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SUBJECT- INDEX AND REPORT
T. O. M. REEL NO. 129-30

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T.O.M. Reels 129 and 130

INDEX AND REVIEW

Prepared by

United States Bureau of Mines

Coal Hydrogenation Demonstration Plant

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The two Reels, #129 and 130, were the subject of a report of the United States Naval Technical Oil Mission in Europe, entitled

"The Production of Synthetic Fuels in Germany by the Hydrogenation of Carbonaceous Substances."

The documents were forwarded to the Bureau of Ships by NAVTECHMISEU letter JJ7 (10/Ma) Serial 631, dated July 20, 1945.

The documents are listed alphabetically as "references". In most cases, the references represent one paper or document on a specific subject. In certain cases, the "references" include a number of papers on one or several topics.

The two reels are listed together in the forwarding letter, but are treated separately in the present report. The page numbers indicate only an approximate location of the articles in the reel.

I. CLASSIFIED INDEX. Reels 129 and 130.

	REEL	REFERENCE
<u>ACCIDENT PREVENTION</u>	130	m-12, gg
<u>ACCOUNTING</u>	129	a1, a3, a4
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<u>EXPERIMENTAL RUNS</u>	130	p, q
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	130	i, q
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	130	m-8
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<u>STALL OPERATIONS</u>	130	m-3, m-4, m-5, m-6
<u>STALL DISMANTLING RESULTS</u>	130	o, r
<u>STEEL</u>	130	aa, cc, dd, ee, ff
<u>TESTING OF MATERIALS</u>	130	m-15
<u>TROUBLES IN OPERATIONS</u>	129	a-7

II. Review of Contents. REEL 129

<u>Refer.</u>	<u>Pages</u>	
a-1	2-51	Reasons for Creating Special Accounting Stations in Hydrogenation. Translated, T-133. W. M. Sternberg.
a-2	52-56	Discussions on March 26, 1943 in Leuna. The NIO Material. Translated, T-191. W. M. Sternberg.
a-3	57-68	Accounting in Hydrogenation. Leuna, 1943. Translation (abridged) T-187. W. M. Sternberg.
a-4	69-79	Directions for Accounting in Hydrogenation Plants. Leuna, July 1943. T-195. W. M. Sternberg.

Ref. a-5; pp. 80 - 133:

Two papers by Bähr on a combination process of hydrogenation and low temperature coking (L.T.C.). The consumption of hydrogen in the hydrogenation of coal is some 2 1/2 times higher than of tar. The utilization of carbon is higher in hydrogenation, the coke produced by L.T.C. is a non-smoking fuel, and has a definite market value, even if high in ash.

The two papers describe in detail the work done during 1938, the first one on a laboratory scale, the second describes a large scale run as well.

The hydrogenation took place either in a coil, or else a coil and an empty reaction space (preheater and 1st converter) with a proportion of 1:2 between the reaction space and the volume of coal. The cost of hydrogen was reduced by substituting for it methanol gas with 25% CO, or water gas, and a utilization of 75 - 82% was obtained in small scale tests, while with one converter of the stall 8, a utilization of 87.3% was reached. The throughput in the latter case was increased from 20 to 27 m³. The HOLD was mixed with dry brown coal, and either coked at low temperature, or else used in a Lurgi gas producer, with good results. The thruput thus was greatly increased, and only one of the four converters were used.

The process could find application for hydrogenation as well as for L.T.C. The "shortened" hydrogenation could be run in conjunction with L.T.C. by mixing dry brown coal to be coked with the HOLD and briquetting it, with a tar output of 27 - 32% instead of the usual 12 - 15%. The oil or tar products distilled over are absorbed in middle or heavy oil. The heavy oil is used in pasting the coal for hydrogenation, and is in turn partially broken down into middle oil. The oil formed in the catchpot of hydrogenation and L.T.C. is also fractionated into middle oil and heavy oil.

