	12 text pages
Frame:	1-20		6 tables
			4 figures

SUMMARY

1. Contact masses for the preparation of gasoline by dehydrogenation at 50 atmospheres were developed. The best contact masses were made of molybdenum and active clay.
2. The addition of alkali to the molybdenum-clay contact masses lowered their activity (Rudoerfer contact mass).
3. The dehydrogenation activity rises with the Mo concentration at least up to a concentration of 100 kg. Mo per cubic meter.
4. An experiment was run for a period of 125 days during which time the contact mass was regenerated five times. The contact mass regenerated once was more active than the fresh contact mass. After the fifth regeneration the same good results were obtained as after the first regeneration. The used contact mass was solid. No disintegration had set in.
5. By dehydrogenation of the fractions boiling above about 100°C. from gasoline samples 6434 or 7019 and later addition of the corresponding low boiling fractions, there was obtained with 4-7% loss (gas and coke, however no distillation residues) gasolines of higher knock rating and of excellent aromatic distribution. It is to be assumed that this gasoline is better than all known aromatized gasolines, including Welheim gasoline.
6. The activity of the contact mass drops off rather slowly. In the preparation of naphthenic-aromatic gasolines (7019) the contact mass could be used for periods of over four weeks. With naphthenic-paraffinic gasolines (6434) periods of 8-14 days are reached with a reduction in the aromatic formation of about 20 per cent.
7. Increased aromatic formation is accompanied by higher gas formation.

Frame 1-20 (Contd.)

8. By increasing temperatures stepwise excellent yields of aromatics are obtained with a relatively small amount of gas formation.

9. In the first experiments the influence of chromium on retarding gas formation could not be confirmed.

TESTING OF CONTACT MASSES FOR
HYDROGENATION IN THE 100 CC FURNACE - 408 NUMBERS 1-8

Ref. No.:	N-16	Report No.:	LU 558
Origin:	I.G. Farben Ludwigshafen	Author:	Nonnenmacher
Date:	8/13/40		Oettinger & Donath
Frame No.:	21-29	Contents:	6 text pages, 6 tables

SUMMARY

In eight 100 cc. furnaces of which four were arranged in a lead bomb, a series of contact masses were compared under the usual dehydrogenation conditions (15 atm. of hydr. pressure, 500°C., 0.5 kg. per liter per hours throughput) according to their tendency to dehydrogenate heavy gasolines rich in hydrogen - with the contact mass 7360 (active alumina and 6% molybdenum trioxide).

By the addition of 1% Cr_2O_3 (7612) or 0.34% as well as 3.4% zinc oxide (7615 and 7614) contact masses were obtained that were somewhat more active in aromatic formation and cracking than 7360.

Compared on equal quality of product contact mass 7612 gives somewhat less gasification than 7360.

The results were confirmed in part in larger autoclaves.

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REPORT OF THE OIL CONFERENCE HELD AT LEUNA 12/22/37

Ref. No.: N-17
Origin: I.G. Farben Leuna
Date: 12/22/37
Frame No: 30-80

Report: 558

SUMMARY

A total of 11 papers were given at this meeting:-

PART A. Discussion of Nitrogen

1. Sale and market conditions for nitrogen fertilizers in Germany and foreign countries. (Oster)
2. Sale and market conditions for technical nitrogen. (Hanser)
3. Food conditions and sale of nitrogen. (Stroebel)
4. Status of the projects in foreign countries. (Fahrenheit)
5. Progress and development of production. (Goldberg)

PART B. Discussion of Oils

1. Sale and market conditions for gasoline. (Fischer)
2. New developments in the field of hydrogenation. (Pier)
3. The development of special steels for high pressure apparatus. (Wyazonirski)
4. The construction of hydrogenation apparatus and their simplification. (Von-Feldert)
5. Significance and position of the soft coal production in Leuna. (Henning)
6. The oil industry in America. (Buetfisch)

THE CALCULATION OF GASOLINE
OCTANE NUMBERS BY ARITHMETICAL MEANS

Ref. No.:	N-18	Report No.:	LU 558
Origin:	I.G. Farben Ludwigshafen	Author:	Wehn
Date:	8/9/40	Contents:	5 pages text
Frame:	81-88		4 tables
			1 figure

SUMMARY

It is often desirable to obtain a quick evaluation of octane numbers of gasolines. The customary evaluation of the octane number of gasoline, particularly when aromatics have to be removed first, takes large quantities of samples and also considerable time. On the other hand the octane number of the original gasoline without the aromatics removed is always available. In this article the author attempts to evaluate the octane number of the pure gasoline without aromatics from the octane number of the original gasoline and its aromatics content. From calculated and experimental data it is shown that the octane number for gasolines can be calculated with an error of ± 3 units if its constituents are known. Also indirectly the possibility of calculating the isoparaffin content from the isobutane content is demonstrated. It is also shown that the octane number of aromatics in gasoline can be calculated rather exactly from certain formulas given in this paper, while in other cases great deviations are found. This is probably due to poor experimental data. (The formulas are not included in this summary).

REPORT ON THE EXCHANGE OF EXPERIENCE
CONFERENCE HELD AT POELITZ FROM 8/7/42 TO 9/5/42

BEHAVIOR OF COAL RETORTS WITH A RISING TEMPERATURE

Ref. No.:	N-19	Report No.:	538
Origin:	I. G. Farben Poelitz	Authors:	Rank and Hahl
Date:	8/17/42	Contents:	11 pages text and 1 figure
Frame No:	89-98		

SUMMARY

(Article is not legible.)

HYDROCARBON SYNTHESIS FROM CARBON MONOXIDE AND HYDROGEN

Ref. No.: M-20
Origin: I.G. Farben Ludwigshafen
Date: 8/14/42
Frame No: 99-102

Report No.: LU 558
Authors: Michael
Contents: 5 pages text

SUMMARY

The emulsion method consists in carrying out the reaction in a sludge characterized by very small particles of gas and contact mass. As sludge the high boiling reaction products are used. The gas distribution was produced by means of foam stones with pores of 0.10 to 0.15 mm diameter. This contacting means can also be fixed in place by using finely divided metal of a particle size 1 to 5 microns in a heavy sludge. In order to counteract a sinking of the contact particles and to conduct away the heat of reaction, the heavy sludge is slowly (about 20 times an hour) agitated between the reactor and the cooler so that the heavy emulsion has an opportunity to be degassed at the highest point in the circulation system. Temperature between 240-280°C can be used usually 250°C. Exact conditions and materials to be used are given in the article.

THE MANUFACTURE OF LUBRICATING OILS FROM SYNTHETIC PRODUCTS

Ref. No.: N-21
 Origin: I.G. Farben Ludwigshafen
 Date: 8/13/42
 Frame No: 103

Report No.: LU 558
 Author: Michael
 Contents: 1 page text

SUMMARY

Lubricating oils can be manufactured from the products of olefin polymerization or from the products of chlorinated paraffin.

The heavy gasoline obtained by polymerization of olefins at 250°C yields a good V.I. oil only if the fraction boiling below 150°C is excluded before polymerization. If this is not done it has to be extracted with SO₂ before polymerization. The middle oil may be used for polymerization without treatment. The paraffin has to be hydrogenated and then chlorinated before use.

Motor oils are obtained by the polymerization of olefins from heavy gasoline and middle oil. These products may also be used as light components of aviation motor oils. Steam cylinder oil was obtained by using chlorinated paraffins as a starting material.

Starting Material	Theoretical Yield of Oily Fraction	Treatment of Starting Material	Product	Yield	
				Referred to Fraction	Total of Oily Fraction
Heavy gasoline 150-200°C	12%	Extracted and polymerized V.I. 86	Automobile oil	30%	4%
Middle oil 200-350°C	42%	Polymerized V.I. 83	Automobile oil	40%	17%
Paraffin 350°C	13%	Hydrogenated & chlorinated	Hot steam cylinder oil	50%	<u>7%</u>
					28%

Four per cent of propylene lubricating oil can be added to this total yield by polymerizing the propylene fraction.

DHD EXPERIMENTS IN THE 40 LITER FURNACE (3 DHD
FURNACE WITH INTERMEDIATE REHEATING. 1 REFINING FURNACE) 8/10/42

Ref. No.: N-22

Origin: I.G. Farben Ludwigshafen

Date: 8/10/42

Frame No: 104-128

Report No.: LU 558

Author: (illegible)

Contents: 7 pages text

14 tables

7 figures

SUMMARY

Scholven 5058/6434 gasoline was dehydrogenated to high-power fuel in the 40 liter oven 601 at pressures of 20, 34 and 40 atmospheres and with an adiabatically falling temperature. At 50 atmosphere and with favorable working conditions using a cycle lasting 20 hours 78% of stabilized gasoline boiling up to 165°C with an aromatic content of 68 to 69% by weight, leaving a distillation residue of about 5%. The total yield of liquid was therefore about 3-1/2% higher than from previous runs made in the 100 liter autoclave.

At 20 atmospheres pressure the same aromatic content was obtained and a 77% yield of stabilized gasoline and about 8% distillation residue.

THE DEPOSITION OF "CAVIAR" IN LIQUID PHASE FURNACE 8/10/42

Ref. No.: N-23

Origin: I.G. Farben Ludwigshafen

Date: 8/10/42

Frame No: 129-133

Report No.: L.U. 558

Authors: Rank, Leonardt
and Wittmann

Contents: 4 pages of text
6 tables

SUMMARY

The properties of all high-ash containing sediments and their coal ash from the liquid phase furnaces of semi-works and industrial plants were compared. The results obtained showed that sediments of highly different types can be formed; a fact which may be based on the common cause that the used coal contains a certain amount of alkali and alkali earth metals which is usually expressed as the alkalinity of the coal. Different types of sediment are formed depending upon the relative proportions of alkali and alkali earth metals present (bound to chlorine or carbonic acid (CO₂)). The results of the experiments are given in a table.

THE INFLUENCE OF TEMPERATURE AND
THROUGH-PUT IN THE 600 ATMOSPHERE
AROMATIZING OF LIQUIFIED BITUMINOUS COAL
MIDDLE OIL WITH "TERRANA" CATALYST 6/29/42

Ref. No.: N-24

Origin: I.G. Farben Ludwigshafen

Date: 6/29/42

Frame No: 134-146

Report No.:

Author: Trefinow

Contents: 4 text pages

2 tables and

7 figures

SUMMARY

1. The aromatization of middle oil obtained by liquifying anthracite coal from Scholven with catalyst 7501 (Terrana with No. 3) was studied.

- (a) At constant through-put and varying the temperatures between 460-510°C. and
- (b) At a constant temperature (26.0 MV) and varying the throughput from 0.8 to 2.0

The results of the two experimental series are shown in the form of tables and figures.

2. With increasing temperature the following can be shown.

- (a) An increase in the total gasoline fraction of from 48% at 24 MV to 90% at 27 MV and an increase in the gasoline fraction boiling up to 100°C. of from 40 to 68 per cent.
- (b) An increase in aromatics in the gasoline from 23% to 38% by raising the temperature from 493° to 510° C.
- (c) A big drop in the B-middle oil aniline point from +28° to -13°.
- (d) An increase in the case of vaporization. The vaporization curve of the gasolines obtained ascends very steeply in the temperature range 493 to 510°C. and reaches the value of 59% at the later temperature.
- (e) A decrease in the butane content from 50 to 20% and of the isobutane content in the total butane from 47 to 25%.

3. With increased through-put the following was observed.

- (a) A decrease in gasoline content from 80% at the through-put of 0.8 to 42% at a through-put of 2.0 and a decrease in the gasoline constituents boiling under 100°C. from 65 to 38%.
- (b) An increase in the aromatics in the gasoline from

THE INFLUENCE OF TEMPERATURE AND
THROUGH-PUT IN THE 600 ATMOSPHERE
AROMATIZING OF LIQUIFIED BITUMINOUS COAL
MIDDLE OIL WITH "TERRANA" CATALYST 6/29/42

(Contd. from Sheet -11-)

25 to 35% and at the same time a decrease of the B middle oil aniline point from 46 to -9°C.

- (c) A decrease in the vaporization from 25% to about 16%.
- (d) An increase in the iodine number of the gasoline from 3.8 to 5.7 and of the middle oils phenol content from 0.25 to 1.6.
- (e) An increase in the boiling point of B middle oil from 294° to 320°C.

