FILM STUDY GROUP

SUBJECT INDEX AND REPORT

T.O.M. REEL NO. 75

Prepared by

PHILLIPS PETROLEUM CO.

PHILLIPS PETROLEUM CO.
RESEARCH DEPT.
BARTLESVILLE, OKLA.

INDEX

POLITZ HYDROGENATION WORKS

Technical Oil Mission Microfilm Reel No. 75

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Report 578-46R

PHILLIPS PETROLEUM COMPANY

RESEARCH DEPARTMENT

BARTLESVILLE, OKLAHOMA

REPORT ON

UNITED STATES TECHNICAL OIL

MISSION MICROFILM REEL 75

Compiled and Edited

by

Technical Information Section

PHILLIPS PETROLEUM COMPANY - RESEARCH DEPARTMENT - REPORT 578-46R

56

UNITED STATES TECHNICAL OIL MISSION MICROFILM REEL 75

Compiled and Edited

by

Technical Information Section

SUMMARY

This report on United States Technical Oil Mission Microfilm Reel No. 75 is a part of the American Petroleum Institute - Technical Oil Mission program devoted to the indexing, abstracting, and translation of the German literature collected by the Technical Oil Mission. Its distribution outside Phillips Petroleum Company includes all companies participating in the program and the Library of Congress. From the Library of Congress, photostat or microfilm duplication is available to anyone making a request.

The general content of this reel is literature on the Pölitz Hydrogenation Works. Specific content of greatest value includes catalysts, charge stocks, operating conditions, products obtained and equipment used.

Albert E. Miller, Chairman
API-TOM Study Group

DEC 24 1946

MIXTURES OF PRODUCTS CHARGED TO CHAMBERS

Frames 12-13

Translation

Chamber 1 a. For the preparation of the polymer, 2,772.5 parts 1-C_4 were added from reservoir #2 in building #610. The chamber was charged with 1,777.7 parts of pure polymer. 1,762.5 parts (of pure polymer) had accumulated. 12

Chamber 21 and 22. (DHD). Charge for tank 157 (fractionation number V. 1 and V. 2) was as follows:

On the 1st and 2nd	40 vol % #5058-gasoline
	35 vol % #5058 spindle oil-gasoline
	7 vol % #6434 spindle oil-gasoline
	18 vol % rich gas condensate
On the 3rd.	65 vol % #5058 spindle oil-gasoline
	10 vol % #6434 spindle oil-gasoline
	25 vol % rich gas condensate
On the 4th to 20th	45 vol % #5058 spindle oil-gasoline
	11 vol % #6434 spindle oil-gasoline
	24 vol % petroleum gasoline
	20 vol % rich gas condensate
On the 21st to 30th	53 vol % #5058 spindle oil-gasoline
	12 vol % #6434 spindle oil-gasoline
	17 vol % petroleum gasoline
	18 vol % rich gas condensate

Note that April 4 was the first time that petroleum from fractionation A 1 and B 1 was mixed for tank 157. Because of incorrect position of a slide value (used in recirculation) during an air raid alarm on April 9, 1944, the contents of tank #157 spilled over to tank 1 and became mixed with it.

Chamber #21 operated during periods 102 to 105. Chamber #22 operated during periods 88 to 93. See page 15 (original German) for boiling points of the mixtures.

Chamber 1 to 4 (#6434). From the 1st to the 30th there was mixed in tank Q:

70 vol % #5058-gasoline
30 vol % #6434-gasoline

See page 16 (original German) for composition of the products injected. Chamber #1 was discharged April 6 to Contact (vessel) #6434.

Chamber 5 to 8 (#5058). The following was added to tank K:

32 vol % Middle oil from coal extraction
10 vol % Middle oil from tar mixture
8 vol % Gasoline from coal and tar mixture

50%

Report 578-46R
April 1944

DISTILLATION

Frame 12

Translation

●

	Raw Material throughput cubic meters per hour	Yield gasoline	Density gasoline	Yield Middle oil	Density Middle oil	Density residue	Product from Chamber Number
A_1	55-75	16	0.74	35	0.84	0.92	Chamber 11 + 12
A_2	65	23	0.77	40	0.97	1.08	tar mix 13 - 16
A_3	40-80	22	0.78	40	0.97	1.09	tar mix 13 -16

Figures are representative for the month of April

Chamber 5 to 8 (#5058). (continued)

15 vol % Middle oil from petroleum fraction
17 vol % Middle oil from rum (ethyl) crude oil
7 vol % Middle oil from brown coal and middle oil
11 vol % Gasoline from petroleum + petroleum fraction
50%

From the 1st to the 14th the brown-coal middle oil was distilled. This brown-coal middle oil had been mixed with petroleum and petroleum stripper product 13 from tank C_1; from the 15th to the end of the month it was mixed with the coal stripper + tar mixture from tank H and I. See page 17 (of original German) for mixtures.

Frame 14 (See figure on the following page)

Mixtures to Tank 157 Charge Stock for Fractionation U. April 1944

German	English
Reichgaskondensat 20/110	Rich gas condensate 20/110
6434 S.Bi 130/180	#6434 Spindle oil gasoline 130/180
Erdolbenzin 55/165	Petroleum gasoline 55/165
5058 S.Bi 130/180	#5058 Spindle oil gasoline 130/180
5058 Bi 45/180	#5058 Gasoline 45/180
5058 Bi 45/130	#5058 Gasoline 45/130

Chamber 11 and 12 (petroleum). From the 1st to the 30th the following was mixed in 16 tank B:

50 vol % heavy oil from Hungarian and Rumanian crude oil
50 vol % heavy oil from petroleum stripper

See page 18 (original German) for mixtures. 17

Chamber 13 to 16. The contents of tank D were as follows:

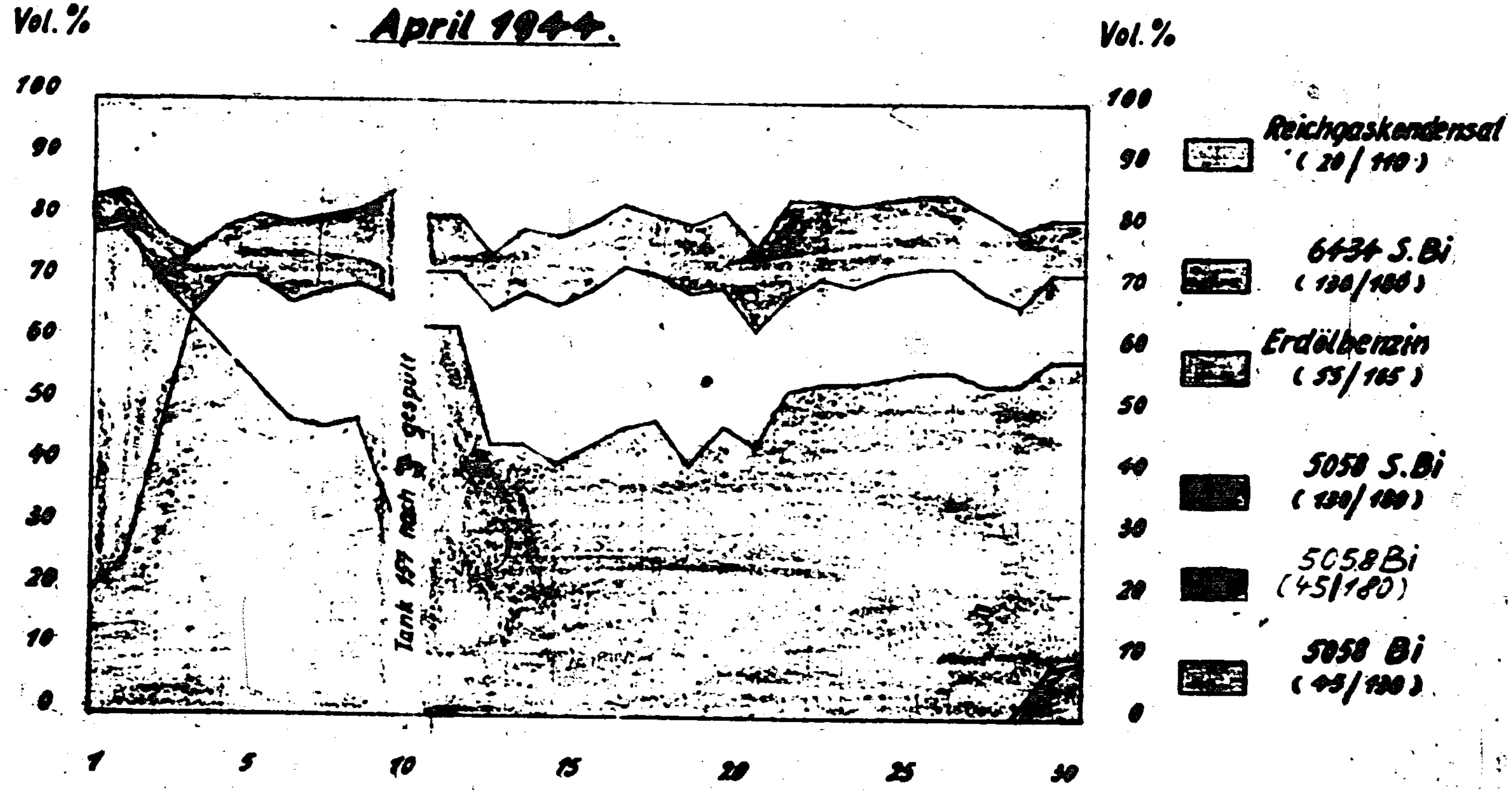
from 1st to 15th	5.0 - 6.0%
16th to 19th	6.0 - 7.0%
20th to 24th	5.5 - 6.5%
25th to 30th	7.0 - 8.0%

The yield was increased about 30 to 35 per cent from the 13th to the 15th by the addition of 15 to 20 per cent heavy oil to the centrifuged oil.

Mischungen nach Tank 157

(Rohprodukt für V_1 Destillation)

April 1944.



Hydrierwerke Pölitz A.G.
Stettin-Pölitz

DISTILLATION

April 1944

Frames 22 - 23

Translation

	Raw Material throughput, Cubic Meters per hour	Yield gasoline %	Density gasoline	Gasoline % (to 100°C)	Octane Number (Benzene)	Yield Middle Oil	Density Middle Oil	Density residue	From Chamber Number
B_1	44	18	0.74	23	-	35	0.85	0.93	petroleum 11 - 12
B_2	58	23	0.72	57	66	12	0.78	0.85	5 - 8
B_3	57	22	0.72	65	67	12	0.78	0.85	5 - 8
C_1	40	60	0.69	70	73	6 - 9	0.77	0.80	2 - 4

Report 578-46E

DISTILLATION

April 1941

Frames 23-24

Translation

Number	Raw Material throughput Cubic Meters per hour	Yield gasoline %	Density gasoline	Gasoline % (to 100° C)	Unsaturates and aromatics %	Naphthenes %	Paraffins	Iodine Number	Octane Number (benzene)	Density residue	From Chamber Number
Distillation	45	70	0.70	60	7.5	33.0	59	-	71	0.80	2 - 4
DHD distillation U	37	14 (to 90° C)	.64	-	12	33	54	-	-	0.77	varies
DHD distillation R	20	93 (to 90° C)	.78	28	60	10	30	2	82	0.98	21 - 22

STABILIZATION

Frames 25-26

April 1944

Translation

No.	Raw Material Throughput cu. m./hr	% gasoline to 70 °C	% gasoline to 100°C	V. P.	D.	b.p. at end °C	Unsaturates and Aromatics	Naphthenes %	Paraffins %	Octane Number		I$_2$ No.	From Chamber
										benzene	gasoline + Pb pentane addition %		
1	34	28	68	0.48	0.71	150	6	38	56	71	10	-	2-4
2	40	28	68	0.48	0.71	150	6	37	56	71	10	-	2-4
3	25	19	41	0.46	0.77	172	50	12	36	79	6	2.5	21-22
4	19	19	43	0.46	0.76	172	49	14	37	79	3	1.7	21-22

Report 578-[illegible]

MONTHLY REPORT ON THE BALANCE OF LEAN AND RICH GASES DURING MONTH OF SEPTEMBER, 1944

Frames 114-127

Abstract

Butane propane, and CO are cracked. The monthly report deals with improvements in measurements and calculations within the industrial plant. Operations are not included.

The following tables are included:

Frame 119	Content of charge and cracked gas.
Frame 124	Propane-butane charge composition.
Frame 126	Composition of "rich" gas.

Example (Frame 119)

GAS CHARGED AFTER CRACKING

CO_2	...	6.91
CO	0.95	10.89
H_2	68.34	78.72
N_2	6.10	2.47
Hydrocarbons	24.61	1.01
Carbon No.	1.82	1.00
	101.82	101.00

Report 578-46R

HYDROGENATION PLANT AT POELITZ

I. V. Diepenbruck

Frames 226-244

Translation

OPERATING EXPLANATION

A. Working Method.

The Hydrogenation Plant at Poelitz has been built, in order to produce refined substances out of coal, coal-tar oil, mineral oil, or cracking residue. The practical method for this production is the perfected catalytic high-pressure hydrogenation of the I. G. Farben Company. 226

In this process, the charging material, which generally consists of "hydrogen poor" coal and hydrocarbon molecules, is accumulated under high pressure and with high temperature hydrogen.

The hydrogen used to this end is either produced out of coke and steam in a generating plant (Building No. 128, 129), whereby water gas forms and/or in a cracking plant (Building No. 207, 208, 209, 210), in which cracked gas forms, which two products are sent through a conversion plant (Building No. 212, 213), in order to convert the carbon monoxide contained in the products extensively into carbon dioxide. This crude hydrogen is then washed under pressure with water (Building No. 240) or copper hydroxide (Building No. 241), in order to get out the carbonic acid and residue in the carbon monoxide, and condensed at the working pressure (Building No. 230, 250), of 300 or 700 atm.

The raw materials used in the hydrogenation, producing benzine and gas oils, are coal, coal-tar oil, coal tars, pitch, cracked tar, petroleum residue, and so forth. Each in accordance with the type of product desired is manufactured in the sump phase (Building No. 335, 350, 355, 360) and/or in the gas phase (Building No. 331, 332, 340). The products which are manufactured in the sump phase (liquid phase) are, by contacting, stirred and mixed with the heavy oil accumulating
in the liquid phase, at 500-700 atm. This mixture is then heated and 227
charged to the high pressure chamber, in which, by the addition of highly compressed hydrogen, the products split or hydrogenize into light volatile compounds with low molecular weight. The liquid mixture of hydrocarbons which results is separated into light, medium and heavy hydrocarbons (Building No. 450, 451, 555, 556, 557, 558, 560, 561, 562, 563). The light and medium constituents go after eventual sulfuring to further treatment in the gas phase (the products are here worked into the gaseous state), while the high-boiling constituents are used for the mixing up of the raw products for the sump phase. The residues which accumulate in the sump phase are either centrifuged (Building No. 275) or distilled

(Building No. 276) and the hereby accumulating residue is either burned (Building No. 277) or treated in a low-temperature carbonization plant (Building No. 280), producing coke and heavy oil. The heavy oils are transferred back to the hydrogenation plant. Each, according to its characteristics, goes into the sump or gas phase.

The light or medium oils which accumulate in the sump phase are, with a suitable mixing of the proper reflux for the gas phase chamber, pressure charged into this vessel with an increase of the temperature, in which they are conducted in vapor form with hydrogen over a compact contacting material, whereby these products are again changed into light low-molecular chemical compounds, after which they are subjected to distillation (Building No. 540, 541, 542, 543, 550, 551, 552, 555) into benzine and medium oil. The benzine goes for further treatment to the wash (Building No. 537) and stabilization (Building No. 586, 539) and transferred as finished product to storage (Building No. 770), while the medium oils are recharged to the high-pressure apparatus, for conversion — exactly as described before — again into benzine.

The gases accumulating in this single working phase are either
charged to the cracking plant, in order to produce cracked gases with
the aid of double-decomposition with water vapor, as already indicated 228
above, and which is then treated further with hydrogen, or the gases are
compressed (Building No. 528) to yield propane, butane and pentane
(Building No. 530, 531, 532). The pentane is added to the accumulated
benzine; the propane or butane is mixed in the corresponding proportion,
in order to produce refined gas.

The finished products are benzines or gas oils. The benzines have a specific gravity between 0.7000-0.7500 and 0.750-0.830. The boiling curves of these benzines lie between 30 and 220.C. The gas oils produced have specific gravities of 0.830-0.890 and have a boiling curve of about 150-400 C. Propane and butane occur as secondary products, which are marketed either simply as propane or butane or in a mixture as a refined gas. The remains do not accumulate in the case of the hydrogenation.

The raw materials and intermediate products are piped from the raw material tanks (Building No. 690) in the tank farm (Building No. 660) and the intermediate tanks (Building No. 450, 770) to the finished-product tanks. From these finished-product tanks marketing products are piped either to the tank car loading rack (Building No. 657) on Street 7 or to the gravity flow (Building No. 60). The tank car loading rack consists of two loading spots with four or five spouts. On the gravity flow is found three loading spots with three spouts each.

Exact computation of yield concerning the finished products in the case of further treatment of the individual raw materials can still not be stated today for lack of experience.

A schematic description of the hydrogenation is enclosed.

In respect to the energy operation a heating gas plant (Building No. 153, 155, 156) is attached, whose low-temperature carbonization tar is jointly manufactured in the hydrogenation.

Explanation of the Building Numbers. 229

Building Number	
60	Warf building
128	Air blowers and pump house for the water gas plant.
129	Water-gas-generation plant with dumping station and inclined platform.
153	Heating gas plant with dumping station and inclined platform.
155	Disintegrator and blast house for the heating gas plant and two pump stations
156	Blower house for heating gas system
207	Hygas-cracking plant II
208	Hygas-cracking plant I
209	Blast house cracking plant
210	Organic sulfur rectification
212	Conversion plant
213	Tower rectification
230	Compression house
240	CO_2-wash
241	CO_2-wash with low-pressure framework
250	Outlet connection condensor.
269	Carbon drier and preliminary disintegrator
270	Carbon bunker
271	Carbon-grinding plant
275	Centrifuge
276	Washing-distillation
277	Washing-boiler house
280	Low temperature carbonization plant
331	Benzine chamber 6434 and 7019 No. 1 to 5
332	Benzine chamber 5058 No. 6 to 8
335	Sump phase chamber No. 11 to 16
340	Machine house-gas phase with instrument house
350	Circulatory gas wash with instrument house
355	Pump house-sump phase with instrument house
360	Pulp pressing house
450	Intermediate-production tanks for light and medium oils 230
451	Pump house for light and medium oils
470	Intermediate-production tanks for heavy oils
471	Pump house for heavy oils
528	Compressor house for hi-content gas
530	Linden plant (Extraction of benzine.)
531	Freezing plant
532	Lurgi wash
536	Pump house for stabilization plant
537	Benzine wash
539	Stabilization plant
540	C_1 and C_2 distillators

Explanation of the Building Numbers

Building Number	
541	Cold oil pump house for C_1 and C_2
542	Hot oil pump house for C_1
543	Hot oil pump house for C_2
550	B_2 and B_3 distillators
551	Cold oil pump house for B_2 and B_3
552	Hot oil pump house for B_3
553	Hot oil pump house for B_2
555	A_3 and B_1 distillators
556	Cold oil pump house for A_3 and B_1
557	Hot oil pump house for B_1
558	Hot oil pump house for A_3
560	A_1 and A_2 distillators
561	Cold oil pump house for A_1 and A_2
562	Hot oil pump house for A_2
563	Hot oil pump house for A_1
660	Extraction of propane
665	Gas blower
667	Benzine-emptying station into rail cars with two track balances
690	Cracking-residue storage tank warehouse
770	Marketing tank warehouse

B. Determination and Retention of the Operation Data 231

Entering raw materials are pumped from the ships and tank cars into the previously defined storage tanks. For each access a pump assignment must be written out, which goes to the controller of operations. Foreign raw materials are registered in the record book under part one. The raw material tanks are measured daily, so that the amount is always visible. The raw materials go then from event to event to manufacture. Exit of foreign raw materials is registered in the record book under part two.

The raw material goes in part to the chamber, in part directly to the distillation. The transfer is ascertained by means of an Eckardt meter and the products are run out of the chamber through the Eckardt meter to the intermediate tanks. The products which are run out of the distillation oven are measured with a coil balance, and the products which run out of the distillation apparatus into the intermediate tanks are measured with the Eckardt meter. Also, this count appears in hourly readings on the plant sheet. The resulting half product (benzine) is pumped to the finished product storage through the benzine wash and the stabilization plant. The entrance is again measured with the coil balance and the exit with the Eckardt meter, and retained on plant sheet. The residue which accumulates with the distillation is run again to the intermediate tanks and the chamber; and in the case of the yield from distillator is likewise measured with the Eckardt meter and noted on the plant sheet. The gases which accumulate in the process are collected in the gas meter, liquified under pressure and separated. The liquid propane and butane are pumped into the refined-gas-storage tank and mixed up to refined gas in a mixing receptacle. The refined gas is pumped to loading stations and loaded on the balances into cylinders. A part of the refined gas is loaded into tank cars and its weight ascertained on the track balance.

The benzine produced at the stabilization plant is transferred to the preliminary tanks No. 201 to 204. For each access a pump assignment is exhibited and the permitted amount is drawn off into tank cars. From the preliminary tanks, the benzine is pumped after preliminary test into the finished product tanks 213, 215, 217 and 220. A pump assignment is exhibited and the permitted amount entered in the tank card index and the operations book under part one. After the release, the benzine itself is drawn off. In the case of tank car loading, the weight is determined on the track balance. In the case of ship loading, the weight is determined out of the initial tank, and the tank is measured before and after the filling of the ship by the controller of technical operations, the temperature is measured and the specific weight is determined. The exportation is entered into the tank card index and the operations book under part two. Besides, there are listed in part two of the operations book samples which go abroad. Samples which are used for maintenance of production operations in the laboratories and testing stages appear in the operations book under part three. For each outgoing shipment is exhibited a shipment announcement and entered in the exportation book, separating according to quality. 252

The finished product tanks are measured daily and the amount entered in the measurement report.

C. Task Range of the Operation Control 253

By means of an automatic recording measurement installation, the division recording is obtained, with whose help as far as possible, are set up energy and material balance sheets, daily for the individual operations and monthly for the whole process. It arrests on the basis of this daily or monthly objective and quantitative compilation any irregularity in the conduct of the operation and makes possible thus a far-reaching certainty and economy in production. The balance sheet, which is produced, furnishes the necessary data for the calculation of the individual intermediate products and the finished products. The removable diagrams give the proper values for each point of time, which can produce, with the help of the planimeters, the summary values necessary for the balance sheet.

INNER ORGANIZATION OF THE OPERATION CONTROL

Under the direction of technical physicists there are, in Building 315, the following divisions, in order:

a. a workshop for the erection, maintenance, and repair of all measuring apparatus and regulating devices, and further for testing and calibrating them before their installation or re-installation into the plant. The workshop has annexed a division for the storage of reserve measuring apparatus with spare parts. The organization of the calibration of fixed measuring apparatus into regular time intervals is therefore of greatest importance.

b. a laboratory for the calibrating and testing of measuring instruments, the testing and calibrating of recording analytical apparatus, the carrying through of tests and the building of new apparatus.

c. a commercial office for the valuation of the instrument readings, the compilation of the results of measurement and the formation of daily and monthly balance sheets for foremen and superintendents. 234

d. a technical office for the control of measuring apparatus for the securing of their accuracy of measurement, the plans of new apparatus, the preparation of calibration curves, and the control of the quantitative measurement of gas and other products. An important task of the technical personnel exists, besides, in the composition, supervision and maintenance of the operation and control curves.

e. a group of technical auxiliaries designated as "field service", who attend to the maintenance of the control and calibrating apparatus within the plant.

One obtains a picture of the magnitude of this division, when he considers that the portion of the cost of the measuring apparatus lies on the average at about 5 per cent of the total capital value. In practice an accuracy of measurement as high as possible must be constantly striven for, and lies in most cases at the average of ± 1 per cent. In order to reach this goal, it is necessary to constantly carry through with care the removal of pollution and the installation of refiners, filters and pressure regulators for the unexceptionable operation of the analytical-calibrating and measuring apparatus.

THE COMMERCIAL CALCULATING OFFICE

For the preparation and supervision of all compilation concerning products and energy motion, and for the planimetering of the exhauctive diagrams (all the ones on gas types, water, vapor, safety gas, heavy and light oils, and finished products) there are technical commercial personnel. In the case of all chief measurements necessary for the balance sheet, for example, of gases, there are recorded near the registered quantitative value, the values of pressure, temperature, heat content, and density necessary for computation. In our work there are already today a hundred recorded quantitative measurements in operation. The amount is measured by registration of the working pressure which forms with the installation of the measuring orifice and the measuring diaphragm. With all chief measurements, the necessary moderating space amounts to 10-15 D before the throttle diaphragm and about 5 D after the throttle diaphragm (D = diameter of tube). All recording quantitative measuring apparatus, for example, the so-called coil balance, are calibrated as exactly as possible and tested on unexceptionable operating conditions. In the case of all important quantitative steam and water measurements, the prevailing pressures and temperatures are continually measured. All measurement dials carry, on the outer side, the exact operation data (Boring, notation if orifice or diaphragm). In practice 235 this often varying data is written upon specific form sheets and can be easily controlled and tried out. The measurement of the amount of products passing in the operation (heavy and light oils, benzine and the finished products) results with the help of the Eckardt meter and the oval rod counter. For our work there are approximately 140 such meters in operation. The readings are without exception both from the factory and the outer service of the operations control recorded hourly in a special book. The correction data of each meter have been determined from the yield and are kept in mind. The weight is ascertained out of the amount by means of the exact establishment of the specific gravity. The specific gravity is thereby determined, that in an Eckardt meter a so-called sample is installed, which is in each case the removal of a small amount (about 10 cm^3) from the chief product, which is collected in a special flask. By measurement of the entering and exiting amount the existing loss can always be determined. The finished products are likewise measured by the Eckardt meter, however, at the same time there results a continuous control through the reading of the tank level of the concerned tank. All tank levels are followed up continually in a central observatory. All measured values are constantly brought together in a book. The plant itself continually supplies pump information, so that the accuracy of the measurement can be tested as quickly as possible. All introduction into the works is controlled by testing incoming tank cars. Out of all the named records finally is formed the numbers which are necessary for the control of the production. These numbers give a picture of the material and energy flow and yield the base for economy and calculation accounts. Therefore, if the operations control can make real and uninfluenced their classification on the basis of measurements, this division is placed directly under the technical transmission of work.