About the double amount of tar is produced during L.T.C. with no changes of power, service and thruput, the costs would not be increased in this process, while hydrogenation would be simplified and cheapened, and the loss in carbon during kilning might be reduced by utilizing the products as grude coke. Economic computations were in progress.

- a-6. pp. 134-141 High Pressure Converters Made by the Wickel Process. T-99. K.C. Braun.
- a-7. pp. 142-164 Operational Difficulties and Experience in High Pressure Work During the Last Months. Leuna, Jan. 15, 1937.

Contents:

- A. Difficulties with gas: carbon monoxide
Remedies: (a) pressure resistant thermo-
couple tubes; (b) elimination of V2A sheets
from the converter; (c) strengthening of
V2A sheathing of cold gas tubes; (d) in
the HOLD unit: mounting separately, bends
and T's replaced with hose connections and
gentle bends.

- B. Non-uniformity of the product and its over-
coming.
 - (1) Sand and caviar.
 - (2) Plugging up of the heat exchanger
II by coarse particles.

- C. Overcoming disturbances by changes in
operating procedure (only in the coal
liquid phase)
 - (1) Erosion of heat exchanger I. Remedy:-
reduction of the velocity of flow;
 - (2) Coking in converter IV. Remedy: a
lower concentration and a higher rate of
flow.
 - (3) Coke formation in the hot catchpot,
Remedy: change to the iron catalyst, and
as in (2).

- D. Elimination of offensive odors by enclosing
the hot catchpot (in the liquid coal and tar
phase).

This document is rather old and largely superceded by more recent information. It has not been considered necessary to either translate it or to abstract it in detail.

a-8 pp. 165-178. Technical Experiences in the Operation of a Converter Stall in the year 1936 (Second Half). Abstracted, T-53. K. C. Braun

- b. pp. 197-203 TEA Report on the Development of Hydrogenation in Leuna. A historical review. Slightly condensed, T-23. W. M. Sternberg.
- c. pp. 204-333 Monthly reports of the Bohlen Works for 1942. A very detailed production report of the works, covering every phase of production. The report will stand very close study, but can not be abstracted.
- d. pp. 334-397 Basis of computations for the Monthly Reports of the Bohlen Works of the Braunkohle-Benzin Aktien Gesellschaft. Curves and data used in the accounting work done. Can not be abstracted.
- e. pp. 398-460 Basis of Computations for the Monthly Reports of the Leuna Works for the month of March, 1944. Similar to ref. d.
- f. pp. 461-563 Monthly Reports of operation of the Sudetenland Treibstoffwerke Aktiengesellschaft, Hydrierwerke Brux. Included are reports on L.T.C., gas production (Linde oxygen installation, water gas, gas compression and pressure conversion, CO₂ and CO scrubbing), hydrogenation, etc. Can not be abstracted.
- g. pp. 564-653 Monthly Report for December 1943, the Pöhlitz Hydrogenation Works.
- h. pp. 654-674 Characteristics of the Different Hydrogenation Works. Translated, T-46. W. M. Sternberg
- i. pp. 675-679 Hydrogenation of Rhenish Brown Coal. Translated, T-196. W. M. Sternberg.
- j. pp. 680-725 High Resistances on the Suction Side of Heat Exchangers in the 5058 Stall. Translated, T-128. K. C. Braun.
- k. pp. 726-776 Fouling on the Suction Side of the 5058 Heat Exchangers. Translated, T-131. K. C. Braun.

- | <u>Ref.</u> | <u>Pp.</u> | |
|-------------|------------|--|
| 1 | 4-36 | <u>Investigation of Two Heat Exchanger Tube Bundles in-Brux. Leuna, 1943.</u>
<u>Hydrogenation of Tar.</u> The heat exchangers were dismantled because of high resistance, and the tube bundles examined. The reasons for the plugging up were diagnosed as follows:

1). Deposition of iron sulfide formed by corrosion of the tubes and apparatus by the sulfur in the feed and from the catalyst.