CATALYST NO. 7846a250 6/26/42

Ref. No.:	N-24	Report No.:	LU 498
Origin:	I. G. Farben Ludwigshafen	Author:	Fuener
Date:	6/26/42	Contents:	4 pages text
Frame:	147-152		2 tables

SUMMARY

Since catalyst No. 7846 (Al_2O_3 - MoO_3 -NiO) gave good results in the semi-works prehydrogenation plant at Ludwigshafen, this catalyst was installed in Scholven but had to be removed after 2-3 weeks due to low activity. Since this reactor was technically different than the semi-works the cause of why the catalyst did not work could not be proved. Later tests on a small scale showed that the catalyst in its original form (containing no sulfur) was active in a small scale plant but had low activity in a large plant. The reason was found to be that in the small plant the catalyst sulfurizes at the start and thereby increases its activity while a technical plant catalyst keeps its original low activity. For a test at Leuna, catalyst 7846 was used in a sulfurized form.

The catalyst was then used at Leuna for about 8 months before it showed a decrease in activity but it still gave production of phenol and aniline compounds.

Then molybdenum was replaced by tungsten. An exact description of the manufacture of the catalyst and its different properties according to the different constituents and modes of manufacture is contained in the paper. The paper briefly compares results obtained with these modified catalysts.

HYDROCARBON SYNTHESIS FROM CO AND H₂ WITH
AN IRON CATALYST ACCORDING TO THE EMULSION METHOD

Ref. No.:	N-25	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	(Not given)
Date:	6/23/42	Contents:	2 pages text
Frame:	153-155		2 tables

SUMMARY

Tables are given (not included in this summary) which summarize the products obtained in a ten week run on a 1.5 cubic meter furnace at 250°C. and 20 atm. with a gas mixture of CO:H (55:45) and a daily yield of 0.3.

In smaller experiments a high percentage of high boiling products were obtained. The paraffin content was as high as 40 per cent. Some of the products were further worked up to yield alcohols, sulfonates, fatty acids, lubricating oils and leather oils.

REMARKS ON THE REPORT: "AN INVESTIGATION OF THE HYDROGENATION
OF A COLLOIDAL COAL SOLUTION" BY PROF. JOSE PERTIERRE 6/11/42

Ref. No.:	N-26	Report No.:	LU 558
Origin:	I.G. Farben Ludwigshafen	Author:	Reitz
Date:	6/11/42	Contents:	3 text pages
Frame:	156-158		

SUMMARY

This work is a summary of a series of experiments by the Prof. Pertierre. Included are experiments on hydrogenation of colloidal solutions of soft coal in anthracene oil, the partial elimination of oxygen from coal through heating under pressure with hydrogen with dispersion agents, softening point of coal after hydrogenation, extraction of coke, with boiling anthracene oil in a CO₂ atm. Autoclave experiments with tetralin, with phenol, and with tetralin-phenol mixtures as solvents were made. Measurements of density, viscosity, surface tension, and immersion heat on introduction of dry coal also were made using these solvents. The author describes measurements of the wetting test, pycnometric measurements of the density of coal samples, the analysis of the chemical structure of colloiddally dissolved coal and compares them with results of other authors.

ALKYLATION OF UNSATURATED GASOLINES 6/8/42

Ref. No.: N-27
Origin: I. G. Farben Ludwigshafen
Date: 6/8/42
Frame: 159-164

Report No.: LU 558
Author: (Not Given)
Contents: 2 pages text
4 tables

SUMMARY

In connection with a conference on hydrocarbons the results obtained in the alkylation of unsaturated gasolines and specifically cracked gasolines were discussed. It was disclosed that the problem was being studied at Oppau, Leuna, and in the Ruhr. Gasolines with remarkable antiknock values were obtained in the Ruhr and at Oppau. The alkylation of unsaturated gasoline instead of butylenes is a fact that can be explained by the higher olefins obtained by thermal cracking contain undesirable diolefins and small demand for the high boiling fractions. Thermally cracked Fisher-and petroleum gasolines, and Michael-gasoline after catalytically cracking contain very little diolefins and are good starting materials for alkylation. In contrast to the C-4 alkylation, the treatment of the higher olefins require a high excess of butane (more than 5 volumes) and a longer reaction time. The reaction temperature should be 0-10°C., the ratio of gasoline: acid (1:1-1:2).

The gasolines alkylated in this study were a primary gasoline (Michael) boiling below 150°C. and refined over bauxite and a similar gasoline boiling below 300°C. and refined over bauxite, cracked gasoline from Michael middle oil and a cracked gasoline from (Reitbrock-petroleum).

THE PROCESSING OF SYNTHETIC MIDDLE OIL
(DR. MICHAEL) BY CATALYTIC CRACKING AND ALKYLATION

Ref. No.:	N-28	Report:	LU 558
Origin:	I.G. Ludwigshafen	Author:	Free
Date:	6/8/42	Content:	6 pages text
Frame:	165-171		1 table

SUMMARY

Middle oils from the CO-H₂ synthesis (Dr. Michael) can be easily converted into highly unsaturated gasoline through catalytic cracking. A high yield is obtained and the resulting gasoline (from b-middle oil) has a higher aniline point than the original oil. Also obtained are liquefied gases with over 50% olefins and a slight amount of dry gas and coke. The losses due to vaporization and coke formation were only 27.2%. Through alkylation of the unsaturated cracked gasoline 15.3 to 11.1% (corresponding to the original oil) of an alkyl gasoline (E=165°) was obtained. This gasoline had an iodine number of 30 to 34, an octane number of 83.2 and of 102 with 0.12% of Pb. Through the combination of catalytic cracking and alkylation 97.9 to 99.4% liquid reaction products could be obtained from the middle oil. With more favorable alkylation conditions the yield and quality of the alkylate could be improved still further.

THE PRODUCTION OF THREE ISOMERS--ETHYLTOLUENE,
DIMETHYLCYCLOHEXANE, AND ETHYLCYCLOHEXANE

Ref. No.:	N-29	Report No.:	LU 558
Origin:	I.G. Farben Ludwigshafen	Author:	Bueren
Date:	6/4/42	Contents:	2 pages text 1 table
Frame:	172-174		

SUMMARY

O-, p-, m-bromotoluene were synthesized by the Sandmeyer reaction and purified. The ethyltoluenes were prepared by the Wurtz-Fittig reaction and the dimethylcyclohexane and ethylcyclohexane were prepared by hydrogenation of the pure dimethylbenzenes and ethylbenzene. The exact procedure for these syntheses are given and tables of their physical properties (including density, boiling point, and refractive index) are given.

THE EFFECT OF THROUGH-PUT ON THE "CRACKING" OF
BRUCHSAL GAS OIL WITH HF TREATED "TERRANA" 7/11/42
(CRACKING AND HYDROGENATING, 11TH COMMUNICATION).

Ref. No.:	N-30	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Trofinow
Date:	7/11/42	Content:	5 pages text
Frame:	175-184		4 tables
			1 figure

SUMMARY

The through-put varied between 0.5 and 2.0 during the "cracking" of Bruchsal gas oil (Pl203) over the contact No. 6109 (Terrana treated with HF). The cracking was carried out in a 200 cc. furnace without regeneration of the contact at a constant temperature of 210 MV or 408°C.

When the through-put was increased from 0.5 to 2.0, the gasoline concentration was reduced from 54% to 20% and the fractions boiling below 100°C. decreased in the gasoline from 58% to 31%. The gasoline yield reached its highest value of 0.36 to 0.38 at a through-put of 1.0. The vaporization increased with the through-put from 18% to 23%, however, the composition of the vapors is independent of the through-put. The gasoline octane number decreases from 71 to 61 with rising through-put. This decrease is explained by the decrease of low boiling fractions in the gasoline and by the decrease of the aromatics in the higher boiling fractions. At a lower through-put the gasoline receives more of the higher boiling aromatics than at a higher through-put. The composition of the whole gasoline is not changed appreciably with a change of through-put.

At a through-put of 0.5 the gasoline fraction boiling up to 155°C. has an octane number of 67.5 measured by the motor method and 60% of this gasoline boiled below 100°C.

The gasoline obtained with a through-put of 0.5 has nearly the same properties as a gasoline obtained under the same conditions which has been cracked in $\frac{1}{2}$ hour cycles, however the former process is less efficient in the production of gasoline, but produces gasoline of lower volatility.

In regard to the yield of gasoline, volatility and the properties of the gasoline, catalyst 6109 produces the same results as catalyst 6434 only at a lower through-put of 0.5 and at a higher

THE EFFECT OF THROUGH-PUT ON THE "CRACKING" OF
BRUCHSAL GAS OIL WITH HF TREATED "TERRANA" 7/11/42
(CRACKING AND HYDROGENATING, 11TH COMMUNICATION)

(CONTINUED)

temperature of 21.0 MV. At a through-put of 2.0 catalyst 6109 produces only 38% of the gasoline produced by catalyst 6434. The gasoline produced by 6109 is also of lower quality than the gasoline from 6434.

250 ATM AROMATIZATION CATALYST NO. 8688 PRELIMINARY RESULTS

Ref. No.:	N-31	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Peters & Tropinow
Date:	3/9/42	Contents:	2 pages text
Frame:	185-187		1 table

SUMMARY

A new aromatization catalyst 8688 was developed which had the following composition: Support: 90 parts activated clay (8500), 10 parts terrana HF (6109) + 1 weight % zinc oxide as acetate + 10 weight % chromium oxide as chromic acid, + 2 weight % molybdic oxide as ammonium molybdate.

This new catalyst has about the same efficiency and produces gasolines of the same volatility as catalyst 7019. However, the vapors contain considerably more butane and approximately twice as much isobutane as those from a 7019 gasoline. The gasoline has 7% less aromatics but considerably more low boiling constituents than the gasoline from 7019. The octane number is eight units better than the gasoline from 7019.

In contrast to catalyst with terrana as carrier the efficiency of this catalyst decreases only slowly. During 2,000 hours of use it decreased from 0.33 to 0.28. This catalyst was tested with oils obtained by the liquefaction of anthracite. Recycling was found necessary.

The catalyst can be regenerated by burning off in air.

DESCRIPTION OF THE PARAFFIN REMOVING PLANT
AT THE BRABOG WORKS IN ZEITZ, THIS HAS BEEN
DONE FROM THE POINT OF VIEW OF EQUIPMENT AND
OF WORKS MANAGEMENT IN ORDER THAT A COMPARISON COULD
BE MADE OF THE PARAFFIN REMOVING PLANT AT LUTZKENDORF

Ref. No. : N32

Origin: I.G. Farben Ludwigshafen

Date: 5/8/42

Frame: 188-193

Report No. : LU 558

Author: Hoehn

Contents: 6 pages text

SUMMARY

The differences between the paraffin removing works at Zeitz and the one in Luetzkendorf, except for the difference in the raw products, are as follows:

1. Mixing of the oil and propane takes place under more favorable conditions in Luetzkendorf than in Zeitz. At L, iso-thermal mixing is more nearly accomplished. The contact time during the mixing is much longer in the L plant.
2. The cooling process takes place under more favorable conditions in the L plant since the steam compressors permit more uniform cooling.
3. At the L plant higher pressures are used in order to transfer the mixture from the cooling tank to the filter reservoir.
4. At the Z plant all tanks are provided with equipment to prevent settling of paraffin by stirring or circulating by means of pumps.
5. At the Z plant the cooled mixture was transported exclusively by means of piston pumps, since the semi-works experiments at Leuna indicated that centrifugal pumps may destroy crystals which have formed.
6. At the Z plant stripping by means of steam is not applied in order to keep water out of the propane current.

THE PROCESSING OF A MIXTURE OF RUHR COAL TARS
PRODUCED AT 600 ATMS. IN A 10-L. FURNACE AND TOPPED AT 325°C.

Ref. No.: N-33
Origin: I.G. Farben Ludwigshafen
Date: 5/18/42
Frame: 194-227

Report No.: LU 558
Author: Hortmann & Hupfer
Contents: 19 pages text
17 tables
2 figures

SUMMARY

In the plant experiment for processing tar residue^(*) P1414 in ka804 several difficulties occurred which were not to be expected after the successful experiment in a 10 liter furnace P1094⁽¹⁾. The main difficulty was caused by insufficient asphalt decomposition which made the combustion of the residue sludge impossible. The carbon separation which occurred at the same time however was probably caused by the apparatus itself.

An accelerated experiment in the 10-L furnace was planned for the tar residue which was poured into the chamber in order to determine the actual nature of the above mentioned difficulties.

