FILM STUDY GROUP

SUBJECT INDEX AND REPORT

T.O.M. REEL NO. 86

Prepared by

SOCONY VACUUM OIL COMPANY

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

Review of Microfilm Reel #86
U. S. Government Technical Oil Mission
I. G. Farbenindustrie  -  Leuna

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Technical Oil Mission Microfilm Reel No. 85


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I. G. Farbenindustrie - Leuna

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Description of Individual Items (#28-31, 53-110)


28-31          4p          frames 1002-1005     various dates

Drawings of the Synthetic Lube Oil Installation

These are the balance of the prints discussed in Reel #85, items 28-52.

| Item | Drawing # | Title | Frames |
|------|-----------|-------|--------|
| 28 | M-3052-2 | Flowsheets of Entire Plant | 1005 |
| 29 | M-4412-2 | Plot Plan | 1004 |
| 30 | M-3977-2 | Ethane Cracking Furnace | 1003 |
| 31 | M-1640-1 | Ethane Cracking Installation Plan | 1002 |

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53          13p          frames 1007-1019     4/21/42

Report on the Carbonization of Metal
Tubings in the Dehydrogenation of
n-Butane and Isobutane

The deposition of carbon on the surface of metal tubes in which normal butane and isobutane are dehydrogenated at elevated temperatures was investigated. Comparative tests show no carbon deposits at 600°C. in the case of isobutane. After two weeks at 625-650°, "Sicromal-8" shows traces of carbon. At 700°, both "Sicromal 8" and copper show deposits even after 1 hour, while "FF-30" alloy shows traces only after 6 days. In all cases, carbonization occurs quicker on rough surfaces than on smooth ones. Using a rapid flow of n-butane at 600°C., the alloys $NCT_3$, Sicromal-8, and Sicromal-9 show, respectively, decreasing deposits of carbon after 8 weeks, while Sicromal-10 remains practically unaffected.

The composition of the steel alloys is given in the following table:

| | %C | %Mn | %Si | %Cr | %Ni | %Mo | %Al |
|------|------|------|------|-------|------|------|------|
| FF-30 | 0.05 | - | 0.6 | 2.80 | - | - | - |
| Sicromal 8 | 0.05 | 0.28 | 0.6 | 6.50 | - | - | 0.45 |
| " 9 | 0.08 | 0.29 | 3.1 | 9.12 | - | - | 0.35 |
| " 10 | 0.084 | 0.29 | 1.0 | 18.15 | 14.0 | - | 0.90 |
| $NCT_3$ | 0.09 | 0.70 | 2.4 | 25.2 | 20.1 | - | - |

54          6p          frames 1020-25          7/2/43

Dehydrogenation of Butane or Propane
in Tubular Reactors over Fixed Bed
Catalysts

        The report compares the results obtained by dehydro-
genating butane in tubular reactors over fixed-bed and mobile
catalysts. In the latter method, the catalyst is sieved through
the reactor, regenerated in an oven to remove coke deposits,
and returned to the reactor. Parallel experiments conducted at
temperatures beginning at 530°C. and a ratio of catalyst to
charge of 1:700 to 1:800, show that the activity of the mobile
catalyst decreases appreciably with the temperature, i.e., the
time necessary to treat the same amount of butane increases.
The fixed bed catalyst, on the contrary, shows no appreciable
failure at 580°C even after 192 hours. The catalyst is very
sensitive to water vapor, which it probably absorbs on cooling
while passing from the regeneration furnace back into the reactor.
This effect does not exist in the fixed-bed procedure, which is
recommended, therefore, for use in the large-scale installations.

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55-76          22p          frames 1026-1047          various dates

        Drawings

        These items are blueprints and flowsheets pertaining
to the equipment and operation of butane dehydrogenation in-
stallations.