Development of the Plant at Stettin-Poelitz

Working Point	Capacity (in aggregate)	Construction Firm	Max. No. of Cases	Investigation No.
High Pressure:				
Compressor 325 atm.	16200m³/hr crude gas = 10900m³/hr refined gas	Schwartzkopff Borsig Khrh. u. Schmer M. Esslingen	15	7 2 (+1 Stettin III) 1 5
Additional Compressor 700 atm.	10250m³/hr refined gas	Borsig	(10)9	9 (+1 Stettin III)
Sump Phase:				
Pulp Press	10m³/hr	Wumag M. Esslingen Schwartzkopff	24	17 8 (+6 Stettin III) 2
Drilling Pump	12m³/hr	M. Esslingen	13	13
Washing Oil Press	4m³/hr	Wumag	15	15 (+1 Stettin III)
Water Pump	5m³/hr	Balcke	7	7 (+1 Stettin III)
Hot Circulation Pump	10m³/hr	M. Esslingen Wumag	8	4 1
Feeder Pump	250m³/hr	Halberg	8	8
Chamber Oven	18/15 m 1000 ∅	Krupp and DHHV	6(0000)	24 (+4 Stettin III) (+5 Res.)
Chamber Regulator	18 m 600 ∅	PWR, Krupp and DHHV	6 x 3	18 (+3 Stettin III) (+2 Res.)
Chamber Washer	12 m 1000 ∅	Krupp	5	5
Gas Circulation Pump	57000m³/hr	Schwartzkopff Halberg	7	7 1 (+2 Stettin III)
Expansion Mach.	65m³/hr	Leuna	4	4
Additional Pressing Pump	12.5m³/hr	M. Esslingen	5	5
Feeder Pump	120m³/hr	Halberg	4	4
Driving Oil Circuit	240m³/hr	Halberg	15	15 (+[illegible] Stettin III)

Development of the Plant at Stettin-Poelitz (continued)

Working Point	Capacity (in aggregate)	Construction Firm	Max. No. of Cases	Investigation No.
Sump Phase (cont'd)				
Necessary Towers	3 Ka. content	Eisenwerk Kaiserslautern	2	2
Gas Phase:				
Pre-hydrogenation				
Injection Pump	25m^3/hr	M. Esslingen	6	6
Injection Pump	3m^3/hr	Balcke	3	3
Water Pump	6m^3/hr	Balcke	2	2
Gas Circ. Pump	75000m^3/hr	Schwartzkopff	5	5
Chamber Oven	18 m 1000 ø	DHHV	3(0000)	17
Pre-hydrogenation Chamber Regulator	18 m 600 ø	PWR, Bock. Verein, DHHV	3 x 3	1
Benzine-Injection Pump	25m^3/hr	M. Esslingen	6	6
Injection Pump	6m^3/hr	Balcke	3	3
Water Pump	6m^3/hr	Balcke	3	3
Gas Circ. Pump	75000m^3/hr	Schwartzkopff	3	4
Chamber Oven	18 m 1000 ø	DHHV	5(000)	13
Chamber Regulator	18 m 600 ø	PWR and DHHV	5 x 2	7
ET 110-Injection Pump	2.5m^3/hr	M. Esslingen	(2)	2
Chamber Oven	12 m 500 ø	PWR	(2(0))	1
Chamber Regulator	12 m 500 ø	PWR	(2 x 1)	1
A-Carbon Plant	1365m^3/hr	Bamag	(1)	1
Dehydrogenation:				
DHD I.-Injection Pump	12m^3/hr	Balcke	-	4
Water Pump	1m^3/hr	Fitzmann u. Pfeiffer	-	2
Gas. Circ. Pump	25700m^3/hr	Halberg	-	3

Development of the Plant at Stettin-Poelitz (continued)

Working Point	Capacity (in aggregate)	Construction Firm	Max. No. of Cases	Investigation No.
Dehydrogenation (cont'd)				
Air Compressor	1600m^3/hr	Halberg	-	2
Chamber Oven	8.8 m 1600 ø	D. Röhrenw	(2(00000))	10
Chamber Regulator	12 m 500 ø	D. Röhrenw	(2 x 2)	4
DHD II.-Injection Pump	33m^3/hr	M. Esslingen	-	3
Gas Circ. Pump	30700m^3/hr	Halberg	-	4
Chamber Oven	10/12 m 1400 ø	Wittkowitz	(2(000000))	12
Chamber Regulator	12 m 600 ø	Wittkowitz	(2 x 3)	6
Air Compressor	2000m^3/hr	Borsig	-	1
Water Pump	3.6m^3/hr	Fitzmann u. Pfeiffer	-	2
Injection Pump	12m^3/hr	Balcke	-	1
Low Pressure:				
Carbon Preparation:	(mt = metric tons)			
Initial Grinding-				
-Hammering Mill	400mt/hr	Krupp, Magdeburg	2	2
-Rolling Mach.	100mt/hr	Krupp, Magdeburg	2	2
Pulverizing-				
-Hammering Mill	80-120mt/hr	Krupp, Magdeburg	2	2 + 2
-Centrifuge Mill	60mt/hr	Krupp, Magdeburg	2	2
Drier	35-40mt/hr	Büttner	2	3
Filling Pumps	30mt/hr	Klaud. Peters	3	4
Mixture Conveyer	30mt/hr	Möller	-	1
Vibrating Strainer	20mt/hr	Krupp, Magdeburg	3	4 + 2
Rolling Mach.	6mt/hr	Krupp, Magdeburg	3	4 + 4
Concentrate Mill	16m^3/hr	Krupp, Magdeburg	3	4 + 2

Development of the Plant at Stettin-Poelitz (continued)

Working Point	Capacity (in aggregate)	Construction Firm	Max. No. of Cases	Investigation No.
Low Pressure (cont'd)				
Residue Preparation:				
Centrifuge	2mt/hr	Bergedorfer Eisenwerke	36	44 + 8
Low-temperature Carbonizing Oven	2mt/hr	Krupp	(10)8	10 + 2
Take-off	3mt/hr	Still	1	1
Pitch-Melting	5mt/hr	HWT	(?)	2
Distillation:				242
A-Distiller	50mt/hr	Koppers	5	3
B-Distiller	38mt/hr	Koppers	5	3
C-Distiller	50mt/hr	Still	2	2
Stabilizing Plant	30mt/hr	Koppers	2	2
Stabilizing Plant	50mt/hr	Port Jérome	-	(+2)
Benzine Wash	50mt/hr	Koppers	2	2
Benzine Wash	50mt/hr	Port Jérome	-	(+1)
Debenzine	7500m^3/hr	Linde	5	3
DHD-predistilled	28mt/hr	Koppers	1	1 + 1
DHD-redistilled	17mt/hr	Koppers	1	1 + 1
Benzine Refinery	60mt/hr	Port Jérome	-	1
Refined Gas Wash	6mt/hr	Lurgi	3	4
Propane Wash	6mt/hr	Lurgi	-	1
Cracking Distiller	1000m^3/hr	Linde	-	1
Gas Production:				
Cracking Oven	9000m^3 pure H_2	Bamag	12	12
Water Gas Generation	6100m^3 pure H_2	Pintsch	4	10
Conversion System	7000m^3 mixed gas	Bamag	24	24

Development of the Plant at Stettin-Poelitz (continued)

Working Point	Capacity (in aggregate)	Construction Firm	Max. No. of Cases	Investigation No.
Gas Production (cont'd)				
CO_2 Wash (26 atm.)	12000m^3 pure H_2	G.H.H.	11	11
CO_2 Rewash	-	Wilke Werke	-	2
CO_2 Injection Pump	1450m^3/hr	Ehrh. and Schmer	7	8
CO_2 Injection Pump	750m^3/hr	Ehrh. and Schmer	4	3
Expension-Centrifuge	1500m^3/hr	Voith	9	9
Rewash Pump	2000m^3/hr	Amag Hilpert	-	2
CO Wash (325 atm.)	30000m^3 pure H_2	Dortm. Union	7	7
CO Expansion Mach.	120m^3/hr	Leuna	6	6
CO Super Press Pump	25m^3/hr	M. Esslingen	6	6
CO Feeder Pump	150m^3/hr	Hacker	5	5
CO Feeder Pump	100m^3/hr	Weise Söhne	5	5
Safety Gas Plant:				
Inert Gas Plant	500m^3/hr	Bamag	2	2
Inert Gas Plant	1000m^3/hr	Bamag	1	1
N_2 Compressor (700 atm.)	1000m^3/hr	M. Sürth	2	2
CO_2 Compressor (4 atm.)	4000m^3/hr	Demag Balcke	4	2 2 (+1)
Energy Operation:				
Fuel Gas Generator	5.6 x 10 heat units	Bamag		243
Steam Tank	92mt/hr	MAN (1-11) Dürr (12,13)	8	13
Waste Heat Tank (Cracking Plant 20 atm.)	4.2mt/hr	Oschatz	12	12
Waste Heat Tank (Cracking Plant 4 atm.)	4mt/hr	Dr. Otto and Co.	2	2
Turbo-generators	24000 KVA	Siemmens (5) Brünner Mach. (3)	5	8

Development of the Plant at Stettin-Poelitz (continued)

Working Point	Capacity (in aggregate)	Construction Firm	Max. No. of Cases	Investigation No.	
Preparations:					
Phenol-containing-Waste-Water I	25m^3/hr H_2O 0.13mt/hr OExtract	Uhde	1	1	
Phenol-containing-Waste-Water II	100m^3/hr H_2O 1.5mt/hr Extract	Uhde	1	1	
Alkazide-Washer:					
1) No Water Gas	20000m^3/hr	Bamag	1	3 (±1)	
2) Gas-Poor	13000m^3/hr	Dr. Otto and Company	2	2 (±2)	
3) Gas-Rich	6000m^3/hr	Dr. Otto and Company	4	4 (±1)	
Claus-Contact System	850m^3/hr	Dr. Otto and Company	2	2	
Supplementary Combustion Plant	9000m^3/hr	Lurgi	1	2	
Mass Catalyser	-	Lurgi	1	1	
H_2SO_4 Concentrate	0.2mt/hr SO_3	Uhde	6	4	
T. 52:					
Butyl-Oven	0.5-0.8mt/hr	Uhde	5	5	
Regular-Oven	-	Uhde	3	3	
Compresser (10 atm.)	3300m^3/hr	Borsig	4	4	
Oil Wash	1800m^3/hr	Bamag	1	1	
Stabilizing Columns (20 atm.)	15mt/hr	Borsig	1	1	
Polymer Towers (50 atm.)	15mt/hr	Uhde	8	8	
C_4-Columns	15mt/hr	Borsig	1	1	
Benzine Columns	5mt/hr	Borsig	1	1	
AT. 244:					
Butyl-Oven	0.5mt/hr Olefine	Uhde	5	5	244
Regular-Oven	-	Uhde	3	3	
Compression-Product Gas	3300m^3/hr	Borsig	5	4	

Development of the Plant at Stettin-Poelitz (continued)

Working Point	Capacity (in aggregate)	Construction Firm	Max. No. of Cases	Investigation No.
AT. 244: (cont'd)				
Compression-Cool Butane	5300m^3/hr	Borsig	7	5
Oil Wash	3600m^3/hr	Bamag	1	1
Mixed Plant-Raw Materials	0.7mt/hr Benzine	Uhde	12	12
Wash	45mt/hr Benzine	Uhde	2	2
Depropanizing	59mt/hr Benzine	Borsig	1	1
Butane-Columns	60mt/hr Benzine	Borsig	1	1
n/l-Columns	20mt/hr Benzine	Borsig	2	2
Benzine Stabilization	10mt/hr Benzine	Borsig	1	1
Benzine Distillation	15mt/hr Benzine	Borsig	1	1

Report 578-46R

Pölitz February 15, 1943

(MARKED CONFIDENTIAL)

THE WORKING OF UPPER SILESIAN COAL CALCULATED ON 1000 GM PURE COAL

Frame 249

Translation

1000 kg	pure coal (81.5 % C 4 g H dispersed)	
75 kg	ash (7% ash, - K_0	
44 kg	contact (2.4% $^{o}FeSO_4$ ° 7 aq + 1.7% Luxmasse) (1)	(T K_0)
71 kg	water	
1190 kg	Crude Coal + Catalyst 1 and 11	

CRUSHING AND DRYING

997 kg	pure coal (loss: 50 kg H_2O + 3 kg pure coal (2)
75 kg	ash
39 kg	contact (30 kg dry 1 and 11 + 9 kg Catalyst 111)
26 kg	water
1137 kg	T K_2 + Catalyst

(1) About 50 per cent H_2O in Fe SO_4 ·7 ag and 10 per cent H_2O in Luxmasse

(2) Loss for complete process from bin to pressure chamber

Frame 249 (continued)

1250 kg oil ground in (9% solids = 113 kg) heavy slurry mixture added
12 kg loss (0.5%) (eventually forming new solids)
1200 kg heavy slurry (about 51.5% liquid +)

1175 kg heavy slurry
450 kg oil ground in (9% = 40 kg liquid) thin slurry addition
1625 kg thin slurry

1625 kg thin slurry (about 40% F)
55 kg oil rinse
2880 kg high pressure addition (2825 kg coal slurry concentrate about 45%)
High pressure 700 atm.: 94% decomposition 24% gas or liquid products; output .23.

803 kg C introduced (12 kg = 0.5%)
755 kg C decomposed
181 kg C vaporized
13 kg C lost (15 kg high pressure loss = 0.5% of material entering)
561 kg C = 648 kg high pressure oil produced (86.5% C)

7 kg distillate lost
58 kg residue lost (10 + 10 + 38)
583 kg volatile oil produced

75 ash
39 catalyst
52 residue (92% C)
153 solids oil ground in
391 solids
960 occluded oil
1279 occluded 25% liquid
857 thinning oil

CENTRIFUGING

2136 mixture 15.4% liquid
= 3 loss (3) (+ 7 newly formed liquids)
+ 1700 centrifute oil (153 liquid)
+ 433 residue (173 liquid)

(3) Mixture should contain 15% solids. Found 15.5% solids

Frame 249 (continued)

433 Centrifuge residue (40% liquid)
6 Catalyst
24 Spindle oil

Low temperature work: 82% yield
179 solids
38 coke
192 + 24 low temperature oil (T)
20 after cooline
10 gbs and loss

DISTILLATION

1290 kg stripper
20 kg oil after cooling
1310 added to distillate
7 lost
583 gasoline + middle oil
55 thinning oil
685 spindle oil (T)

Product factor 1.72 raw coal per metric ton of oil produced dated (stamped) February 16, 1943.

Stettin-Pölitz June 11, 1942

CALCULATION FOR FUEL OIL FROM COAL

Frame 252

Translation-

Proceding from the present price of 432 Rm (Reichsmarks) /metric ton V T 708 (First quarter of year 1942) end profit of 100 Rm/metric ton propelling gas (that is a cost of 100 RM and a savings of 250 RM).

Basis for these figures: Factor for yield of fuel oil to light gasoline + propelling gas = 1 to 0.77 + 0.1. That is instead of one metric ton fuel-oil, 770 kg light gasoline and 100 kg propelling gas may be used. H_2 requirement: 1200 meters H_2/metric ton fuel oil and 5200 cubic meters H_2 /metric ton light gasoline.

(T) Translator's note: Original German has a line running back to "857 thinning oil" prior to "Centrifuging"; see above.

Report 578-46R

Poelitz Hydrogenation Plant, A. G. Stettin-Poelitz, December 17, 1941

BALANCE FOR GASOLINE AND MIDDLE-OIL FRACTIONS

Frame 256

Translation

Time	Raw Coal	Metric Tons Coal %0	Metric Tons Coal %2	Spindle Oil (excess or difference)	Gasoline +Middle-Oil +Gasified Material	Gasified Material +Gasoline +Middle-oil +Tar	Fresh Gas Gasoline Middle-Oil	Fresh gas Gasoline + Middle-Oil +Tar
September	2,656	2,832 5.2	2,931 3.4	+ 72.9	41.8	33.1	2,696	2,345
October	2,096	2,242 5.53	2,323 3.48	- 36.8	42.2	34.1	3,028	2,657
November	2,006	2,133 5.97	2,209 3.3	- 43.9	42.2	34.4	3,202	2,825

Lowest requirement of H_2:

3200 x 0.77 - 1200 = 1250
Meters H_2/metric ton fuel oil

Saving for 770 kg light gasoline,	332.8 RM
Obtained per 100 kg fuel oil,	10.0
	342.8 RM

~~Minimum requirement H_2: 1250 cubic meters at 5.07 pfennig = 63.40 RM~~
Gases under high pressure: oxygen: 0.1208 x .77 =
0.0930 Metric tons at 5.08 RM = 0.45

Minimum requirement

Gases under low pressure: Oxygen 0.749 x 0.77 = 0.5767 Metric tons at 4.49 RM =	2.60 RM
Hot gases 0.739 x 10^6 x 0.77 = 0.569 x 10^6 heat units at RM 7.09 =	4.00 RM
Water: 71.55 x 0.77 = 55.09 Cubic Meters at 1.333 pfennig =	0.75 RM
Electrical energy 227 x 0.77 = 174.7 kilowatt hour at 1.832 pfennig =	3.20 RM
	74.40
	342.80
	74.40
	268.40 RM
License, differences ----------------------------------(Minus)	3.30 RM
	265.10 RM

These calculations are based on the conditions prevailing in Pölitz, are especially affected by the fact that the vapor phase capacity is not completely utilized because of a deficiency in the sump (liquid phase) middle oil, while salaries and expenses continue on.

The prices for H_2, high density and low density liquids, water, hot gases and electrical energy for first quarter of 1942 are used for these calculations. The energy used per metric ton of light gasoline in the gaseous phase is that energy actually used during the last half of the year 1941 in Pölitz.

The license for fuel oil is assumed to be 3.00 RM.

Pölitz February 15, 1943

Frame 288

Translation

Frame 288 is identical with frame 249. Frame 288 is almost illegible.

EXPLANATION OF HYDROGENATION-PLANT ACCOUNTING

Frames 289-361

Translation

Every accounting ("Kalkulation") should be along the lines and rules set up by the management. Within the bounds of these rules, the accounting must be adapted to the process whose costs are to be ascertained. An accounting scheme set up for hydrogenation is explained in the following. 290

I. ACCOUNTING PROBLEMS

The scope of the accounting depends upon the problems involved. The less that is required, the shorter can the accounting be, and vice versa. In general, the following three things are required for hydrogenation:

1. Designation of the most important cost factors (yield, hydrogen consumption, etc.).

2. Separate evaluations for the parts of single product produced simultaneously from different raw materials (coal, tar, etc.).

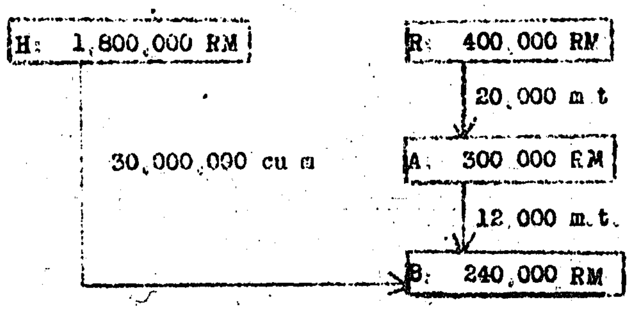
3. Separate evaluations for different products produced simultaneously (gasoline, diesel oil, etc.).

II. SOLUTION OF ACCOUNTING PROBLEMS FOR SIMPLE CASES

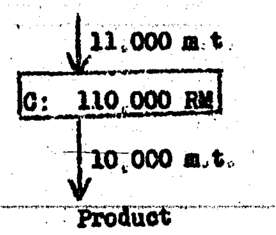
The following simple cases show how the above-mentioned accounting problems can be solved. Although the proportions are much simplified, the essentials are retained. It is assumed that the total cost is properly approportioned among the hydrogenation operations (cost points), this operating cost-accounting problem is not further considered, for it follows general cost-accounting lines. It is appropriate to add the costs for auxiliary materials (catalysts, etc.) to the operating costs. In contrast, it is necessary to obtain the hydrogen cost separately and to carry it independently (as if resulting from an operation other than the hydrogenation itself). In contrast to the operating costs, the hydrogen cost cannot be spread over the various operations or cost points (in this case the furnace chambers) inasmuch as different raw materials can consume different amounts of hydrogen at the same furnace-chamber throughput. Furthermore, setting forth the hydrogen cost separately is especially appropriate because this cost forms a large part of the total cost, this is similarly true for Hy-gas. For simplicity, the following is for only three operations (A, B, C) and for hydrogen (H, as if resulting from an operation other than the hydrogenation itself).

1. Case I. One Raw Material, One Product, No Change in Supply of Intermediates 291

Flow diagram:



Flow diagram: (continued)



An especially simple case is set forth in this flow diagram. The figures in the rectangles give the monthly costs in Reichmarks (RM) for raw material for hydrogen, and for the various operations; the other figures give the amounts of the charges and of the products in metric tons (m.t.) per month. The accounting can be carried out in the easily understood manner given below.

	Per month			Per m.t. product	
	Amount, m. t.	RM/m.t.	RM	Amount, m.t.	RM
Raw Material	20,000	20	400,000	2.0	40
Hydrogen, H	30,000,000[a]	60[b]	1,800,000	3,000[a]	180
A	20,000	15	300,000	2.0	30
B	12,000	20	240,000	1.2	24
C	11,000	10	110,000	1.1	11
Mfg. cost = H+A+B+C			2,450,000		245
Total or product	10,000	285	2,850,000	1.0	285

a. Cubic Meters (cu. m.).
b. Per 1000 cu. m.

This accounting scheme contains all the quantities needed for figuring the total cost or value of the product. These quantities are broken down according to raw material, hydrogen consumption, and the separate operations, and also according to amounts and to prices. Moreover, the costs are based on metric tons of charge or on cubic meters of hydrogen. It can be readily recognized, for example, whether an increase in the hydrogen cost is caused by a higher hydrogen consumption or 292
by a higher price for hydrogen. If desired, the costs can be broken down further into labor, utilities, and so on, as far as these are known for the individual operations or for the hydrogen. The first requirement, "designation of the most important cost factors," is thus completely fulfilled.

It is advantageous to change, purely formally, the above accounting scheme when the accounting involves a large number of raw materials, intermediates, and products. Illustrating this principle, this will be shown for the above simple case, even though it seems trivial. The new representation takes place in the following four steps:

Step 1. Yield account, on raw-material basis:

	Per m.t. raw material	Total
Hydrogen, cu.m.	1,500	30,000,000
Charge to A, m.t.	1.00	20,000
Charge to B, m.t.	0.60	12,000
Charge to C, m.t.	0.55	11,000
Product, m.t.	0.50	10,000

In contrast to the former representation, the specific values are referred to one metric ton of raw material instead of to one metric ton of product. This is often advantageous.

Step 2. Account of specific costs per unit of charge:

	m.t. charged or cu.m. consumed	RM/m.t. or RM/1000 cu.m.	RM/month
Hydrogen	30,000,000	60	1,800,000
Operation A	20,000	15	300,000
Operation B	12,000	20	240,000
Operation C	11,000	10	110,000
Mfg. cost			2,450,000

Step 3. Account of manufacturing costs per metric ton of raw material (from Steps 1 and 2):

	RM/m.t. or RM/1000 cu.m.	Per m.t. raw material	
		m.t. or cu.m.	RM
Hydrogen	60	1,500	90
Operation A	15	1.00	15
Operation B	20	0.60	12
Operation C	10	0.55	5.50
Mfg. cost			122.50

Check: Total mfg. cost = 20,000 x 122.50 = 2,450,000 RM.

Step 4. Account of total cost (value of product):

The following relation holds: total cost = raw material cost + manufacturing cost. Accordingly,

Raw material charged			Mfg. cost	Product		
m.t.	RM/m.t.	RM	RM	m.t.	RM/m.t.	RM
20,000	20	400,000	2,450,000	10,000	285	2,850,000

The result is the same as before. Further analysis of this simple account will 293
be given later; let it be anticipated only that the first step, the yield account, is separated by itself because this is generally obtained not from the main accounting but from the operation balance.

For the example being carried out, the intermediates can also be indicated, as is often necessary (e.g., in taking inventory and in making deliveries). If, for example, an intermediate leaving operation B (11,000 m.t. per month) is to be evaluated, it is necessary only to ascertain the manufacturing cost up to and including operation B, and then obviously

Raw material	= 400,000 RM
Mfg. cost = H+A+B	= 2,340,000 RM
Value of intermediate	= 2,740,000 RM for 11,000 m.t.

or 2,740,000/11,000 = 249.09 RM/m.t. The same result is obtained if from the cost or value of the product is deducted the cost of operation C (i.e., the manufacturing cost needed to convert the intermediate to the product); thus

Product	2,850,000 RM
Mfg. cost to convert intermediate to product	- 110,000 RM
Value of intermediate	2,740,000 RM

for 11,000 m.t., or 2,740,000/11,000 = 249.09 RM/m.t. This obvious way of evaluating an intermediate from the product is noteworthy because in most instances it is the only possible way (see Case III).

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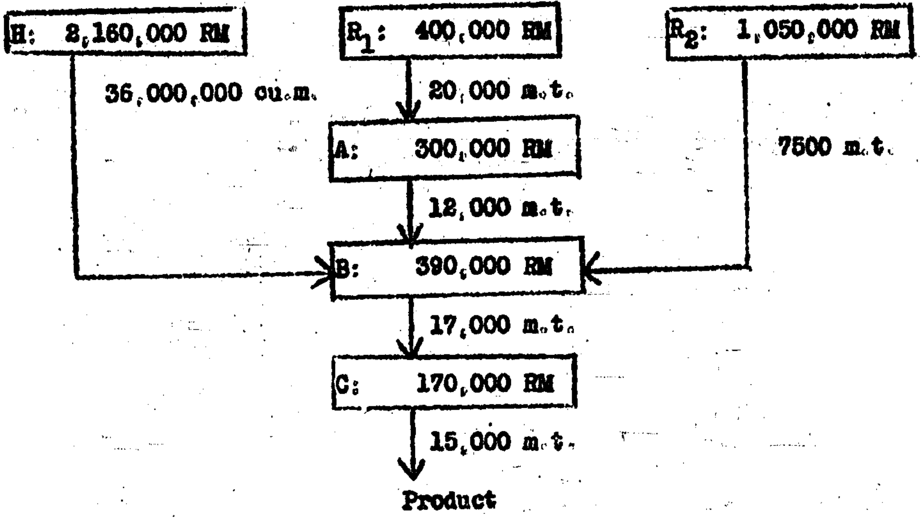
2. Case II: Different Raw Materials for a Common Processing.

Flow diagram:

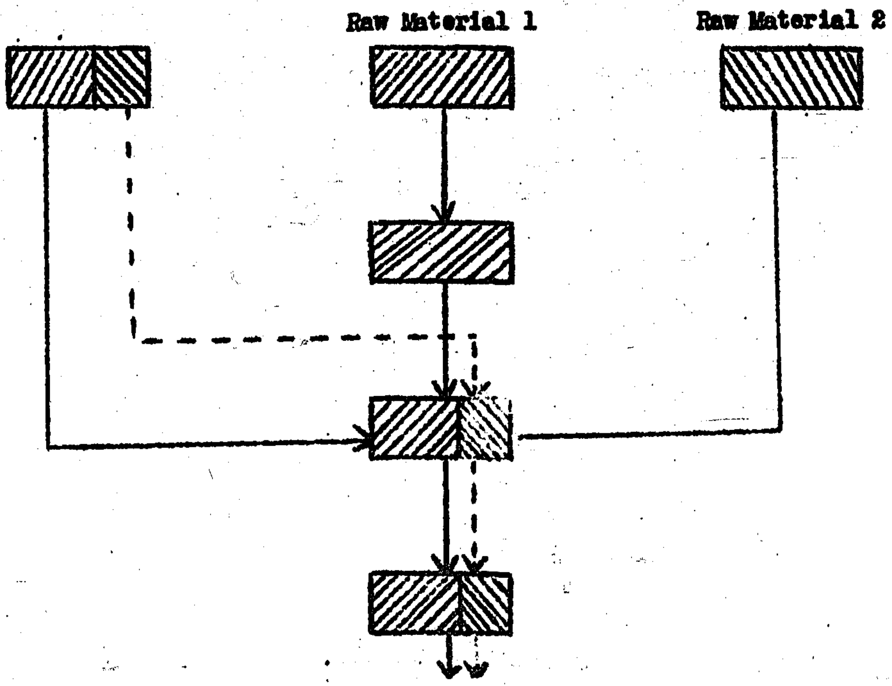


If now two raw materials, one introduced into operation A and the other into operation B, are processed simultaneously, the accounting might again be as in the first scheme in Case I:

	Per month			Per m.t. product	
	Amount, m.t.	RM/m.t.	RM	Amount, m.t.	RM
Raw Material 1	20,000	20	400,000	1.333	26.67
Raw Material 2	7,500	140	1,050,000	0.500	70.00
Total raw materials	27,500	52.73	1,450,000	1.833	96.67
Hydrogen, H	36,000,000[a]	60[b]	2,160,000	2,400[a]	144.00
A	20,000	15	300,000	1.333	20.00
B	19,500	20	390,000	1.300	26.00
C	17,000	10	170,000	1.133	11.33
H+A+B+C			3,020,000		201.33
Total	15,000		4,470,000	1.000	298.00

a. Cubic meters
b. Per 1000 cu.m.

However, the value, 298.00 RM/m.t. product, is a "mixed" value, whereas it is desired to know separate values for the parts of the product from raw material 1 and from raw material 2. In order to determine these values, the load imposed by each raw material upon the operations must be known, i.e., how much hydrogen is consumed by each raw material. This is schematically indicated in the following flow diagram, in which the raw materials are differentiated by different shadings:



If there were such a separation, it would be simple to make a separate accounting for each individual raw material. But it must be emphasized that the important thing in this case is the correct breaking down of the value. Consequently, making separate accountings will be omitted, and the accounting will proceed at once according to the second scheme.

Step 1. Yield account:

	Per metric ton		
	Raw Material 1 (20,000 m.t.)	Raw Material 2 (7,500 m.t.)	Total (Check)[a]
Hydrogen, cu.m.	1,500	800	36,000,000
Charge to A, m.t.	1.000	---	20,000
Charge to B, m.t.	0.600	1.000	19,500
Charge to C, m.t.	0.550	0.800	17,000
Product, m.t.	0.500	0.667	15,000

[a] Example: 1,500 x 20,000 + 800 x 7,500 = 36,000,000

Step 2. Account of specific costs per unit of charge:

	m.t. Charged or cu.m. Consumed	RM/m.t. or RM/1000 cu.m.	RM/month
Hydrogen	36,000,000	60	2,160,000
Operation A	20,000	15	300,000
Operation B	19,500	20	390,000
Operation C	17,000	10	170,000
Mfg. cost			3,020,000

Step 3. Account of manufacturing costs per metric ton of raw material:

	RM/m.t. or RM/1000 cu.m.	Per m.t. raw material 1		Per m.t. raw material 2	
		m.t. or cu.m.	RM	m.t. or cu.m.	RM
Hydrogen	60	1,500	90	800	48
Operation A	15	1.00	15	--	--
Operation B	20	0.60	12	1.00	20
Operation C	10	0.55	5.50	0.80	8
Mfg. cost			122.50		76

Check:

20,000 x 122.50 = 2,450,000 RM
1,500 x 76.00 = 570,000 RM
3,020,000 RM

Step 4. Account of total cost:

	Charge			Mfg. cost,	Product		
	m.t.	RM/m.t.	RM	RM	m.t.[a]	RM/m.t.	RM
Raw material 1	20,000	20	400,000	2,450,000	10,000	285	2,850,000
Raw material 2	7,500	140	1,050,000	570,000	5,000	324	1,620,000
Total	27,500		1,450,000	3,020,000	15,000	298	4,470,000

[a] Calculated from values in Step 1: 20,000 x 0.500 = 10,000; 7,500 x 0.667 = 5,000.

This account gives directly separate values for the parts of the product from 297 raw material 1 and from raw material 2; the mixed value (298 RM/m.t.) agrees with that previously obtained. However, the labor involved in this four-step accounting is scarcely any more than in that of making a separate accounting for each raw material.

A variation of this case arises when one of the raw materials is replaced by an intermediate; if the intermediate has the same characteristics as raw material 2, it will have the same manufacturing cost. However, the value of this intermediate is not known and should first be ascertained; it can be simply calculated from the fact that the product from the intermediate has the same value as from the raw material (in this case, 285 RM/m.t.):

	Amount, m.t.	RM/m.t.	RM
Product	5,000	285	1,425,000
Mfg. cost from intermediate			- 570,000
Intermediate	7,500	114	855,000

Alternatively, this calculating can be done directly in Step 4:

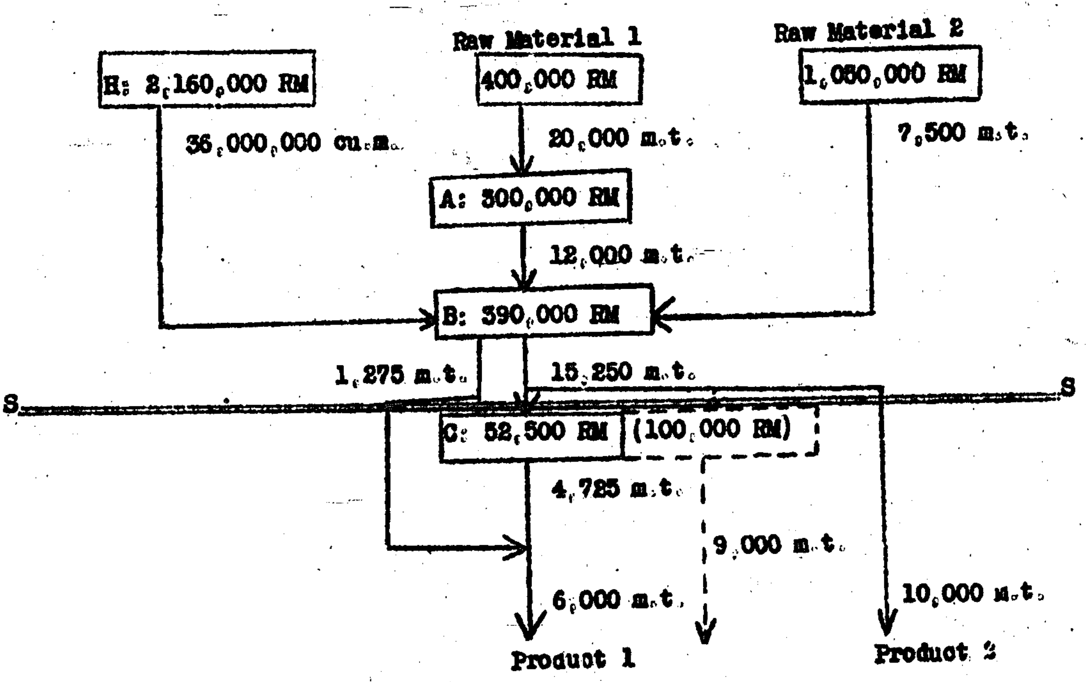
	Charge			Mfg. cost	Product		
	m.t.	RM/m.t.	RM	RM	m.t.	RM/m.t.	RM
Raw material 1	20,000	20	400,000	2,450,000	10,000	285	2,850,000
Intermediate	7,500	114	855,000	570,000	5,000	285	1,425,000
Total	27,500		1,255,000	3,020,000	15,000	285	4,275,000

For raw material 1, the account proceeds in the usual manner from left to right and gives the product value of 285 RM/m.t. This same value is inserted for the product from the intermediate, and the value of the intermediate is calculated by proceeding from right to left. The scheme can be carried over directly to additional intermediates and determination of their values, so that any number of raw materials or intermediates can be present.