According to its qualities the so tested product distinguished itself from P1094 by higher softening point as well as by a higher content of solid hydrocarbons (solid benzines) and above all by s-asphalt as shown in the following table:

	<u>P 1416 get.</u>	<u>P 1094 get.</u>
Kraessar °C.	36.7	29.7
Solid hydrocarbons, %	9.6	8.4
s-asphalt in 0.1%	26.8	23.5

The product is therefore much more difficult to hydrogenate. It was not possible to obtain a residue sludge which could be completely burned in the 10-L furnace using an iron-titanium catalyst.

Trouble free processing could only be obtained with a molybdenum-catalyst. In order to accomplish this result the charge had to be 15% smaller when compared with P1094 at the same temperature (4780). As shown in the following table a higher asphalt decomposition was obtained and as was expected a lower efficiency and a somewhat higher yield of gas. The decomposition of the solids was poorer in spite of a longer reaction time.

(*) Distillation tar "Auguste-Viktoria".

(1) Composition 202401, 4/24/42

Frame 194-227 contd.

TABLE

	<u>P 1416 get.</u>	<u>P1094 get.</u>
Efficiency	0.47	0.39
Yield of gas	13.2	12.6
Asphalt decomposition%	99.1	99.3
Decomposition of solid%	93.7	100.0

A heavy viscous oil was obtained from P1416 by hydrogenation.

Increase of the catalyst up to 1% did not show any advantages.

The treatment of the tar residue with chlorine and iron-titanium was tried in order to eliminate molybdenum. After a short experiment using elemental chlorine, which had to be stopped, 85% of carbon tetrachloride diluted with middle oil was forced into the furnace with pressure. Even if an essential improvement in the total result could be noticed when compared with the use of the iron-titanium catalyst without chlorine, it was not sufficient to obtain a trouble free combustion of the residue sludge. Probably decreasing the size of the charge to approximately that which was used with molybdenum would have been more successful. It is probable very similar results as obtained with molybdenum would have been obtained in this case except for the gas yield which when using chlorine appeared to be less favorable.

However, it was impossible to prove these conclusions because a leakage appeared after seven days at the chlorine inlet to the furnace. Apparently the pressure of 600 atmosphere at the furnace temperature was too high.

The experiment was carried out by Dr. V. Hartmann in cooperation with Dr. Hupfer.

Processing of the residue was done by Dr. Gieg

Analysis: Dr. Schiffmann
Dr. Fuener
Dr. Meier
Dr. Hirschberger

STATUS OF EXPERIMENTAL CHAMBER 804 ON
MAY 4, 1942-PRODUCTION OF PRIMARY BITUMEN AT 700 ATMS.

Ref. No.:	N-34	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Not given
Date:	5/4/42	Contents:	4 pages text
Frame:	228-234		2 tables

SUMMARY

The experiment to prepare primary bitumen from Silesian coal at 700 atmospheres pressure showed that ovens with a small gas capacity are not suitable for semi-works scale due to the excessively small rate of gas flow. With pure through-put of .86 and 1.63 cubic meter gas per kilogram of pure coal, a yield of 0.49 of ash free bitumen and a moderate amount of production middle oil was obtained.

Due to the too large volumes of gases the filter efficiency was worse than in the 300 atmosphere test.

The conversion of the product into ash-containing bitumen (for briquetting) gave a yield of material for briquetting of .71.

THE STATUS TO DATE OF HYDROCARBON
SYNTHESIS ACCORDING TO THE EMULSION METHOD

Ref. No.:	N-35	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Michael
Date:	5/18/42	Contents:	5 pages text 2 tables
Frames:	235-241		

SUMMARY

The emulsion method, using 1.5 cubic meter oven did not show any technical difficulties. The oven can be charged for a period of several months without any difficulties. There is no doubt that also in an industrial plant, using ovens of 40-50 cubic meter capacity, the same results would be obtained.

The essential parts of the plant are the following (see picture): a vertical standing cylindrical oven which is equipped on the bottom part with several foamstones or candles; a chamber for the exit gases, a condenser to remove the heat of reaction and a centrifugal pump.

Chamotte-stones (baked but not glazed porcelain) are used as porous foam-stones. Using an oven of a height of 8 meters as represented by the 1.5 cubic meter oven, it has been found that a pore diameter of 0.2 mm. is too large and pores of the diameter of 0.1 mm. were found to be too small. Foam-stones having pores of 0.15 diameter have been ordered.

The question whether the height of the oven of approximately 8 meter is the right one or if smaller height would be sufficient could not be answered at this moment. At 250° using ovens of 8 meter height a daily yield of 0.3 was obtained. With a 3 meter height, 10 liter oven yields of .5 were obtained. However, using the 8 liter ovens then the optimum conditions of small ovens could not be maintained. On the other hand there exists a relation between the fineness of the foam and the yield and also between the foam fineness and the height of the oven. This question will be answered by a test using an oven of four meter height and 30 liter capacity.

CURRENT STATUS OF CATALYSTS. GAS PHASE CATALYST.

Ref. No.: N-35 Report No.: LU 498
Origin: I.G. Farben, Ludwigshafen Author: Piiner
Date: 1/6/42 Contents: 2 pages text
Frame: 242-243

SUMMARY

1. Prehydrogenation:

The preparation of a replacement for catalyst 5058 is described. It has a density of 1 and contains 250 kg. of WO_3 per cubic m. of catalyst in addition to 45 kg. Ni_2O_3 . The remainder consists of 700 kg. of activated clay. As starting materials technical aluminum sulfate (18% Al_2O_3), yellow clay from Bitterfeld and $NiCO_3$ are used. Details of the preparation are given.

2. Aromatization:

Laboratory experiments on catalyst 7019 are proceeding.

3. DHD catalyst 7360 (MoO_3 on activated clay from Oppau) is being produced at a rate of 30 cu. m. per month. The firmness of the pelleting is still faulty. The regeneration of used 7360 has been developed by Dr. Wittman and can now be tested on a commercial scale.

4. Catalytic cracking:

The catalyst consisting of 35% Al_2O_3 and 65% SiO_2 which has been tested and found to be satisfactory for catalytic cracking in 50 l. cases has been prepared in the laboratory in 200 l. quantities. The commercial scale manufacture is so similar to that of activated clay that the same apparatus can be used.

STATUS OF EXPERIMENTS ON SYNTHETIC OILS

Ref. No.:	N-36	Report No.:	LU 558
Origin:	I.G. Farben Ludwigshafen	Author:	Michael
Date:	1/6/42	Contents:	5 pages text
Frame:	244-249		1 table

SUMMARY

The main work of the recent period is the conversion to a commercial scale of the foam process in the foam plate oven. For this purpose an oven with 1.5 cu. m. capacity was ordered in the early summer of 1941. Successful experiments were carried out in the preceding months with a smaller oven (25 l. capacity) of similar construction which is still in operation. Experiments were carried out with a third oven of 300 l. capacity using intensive stirring in place of the foam plates. Difficulties arose at the stuffing boxes of the stirring well. The experiments were discontinued for the present and the oven was changed over to the foam plate type process.

Details of the oven construction are given, the preparation of the catalyst is described, and also its suspension in oil, and its regeneration. The conditions for operation of the ovens are: 20 atm. pressure, 55/45 ratio of CO to H₂, and temperatures in the range 250-300°. 280 is the optimum temperature. Sulfate containing foam plates must not be used. Quartz and glass powder plates of 0.1 and 0.2 mm. porewidth were used, but are too sensitive to thermal expansion on a large scale. Chamotte plates will be used in the future.

CATALYTIC CRACKING OF MIDDLE OILS WITH ALUMINUM CHLORIDE

Ref. No.:	N-37	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Free
Date:	2/9/42	Contents:	10 pages text
Frame:	250-264		4 tables
			1 figure

SUMMARY

With aluminum chloride cracking, middle oils of high hydrogen content are the best starting materials. With decreasing aniline point of the feed oils, the yield of gasoline falls and that of tar increases. Oils with aniline point of 65 yield as much tar as gasoline. Naphthene and asphaltic oils of aniline point of about 55 to 65 which are well suited for cracking over silica catalysts cannot be cracked commercially with aluminum chloride. Although a 70% yield of L gasoline is obtained by cracking these oils over a silica catalyst (with hydrogenation of the residue) only a 30-35% yield is obtained with aluminum chloride cracking. Oils outside the boiling range 200-250°C have not been investigated yet.

REPORT ON EXPERIMENT IN PREHYDROGENATION OF MIDDLE
OILS FROM LIQUEFIED BITUMINOUS COAL WITH CATALYST 7846.

Ref. No.:	N-38	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Not given
Date:	2/7/42	Contents:	9 pages text
Frame:	265-287		13 tables

SUMMARY

The prehydrogenation qualities of the contact 7846 W250 before hydrogenation were tested in a semi-works scale furnace having a catalyst volume of 750 liters for a period of 31 days. The charge was oil obtained by liquefaction of Silesian coal in a large scale unit in chamber 804, an a-middle oil from the Dilsenberg gasoline A.C., and likewise a mixture (1:0.5) consisting of a-middle oil obtained from the hydrogenation works in Schelden and s-gasoline obtained from the industrial plant chamber 804.

The oils were hydrogenated at a total pressure of 280 atm. which corresponds to a partial pressure of 240 atm. for the major part of the experiment. Near the end of the run the total pressure was increased to 290 atm. which corresponds to a partial hydrogen pressure of 247 atm. The temperature was 408 to 418°C. (21.6 M.V. at 40°C.), and a production rate of 0.60-0.81.

From the above charge was obtained b-middle oils with an aniline point of 46-51°C., nitrogen content of 0.006 to 0.01%, and a phenol content under 0.01%. These products could easily be "benzenated" over 6434 for the production of gasoline.

Previously prepared b-middle oils with lower aniline points and with a higher nitrogen content were not suitable for the production of gasoline.

From these experiments the following rules in the operation of commercial units should be followed. Due to fluctuation in the quality of the charge material in gasoline production, it is necessary to regulate the reaction temperature and production rate so that the desired aniline point and nitrogen content of the b-middle oils are obtained. It is necessary that catalysts be regulated to correspond with the through-put. It is therefore recommended to start with a low through-put and to change from hydrogen-poor to nitrogen-rich starting materials. The catalyst was of good quality at the start. Hardness tests on the catalyst were adequate.

MEMO ON THE CONFERENCE HELD IN LUDWIGSHAFEN
ON 1/30/42 ABOUT PROCESSING OF BRUEKTER-TAR

Ref. No.:	N-39	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Hupfer, Hartmann and Simon
Date:	1/30/42	Contents:	6 pages text 1 table
Frame:	288-294		

SUMMARY

1. Distillation. According to the experiences of Dr. Dallein in Leuna, it was found that tar is too sensitive with regard to temperature to be suitable for continuous distillation in a tube still.

2. High Pressure.

- (a) In an experiment in Leuna the tar with an addition of sulphur was charged without noticeable crusting at low temperatures for a period of 3 weeks.
- (b) Experiments in Leuna without the addition of sulphur resulted in crusting mainly through the formation of inorganic substances (iron sulfide) which probably were due to the decomposition of iron selenate.
- (c) In the experiment at Ludwigshafen and Poelitz at higher temperatures the crust formation was greater. The crusting consisted in the main part of organic substances. The crusting probably could have been reduced by improving the flow proportions which would have resulted in a sacrifice of the high yields.

The results were nearly always the same (yields 0.5 to 0.6).

On the basis of these experiments, it was agreed that it must be possible to find experimental conditions for processing Bruexter-tars without trouble, particularly since the tar shows a very high heat sensitivity and therefore does not require much heating, and since the flow conditions in an industrial plant does not favor crusting.

For this reason it was agreed to not make further experiments using lower preheating temperature with the addition of elemental sulphur.

The unfavorable experiment with addition of sulphur in Poelitz was not conclusive since the contact mass was not clean and the preheater was run at too high a temperature.

QUALITATIVE COMPARISON OF GASOLINE
PRODUCED FROM BITUMINOUS COAL "B" MID-
DLE OIL BY CRACKING AND BY HYDROGENATION.

Ref. No.:	N-40	Report No.:	LU 558
Origin:	I. G. Ludwigshafen	Author:	Reitz
Date:	1/28/42	Contents:	12 pages text
Frame:	295-317		15 tables
			6 figures

SUMMARY

For an exact comparison of quality the following gasoline samples were made from 8376 prehydrogenation middle oil obtained from upper Silesian coal:

Cracked gasolines made using terrana and with synthetic aluminum silicate catalysts and the same samples were hydrogenated and tested.