        The titles, items, and drawing numbers are listed
below:

| Item | Drawing # | Title | Frames |
|------|-----------|-------|--------|
| 55 | M-3364-1 | Flowsheet | 1047 |
| 56 | M-3075a-1 | Assembly of Tube Bundle; Dehydro-genation Furnace | 1046 |
| 57 | M-9077a-2 | Tube Bundle; Dehydrogenation Furnace | 1045 |
| 58 | M-3074b-1 | Tube Bundle mid-section; Dehydro-genation Furnace | 1044 |
| 59 | M-6776b-2 | Details of catalyst outlet | 1043 |
| 60 | M-4091c-1 | Experimental catalyst outlet | 1042 |
| 61 | M-8222-4 | Details of Dwg. #M-4091c-1 | 1026 |
| 62 | M-2742-8 | Driver for catalyst outlet assembly | 1027 |
| 63 | M-8320a-2 | Details of catalyst outlet assembly | 1041 |
| 64 | M-4023-1 | Details of catalyst outlet assembly | 1040 |
| 65 | M-4000-1 | Housing for catalyst outlet | 1039 |
| 66 | M-4047a-1 | Details of catalyst outlet | 1038 |

| Item | Drawing # | Title | Frames |
|------|-----------|-------|--------|
| 67 | M-8354-2 | Assembly of catalyst outlet; arrangement | 1037 |
| 68 | M-3078b-1 | Catalyst outlet assembly (Cast Iron) | 1036 |
| 69 | M-9620-8 | Catalyst outlet assembly (Cast Iron) | 1028 |
| 70 | M-9883a-2 | Pneumatic valve for catalyst outlet | 1035 |
| 71 | M-4794-16 | Sand-Pocket for tube bundle | 1029 |
| 72 | M-4295-16 | Nozzle details | 1030 |
| 73 | M-2959-1 | Catalyst receiver - dehydrogenation furnace | 1034 |
| 74 | M-5070-1 | Regeneration furnace | 1033 |
| 75 | M-5181-1 | "          " | 1032 |
| 76 | M-5071-1 | "          " | 1031 |

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77          9p          frames 1048-1056          3/31/43

Investigation of Esthonian Shale Oil
as Raw Material for Oxo-Synthesis and
Chemical Processing

Estonian shale is considered to be an interesting raw material for Oxo-synthesis due to its high content of aromatics and olefins (about 36%). Upon distillation, it yields several liquid fractions, boiling up to 340°C. The fractions below 200°C can be directly subjected to Oxo-synthesis to yield alcohols. The higher fractions must first be treated with sulfuric acid and water, or selectively refined with liquid sulfur dioxide and butane. The purified fractions can then be used in the Oxo-reaction or directly sulfonated. The comparative results are summarized in the following table:

| Fraction | Weight % of Shale Oil | Yield of OH-Containing Products from "Oxo" Synthesis of Shale Oil Cuts | Mol. Range of Oxygenated Products | Possible Uses of Alcohols |
|----------|------------------------|-----------------------------------------------------------------------|------------------------------------|----------------------------|
| Gasoline to 200° | 10 | 40-45% | $C_8$-$C_{12}$ | Plasticizers or Ester Oils |
| Oil 200°-250° | 10 | 20-22% | $C_{12}$-$C_{15}$ | Wetting-agents |
| Oil 200°-300° | 12 | 20% | $C_{12}$-$C_{17}$ | Wetting-agents |
| Oil 230°-340° | 25 | 20% | $C_{14}$-$C_{20}$ | Wetting-agents |

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### Experiences with Semi-Technical
### "Synol" - Installation Me458

The report describes the handling and operating procedures of a synol installation in Leuna, consisting primarily of a catalyst reducer, a reactor, a carbon dioxide pressure scrubber, an active-carbon absorber, and a distillation unit. The general procedure, as shown in a series of flowsheets, can be summarized as follows:  The catalyst (WK-17) is reduced in a stream of hydrogen at 450°C. and stored under an atmosphere of hydrogen to prevent any action of oxygen.  The synthesis gas, containing 5% of carbon dioxide, 52% of hydrogen and 43% of carbon monoxide, flows over the catalyst at 20 atms. pressure. The mixture of gaseous and liquid products is then expanded into a "hot" separator where a 20% alkaline solution is added to prevent corrosion.  The gases proceed through an air condenser and a water cooler and finally an ammonium refrigerator to the active-carbon tower; the condensed liquids go to a cold separator where reaction water is withdrawn.  Finally, the liquid products recovered in the expansion vessel go to a second hot separator. The active carbon tower is periodically regenerated with high-pressure steam and the liquid products recovered.  The reactor is also periodically cooled and the accumulated paraffin wax extracted with a solvent.  The liquid fractions go to a batch distillation, where they are distilled to 160C at atmospheric pressure.  Above 160°C, a vacuum of 5-8mm. Hg is used.