The second requirement, "separate evaluations for the parts of a single product produced simultaneously from different raw materials" is thus fulfilled.

3. Case III. Different Products from Simultaneous Production, or Evaluation of Withdrawn Intermediates. 298

Flow diagram:



Going beyond Case II, it is now assumed that before operation C 10,000 m.t. of intermediate is withdrawn as a product (e.g., B-middle oil = Diesel oil, or crude gasoline = DHD fore-product). Besides, it is assumed, as often happens in practice, that a part of the material formed in operation B has the characteristics of the product and need not be subjected to operation C (e.g., pentane). In addition to the value of the product, that of the intermediate (= product 2) is to be ascertained.

This is not done directly. The costs up to line SS can be first determined; 299
these are:

	RM
Raw material 1	400,000
Raw material 2	1,050,000
Hydrogen	2,160,000
Operation A	300,000
Operation B	390,000
	4,300,000

These costs include those for 1,275 m.t. of a product having the still unknown value of product 1, and 15,250 m.t. of an intermediate, of which 10,000 m.t. is to be withdrawn as product 2, also still of unknown value. Thus there is an aggregate value for two different products to be broken down, and this problem is at first indeterminate and incapable of a single-valued solution. Further progress is made as follows:

If the 10,000 m.t. were not withdrawn but were worked up further into product 1, the amount of product 1 and the cost of operation C would be increased. The amounts involved can be readily computed, as follows:

	Actual (per month)	RM/m.t.	Computed (per month)
Charge to operation C_1	5,250 m.t.	--	10,000 m.t.
Product 1 from C	4,725 m.t.	0.90	9,000 m.t.
Cost for operation C	52,500 RM	10.00	100,000 RM

That is, product 1 would be increased to 9,000 m.t., and the cost for operation C to 100,000 RM; and the 10,000 m.t. of intermediate would not be withdrawn (in the flow diagram, this is indicated by a broken line). On this basis, only <u>one</u> product would be obtained, and its value can be simply ascertained, as follows:

	Per month			Per m.t. product	
	Amount or Charge	Price per unit, RM	Cost, RM	Amount	Cost, RM
Raw material 1, m.t.	20,000	20	400,000	1.333	26.66
Raw material 2, m.t.	7,500	10	1,050,000	0.500	70.00
Total raw materials, m.t.	27,500	52.73	1,450,000	1.833	96.66
Hydrogen (H), cu.m.	36,000,000	60	2,160,000	2.400	144.00
To operation A, m.t.	20,000	15	300,000	1.333	20.00
To operation B, m.t.	19,000	20	390,000	1.300	26.00
To operation C, m.t.	5,250	10	52,500	0.350	3.50
H+A+B+C			2,902,500		193.50
Product 1, m.t.	6,000		4,352,500	0.400	290.16
Product 2, m.t.	10,000			0.667	
Conversion in C of 10,000 m.t. product 2 into 9,000 m.t. product 1	10,000	10	100,000	0.667	6.67
Computed product 1	15,000	296.83	4,452,000	1.000	296.83

Thus the value per metric ton of product 1, as a mixed product from raw materials 1 and 2, is 296.83 RM (this value results from dividing the computed total cost, 4,452,000 RM, by the computed production, 6,000 + 9,000 = 15,000 m.t.). Product 2 can now be readily evaluated, by difference:

	Amount, m.t.	RM/m.t.	RM
Total product	16,000	272.00	4,352,000
Product 1	-6,000	296.83	-1,781,000
Product 2	10,000	257.15	2,571,500

That is, product 2 has a value of 257.15 RM/m.t., which may be also obtained in a check as follows: From the fact that 1 m.t. product 2 corresponds to 0.9 m.t. product 1, 0.9 x 296.83 = 267.15 RM/m.t.; subtracting from this value the cost of operation C for converting product 2 to product 1 gives 267.15 - 10.00 = 257.15 RM/m.t.

Thus, it has been shown that when more than one product results from one operation, the evaluation of the different products is at first indeterminate and not unequivocally solvable, but it becomes solvable by computing what the yield and the manufacturing cost would be if the various intermediates were worked up into a single product.

The accounting carried out above has the disadvantage that the value of product 1 appears as a mixed value (from raw materials 1 and 2). This mixed value can be broken down as in Case II, appropriately by the four-step accounting scheme, as follows:

Step 1. Yield account:

	Per metric ton			
	Raw material 1 (20,000 m.t.)	Raw material 2 (7,500 m.t.)	Intermediate = product 2(10,000 m.t)	Total (check)[a]
Hydrogen, cu.m.	1,500	800	--	36,000,000
Charge to A, m.t.	1.00	--	--	20,000
Charge to B, m.t.	0.60	1.00	1.00	19,500
Charge to C, m.t.	0.50	0.70	1.00	5,250
Product 1	0.50	0.667	0.90	6,000

[a] Example, for product 1: 0.50 x 20,000 + 0.667 x 7,500 - 0.90 x 10,000 = 10,000 + 5,000 - 9,000 = 6,000

Step 2. Account of specific costs per unit of charge:

	m.t. charged or cu.m. consumed	RM/m.t. or RM/1000 cu.m.	RM/month
Hydrogen	36,000,000	60	2,160,000
Operation A	20,000	15	300,000
Operation B	19,500	20	390,000
Operation C	5,250	10	52,500
Mfg. cost			2,902,500

Step 3. Account of manufacturing costs per metric ton of raw material (or product 2 or intermediate):

	RM/m.t. or RM/1000 cu.m.	Per metric ton of					
		Raw material 1		Raw material 2		Intermediate = product 2	
		m.t. or cu.m.	RM	m.t. or cu.m.	RM	m.t.	RM
Hydrogen	60	1,500	90	800	48	--	--
Operation A	15	1.00	15	--	--	--	--
Operation B	20	0.60	12	1.00	20	--	--
Operation C	10	0.50	5	0.70	7	1.00	10
Mfg. cost			122		75		10

Check: 122 x 20,000 + 75 x 7500 - 10 x 10,000 = 2,902,500 RM.

Step 4. Account of total cost:

	Charge			Mfg. cost,	Product		
	m.t.	RM/m.t.	RM	RM	m.t.	RM/m.t.	RM
Raw material 1	20,000	20	400,000	2,440,000	10,000	284	2,840,000
Raw material 2	7,500	140	1,050,000	562,500	5,000	322.50	1,612,500
"Mixed" value	27,500	52.73	1,450,000	3,002,500	15,000	296.83	4,452,500
Intermediate = product 2	-10,000	257.15	2,571,500	- 100,000	-9,000	296.83	-2,671,500
Product 1	17,500	--	---	2,902,500	6,000	296.83	1,781,000

The same values as before are obtained, 257.15 RM/m.t. for product 2 and a 302
"mixed" value of 296.83 RM/m.t. for product 1; the latter is broken down to 284 RM/m.t.
from raw material 1 and to 322.50 RM/m.t. from raw material 2.

The scheme used can be readily applied to any number of raw materials and intermediates, provided only that these can be worked up into a single product and that the yields and the manufacturing costs for doing so are known.

With this provision, which in most cases is fulfilled, the third requirement, "separate evaluations for different products produced simultaneously", is fulfilled.

III. PRACTICAL APPLICATION

In practical application of the above-described accounting, suitable forms are used.

The first step (yield account) is obtained from the operation balance and is submitted with the monthly report of the main accounting. Its value lies in that the form for the yield account from the operation balance differs from that in the main accounting.

The second step (account of specific costs) results in accounting sheet 1, whose makeup is readily understandable from the above-described main scheme. Operating costs and auxiliary materials are specifically indicated.

The third step (account of manufacturing costs per metric ton of raw material and of intermediate to a single product (VT 708) is the essential part of the accounting. It results in the forms formally designated "helpsheets" 1-3. From them can be obtained all data of importance for evaluating the accounting.

The fourth step (account of total cost) results in accounting sheet 4. In order not to overburden this sheet, the manufacturing costs for the various raw materials and intermediates are broken down on the preceding accounting sheets 2 and 3, according to auxiliary materials, operation costs, hydrogen (pure) and Hy-gas. Besides providing many checks, this arrangement gives a breakdown of the manufacturing costs according to all the raw materials and intermediates, which is useful for many purposes. Especially to be mentioned are the items on accounting sheet 4 that are labelled "special product operations" and "value differences", which must be entered if the results of the accounting are to agree with the recorded values.

"Special product operations" result, in the sense of accounting sheet 4, 303
when an intermediate is introduced, intentionally or otherwise, into an operation not designed for it (as when coal paste is mixed into the crude product, or crude gasoline into the raw material), so that it is processed twice or perhaps not at all.

"Value differences" refer to intermediates that, for any reason whatever, are given a value other than the calculated one.

The accounting proper ends with accounting sheet 4. The important results are summarized in accounting sheet 5.

IV. ACCURACY OF THE ACCOUNTING

It must be pointed out that the accuracy of the breakdown according to raw materials and intermediates depends on the accuracy of the yield account.

Certain assumptions made in the yield account, such as similar treatment of different 5058-injection products, must be considered in evaluating the accounting results. If a finer breakdown of the accounting is desired, its attainment depends on whether the yield account can meet the requirements.

V. EXTENSION OF THE ACCOUNTING

It is possible by the foregoing method to separate the costs for different products from simultaneous processing of various raw materials with the accuracy attainable in technical practice. The manufacturing cost of any individual product is broken down according to hydrogen consumption, Hy-gas, auxiliary materials, operation costs, and according to operations. If necessary, a further breakdown of operation costs into kinds of costs (labor, utilities, etc.) can be made. The method separates the "technical part", the yield account, from the "commercial part". For many purposes, still more is needed; for example, in preliminary appraisals and in design-cost accounts, it is necessary to know the relation of costs to the production level, which is not directly obtainable from the accounting results. However, it is possible, by a rational evaluation of the individual accounting results especially with the help of statistical methods, to draw some conclusions about the relation of costs to the production level. A necessity for any extension is that the proper accounting has been set up.

ABSTRACT OF FRAMES 304-361

Frames 304-361 present detailed monthly cost data relating to the production of various products (gasoline, aviation gasoline, etc.) from various raw materials (coal, tar, petroleum) by the hydrogenation plant at Stettin-Pölitz during most of the period from the beginning of 1940 to the early part of 1943. The data, which are in many voluminous tabulations conforming to the foregoing explanation and in a few graphs, appear to be of little or no present technical interest in this country. However, frames 337-339 may have some significance at the present time and therefore have been translated since cost data is presented for the manufacture, from coal, of the two more important petroleum products, aviation gasoline and motor gasoline. Anyone interested in further cost details per se should examine the German presentation.

CALCULATIONS FOR MANUFACTURE OF GASOLINE FROM COAL - S-81 + M°OIL

Translation of Frames 337-339

AVIATION GASOLINE

337

Basis:

Sump phase: 4 coal chambers = 21,000 m.t./month = 252,000 m.t./year S-gasoline middle oil

Gas phase: 2 (000) pre-hydrogenation chambers = 40 m^3 5058-7846
2 (000) gas-producing chambers = 40 m^3 6434, charge

Production: 100,000 m.t./yr of aviation gasoline (VT 708)

Charge: coal, S-gasoline + middle oil	250.-- RM/m.t.	
tank operation cost	---.75 "	
Charge product for hydrogenation	250.75 RM/m.t.	
Yield in pre-hydrogenation = 97.1%	258.30 "	258.30 RM/m.t.
+ H_2 - consumption = 608 m^3 at .055	33.45 "	
at .050		30.40
+ Catalyst replacement	2.70 "	2.70 "
+ Operations costs for high pressure	14.50 "	14.50 "
+ Operations costs for distillation	2.50 "	2.50 "
Prehydrogenation of gasoline + middle oil	311.45 "	308.40 "
Prehydrogenation of middle oil (B-middle oil)	311.45 "	308.40 "
Tank Operations costs	0.75 "	0.75 "
Sulfurizing costs	0.10 "	0.10 "
6434 refined product	312.30 "	309.25 "
Yield in 6434 = 76.2%	410.00 "	400.00 "
+ H_2- consumption = 473 m^3 at .055	26.00 "	
at .050		23.75 "
+ Catalyst replacement	0.95 "	0.95 "
+ Operation costs for high pressure	27.50 "	27.50 "
+ Operation costs for distillation	5.80 "	5.80 "
6434 - crude gasoline	470.25 "	463.90 "
Mixed product: 24.7% prehydro. gasoline	76.95 "	76.20 "
75.3% 6434 gasoline	354.20 "	349.20 "
	431.15 "	425.40 "
Benzine wash	0.50 "	0.50 "
	431.65 "	425.90 "
Stabilization	1.75 "	1.75 "
Working price without credit	433.40 "	427.65 "

Credit:

Hygas:	1705 International Heat Units (IHU)/m.t. of L-gasoline at 6.00	10.70 RM/m.t.	
	at 5.50		9.80 RM/m.t.
Triebgas:	0.12 m.t./m.t. of L-gasoline at 150.00	18.00 "	18.00 "
Working price after deduction of the credits		404.70 RM/m.t.	399.85 RM/m.t.

Hy gas credit:	Total hygas amount:	3.105 IHU/m.t. of L-gasoline
	Triebgas obtainable:	0.72 m.t. = 1320 IHU/m.t. of L-gasoline
	Credit of hygas:	= 1785 IHU/m.t. of L-gasoline

Triebgas credit: 0.12 m.t./m.t. of L-gasoline at 150.00 RM/m.t.

H_2 - Consumption: 1107 m^3/m.t. of L-gasoline for the gasphase
i.e. 750 " " " = 608 m^3/m.t. of pre-hydro. stripper
and 357 " " " = 473 " " 6434-gasoline

AUTO GASOLINE

Basis:

Sump phase: 4 coal chambers = 21,000 m.t./month = 252,000 m.t./yr of S-gasoline + middle oil

Gas Phase: 2 (000) pre-hydrogenation chambers = 40 m^3 5058-7846
2 (000) gas-producing chambers = 40 m^3 6434, charge

Production: 233,000 m.t./yr of auto gasoline

Charge: coal, S-gasoline + middle oil	250.-- RM/m.t.	
tank operation cost	0.75 "	
Charge product for hydrogenation	250.75 RM/m.t.	
Yield in pre-hydrogenation = 97.1%	258.30 "	258.30 RM/m.t.
+ H_2 - consumption = 608 m^3 at .055	33.45 "	
at .050		30.40 "
+ Catalyst replacement	2.70 "	2.70 "
+ Operations costs for high pressure	14.50 "	14.50 "
+ Operation costs for distillation	2.50 "	2.50 "
Pre-hydrogenation of gasoline + middle oil	311.45 "	308.40 "

Prehydrogenation of middle oil (B-middle oil)	311.45 RM/m.t.	308.40 RM/m.t.
Tank operation costs	0.75 "	0.75 "
Sulfurizing costs	0.10 "	0.10 "
6434 refined products	312.30 "	309.25 "
Yield in 6434 = 98%	334.30 "	331.00 "
+ H_2 - consumption = 370 m^3 at .055	17.05 "	
at .050		15.50 "
+ Catalyst replacement	0.90 "	0.90 "
+ Operation costs for high pressure	25.70 "	25.70 "
+ Operation costs for distillation	5.40 "	5.40 "
6434-crude gasoline	383.35 "	378.50 "
Mixed product: 31.5% prehydro. gasoline	98.40 "	97.45 "
69.5% 6434 gasoline	262.50 "	258.25 "
	360.90 "	355.70 "
Benzine wash	0.45 "	0.45 "
	361.35 "	357.15 "
Stabilization	1.50 "	1.50 "
Working price without credit	362.85	358.65 "
Credit:		
Hygas: 0.507 IHU at 6.00	3.05 "	
at 5.50		2.80 "
Triebgas: 0.042 m.t./m.t. of auto gasoline at 150.--	6.30 "	6.30 "
Working price after deduction of the credits	353.50 RM/m.t.	349.55 RM/m.t.

Hygas credit; Total hygas amount: 0.957 IHU/m.t. of auto gasoline
Triebgas obtainable: 0.042 m.t. = 0.46 IUH/m.t. of auto gasoline
Credit of hygas: 0.507 IHU/m.t. of auto gasoline

Triebgas credit; 0.042 m.t./m.t. of auto gasoline at 150.00 RM/m.t.

H_2 Consumption; 850 m^3/m.t. of auto gasoline for the gas phase
i.e. 638 " " " " = 608 m^3/m.t. of prehydro. stripper
and 212 " " " " = 310 " " 6434 gasoline

CALCULATION OF MASS: MARCH, 1943

Charge:

	kg.	RM per 1000 kg	RM
Raw material:			
a) for Coal phase:			
Hydrogenation coal	35,840,000	27.--	967,680
b) for Tar phase:			
Generator tar	546,100	72.--	39,319
Generator tar 133	2,233,500	47.50	106,091
Tar oil 133	228,100	110.--	25,091
Low-temperature-carboniaztion tar from Brüx	24,529,700	153.--	3,753,044
Low-temperature-carbonization middle oil from Brüx.	219,880	150.--	32,982
Low-temperature-carbonization benzine from Brüx	132,270	145.--	19,179
Pitch-containing residue	105,340	70.--	7,374
Hot tar	514,630	80.--	41,170
Coal-tar pitch	116,700	105.--	12,254
c) for Petroleum phase:			
Rum.crude oil	16,079,025	197.--	3,167,568
Rum.crude oil increase Jan.-Feb.			129,043
Unrefined crude oil	2,160,120	240.--	518,429
			8,819,224
Intermediate products:			
DHD-middle oil	572,400	255.461	146,226
T52-pentane	15,400	516.818	7,959
T52-residue oil	6,900	256.667	1,771
Puregas 241	88,282,000	46.21	4,079,291
Auxiliary materials:			
Iron sulfate	730,000	38.--	27,740
Bayer masse	1,140,000	27.--	30,780
Sulfigran	170,100	300.--	51,030
Soda	44,000	100.--	4,400
Low-temperature-carbonization process	376,510	119.--	44,805
Caustic Soda			1,670
H_2S gas	164,400	39.63	6,515
Purification mass 10,927	180,000	332.--	59,760
Catalyst 5058			196,531
Catalyst 6434			19,049
			442,280
Operations costs:			
Cost division-hydrogenation			4,634,895
Cost division-hygas manufacture			60,243
Costs not divided			85,807

	kg.	RM per 1000 kg	RM
Stock variation:			3,713,066
Self-consumption and delivery:			23,334
Credit:			
Hygas			1,131,693
Washing gas for hot gas			174,263
Deliveries:			
Crude benzine in DHD	20,015,400	345,620	6,917,725
Pentane in DHD	8,200	516,707	4,237
Production of VT 708:	16,458,400	383,596	6,313,378

COMPARISON OF MODE OF OPERATION AND OUTPUT OF A COAL-CHAMBER WITH 4 AND 5 FURNACES

Frames 367-373

Translation

Summary: A coal-chamber with 5 furnaces maintains a higher throughput than a normal chamber. The preheater is operated in such a way that the discharge into the 1st. furnace of the 5-part chamber is at an even temperature. The output of a 5-part chamber, calculated on the reaction volume, is less than that of a chamber with four furnaces.

By experiment and continuous operation it can be determined that a 5-part chamber maintains a higher throughput than a chamber with four furnaces. The more even the temperature curve proceeds in the 1st furnace, the more nearer does the throughput of the 5-fold chamber approach that of a normal 4-part chamber. In the most extreme cases, the 1st furnace acts as a starting furnace and is almost omitted in the performance of the chamber.

The 5th furnace is of special importance for the life-period of a coal-chamber, which in the case of the instantaneous operation is tied in with the extent of crust formation formed in the preheater. With increasing crust formation the output of the chamber diminishes, since after the maximum (Nadel) temperature is reached the hot quantity of gas must be returned slowly but continuously. Therefore the starting temperature of the preheater diminishes. The temperature curve in the 1st furnace is more level, whereby the pure coal decomposition is less. By retraction of the injection the decomposition can be brought again up to the same high. In the case of a chamber with 5 furnaces the preheater starting temperature can be lowered extensively (about 21.5 MV), without the chamber differing essentially from a 4-part chamber.

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Calculated on the reaction volume the output of the coal-chamber is considerably less than that of a 4-part chamber.

In the operation of the 5-part chamber in contrast to a 4-part chamber it is to be noted, that the chamber on account of the increased reflection of the 5th furnace is approached with difficulty. Temperature variations take place longer and stronger on account of the longer duration. Good temperature variation attain, however, a calm operation. The 5th furnace, therefore, works out exceedingly favorably, in that the chamber displays a proportionate output and is not so strongly subjected to fluctuation in decomposition as is a 4-part chamber.

For the purpose of comparison of a chamber with 4 and 5 furnaces, two chambers are operated at about the same temperature level. The temperatures and operation data of the chamber have been compiled into a table.

The balance sheet for the two chambers gives the following values:

	Chamber 15 (4 furnaces)	Chamber 16 (5 furnaces)
Injection of Thick Pulp	14000 1/hr	16000 1/hr (53.3% Fe)
Injection of Thin Pulp	18000 1/hr	19000 1/hr (41.6% Fe)
(zw. of 4 u.h.-Absch. t/61)	2000 1/hr	2000 1/hr
Mud	19.1*m.t./hr	20.8*m.t./hr
Liquid Separator	16.9*m.t./hr	18.22*m.t./hr
Gas and Middle Oil	37.8 %	38.6 %

The balance sheet for the two chambers: (continued)

Separator-325°C	Chamber 15 (4 furnaces)	Chamber 16 (5 furnaces)
Gas and Middle Oil	6.37* m.t./hr	7.02* m.t./hr
Output as regards 4 furnaces	0.18* m.t./hr	0.20* m.t./hr
Output as regards 5 furnaces	---	0.16* m.t./hr

The output of the 5-part chamber lies therefore, calculated on the reaction volume, lower than that of the 4-part chamber. Quantitatively there is produced in the 5-part chamber 0.65 m.t. per hour of gas and middle oil. It 369 is thus shown that the 4 chambers with a fifth furnace produce only about 25 per cent more of the gas and middle than if the 4 furnaces were put in for a new chamber. Therefore it is to be born in mind that the increased output of 4 chambers with 5 furnaces demands no increase of operation personnel and signifies in the manner of energy a proportionally slight increase in load.

Vielen

Plant I

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		Chamber 15 (4 furnaces)	Chamber 16 (5 Furnaces)
Temperature of the Preheater		22^2	22^3
Maximum Temperature of Furnace	1	25^3	25^3
" " " "	2	25^3	25^3
" " " "	3	25^3	25^3
" " " "	4	25^2	25^2
" " " "	5	--	25^1
Gas Entrance		30000	30000
Cold Gas: Furnace	1	4700	4100
"	2	5800	7500
"	3	4600	3400
"	4	2700	2700
"	5	----	2100
(zw. of 4 + H.-Absch)		6900	8900
		24700	28700
		Heating Value	Heating Value
Hot Gas		3700 (1672)	3800 (1672)
(Am. Olzw. of 4 + H-Absch.)		2000 l.	2000 l.
Mud Solid		18.8 %	19.8 %
Ash		50.2 %	49.1 %
Decomposition Regarding Ash		95.0 %	95.0 %

*Metric ton/hour

DIFFERENT METHODS OF OPERATION OF THE SUMP PHASE WITH PETROLEUM-, TAR-, AND COAL-CHAMBERS

General Information

Hydrogen Consumption:	For 1 m.t. Petroleum,	300 m^3/hr
	For 1 m.t. Tar	600 m^3/hr
	For 1 m.t. Pure coal	1200 m^3/hr
Medium Throughput:	Petroleum Chamber	22 m^3/hr
	Tar Chamber	27 m^3/hr
	Coal Chamber	16 m^3/hr
Output:	Petroleum Chamber	7000 m.t. Gas and Middle Oil per month
	Tar Chamber	8600 m.t. Gas and Middle Oil per month
	Coal Chamber	6500 m.t. Gas and Middle Oil per month

Case I: 2 Petroleum-, 2 Tar-Chambers

Production: 31000 m.t./month

Hydrogen Requirement: 40,000 m^3/hr

Case II: 1 Petroleum-, 2 Tar-, 1 Coal-Chamber

Production: 30,000 m.t./month

Hydrogen Requirement: 51,000 m^3/hr

Case III: 2 Petroleum-, 2 Tar-, 1 Coal-Chamber

Production: 37,700 m.t./month

Hydrogen Requirement: 57,000 m^3/hr

Case IV: 1 Petroleum-, 2 Tar-, 2 Coal-Chambers

Production: 37,200 m.t./month

Hydrogen Requirement: 68,000 m^3/hr

Necessary changes in apparatus or number of operation-ready machines needed:

Case I: Suction for the sludge-recycle of Chambers 13 and 14 until the paste press extends 15 a.

Hot cycle for Chamber 13 is necessary (Chamber 14 runs without hot cycle).

Three water driven tops assembled for the paste presses (exhausted in Königsfeld) be changed in the preheating cracker Chambers 13 and 14 like Chambers 11 and 12. The oven passage be isolated in the case of Chamber 14.

Hot separator for Chambers 13 and 14 fitted out with a line as in the Tar-Chamber.

Pointed vessel for Chambers 11 and 12 as well as Chambers 13 and 14 bound to a separate container.

Case II: Preheating cracker of Chamber 14 remains unchanged.

Case III: A booster compressor finished as in case I.

Case IV: Two booster compressors, 1 circulation pump and 3 other tops as column 2.

CHARGE OF BROWN-COAL TAR ALONG WITH PETROLEUM RESIDUES

No. of Sump Chambers	Chambers 2 Tar- and 1 Petroleum		Chambers 2 Tar- and 2 Petroleum		Chambers 3 Tar- and 1 Petroleum	
Raw Materials: Description	Tar (35%-325°)	Petroleum Residue (0%-325°)	Tar (35%-325°)	Petroleum Residue (0%-325°)	Tar (35%-325°)	Petroleum Residue (0%-325°)
Raw Materials:						
Insertion: *m.t./mo.	27,000	10,000	27,000	20,000	40,000	10,000
H$_2$-Consumption: (Sump and Gasphase)						
Separately-m^3/hr	56,000	17,000	56,000	34,000	84,000	17,000
Collectively-m^3/hr	73,000		90,000		101,000	
Sump-phase alone-m^3/hr	33,000		39,000		47,000	
Production:						
Parts by volume-*m.t./mo.	19,000	7,000	19,000	14,500	29,000	7,000
Refined Gas-*m.t./mo.	4,000	2,000	4,000	4,000	6,000	2,000
Collectively:						
Parts by volume-*m.t./mo.	26,000		33,500		36,000	
Refined Gas-*m.t./mo.	6,000		8,000		8,000	
or Collectively:						
High-pressure dehydrogenation-*m.t./mo.	13,000		13,000		13,000	
Auto Gas-*m.t./mo.	14,000		22,000		25,000	
Refined Gas-*m.t./mo.	5,000		7,000		7,000	

*Metric tons/month

TREATMENT OF TAR AND PETROLEUM RESIDUE*

Basis:	Chambers 2 Tar-, and 1 Petroleum-	Chambers 2 Tar-, and 2 Petroleum-
Sump Phase: Chamber	2(0000) + 1(0000)	2(0000) + 2(0000)
Pulp press-m.t./hr	42 + 24 = 6 mach.	42 + 48 = 9 mach.
Contact press-m.t./hr	12 + 7 = 3 "	12 + 14 = 4 "
Gas Circ.-m³/hr	84,000 + 40,000 =	84,000 + 80,000 =
" " collectively-m³/hr	144,000 = 3 mach.	184,000 = 4 mach.
Compression-200 at.	33,000 = 3 mach.	39,000 = 4 mach.
Water pump-m.t./hr	3 = 1 mach.	4 = 1 mach.
Washing Oil-m.t./hr	3 = 1 mach.	4 = 1 mach.
Mud-m.t./hr	3.6 + 1.4 = 5 m.t./hr	3.6 + 2.8 = 6.4 m.t./hr
Sump Phase Distillation:		
Liquid Separator-m.t./hr	36 + 21 (= 57 m.t./hr	36 + 42(= 78 m.t./hr)
Raw tar-m.t./hr	38 + --	38 + --
Collectively	74 + 21 = 2B + 1B-Distillors	74 + 42 = 2B + 1B-Distillors
Debenzine	about 6,000 m³/hr-Raw gas	about 8,000 m³/hr-Raw gas
Gas Phase: Chamber		
Pre-hydrogenation Chamber	2(0000)	2(0000) + 1(0000)
Injection Pump-m.t./hr	13 cm + 39 sm = 2 mach.	13 cm + 45 sm = 2 mach.
Contact vol. (0.7) m³	64 = 8 furnaces	83 = 10 furnaces
Gas circulation-m³/hr	200,000 = 3 mach.	250,000 = 4 mach.
Water pump-m³/hr	2 = 1 mach.	3 = 1 mach.
Benzine chamber	2(0000)	2(0000) + 1(0000)
Injection Pump-m.t./hr	34 cm + 18 cm = 2 mach.	34 cm + 23 cm = 3 mach.
Contact vol. (0.45)-m³	60 = 7-8 furnaces	78 = 10 furnaces
Gas circulation-m³/hr	120,000 = 2 mach.	150,000 = 2 mach.
Water pump-m³/hr	5 = 2 mach.	7 = 3 mach.
Gas Phase Distillation:		
6434 liquid separator	45 (= 1 B - Distillor)	58
7846 " " m.t./hr	43 (= 1 B - Distillor)	56
Collectively m.t./hr	88 = 2 B-Dist. (?)	114 = 2 B + 1 C-Dist.
Stabilizer and Washer	about 36 parts by volume or 18 hi-pressure dehydrogenation + 19 auto fuel	about 47 parts by volume or 18 hi-pressure dehydrogenation + 30 auto fuel
Debenzine apparatus	about 6,000 m³/hr rich gas	about 8,000 m³/hr rich gas
Hi-pressure Dehydrogenation:		
Chamber	2(00000)	2(00000)

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*Reader will do best to consult original.

Report 578-46R

REDUCING THE HEATING LOAD ON SUMP-PHASE PREHEATERS.

Frames 378-391

Abstract

The material included is as follows:

Part 1. A transmittal letter from I. G. Farbenindustrie, A. G., Ludwigshafen, dated April 14, 1943, to the Pölitz Hydrogenation Works, Stettin-Pölitz, concerning a report on a conference which was held at the Ludwigshafen Plant No. 558 on March 22, 1943. This conference was held to study means of reducing the heating load on the sump-phase preheaters of high pressure coal hydrogenation plants in order to effect heat economies and to increase the life and safety of operation of the hairpin preheater tubes, which were made of the relatively scarce N 10 steel alloy. (Frame 378.)