Gasolines obtained by hydrogenation at 600 atm. and 250 atm. using the terrana catalyst were also tested.

ND-gasolines containing 30% aromatics were obtained by hydrogenation of gasoline at 250 atm. The octane numbers were determined on 20°C. cuts boiling up to and above 100°C. as well as the residual gasoline. The octane numbers of the fractions were also calculated. The super-charge characteristics of the gasoline was determined in Oppau. The attached table contains some characteristic results.

Frame 295-317 (Contd.)

TABLE

	Catalytic Cracking				Gasoline Production		Gasoline Production + DMD
	Terrana After Hydrogenation of the un-sat.		Synth. Al-SiO ₂ Cat. After Hydrogenation of the Unsat.		250 atm.	600 atm.	
Gasoline endpoint °C.	158	153	157	157	152	144	165
Gasoline % _{100°}	58	58	51	41	44.5	53.5	57
Gasoline Vol. % Atom.	20.5	15.5	21.5	21.5	5	7	31
Oct. No. Engine Method	77	77.4	79.5	77	74.1	76.3	80.2
E.M. + 0.12Pb	91.1	93.2	94	93.5	91	90.6	95
75-100° E.M.	78	77.6	77.5	76	77.2	77.8	76.3
120-140° E.M.	75	74.7	76.8	74	68	68.5	87.4
Residue-Gasoline % _{100°} C	64.5	66	63	59	43	57.5	61
O. No. E.M.	75.4	76.3	74.5	75	73.7	73.5	75.8
E.M. + 0.12 Pb	92.5	-	93	92	-	-	-
-100° E.M.	79.9	79	-	79	77.4	79.1	79
100° E. M.	63.2	67	66.5	61.4	68	67.1	67
Supercharge-Behavior (Minimum)	Between B ₄ & C ₃		In min. as C ₃		As B ₄	Slightly better than B ₄	As C ₃
% Gasification (coke gases) for gasoline gases	30	-	27	-	17	15	22
% gasoline (Cal. from liquid end prod.)	30	-	45	-	100	100	100

Frame 295-317 (Contd.)

For cracked gasoline, the octane number of fractions remain practically constant between 100 and 135° (terrana) and 65-150° (aluminum silicate) because the higher fractions contain more aromatics, whereas the lead number decreases only slightly in this range. When the unsaturated compounds are hydrogenated, the terrana gasoline shows a slight improvement, particularly in the lower fractions, whereas the gasoline from the synthetic cracking catalyst shows a slight decrease in quality. It is to be noted that in the first case the supercharge curve is practically unchanged, whereas in the second case despite the lowering of octane number there is a definite improvement.

The hydrogenated gasolines (250 to 600 atm.) were very similar to each other, and in the fractions up to 85°C. were practically identical with the cracked gasoline. In the higher fractions the octane numbers decrease very steadily because of the lack of aromatics.

The 6434 gasoline dehydrogenated to 35% aromatics content was one to two octane numbers units poorer in the cuts under 100°C. whereas in the fractions over 100° the octane numbers increased very rapidly.

The octane numbers of the residual gasoline compound at the same boiling points are about one to two units better for the hydrogenation gasoline than in the cracked gasoline and a little poorer for the Dhd-gasoline fractions boiling over a 100° and possibly also the fraction under 100°C. Since the Dhd residual gasoline contains only a little material boiling above 100°C., the octane number of the residual gasoline in this experiment is not particularly significant.

The supercharge behavior of the Dhd-gasoline is practically as good as the corresponding samples from cracked gasoline hydrogenated over an aluminum silica catalyst. It is remarkable that the Dhd gasoline containing 30% aromatics without additives gave a curve identical with the C₃ curve (60:10:10). The good quality of the Dhd gasoline is due to the fact that it was manufactured from 6434 gasoline solely. By comparison with cracked gasoline it must be taken into consideration that in producing gasoline by dehydrogenation, the original oil can be totally converted into gasoline whereas in cracking, one can only convert a part of it. "B" middle oil cannot be cracked completely to gasoline.

THE CRACKING OF CRUDE MIDDLE AND
HEAVY OILS (PRESSURE AND VACUUM
DISTILLATES) OVER CATALYST 7935.
(ACCORDING TO TESTS CARRIED OUT
IN A 10 L. FURNACE WITH GAS CIRCULATION.)

Ref. No.:	N-41	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	(Not given)
Date:	9/28/42	Contents:	7 pages text
Frame:	318-337		19 tables
			3 figures

SUMMARY

In addition to experiments in the 1 liter furnace, experiments were also made in a 10 liter furnace with gas circulation, in which middle oils and heavy distillate oils from hydrogen rich and hydrogen poor petroleum were subjected to dehydrogenation cracking. The experiments were made as is shown in the table attached under 40-50 atm. hydrogen pressure at a through-put of 1.5 using an $Al_2O_3 \cdot MoO_3$ catalyst which can be regenerated.

(See attached table)

TABLE TO SUMMARY: FRAME 318-337

Charge	MIDDLE OIL		DISTILLATE 350°	
	H-Rich	H-Poor	H-Rich	H-Poor
Sp. Gr./50% Product	0.814/270	0.870/295	0.880/460 Cal	0.927/470° Cal
Aniline point/°H	72.7/13.9	48.5/12.2	-/12.6	-/11.3
			66% 325° Vac	50% 325° Vac
Total Pressure°	70 (90)	68 (80)	70 (90)	75 (90)
H ₂ Pressure	41	53	50	40
Temperature MV/°C	25/476	25/476	25/476	25/476
	26.3/501			26/493
Through-put	0.5	0.5	0.5	0.5
Cbm. Gas/kg.	2.0	2.0	2.0	2.0
Cycle-period-hours	40	12	12	20-33
Estimated attainable cycle-period-hours	Ca. 100	Ca. 50	Ca. 50	Ca. 25
Yield-Weight %				
Gasoline-170°	44	27.5	16.5	16
Middle-oil 170-350°	46	67.5	45	39
Heavy oil 350°	-	-	31.5	34
Gas (C ₂ or C ₃ =C ₄)	9.7	4.1	3.5	7
Coke	0.15	0.75	1.5	4
Gas+coke/gasoline+gas+coke	18%	15%	29.5%	40%
Gas+coke/fract.-350°+gas+coke	-	-	9%	16.5%
Consumed-H ₂ cbm/to new oil	90	115	95	163
Analysis of products:				
Gasoline:				
Sp. Gr.	.728	.740	.718	0.724
Aniline pt.	48.5	45.5	56	52
70/100°/20°	11.5/35	4.5/25	7/33	3/28
End point	174	172	173	175
% Aromatics	20	17	10.5	14.5
% Naphthenes	12	28	22.0	16.0
Iodine No. (Hanns 38)	5.8	4.4	-	-
Octane No. H/H+C ₁ 12 Pb	52.5/78.5	62.5/82	46/-	32/-
Middle-Oil				
Sp. Gr.	0.848	0.854	.832	.859
Aniline pt.	41	43.5	60.5	48
Boiling start/endpoint	187/333	192/336	191/350	193/350
50%	238	253	267	270
Cetane no.	38	34.5	48	42.5
Solidification pt.	-	-	-19	-25°
Viscosity 0E/20°	-	-	1.28	1.28
Residue:				
Sp. Gr. 120°			.917	.969
Viscosity E/50°			2.0	3.0
Flash Pt.			188°	194°
Solidification Pt.			+25°	+17°
Coke-test			0.17	0.65
Boiling Start/E.pt. Vac.			206/-325°	203/325-92.2
50% P.			-948	4050
			400°	

*Calc. total pressure if loss of vacuum is small.

Frame 318-337 (Contd.)

The yield of gasoline from the middle oils was 30-45% while the heavy oils yielded 10% gasoline, 40-45% middle oils, and 15-20% of new products and loss. The oils rich in hydrogen can be worked upon more easily than those poor in hydrogen.

The gasolines obtained have up to 20% aromatics, are saturated, but do not have a high octane number. The octane number of the products from heavy oils is from 46-52, and 52-63 for the gasoline from middle oils. However, the gasolines do not contain many naphthenes and are therefore less suitable for the Dhd procedure.

The middle oils are dehydrogenated to a greater extent. They are not very suitable for catalytic cracking, but, for gasoline production using catalyst 6434 they are very good. They produce twice as much gasoline as straight-run middle oil. The octane number of the 6434 gasolines is at least one to three units higher than the gasoline obtained from the same crude by normal hydrogenation. The octane number from the dehydrogenated middle oil was between 35 and 48, it is higher if heavy oil is used since as a general rule the dehydrogenation proceeds further.

The residue boiling above 325°C. has a specific gravity of 0.92-0.97 which is about 0.04 higher than the initial material. The coke test is low (0.17-0.65), however, the solidification temperature of the residue is +25°C. to +70°C. which is high.

The hydrogen consumption per ton of gasoline from hydrogen poor and hydrogen rich middle oil is 200-400 cubic meters per ton, and the hydrogen consumption per ton of gasoline plus middle oil obtained from heavy oil is 150-300 cubic meters. The gas formed is saturated and contains approximately 22 weight per cent methane, 26 weight per cent ethane, 15 weight per cent propane and 37 weight per cent butane. 22% of the butanes are isobutanes.

INFLUENCE OF THE DEGREE OF HYDROGENATION (A.P.
OF S-MIDDLE OIL) ON THE RESULTS OF PROCESSING
S-MIDDLE OIL FROM SCHOLVEN BY MEANS OF 8376/6434

Ref. No.:	M-42	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Guenther
Date:	9/28/42	Contents:	4 pages text
Frame:	338-347		4 tables
			5 figures

SUMMARY

I. Middle oil from Scholven was hydrogenated over catalyst 8376 at different temperatures until it had an average aniline point of 50, 47, 46, 39, and 32.

The B-middle oils were converted to gasoline over catalyst No. 6434.

II. The influence of the pre-hydrogenation temperature on aniline point of the B-middle oil, on the phenol content, acid number, and ease of conversion to gasoline, is as expected. The influence of the prehydrogenation temperature is very small, between 21 and 22.5 mv., and it is very large below 21 mv. It can be assumed that the maximum hydrogenation lies slightly above 22.5 mv.

III. The influence of the prehydrogenation temperatures on the qualities of the 8376 gasoline is much less than on the qualities of the middle oil.

IV. The aniline point of the B-middle oil has the following influence on the quality on the 6434 gasoline: with decreasing aniline point of the B-middle oil the aromatic content increases in the 6434 gasoline at the expense of the paraffin content. This has practically no influence on the octane of the 6434 gasoline, however, it has some influence on the lead sensitivity which decreases for a decrease of the aniline point of the B-middle oil.

V. The same can be said as under No. IV as to the quality of the finished gasoline (prepared from a mixture 8376 and 6434 gasoline.)

VI. The technically important range for the aniline point of the B-middle oil is between 50 and 46. In this range the ease of conversion to gasoline is very good to satisfactory. There is little relation between the composition and the basic octane number and the lead sensitivity of the gasoline to the aniline point of the middle oil. It can also be assumed that there is little relation between the aniline point of the B-middle oil (42 and lower) and the quality of the gasoline.

EXPERIMENTS IN THE AUTOCLAVE FOR THE
CONVERSION OF CaCO_3 (CAVIAR) INTO CaSO_4 DURING
THE HYDROGENATION OF BROWN-COAL FROM THE RHINELAND

Ref. No.:	N-43	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Grasel
Date:	9/28/42	Contents:	2 pages text
Frame:	348-349		1 table

SUMMARY

During the hydrogenation of Rhineland brown coal the calcium in the coal precipitates as calcium carbonate forming caviar-shaped particles. Since this is undesirable, an attempt was made to convert the calcium carbonate to calcium sulfate.

For this purpose, instead of the usual catalyst (3% Bayer mass) the coal was impregnated with 10.5% $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. The sulfate contained in this amount of FeSO_4 would be sufficient to combine with 80-85 per cent of the calcium oxide (CaO), present in the coal; the amount of iron in this catalyst is approximately twice as large as that contained in the Bayer mass.