The total yield is about 170g. of liquid product per $m^3$ of converted gas.  The catalyst lasts over 7 months in a recycle process, and 3-4 months in a 3-step single pass operation. The yield of alcohols and olefins is quite considerable, ranging from 50-65% in the lower boiling fractions to 30% in the $C_{18}$ cut. The ester yield increases with molecular weight to about 20% in the $C_{18}$ cut.  Aldehydes and ketones average 1-2% in each fraction.  The alcohols are all primary and straight chained, the olefins are alpha-unsaturated.  This article contains very precise details for the step-by-step operation of various units of the installation.

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79-93             16p           frames 1097-1112        various dates

### Drawings of Oxo- and Synol Units

Items 79-86 and 87-93 are blueprints and flow diagrams referring, respectively, to the equipment and functioning of the "Oxo" and the "Synol" installations at Leuna.  A list of their

titles is given below:

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94                53p            frames 1113-1165              5/15/39

## The Use of Liquefied Petroleum Gas for Priming and Propulsion of Airplane-Engines

The manifold advantages of liquefied petroleum gas promoted its early use for car and airplane motors. This gas, consisting chiefly of propane, n-butane, and isobutane, is easy and safe to handle, furnishes more heat per unit of mixture-volume, and has a better octane ratio than the current gasoline. This is of particular advantage in airplane motors, which can be operated thus with equal efficiency while using poorer mixtures. The liquefied petroleum gas is non-toxic and its combustion products are smoke and carbon monoxide-free. The gas allows an easy cold start and does not cause any dilution of the motor oil. It is very useful, therefore, in starting the engine and in the take-off.

This report describes the feeding of liquefied petroleum gas to the motor through a carburetor or through an injector. In the latter case, the liquefied gas may be used with a 10% gasoline addition or may be partially vaporized and both phases used simultaneously. Technical data on consumption, efficiency, performance in motors, costs of installation, storage, transportation and blueprints of regulating and feeding devices complete the report.

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95          53p          frames 1167-1219          5/26/39

This is a repetition of Item 94,
probably included by mistake.

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96          15p          frames 1221-1237          12/14/39

Conversion of a Diesel Truck Engine
for Use with Liquefied Petroleum Gas
(Report from the Experimental Station
  at Ludwigshafen)

The report gives data on the use of liquefied
petroleum gas in a Diesel engine, at a compression ratio of
14:1. A device is described, by which the liquefied petroleum
gas and air mixture is fed into the motor, together with an
igniting Diesel oil of cetane No. 90 to 94. This device also
enables the motor to be quickly switched back to oil use only.
Blue prints and photographs of the mechanical installation,
data of performances, and cost estimates are enclosed.

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97          61p          frames 1239-1299          various dates

Miscellaneous Data on the Use of
Liquid Petroleum Gas

This item is a series of commercial and technical
pamphlets, blueprints and curves dealing with the use of
liquefied petroleum gas, its motor properties and performances,
and the mechanical details of installation and feed, with
particular reference to the vaporizing and carbureting unit.

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98          1p          frame 1300          12/31/41

New Type Vaporizer

Blueprint describes a new vaporizing unit for lique-
fied petroleum gas used as a motor fuel.