Part 11. Report on the conference at the Ludwigshafen hydrogenation plant No. 55 of I. G. Farbenindustrie, A. G. on March 22, 1943. This report was divided into a section relating to precautions to be taken in the heating technique, a second section relating to chemical precautions, and a third section relating to technical considerations (largely metallurgical), designed to improve the life and safety of operation of the N 10 steel alloy hairpin preheater tubes. To this report are appended four pages containing two tables, two graphs, and two flow diagrams, which are used to present data which were discussed in this report. (Frames 379-389.)

Part 111. A supplementary note by Mr. Schappert of Ludwigshafen discussing the thermal advantages of substituting coal paste for cold gas in the coal chambers. (Frames 390-391.)

RESULTS OF THE LARGE-SCALE PASTE REGENERATION EXPERIMENT K 804.

Frames 392-399

Abstract

This is a report by Mr. Schappert of the Ludwigshafen coal hydrogenation plant of I. G. Farbenindustrie, A. G., dated October 8, 1941. It is concerned with efforts to increase the output of existing coal hydrogenation plants, particularly the one located in Upper Silesia. Interpretation of the results reported is made difficult, because in this report references are made to twelve appended figures which are not included in Reel 75; however, an attempt has been made to present the more important data and conclusions given in this report.

The stimulus for carrying out this large-scale experiment was the requirement that the Upper Silesian coal hydrogenation plant must have a throughput of 50.2 metric tons of coal paste per hour, corresponding to a space velocity of 0.59 kg of pure coal/liter per hour. Compared with the throughput at Nordstern at that time of about 30 metric tons per hour, this represented about a 70 per cent increase in throughput. Considering that it was the opinion of most engineers that no preheater appreciably larger than the one in existence at Nordstern should be constructed, some other or a supplementary method of heating the tremendous amount of paste required had to be found.

Various procedures for heating the coal paste were considered, which are as follows:

Procedure I-The Nordstern Procedure.

In this procedure, the incoming gas is heated practically to reaction temperature in a gas regenerator and is added to the coal paste, heated to about 280 C, after the latter has passed through the low temperature regenerator. The mixture is then heated to reaction temperature in the preheater hairpin tubes, which are heated by combustion gases. If the great charge of the Upper Silesian chamber were heated by this procedure, a preheater with 45 hairpins (90 individual tubes) would be required, and the pressure drop across the preheater would amount to 48 atm. The heat requirement with an efficiency of 70 per cent would amount to 14 x 10^6 kg-cal/hr.

If the throughput for the Upper Silesian plant were increased to 60 metric tons per hour, more than 50 hairpins would be required. By increasing the inside diameter of the hairpin tubes to 100 or 110 mm, a preheater which is not much larger than the one at Nordstern could be used.

Procedure II-The Leuna Procedure.

This procedure consists of the provision of paste regeneration for all the paste, as has been successfully accomplished for over 10 years at Leuna in hydrogenating brown coal. With a K-value (heat transfer coefficient) of 130, heating the entire amount of paste to 332°C was accomplished. Heat consumption amounted to about 6 x 10^6 kg-cal/hr and 26 hairpins were required for the preheater.

However, it is known that the required paste regeneration can not be carried out in the above manner, which was also confirmed by the large-scale paste regeneration experiment K 804 at Ludwigshafen. The reason for this is that this type of coal must be introduced into the preheater at about 250 C. It was possible to effect paste regeneration to about 250 C with only two regenerators. However, it is very difficult to effect this regeneration in such a way that the preheater can be operated at safe tube surface temperatures, because of the greatly increased heat requirement placed upon the preheater. The heat requirement and resistance of the preheater would increase by about 30 per cent. Therefore, because of the high throughput of the Upper Silesian plant, this procedure can not be considered, principally because of the high resistance to flow through the preheater, for which it is estimated that the pressure drop would amount to 52 atm.

Procedure III-The Pölitz Procedure.

The procedure used at Pölitz at the present time incorporates the following advantages of Procedures I and II:

1. The paste regeneration is carried out with a lower concentration of fresh coal than was used in Procedure II, which will probably result in less trouble with the regenerator.

2. The correspondingly smaller amount of fresh coal is heated in a paste of higher concentration at a high temperature differential by the combustion gases leaving the preheater as in Procedure I. Both streams then mix and the mixture is brought to reaction temperature in the preheater, which is heated by hot combustion gases.

3. By the division of the paste into two parallel streams, the pressure differential of the entire system is considerably reduced. It is only by this means that the control of the resistance is made possible for this high throughput. The preheater requires 23 hairpins; the pressure differential amounts to 28 atm; and the heat requirement amounts to about 8×10^6 kg-cal/hr for a throughput of 50 metric tons of coal paste per hour.

Procedure IV.

This is the same in principle as Procedure III only it is carried out with equal concentrations in both streams. The preheater gave the most favorable performance with this procedure, requiring only 18 hairpins, with $\Delta P = 8$ atm, and with a heat requirement of 6.5×10^6 kg-cal/hr for a throughput of 50 metric tons of coal paste per hour. It was erroneously believed-as was proved by this large-scale paste regeneration experiment-that with the process yielding gasoline and heating oil, paste with an appreciably higher concentration of solids could be used in the regeneration than with the process yielding gasoline and middle oil, principally because the paste contained material capable of swelling because of the sludge recycling of less fresh coal. As compared with Procedure III, this procedure would also have advantages on the low pressure paste side for the installation of the paste presses.

The principle objective of the large-scale test K 804 was to clear up questions relating to regeneration, so it was arranged in such a way that it could be operated according to either of the Procedures III or IV.

Reference was then made to a figure which compares the relationships of the regeneration in experiment K 804 with that which is used at Pölitz and that which is planned for the hydrogenation plant in Upper Silesia.

Reference was also made to another figure which shows the course of the heat transfer coefficients or K-values, K I, K II, K III, and K total, during experiment K 804. The fact that the curve for total regeneration is the lowest and not even a median curve is related to the fact that the relatively high radiation losses in the piping between the individual regenerators greatly influence the total K-value, whereas these radiation losses do not show up in the K-values for the individual regenerators.

The following results were established by this paste regeneration experiment K 804:

1. The K-value of about 350 obtained is very high for a paste containing about 40-45 per cent of solids.

2. There was no decrease in the K-values observed toward the end of the test. Operation at the end of the test was the same as at the beginning.

3. The K-values are greatly dependent upon the concentration of solids in the paste, but more particularly, upon the concentration of fresh coal therein, which contains the substances which are capable of swelling.

For example, in one case in which the oil press which furnishes the oil for the control of the concentration of the thin paste had to be stopped for one hour, the concentration increased rapidly from 41 to 51.6 per cent of solids or 34 to 45 per cent of fresh coal. This resulted in a decrease in the K-values during this hour from 300 to 100 and they were still decreasing. Likewise, the pressure differential increased just as rapidly from about 2 atm to 4 atm for the combined regenerators I and II. By means of this unintentional test, proof was given of the great dependence of the K-values upon the concentration. When the oil press was restarted after an hour, the normal K-values reappeared.

Subsequently, the concentration was intentionally increased to 47 per cent of solids or 39 per cent of fresh coal, which resulted in a decrease in the K-values from about 300 to 120. These returned to the former value of 300 when the original concentration of 41 per cent of solids was reestablished.

The course of the pressure differential for regenerators I and II during the increase in concentration from 41 to 47 per cent was indicated graphically in one of the missing figures. Not only the fact that the absolute value of this pressure differential increased from 1.8 to 2.5 atm but the way in which the differential pressure recorder operated during this increase in paste concentration indicated a condition of local stoppage in the regenerator. When the paste concentration is reduced again, the obstruction is removed, the K-values return to the original value, and the differential pressure recorder returns to normal operation. These effects indicate that the regenerator was not seriously clogged; however, during such periods of abnormal operation, the equipment deteriorates.

The extremely important conclusion was reached from experiment K 804 that, although it will not be possible to maintain a desired paste concentration in large-scale operation without having fluctuations of a least one per cent, it will still be possible to operate at a paste concentration close to the upper limit, beyond which continuous operation becomes injurious to the equipment.

Heat Transfer Coefficients (K-Values) of the Preheaters.

Data are presented in a graph, which is missing, and in tabular form showing the dependance of the heat transfer coefficients (K-values) upon the paste concentrations in the three parts of the preheater for which the operating conditions are as follows:

Part I - Thin paste is heated to about 300 C.
Part II - Thick paste is heated to about 450 C.
Part III - Total paste is heated to about 450 C.

The conclusion reached concerning the preheater was that the thick paste components should be preheated to about 300-330 C in the cold section and then by admixture with the part of the stream coming from the regenerator, the real heat source, the temperature is confined within the limits, 300 to 360 C. It was stated that this procedure was found to be the most satisfactory at Pölitz, but that additional data to clear up the matter are being obtained at Stettin.

Summarizing, the large-scale test K 604 demonstrated the significant result that even with the process yielding gasoline and heating oil, the regeneration can be carried out only with a thinner paste. In comparison with the mean concentration of 47 per cent of total solids at the furnace inlet, a concentration of 41 per cent in the regeneration step is safely controllable; it is to be expected, however, that a concentration as high as 43 per cent would cause no difficulty. Correspondingly, the concentration of the thick paste should be increased, for example, to 54 per cent. Even a concentration as high as 56 per cent may be used, the only necessary requirement being that the temperature of the paste does not fall below 115 C in the paste presses, and that, by the use of suitable piping, the linear flow velocity of the paste must be kept below 1.5 m/sec.

Economics of the Process.

An economic survey of the paste regeneration process was made and the results were reported in some detail. Although additional capital investment is required for two additional regenerators and an additional paste press which are required for the process, this is partially counterbalanced by the lower expenditure for a smaller preheater with considerably less hairpin heater tubes and a smaller mill. However, of the greatest importance is the fact that the heat economy of the coal hydrogenation process is so greatly improved by the use of paste regeneration that the savings resulting from diminished fuel gas requirements for the preheater will pay for any increased investment costs and still pay a considerable profit. The reader is referred to the original data (Frame 397, p.6) for details. In this connection, the similar economic survey which was made at Pölitz, entitled "Economics of Paste-Regeneration for One Coal Hydrogenation Chamber", Reel 75, Frames 400-405, which is abstracted in some detail subsequently in this report, may be of interest.

Consideration of Flow-Resistance.

It was pointed out that, because of the high resistance to flow of the paste, it would be in possible, without paste regeneration, to build a 3 or 4-cell hydrogenation chamber for producing gasoline and heating oil unless one used much larger preheater tube dimensions than have been tested in commercial plants. Therefore, it would be necessary to install considerably more hydrogenation chambers in order to attain the output required.

Pressure differentials which were measured in the large-scale experiment K 804 were compared with values which were computed for the plant in Upper Silesia. These data were presented in a graph, which is missing, and in tabular form; however, the latter data appear to be of little significance.

Conclusions.

The final conclusion reached was that by the use of thin-paste-regeneration, it is possible to operate a 4-cell coal hydrogenation chamber with a high throughput and relatively small preheater.

Report 578-46R

ECONOMICS OF PASTE REGENERATION FOR ONE COAL HYDROGENATION CHAMBER

Frames 400-405

Abstract

This report, dated October 14, 1941, was submitted by the High Pressure Technical Division of the Pölitz Hydrogenation Plant.

With regard to the economics of paste regeneration, the concentration limits ~~of both the thick and thin pastes are of considerable importance. In this particular~~ study, a concentration of 38 per cent of coal was regarded as the lower concentration limit for thin paste, economic data for which were compared with corresponding data for a thin paste having a concentration of 40 per cent of coal although the latter concentration is not to be considered as the upper limit. Associated with these thin paste concentrations of 38 and 40 per cent in this study were thick paste concentrations of 52 and 54 per cent, respectively.

Data relating to the total throughput of various materials handled in this hydrogenation chamber are presented first. Then data for the paste regeneration procedure are tabulated in column (a) for thin paste concentrations of 38 and 40 per cent, which are compared with data for the Nordstern procedure in column (b), in which procedure only the inlet gas is regenerated. Following this, a cost comparison for the two thin paste concentrations of 38 and 40 per cent used in the paste regeneration is given under the two headings: a. Plant Costs and b. Operating Expenses. Under the former, it is indicated that with paste regeneration, two additional regenerators and one additional paste press are required, but that the additional cost for these is largely cancelled out by the diminished cost for the smaller requirements of preheater hairpin tubes and other piping. The latter savings amount to 92 per cent of the additional cost for new equipment in the case of the 40 per cent thin paste concentration, but only to 68 per cent for the 38 per cent concentration. In the tabulation of operating expenses, it is shown that savings for heating gas with the use of paste regeneration are of real significance, although those for cooling water and electrical energy are relatively insignificant. These savings for fuel gas are given in a table (Frame 403), wherein comparisons are made for the 38 and 40 per cent concentrations of thin paste for various costs of the heating gas per heat unit, on an hourly and an annual (8000 hours) basis. Also, the time that the chamber has to be shut down for repairs when operating with paste regeneration is indicated in this table. Data contained in this table are also presented in graphical form (Frame 405). From these data, it is apparent that the savings for fuel gas are much greater for the thin paste concentration of 40 per cent solids than they are for the lower concentration of 38 per cent.

With regard to the possibility that the savings from paste regeneration might be absorbed by additional expense resulting from plant shut-down, required to make repairs to the regenerators, the following three statements were made:

1. The experience with this type of operation to date is so slight that no final fundamental conclusions can be drawn. However, judging from the slight experience with this type of operation to date, it appears that if the shut-down is required only to repair the regenerators, a period of three to four days should suffice to make the necessary repairs.

2. The savings for fuel gas are so great with paste regeneration that it is feasible to make the additional capital investment for whatever stand-by equipment is required so that the hydrogenation plant can be kept in continuous operation without decreased production while any necessary repairs are being made.

3. It was concluded from the above statements that the question of paste regeneration as such is subordinate to the fact that for each metric ton of finished gasoline produced, having a calorific value of 1 x 10^7 kg-cal, a decrease in the expenditure of heat energy amounting to 1 x 10^6 kg-cal is effected because of the greater efficiency in plant operation. This saving is of the greatest significance for the energy economy of the hydrogenation process.

Report 578-46R

High Pressure Investigations
WK/Lu 558

May 17, 1938

PRESSURE HYDROGENATION OF PETROLEUM CRACKING RESIDUES

SUMMARY

With the objects of providing a basis for the full scale commercial process and of confirming the results obtained in smaller systems (0.5 and 1.4 liter furnaces), experiments were carried out whereby two gas-oil-cracking residues and three crude-oil-cracking residues were hydrogenated under pressure in a 10-liter furnace employing a pressure of 600 atmospheres of hydrogen.

The most important properties of the cracking residues investigated are presented in the following table:

	Gas oil residue no. 2	Gas oil residue no. 3	Crude oil residue no. 3	Crude oil residue no. 1	Crude oil residue no. 2
Series	A	D	E	B	C
Specific gravity at 0°C.	1.028/50	1.012/100	0.970/70	0.956/100	1.000/50
% at 350°C	26.6	19.5	17.2	33.8	24.4
% asphalt	3.7	7.1	15.1	13.1	13.4
% C	88.53	89.31	86.04	85.92	86.69
% H disposable per 100 g C	9.64	8.90	11.57	11.22	10.93

Every oil was three to five weeks on the way. The experiments were carried out without difficulty.

As catalyst in most cases 6612, charge 12, was used. This is 10 per cent $FeSO_4$ based on the charge and is made by adding 10 per cent 1181 ($FeSO_4$) to 5998, charge 12. The amounts of catalyst used were 1 to 1.5 per cent on the fresh oil. Temperatures were in the neighborhood of 500°C and the throughput was 0.75 to 1.0 kilogram of charge per liter of reaction volume per hour. For 100 kg. of charge about 7 kg. of sludge was obtained on the average. The yield of gasoline plus 350°C-middle oil was between 0.64 and 0.82 kg. per liter of reaction volume per hour and about 11 to 13.5 per cent gas (based on the products).

The results are in complete agreement with those obtained in experiments with smaller apparatus.

Results of the experiments with various oils are summarized in the table. As was to be expected, the less aromatic crude-oil-cracking residues were easier to split than the gas-oil-cracking residues. Thus the gasoline fraction of the 350 C endpoint material obtained from the gas-oil-cracking residue, which had been severely cracked, was only 15 to 17 per cent, while that obtained from the crude-oil-cracking residue was 24 to 30 per cent.

The quality of the gasoline obtained from the gas-oil-cracking residue is higher, again corresponding to the more aromatic character of these oils. The octane number was 73, as compared to 53 and 62 for the gasoline from crude oil cracking residues. On the other hand the quality of the middle oils obtained from the gas oil residues is poorer, with cetane numbers of, 9.5 and 14 as compared with 33 and 43 for the middle oils obtained from crude oil residues.

The asphaltic material in gas oil cracking residues is readily processed with the result that with these residues an almost asphalt-free product is obtained. The asphaltic material of higher molecular weight contained in the crude oil cracking residues is hydrogenated only with somewhat greater difficulty so that the use 421
of greater amounts of catalyst is indicated. Accordingly in the commercial process it will be an easier job to convert the gas oil cracking residues than to convert the crude oil residues.

At the end of the summarizing table are given some pertinent observations as well as other data not included in the table. Finally a detailed description of the experiments is presented. 422

Summary

Five cracking residues were hydrogenated in a 10-liter tube furnace with colloidal catalyst in suspension, the operating pressure being 600 atm. The results are summarized below (although the experiments were not carried out under strictly comparable conditions):

Charge	Gas oil residue no. 2	Gas oil residue no. 3	Crude oil residue no. 3	Crude oil residue no. 1	Crude oil residue no. 2
Series no.	A4	D4	E4	B4	C2
Catalyst	1% 6612 chg 12 - 1% P 471	1% 6612 chg 12	1.5% 6612 chg 12	1% 6612 chg 12	1% 6612 chg 12 + 1% recycle catalyst from centrifuge residue
Furnace temperature	26.3 MV	26.6 MV	26.6 MV	26.4 MV	26.6 MV
Operated for (Abschlaum)	Slight separation	Slight sepn.	Slight sepn.	Slight sepn.	Much sepn.

(continued)

Charge	Gas oil residue no. 2	Gas oil residue no. 3	Crude oil residue no. 3	Crude oil residue no. 1	Crude oil residue no. 2
Series no.	A4	D4	E4	B4	C2
Yield of gasoline and middle oil combined	.735	.639	.822	.767	.816
Gas, % on gasoline + middle oil + gas	12.9	13.5	10.7	13	11.8
Per cent gasoline, 200°C end-point, on gasoline + 350°C middle oil.	14.9	17.1	29.8	25.6	24.2
Gasoline, 200°C(1)					
% 100°C	15	28	28	18.5	20.8
Specific gravity (chem. refined)	.785	.761	.743	.760	.755
Octane no., res. method (chem. refined)	ca. 73	73	ca. 53	62	ca. 62
Middle oil 200-350°C (Diesel oil)					
Specific gravity	ca. .955	.958	.892	ca. .898	927
Aniline point, °C	-	-	41.5	ca. 34	19.5
Cetane no.	ca. 14	9.5	43	ca. 36.5	33
Abschlamm-Note	180	160	80-90	40	ca. 40

The following observations are arrived at on the basis of the above table:

1.) Catalyst number 1181 ($FeSO_4$) on support number 5998, charge 12, is satisfactory but catalyst number 3496 (MoO_3) is better. For gas-oil- cracking residues 1 per cent of catalyst number 6612 ($FeSO_4$), based on the charge, is more than necessary, while for crude oil cracking residues the use of 1.5 per cent is recommended.

2.) The threshold temperature are quite sharp around 480-490 C. so that the safety devices which prevent the reaction from "running away" must be fast-acting and reliable. Increasing the "abschlamm" reflux facilitates control of the temperature.

3.) Runs were carried out with little and with much diluent (abschlamm). The results were similar with both methods but, of course, there is danger of high asphalt formation when running at high dilution.

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1. Gasoline in the gas not determined in runs A4 and B4.

4.) The yield of desired products was above 0.6 and, in some cases, above 0.8, the poorest result being obtained with gas oil residue number 3.

5.) The yield of gas was very satisfactory at 10.7 to 13.5 per cent.

6.) The 200-degree gasoline in the 350-degree cut was between 14.9 and 29.8 percent, being lowest for gas oil residue number 2 and highest for the mildly cracked crude oil residue number 3.

7.) The 200-degree sludge-phase gasoline was always "undercut" since this procedure produces a gasoline of the proper boiling range.

8.) The knock-rating of the "undercut" sludge-phase gasoline was quite good for the gas-oil residues at 73, research method; for the crude-oil residues the octane number was less satisfactory at 58-62.

9.) For the diesel oils (for which the "main points" are very good) the cetane rating is just the reverse of the octane rating, the gas-oil residues yielding material with cetane numbers between 9 and 14, and the crude-oil residues yielding material with cetane numbers between 33 and 43. In the first case it is necessary to include the whole oil beyond the gasphase in order to obtain a vehicle diesel oil, while in the second case it is sufficient to include only the admixture (Zumischung) of gas-phase middle oil. ??

A part of the organic material was deposited on the catalyst.

The separation of diluent (Abschlamm) from stripper (Abstreifer) in the separator is not very sharp at 600 atmospheres.

Also at 600 atmospheres under dehydrogenation conditions the splitting increases for the higher boiling cuts.

For the sludge-phase (Sump-phase) gasoline from gas oil residues the octane number increases for rising boiling points, while for that from crude oil residues the octane decreases with rising boiling point. The curves for octane number by research and motor methods run practically parallel for the sludge-phase gasoline, while for that from crude oil residues the curves intersect.

The resulting liquor for both kinds of residues is weakly acid.

The percentage of asphalt formation is appreciably greater for the gas oil residues than for the crude oil residues; this can be attributed to the readiness with which asphalts arising from polymerization of lighter molecules can be hydrogenated. This also applied to the formation of solids and to the residue remaining after vacuum distillation of the diluent molecules. In the case of the mildly cracked crude oil cracking residues the higher asphalts are more readily decomposed than in the case of the gas oil cracking residues.

The most mildly cracked residues yield the most carbon monoxide in the gas.

Recycle of the light fraction (Hochtoppen) of the diluent-distilled-heavy-oils (Abschlamm-Destillat-Schweröle), which are precipitated out of the diluent, through the furnace appears to work poorly with respect to the yield of cracked products.

Because of the curdling point (Stockpunkt) the sludge-phase diesel oil cannot be carried much above 350 C.

The heat interchange in the preheater appears to be better with the gas-oil-cracking residues than with the crude-oil-cracking residues. The preheater performed throughout without trouble.

If concentration of solids (catalyst) occurred in the oven, oven temperatures became locally unstable.

Intrinsically (an sich) the recycling of the diluent is undesirable from the standpoint of the hydrogenation resluts; however, it is necessary in the technical process in order to avoid drying out (Austrocknen) of the furnace.

By SO_2 extraction of the sludge-phase middle oil from gas-oil-cracking residues one obtains 12 per cent of a diesel oil which is somewhat unsatisfactory in cetane number. The curdling point (Stockpunkt) of the material is bad, therefore, SO_2 extraction is out of the question in this case. The situation is appreciably better with respect to the crude oil cracking residues; here one obtains a 41 per cent yield of a diesel oil with cetane number 53 and, in the other layer an extract with the hydrogen content of middle oil from gas-oil-cracking residues. From the latter good hydrogenation-gasoline is to be expected.

The filtering qualities of the diluent efrom crude oil cracking residue is unsatisfactory. 426

PRESSURE HYDROGENATION OF PETROLEUM CRACKING RESIDUES WITH FINELY DIVIDED CATALYST IN A 10-LITER TUBE FURNACE AT 600 ATMOSPHERES.

Record of Experiments

Apparatus and method: Gas and slurry preheater: Back-and-forth snake coil 10 x 23 x 10000 N 8 in a gas-heated lead bath. Furnace: 70 x 136 x 3000 N 8 with back and forth tubes. Turn-number (Tourenzahl): 14/minute. Reaction volume: 11.2 liters. Direction of flow: Upwards. Separation vessel. 58 x 140 x 1500 V_2A in an electrically heated lead bath. Direction of flow; Upwards. Arrangement: Look-glass, diluent through horizontal coil (28 turn (Touren) / minute) (Combined with a look-glass) over 400 "atu" intermediate-flask "3-liter capacity) into a diluent receiving flask at atmospheric pressure. Gas and product over a cooler with a look-glass at the side. Gas released. Furnace 411 from August 8 to November 11, 1937 and from March 3 to April 29, 1938.

Course of the experiments:

August 13, 1937: Under pressure inert gas 600 atm., 8000 liters on the roof (Dach). Conditions (mit Einlauf): Gas-oil-cracking residue no. 2 plus separator heavy oil (at first from a previous experiment, later from the present experiment) in the ratio 60:40; 1 per cent (based on the charge of fresh oil) catalyst 5475, charge 16, mixed to a colloidal slurry.

August 14, 1937: To 18 MV with diluent recycle, ratio of fresh oil to diluent: 1/0.5. At the outset: furnace 25.5 MV, separator 0.7 MV under the furnace, fresh oil throughput 6 kg/hr., gas = fresh oil x 2.0.
Heat interchange in the snake coil: Good. 427

August 15, 1937: Temperature rises: Furnace 26 MV. Work up: The product was distilled to give gasoline 200°C, middle oil 200-350°C, heavy oil above 350°C. The diluent was weathered off and the oil so obtained was added to the distillation products. The weathering process was carried out in such a way as to lose the least possible oil. This step disturbed the distillation so that middle oil was carried back into the furnace; In the following experiment the furnace was set up for gas oil cracking residue no. 2 without separator-heavy-oil because of this difficulty with the distillation.

428-449

TRANSLATOR'S NOTE: There follows 20 pages of notes similar to those just given. These seem to be unimportant although anybody wishing to repeat the experiments exactly might find these notes helpful. These 20 pages are omitted from the translation.

Page 23

REMAINING OBSERVATIONS

When operating at low diluent percentages the temperature of the furnace is steadier for higher diluent recycle.

With crude oil cracking residues which have high molecular weights the heat transfer in preheating seems to be somewhat poorer than with the gas-oil-cracking residues which have lower molecular weights.

With crude-oil-cracking residues of high molecular weight the danger of drying out in the furnace is less than with gas-oil-cracking residues of lower molecular weight. Therefore it is possible to operate in a narrower temperature range without danger of drying out than with gas-oil-cracking residues. The build up of solids in the furnace seems to be of the same order of magnitude as in the hydrogenation of coal at 600 atmospheres. When the depositions are too great the furnace temperature becomes locally unsteady.

When operating at high percentages of diluent with corresponding recycle of centrifuge heavy oil there is a saving of diluent (Abschlamm) recycle.

The preheating of all oils proceeds without difficulty.

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The following tables are appended:

Index of Tables

Balances (Furnace 411), (four tables)

Gasoline Inspection Data

Middle Oil, Separator Heavy Oil and Diluent (Abschlamm) Heavy Oil

Charge-Oil Inspection Data, Summary of Inspection Data on Charge, Middle Oil and Heavy Oil

Viscosity of Fresh Oils

(continued)

Nitrogen Determinations on Fresh Oils

Gasoline Fractions for Balance A4
(boiling range, gravity and knock rating).

Ditto for Balance B 2.

Ditto for Balance K 7.

Diesel Oil Inspection Data

SO_2-Extraction of S-Middle Oils from Balances A and B.

Heater Oil Inspection Data

Analyses for Carbon, Hydrogen, Oxygen, Nitrogen and Sulfur
(fresh oil contained 2.2 to 2.7 per cent sulfur)

Schwel analysis (Evaporation analysis ?)
(oil, coke, decomposition water, gas and loss on fresh oil, diluent and centrifuge residue). (two tables)

Light Hydrocarbon Analyses

Filtration Experiments for Balance C 1.

Asphalt Inspections.
(two tables).

Liquor Inspections
(NH_3, CO_2, and H_2S also yield of liquor, 10 to 30 per cent of charge)

Pölitz Hydrogenation Plant, Inc.

Pölitz, Dec. 1, 1944
BH/Kö/Ht

Mr. Director Dr. Wissel
Building 11

562

File Note

Concerning: Influence of the coal catalyst (Bayermasse and Sulfigran) as compared with the ordinary grude catalyst in the treatment of pitch (Chamber 12).

SUMMARY

1. The transition from grude catalyst to Bayermasse and Sulfigran for pitch treatment produced an increased throughput, expressed in both a higher Bi/Mi and a higher heavy-oil yield.
2. It appears that if the coal catalyst is used a heavier incrustation of the preheater with iron sulfide must be reckoned with.

Point 1.

In the last unfinished period the pitch chamber was operated -- first, from Sept. 9, 1943, with the usual catalyst 10927 (grude catalyst) and then, from Sept. 26, 1943 on, with Bayermasse and Sulfigran alone or in mixture with catalyst 10927. The catalyst compositions and the more important operating data are given in the attached graph.

The ratio of injection-mixture to pitch-mixture was held constant at 70:30 until Nov. 17, at which time addition of varying quantities of tar-mixture and grinding oil from the coal chamber was started. On Dec. 20 the ratio of 70:30 was again resumed. For the present investigation only, the data from the periods during which the pitch-mixture alone was charged will be used, since the pitch-tar-grinding-oil-mixture--because of large variations in the composition--does not permit any clear conclusions. 563
The curves show that during the time Bayermasse and Sulfigran were used, either alone or with catalyst 10927, more injection-mixture could be charged, the amount accumulating in the stripper increased, and sludge production in the finished product decreased. This was particularly evident from Oct. 28 to Nov. 12. The supposition that the coal catalyst favors primarily the formation of heavy oil does not prove to be correct, as the heavy-oil balance during this time proves.

HEAVY OIL BALANCE

	Average Daily Values	
	Coal Catalyst 10-30 to 11-5	Catalyst 10927 11-7 to 11-12
Total injection mixture, metric tons	511	485
Heavy oil added, metric tons	197	189

(continued)

Asked about the probable reasons for the better fractionation procedure at Pölitz, the representatives of this plant hinted that in their operation at high pressure, the asphalt equilibrium value was held at about 8 per cent, and indicated that by uniform dilution in the centrifuge, the asphalt concentration in the oil and residue coming from the centrifuge was kept equal. The representatives of the Pölitz plant evidently withheld more fundamental information than the above regarding the fractionation step from the representatives of the other plants, because a statement was made in parentheses in the report that addition of catalysts in the fractionation step was not mentioned to the other representatives.