2). Deposition of sodium chloride from the chlorine in the tar and the sodium bicarbonate in the catalyst.

3). Sedimentation of especially high silicate and iron particles from the catalyst.

4). Tar formation through deposition of high asphalt materials in heat exchanger I.

5). Coking in the heat exchanger. The percent of the individual components of the deposits was presented diagrammatically. The simple assumption of a deposition of catalyst was disproved. The principal reason for the plugging up must be found in the properties of the Brux tar, and the unsteady operation of the tar stall, subjected to strong fluctuations, may have played an important role. |
| m | 37-207 | <u>Typical Operating Sheets. Leuna, 1937 - 1938.</u>

The title was supplied by the Navy Department Bureau of Ships. This really is a collection of papers on tar hydrogenation in Leuna, with some drawings, flow sheets, operating details and data. Some of the individual articles have been translated and Bureau of Mines translation numbers given. |
| m-1 | 37-52 | <u>Preparation of Tar and Vapor Phase Hydrogenation.</u>

The composition is given of the tar used, the catch pot products and HOLD produced, also typical operating sheets and data. |
| m-2 | 53-55 | <u>Centrifuging of Tar.</u> Centrifuging of tar and off-oils for the removal of water and solids. Details of operations and a flow sheet. |

<u>Ref.</u>	<u>Pp.</u>	
m-3	56-75	Tar Liquid Phase Stall. Flow sheets and some operation data of the tar stall; includes a sketch of thermocouple connections in the gas preheater.
m-4	76-111	Vapor Phase Stall. A brief description and operating data on different vapor stalls; also details of filling and emptying the converter with the catalyst.
m-5 & m-6	112-134	Starting & Shutting Down Stall. Abstract T-164. K. C. Braun.
m-7	135-157	Tar Injection and Pump House Description. Flow sheets and very brief description of the pumps used in the liquid and vapor phases.
m-8	158-166	Operational Disturbances. Trouble during operation of hydrogenation plant. Translated, T-203. W. M. Sternberg
m-9	167	Analytical Values for NH_3 , H_2S and CO_2 .
m-10 & m-11	168-174	Gas Analyses. Typical gas analyses and apparatus for sampling gas.
m-12	175-179	Accident Prevention. Translated T-85. W. M. Sternberg.
m-13 & m-14	175-180	Connecting Motors to Different Circuits & Bringing the Gas Circuit Pipe Lines to the Stalls.
m-15	181-189	Testing of Materials. Translated T-84. W. M. Sternberg.
m-16		Controls of Electric Preheaters.
m-17-a	190a- 197a	Temperature Measurements by Means of Thermocouples. 1). Iron-constantan, silver soldered, iron positive pole; up to 600° . 2.) Cr-Ni.-B-Thermominus from 600 to 1000°C . welded, Cr-Ni-B-is positive. 3). Platinum-platinum rhodium up to 1600° . Diagrams of connections are given.

Ref. Pp.

m-17-b 198a-199a

Table for Converting mv. with the iron-constantan couples °C. The iron-constantan scale is referred to 40° C. cold junction:

<u>Deg.C.</u>	<u>mv.</u>	<u>Deg.C.</u>	<u>mv.</u>
-195.8	-9.78	270	13.01
- 78.5	-5.77	280	13.58
0	-2.07	290	14.16
10	-1.56	300	14.74
20	-1.05	310	15.31
30	-0.53	320	15.89
40	0	330	16.46
50	0.52	340	17.04
60	1.04	350	17.62
70	1.58	360	18.20
80	2.13	370	18.78
90	2.69	380	19.37
100	3.25	390	19.95
110	3.81	400	20.53
120	4.37	410	21.12
130	4.93	420	21.70
140	5.50	430	22.29
150	6.06	440	22.87
160	6.62	450	23.46
170	7.18	460	24.05
180	7.75	470	24.64
190	8.33	480	25.23
200	8.92	490	25.82
210	9.50	500	26.42
220	10.09	510	27.01
230	10.67	520	27.60
240	11.26	530	28.20
250	11.85	540	28.80
260	12.43	550	29.40
		560	30.00
		570	30.60
		580	31.23
		590	31.86
		600	32.49
		610	33.12

Chrome-Nickel B-Thermominscuple. Junction at 20°.