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99          17p          frames 1304-1320          2/22/43

Isomerization of n-Butane with Aluminum
Chloride - Report by Drs. Pohl and Nowotny

Isobutane necessary for the production of isooctane

by alkylation can be prepared by isomerizing n-butane at 400-500°
under 100-200 atm. pressure with a tungsten sulfide catalyst, or
at low-pressure and temperature over aluminum chloride, activated
by gaseous hydrogen chloride. The latter process operates at
95-100°C under 16 atm. pressure, using butane free from olefins
and higher hydrocarbons, anhydrous hydrogen chloride, and 8-13%
anhydrous, iron-containing aluminum chloride. It has been found,
that the aluminum chloride sludge is also very active as catalyst.
At a conversion per pass of 25-30% of butane, a 95% yield of
isobutane can be obtained.

In actual operation, fresh and recycle butane are mixed
in an absorber with a recycle mixture of hydrogen chloride gas
and propane, and introduced at the lower part of the reactor.
Dry aluminum chloride is placed at the top of the reactor and the
slowly forming liquid sludge flows down, counter-current to the
gas, and is collected at the bottom. One catalyst charge can
last 18-24 days. The reacted gases are partly condensed in a
column to remove the HCl. The uncondensed HCl and propane are
recycled to the absorber, and the liquefied hydrocarbons are
fed into a fractionating unit. Isobutane and some propane are
vaporized and sent to the alkylating unit, while the residue,
consisting mostly of n-butane is recycled. The propane, which
serves as a vehicle for HCl gas, is formed as a partial cracking
product of n-butane.

Flowsheets and material balance data accompany the
report.

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100          13p          frames 1322-1334          July 1942

Synthesis of Aviation Gasoline by
Catalytic Cracking - Report by Dr. Kaufmann
of Leuna

The report mentions four ways of processing crude oil
to obtain good yields of high octane aviation gasoline. They
are:
     (a)  Distillation of the topped residue under vacuum,
catalytic cracking of the middle oil and gasoline refining.
This method also yields appreciable amounts of diesel oil,
lubricating oils, and heating oils.

     (b)  Hydrogenation and catalytic cracking of the middle oil,
gas stabilization, and refining.

     (c)  Hydrogenation of the residue, cracking of combined
middle oil fractions from topping and hydrogenation, gas stabili-
zation and refining.

(d) Hydrogenation of the topped and cracked residues, cracking of topped and hydrogenated middle oils, gas stabilization and refining. These last three methods, particularly the last one, yield the largest amounts of topped and cracked gasoline.

As for actual cracking procedure; four methods, all employing an alumina-silica catalyst, are mentioned. The first one is fully described and is the one recommended for large-scale installations, since it is the only one so far that is practically developed. It is an intermittent batch process, while the other three are continuous.

## (1) Fixed Bed Catalyst Process

An alumina-silica catalyst of molar ratio 1:9 is used which allows runs of 1-3 hours. Increased cracking time and load favor high yields of high quality gasoline; increased cracking temperature decreases this yield. The gases formed consist chiefly of isobutane, normal butane and propane with some $C_2$-$C_4$ olefins and hydrogen. The most favorable temperature is around 400°C. The catalyst is regenerated periodically by burning off the coke deposits.

## (2) Mobile Catalyst Process

The catalyst is used in bead form, in a special furnace consisting of a catalyst-regeneration zone, a flush zone and a reaction zone. The beads cross the entire unit at an adjustable speed, counter-currently to the material to be cracked, and can be recycled. This process has the one advantage of being continuous.

## (3) and (4) Powdered Catalyst in Gas or Liquid Phase

These are both in the development stage. Their advantages seem to lie in the ease and economy of operation, due to the continuous character of the processes.

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101          41p          frames 1337-1376          June 1944

Catalytic Cracking of Pure Hydrocarbons
Report by - Dr. Weiss

A certain number of pure synthetic or commercial hydrocarbons were submitted to cracking at 420° over a synthetic catalyst #3248 for periods of 60 minutes and the reaction products identified or determined on the basis of analytical data described. Experiments were performed on the following materials: n-decane, n-heptadecane, isododecane, n-decene, n-cetene, xylene,

durene, isoamylbenzene, diphenyl, diphenylmethane, alpha-methyl-naphthalene, tetrahydronaphthalene, isoamycyclohexane, dicyclo-hexyl, dicyclohexylmethane and decahydronaphthalene and led to the following conclusions:

The olefins are the least thermally stable and are almost completely cleaved to lower-boiling compounds. The paraffins are harder to crack, but their stability decreases as the molecular weight increases. The naphthenes can be cleaved and isomerized to cyclopentane derivatives. The aromatics are the most stable, as a class. In polysubstituted aromatics, such as durene, disproportionation into more and less substituted compounds occurs. The introduction of a paraffinic side chain decreases the thermal stability of an aromatic hydrocarbon.