LIQUID-PHASE CATALYSTS

Frames 555-565

Translation

Bag 2 555

30/4.13

12 Liquid-phase Catalysts

Pölitz Hydrogenation Plant, Inc. 556
High pressure research

LV(?) 558 September 29, 1938

On the problem of smp-phase catalysts for Stettin

In the 10-liter oven (classification No. 134371, from May 17, 1938) the following catalysts were studied:

1. Steam-activated brown coal-grude, neutralized, impregnated with MoO_3,
2. Steam-activated brown coal-grude, not neutralized, impregnated with 10% $FeSO_4$,
3. Brown-coal producer soot Me, not neutralized, impregnated with 10% $FeSO_4$,

In all catalysts molybdenum is somewhat better than iron; but iron is also satisfactory in all cases. For gas-oil cracking 1 per cent of catalyst is satisfactory; for crude-oil cracking 1.5 per cent is recommended.

In the 100-liter oven brown-coal producer soot Me, not neutralized, impregnated with $FeSO_4$, likewise proved satisfactory; as in the 10-liter oven. In comparison, an experiment with 1 per cent Luxmasse, impregnated with 0.2 per cent MoO_3, ended unsuccessfully, because this catalyst agglomerated into a large ball, which naturally remained in the oven and filled the reaction space (it was observed in the 10-liter oven that upon reaching a determined catalyst concentration the local oven temperature fluctuated).

According to information from Dr. Wissel the following difficulty exists for supplying catalyst from Merseburg for Stettin:

1. The unworked producer soot, of which there is a sufficient quantity at Merseburg, must be taken to Stettin in large trucks. It is necessary to buy these trucks at Stettin.

2. Merseburg can impregnate the producer soot with $FeSO_4$ and then send it to Stettin in a moistened state in ordinary trucks. Stettin must pay the freight for the water (about 40 per cent of the catalyst) and then dry the catalyst (we have found that ~~the mealiness of the moist catalyst is unsatisfactory).~~ Either possibility will require the maintenance of a very large stock pile at Stettin, to prevent a plant shutdown in case of traffic stoppage.

I suggest that, if coke is used as a catalyst support, Stettin could obtain large quantities from Silesia. Moreover, the erection of an impregnator and drier at Stettin would be necessary. Dr. Wissel asked to test one such catalyst in the small apparatus.

Whether it is necessary to use a high catalyst concentration and recycle of the catalyst through the centrifuge to get useful results cannot be decided without study. The fact that cracking of the high-boilers proceeds under dehydrogenating conditions when operating at 600 atmospheres makes this way appear unpromising, since in earlier studies a high catalyst concentration maintained a good activity, when operated under hydrogenating conditions.

The suggestion of Dr. Wissel that vanadium-containing ash 558
(about 0.1 per cent) be used as catalyst for crude-oil cracking appears valid to me, since it is recycled through the centrifuge so that 1.0 per cent of catalyst is carried over with the fresh oil. The catalyst must be very stable, so that it will still work under dehydrogenating conditions after ten recycles. In studies in the 10-liter oven of the single recycle of a grude catalyst, it could not be determined whether the recycled catalyst was still active.

A further suggestion of Dr. Wissel, namely, the use of coal-hydrogenation residue for a catalyst carrier, appears of little promise. For, as is well known, the real function of the sump-phase catalyst support is the absorption in its pores of the adhesive asphalt formed by the cracking hydrogenation; when however, as in the hydrogenation-residue, all the pores are already filled with asphalt or asphalt-coke, the necessary function cannot be fulfilled. Opening the pores by activation with steam at about 900 C is too expensive.

The proofs taken in the arrangement of Dr. Pier, concerning Op as purveyor of catalyst support, showed the following according to a conference with Dr. Gloth:

1. A producer operated with Rhenish brown coal yields a powdery
   slag with 95.99 per cent ash (analyzed by Dr. v. Füner).
   Operating to produce less ash was of no interest. It is 559
   highly probable that this material is not suitable for us.

2. A producer operated with coal-coke in one particular place yielded a more sintered residue (analyzed by Dr. V. Füner) in comparison with that mentioned under (1).

3. Moreover, the coke-producer yields a soot, of which the ash content is considerably less than that from (1) and (2). This material may have considerable promise for use as a catalyst support. If necessary (which a local inspection must show) the still-glowing soot can be activated with more efficient use of its heat by contacting with steam at about 900 C in a tube sealed to the producer. This material is probably also easily obtained in Stettin.

SUMMARY

Soot (in some cases easily activated) can be supplied from coke producers for urgently needed catalyst carrier for KaSO4 and Stettin.

Coke itself can be used as a secondary catalyst support.

In partnership with Dr. Rank.

(signed): W. Krönig

Report 578-46R

Pölitz Hydrogenation Plant, Inc.
Research Works

February 28, 1944 My 560
BV/Siew.

Concerning: Use of catalyst 1181 (ferrous sulfate) in the hydrogenation of coal.

In the different hydrogenation plants two methods are used for adding catalyst 1181 (ferrous sulfate) to coal. The first consists in impregnating the rough-ground coal with a saturated water solution (about 30 per cent) of the catalyst and drying, the second in dry addition of the catalyst to the coal from the analogous drying of catalyst 6512 (Bayermasse).

To test whether these different addition possibilities had any influence on the Bi/Mi--capacity or the decomposition, we have made two experiments with an O.S.-hydrogenation coal mixture (30 per cent Jupiter, 25 per cent Heinitz, 15 per cent Beuthen lump, 10 per cent Beuthen fines, 20 per cent Donnersmark), as was made in the middle of November, 1943, in the chamber works, whereby in one case the catalyst 1181 (ferrous sulfate) was added according to our usual impregnation method and in the other according to the practically dry process used in the plant. The second experiment was run only 168 hours, because the oven was needed for a more pressing experiment.

As can be seen in the attached tabulation of the experimental data, the Bi/Mi capacity of the impregnated ferrous sulfate catalyst was 0.26 kg/liter of reactor volume/hour and that of the dry mixture was 0.25 kg/liter of reactor volume/hour at 325 C. The difference of about 4 per cent was insignificant. The decomposition, 94.0 per cent and 94.5 per cent, respectively, was practically identical in both cases.

Although these results should be taken with some reserve because of the shortness of the tests (which will be repeated at the first opportunity), the indications are that a clear-cut difference cannot be expected in the working out of the two methods of addition of catalyst 1181 (ferrous sulfate).

(Signature not decipherable)

Attachment to report of Feb. 28, 1944, concerning: Use of catalyst 1181 (ferrous sulfate) in the hydrogenation of coal. Experimental conditions and results (average values)

Experiment		Addition of catalyst 1181 (ferrous sulfate) by impregnation	Dry addition of catalyst 1181 (ferrous sulfate)
Pressure, atm		600	600
Max coil temperature, (S_4), mv		23.5	23.5
Max oven temperature, mv		25.0	25.0
Hot separator temp (W_4), mv		24.3	24.3
Coal sp vel, kg/litr (reactor)/hr		0.48	0.48
Parts (or tons) of coal		0.51	0.51
Parts (or tons) coal: grinding oil		46:54	46:54
Parts (or tons) coal: sludge recycle		1:0.3	1:0.3
Catalyst	% 1181 (ferrous sulfate)	1.2	1.2
	% 6512 (Bayermasse)	1.8	1.8
	% 6709 (Sulfigran)	0.3	0.3
Kind of sludge-diluting oil		Heavy oil	Heavy oil
Kind of grinding oil		Strained oil	Strained oil
Grinding oil	% solids	9	7.5
	% ash in solids	41	30
	% asphalt in oil	7	8.5
Sludge	% solids	26	22
	% ash in solids	55	52
	% asphalt in oil	13	12
Stripper	Sp gr/20C	0.990	0.992
	Wt %-160C	4	4
	Wt %-160-325C	46	47
Practical oil yield/raw coal, %		67	65
Practical oil yield output, kg/litr/hr		0.32	0.31
Bi/Mi-capacity, kg/litr/hr		0.26	0.25
Oil yield distribution in the practical oil yld	% Bi/Mi	81	80
	% S°oil	19	20
% decomposition		94.0	94.5

(Signature indecipherable)

Report 578-46R

REPORT CONCERNING A CONFERENCE ON COAL HYDROGENATION

Frames 479-480

Abstract

This report, dated September 30, 1941, concerning a conference held at Pölitz on September 26, 1941 was made by Mr. Schmitt of the Pölitz Hydrogenation Works. The conference was attended by six representatives of the coal hydrogenation plants located in Gelsenberg, Scholven, and Pölitz, the representatives for each plant being designated in the report.

The purpose of the conference was to compare the results of coal hydrogenation work carried out at Pölitz with similar results obtained at Gelsenberg. The points of special interest were the coal throughput, the fineness of grinding of the coal, and the results obtained with the tar fractionator. Operating data for Gelsenberg for the month of August, 1941, which are mean values for the three-and four-cell chambers, and tentative data for Pölitz covering a period of 8-14 days, are presented in the following table:

	Pölitz	Gelsenberg
Chamber Arrangement: (In operation)	2 x 4-Cell Chambers	3 x 3-Cell Chambers* 1 x 4-Cell Chamber
Throughput: Thin paste	19.6 metric tons/hr (38%)	-
" Thick paste	22.4 " " " (53%)	32 metric tons/hr (54%)
Coal Throughput, TK.	16.8 " " "	14.1 " tons/hr
Results:		
Disintegration	94-95%	96.3%
Oil production, kg/l	0.30	0.265
Gasoline + middle oil in oil	85%	91.4%
Gasification	24%	22.5%
Oil/metric ton of pure coal	665 kg	697 kg (?)
Hydrogen used/metric ton of oil	-	1.657 m^3(?)
Coal-Grinding:		
Before Concerta-mill, under 0.1 mm	23%	50-55%
After " ", " " "	85%	72%
Fractionator Oil yields:	Ca. 80-85%	80.3%

*Throughput of the 3-cell chambers: 22-25 metric tons/hr of paste =
10-11 " " " of TK.

It was indicated that the production rate at Pölitz is about 10 per cent higher than at Gelsenberg, but that at Gelsenberg a preheater reserve of about 10°C was being retained, which, if utilized, might result in equalization of the two production rates. Also, it was stated that the production rates presented in the table were based on chamber volumes which are probably somewhat too large at Gelsenberg and too low at Pölitz. Correcting for these differences, the production rate increases to 0.270 kg/l for Gelsenberg, and decreases to 0.295 kg/l for Pölitz.

Heavy Oil Balance (Continued)

	Average Daily Values	
	Coal Catalyst 10-30-to-11-3	Catalyst 10927 11-7 to 11-12
Pitch concentration	72 %	72.9 %
Stripper without water	427	389
Bi/Mi in stripper	157 (30.8% yield on inject. mixt)	152 (31.2% yield on inj. mixt.)
Total heavy oil leaving	316.7	297
Heavy oil excess	115 (22.5% yield on inj. mixt.)	107 (22.1% yield on inj. mixt.)

Both the Bi/Mi capacity and the heavy oil excess in both cases are the same percentage of the injection mixture. There is an increased throughput of 26 metric tons per day, or 5.4 per cent based on the injection mixture, with both the Bi/Mi ratio and the heavy-oil excess remaining equal.

Point 2.

It appears that the preheater needles of the coal chamber acquire a considerable incrustation (about 10 mm) through iron sulfide formation after quite long operating times. This fact can be traced back to operation with the above-mentioned coal catalyst and is probably due primarily to the presence of ferrous sulfate and Sulfigran. 564

In the petroleum and pitch chambers, which in the past operating periods were operated with catalyst 10927, an iron sulfide incrustation of about 2.6 mm was found in one particular case. It now appears that with the transition to Bayermasse and Sulfigran in the pitch chamber an increase in the rate of iron sulfide formation resulted.

As an indication of the incrustation in the preheater, the temperature difference between the product temperature in the preheater outlet and the highest needle temperature can be studied. The larger the incrustation, the larger this difference becomes.

This difference is shown graphically in the curves in column 1. It is constantly increasing and, in the periods Oct. 5 to 19 and Oct. 31 to Nov. 25, during which both Bayermasse and Sulfigran were used, there was an increase in the rate of rise of the curve. However, the time of observation is too short for any conclusive statement to be made. The heat balance of the heating-up (see page 4 (frame 565)) on Sept. 22, 1943, and Jan. 2, 1944, likewise indicate a heavy incrustation of the preheater needles.

The heat balance indicates a great impairment of the regeneration. The preheater is not able to compensate for this heat loss, because of loss in heating capacity due to incrustation of the needles. The decreased heating capacity is expressed in the decreased throughput of 22 per cent.

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Heat Calculations for Chamber 12

		Sept. 22, 1943 22 o'clock	Jan. 2, 1944 12 o'clock
Injection mixture, cu.m./hr		18.0	14.0
Separator Reflux, " " "		7.0	8.0
Hot circulation, " "		8.0	5.0
Entering gas, " "		18,000	18,000
Cool gas, " "		4,000	4,000
Temperature formation, C	Entering	184←305←382	219←256←305
	Leaving	306→391→442	348→415→446
Δt,m		86	134
K value, K cal/m^2/C/hr		125	30
Regn heat cap., 10^6 kcal		3.8427	1.695
Preheater outlet, C		476	415
η of regn, %		48.7	32.4
Heat capacity, η = 100%, 10^6 kcal		6.11	5.75
Heat capacity, η = 65%, 10^6 kcal		3.97	3.74
Additional heat for η = 65%, kcal		105,000	116,000
Heat change, kcal/kg injec. mixt./hr		166.9	138.5

BH-Sump-phase,

(Signature indecipherable)

CATALYST 6612 (SEE THE RECORD OF THE I. G. FARBEN CO. HIGH-PRESSURE EXPERIMENT OF DECEMBER 3, 1940). 580

Translation Frames 580-582

Out of the above mentioned report of the I. G. Farben Co., the section pertaining to the high-pressure experiment, it has resulted, that the catalyst 6612 Ch 12, which was first foreseen in the case of the treatment of cracked residue, is no longer in question for the treatment of pitch, hard coal, and hard-coal-tar oils. As I have personally acknowledged, the experiment at Ludwigshafen, which has not been officially recognized, achieved no success with the above catalyst.

After the report of Dr. Krönig, the suitability of catalyst 7509 Ch 12 in the case of the experiment at Ludwigshafen, which contained on ashes about 5 per cent Titanium sulfate (water free) in addition to 7 per cent Iron sulfate, will be proved in contrast to catalyst 5475 Ch 12 (Molybdenum oxide). A tar residue boiling above 325°C, obtained from the Augusta Victoria Plant, which incidentally was at Ludwigshafen, was converted into benzine and hot oil. The results show that the catalyst 7509 Ch 12 cuts off poorer in contrast to the catalyst 5475 Ch 12.

Under consideration of the fact that -- as explained in the experiment report -- the washing amount in the daily report in the case of operation with catalyst 7509 Ch 12 is unusually high and has been calculated in common with low washing percents, the report can decide, however, out of the oil-production distribution, that the catalyst 7509 Ch 12 yields a lesser amount in products boiling up to 325°C than the operation with the catalyst 5475 Ch 12. 581

Therefore the supposition is not on hand to prove, that the output in the case of operation on benzine and middle oil with the new catalyst lies at a low piont than with the approved Molybdenum.

Vaporization in the case of operation of hard-coal-tar residue on benzine and hot oil with the new catalyst lies about 1 per cent higher than in the case of operation with the Molybdenum catalyst. This distinction in the vaporization is probably still increased in the case of operation of these same products on benzine and middle oil and therefore acts still further toward the unfavorableness of the new catalyst.

Further disadvantages of the new catalyst, for example, with regard to low-temperature carbonization yield, are to be directly found out of the report.

Out of personal and confidential communication I learned, that in the case of the experiment in Ludwigshafen the stripping product had a specific weight, which in the case of a stripping temperature of 40°-50°C came so close to the specific weight of the stripping water, that the separation of the water appeared difficult. In case this disadvantage in the working method with the new catalyst showed up likewise in large-scale operation, it would be extremely difficult to accomplish the distillation.

Summarizing, it can be said, that it appears unconditionally necessary to prove the equivalence of the new catalyst 7509 Ch 12 with regard to its action in comparison to the catalyst 5475 Chamber 12 in the case of treatment of tar on benzine and middle oil, before an introduction for our operation comes into question. Since we have foreseen in our working program also the treatment of pitch of various sources, the new catalyst in the case of the operation of this product, which in the case of a higher solid substance content and a considerably longer standing time acts as the initial material which stood in Ludwigshafen for arrangement, if necessary in a mixture with tar to test on its suitability.

Concerning the occurrence of titanium ores inside of the German territory it can in the case of the present stand of our books no nearer determination can be taken. There is present only a notice, according to which in the year 1929, 1305 metric tons of titanium ores were exported out of the territory of Czechoslovakia

Dr. Schmitt

SULFUR TREATMENT OF MIDDLE OILS

Frames 590-7

Translation

EXPERIMENT INTERCHANGE-SULFUR TREATMENT OF MIDDLE OILS

In disposal of your foregoing letters, we hereby submit the answers to the question sheet based on our knowledge of the subject. 59

As you found from the lists, we have changed over experimentally into gas phase operations for the treatment of the injected materials with sulfur, a simple operation for the pre-hydrogenation with elementary sulfur and the use of H_2S in gasoline production.

Attempts are not being made at present by us to sulfurize with a gas that contains as much as 80 per cent H_2S; yet we are of the opinion that with a smaller per cent of H_2S not unlimited harm is brought about by the contact. We have used for some time for sulfurizations 6434 composed of about 60 per cent H_2S, the absence of which, at any time, caused a dying out to occur.

Heil Hitler !

ANSWERS TO THE QUESTION SHEET CONCERNING THE EXPERIMENT INTERCHANGE-THE SULFUR TREATMENT OF MIDDLE OILS

1.	S Content of Injected Products:	5058 - ca. 0.05-0.10 % 6434 - ca. 0.00 %
2.	Phenol and Oxygen Content of the Injected Products:	5058 - ca. 12-15 % Phenol 6434 - ca. 0.01 % Phenol 5058 - ca. 2 % Oxygen 6434 - ca. 0.00 % Oxygen
3.	Sulfur Adsorption in per cent S:	
	a. Because of Elementary Sulfur in:	5058 - 0.2-0.3 %
	b. Because of Elementary H_2S in:	5058 - 0.2-0.3 % (Free Operation Methods)
	c. Because of Elementary H_2S in:	6434 - 0.2-0.3 %

591

4. Data of Temperature and Pressure in Sulfur Treatment:
   a. Elementary Sulfur in: 5058: $t = 40\text{-}50°$, no pressure
   b. H_2S in: 5058: $t =$ ca. $40°$, $p = 400$ mm
   c. H_2S in: 6434: $t = 40°$, $p = 400$ mm

5. H_2S Analysis: 50-60 % H_2S
   50-40 % CO_2

6. S in Circulating Gas: 5058 - 0.02-0.05 g/mm^3
   6434 - 0.8-1.7 g/mm^3

7. a. Circulating Gas: 5058 - ca. 1.5 + 1.0 = 2.5 m^3/kg Injected Material
   6434 - ca. 1.3 + 0.7 = 2.0 m^3/kg Injected Material
   b. Refined Gas: 5058 - ca. 400-450 m^3/t Injected
   6434 - ca. 300-350 m^3/t Injected

8. Special Observations in the Regeneration and Extraction

A powerful dying out occurs in the heat transfer for the same heating during the prehydrogenation occurring generally in connection with gasoline production, likewise with or without sulfur treatment and, of course, caused by iron sulfide deposits on account of the phenolic content of the injected products. These deposits appear through the addition of sulfurizing mediums, especially promoted by H_2S.

After three years' work with H_2S sulfur treatment during gasoline production, a diminishing of the K-value occurred with 6434 in the reclaimed rubber and extracted material; there existed with 6434 in contrast to this under certain circumstances (after free observations) deposits of sulfur in the reclaimed rubber at the sulfurization, when the material injected was too light.

9. Special Observations on the Material Injected after the Sulfur Treatment

The action of H_2S (in the presence of phenolates) occurs not only under conditions of high pressure, but also under reduced pressure during the sulfurization process. A part of the phenolate decomposes here at a reduced temperature (about 40) and fine particles of iron sulfide precipitate out.

In the sulfurization of 6434, a product injected with H_2S, this was not observed.

S in pieces is used; its impurities occasionally cause difficulties in the filtration of the product.

10. Results with H_2S Concentration Under 80 Per Cent

Sulfurisation with H_2S was always carried out in Poelitz with an H_2S concentration below 80 per cent; a dependence of this concentration on contact damage was not observed.

Sulfur - Balance "B.S." Month: March 592

			S in "B.S." Kg.	S Waste Kg.	S Produced Kg.
Refined Gas 520	27 551 600	m^3			
Crude Gas 2.8 g S/m^3			76 400		
Pure Gas .013 g S/m^3				355	
Rich Gas 520	10 496 000	m^3			
Crude Gas 15.3 g S/m^3			161 000		
Pure Gas .012 g S/m^3				126	
Phenol Water	55 383	m^3			
	3.84 g S/1		212 000		
Dirty Water	21 675	m^3			
	0.005 g S/1		108	108	
Final Water	70 880	m^3			
	0.381 g S/1			27 700	
Recovered Gas 518	119 927	m^3			
	18.5 % H_2S		29 100		
Amount S in 520 + 576			478 608	28 289	450 310
OWG 770	20 159 000	m^3			
Crude Gas 4.1 g S/m^3			82 000		
Pure Gas 0.084 g S/m^3				2 000	80 000
Total			560 608	30 289	530 319
H_2S Production					
Separated Gas 520	483 175	m^3			
	57.5 % H_2S				364 000
Separated Gas 770	193 410	m^3			
	31.7 % H_2S				
Blast for Reburning	2 008 800	m^3			80 900
	30.0 g S/m^3				62 790
H_2S - Evolution					
Bi-Sulfur Treatment 518	322 000	m^3			
	57.5 % H_2S			242 000	
Claus Furnace:	345 020	m^3			
	44.7 % H_2S			202 000	

Sulfur - Balance "B.S." (Continued)

	S in "B.S." Kg.	S Waste Kg.	S Produced Kg.
H_2S over Roof in 516 + 520 (concentration)		25 619	
Blast for Reburning 2 008 800 30.0 g S/m^3		62 700	
		550 519	
S Production (Claus Furnace)			144 000
Claus Oven Waste Gas for Reburning		58 000	
Claus Oven-Yield 71.4 %			
Blast for Reburning			62 700
Claus Oven Waste Gas			58 000
			120 700
H_2SO_4 Production 10			
H_2SO_4 Yield %			

Composition

	Kg.	%
S Introduced in "B.S."		
In 520 and 576	478 608	85.0
In 770	82 000	15.0
Total	560 608	100.0
S Removed in "B.S."		
S Produced (Claus Furnace)	144 000	25.8
S for the Bi-Sulfur Treatment 1. as hydr.	212 900	57.9
2. as recovered gas	29 100	5.2
Claus Furnace Waste Gas for Reburning	58 000	10.4
Blast for Reburning	62 700	11.2
H_2S in 526 + 520 (Conc.) over Dome	29 619	4.2
S in Pure Gas	2 481	0.4
Total	560 608	100.0

S for the Elementary Sulfur Treatment	106 790 kg.
S at Building 110	1 700 "
S At Zell and Zellulose at. wt.	77 090 "
	185 580 "
S Amount on 1-3-44	24 690 "
S Produced in March	144 000 "
S of the Residue	80 120 "
	248 810
S Delivered in March	185 580 "
S Amount on 31-3-44	63,230 "

Sulfur - Balance "B.S." Month: February 594

	S in "B.S." Kg.	S Waste Kg.	S Produced Kg.
Refined Gas 520 21 880 000 m^3			
Crude Gas 2.5 g S/m^3	54 700		
Pure Gas 0.012 g S/m^3		260	
Rich Gas 520 8 724 000 m^3			
Crude Gas 14.5 g S/m^3	124 800		
Pure Gas 0.015 g S/m^3		140	
Phenol Water 68 150 m^3			
3.18 g S/1	216 200	36 600	
Dirty Water 20 100 m^3			
0.003 g S/1	60	60	
Final Water 65 900 m^3			
0.174 g S/1		11 500	
Recovered Gas 518 82 650 m^3			
17.7 % H_2S	19 100		
S - Amount in 520 + 518	414 860	48 560	366 300
OWG 770 21 114 000 m^3			
Crude Gas 4.5 g S/m^3	94 700		
Pure Gas 0.081 g S/m^3		1 700	93 000
Total	509 560	50 260	459 300
H_2S Production			
Separated Gas 520 564 000 m^3			282 000
56.1 % H_2S			
Separated Gas 770 219 600 m^3			
32.4 % H_2S			93 000
Blast for Reburning 1 880 000 m^3			
26.1 g S/m^3			49 100
			424 100
H_2S Evolution			
Bi-Sulfur Treatment 518 270 000 m^3			
56.1 % H_2S		199 000	
Claus Furnace 315 950 m^3			
42.5 % H_2S		176 000	
H_2S over Roof in 576 + 520 (Conc.)		35 200	
Blast for Reburning 1 880 000 m^3			
26.1 g S/m^3		49 100	
		459 300	
S Production (Claus Furnace)			134 400
Claus Oven Waste Gas for Reburning		41 600	
Claus Furnace Yield: 76.4 %			
Blast for Reburning			49 100
Claus Oven Waste Gas for Reburning			41 600
			90 700

H_2SO_4 Production 10

H_2SO_4 Yield %

+ 11 500 m^3 as Refuse

Composition

		Kg.	%
S Introduced in B.S.			
S Produced (Claus Furnace)		134 400	26.4
S for Bi-Sulfur Treatment	1. for hydr.	179 900	35.4
	2. for recovered gas	19 100	3.7
Clause Furnace Waste Gas for Reburning*		41 600	8.2
~~Blast for Reburning*~~		~~49 100~~	~~9.6~~
H_2S in 576 + 520 (Conc.) over (Roof)		55 200	6.9
S in Final Water		48 160	9.4
S in the Pure Gas		2 100	0.4
*as SO_2 to the chimney			
	Total	509 560	100.0

S for the Elementary Sulfur Treatment	91 470 kg.
S at Building 110	1 750 "
S at the Zell and Zellulose, Wittenberge	87 690 "
S at Kausch and Co., Hbg.-Ld.	29 150 "
S at Bark and Schülte, Hbg.	100 330 "
	310 370 "
S Amount on 1-2-44	109 240 "
S Production in Feburary	134 400 "
S in Residue	91 470 "
	335 110 "
S Amount in February	310 370 "
S Amount on 29-2-44	24 740 "

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Sulfur - Balance "B.S."

Month: January

			S in "B.S." Kg.	S Waste Kg.	S Production Kg.
Refined Gas 520	20 988 000	m^3			
Crude Gas 2.3 g S/m^3			48 200		
Pure Gas 0.014 g S/m^3				300	
Rich Gas 520	7 637 000	m^3			
Crude Gas 14.8 g S/m^3			113 000		
Pure Gas 0.016 g S/m^3				100	
Phenol Water	60 765	m^3			
	3.18 g S/l		193 000	10 200	
Dirty Water	17 015	m^3			
	3.000 g S/l		-	-	

Sulfur - Balance "B.S." (Continued)

	S in "B.S." Kg.	S Waste Kg.	S Production Kg.
Final Water 66 150 m^3			
1.27 g S/l		84 000	
Recovered Gas 518 90 000 m^3			
14.4 % H_2S	17 000		
S Amount in 520 + 576	371 200	94 600	276 600
OWG 770 24 745 000 m^3			
Crude Gas 4.0 g S/m^3	99 000		
Pure Gas 0.080 g S/m^3		2 000	97 000
Total	470 200	96 600	373 600
H_2S Production:			
Separated Gas 520 349 000 m^3			
52.4 % H_2S			240 000
Separated Gas 770 241 000 m^3			
30.7 % H_2S			97 000
Blast for Reburning 2 009 000 m^3			
13.9 g S/m^3			28 000
			365 000
H_2S Yield:			
Bi-Sulfur Treatment 518 269 000 m^3			
52.4 % H_2S		180 000	
Claus Furnace 321 000 m^3			
37.4 % H_2S		157 000	
H_2S over Roof in 576 + 520 (Conc.)		8 600	
Blast for Reburning 2 009 000 m^3			
13.9 g S/m^3		28 000	
		373 600	
S Produced (Claus Furnace)			121 600
Claus Oven Waste Gas for Reburning		35 400	
Claus Furnace Yield			35 400
Blast for Reburning			28 000
Claus Oven Waste Gas for Reburning			63 400
H_2SO_4 Production 10			
H_2SO_4 Yield %			
3 200 m^3 Sludge to the Refuse			

Composition

	Kg.	%
S Introduced in "B.S."		
S Production (Claus Furnace)	121 600	25.9
S for the Bi-Sulfur Treatment (1. to the hydr.	163 000	34.4
2. recovered gas 518	17 000	3.6

Composition (Continued)

	Kg.	%
Claus Oven Waste Gas for Reburning*	35 400	7.6
Blast for Reburning*	28 000	6.0
H_2S in 576 + 520 (Conc.) over Roof	8 600	1.8
S in Final Water	84 000	17.9
S in Sludge to Residue	10 200	2.5
S in the Pure Gas	2 400	0.5
Total	470 200	100.0

*as SO_2 to the chimney

S for the Elementary Sulfur Treatment (518)	97 710	kg.
S at Building 110	1 700	"
S at Fa. Bark and Schälte, Hbg.	30 330	"
S at Zell and Zellulose, A. G. Wittenberge	67 040	"
	196 780	"

S Amount on 1-1-44	96 710	"
S Produced in January	121 600	"
S as Residue	87 710	"
	306 020	"

S Amount in January	196 780	"
S Yield on 31-1-44	109 240	"

ANALYSIS OF SULFIGRAN

Frame 617

Translation

Experiment: Sulfigran, Ore Smelted

Experiment Number	4197
Beginning of Experiment:	12-7-43

% Ash	13.5	
% Na	39.4	
% Saturated Sulfide	30.1	
% SO_4 - S	0.45	
% Polysulfide	0.5	
% SiO_2	3.1	
% Fe	1.1	
% Al	0.5	
% Ca	1.2	
% Acid Solution	15.8	(carbon)
% Insoluble Ash	7.0	(from starting material)

Distribution:

Dr. Wissel
Dr. Schmitt
Dr. Diepenbruck
Dr. Kiehn
Dr. Horn
Dr. Berger

FERROUS SULFATE

Frame 625

Translation

Ferrous Sulfate

Herr Heibach developed the following mixtures based on a meeting with the consultants on high-pressure experiments on December 9, 1938, for the preparation of ferrous sulfate and the manufacture of oil as carbon:

1. Manufacture of crack-residue with maximum 1.5 per cent contact number, 6612. Ch. 12, that is 1.5 per cent (1.14 "stuto")* hot ashes saturated with 10 per cent $FeSO_4$; estimated as water free, that is, 20 per cent $FeSO_4 \times 7H_2O$ corresponding to 220 kg/h = 1800 "jato".*

2. In the manufacture of carbon at first is the case A + E 1.2 per cent dry $FeSO_4$ corresponding to 6500 "jato" $FeSO_4 \times 7H_2O$, later for the case F + G + E, 11,000 "jato".