<u>°C.</u>	<u>mv.</u>	<u>°C.</u>	<u>mv.</u>	<u>°C.</u>	<u>mv.</u>
500	11.70	720	18.42	940	26.14
510	11.98	730	18.75	950	26.50
520	12.26	740	19.09	960	26.86
530	12.54	750	19.43	970	27.22
540	12.82	760	19.78	980	27.58
550	13.12	770	20.13	990	27.95
560	13.42	780	20.48	1000	28.32
570	13.72	790	20.83	1010	28.69
580	14.03	800	21.18	1020	29.06
590	14.34	810	21.53	1030	29.44
600	14.65	820	21.88	1040	29.82
610	14.96	830	22.23	1050	30.20
620	15.27	840	22.58	1060	30.58
630	15.58	850	22.93	1070	30.96
640	15.89	860	23.28	1080	31.34
650	16.20	870	23.63	1090	31.72
660	16.51	880	23.98	1100	32.10
670	16.82	890	24.34	1120	32.86
680	17.13	900	24.70	1140	33.62
690	17.45	910	25.06	1160	34.38
700	17.77	920	25.42	1180	35.14
710	18.09	930	25.78	1200	35.90

Ref. pp.

- m-17-c 190-199 Measurements of Gases & Liquids by means of Throttling Discs. Rough sketches and formulas are written in pencil and frequently illegible.
- m-17-d 200-201 Corrections for Pressure Flow Meter Readings.
- m-17-e 202-207 Air Activated Recording Manometers of Siefert; Miscellaneous Data on Stall Operations.
- n 208-228 OVERCOMING DIFFICULTIES WITH CAVIAR FORMATION BY DESANDING OF CONVERTER I. Leuna 1941. Translated T-109. W. M. Sternberg.
- o 229-245 REPORTS OF DISMANTLING OF STALL V, Leuna 1941. Translated T-165. W. M. Sternberg.
- p 246-320 LARGE SCALE FUEL OIL PRODUCTION TESTS. Ludwigshafen 1941. Translated T-52. W. M. Sternberg.
- q 321-339 RESULTS OF LARGE SCALE TESTS OF PASTE HEAT EXCHANGE. Ludwigshafen 1941. Translated T-32. W. M. Sternberg.
- r 340-372 PRELIMINARY REPORT ON DISMANTLING OF HIGH PRESSURE STALL AFTER THE LARGE SCALE FUEL OIL EXPERIMENTAL RUN. Ludwigshafen 1941. Translated T-55. W. M. Sternberg.
- s 373-383 DISCUSSION OF 300 AND 700 ATMOSPHERE HYDROGENATION IN UPPER SILESIA. Leuna 1941. Dr. Peters' report gives a numerical tabular survey of the different methods of operation in the vapor phase at 300 and 700 atmospheres.

When operating for the production of 50-55% aromatics in gasoline, using S gasoline was only possible at 250 atm. S gasoline gives low aromatics and high gasification at 300 to 700.ats.

One and two-stage benzination at 700 ats. produces the least gasification in the production of DHD feed.

The highest proportion of iso octane is produced at 300 ats. in the 2-stage process, if the liquid phase be operated for fuel oil production.