Comparative experiments show that by using FeSO_4 the carbon dioxide content of the benzene insoluble part can be greatly reduced. As shown in the table, the CO_2 content of the benzene insoluble part can be reduced to less than half by using 10.5% FeSO_4 . By using Bayer mass as a catalyst or ferrous sulfate, there is always an excess of calcium oxide in the benzene insoluble part so that only a part of the Ca may be present as CaCO_3 . Using the Bayer mass about 77% of the calcium oxide is combined with CO_2 , but using iron sulfate only about 39% of the calcium oxide is present as CaCO_3 . In both cases practically all the calcium oxide introduced with the coal was recovered in the benzene insoluble portion.

The hydrogenation results may be approximately the same if an equivalent amount of iron in the Bayer mass and iron sulfate-catalyst is used. In the case here presented in which twice the amount of sulfate was used in order to bind as much calcium oxide as possible, a more favorable reduction of asphalt and a slightly higher gasification was observed. The cracking was practically the same.

According to these results it seems possible by use of sulfates to decrease the tendency of forming calcium carbonate.

NOTES ABOUT THE PRESENT STATE OF THE DEWAXING PLANT IN ZEITZ

Ref. No.: N-44
Origin: I.G. Farben Ludwigshafen
Date: 9/28/42
Frame: 350-358

Report No.: LU 558
Author: Hoelscher
Contents: 9 pages text

SUMMARY

There are two main difficulties at the present time in the dewaxing plant in Zeitz. First the filter efficiency is only 50% of the projected value.

Second, the quality of the paraffins was inferior when the T.T.H (see figure I) was followed closely and therefore it was necessary to change over to the MTH (see figure II) method. On the otherhand, this change caused a precipitation of lubricating oil which the RWM (Reich War Ministry) thought undesirable for production. The possible causes of these troubles and the possible means of prevention are described and summarized as follows:

(a) In Zeitz the following work was planned and started:

- (1) Construction of additional filters.
- (2) Addition of more wash containers.

(b) In Ludwigshafen is being investigated:

- (1) The relation of the thickness of the filter bed to the counter current solvent.
- (2) The influence of other oils in the counter solvent.
- (3) The refining in a propane solution by means of $AlCl_3$ or H_2SO_4 .
- (4) The refining of different products with H_2 .

(c) The following proposals are made for the Zeitz plant.

- (1) Increasing the rotation speed of the filter.
- (2) Improved cooling for the filter tank.
- (3) Increasing the counter current flow of solvent.
- (4) Processing for a short time according to diagram 3.
- (5) Either doubling the filter capacity or installing an H_2 -refining plant.

See Figure III

Frame 350-358 (Contd.)

FIGURE I

TTH Method:

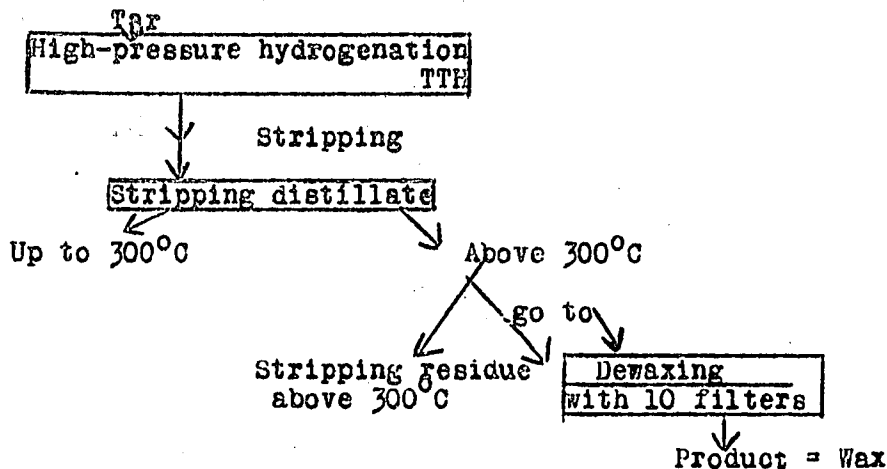
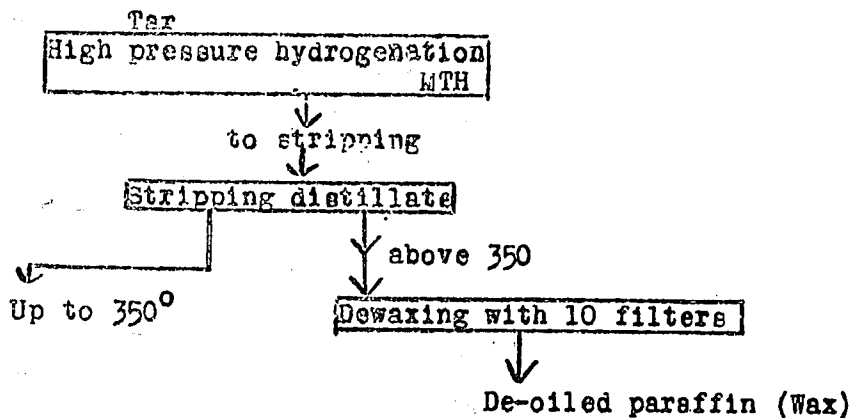


FIGURE II

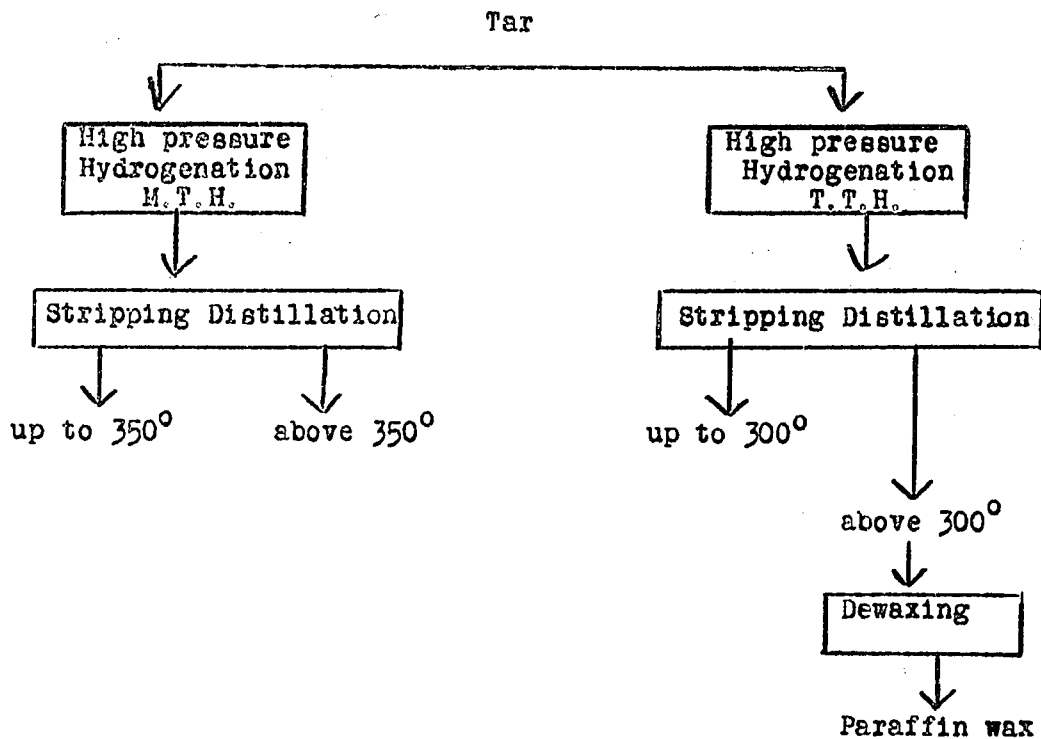
MTH Method:



Frame 350-358 (Contd.)

FIGURE III

PROPOSED PROCEDURE:



NEW TTH TESTS WITH CATALYST 5058 AND 7846

Ref. No.: N-45
Origin: I. G. Farben Ludwigshafen
Date: 9/24/42
Frame: 359-360

Report No.: LU 558
Author: Eisenhut
Contents: 1 page text
1 table

SUMMARY

The total product of each run was separated by fractionation into gasoline, middle oil, and a stripped residue. The residue was dewaxed in two steps by means of propane.

The dewaxing and deoiling was done by using tilting filters. The results are a little better than those obtained in the industrial process. The pure paraffins obtained by this method were sent to Oppau for oxidation. The result of this investigation are not available as yet, however, it is thought that the paraffins are technically useful.

It is to be noted that for both catalysts the absolute amount of paraffin is the same in both residues even though catalyst 7846 is a better cracking catalyst and a better hydrogenation catalyst. The paraffin obtained by catalyst 7846 has a slightly lower specific gravity (0.007) than that obtained with contact 5058. The first step in the deoiling of the product is easier if catalyst 7846 is used.

The oil obtained from catalyst 7846 represents a spindle oil while the dewaxed oil from catalyst 30 was a light machine oil.

It could not be determined if there was a difference between the gravities of the wax (paraffin) obtained by the two de-oiling steps (1) redissolving (2) mashing, because the amount of propane used for the two steps was not the same. 300% of propane was used for redissolving, while 200% of propane was used for de-oiling by mashing. The density of waxes obtained with the catalyst 7846 and deoiled by the two steps varied somewhat.

d_{70} = 0.770 by redissolving

d_{70} = 0.782 by mashing

Using the catalyst 5038 these differences were less.

EXTRACTIVE HYDROGENATION IN A 10 LITER FURNACE

Ref. No.:	N-46	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	V. Hartmann
Date:	9/18/42	Contents:	2 pages text
Frame:	361-365		2 tables
			2 figures

SUMMARY

In order to investigate the dependence of the ease of filtration of a product on the applied working conditions, extraction experiments were made in a 10 l. furnace. The experiments based on the known conditions of the so-called "Uhde extraction" could be reproduced in a 10 l. furnace. The results of these experiments led to the development of a new process which however has nothing to do with extraction itself and is therefore better named "extractive hydrogenation".

The results may be summarized as follows: A higher degree of hydrogenation may be favorable for the ease of filtration of the product if the working conditions are kept above those of the "Uhde-extraction", that means, higher pressure and higher temperature. The temperature has a considerable influence on the ease of filtration. The ease of filtration improves considerably by decreasing the temperature under otherwise the same conditions. The pressure has likewise a distinct but not as great an influence on the filtration. With rising pressure the ease of filtration is decreasing uniformly over the whole temperature-range. The through-put has a smaller and not as uniform influence. An increase of the through-put impairs the ease of filtration. The amount of gas, the recycling of the residual sludge, and the admixed oils have only a small influence. However processing without a catalyst shows a distinct improvement in the ease of filtration. The formation of middle oil increases as normal hydrogenation increases with rising pressure; but it decreases with increasing through-put, reducing the gas volume, and by eliminating the catalyst.

These relationships show that the ease of filtration is dependent on two counter-reacting effects. A hydrogenation of the primary decomposition products of the coal causing little or no cracking is detrimental for the ease of filtration. Such conditions are mainly caused by the catalyst and by higher pressure. Cracking hydrogenation, or the cracking itself, is caused by higher temperature and by longer contact time, however it is favorable for the ease of filtration. The most favorable working conditions are therefore obtained by compromising and reacting the coal under high pressure and high temperature and high through-put using no

Frame 361-365 (Contd.)

catalyst. However, by this method the recycle middle oil produced is insufficient and a small percentage of it has to be replenished from the coking of the residue (manufacturing of electrode carbon.) If this new method of producing middle oil is not desirable, then a catalyst has to be used at a lower through-put and in so doing sacrificing ca. 30% in the ease of filtration. It might be possible to obtain the same result by cracking without a hydrogenating catalyst and thereby not impair the ease of filtration.

Extraction itself as used by Stinnes cannot be applied in a 10 l. furnace. The conditions of flow in the 10 l. furnace at small through-put rates without gas and without recycling of the sludge cause deposits and thereby reduce the efficiency of the furnace.

Frame 366-382 (Contd.)

Especially favorable results were obtained with a gasoline marked 840 to 5058-Rheinbromgasoline. Compared with the usual 50-60 hours through-put time, the through-put time of the DHD period with this product was 120 hours, without starting the IMD-period. A technical yield of 85.6% was obtained. Due to these especially favorable circumstances, it was agreed with the RWM, that beginning September the whole production (approximately 3500 tons) of No. 5058-gasoline from Rheinbrom was to be issued in plant DHD; while the same amount of Rumanian or Hungarian gasoline was to be used for the production of automobile gasoline.

The qualities of the product are given in the attached tables and graphs. (not included in this summary).