Distribution:

Dr. Wissel
Dr. Schmitt
Dr. Hermann
Dr. Heibach

* Possible translation of "stuto" = metric tons per hour.
" " " "jato" = metric tons per year.

DEVELOPMENT OF THE SUMP-PHASE CATALYST WITH LIGNITE

Frames 638-639

Translation

DEVELOPMENT OF THE SUMP-PHASE CATALYST WITH LIGNITE

Report on the Meeting at Leuna on January 1, 1944

Information has been furnished on the development of catalyst addition by Dr. Döllein with the aid of Dr. Becker. Lignite went without catalyst addition in Leuna in 1927-1929. It appears that with these manufacturing methods of lignite the analysis is in the order of magnitude of about 60 per cent, and, moreover, the asphalt of this washing ranges so high that further washing causes difficulties. As hydrogenation was discontinued to a great extent during the economic crisis of 1929, experiments were conducted for the hydrogenation of lignite in the presence of sump-phase catalysts. It appears that an addition of 2 per cent molybdenum, calculated as MoO_3 and added as the ammonium salt, gives good results. The analysis goes resistingly upon the merit that it was economically productive. It became apparent after further experiments, that after its neutralization in the alkali medium with lignite present (to about 25 per cent complete neutralization) only about 0.05 per cent molybdenum (calculated as molybdic acid) was discovered in the sulfuric acid medium, for the same decomposition was discovered as with larger molybdic acid addition in the absence of neutralization. (Probably from these experiments, the theory has developed that hydrogenation and division of carbon in acid medium gives better results of analysis. Among others, experiments were conducted with nickel salts that likewise gave good results. Since the above conducted combination was not present in satisfactory medium; so, the others, from the economical viewpoint, caused no stimulation, but good results were also obtained with the addition of iron salts. It was further shown that in a neutralization of carbon with sulfuric acid using iron, as bad results were obtained as when iron salts alone were used. It was therefore established that the best results are from the addition of iron in the presence of alkali. From these considerations the catalyst Me 10927, that is, alkalized iron sulfate ash, was developed. In Leuna the carbon was saturated with iron sulfate solution, which was added in portions of 0.8 per cent, calculated for this purpose as Fe. This iron sulfate was then neutralized with equivalent amounts of alkali. Lye, sulfigran, soda, etc. were used as the alkali. It is assumed that the ferrous hydroxide first formed is transposed with the sulfur of the carbon into iron sulfide, which results in the especially effective catalyst reaction. Since iron sulfate did not exist in satisfactory amounts for disposal, "lautemasse, luxmasse and bayermasse", respectively, were tried. Difficulties resulted in respect to patent rights since Bergius, for his part, had specified "luxmasse" as an effective catalyst in the original patent. These experiments showed the result that these products also had the effect 639 of a catalyst; yet, in their effectiveness, they remained behind the hydroxide precipitated from iron sulfate and alkali. As a rule it can be said that in case of doubling the amount of "bayermasse", the iron content refers to the attainment of the same effectiveness as with iron sulfate that must be added to the carbon. Leuna accepts "bayermasse" and recognizes the addition of 1.8 per cent, calculated as Fe, as being effective. These amounts correspond to approximately 5 per cent "bayermasse". No especial effect was reported with larger additions. Sulfigran and iron sulfate were not used in Leuna.

At Ludwigshafen in 1939 in the hydrogenation experiments on coal the following ingredients were mixed: 1.2 per cent iron sulfate as $FeSO_4 \cdot 7H_2O$ + 1.5 per cent "bayermasse" + 0.5 per cent Sulfigran, all referred to "TKo." That is: 0.20 per cent Fe as iron sulfate and 0.40 per cent Fe as "bayermasse". The calculated Fe yields an average addition of 0.6 per cent. The amount of "sulfigran" based upon "TKo" is in the case of iron equivalent to the iron sulfate. These addition catalysts developed in Ludwigshafen for coal are considerably reduced as to the amount, which has proved necessary for lignite. The amount of iron sulfate was probably reduced with respect to the amount mentioned from the unsatisfactory offer, thereby probably the amount of water needed for the dissolving of the iron sulfate might offer important restrictions upon the drying of coal. Dr. Döllein and Dr. Becker are not authorized to give special information concerning these catalysts for coal.

SITUATION WITH IRON SULFATE

Frame 640

Translation

Situation with Iron Sulfate

640

Concerning your inquiries of the partnership work for hydrogenation in your letter of November 24, 1944, the following statements are made:

1. Which demand on iron sulfate have you foreseen for 1945?

On the assumption that there were three coal chambers in operation at the beginning of the year and four at the end of the year so that three coal chambers operated in the middle of the year, there would be from 17 to "T.Ko" with an hour's infiltration for each chamber and 8,000 hours per year the use of 480,000 to "TKo" per year. In the middle 1.5 weight per cent iron sulfate were added so that in 1945 the demand on iron sulfate amounted to 7200. tons.

2. What amount have you stored?

According to the data on December 1, 1944, the amount stored in the coal room and in building 720 was 850 tons.

3. How much can yet be stored by you?

Approximately 100 tons.

4. How has your demand been covered up to now?

The iron sulfate was ordered from a contractor from the "Bayewerke" in Leverkusen.

Operations Manager

CATALYST FOR COAL HYDROGENATION

Frames 641-644

Translation

641

On the question regarding the demand and the possibilities of preparation, transportation and storage of the catalyst in Stettin in the sump-phase for cracked residue, Dr. Krönig gave the following answer:

For gas-oil cracked-residue, 1 per cent is used, for crude-oil residue 1.5 per cent of fresh catalyst. The high demand in regard to the fresh catalyst is explained by the much higher content in high molecules in the residue in contrast to the tar, where only about 0.1 per cent of the fresh catalyst (Molybdenum ash in Magdeburg) is used.

Ash, saturated with 10 per cent $FeSo_4$ and 1 per cent MoO_3 acts somewhat the same. Out of the economically devised ashes, iron ash is preferred.

The generator dust is prepared in Leuna. Through a somewhat changed method of operation of the generator (poorer combustion) a dust deposits with 38 per cent ash in contrast to 60 per cent ash in the case of normal operation methods.

This dust is saturated in Leuna for use in suitable operation with 4 per cent Molybdenum (calculated as MoO_3). The saturated generator dust has an apparent density of about 0.35 and a grain size of 61-93 per cent passing through a 254 mesh screen. The grain size goes up to 2 mm. For transportation and storage a suitable container must be provided.

642 For the hinderance of oxidation from ferro-iron to ferric-iron the catalyst must be loaded, shipped and stored under protective gas (supply for at least a week = 191 m.t./day = 550 m^3). This supply would be stored in two of the twelve chambers which should be established for coal hydrogenation. As a protective gas carbonic acid came into question. For the avoidance of oxidation either the saturation with iron should be undertaken in Stettin or the molybdenum ash should be ordered.

For saturation 2.9 kg $FeSo_4 \times 7 H_2O$ in 13 kg of water is injected on 18 kg of ashes; the saturated catalyst has therefore 38 per cent water. This is to be dampened again, since the catalyst forms lumps in the case of mashing. For drying the catalyst, best suited is a small rotary-tube furnace, which can be brought with gas-heating up to about 250°C.

The catalyst so prepared would then have to be ground to such an extent that a maximum of 2.5 per cent would be retained in a 254 mesh screen. In Magdeburg the contact paste was maintained 20-35 per cent solid. For achieving the solid concentration of 35 per cent there would have to be mixed 1.2 m.t./hr of the catalyst with 3.8 m.t./hr mud = 5.0 m.t./hr catalyst-paste in a spherical mixer. For investigation, Herr Kreiselmaier asked to have 200 kg. ash or catalyst sent to Gruson-Magdeburg. For mashing in the case of refining of mud there came into question the sulfuric-acid-containing compounds. Therefore the catalyst paste could be somewhat released.

643

In the case of operation of coal is the iron sulfate (about 2.4 per cent $FeSO_4 \times 7 H_2O$) saturated before the drying of the coal. The other catalysts (2.0 per cent Luxmasse and 0.8 per cent Na_2S) are added without special treatment before the mashing. A catalyst preparation (generally about 3.0 per cent dry catalyst) is dropped, therefore to provide for a stock holder. By means of saturation of the catalyst on the coal, the coal and past drying is not accidental. The water content of the coal goes, for example, from 8 per cent to 12.6 per cent.

According to the experiment of Dr. Krönig in furnace 703 only about one-tenth of the amount of oil now put in for circulation wash for cracking residue is necessary. It is not known, how great the leaks are in furnace 703.

Continuation of the conference without the men from Hochch. Vers.

The question was proposed, how the tar deposited in the power gas manufacture was to be utilized. A sale is unprofitable on account of the small proceeds. Besides on account of the high aromatic-content a good gasoline can be prepared out of it. In the case of operation of coal, the tar together with the coal-stripper is liberated from the particles of middle oil, whose residue goes with the stripper oil to rubbing oil. Precipitation danger no longer exists. In the case of operation of oil, alone, the tar must be given without distillation to the injection product, since in the case of the bringing together with hydrogenation cracked-products the asphalt of the tar would precipitate.

For the working up of the mud from the cracked residue two procedures come into question: the refining with sulfuric acid by attaching the centrifuge and the distillation at 440° or 350°C. In the case of the two processes it would not be easy to burn the resulting refined or distilled residue in a reservoir house together with coal. One must therefore think, especially for later obtaining of the ash out of the residue, about a rotary-tube furnace for the combustion and waste-heat-utilization.

644

As an advantage consider the slight manufacturing cost and manufacturing waste of the refining process in contrast to the distillation. The sulfuric acid refining is almost solid free; it can, however, on account of its content of sulfur compounds, not be given in the distillation, so that a certain loss in oil residue operation must be taken in the cost.

As regards the mass-catalysts-process for utilization of waste gas from the Claus furnace with a content in H_2S and SO_2 up to 10 per cent, it is established, that the working temperature of the catalyst in the case of uncertain S-content of the gases can be regulated by means of throttling the supply of power gas to the combustion furnace as also by means of cooling the catalyst casing. For further certainty a connection from the combustion furnaces, which correspond to the proposed re-combustion furnaces at Leuna, to the chimney should be provided. For the case that the catalyst furnace is omitted, the gases freed by H_2S through re-combustion can go directly to the chimney. An acid-proff lining of the chimney is probably not necessary, since the operating condition is temporary and the temperature of the waste gases can be situated so high through regulation of the power-gas supply, that no condensation occurs.

ON THE QUESTION OF THE SUMP-PHASE CATALYST FOR STETTIN.

Frames 648-53

Translation

648

In a 10-liter furnace (1) the following catalysts were investigated:

(1) Water-vapor activated brown coal ashes, neutralized, and saturated with MoO_3.

(2) Water-vapor activated brown coal ashes, not neutralzied, and saturated with 10 per cent $FeSO_4$.

(3) Brown coal generator dust Me, not neutralized, and saturated with 10 per cent $FeSO_4$.

All catalyst behave unobjectionably; Molybdenum is somewhat better than iron, but iron is completely sufficient. Likewise it is sufficient for all cases of the generator dust at Merseburg. For gasoline-cracking residue 1 per cent catalyst is more than sufficient; for crude-oil-cracking residue 1.5 per cent catalyst is recommended.

In the 100-liter furnace the brown coal generator dust Me, unneutralized, and saturated with 10 per cent $FeSO_4$, likewise proved unobjectionable, mixed as with the 10-liter furnace. On the other hand, an experiment with 1 per cent Luxmasse, saturated with 0.2 per cent MoO_3, proceed catastrophically, by gathering this catalyst into a great ball, which naturally remained lying in the furnace and thus took up the reaction space.(2)

After the statement of Dr. Wissel the following difficulties exist for the supplying of the catalyst out of Merseburg to Stettin:

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(1) The untreated generator dust, which is present in sufficient amounts in Merseburg, might be sent to Stettin in dust wagons of great size.

(2) At Merseburg the generator dust for Stettin might be saturated with $FeSO_4$ and then sent in the moist state to Stettin in the usual conveyances. At Stettin the freight must be paid for the water (about 40 per cent of the catalysts) and a drier(3) must be set up. Both possibilities demand the setting up of great storage tanks in Stettin, in order to keep the commercial supply from being exhausted.

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(1) Classification #34371 of May 17, 1938.

(2) The observation of the 10-liter furnace confirms, that in the case of the establishment of a fixed catalyst-concentration in the furnace, the furnace temperature is locally unsettled.

(3) The ability of the moist catalyst to be ground is, as has been ascertained, unsatisfactory.

It would be proposed by me to take coke as a catalyst carirer, which Stettin can order in great amounts out of Schlesien. Moreover, the setting up of a saturator and drier in Stettin would be unnecessary. Dr. Wissel offered to test such a catalyst in a small apparatus.

If an operation with higher catalyst-concentration and catalyst refluxing about the centrifuge leads to useful products, nothing can be decided without experiment. The fact, that in the case of operation at the accelerated high output at 600 atmospheres, the cracking proceeds under dehydrogenation conditions for the high-boiling products, ~~causes this way to appear not very promising, since in the earlier experiments the high-~~ concentrated catalysts retained good activity only as long as it was operated under hydrogenation conditions.

650

These observations apply also to my opinion for the proposal of Dr. Wissel, to use the V-containing ashes (about 0.1%) of the crude-oil-cracking residue as a catalyst, by so sending it back over the centrifuge, that with the fresh oil 1 per cent catalyst is led in. The catalyst must already be very stable, in order to work under dehydrogenation conditions still sufficiently after 10- fold refluxing. With the experiment in the 10-liter furnace in the case of single-fold refluxing of an ash catalyst it could not be determined if the refluxed catalyst possessed activity or not.

A further proposal of Dr. Wissel, namely to use the hard-coal-hydrogenation residue as a catalyst carrier, shows little promise. For, as is known, it is with the most essential problem of the sump-phase-catalyst carrier, that the sticky asphalt formed in the case of the hydrogenation is absorbed in its pores; if, however, as in the case of the hydrogenation residue, all the pores are already stopped up with asphalt or asphalt-coke, the necessary task can not be fulfilled. An opening of the pores (activated with water vapor at about 900°C) would be, however, too expensive.

The experiment undertaken on the order of Dr. Pier, in so far as Op is concerned as supplies of a catalyst carrier, yielded the following after consultation with Dr. Gloth:

(1) In the case of the generator operated with purest brown coal, there accumulates a powderous slag with 95.99 per cent ashes.(4) Operation on less ash content is out of the question. The material in the highest possibility is not useful for us. 651

(2) In the case of the generator operated with hard-coal coke there accumulates in a particular place a sintered residue,(4) from that concerned in statement 1).

(3) In the case of the coke generator there accumulates, besides, a flue dust,(4) whose ash content is less than in the case of 1) or 2) These materials may have considerable use as catalyst-carriers. If necessary, the still glowing flue dust (which a local inspection must show) can be somewhat reactivated with water vapor by utilizing its heat in a cheap way in a directly joined tube at about 900°C. These materials were probably also easily obtained in Stettin

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(4) The sample is thereby analyzed by Dr. Von Fümer.

SUMMARY

First, as a catalyst carrier for Ka 804 and Stettin, flue dust should be provided out of the coke generator.

Second, coke could be used as the catalyst carrier in question.

In partnership with Dr. Rank

Dr. Krönig

Translator - Robert Doan

652

HOT ASHES OR LUXMASSE AS CATALYST CARRIERS IN THE CASE OF THE HYDROGENATION AT 600 ATM. OF CRACKING -RESIDUE IN 100-LITER FURNACE 703

(Fresh oil = 1:1 mixture of crude oil and gas-oil-cracking residue)

(P 1307 + P 1329)

	Date	9-21-38	9-25-38
Fresh Oil	% organic solids	0.48	0.48
	% asphalt	7.4	7.4
	stripping-heavy oil	1:0.2	1:0.4
Fresh Oil	mud	1:0.7	1:0.7
	throughput	1.1	0.704
Passage of Circulation Gas	m^3/kg Fresh Oil	1.95	2.2
Catalyst		Brown coal generator dust Me Operating with 10% $FeSO_4$ (K 6612, Ch 12)	Luxmasse operating with 0.2% MoO_3 (K 6954)
Fresh Oil		1	1
Temperature °C of Furnace		484	481

% Solid Decomposition		114[1]		48.2	
% Asphalt Decomposition		93.8		89.9	
Washing Note		50		40-50	
% Mud in Fresh Oil for Distillation		15.2		23.3	
% Highly Superheated Mud in Fresh Oil		3.7		5.9	
	Benzine-150°-	6.6	66.1	7.6	66.1
	Middle Oil 150-350°	59.5		58.5	
Inclusive of Washing and Ash-free Fresh Oil	Stripper-Heavy-Oil 350°	18.3		3	
	Mud Oil	14.5		22.5	
	Highly Superheated Mud in New Contact	2.8		4.9	

---

(1) That is, a small part of the organic portion of the catalyst carrier has been disintegrated.

(continued)

Date		9-21-38		9-25-38	
Vaporization of Benzine and Middle Oil		12.1		14.7	
Output inclusive of Washing Treatment	Benzine-150°	0.072	0.722	0.054	0.466
	Middle Oil 150-35°	0.650		0.412	

The Luxmasse-Molybdenum Catalyst is therefore extraordinarily much worse 653 than the iron-ash catalyst, as is shown primarily in the output and vaporization. With the ash catalyst the furnace remains on a high proportionate yield, after the replacement by Luxmasse catalyst, the yield falls off on every level. Moreover, the Luxmasse catalyst is worse in asphalt and solid decomposition. These latter unfavorable characteristics have worked out disagreeably, as emphasized earlier (1), since the catalyst, although it was pound collodially fine (2) (Passage through a 254-mesh screen: 90.5 per cent, through a 279-mesh screen: 75.5 per cent, through a 330-mesh screen: 70 per cent) with coke (4) it gathered together in the shell-shaped form of 0.8-1 mm size, which naturally was too great, to carry the stream out of the furnace. The Luxmasse catalyst is not, therefore, in question for Stettin.

W. Krönig

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(1) Classification #134521 of September 29, 1938.

(2) What the A.N.L.C. had emphasized again especially for this type of catalyst.

(3) See the adjacent photo

(4) The grains in the furnace have only 45 per cent ash insoluble in benzine in contrast with 100 per cent in the case of the entering catalyst; and indeed it does not concern the agglomerate but the coke, that is, intermediates have exhibited a probable agglomerate.

CATALYST FOR COAL HYDROGENATION

Frames 656-61

Abstract

Concerning the important catalyst for coal, conferences were held with Drs. Simon and Funer. Herr Dr. Simon reported that for the treatment of coal only unused Lux-, Lauta-, or Bayer-product would be used. (The weight of Lux-product used amounted to about 5 per cent of the total charge.) In the hydrogenation process Lux- and Lauta-product behaved equally well, while unused Bayer-product was poorer than these two. Lauta- and Lux- products, like iron ore, can be hydrated, while Bayer-product is oxidizable. Prices for these products are as follows:

Iron Ore	16.60 Reich	marks	per	ton
Lauta product =				
Lux product with 50-55% water	19.00 "	"	"	"
Bayer-slurry (18-20% water)	22.30 "	"	"	"

In Leuna a mixture of 40 per cent Iron Ore and 60 per cent Bayer product was used, for which Leuna paid 16.60 and 22.30 Reich marks per ton, respectively, according to statements of Dr. Funer.

Dr. von Funer made the following remarks concerning the composition of the individual materials:

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	Lux-Product %	Bayer-Product %	Lauta-Product %
SiO_2	1.05	7.48	1.71
TiO_2	8.18	7.60	3.92
Al_2O_3	7.1	17.86	3.80
Fe_2O_3	47.78	51.27	13.90
Cr_2O_3	0.18	0.12	---
NiO	0.09	---	---
CuO	0.03	---	---
CaO	2.23	0.88	4.84
MgO	0.58	0.09	0.33
K_2O	0.10	0.12	0.11
Na_2O	4.61	5.46	1.32

(continued)

	Lux-Product %	Bayer-Product %	Lauta-Product %
CO_2	3.45	---	1.82
SO_4	0.95	1.01	0.09
P_2O_5	0.20	---	---
H_2O	11.0	0.55	47.37
Cl	---	0.12	---
Ignition Loss	18.76	8.23	59.0

The alkali decomposition of red bauxite (containing considerable quantities of iron) gave a red sludge which had the following analysis:

Fe_2O_3	38%
TiO_2	5.5%
SiO_3	3%
Al_2O_3	3%
Na_2CO_3	0.5%
H_2O	50%

The differences in individual sludges resided in the procedure for the bauxite decomposition. For example the so called Lauta-product resulted from the old procedure in which treatment is carried out at 600-700° with Na_2CO_3 melt. In this treatment aluminum as well as iron is removed. The new procedure, in which the so called Bayer-product results, is conducted with NaOH at about 700° and 7 atmos. pressure. In this procedure only the aluminum is removed; the iron is not.

The following supply sources for the various sludges were suggested by Dr. Funer.

Bayer product	Lauta factory
Lux product	Giuline
Lauta product	Lauta factory
$FeSO_4 \cdot 7H_2O$	Leverkusen

Report 578-46R

4.1.1939

659

To Director Pier,
High Pressure Research

In order to avoid the drying of wet coke, studies were made in grinding moist coke with asphalt-free-wash-heavy oil distillate and asphalt free oil. The material prepared from the asphalt free oil is less viscous than that pre~~pared from asphalt containing oil. It appears possible from a commercial view~~point to treat moist coke with asphalt-free heavy oil.

We ask in this communication whether the use of the product prepared from moist coke and asphalt free oil might have any particular disadvantages with reference to work techniques at Pölitz.

660

MATERIAL PREPARATION WITH USE OF HEAVY OIL DISTILLATES

By use of practical asphalt-free wash-heavy oil distillate for the preparation of product, there is obtained a slightly less fluid product when mixed with wet coke (40 per cent water content) than when dry coke is used.

On long standing the viscosity of product samples prepared with practical asphalt free heavy oil did not increase (both water free and water containing samples).

With use of material prepared from untopped sump sludge it was shown that no essential viscosity difference existed between samples prepared from dry and moist coke.

In the preparation of material by use of practical asphalt-free heavy oil distillate in which further dilution with untopped sump oven sludge is used, according to preliminary studies at this laboratory - a pre-drying of coke is not necessary.

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(1) Heavy Oil Preparation (wash oven 703)

(a) Wash Properties

Content of benzene insolubles	4.2%
Ash content of benzene insolubles	33.6%
Asphalt content	3.2%
Viscosity (50°C)	8.6°Engler

(b) Wash Distillation

Middle oil (up to 350°C)	21.7%
Heavy oil (350-440°C)	49.8%
Residue (above 440°C) (softening point (+ 86.5°C benzene insolubles = 24.4%)	24.3%

(continued)

(2) Wash-Heavy Oil Distillate
Properties

Specific Gravity at 50°C	1.040
Asphalt content	0.65
Viscosity at 50°C	13.1°Engler

Boiling Curve

Initial	355°
14.2%	375°
52.5%	400°
47.5%	Residue

(3) Product Preparation

(a) 66.6 wt % heavy oil distillate to 33.3 wt % Winkler generator powder

(b) Mixing temperature about 50°C

(c) Mixing time: short grinding in ball mill - main mixing 4 hours in disintegrator

(4) Viscosity of Slurry

Water Content of Coke (In flow time for 200 cc slurry at 100° through a 6 mm tube in seconds)	0%	40%
After one day standing	41.7	59.[illegible]
After 5 days standing	40.7	5[illegible]
Mixing with wash in proportion of 1:2	7.2	8.[illegible]
" " " " " 1:9	5.8	7.0

Report 578-46R

EXAMINATION OF BAYERMASSE, FERROUS SULFATE AND SULFIGRAN

Frames 678-680

Translation

Examination of Catalysts I, II and III

678

Catalyst I (Bayermasse)

H_2O	12.9 %
Fe_2O_3	37.9 % (in the dry test)
Ash	82.8 % (in the initial test)
Ash	93.8 % (based on initial tests)

Catalyst II (Ferrous Sulfate)

$FeSO_4 \cdot 7H_2O$	96 %
Fe^{+++}	Trace
Free H_2SO_4	0.29 %
Ash	29.9 %

Catalyst III (Sulfigran, Smelted Ore)

Total S	26.9 % (= 65.5 % Na_2S , 1.20 % Ne_2SO_4)
SO_4	4.5 %
Ash	12.2 %
Dissolved Acid (Organic)	22.3 %

Distribution:

Dr. Schmitt
B.L.

Examination of Catalysts I, II and Sulfigran

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Catalyst I (Bayermasse) on 7-16-43

% H_2O	8.0	
% CO_2	6.6	1.4
% Ash	86.5	90.0

Sulfigran

% SiO_2	0.17
% Al_2O_3	0.10
% Fe_2O_3	0.56
% CaO	0.75
% MgO	Trace
% SO_4	2.40
% Cl	0.10
% Ash	17.1
Na_2S	Rest

Examination of Catalysts I, II and Sulfigran (Continued)

Ash Analysis

% SiO_2	13.61	13.63
% Fe_2O_3	39.28	42.32
% Al_2O_3	23.35	26.62
% TiO_2	5.55	5.55
% P_2O_5	0.38	0.43
% CaO	1.96	0.62
% MgO	1.12	0.69
% SO_3	9.55	1.14
% Na_2O	3.85	8.90
% Loss on Ignition	1.1	

Ash Melting Point	113.0 C
Sintering Point	88.0 C

Catalyst II

% $FeSO_4 \cdot 7H_2O$	95.1
% Fe^{+++}	0.23
% H_2SO_4	0.33
% Ash	ca. 29.0

Examination: Sulfigran, Ore Smelted

680

% Ash	135
% Na	39.4 (= 27.4 % S, 67.0 % Na_2S, 122 % Na_2SO_4)
% Total S	30.1 (= 134 % Na_2SO_4)
% SO_4 - Sulfur	0.45
% Polysulfide	0.5
% SiO_2	3.1
% Fe	1.1 (= 0.6 % S as FeS)
% Al	0.5 (= 0.9 % S as $Al_2(SO_4)_3$
% Ca	1.2 (= 1.0 % S)
% Dissolved Acid	15.8 (carbon)
% Undissolved Ash	7.0 (upon starting material, 1.1%)

S - Combination

Na	27.4
Fe	0.6
Al	0.9
Ca	1.0
	29.9 found 30.1

Na	39.4
S	30.1
SiO_2	3.1
Fe	1.1
Al	0.5
Ca	1.2
C	15.8
	91.2

GASPHASE EXPERIMENT 1939
IN CHAMBER 804
MAY, 1940

I.G. FARBEN INDUSTRIES INC.
LUDWIGSHAVEN-ON-RHINE 687

THE CONVERSION OF COAL-LIQUEFACTION MIDDLE OIL INTO AROMATIC GASOLINE WITH CATALYST 2019-L-8.

SIMON AND SUSSENGUTH 688

Frames 687-698

Translation

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SUMMARY 690

In chamber 804 in a furnace of 760 ltr. reaction space, coal-liquefaction middle oil from the hydrogenation plant at Scholven was converted with catalyst 7019-L-8 at 300 atm. under aromatizing conditions into L-benzine with an end point 160°C. The experiment could be practically conducted for seven weeks without trouble.

The results obtained are compiled with the chief data in Table I.

Table I

I. Operation conditions:

Reaction volume	760 ltr.
Injection amount/hr.	760 kg
Reflux proportion	2:3
Total pressure of furnace mouth	300 atm.
H_2 partial pressure in furnace introduction gas	240 atm.
Total gas/kg of injection	2.6 m^3
Average temperature in the furnace	26.8 MV = 507°C.
Wash oil amount/1000 m^3 of gas	600 ltr.

II. Operation results:

Benzine yield-160°C	0.28-0.30
Benzine concentration %	28-32
% C-gasification or in benzine + gasification	21
H_2 consumption/ton of benzine (15°C, 735 mm.)	720 cm

III. Benzine characteristics:

Specific weight at 20°C.	0.794
Aniline point I °C.	-6.8
Aniline point II °C.	+49.0
% Aromatic substances in benzine	53
%-100°C in benzine	27-35
Endpoint of benzine °C.	160-168
Octane number by the motor method	76.5
Motor method + 0.12 lead	90.0
Research method	88.0
Research method +0.12 lead	99.0

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The benzine produced from the coal-liquefaction middle oil with 53% aromatic substances proved to be superior in motor tests to the commercial C1-fuel. The products are therefore especially suitable as high-output fuels for special purposes.

The catalyst 7019-L-8 in the form of very porous catalyst grains used for the preparation of the products has attained the results expected after the small experiments. Its mechanical stability was, however, as the conditions at the close of the experiment showed, not wholly satisfactory. Through the preparation achieved in the meantime of the catalyst in the usual compact cylinder-shaped pulp, a satisfactory solution has been found in this respect. With this new 7019-L-8 catalyst, CV2b special fuel was prepared from the end of January, 1940, in our large V apparatus.

The experiment was carried out in August and September, 1939.

I. Purpose of the Experiment. 693

According to the experiment in the single cylinder, the aromatic benzine-180°C, CV2a, did not have the expected high performance in the lean mixture testing as did the commercial 100-octane gasoline. The single-cylinder experiment with benzine from an end point of 140°C and 160°C shows, that a maximum of the performance lies at the 160°-benzine, and that the performance of the 160°-benzine exceeds that of the

100-octane fuel. For motor experiments and for the aviation tests greater amounts of 160°-benzine (CV2b) should now be prepared with catalyst 7019.

At the same time the production to be obtained from our 500-furnace (800 ltr. contact space) should serve as the semi-technical first step for the later carrying through of the aromatization in the larger experiment. It would operate with coal-liquefaction middle oil as initial material.

II: Introduction.

The experiment was carried out in the newly installed sump-phase apparatus for 700 atm., since the existing 300-atm. gas-phase apparatus was needed for another purpose. The apparatus was used practically unchanged; only an electric preheater was installed in place of the sump-phase gas-preheater, and in the 12 m.-long, 850/600 mm diameter sump-phase reaction furnace of 955 ltr. content, a catalyst tube with 5 694
stops was installed, so that the reaction volume was reduced to 770.5 ltr. The radiation proportions in regenerator and furnace are naturally different in the 700-atm. apparatus operated only at 300 atm. than in the normal 300 atm. plant.

The apparatus used is seen in detail from the accompanying drawing RSK 4528 d-4/1575 d. The principal data of the individual high-pressure apparatus is the following:

1.) A preheater coil operating with 20 atmospheres excess pressure, 44.5 mm diameter, 12 m. long, content 55 ltr., heating surface 13 m^3; (the coil arrangement is designed to obtain a temperature of approximately 100°C) through open heating of the gases before entering the furnace.