<u>Ref.</u>	<u>Fp.</u>	
t	384-411	CHLORINE IN THE VAPOR PHASE INJECTION FEED. ITS REMOVAL, AND GENERAL ORIENTATION OF OPERATION DETAILS AT HIGH PRESSURE IN SCHOLVEN. Leuna, 1939. Translated T-183. W. M. Sternberg.
u	402-434	SULFIDIC VAPOR PHASE CATALYST, ESPECIALLY TUNGSTEN SULFIDE, IN INDUSTRIAL COAL HYDROGENATION. Lecture delivered by M. Pier in Ludwigshafen, 1943. Translated T-184. W. M. Sternberg.
v	435-458	DEVELOPMENT OF PREHYDROGENATION CATALYST. By Gunther, Dec. 1940. Also on Reel 167, fr. 624-643 and Reel 165, fr. 199-221. Part III of series of articles. Part I, reel 167, fr. 657-664 on catalyst 5058, 6719 and 7745. Part II, reel 167, fr. 646-656, on a Al_2O_3 - Mo with W catalyst. Part III Al_2O_3 - Mo - Ni catalyst. Summary of Dr. Gunther's paper:

1. No entirely satisfactory substitute for 5058 catalyst is found in the dilute tungsten catalysts, nor in the combination of Mo-W (-Fe-Ni), and experiments were therefore undertaken with alumina catalysts alone. Dr. Stöwener's in Oppau alumina was used as the active alumina.
2. The aniline point of the B middle oil rises in the alumina-Mo catalysts with increasing Mo contents. No sufficient hydrogenation effect is obtained with as much as 30% MoO_3 upon alumina.
3. The a. p. of alumina-Ni catalysts (no Mo) of the B middle oil rises also with the Ni content of the alumina-Ni catalysts (no Mo). The hydrogenating action is less than with the corresponding Mo catalysts.
4. The addition of 3% Ni_2O_3 to the alumina-Mo catalysts increases the hydrogenating effect very strongly. The same relationship of a. p. of the B middle oil from the Mo concentration as in 2. is found. Clay catalysts with 3% Ni_2O_3 and 10% MoO_3 have very good hydrogenation activity. Catalysts of this type (7846) can very well replace the 5058 catalyst.
5. The maximum hydrogenation efficiency in catalysts composed of active alumina, 10% MoO_3 and X % Ni_2O_3 is found with X = 6% Ni_2O_3 .
6. The relationship between catalytic efficiency and the ratio of Mo : Ni has been determined for catalysts composed of

activa alumina + 9% (MoO₃ + Ni₂O₃). Mo and Ni are found to be activating each other. A sharp maximum of activating efficiency is found with 1 mol Mo : 0.63 mols Ni.

7. Sulfurizing Mo-Ni catalysts results only in an initial rise of activity. When sulfur-containing raw materials are used, the catalysts become spontaneously sulfurized in the course of a few days. Catalysts sulfurized under pressure apparently adjust themselves to the same sulfur content. One may no longer be able to tell after a few days of operation, whether the catalyst had been sulfurized under pressure or not.

8. Catalyst 7846 (act. alumina + 10% MoO₃ + 3% Ni₂O₃) produces an exceptionally well refined middle oil with a. p. of 38 - 40° from a coal hydrogenation middle oil with an a. p. of -15°, which can be used over 6434 as well as the 5058 B middle oils. Somewhat less gasoline is obtained during the prehydrogenation than with 5058.

The quality of the 6434 gasoline is the same as with the 5058 prehydrogenation. The 7846/6434 system produces as much gasoline and of the same quality as the 5058/6434 system.

9. A run with 7846 has now been continued for 50 days with no loss in effectiveness. Benzination of the B middle oils produced has been run for 26 days without the least loss in efficiency and with exceptionally good productivity. This is discussed later in greater detail.

10. Acknowledgement of the work of co-workers, Drs. Peters, Grassl, Trofimow, Donath, Fünser, Dehn and Meyer.

Ref. Pp.