2. DND-Experimental chamber 504:

In the 1 cbm. DHD-experimental apparatus Ka504 during the last month 18 high-power gasolines (aviation gasolines) (high efficiency gasoline up to 180° with a vapor pressure of 0.2) were produced from a gasoline marked Scholven 5058-6434 strippings. The desired vapor pressure could be obtained only by partial removal of the C₃ fraction by stabilization. As first produced the end point was somewhat low, and the fraction between 160-180 is very small. Therefore this test was repeated with a higher boiling gasoline; no results of this test are so far available. The first mentioned product in a mixture with 20% iso-octane has a good supercharge quality.

3. 700 atm.-gas phase experimental chamber 805.

In the experimental chamber 805 during the last month a first gas phase experiment has been made over the catalyst 7421 at 700 atm. The charging material was the middle oil obtained by liquefying coal from Scholven, which had been suspended in the DHD residue. With a contact volume of 770 liters the chamber was operated (September 21, 1942) first, with 600 atm. starting pressure, an injection rate of 200 cbm/hour, and a gas inlet temperature of 19 MV. The injection product consisted of 60% of a-middle oil and 40% of 6434 b-middle oil, the latter was replaced after the second day of operation by (our) own b-middle oil. The through-put was increased up to 620 kg. September 23, the temperature up to 23.5 MV, and the pressure up to 700 atm. The other data are summarized in a table and show, that with an increase of the average furnace temperature up to 24.5 MV, a very poor efficiency (0.325) was obtained. The phenol-number of the middle oil was quite low. On September 29 at 1:55 P.M. the temperature in the furnace increased in two minutes time up to 50 MV, accompanied with sudden collapsing of the tubes in the chamber. Although the furnace was flooded with cold gas, while the furnace was going back to normal temperature, a connection in the

Frame: 366-382 (Contd.)

charging line between regenerator and preheater broke, which put the chamber out of operation. Closer examination showed that (evidently by the sudden increase of temperature) a flange on the regenerator leaked and ignited the escaping gas, which caused the connecting line to break. After replacing the loose pipes in the regenerator, the chamber was repaired and was put into operation in approximately 8 days.

Parallel experiments in the small apparatus under the same conditions as used in the large scale apparatus, did not confirm the above mentioned low phenol reduction as well as the very low aniline points of the stripping product and b-middle oil. This result indicates a short circuit in the regenerator existed from the start which became noticeable when the furnace burned out and caused the sudden collapse of the chamber construction.

THE USE OF THE ALUMINUM Al_2O_3 -W-Ni-PREHYDROGENATION
CATALYST 8376-7846 W250 FOR VARIOUS PRODUCTS

Ref. No.:	N-48	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Guenther
Date:	9/10/42	Contents:	7 pages text
Frame:	383-394		5 tables

SUMMARY

I.

1. Middle-oil-a obtained from coal tar was hydrogenated at a pressure of 250 atm. over the catalyst No. 8376 at a temperature of 434 and 375°. The oil distinguishes itself from all other by a high content of oxygen (9%) in the gas phase and by a high content of acid-compounds (40%) in the liquid phase. Furthermore, the oil has a high iodine number (124) and a high aniline point (+5.5°C). The hydrogen content of the oil however is lower than could be expected from the aniline point; it is even lower than that of the middle oil aniline point (-17°C) obtained by the liquefaction of coal.

2. The extraordinary properties of this coal-tar-middle-oil when compared with other middle oils from coal products, also is observed if hydrogenated.

3. The prehydrogenation is characterized by:

- A. A high yield of gasoline (30%, mostly from oxygen and hydrogen compounds) of a high paraffin content (18%) and of low octane numbers (66-84.5)
- B. By extraordinarily high aniline point of the B-middle oils (+61°C) and relatively high acid number (10.8 and 74.4).

4. For producing gasoline it is surprising that:

- A. The B-middle oils in spite of their high acid-numbers give good yields.
- B. The 6434-gasolines in spite of their high paraffin content, have surprisingly high octane numbers.

5. The so-called "gasphase gasolines" obtained by mixing 8376 and 6434 gasolines show a little lower boiling point (15%-100°C) due to the high percent of 8376 gasoline (35%) and the extraordinary qualities of this gasoline.

Frame 383-394 Contd.

II.

1. Distillate obtained by cracking coal-tar, under pressure and containing 70% of a fraction boiling lower than 350°C. 5% of asphalt, 0.35% of solids and 0.06% of ashes, was hydrogenated at 600 atm. using a catalyst 8376 which was arranged in a stationary location in the reactor. The product was very similar to the coal tar-a-middle oil but contained a higher boiling fraction. The product contained 92% of a fraction boiling lower than 330°C. which was very similar to the 8376-b-product obtained from a-middle oil; it also shows good results if reacted over catalyst 6434.
2. Diesel-oils could be made from cuts of the products obtained by reacting the distillates from pressure-cracking, over the catalyst 8376 at 600 atm. The diesel-oils have a cetane-number of 45 and 48 and a solidifying point of -39 and -17°.

THE STUDY OF THE HYDROGENATION OF SILESIA COAL K1197

Ref. No.: N-50
Origin: Griesheim I. G. Central Library
Date: 10/24/42
Frame: 395-396

Report No.: 515/III-B-5
Author: Hartmann
Content: 2 pages text

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*Page number and tables from article; Processing of Coal for Gasoline, Middle-Oil and Heavy Oils: Frame 397-508

PROCESSING OF COAL FOR GASOLINE, MIDDLE OIL AND HEAVY OILS.
PROCESSING OF SILESIAN COAL "K1197" FROM THE "HEINITZ MINE" IN
A 10 LITER FURNACE AT 600 ATM. PRODUCING AN EXCESS OF HEAVY OIL

PART NO. I

Ref. No.:	N-51	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	V. Hartmann
Date:	10/24/42	Contents:	53 pages text
Frame:	397-508		22 tables
			5 figures

SUMMARY

Experiments are made with Silesian coal in a 10 liter furnace in order to obtain fundamental knowledge about the possibility either to manufacture an excess of heavy oils (gasoline + fuel oil), or on the other hand gasoline, middle-and heavy-oils. Especially a new process was tested which takes a middle position between the preparation of gasoline and middle-oil and the previous procedure of gasoline and fuel oil. This new method is, especially applicable for coal, containing asphalts which are difficult to crack. Due to low cost the process is technically more desirable than the process producing a maximum of heavy oils.

As expected from the analysis as shown in the preparation of gasoline and middle-oil, the coal from the Heinitz-mine was inferior to coal from Gelsenberg. With an efficiency higher than 0.4 and a formation of gases of less than 22%, the quality of the Heinitz-coal is still sufficient, for processing a maximum of heavy oils with sufficient asphalt-cracking. (Group of experiments No. Ey).

During the whole series of experiments which extended over a period of more than 8 months, the following fundamental problems were also investigated:

1. The influence of through-put and temperature on the processing of such an inferior coal for producing an excess of heavy oils. (Group of experiments No. Ex-Ey.) The main result was, that more vigorous conditions had to be applied (less through-put at high temperature) in order to crack the asphalts to such a degree, that they would not cause any difficulties in processing the residue.
2. Influence of the amount of gases. (Group of experiments B_{IX} AND B_X) the decrease of the amount of gases formed

Frame 397-508 (Contd.)

essentially influences only the cracking of the asphalt.

3. The influence of the separator temperature (Group of experiments B_{VIII}.) It is of no importance in the present case.
4. Catalytic additives.
 - A. Sulfurizing of the "Bayer-Matter" (Group of experiments B_{VII}). The catalytic-effect of the sulfurizing of the Bayer-mass is extremely small and therefore scarcely justifies its cost.
 - B. Addition of H₂S (Experiment B_{VII}) By adding 5% of H₂S no effect could be seen.
 - C. Addition of chlorine (Group of experiments No. A_{XI}) Adding chlorine as CCl₄ into the acid-section at the bottom of the furnace did not show any effect on the production of heavy oils at 600 atm.

Frame 509-510 (Contd.)

In order to increase the caloric efficiency the following improvements can be made:

1. The installation of a Linde-equipment in which the residual water-gas and H_2 are separated (H_2 -39%). The efficiency rises from 37.2 to 42.6%.
2. The utilization of the carbonization residue increases the efficiency from 40.6 to 44.3%.
3. The hot steam condensers for the utilization of the heat exchange in the sludge and product increases the efficiency from 41.3 to 43.2%.

EXPERIMENTS WITH H₂-CO₂ MIXTURES UNDER DHD CONDITIONS

Ref. No.:	N-52	Report No.:	LU 558
Origin:	I. G. Farben Ludwigshafen	Author:	Nonnenmacher and Donath
Date:	3/6/42	Contents:	6 pages text 2 pages tables 1 page figures
Frame:	511-519		

SUMMARY

Experiments were made with a mixture consisting of 95% H₂ and 5% CO₂ at a pressure of 25 atm. in a 1000 cc. furnace containing built-in DHD catalyst. The heavy gasoline 6719/6434 was sprayed first into the CO₂-H₂-gas mixture and then into the CO₂ free hydrogen. At a temperature of 510°C. and a gas rate of 200 liters per hour, about 26.5% of the CO₂ was reduced to CO and about 36.4% to CH₄. A noticeable decrease in the dehydrogenation activity occurs only while operating with an H₂-CO₂ mixture; after changing back to H₂ the catalyst regains its original activity.

From these results a few conclusions may be drawn concerning a reduction in the off-stream time for regenerating and return to operating conditions in technical DHD plants. (DHD-standard-chambers). If during the off-stream period the pressure of the regeneration gas is reduced to about 10 atm., and if the chamber pressure is quickly restored to its original pressure with hydrogen, the CO₂ is quickly and completely converted to CH₄.

The hydrogen consumed is about 400 cbm. per hour; the heat developed in this reaction is 400 x 400 = 160,000 cal. If the hydrogen is distributed equally over the 5 furnaces and if cold gas is fed to the last four furnaces, then the reduction of the CO₂ may be completed in half an hour. It should be considered, whether it would be more expedient to remove at least part of the CO₂ by injecting an aqueous solution of Na₂ CO₃ before restoring the pressure in the chamber. By using an injection of 3 cbm. of solution per hour the total amount of CO₂ can be removed in approximately 3 hours.

PROPERTIES OF SOME RUMANIAN OILS

Ref. No.:	N-53	Report No.:	(Not given)
Origin:	Corporation for Petroleum Production and Processing in Germany	Author:	" "
Frame:	520-550	Contents:	33 tables

SUMMARY

The report contains the following tables:

Survey of the analyses of the oils from Grosny.

General analysis (physical properties, etc.) of: paraffin-free oils from the Old-Grosny territory and the New-Grosny territory.

General analysis of heavy oils, light oils and middle oils from different parts of Rumania.

BID FOR A DEASPHALTING INSTALLATION WITH
A CAPACITY OF 50,000 TONS OF VACUUM RESIDUE PER YEAR

Ref. No.: N-53
Origin: I. G. Ludwigshafen
Date: 3/4/42
Frame: 551-559

Report No.: LP 109 Dr/Bl & Dr/Koe
Author: Not given
Content: 9 pages text

SUMMARY

A plant for removing asphalt and resins from 50,000 tons of vacuum residue is discussed. At 40°C. the asphalt is extracted with 650% of propane, and at 80°C. the resins are extracted with 800% of propane. The installation can be supercharged 10% and the solvent is recovered separately from the asphalt and resin extractions. A complete list of the different apparatus to be found in this plant are given under the following headlines:

- I. Columns and containers.
- II. Preheaters, heat-exchangers and condensers.
- III. Compressors and pumps.
- IV. Pipelines, fittings and accessories.
- V. Complete regulation-installation.
- VI. Complete measuring equipment.
- VII. Other types of installation.
- VIII. Insulation.
- IX. Iron construction and scaffolding for equipment.
- X. Electrical installations.

COMPARISON OF THE MANUFACTURING OF GASOLINE USING
NATURAL BLEACHING EARTH (FULLER'S EARTH) (6109) AND
SYNTHETIC ALUMINUM SILICATE (6752) AT 600 ATM. AS CATALYSTS

(5th Communication)

Ref. No.:	N-54	Report No.:	High pressure tests
Origin:	I. G. Ludwigshafen		LU 558
Date:	3/2/42	Author:	Rotter
Frame:	560-564	Content:	2 pages text 2 pages tables 2 pages figures

SUMMARY

As reported in the 4th communication (2/24/42, No. 199921), natural bleaching earth treated with HF is distinguished from all other cracking catalysts containing no additives by its efficiency and by the octane numbers of the gasoline cuts.