2.) A regenerator, 850/600 mm in diameter, 12 m. long, 12 tubes 18/10 mm in diameter, a central heating surface of 61.3 m^2; stream cross-section in the outer part is 279 cm^2, in the inner part 100 cm^2;

3.) An electric preheater; 6 tubes with a diameter of 48.25/24mm. and length of 10.1 m, 10 tubes with a diameter 44.5/24 mm and length of 10.1 m, and an inner heating surface of 12.2 m^2;

4.) A furnace, 850/600 mm in diameter, 12 m long, with a 5-stop catalyst tube with 5 inlets, a reaction volume of 770.5 ltr., method of production from top to bottom;

5.) An air cooler, 24 mm in diameter, 70 m long, cooling surface of 142 m^2;

6.) A shelved cooler, 24 mm in diameter, a central cooling surface of 43 m^2;

7.) A horizontal product stripper, contents 600 ltr.;

8.) A shelved washer, 34 mm in diameter, 50 m long; 695

9.) A vertical water stripper, contents 346 ltr.

The product injection was done by our normal coal paste press, which worked very well for the purpose during the whole experiment. The sulfuring needed for the maintenance of the catalyst activity was done by the dissolving the determined amount in respect to elementary sulfur in the injection product, in the suction pump before the injection press.

Rinsing water can be injected before and after the regenerator, after the air cooler and after the product stripper; in general the water injection before and after the regenerator was satisfactory. The remaining inlets were suitable for the sump-phase experiment in preparation, but were used only occasionally.

The product expansion from the high-pressure stripper was undertaken as usual in two steps:

Step 1. Expansion of the product in a horizontal 300 atm. high-pressure flask from 300 to 50 atm. of nascent gas = waste gas I.

Step 2. Further expansion of the product to 1 atm. nascent gas = waste gas II.

The impurities in the circulation gas were, in the first week of the operation of the apparatus by expansion of the circulation gas behind the water stripper and by increased fresh-gas addition, decreased to contamination degree of about 10% N_2 corresponding to 0.19 kg/m^3 density in the furnace entrance gas. After the final regulation of the water plant, the circulation gas was no longer expanded, but the 696
gas was washed with about 600 ltr. of wash oil per 1000 m^3. The arrangement of the wash plant can be found essentially from the accompanying drawing RSK 4315/1528. The chief data on the high pressure apparatus is as follows:

1. A washer, 640 mm in internal diameter, 5 m long, about 3/5 filled with Raschig rings;
2. A stripper, 145 mm in internal diameter, 3.5 m long;
3. A horizontal oil expansion flask.

The wash oil expansion was in the same manner as the product expansion carried out in two steps 300/50 atm. and 50/1 atm. under the freeing of wash gas I and wash gas II.

In place of the washing with wash oil in the washing plant, for a few days the washing was conducted by means of compression and re-injection of the expanded product stripper products into the wash-water injection-stream after the regenerator. The wash effect was likewise good and was used abundantly.

In the experiment apparatus the different liquid-position instruments should be examined (See the technical report of Dr. Hamacker's high-pressure experiment). They were installed:

1.) in the product stripper: the Induction-liquid-indicator at Leuna,

2.) in the water stripper after the product stripper: the Berger-liquid-indicator,

3.) likewise in the water stripper as a parallel measurement to 2.): the Pfleiderer-liquid-indicator,

4.) in the oil wash: the Gerwig-indicator.

Collectively the four indicators give unvarying and uninterrupted measurements of the liquid level. So far as a distinct valuation can be taken, the Induction-liquid- 697
indicator and the Pfleiderer-liquid-indicator show the liquid level most quickly and most sensitively, so that the operators can work best with these two pieces of equipment.

The starting up of the apparatus, which was delayed somewhat by trouble in the electric heater and by unexpectedly high radiation-loss in the 700-atm. apparatus, took place the beginning of August, 1939. It is still to be mentioned as a special precaution, that we had arranged between the catalyst tube and the furnace casing a small gas washing, since in the case of the preliminary experiment this intermediate space was filled with the above boiling fractions of the injection products which produced a high heat radiation of the furnace on the whole extent of the housing.

III. Procedure.

The initial product for the production explained in detail in the following was coal-liquefaction middle oil Scholven 180-32°C (without sump-benzine). The products were injected, in mixture with a particular amount in b-middle oil after 696
sulfuring the mixture to about 0.3% amt. of sulfur, into the high-pressure chamber maintained at 300 atm. entrance pressure; and were pressure hydrogenated under heating in the regenerator and electric preheater to 500°C with about 2.5 m^3 circulation gas/m^3 of injection over catalyst 7019-L-8. The product which accumulated in the liquid form in the high-pressure stripper was expanded in two steps 300/50 and 50/1 atm. and was decomposed in the distillation into benzine-160°C and middle oil-160°C. The accumulating middle oil-160°C went as b-middle oil again into the high-pressure chamber.

Report 578-46R

Hydrogenation Works, Poelitz.
Plant report No. LU-558
Dated May 1940

BALANCES AND PRODUCT INVESTIGATIONS
PROCESSING of ANTHRACITE COAL LIQUEFACTION'S MIDDLE OIL INTO AROMATIC BENZINE OVER CATALYST 7019-18.

Frames 698-756

Abstract

This paper contains data on a major hydrogenation experiment in the gas-phase conducted at the Poelitz plant in 1939. For this purpose the high pressure chamber No. 804 was used and operated at 300 atmospheres pressure. This particular report is designated as No. IV and describes mainly the experimental procedure with a detailed analysis of the results obtained. The numerous tables, graphs and flow sheets attached furnish complete data on heat balances, yields and specifications. The benzine manufactured as a result of this experiment has proved itself in actual motor test to be superior even to the customary "C-1" benzine with an octane number of 100, which is evident in the graph illustrated in table No. 7. This heavy duty benzine designated as "CV-2b" is therefore particularly suitable for special purposes and as aviation gasoline. It is of interest to note that the nature of the catalyst bulk material used in this experiment and designated as Contact #7019 I-8 consisted chiefly of coal "A", formed and pressed into small cylinders 8 mm long by 5 mm in diameter, after which it was penetrated with a single contacting substance. Item "A" of this report outlines the conditions for the anticipated run and describes the initial preparations necessary for bringing the unit on stream. Item "B" contains a series of tables and graphs giving data on daily results of charges and yields. Item "C" shows total production balances in tables and charts. Item "D" furnishes data on solubility of gases generated. Item "E" contains tables of heat balances for the process and also discusses the effects of using quenching oils in controlling the reaction temperatures. Item "F" consists of daily production sheets giving an analysis of the initial charging stocks, stripper products and final products. The report is concluded with Item "G", an analysis of the finished benzine, CV-2b.

CONDITION OF THE CATALYST

Frame 734

Abstract

1. Catalyst Condition

The catalyst 7019 LB, containing activated Charcoal, had been shaped in a press for wire cutting to the customary cylindrical form, about 8 mm. long and 6 mm. in diameter. Furnished to us by Leverkusen a Catalyst factory in Ludwigshafen, this activated charcoal catalyst had been saturated by a separate catalyst and dried again in a reduced atmosphere. These individual catalyst particles could be easily pulverized by only moderate pressure This was due to many cracks and crevices in their surface. The catalyst which could be successfully prepared by pressing tight in a mold, yet was not very suitable for technical use in this condition, had the following developement evaluations to be considered.

Hydrogenation-Pölitz
Joint Stock Company
Dr. Kurt Wissel

Stettin-Pölitz 1/12/44

STABILIZATION OF THE PRODUCT FROM THE LIQUID SEPARATOR UNDER PRESSURE WITHOUT INTERMEDIATE EXPANSION AND COMPRESSION OF RICH GAS

Frames 757-767

Abstract

In the period following the destruction and damage inflicted on the Hydrogenation Works at Poelitz by the last bombing attacks, it was necessary in order to put the plant back in operation to partly rebuild the old stabilization equipment used in the manufacture of DHD gasoline and similar products. It was estimated that the time required for rebuilding these units on the basis of the old operating methods was entirely too long and in addition it was felt that the old process would no longer meet future requirements for either specifications or yields. A new method was therefore planned which employed considerably less equipment, shortened the operating time and generally resulted in a higher yield and much better products at a lower cost.

The paper proceeds to describe in some detail both the old and new stabilization treatment in both the gas-phase and sump-phase stages with a comparison of results. Six comprehensive flow sheets attached to this report show the essential differences in the old and new stabilization process.

REFINING CATALYST 6108 FROM DHD CHAMBERS

Frames 769-770

Translation

SUMMARY

As the plant yield shows, the production of Catalyst 6108 can be assured as an additional charge to Catalyst 7360 in the refining oven of the DHD chambers. Therefore, the development of the catalyst is simplified. Besides the reservage that originates through exposure to the high temperatures of the DHD oven, then as a result of the method by which Catalyst 6108 is attained, the refining oven and the dehydrating oven both contain the same catalyst.

DETAILED STATEMENT

Catalyst 6108 is alumina in the form of 10 mm. pills. In each refining oven, which contains a deposit of 6 m^3 of Catalyst 7360, 2 m^3 of these pills are installed. Catalyst 7360 is hydrogenated to give olefinic double bonds. An improvement of the durability of the stock of DHD-gasoline will agree with Catalyst 6108 because of the polymerization action. These will be analytically characterized by the aging test, where a value of 10 mg. of resin per 100 ccm gasoline shall not be exceeded after aging.

The values cannot be obtained by addition of Catalyst 6108 only. One is forced to add the gasoline inhibitors which effect a sufficient improvement in durability of stock when added in the amount of 0.01 volume per cent. Therefore, the use of Catalyst 6108 is in addition to the other.

For the operation, Catalyst 6108 is used in the charge. Since the catalyst is regulated only in one compartment, passing to the refining oven during the development of the catalyst, an intermixing occurs between Catalysts 7360 and 6108. This makes necessary a time-consuming sorting by hand. It has been additionally proved that Catalyst 6108 is regenerated insufficiently by firing with air.

Upon repairing of chamber 22 on August 15, 1943, the installation of Catalyst 6108 F770 was assured. A similarity of the finished gasoline mixed with an inhibitor, which was prepared both before and after the fifteenth of August, shows no deterioration during the aging test.

AGING TESTS OF THE DHD-FINISHED GASOLINE
(mg RESIN/100 ccm GASOLINE)

"T.K.No."	July 15 - Aug. 15 Oxidation Test after Aging	"T.K.No."	Sept. 1-30 Oxidation Test after Aging
1170	0.8	1185	3.2
1171	5.4	1186	2.2
1172	2.0	1187	4.0
1173	3.0	1188	3.2
1174	5.8	1189	2.4
1175	2.2	1190	2.0
Average Value	2.9		2.8

In addition, in October the gasoline of both chambers was investigated, of which chamber 21 was supplied with Catalyst 6108 while chamber 22 was not. The following table shows the values obtained for the gasoline containing inhibitor.

	Chamber 21 (with Catalyst 6108)	Chamber 2 (without Catalyst 6108)
Iodine No.	0.4	0.6
Oxidation Test after Aging	2.4	1.0
% Pb Destroyed	27.6	23.4

PROPERTIES OF DHD-STRIPPED GASOLINE BEFORE AND AFTER SUBJECTION TO THE REFINING OVEN WITH AND WITHOUT THE ADDITION OF CATALYST 6108

Frame 771

Abstract

The refractive index of the DHD-stripped gasoline remains approximately the same before and after the addition of Catalyst 6108. However, with the addition of 2 m^3 Catalyst 6108, the initial distillation point seems to be higher, as are the first few values in the distillation. These values are somewhat higher before subjection to the refining oven than afterwards for the material with catalyst added. Two aniline points are given; in one, the values are much higher than in the other. There is no explanation given as to the wide range in values, but it could be accounted by the fact that one was determined before aging and one after aging of the sample. For each of these aniline points the values are higher for the material without catalyst. The per cent aromatics is lower with the catalyst-treated material than with the other either before or after subjection to the refining oven; the per cent naphthenic materials is higher for the catalyst-treated material; and the per cent paraffins remains about the same. The iodine number is lowest for the material treated with Catalyst 6108, next lowest for the non-treated material after subjection to the refining oven, and much higher for the non-treated material before subjection to the refining oven.

Aging tests were performed on the sample containing lead, with and without the addition of inhibitor. The percentage of Pb destroyed appeared to be higher without inhibitor than with its presence. Exact and complete results are given in the evaluation sheet.

DATA SHEET ON CATALYST 7360

Frame 772

Abstract

Two samples were run in order to determine the properties of a substance to which Catalyst 7360 had been added. The values for the various properties, aniline point, distillation points, specific gravity, iodine number, per cent aromatics, etc., appear to agree fairly well. For comparison, the same tests were performed on a sample without this catalyst present. Here the difference in values is sometimes great. Data are given for the results.

NEW DHD CATALYST - 10 mm. PILLS

Frame 773

Abstract

This is a letter from the hydrogenation plant at Pölitz to I. G. Farbinindustrie in Ludwigshafen requesting a replenishment of their supply of the new DHD catalyst. It is stated how long the catalyst has been in use at this plant, and some of the results of its use are given. There seems to be a rise in temperature after 20 hours operation period with the catalyst, because of its poor regeneration. This causes a rise in gas density and a vaporization of about 3 weight per cent.

NEW DHD CATALYST 7360 L - 10 mm. PILLS

Frames 774-777

Abstract

A comparison was made of the operation periods of chambers 21 and 22 containing the same charge and maintained at the same pressure, but each containing Catalyst 7360 in the first three ovens and Catalyst 7360 L in the fourth. The density of the circulating gases in chamber 21 ranged

higher than those in 22, and the reaction in oven 4 of chamber 21 was powerfully exothermic in contrast to oven 4 of chamber 22. Reasons are given for the higher vaporization in chamber 21.

The following measures were set up as a goal: to determine whether oven 4 of chamber 21 contained a greater portion of high-boiling hydrocarbons than the corresponding oven of chamber 22; to compare the aromatic content of oven 4 with that of ovens 1-3; to determine the power of regeneration of this catalyst, therefore the demand for it.

The advantages are given for the use of Catalyst 7360 L, and it is stated that this catalyst might replace Catalyst 7360.

Data are included in tabular form on the results of these experiments.

DHD CATALYST - RECONSTRUCTION CHAMBER

Frame 778

Abstract

This is a letter from I. G. Farbinindustrie to the hydrogenation plant in Pölitz concerning tests made on DHD catalysts from their reconstruction chamber. Results of these tests are given, and it is suggested that these catalysts be sent to I. G. Farbinindustrie for regeneration. It is stated that the catalyst from oven 4 could be used, in extreme cases, as a refining catalyst.

DHD CATALYSTS

Frames 779-780

Abstract

This letter is from the hydrogenation plant at Pölitz in answer to one from I. G. Farbinindustrie. The writer stated that the plant was in agreement with the suggestion that the catalysts from the first three ovens of the reconstruction chamber be discarded. The writer seemed anxious to receive the results from tests that were underway at that time by I. G. Farbin-

industrie on these catalysts.

It is stated that other samples of catalyst taken from ovens 3 and 4 are being sent for activity tests.

The success of Catalyst 7360 L is mentioned, and it is predicted that ~~this new catalyst will soon replace other catalysts.~~

DHD CATALYST

Frames 786-739

Translation

Your tests on our catalysts inserted in chamber 21 have shown that these are in order after one year of operation time. Since further gas formation is normal in the chamber, we disregard the change of catalysts for a time. No residue is present for the dead catalyst 7360 L of oven 4; thus, we were not able to comply with your wish for a three liter sample. We wish to offer you our sincere thanks for your investigation of the catalyst samples. 786

At the same time, we are sending you an outline of DHD yield during 1942. The initial products were greatly varied in your summary; thus it is that heavy gasoline, with an aniline point between 55° and 80°C, was dehydrogenated. This made possible a comparison of the gas formation in dependence on the injected material (See Outline). In November of the foregoing year the reversing of ovens 4 and 5 of both chambers was accomplished. A lowering of the gas formation was reported for a corresponding prolonging of the production period; thus the yield of gasoline specified by you will be attainable.

Hydrogenation Works, Pölitz

CONCERNING: DHD CATALYSTS

The following communication was obtained from Dr. Donath, occasioned by an address in Leuna on November 22.

1. Harmful Effects to Catalyst 7360

In Pölitz it was observed that in the manufacture of Rumanian straight-run gasoline there is a much shorter reaction period than with that of refined gasoline based on the injected material with the same content of paraffinic hydrocarbons. These facts were reported to Ludwigschafen, and thereby the supposition was announced that certain nitrogen compounds in the straight-run gasoline might be responsible. A tank of Rumanian straight-run gasoline was shipped in August.

Ludwigshafen has established through experiments that organic oxygen, nitrogen, and certain sulfur compounds have an influence on the length of operation period of DHD. This can be recognized from the following table.

ADDITION	Shortening of DHD Operation Period	Duration of Operation after Regeneration
Phenol, 1%	From 20 hrs. to 94 hrs. (About 22%)	Regeneration after normal operation period
Phenol, 4%	From 48 - 22 hrs. (About 55%)	Regeneration after normal operation period
Aniline, 0.25%	From 50 - 26 hrs. (About 50%)	Regeneration after normal operation period
Aniline, 0.7%	From 50 - 20 hrs. (About 60%)	Regeneration after normal operation period
Carbon Disulfide, 0.2%	From 48 - 24 hrs. (About 50%)	Regeneration after normal operation period
Carbon Disulfide, 1.0%	From 120 to 17 hrs. (About 86%)	First regeneration after 3 hrs., again after 58 hrs.
Diisobutylene, 10%	From 50 - 50 hrs.	
Isooctane	From 50 - 43 hrs.	

Because nitrogen and sulfur compounds in amounts as small as 0.2 weight per cent are harmful to DHD Catalyst, the operation period is cut down to half its normal time. While the injurious effects are raised by regeneration with nitrogen compounds, large amounts of sulfur cause so much harm that these are still present after repeated regeneration.

A report concerning these investigations may be expected the next time.

(2). The New Catalyst

Ludwigschafen has developed a new catalyst which is effective in the presence of hydrogen at 400° C and 300 atm. pressure. It is a pure aromatic middle oil with a distillation end point of 325° C. unchanged in gasoline with 60 weight per cent aromatics. The catalyst is sensitive to nitrogen and phenolic compounds and effective with a performance of 0.3 - 0.4 kg./Ltr./h. The vaporization is about 15 weight per cent. This catalyst is preferred above all middle oils for the dehydrogention. 789

(3). Graph Included in Report

Aniline Point of the Manufactured Heavy Benzines 787

A. Gas, Coke + Loss from Chamber in Wt. % of Injected Material.

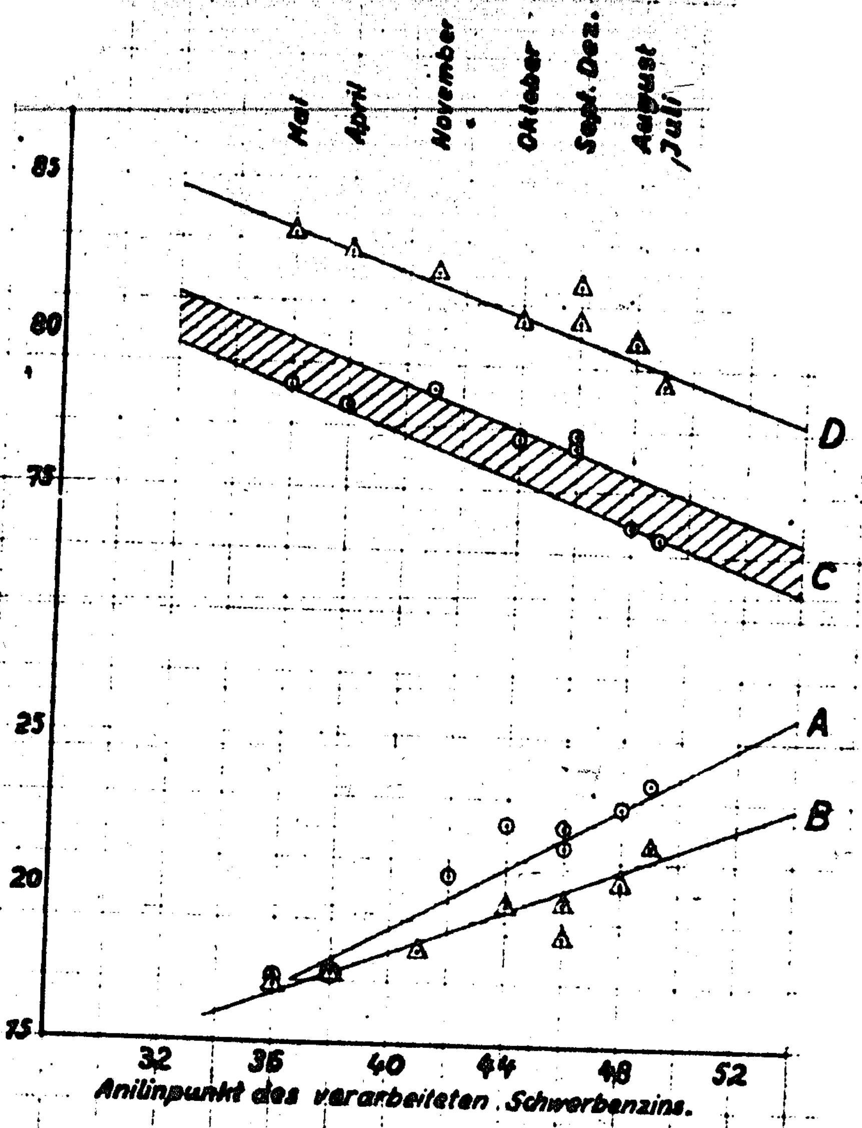
B. Gas, Coke + Loss from Chamber and Distillation in Wt. % of Free C_4 Raw Benzines.

C. Yield in Finished Benzine of Free C_4 Raw Benzine.

D. Yield in Finished Benzine plus Middle oil in Wt. % of Free C_4 Raw Benzine.

D-H-D Benzin. Ausbeute u. Ausgangsmaterial.

D-H-D Anlage Pölitz. Monatswerte 1942.



A. Gas, Koks + Verlust der Kammern in Gew.% der Einspritzung.

B. Gas, Koks + Verlust der Kammern und Destillationen in Gew. % des C_4 freien Rohbenzins.

C. Ausbeute an Fertigbenzin in Gew.% des C_4 freien Rohbenzins.

D. Ausbeute an Fertigbenzin plus Mittelöl in Gew.% des C_4 freien Rohbenzins.

Hydrierwerke Pölitz A.G.
Pölitz bei Stettin

Eich.

Report 578-46R

CATALYST 6434 (VK) in RECYCLE GAS STEAM

Frames 791-805

Translation

SUMMARY OF CONTENTS

Introduction

Diagram:

CATALYST 6434 (VK) in RECYCLE GAS STREAM

Since the introduction of Catalyst 6434 in the hydrogenation plant, its use has been established in these chambers. Of course, the question is as to a permanent deterioration of regeneration, which always limits a rising heat of addition by the heat exchange. These represent, with necessary pre-heater, the limited performance of the chamber after a while. These decreases in heat of transition values, especially in heated regenerators, were noticed in all cases. Powder, which plainly came from the catalyst, was found in the pipes when the regenerators were opened. Shale was observed in many cases at the inlet side and around the regenerator pipes; especially was this true at Leuna, where the product was sulfurized with elementary sulfur. (1)

The following experiments, performed in Leuna, treat the sulfur problem and deal exceptionally with the requirements of Catalyst 6434. This catalyst is best used in pills with an approximate diameter of 10 mm and height of 10 mm; whereby, the cylinder has "Kugelkolotten" an upper and lower boundary area. After a solidification of these pills, which under these new conditions present a porous impression and contain 4 - 6 per cent water, it was observed during the operation that this appeared to be the same one that came into use in 1937 and was referred to as an "improved catalyst." and that it had to its credit in Leuna in oven 216 of Chamber 11 an operation time of 58 hours. To both these quantities of catalysts from later experiments was added a third (Section 111) which had previously been treated at Ludwigshafen. It was treated in Ludwigshafen in order to determine its capability of resistance to moisture at about $400^\circ C$ in nitrogen (later in hydrogen). In contrast to the porous form, the catalyst not previously treated showed itself to be a much more resistant form and apparently insensitive to water.

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(1) Entered in report: Kimmerle, Leuna, September 6, 1937. "Concerning the Content of the Regenerators of 6434 Chambers".

Kuppinger, Leuna, October 22, 1937, "6434 and the Sulfurization of B-Middle Oil"

1. Direction of Flow (2)

The usual direction of flow in the gasoline oven at present is from below to above, not only with respect to the 794 blends installed in the construction at present, but also regarding the favorable temperature distribution with these streams. Therefore, the first series of experiments will pertain to the influence of the direction of flow upon the powder formation and will correspond to Diagram 1. The gas taken from the recycle gasoline underwent several cycles by a rotary pump at 250 atm. pressure.

Before introduction into the apparatus the gas was expanded somewhat, then measured and carried to Tower 1. where it flowed from the upper to lower part and to Tower 11 where the stream was directed to the upper portion. These arrangements had the advantage that both towers exhibited absolutely identical infiltration ratios. The powder formed here was collected as a nettle material in two double sieves and weighed. The determination of the powder followed this method very exactly. The second double sieve normally showed no increase in weight. Indeed the amount of gas, also the resistance in the catalyst column and the first powder sieve, can be controlled by pressure methods. The catalyst tower consists of 120 mm. high-pressure pipes. The corresponding layers of coke are found above and below the approximately 1 m high catalyst column with about 9 kg catalyst.

Before the experiment was begun, the catalyst poured in was blown out from above to below in the customary operation method with nitrogen. After a determination, it was evident that with a careful blowing out about 0.3 per cent of the catalyst weight poured into the tower was lost as powder in a week. With the experiments that followed, which comprised a duration of between 35 and 60 hours, it resulted regularly that little powder disappeared from the first tower in contrast to the amount collected behind the second tower where the catalyst was inserted from the lower part to the upper part. Apparently great amounts of coke were delivered out of the powder. While the powder from Tower 1 (↓) in the first experiment amounted to 2.3 g. and in the following experiment to below 0.2 g., the amount of powder from Tower 11 (↑) was always between 88 and 21 g. In order to hasten the experiment, a velocity of 0.56 m/sec. was selected based upon the free cross - sectional area. (Since velocities were based collectively on the free cross - sectional area, the technical value really amounted to about 2.4). Since catalysts are started out always at these velocities, the limit for the drawing over was found to be 0.48 m/sec. In a farther experiment with 0.43 m/sec. velocity, no more catalyst was drawn over; after 39 1/2 hours passage of gas, the sieve behind Tower 1 (↓) was completely clean. (Increase in weight + 0.12 g), while the sieve behind Tower 11 contained 18.9 g of pure powder.

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(2) Sections landll to the extensive reference on Dec. 12, 1937 upon the experiment exchange in Magdeburg.

11. Flow Velocity

After these experiments, which clearly showed the advantage of downward flow with larger velocities, experiments were conducted with the absence of pressure in order to clarify the influence of the velocity on upward flowing. In glass tubes with 52 or 178 mm diameter it appears that the catalyst layer lying calmly ~~at first begins to be disturbed on the surface with rising velocity. The pills~~ in the middle of the tubes are moved most, as though over a subterranean source. With further rise in velocity, the upper surface is always moved more until they provoke periodic eruptions and the pills are hurled 50 cm. high.

Since all these phenomenons represent a question of equilibrium between the downward effective pill weight and the high pressure of the dust, in order to obtain a dimensionless volume, all velocity is based upon a theoretical suspension volume for an absolutely cylindrical pill. And, of course, the pills are blown in on the front side (of the circle) since they came as freely as if blown in from the cylindrical side. For the value of resistance, 0.91 is set up. Therefore it is that: $V_{sch} = \frac{2.9\ G\ tats \cdot 4}{\gamma' tats \cdot 0.91\ \pi\ r.^{2}}$ (m/sec), where G tats is the effective pill weight in kilograms with respect to the buoyancy, d the pill diameter in meters, and γ'tats the effective weight of the flowing medium in kg/m^3. These mathematically ascertained values do not absolutely correspond to the actual suspension velocity, since sometimes the pills are not a geometrically defined cylinder. Each deviation of the sharp-edged cylinder means a raising of the suspension velocity. In order to get a conception about this we will experimentally take an aluminum cylinder 10 mm in diameter and 10 mm high and set up the air current. As a result, the first such body which had a technical air velocity of 1.07 in contrast to the theoretical suspension velocity, caused about 1 m deep to be suspended. After several repetitions these rise to 1.14, the technical value of the theoretical suspension velocity, since, because of numberous collisions, the boundaries of the aluminum cylinder have become "rounder". An aluminum cylinder with lightly broken edges is necessary for the setting in motion of the velocity, which corresponds first to the value of 1.18 and later to the 1.22 technically theoretical suspension velocity. It can be further observed that fresh 6434 pills need a theoretical suspension velocity value of between 1.40 and 1.55. One is in position to compare the experiments without pressure with the proportions under high pressure by introduction of these theoretical suspension velocities as relative amounts. Since not enough is known of the data found for the chamber in operation, average values will be used for the following calculations, that is, 1.35 g for the weight of the pill and 11.2 mm for the pill diameter.

It has been the result, in the case of experiments in the absence of pressure, that at about 15 per cent of the theoretical suspension velocity the upper surface of the filled in catalyst layer begins to be agitated; after further rise in velocity, finally at 20 per cent of the theoretical suspension velocity, the catalyst pills are flung high into the air, a circumstance which certainly corresponds to the overflow. This knowledge could be transmitted in the first experiment so that it gives 0.48 m/sec. exactly 20 per cent of the theoretical suspension velocity for the mentioned boundary velocity. The relative values of the actual velocity for maximum additions (always based upon a free cross-section) to the suspension velocity can be ascertained for the chambers run in Leuna, Böhlen, and Magdeburg with 6434 catalyst. The ratio of the flow in the chambers with VK 6434 will be compared to a certain extent in Illustration 11. Equipment is specified for all chambers that have been in operation. It can be seen that many experiments have already been carried out in Leuna. Here the picture is not very clear, since the product of regeneration, which has been sulfurized with elementary sulfur, has an unfavorable effect upon the catalyst powder and always causes a shale to form in the outer part of the regenerators. In the cracking that follows the nearest definition of flow of gas and injected material is introduced. Whereupon, it yields at the outlet of the last oven, in combination with the specific gravity, pressure, and temperature, the actual specific gravity, γtats(Kg/m^3), and the effective velocity (m/sec). The theoretical suspension velocity will be calculated according to the usual equation. Finally, the effective velocity will be determined upon the suspension velocity and the relative value, introduced heretofore, will be obtained (in %).

Oven 12 of the chamber in Leuna is to be used for a checking of the relative value. If a large gas-product ratio is found at the beginning, it indicates a high theoretical suspension velocity (2.4 m/sec) and causes a rash increase of the effective velocity so that a maximum relative value of 16 per cent of the theoretical suspension velocity is very extraordinary. If this series causes an unusually rash and strong pollution of the regeneration, effecting also the resistance and heat of transmission, it can only be eliminated by water methods. The K-value in regenerator 1 in the first 3 weeks was lowered to half its number, (300 to 150).