- w 460-483 OPERATING EXPERIENCE WITH CATALYST 7846-W-250 (6376) IN COMPARISON WITH 5058 AND COMBINATIONS OF THE TWO CATALYSTS. Some of the article is illegible; the rest translated, T-185. W. N. Sternberg.
- x 484-507 REPORT ON THE FILTRATION OF COAL HYDROGENATION LET-DOWN, Leuna July 21, 1938. Abstracted T-177. K. C. Braun.
- y 508-558 BID ON PLANT PRODUCING 180,000 TON PER ANNUM OF AVIATION GASOLINE AND 30,000 TON PER ANNUM OF LIQUID GASES FROM BROWN COAL BY CATALYTIC PRESSURE HYDROGENATION FOR RUSSIA. I. G., Ludwigshafen, December, 1939. Abstracted T-182. K. C. Braun.

<u>Ref.</u>	<u>Pp.</u>	
z	559-587	I.G. CONSTRUCTION STEELS. Abstracted T-179 K. C. Braun.
aa	588-590	PHYSICAL AND CHEMICAL PROPERTIES OF STEEL. Table not readable.
bb	591-596	REPORT ON CONSTRUCTION MATERIALS FOR HYDROGENATION BY DR. DINKLER. Leuna, Oct. 1942. Abstracted T-96. K. C. Braun.
cc	597-611	HIGH PRESSURE STEELS. Spring, 1944. A lecture by Dr. Dinkler. Translated T-98A. K. C. Braun.
dd	612-622	RESULTS OF INVESTIGATION OF NiO STEEL, Leuna, 1943. Translated T-172. W. M. Sternberg.
ee	623-627	REMARKS ABOUT MATERIALS IN HYDROGENATION AND THEIR SUPPLY BY STEEL WORKS. By Dr. Class, Leuna, Oct. 1942. Abstracted T-93. K. C. Braun.
ff	628-635	HYDROGEN EMBRITTLEMENT TESTS, THEIR DEPENDENCE ON TEMPERATURE, LENGTH OF TEST AND PRESSURE. Leuna, 1944. Translated T-94. W. M. Sternberg.
gg	636-638	ACCIDENTS AND DAMAGE IN HYDROGENATION PLANTS, Ludwigshafen, 1942. Translated, T-178. K. C. Braun.
hh	639-761	OPERATION OF HYDROGENATION PLANT. Translated T-45. K. C. Braun. W. M. Sternberg.
ii	762-771	REPORT ON ADVANCES IN MEASUREMENT, REGULATORS AND AUTOMATIC CONTROL OF OPERATIONS IN HIGH PRESSURE PLANTS, Leuna, Oct. 1942. Abstracted T-91. K. C. Braun.
jj	772-784	REPORT ON PHYSICAL CONTROL OF OPERATIONS IN THE DEVELOPMENT AND OPERATION OF HYDROGENATION WORKS. Ludwigshafen, by P. Gmelin. Translated T-92. K. C. Braun.
kk	785-797	TEMPERATURE MEASUREMENTS IN HYDROGENATION. A general discussion of the technique of measure- ments. An article by Weiss, written in 1935, it contains well known information on the subject.
ll-1	798-815	ELECTRIC LIQUID LEVEL INDICATORS FOR CONTAINERS UNDER PRESSURE. By W. Weis. A description of an electric level indicator for containers under pressure. The level is indicated by an aluminum rod suspended in a high pressure space on a spring,

Ref. Pp.

and the buoyancy of the liquid reduces the weight of the rod. The position of the aluminum rod is raised or lowered depending on the level of the liquid. The position of the rod is transmitted without friction to a self-induction distance indicator. For this purpose, a small iron rod is fastened to the aluminum rod and the iron will affect the self-induction of a coil outside the pressure space. Such level indicators have at present been constructed for a maximum pressure of 700 ats. and for a range of measurement of 300 or 500 mm.

- 11-2 816-845 ELECTRIC LIQUID LEVEL INDICATOR FOR CONTAINERS UNDER PRESSURE. Supplement to Reference 11-1, consisting of drawings and curves.
- mm 846-874 AUTOMATIC REGULATORS OF THE LIQUID AND VAPOR PHASE STALLS AT LEUNA. By Weis, January 1944. Abstracted T-88. K. C. Braun.