The cracking of naphthenes yields much more aromatics than the cracking of olefins or paraffins under similar conditions, due to the fact that a dehydrogenation is usually easier than a cyclization. The amount of olefins formed in cracking depends also on the constitution of the material and increases in the following sequence: aromatics, naphthenics, paraffins, olefins. These two last points are of importance in the study of cracked gasoline. The production of coke increases, naturally, with the thermal instability of the hydrocarbon. In the case of aromatics, however, polymerization leads to highly condensed, polynuclear compounds.

The catalyst also has a strong isomerizing influence which explains the predominance of ramified products, mostly mono-methylalkanes, in catalytically cracked gasoline.

Tables and analytical curves accompany the report.

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102          25p          frames 1378-1402          April 1940

Quantitative Determination of Lower-Boiling
Phenols by Fractionation - Report by
Dr. Kolb

The report deals with the separation and determination of phenol and of three cresols, in the phenolic oil fractions from coal-tar. Synthetic mixtures of pure phenols were submitted to precise fractionation and the distillation curves and analysis showed the possibility of separating the pure compounds within a ± 2% error. Meta-cresols can be determined as the trinitroderivative. The solidification points of the phenolic fractions provide a very useful check of the fractionation, except in the case of the simultaneous presence of meta and para-cresols. This is due to the closeness of their boiling points

and to the presence of a maximum melting point in the binary
system. Xylenols can be determined by the same method, in the
absence of other phenols of the same boiling range, such as
ethyl phenols, propylphenols, etc.

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103          12p          frames 1404-1416          11/21/38

### Enrichment of Phenolic Waters

In order to increase the amount of phenol contained in
the waters from tar hydrogenation to make its recovery with
tricresylphosphate more profitable, the following enrichment
methods have been suggested and investigated:

(a) Injection of phenolic water in the high-pressure tar
hydrogenation reactors instead of the normal condensate to
increase the concentration of phenol by recirculation. This
method can be applied without disturbing the production only
in the liquid-phase hydrogenation. The small gain in phenol
concentration does not, however, justify the complication of
the installation and the increased cost of operation.

(b) Extraction of phenol-containing oil-fractions with
phenolic waters. This method is cheaper and easier than the
previous one and allows a substantial recovery of phenol.

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104          77p          frames 1418-1497          5/30/41

### Extraction of a Gas Oil from a Rumanian
### Crude with Phenol - Report by Dr. Pohl

In order to prepare surface active agents, so called
"Mesapons", by sulfochlorination, gas oil fractions must be
solvent refined and hydrogenated. This can best be achieved by
extracting the gas oil with 250% of phenol containing 4-10% of
water, at 65-70°C. The process can be performed either in a 5-
stage batch installation or in a 5-stage continuous column in-
stallation. In this latter way, the oil is extracted with
portions of phenol of successively higher water content. The
yield is 55-70% of raffinate containing 80-90% of paraffins.
The recovery of phenol is not quantitative due to the fact that
its boiling point lies within the lower distillation limit of
the extract.

The report is illustrated by technical data and curves,
mostly illegible.

105        12p          frames 1499-1512          March 1944

### Study of an Austrian Petroleum

A series of crude-oil samples from various Austrian drillings was investigated. The following tests were performed: Engler distillation, specific gravity, determination of paraffins by the butanone method, of olefins using the Winkler-bromine number, of olefins and aromatics by sulfuric acid, of naphthenics by the aniline point method, elementary analysis, and sulphur content. The various samples were then classified according to the oil base.