In this report a comparison is made of this catalyst with synthetic al-silicate (K-6752) for cracking b-middle oil from coal liquefaction and Bruchsal gas oil.

The experiments were typical runs made under conditions used for the preparation of gasoline (without regenerating the catalyst). The results are compared with those obtained using catalyst 6434 at 250 atm. using the same charging stocks in tables and figures that are not included in this summary.

The tables and figures show the following:

1. The maximum efficiency of catalyst 6109 at 600 atm. is considerably higher than that of catalyst 6752. However, at 250 atm. the efficiency of 6109 is only half that of catalyst 6434 in spite of higher temperatures of operation.
2. The C_{11} production and the $i-C_{11}$ content in the total $-C_{11}$ fraction is of about the same magnitude as that of 6434 and are much higher than from 6752.
3. The octane numbers of the gasoline fraction distilling below $150^{\circ}C$. are approximately the same for the three catalysts, although the aromatic content is somewhat higher for the synthetic catalyst.
4. The octane number-curves of the gasoline fractions from coal

Frame: 560-564 (Contd.)

liquefaction using catalysts 6109 and 6758 are almost superimposeable in spite of a much higher content of aromatics and a higher iodine number for the fractions obtained with catalyst 6752. The octane number-curve of the gasoline fractions from Bruchsal gas oil using catalyst 6109 in spite of a lower content of aromatics lies above that of the fractions obtained with catalyst 6752.

5. The octane number of the residual gasoline (Bruchsal gas oil boiling range 140-150°C.) using catalysts 6434 (at 250 atm.) and 6752 (at 600 atm.) is approximately 29.0; if catalyst 6109 is used (at 600 atm.), the octane number of the residual gasoline is about 35.5.
6. The comparison of catalyst 6752 with 6434 and 6109 shows that, apart from its poor cracking efficiency under gasoline manufacturing conditions, it has more disadvantages than advantages.

FURTHER EXPERIMENTS IN THE
FIELD OF ALUMINUM-Mo-Ni-CATALYSTS

Ref. No.:	N-55	Report No.:	LU 558
Origin:	I. G. Ludwigshafen	Author:	Guenther
Date:	2/4/41	Content:	7 pages text
Frame:	565-572		4 tables
			1 figure

SUMMARY

1. Starting with catalyst 7846, the ratio of Mo:Ni as well as the absolute quantities of Mo and Ni were varied. The result was, that a better catalyst than 7846 could not be found.
2. The preparation of 7846 could be simplified, without impairing the activity of the catalyst.
3. Intermediate and waste products obtained from Dr. Stoewener in Oppau can also be used for preparing 7846.
4. Catalysts, just as good as 7846, could be prepared from several other aluminum compounds (activated Giulini-earth and some aluminum earths prepared under high pressure).
5. Catalyst carriers prepared by activation of bauxites could not completely replace the active aluminum prepared by Dr. Stoewener for the 7846 catalyst.
6. The production of 7846 was continuously tested. All tests were satisfactory.
7. The catalyst 7846 recovered from a furnace (ka 12 furnace II) in Beholven on December 12, 1940, had practically the same activity as freshly prepared catalyst 7846.

Regeneration of the catalyst did not change its activity.

PRODUCTION OF LIGHT GASOLINE

Ref. No.:	N-56	Report No.:	LU 558
Origin:	I. G. Ludwigshafen	Author:	Reitz
Date:	2/1/41	Contents:	2 pages text
Frame:	573-582		1 table
			8 figures

SUMMARY

Experiments were made in a $\frac{1}{2}$ l. furnace at 200°C and 600 atm. with gas oil and prehydrogenated middle oil from anthracite and brown coal (Schelven and Meresburg). The total material of these tests is still being edited and is given here only in extract form.

A. Pressure of runs. (600 and 200 atm.) The values for gasification, obtained at 600 and 200 atm., are plotted in the Graph #1 giving the boiling point curve of the product boiling above gasoline. None of the graphs have been included in this summary. It is shown that for middle oil from anthracite and brown coal and for gasoline of end point at ca 100°C, the gasification at 600 atm. pressure is about 6% lower than at 200 atm. With a gasoline of endpoint of 150° and 20% of gasoline (b.p. up to 70°C) the difference is only about 3%. Since these experiments showed up to 10% of loss, the results must be verified by further experiments. The following table contains average data for gasoline production from brown coal middle oil. The gasoline contains 45% of components boiling point up to 70° and has an approximate endpoint of 100°C.

TABLE

Pressure (Atm.)	600	200	
Gasoline efficiency	0.3	0.4	0.3
Gasoline Concentration	22	29	23
Temp. M.V.	18.5	19	19.5
% Gasification/gasoline+gasification	26		32
%C ₄ in the gasification	84		56 ?
% Isom. C ₄ in total C ₄	74		72
% C ₅ in stab. gasoline	ca 32		ca 28
% Isom. C ₅ in C ₅ of the gasoline	89		87

Frame 573-582 (Contd.)

As said before, the advantages of the run at 600 atm. have to be verified by further experiments.

In the Graphs No. 2 and 3, the knocking values of different fractions are plotted. It is shown that fractions to be compared at 600 and 200 atm. are practically identical. Therefore, the gasoline quality does not offer a possibility of deciding the question of running at 600 or 200 atm. in the production of light gasoline.

B. Comparison of several crude materials. The data of the octane numbers of Graphs No. 2 and 3 are plotted on a larger scale on Graph #4, for gas oil and prehydrogenated middle oil from anthracite and brown coal. The octane numbers of these products without lead are practically the same, while that of petroleum is lower. With the addition of lead the octane numbers of the anthracite products are apparently somewhat higher than those of brown coal. This fact does not apply in all cases to the octane numbers of the total light gasoline, which are almost identical for anthracite and brown coal. The curve for the octane numbers of light gasoline from gas oil lies under the curve for the products from anthracite and is similar to the curve from brown coal.

THE U.S.A. MILITARY ISSUE SPECIFICATIONS FOR AVIATION GAS

Ref. No.:	N-57	Report No.:	LU-558
Origin:	I.G.Ludwigshafen	Author:	N. P. N. Bureau
Date:	1/31/41	Translator:	Henkels
Frame:	583-586	Contents:	4 pages text

SUMMARY

This article is a translation from the National Petroleum News 10-30-40.

Newly revised specifications for Army-Navy aviation gasoline, covering four grades: 100, 91, 73, and 65 octane number fuels have been made public. The new specifications, AN-VV-F-781, -776, -761, and -756 supersede Army-Navy specifications AN-9530, AN-9527 and AN-9526 which were not disclosed to the general public. The octane rating is determined by knock test method No. AN-VV-F-746, which is known as the Aviation Fuels Division method or simply the AFD method, and is not confined to Army-Navy use.

SILESIA-GASOLINE DEPHENOLIZATION.
REASONS FOR CHOOSING THE PHENOSOLVAN PROCESS.

Ref. No.:	N-58	Report No.:	Not given
Origin:	I.G. Ludwigshafen	Author:	Greif & Kroenig
Date:	Not given	Contents:	3 pages text
Frame:	587-589		

SUMMARY (Abstracted)

The waste-waters of the Blechamer plant showed a rather high phenol content (ca 10 g. crude-phenol/liter) which made a removal of the phenols necessary before disposal of the water into the river.

All the known dephenolization methods were therefore carefully tested. The so-called Phenosolvan-method which was developed by the I.G. and Lurgi Co. was found to be the best. The cost of this process for purifying lm^3 of waste water is 1.50 Mark, but the price of recovered phenols is higher, and is therefore still a profitable undertaking.

The process is based on the extraction of the waste water, (which is first freed from H_2S by treating with CO_2) with a mixture of amyl- and butyl-acetate of a boiling point range from $110-130^\circ$. This solvent mixture is only slightly soluble in water but dissolves phenols very readily. The loss of solvent per 1 ton of solvent is less than 200 g. per lm^3 waste water.

The special reasons for the preference of this method are as follows:

1. The procedure is very simple and easy to control.
2. The amount of solvent for recycling is very small. Due to the high solvent capacity of the Phenosolvan only 10% of the solvent calculated for the waste water to be treated is necessary. Other extraction solvents require amounts from 100-200% of the waste water. This point was very important for the protection of the plant against air raids, because large recycling amounts of ignitable solvents represent a great fire hazard.
3. In the "Tri-Process", in which the solvent is recovered as a distillation residue, the solvent is easily contaminated. In the Phenolsolvan method however the solvent is distilled after each extraction and no contamination is possible.

Frame: 587-589 (Contd.)

4. All side installations, necessary for the regeneration of the sodium hydroxide used in other methods, are unnecessary for this Phenosolvan process. This point is very essential for the plant in Blechhammer, since the total plant requirement of caustic is so small, that even if all phenols would be extracted by the caustic it would not be profitable to use a method using caustic in a continuous process.
5. Rather large amounts of phenols can be extracted by the Phenosolvan-process from middle oils without additional cost. The oil fraction is contacted with the phenol containing water. By this contact an artificial enrichening of the water with valuable phenols is achieved and likewise an exchange of higher phenols with lower and more valuable ones is made possible.
6. The purifying efficiency of the Phenosolvan is very great. The phenol content of the treated water is about 200 mg/liter, whereas by other dephenolization methods, for instance using steam, about 1000 mg. phenols remain in the waste water. Furthermore all other types of oily and other impurities are efficiently removed which causes the lowering of the permanganate number of the water. This lowers likewise the cost of the following biological purification of the waste water considerably. In order to dispose the water finally into the river, all traces of phenols, left in the water, have to be decomposed in a biological way. The smaller the amount of phenols to be disintegrated, the cheaper and the simpler is this additional purification.
7. As already pointed out, the Phenosolvan removes also oily-and other contamination, which are also quite useful.
8. The Phenosolvan-method is a very economical process.

PROSPECTS FOR FUELS WITH HIGHER OCTANE-
NUMBERS IN THE FIELD OF COMMUNICATIONS.

Ref. No.:	N-59	Report No.:	LU 558
Origin:	I. G. Ludwigshafen	Author:	Hubner
Date:	6/30/41	Contents:	32 pages text
Frame:	590-623		17 tables
			2 figures

SUMMARY

The article is a translation of a paper given by W. H. Hubner at a meeting of the Aeronautical Engineers on Jan. 25, 1940 in New York (Journ. Aeronautical Sciences I, Nr. 8, 319).

The technical development of fuel oils with high octane numbers is progressing so rapidly, that it is difficult to follow all these changing conditions and to make an intelligent plan for the proper and economic usage of these developments. Besides different types of fuels with high octane numbers and of customary volatility there is now a possibility for the usage of the so-called safety fuel oils. It is mostly an economic question on how to decide, how and how much some of these fuels may be technically used.

The purpose of this meeting is therefore to give a survey of the methods, which now are used or are being developed for the manufacturing of such fuels, to give an estimate of the quantities of fuels which are or soon will be at hand, and to report the opinions, which were made about the fuel problem. All statements were either taken from the technical literature or were obtained from private communications of aviation fuel technicians, who took part at this conference. The paper is discussing the following subjects:-

- A. Fuels of High Volatility
 1. Basic Gasolines
 - a. Straight run gasoline
 - b. Natural gasoline
 - c. Catalytically cracked and reformed gasoline
 - d. Hydrogenated gasoline
 2. Mixing Components
 - a. Isopentane
 - b. Isooctane
 - c. Alkylate
 - d. Aromatics

Frame: 590-623 (Contd.)

3. Anti-knock compounds
 - a. Lead tetraethyl
 - b. Aniline
 - c. Water

- B. Safety Fuels
 1. Aromatic

 2. Paraffinic

- C. Cost and Available Amounts of Finished Aviation Gasoline of Octane Numbers 95-100.

RECENT TESTS IN PRELIMINARY HYDROGENATION FOR THE LIQUEFICATION OF ANTHRACITE AND BROWN COAL, USING A Mo-CONTAINING CATALYST IN A ONE LITER FURNACE.