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106        20p          Frames 1514-1534          11/1/41

### Dewaxing Experiments in Ludwigshafen

The experiments were carried out in order to explain the failure of a dewaxing installation in the Lützkendorf factory. The raw product had been deasphalted and refined with phenol. The dewaxing was effected by a liquid propane treatment, crystallization of paraffin and filter-pressing. The Ludwigshafen experiments pointed conclusively to the necessity of stirring during the chilling and crystallization period, to assure the separation of easily filtered paraffin.

The report is illustrated by curves and photomicrographs.

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107        30p          frames 1535-1567          8/12/44

### Recovery of Lower Olefins from Paraffin-Olefin Mixture with Silver Nitrate Solutions - Report by Dr. Pohl

This report refers particularly to the recovery of butene from butane-butene mixtures produced by dehydrogenation of butane. The butene is used for the alkylation of isobutane, from the catalytic isomerization of n-butane, to produce iso-octane. (Refer to items 99 and 54).

It is shown that among salt solutions that form complexes with olefins, a 50% silver nitrate solution is particularly active. Butane-butene mixtures from butane de-hydrogenation can be extracted counter-currently in an iso-thermic, multistage washing installation. The absorbed butene can then be desorbed from silver nitrate solutions by passing through them a stream of isobutane. Thus one obtains directly

butene-isobutane mixtures for use in the alkylation unit.

The report is supplemented by equilibrium computations, flowsheets and cost calculations.

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108          18p          frames 1569-1586          July 1944

Investigation and Comparison of
Redistillation Residues from DHD
and HF Processes - Report by
Dr. Welz and Dr. Ebeneder

The constitution of residues from the redistillation of several DHD and HF products has been investigated and determined qualitatively. The following residues have been investigated:

(1) DHD residue from Leuna; the raw material for that process was hydrogenated gasoline from lignite.

(2) DHD residue from Ludwigshafen; raw material was a mixture of straight-run gasolines from Rumanian and Hungarian crudes. Analysis showed the predominance of polycyclic ring systems, such as naphthalene and its alkyl-derivatives in both DHD residues, while the lower-boiling HF residues contained, primarily, alkylbenzenes.

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109          55p          frames 1588-1642          5/26/41

Laboratory Research on the Synthesis of
Adipic and Alkyl-Adipic Acids -
Report by Dr. Fröhlich

This report, for the most part hazy and illegible, describes attempts to find a cheaper raw material for the synthesis of adipic acids. So far, a hydrogenated phenol oil (SR-II) containing cyclohexanol, methyl- and dimethyl-cyclohexanol, was treated with an eight-fold excess of 62% nitric acid at temperatures not exceeding 80°C., to yield 80-90% of corresponding adipic acids.

Since phenolic oils are rather expensive, experiments were carried out on hydrogenated benzene derivatives. Cyclohexane proved to be very resistant to the action of nitric acid. Cyclohexylamine, cyclohexylchloride, cyclohexene and organic esters of cyclohexanol, prepared by heating cyclohexene

with acids, gave poor yields of adipic acid. The best method
was found to be the hydration of cyclohexene with 80% sulfuric
acid followed by oxidation of the resultant cyclohexanol with
nitric acid.

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110          78p          frames 1644-1721     September 1939

Progress of the Research on Hydrocarbon
Synthesis in Leuna - Report for the
first half year of 1939

In the Fischer process carbon monoxide is hydrogenated
to give mixtures of predominantly straight-chained, aliphatic
hydrocarbons, both paraffinic and olefinic. The most important
factors governing the properties of the product are the nature
of the catalyst, the composition and rate of the charge gas,
pressure and temperature. Semi-technical experiments carried
out in a 4-stage continuous installation yielded 70-81g. of
liquid and solid products per $m^3$ of gas. From 48-56% of the
product consisted of paraffin. Catalysts 3440 and WK5b, not
closely described, gave yields of 67% to 37%, of products boil-
ing over 300°C depending on the reaction time; catalyst KO 30
(from Oppau) gave constant yields. The regeneration of catalysts
resulted, usually, in a decrease of the paraffin formation.

The report is substantiated by detailed tables
describing the results obtained in the laboratory and in the
semi-technical unit. An extensive cost estimate for gasoline
synthesis is also included.

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2/21/47