Ref. No.:	N-60	Report No.:	LU 558
Origin:	I.G. Ludwigshafen	Author:	Reitz
Date:	1/27/41	Content:	17 pages text
Frame:	624-646		5 tables
			1 figure

SUMMARY

A series of Mo-containing catalysts (partly in combination with catalyst 5058 or other W-containing catalysts) were tested at Scholven for preliminary hydrogenation of middle oil from anthracite liquefaction, and at Leuna for the liquefaction of brown coal. The attached table gives information about the prehydrogenation conditions and the possibility of preparing gasoline from B-middle oil. Data on quantities of metals necessary for 1 cbm. prehydrogenation contact space are also given.

The best results up to date were obtained with an aluminum Mo-Ni-catalyst in combination with 20 Vol. % of catalyst 5058 which was added before the first mentioned catalyst.

The qualities of the prehydrogenation-gasoline and of the 6434 gasoline are not influenced by the catalyst used.

A great dependency of pressure upon the hydrogenation reaction was noticed with Mo-containing catalysts, so that e.g. for the prehydrogenation of brown coal at 190 atm. of H₂ pressure catalyst 5058 had to be diluted.

In order to use diluted prehydrogenation catalysts more easily, and also to use such catalysts with weaker refining and hydrogenation qualities, it is strongly recommended that the prehydrogenation middle oil be washed with diluted sulfuric acid in order to remove the amines.

Frame: 624-646 (Contd.)

TABLE

Catalyst or Combination of Catalysts	Kg. Metal per Cbm. Catalyst Space				Anthracite Lique- fication Middle Oil (Scholven)				Brown Coal Lique- fication (Gasoline Middle Oil) Leuna			
	W	Mo	Ni	Fe	In. Atm.	Temp. OC.	Thru- put	*	In. Atm.	Temp. OC.	Thru- put	*
	50% 7360 (active aluminum + Mo) + 50% 7745 (Fe, Ni, W, Al ₂ O ₃)	202	14	23	324	250	442	0.8	good			
80% 7360 + 20% 5058	550	22	-	-	250	442	1.0	good				
80% 7424 (active aluminum + 10 Mo) + 20% 5058	335	37	-	-	250	434	1.0	good	200	434	1.0	bad
7846 (active al- uminum 10 Mo + 3 Ni	-	56	20	-	250	434	1.0	very good	200 190	434 434	1.0 1.7	good very bad
20% 5058 + 80% 7846	388	45	16	-	230	434	1.0	very good	190 190	434 434	0.7 1.0	very good suffi- cient

* Suitability for manufacturing of gasoline

COMPARISON OF DEHYDROGENATION CATALYSTS 7911
(LEUNA) AND 7360 CH.6 (LUDWIGSHAFEN) AT 10 AND 25 ATM.

(PART 2)

Ref. No.:	N-61	Report No.:	LU 558
Origin:	I.G. Ludwigshafen	Author:	Nonnenmacher & Donath
Date:	1/22/41	Contents:	6 pages text 1 table 6 figures
Frame:	647-658		

SUMMARY

In comparative tests of the active dehydrogenation catalysts 7911 (Leuna) and 7360 Ch-6 (Ludwigshafen) at graded temperature but otherwise under the same conditions as with the first series of tests (see report No. 1, 12/7/40), the two catalysts show the same activity and the same yield of aromatics.

Decreasing the furnace temperature causes a decrease of the yield for both catalysts. At a temperature drop from 37-26°C the yield decreases 1-2%, at a temperature drop from 50-40°C however the yield decreases 4-5%. The yields obtained in a 100 liter furnace and in chamber 304 using technical catalyst are about 3% smaller than those obtained in the 1 liter furnace using catalyst 7360 Ch.6 or 7911 at a much lower temperature.

CONCERNING THE PROBLEM OF OVERLOADING CURVES.
THE LIMIT OF ERROR IN THE DETERMINATION OF OVERLOADING CURVES

Ref. No.: N-62
Origin: I. G. Ludwigshafen
Date: 1/20/41
Frame: 659-662

Report No.: LU 558
Author: Fromherz
Content: 3 pages text
1 figure

SUMMARY

1. Theoretical Considerations. According to the report from 1/9/41: "Methods for the Approximate Calculation of Overloading Curves from the Octane Number of the Residual Gasoline from the Aromatic Content", the error in the determination is caused by 3 almost equal factors.

- 1) Fluctuation of the octane number of the residual gasoline of ± 2 units causes ca. ± 0.5 in the total error.
- 2) Fluctuation of the aromatic contents of $\pm 2\%$ causes likewise a difference of ± 0.5 in the total error.
- 3) Inaccuracy in the measurement of the gas volumes up to $\pm 5\%$ results in an error of $\pm 0.05 \lambda$ (the excess of air) which again causes an error of ± 0.5 in the total error.

The total maximum error therefore could be ± 1.5 and the probable error would be ± 1 .

If for a series of single determinations for plotting an overloading curve the same gasoline is used, then only the source of errors No. 3 has to be considered, while the errors caused by No. 1 and No. 2 are of a systematical nature, which are the same throughout the whole series of determinations.

2. Experimental Confirmation. In this connection it is stated that such comparative measurements of whole series with the same gasoline on different days are very interesting. Details are given in a report No. 1274 of 12/23/40 by Prof. Wilke, and in another report of the D. V. I. (German Society of Engineers) from 12/19/40 which is referred to by Prof. Wilke. These reports are not summarized here.

Frame 659-662 (Contd.)

CONCLUSION

As already pointed out in a previous report, the experimental data show the following conclusion.

Evaluation of differences of ± 1 units in the overloading curves leads to a discussion of the limits of error.

Something conclusive can be said of such differences only if the same effect occurs in 3-5 independent overloading curve-determinations.

PROPYLATION OF ANTHRACENE FOR THE PRODUCTION
OF OILS WITH DRYING PROPERTIES, ACCORDING TO DR. VIERLING

Ref. No.:	N-63	Report No.:	LU 558
Origin:	I. G. Ludwigshafen	Author:	Bueren, Christmann, Conath
Date:	5/6/41	Contents:	2 pages text
Frame:	663-664		

SUMMARY

According to the reports of Dr. Vierling anthracene can be alkylated with propylene, $AlCl_3$, as catalyst, giving oils which dry in remarkably short time.

Experiments were performed to find the best conditions for the reaction. Sublimed technical anthracene was alkylated with propylene in the molar ratio of 1:2, 1:3, and 1:4 using aluminum chloride to the extent of 5% of the weight of anthracene at a temperature of 160-180°C. for 2-3 hours. Comparative experiments using o-dichlorobenzene as solvent showed that the reaction goes to completion only when no solvent is used. The reaction product has the consistency of a high viscous cylinder oil. In order to remove the catalyst it was dissolved in benzene and distilled at high vacuum in a nitrogen atmosphere. The distillates are very viscous, yellowish-brown clear oils which dry completely in 3-4 days when applied to a glass plate and exposed to air at room temperature. Dr. Vierling claimed drying periods of only 15-30 hours. These experiments yielded oils with longer drying periods probably because stabilizers to prevent polymerization during distillation were not added.

Dr. Christmann made similar experiments causing the condensation of anthracene with propylchloride (molar ratio 1:2, 1:3, and 1:4) using carbon disulfide, dichlorobenzene and ethylchloride as solvent at 20-70°C. The reaction product had good drying properties (12-48 hrs.), however they could not be purified by distillation because of decomposition and excessive foaming. This difficulty might be prevented by using polymerization preventing stabilizers. Alkylation products from the condensation of anthracene with higher olefins (Michael gasoline) can be distilled without difficulty and also have drying properties.

OCTANE NUMBERS OF SOME HYDROCARBONS

Ref. No.:	N-64	Report No.:	LU 558
Origin:	I.G. Ludwigshafen	Author:	
Date:	4/29/41	Contents:	2 tables
Frame:	665-670		4 pages figures

SUMMARY

The tables contain:-

Octane numbers of hydrocarbons: paraffins, olefins, diolefins, naphthenes, aromatics.

They were taken from a report by Schmittenberg, Hoog, Moerbeck, and Zijden of the Journal of the Institute of Petroleum 26, 294 (1940).

HYDROGENATION OF SILESIAN COAL "K1197" OF THE HEINITZ MINE
FOR GASOLINE AND MIDDLE OIL AT 600 ATM. IN A 10 LITER FURNACE

Ref. No.:	N-65	Report No.:	LU 558
Origin:	I.G. Ludwigshafen	Author:	Hupfer & Kroenig
Date:	3/9/42	Contents:	50 pages text
Frame:	671-761		2 figures
			23 tables

SUMMARY

Older Silesian anthracites have given poor results on hydrogenation for the manufacturing of gasoline and middle oil. For this reason younger coals from Blechhammer, from the Beuthen-mine (K1180) and especially from the Heinitz mine were tested. In a table 2 (not given in this summary) the properties of the Heinitz mine coal are compared with those from Beuthen (K1180) and of those from Gelsenberg (Ruhr K1090). The Heinitz mine coal is very much like the coal from Beuthen (K1180). It differs from it by its higher oxygen content, by somewhat less volatile components on combustion, by less tars and less carbon. For these reasons somewhat poorer hydrogenation results were to be expected.

The Ruhr coal (K1090) differs disadvantageously from the Silesian coal by higher oxygen content and higher alkalinity of the ash, besides containing small percentages of S, Cl and H₂ and by small yields of combustion tar. It was to be expected that these unfavorable qualities could not be compensated for by the low C-content.

The coal could be easily processed in a 10 liter furnace using the customary catalysts; iron sulfate, sulphurized Bayer-mass, sodium sulfide at various rates and temperatures. The results with high through puts can be seen in the following table:-

TABLE 1

Coal	1197		For Comparison	
	1180	1090	1180	1090
Furnace Temp. °C	471	473	474	468
HC-throughput	0.57	0.57	0.57	0.44
Decomposition %	94.0	95.7	95.9	96.1
Bi+Ni-efficiency	.31	.32	.32	.27
Gasification	26.8	27.7	26.6	24.7
Asphalt per mole				
H. C. %	7.8	7.6	7.5	7.4
Asphalt in the				
admixed oil %	7.5	7.8	6.7	6.7

Frame: 671-761 (Contd.)

About the hydrogenation qualities of coal 1197, the following can be said:

1. At 600 atm. the coal gives a high yield which at 0.31 is approximately the same as coal 1180. The maximum yield may be obtained in the neighborhood of 0.57 without impairing the chemical results.

Decreasing the throughput to 0.48 causes a lowering of the efficiency from 0.24-25 depending upon the temperature used without changing the yield of the asphalt--and of the gaseous products, except the decomposition increases ca. 1% with decreasing throughput.

2. In the range of 471-474°C the furnace temperature has little influence on the efficiency and on the asphalt decomposition. This is a common quality for both kinds of coal (1197 and K1180). However decomposition of coal (1197) seems to be influenced to a greater extent by the temperature. Also the volatility of coal 1197, which is by itself unusually high, rises rapidly with the temperature--more than with coal 1180--and at 474°C reaches an extraordinarily unfavorable value. The optimum temperature of gasification seem to lie in a very narrow range, near 471°C. The oils obtained at different temperatures had practically the same qualities. With rising temperatures the methane content increases in the gaseous products.
3. Coal 1197 greatly resembles Silesian coal K1180 in decomposition and efficiency. However the heavy oils obtained from coal 1197 contained less oxygen. The gaseous products of 1197 contained more C₃-hydrocarbons and less butane, however more of the butanes seemed to be isobutane.
4. The coal 1197 (Silesian), in spite of its lower C-content, requires a higher reaction temperature than Ruhr-coal, in order to achieve approximately the same decomposition of the solids and of the asphalts. The higher temperature also causes a little higher efficiency. The results of the gasification however were unfavorable.

It can be summarized that the coal 1197 is still useful for hydrogenation, however it is inferior to coal 1180 in regard to the volatility.

Frame: 671-761 (Contd.)

The catalyst experiments gave the following results:

1. Non sulphurized Bayer-mass differs from the sulphurized Bayer-mass by possessing a greater decomposing quality and a smaller hydrogenation quality. The two catalysts differ so slightly that results could not be clearly established.
2. Soaking the coal with sodium sulfide instead of adding it with the oil seems to give favorable hydrogenation results. Further experiments are necessary.
3. Sulphurized Bayer-mass, soaked with titanium amyl sulfate is much less favorable from the standpoint of decomposition and volatility than the catalyst itself without adding titanium.

The following results were observed concerning the processing apparatus:

- 1.
2. The results with different 10 liter furnaces are comparable.
3. There are indications that fast-stirring may aid decomposition.