The chamber with four 1200(er) ovens will definitely be equipped. Because 796
of the extremely small gas-product ratio the specific gravity of the mixture will be higher and the theoretical suspension velocity much lower, so that with maximum additions, in spite of the thick ovens, the effective velocity of 10 per cent of the theoretical suspension velocity will be attained. The K-values in regenerator 1 fall from 400 to 160 in the first 3 months and remain constant after another 1 1/2 months.

Chamber 11 was equipped at first with 3 "1200(er)" ovens so that the velocity was slight in each respect. The K-value of regenerator 1, which already had previous operation time, decreased continuously. Then this chamber was equipped with "2 x 800er" and "2 x 1 200er" ovens. It is seen that oven 1, which has an upward flow, has relative values for flowing like those with the first execution of chamber 12. The resistance that occurs and the development shows that the actual catalyst has been drawn over. Correspondingly the chamber with "3 x 800er" ovens has downward insertion. Each possibility of movement and, with that, of grinding of the catalyst will be taken with higher charge. The K-values of regenerator 1 in the first month fell from 350 to 200 then held at this value for 1 1/2 months. The flow technique of the charge with a relative value of 15.9% is so high, especially in the last oven, that it has lead to acute pollution.

The ratios are about the same both in the Brabag Works at Böhlen and at Magdeburg. The A-chambers, which are charged up to an extreme show a continuous change in regenerator 1, while the charged B-chambers, not entirely arranged in layers, (also with VK) show no regenerator change up to 10 per cent and prove to have only a very slow decrease in K-value (by FeS). It is interesting that with both A-chambers trouble with the catalyst has occurred with the same flow ratio. In Böhlen, a sudden deterioration of the K-value together with a rise in resistance has occurred with chamber 3 after installation of a fourth oven in the middle of August, 1937 with a relative value between 13.4 and 13.9 of the theoretical suspension velocity. Agreeing well with this were the results in chamber 4 in Magdeburg on the twenty-sixth of September, 1937. The catalyst of the oven, with a relative value of 13.6% of the theoretical suspension velocity, overflowed and obstructed the pipes.

If all the results of Table 11 are considered together, it can be seen that 797
~~the boundary for the overflow of the catalyst is subject to certain variations.~~ While in Leuna, in experiments both with and without pressure, an overflow was first observed at 20 per cent of the theoretical suspension velocity; the same thing occurred in Leuna (Chamber 11) at 16.7 per cent and in Magdeburg at 13.6 per cent. For flawless flowing technique, the shape of the pills is of decided significance. The catalyst was always ground up for the experiments. However, as with the model experiment on the aluminum pills, this caused a demand for a higher effective suspension velocity. Perfect spheres would later begin to float. The pipe diameter is a further geometric point of interest since the possibility of bridge-atom formation decreases with increasing diameter. In the experiments, the diameter was 120 and 178 mm; in the oven at Leuna (Chamber 11), 655 mm; in the Brabag oven, 860 mm. The influence of the experimental methods had, finally, to be determined, especially on the overflow, to see whether it was smaller for the grindings. A small additional blast of cold gas was sufficient for the overflow with the disturbed upper layer of catalyst. (Sentence illegible)

111. Moist Recycle Gas

After the experiments on the perfect flowing technique, it was desirous to determine the effect of the cold, moist recycle gas upon the catalyst. Comparison experiments were carried out on "new" (run in 1937), "treated", and "developed" catalyst in from 19 up to 33 hours with an uncontrolled, slight stream of cold recycle gas. The results show a powerful decrease in resistance to decomposition (up to 50%) in the "new" catalyst. The treated catalyst has lost its resistance, while the "developed" catalyst has property lost its property of turning to powder. Values for these tests are to be found in Table 11.

In the experiments that followed 100 pills of each of the three qualities of catalysts were decomposed and installed together in a 45 mm. high-pressure pipe with a controlled flow of 0.2 m/sec. The filling up and release of the apparatus took place in the customary 4 1/2 hours. As shown in Illustration 111, the gas from the recycle gasoline was withdrawn and placed in a double-necked flask with a volume of 0.68 m^3; its moisture was taken away in order that it might flow through the coils and the experimental apparatus. Coils and apparatus were installed in tanks, which, when used as water-baths, made possible a series of experiments at higher temperatures. The amount measured was run before the return of the gasses from the suction side of the circulators and then held constant.

The first of the two experiments was carried out at room temperature. The recycle gas flowed through the catalyst at the temperature prevailing in the double-necked flask. The gas temperature in the flask remained beneath that in the circulators. The decreased solubility in the flask was expanded. The experiment time amounted to 0 and 42 hours in experiments with the cold gas. In a second series of experiments the gas was heated to about 40° C in contrast to the temperature in the flask. The average experimental time in a series of 4 experiments amounted to between 12 and 50 hours.

For the estimation of the catalysts its tenacity serves for the time being. It has been determined as the average value out of 10 pills in the material testing of Me 175. Since the tendency toward powder formation cannot be uniformly connected with the resistance, a direct powder measurement was developed. Based on previous experiments, a drum was constructed with two chambers of about 100 mm in diameter and 25 mm broad. (Fig 4).

Abb. 4 (Illustration 4)

Abrieb-Trammel (Dust Drum)

With a 20 pill content of each chamber and 70 revolutions per minute, these pills are continuously being rolled over one another. It was established by experiment that the powder formed, under ordinary conditions, is a square root between 0 and 100,000 revolutions. Normally the dust was fixed with groups of pills of 20 (each 20-30 g.) with 50,000 revolutions and converted after slight deviation from the usual function. The measurements were always carried out so that 2 x 20 pills were first blown powder free; dried 24 hours over phosphorous pentoxide; then weighed exactly; and, finally, set up in both drums of 50,000 revolutions. Following this, the pills were again blown powder free, again dried for 24 hours over phosphorous pentoxide, and again weighed exactly. The difference represents the amount of powder. After each measurement, the drums were blown out.

In spite of the same procedure being used each time, the "treated" catalyst showed a slight addition in weight after the 50,000 revolutions. The powder measurements were carried out separately in both chambers and reduced by 50,000 revolutions. The average values of both measurements gave the final amount of powder.

For the results it had to be established whether all three catalyst types showed a large variation, especially in resistance. During the experiments, samples were investigated before the installation The average values found as well as the range in resistance will be indicated in curves 5 and 6 and also in the following table.

Table 1. Data for the Catalysts before Installation in the Experiment (Average Values).

	Resistance (kg/cm²)	Powder (g with 50,000 rev.)	Weight of 20 pills
"New"	150(126-163)	-0.585	28.4
"Treated"	241(208-260)	+0.290	20.4
"Developed"	193(155-225)	-0.241	23.7

A cold recycle gas flow of 0.2 m/sec was used in the first series of experiments. In this way the mechanical capability of resistance of all three catalysts was lessened. If the resistance decreased, the amount of powder formed was larger.

Table 11. Results with Cold Recycle Gas.

Time of Experiment; (Hrs. Gas Passage)	Preliminary Tests 19	33	0	3	6	12	20	40	42
Resistance "New" Dust	98	85	152	148	135	100	94	92	90
	-	-	-1.8	-2.12	-2.25	-2.90	-3.51	-5.27	-
Resistance "Treated" Dust	185	197	206	208	224	214	208	179	164
	-	-	-0.001	-0.15	-0.44	-0.73	-0.76	-1.01	-1.26
" Resistance "Developed" Dust	175	177	200	191	194	190	188	163	180
	-	-	-0.72	-0.83	-0.86	-1.23	-1.30	-1.80	-2.03

The results concerning the experimental time were plotted in the supplementary illustrations 5 and 6. And, of course, at the same time the installation values are given and those values after the hot and cold experiments were made. Illustration 5 shows the influence upon the resistance. Since it is known to take 4 1/2 hours for the filling up and expansion, the values and range before the installation will be plotted to the left of the abcissa. The irregularity of the outlet material caused by the range of deviation will be recognized, thus a certain deviation may be expected in the results (surprisingly small, however).

In the experiments using cold recycle gas, the "new" catalyst shows a marked decrease of resistance, which is powerfully steep at first, then levels off after the first twenty hours. The "treated " catalyst also shows a decrease with an increasing time of flow, although it is not nearly so great as with the "new catalyst. (This is founded on its remaining solidification in its saturation with oil). The values already given for both of the previous experiments match well with the new values. The same curves show the results of the second series of tests, with the recycle gas heated to about 40 C. No influence on the experiment time is more to be observed with all three catalysts. Especially with the "new"catalyst, 802
a heating causes the resistance to be observed in contrast to the installation value, probably by evolution of moisture which the porous catalyst has absorbed in the course of time. The "treated" catalyst similarly has an installation - resistance relationship. These various tendencies of the cold and heat - treated catalyst with respect to their resistance are extremely interesting. The gain in resistance by heating only to about 40°C is therefore especially indicated in the curve?

A sensitive designation of the catalyst in the form of the powder formation has been developed for the experiment, as was previously mentioned. The results concerning the experiment time were again plotted (Ill. 6). Each point was obtained as an average of determinations on 40 pills, in contrast to only 10 pills in the resistance determinations. The average value before the installation was again drawn in to the left. In the experiments with cold gas, all the catalyst show a strong time-dependence and the "new" catalyst decreases especially. At the best the "treated" catalysts remains the same. In the second series of experiments with warm gas ($\Delta t \neq 40^\circ$), there is with a much smaller value of powder formation, yet only a very slight dependence on time. With the new catalyst this was made essentially better by treatment with the warmed gas than it had been under the installation conditions. The "treated" catalyst shows hardly any powder formation. The gain by treatment with the slightly warmed gas and the resistance - values are indicated in the curve.

IV. Setting Up of a Chamber with VK 6434

Three viewpoints are to be considered in regard to the chambers set up with VK 6434 which were not necessary with 5058. The experiments have clearly indicated that the downward direction of flow carries with it certain advantages, since the very light catalyst will take each motion possibility and powder formation possibility. In Leuna chamber 11 was installed, according to these viewpoints, with downward flow. The success is clear since these chambers are in the position to take up a technical flow velocity of nearly 15 per cent of the theortical suspension velocity. Such charges have a continuously acute pollution of regenerator 1 as a result of the upward flow, which is procured mostly in a deterioration of the resistance and the heat of transition. The poor temperature-distribution with downward flow in thick ovens must be removed by corresponding blend construction.

Another viewpoint to be considered is stopping of the oven filled with 803 VK in order to remove amounts of dust that form with the filling up of the oven. Since the catalyst that was treated in Ludwigshafen has higher resistance and essentially slighter power of dust formation, the amount of powder formed at the introduction of this catalyst is very small in contrast to the amount formed by the new catalyst (run in 1937). The continuous circuit flowing through the cables has a higher temperature than that existing in the high-pressure apparatus, which is filled up from the warm circuit. In consideration of the high hydrogen content of the recycle gas, no cooling effect may be expected. The gas cools with the filling up of the chamber and then condenses; this causes a steady amount of harm to the catalysts. With the "new" catalyst, the extent of the harm is great; with the "treated" catalyst, smaller yet always noticeable. The possibility of condensation must be avoided, then, in all cases.

Upon demand for the charging of the chamber with VK, the following method was used for setting it in motion. The chamber was filled up to 30 atm. from the coiled tube (cable) warmed to 40°C. The water content fell, with the previously complete saturation at the most to 0.00635 kg/m3/eff; that is, the thaw point of these gasses is reached below 4°C. At the temperatures around this point, the gas is relatively dry and not inclined to condensation. In winter with lower temperature, a lower mid-temperature must be selected. The oven filled with catalyst must be heated before the chamber can be further filled. ~~This is only possible by rotation corresponding to the gas measurement in~~ combination with the pre-heating. For the starting, a suitable circulator had to be made; a small Turbo blast apparatus was favorable and was installed only for this purpose. As shown in Ill. 7, which represents the plan of a VK chamber, the blast apparatus was installed between the suction side and the pressure side. It is important that the connection on the suction side behind the stripper in order to make possible the water separation; on the pressure side, before the inlet measurement in order to enable a control of the amount. With this blast apparatus there must be selected under 30 ats. about 260 m^3/eff/h with a eff = 6.5 kg/m^3 compared with 2 - 3"at." resistance in the chamber. For the heat of trasport (existing because of the lower pressures) only about 8000 n m^3/h is left with 1 "at" in contrast to 20,000 n m^3/h with high temperature under 220 "at." (chamber 11, blended Jan. 38). In spite of this the chamber operates in the same way. With the same conductance in the pre-heater, the temperature rise in the pre-heater and the temperature decrease in the oven almost always have the technical value, 2.5. Therefore, the temperature ratio in the regenerators does not need to be changed.

The temperature ratio for two cases of high heating of chamber 11 on Jan. 19, 1938 was plotted. The heavy curve to the left indicates the temperature course in about 5 hours. The temperatures under 40°C in regenerator 11 and the gas cooler are not known. It is essentially the range between the preheater and the third oven. The gas (20,000 n m^3/h) passes from the first regenerator into the pre-heater at 110°C, into the first oven at 160°C, and leaves the last (3) oven at 130°C. In the usual case (8,000n m^3/h), the temperatures change in the sense of the (getstrichelten) curves. In consideration of the increased reflection in the hotter part, the gas in the first oven has a temperature of 225°C in order to leave the last oven at 145°C. The hourly heat of transport was almost the same, therefore. The curve drawn to the right shows the corresponding temperatures for a period of about 10 hours. (140-210-176°C up to 140-295-200°C).

Even more important than the question of the temperature difference is the rapidly increasing capacity of the heated gasses to absorb water, and this without removing condensation about the catalyst. The following table gives the dependence of the temperature on the water saturation, drawn up in effective m^3 of gas,

Temperature, °C	40	80	120	160	200
Water Saturation	0.0509	0.293	1.124	3.262	7.763

If the chamber should happen not to be totally dry at the start, then the gas always has more capacity to take up moisture with rising temperatures. In unfavorable cases, the gas is saturated upon escape from regenerator 1 after moist isolation in that regenerator. This gas flows at first by the pre-heater so that it becomes very dry in the oven. The gas gives off its temperature so that it has an over temperature from the last oven (El. 208) in contrast to El. 18 (See Ill. 8). At this temperature it can be unfavorably saturated where the pressure reduction by the oven is not considered. This makes it impossible for condensation to occur. No moisture enters into the oven from this side with the treated, now absolutely dry, catalyst. The water contained in the gas must result with the lowering of the temperature in the regenerator and again in the gas cooler in consideration of the steep saturation curve; it separates from the gas in the stripper. There is no doubt with other catalysts on the work of the stripper after the foregoing event of the separation of the reduction water.

After the chamber has been warmed up to 250-300°C and dried out simultaneously, the remainder of the pressure filling (up to 220 atm.) can take place after disconnection of the blast apparatus. In Ill. 9 (under Ill. 7) the dependence of the course of the pressure and temperature on the time has been diagrammatically indicated. First the chamber is filled up to 30 atm. in the usual operation time and the chamber warmed up to 250°C. When the chamber was filled up to 220 atm., the temperature sinks to about 25-40°C. Then the further preheating can be correspondingly set up. The moisture coming out of the circulator will be taken up by the warm gas without difficulty.... The temperature will be lowest when one draws on the outlet temperature of the last oven to the heat operations and the over temperature caused by the small amount of gas in oven 1 is not considered. The consideration that the gas must be heated and thereby dry at the escape from the last oven and, also, the entrance into the pre-heater does not give off moisture of the catalyst, remains fundamentally consistent.

REGENERATION OF CATALYST 6434

Frames 814-815

Abstract

A discussion is included on the method of regeneration of catalyst 6434, the price of regeneration, and the effectiveness of the regenerated catalyst. Directions are given for obtaining further information from I. G. Farbinindustrie on this subject.

DELETERIOUS EFFECT OF TEMPORARY CONTAMINATION OF INJECTION MATERIAL IN THE GASOLINE UNIT BY INJECTION MATERIAL FROM THE PREHYDROGENATION UNIT

Frames 816-819

Abstract

The damaging effect of temporary contamination of the injection material from the gasoline unit with injection material from the prehydrogenation unit is discussed. The effect of this contamination (in 25 and 50 per cent amounts) on the specific gravity, the phenolic content, and the base number was determined. Results are given in tabular form. It is stated, in summarizing, that in neither case is irreversible damage done to the activity of the material by this contamination. There is some harm done, but the former activity of the catalyst is attained after a few hours.

PREHYDROGENATION CATALYST

Frame 825

Abstract

This article was illegible in places and, therefore, could not be translated. It is a letter discussing the merits of a prehydrogenation catalyst developed by I. G. Farbinindustrie. A summary is given of materials shipped to Pölitz.

CATALYST 7846-W-250

Frames 826-9

Translation

REPORT ON THE CONFERENCE IN LUDWIGSHAFEN ON JANUARY 30, 1942

Catalyst 7846-W-250

826-

The procedure with Catalyst 7846-W-250 was occasionally spoken about at the conference about "Brüxer" tar in Ludwigshafen. The Ludwigshafen men reported that this catalyst acts, generally, as Catalyst 5058, that a high oven temperature must be selected in order to obtain a good reduction of nitrogen and phenol. Moreover, the aniline point of these catalysts must be standardized in B-M oil so that an unobjectionable manufacture of the product amount of Catalyst 6434 is guaranteed. It has been shown that a great fluctuation of the aniline point presents a harmful effect when a sufficiently average aniline point was attained in the injected material; that is, Catalyst 6434 is extremely sensitive to a fluctuation of the aniline point. Since no clear information is on hand at present concerning the amount of the aniline point in the resulting product, it is recommended that the product be passed into the prehydrogenation chamber of a small experiment oven filled with Catalyst 7846-W-250. It is further mentioned that the injection product must be well sulfurized. In the majority of the experiments in Ludwigshafen, a sulfur content of 0.3 per cent in the injection material has proved sufficient. The addition is about 0.6 at the starting period, and this may be raised to 0.8 after a time.

Dr. Peters reported concerning small experiments with Catalyst 7846-W-250. These experiments, carried out with an addition of 0.80 at a pressure of 250 atm., a product/gas ratio of 1.3 with an oven temperature of 22.5 m and a CS_2 addition of 0.4 per cent were passed into the injection product.

A coal - s - Bi + M' oil with an aniline point of -10°, tested under the usual conditions, showed that the B middle oil obtained therefrom can be tested without difficulty about Catalyst 6434. The same favorable results were obtained in the manufacture of a Rumanian "Erdöl" - s - middle oil with 94 per cent, 350°, which had an aniline point of 58°C in the injection product and an aniline point of 62°C in the resulting B-middle oil. A B-middle oil from pitch, which was also maintained under the same conditions as Catalyst 7846-W-250, had an aniline point of 36°C... 827

Dr. Simon reported concerning a large-scale experiment which was conducted in chamber 801 with the new catalyst. An s-benzine and s-middle oil from Upper Silician coal with an aniline point of -7° was chosen as the injection product. The injection product had preserved a sulfur addition of 0.3 per cent. The average oven temperatures amounted to 21.4-2.15 MV. The product/gas ratio was about 1:2.7. The resulting middle oil with a phenol content of below 0.01, a nitrogen content of below 0.005, and an aniline point of from 47-50° lends itself unexceptionally to the working of Catalyst 6434.

In a second large-scale experiment, an s-middle oil from Gelsenberg coal with an aniline point of -13° was used with an addition of 0.8 at a temperature of 21.6 MV. An aniline point in B-middle oils of + 37° does not make it suitable for a benzination with good performance of Catalyst 6434. A retraction of the addition to 0.6 results in a rise of the aniline point in the B-middle oils from 37 up to 47-50°. These B-middle oils are very effective in the working up of Catalyst 6434.

Mr. Schappert started a report at the time on the recognition of the favorable connection of the regenerators and the preheaters with the combined operation methods with Catalysts 5058 and 7846. These results will be given later.

Catalyst 7846W

Dr. Becker has communicated that an injection product with an aniline point of +20° has been developed which consists partly of a mixture of coal and petroleum products in the ratio 70:30. The oven temperatures were regulated so that after the first oven, the phenol reduction reached at least a third of the corresponding oven volum. (x)The aniline point of the products rises up to 25-26°, and after the passage through the second oven, containing 7846, up to 54-56°. This is true for an addition of 0.5 kg/Ltr. For the best performance of the small oven in connection with these chambers used in operations with Catalyst 6434 has shown that probably the aniline point would not be allowed to fall below 52°.

Dr. Becker states that Leuna has introduced a ligniteous product with an aniline point of +15° and after an addition of 0.8 kg/Ltr. (direct passage), an aniline point of 46-48°. In Leuna until now the phenol content has been experimented with so that after oven 1 approximately 30 per cent phenol reduction occurs (4 fold chamber with a 5058 oven) and after oven 4, it is cut to below 0.1 per cent. It has been a practise that the base in the stripper should not exceed 3-4 mg. NH_3/Ltr.

Dr. Becker has taken notice of our results with interest, and he intends to see that the experiments on Catalyst 6434 (which until now have been on a very small scale) be resumed. Leuna has only allowed one chamber with 6434 to be run as a larger experiment; indeed, the B-middle oil of the 7846-chamber was mixed 1:1 with the 5058-B-product. In this manner the 7846-chamber has been in operation for about a half year without the loss in activity of the catalyst. Dr. Becker believes that a somewhat higher aniline 829 point is probably necessary for the mixture of coal and petroleum products in order to attain a sufficient nitrogen reduction; Leuna is trying to determine the connection between phenol reduction, nitrogen reduction, and 6434 damage.

It appears from the present knowledge, that a certain aniline point difference must be (unterwunden) in order to attain a sufficient reduction, for example:

Pölitz for +20° up to +55° = 35 points
Me. " +15° " " +48° = 33 points

Under the conditions of a higher nitrogen content, the raw products require a somewhat more powerful upward hydrogenation with a mixture of coal

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(x) The tests after oven .1 in Leuna were only taken on 3 hours, only in the starting period tests were taken at intervals.

and petroleum products and not only a higher absolute aniline point, but also a higher aniline point difference is necessary. This is true only in small limits with practically identical initial aniline points. In the majority of the cases a certain relationship holds true between the initial product and the aniline point difference conforming to an added curve where the lowest absolute aniline point and the highest aniline point difference ~~of the B-middle oil is obtained with a pure coal product, and, conversely,~~ the highest absolute aniline point and the lowest aniline point difference with a pure petroleum product.

These possibilities are to be made into an experiment by Dr. Huthwelker, and after closing of the investigation are to be presented for observation to both Leuna and Ludwigshafen.

CATALYST 7846-W-250

Frames 830-833

Abstract

This letter from the Pölitz Hydrogenation Plant discusses the large-scale operations there using the new prehydrogenation Catalyst 7846-W-250 as a replacement for Catalyst 5058, which had become increasingly difficult to obtain in sufficient quantities. A detailed explanation is given of the operatious method, the experiments run, and the conditions under which the experiments were carried out Results are presented and an evaluation is made of this catalyst for use as a prehydrogenation catalyst.

CATALYST 7846-W-250

838-850
Frames [illegible]

Abstract

This is a series of letters, articles, and reports concerning the development of Catalyst 7846-W-250, its properties, power as a refining agent, operation characteristics, etc.

The harmful effects of Catalyst 6434 on the effect of this catalyst are reviewed. The relationship of these harmful effects to the N and phenol content has not yet been established. It is stated that to offset these effects, the injection product (composed of 30-40 per cent of mixture of crude oil distillations and sump-phase products and 60-70 per cent coal and sump-phase products) must be hydrogenated in the prehydrogenation to an aniline point of 52-53°C. Results of both small - and large-scale experiments along this line are given.

A report is made from Pölitz on the stage of experiments in their test chambers at the time. This includes the conditions under which the experiments were carried out, conditions of the tests made on the products from the test chambers, and the results of these tests.

The decomposition of Catalyst 7846 is reported from both Pölitz and I. G. Farbinindustrie. From experiments at one of these plants, it is reported that after 3 months 0.6 addition, operations remain uninterrupted. The decomposition of this catalyst is not nearly so great as with 5058.

A comparison of Catalyst 7846-W-250 with Catalyst 5058 as a refining agent proves 7846 to be equivalent to 5058, in all respects. Analytical data are given for the injection products and for the tests made on the refined products.

The conditions, procedures, and results for operation experiments made on Catalyst 7846-W-250 are presented. In summary, it can be said that this catalyst has proved satisfactory chemically and technically, but that its analytical regulation offers more difficulty than that of 5058.

RESULTS WITH NEW PREHYDROGENATION

CATALYST AT LEUNA

Frames 858-861

Abstract

A report is made on the results with the new prehydrogenation catalyst at Leuna in answer to a request from I. G. Farbinindustrie for information concerning it. Curves are included showing the essential analytical and operational data obtained. A discussion is made and data are presented for the experimental conditions and results.

CATALYST 7846

Frame 869

Abstract

This letter answers several questions from Dr. Simon of I. G. Farbinindustrie concerning the effect of Catalyst 7846 on operations underway at the hydrogenation plant at Pölitz. It is compared to operations using 5058.

CATALYST 7846-W-250

Frames 870-871

Abstract

Acknowledgement is made herein of the receipt of reports on experiments at Scholven and Leuna. It is stated that, in the outline included, a report is given on the experiments in Pölitz with Catalyst 7846-W-250, which had been used in combination with 5058 in the prehydrogenation of middle oils and in the refining of gasoline.

A statement is made to the effect that, in all experiments to date, 7846-W-250 has proved good in combination with 5058.

OPERATION WITH CATALYST 7846-W-250

Frames 872-874

Translation

1. Summary:

Catalyst 7846-W-250 has proved satisfactory in about 5 months operation with a connected 5058-oven in a threefold chamber, in respect to both chemical and technical methods. An essentially intensive supervision was necessary with the greatly changed injection product in Pölitz, in contrast to the 5058 catalyst, in order that a harmful effect on the refining catalyst 6434 would not occur. 872

Catalyst 7846-W-250 acts as a refining catalyst in regards to its individual property, that of strong upward hydrogenation of low fractions, with very low temperatures the same as 5058, so that the method appears right, to produce possibly little gasoline above the prehydrogenation with powerful injection of 7846 W. Therefore, the gasoline refining is to be drawn upon greatly (with further distillation limits).

11. Installation of the Chambers:

The new Catalyst 7846-W-250, thought to be a substitute for 5058, has been in operation since the middle of February, 1942 as a prehydrogenation catalyst for middle-oil mixtures in chamber 5(threefold chamber = 24 cbm. R. R.); also, it has been in operation since the beginning of June, 1942 as a refining catalyst for gasoline mixtures in chamber 1 (threefold - chamber = 24 cbm. R. R.). The installation of the chambers is visible from the added plan (Design 1.). In both chambers the catalyst is combined with 5058; in time it is installed in the second and third ovens, while the first oven is supplied with 5058. The 5058-oven was set up between the regenators and preheater. These insertions have proved good with both chambers. The prehydrogenation chamber 5 is run with a product having distillation limits from 200-360° (coal portion -325°); chamber 1 refines sump-gasoline and straight-run gasoline with an end point of 170-210°.

111. Operation Conditions and Results:

A. Prehydrogenation

The operation conditions were so fixed that on one hand, with respect to the knock-value of the flying gasoline, a lower aniline point was obtained, while on the other hand the upward hydrogenation was sufficient to exclude any harmful effect of the catalyst 6434 added afterwards. For this purpose it was necessary, for the correct comprehension of changes in the operation relations,(especially, layers of raw products), to carry out a proportionally sharp test analytically and by hydrogenation on the strippers (7846 compared with 5058).

The operational conditions of the chambers are visible from the added curves. (11 and 1V).

The oven temperatures were so maintained, that the 5058-oven took charge of 1/3 of the phenol content, corresponding to its portion of the reaction ~~volume. With the most common mixture in Pölitz of 2 parts coal and 1 part petro~~leum product (including S-gasoline), the temperature of the 5058 at nearly 19 mV (addition, about 0.65 kg/l); with pure middle oil (containing no S-gasoline) it must be increased to 20 mV. The temperature of the 7846-oven is 22-22.5 mV, (gestaffelt) in the single oven.

With 30,000 cu. m. of entering gas (beginning period, 35,000 cu. m.) the cold gas varies after the added product proportion between 20 and 30,000 cu. m./h. With an increase in the portion of petroleum, the amount of cold gas goes back in an intelligible manner without injuring the operation methods of the chamber.

Besides the normal investigation of the first and third chambers for phenol and nitrogen content and aniline point, the stripper was run (layer on layer) in a 100 ccm oven in order to prove its cleevability over 6434.

The pressure difference of the chamber had not increased during the operations; therefore, one could say that catalyst decomposition had not occurred.

Refining:

The second chamber was set up as a refining chamber with the catalyst combination 5058/7846 upon the basis of results with regards to upward hydrogenation of single fractions. (See report of March 27, 1942). The endpoint of the gasoline to be refined - bearing in mind the quality of the finished product - was in the limits of 170°-210°.

The refining of S-gasoline and straight-run gasoline carried out in chamber 1 (gestaltet) naturally essentially simple as the prehydrogenation. Here, as in the prehydrogenation, ovens 1 and 3 are constantly controlled, so lower temperatures are necessary (5058-17.5 mV, 7846-18.5 mV) that enough working room is present for the collection of the operations fluctuations (raw products).

In spite of the lower temperature of 5058, these cause a powerful reduction as is observed and appears necessary with respect to its volume-portion. A powerful retraction of temperature of 5058 would not take up an eventual catalyst decomposition.

The hydrogenation effect of catalyst 7846 is, compared with that of 5058, very powerful especially in the lower fractions. This is occasioned by the necessity that the higher fractions, that is, in the same stripper and in the middle oils, must be used with higher aniline points as with 5058 in order to offset the bad effects of gasoline catalysts (6434). The testing upon sufficient phenol and nitrogen reduction and sufficiently high aniline point gives no safe stopping point, in our opinion, but only the testing over 6434 itself.

Thus it results that, in spite of sufficient reduction (under 0.01 per cent phenol) with the prehydrogenation of pure middle oils (without S-gasoline), the addition must be reduced to 10 per cent if no permanent harm of 6434 is to occur. The sufficient reduction of the various product mixtures is to be recognized for the beginning of a new operation period.

REGENERATION OF CATALYST 8376

Frames 881-883

Abstract

This is a letter from Pölitz to I. G. Farbinindustrie giving information on the properties of Catalyst 8376 after it had been regenerated by the former. Its effectiveness in operations is discussed. It is stated how much of the spent catalyst is on hand at Pölitz at the time, how long it had been in use, and under what conditions. A report is included with data on the working conditions, hours, number of employees (native and foreign) and other vital statistics for operation of the plant.

DISINTEGRATION OF CATALYST 5058

Frames 884-893

Abstract

In the form of several reports, a lengthy discussion is given on the decomposition of Catalyst 5058 and the experiments conducted in connection with this decomposition. Several causes are given for this disintegration; data from experiments with the purpose of proving these causes are presented.

The more heavily supported assumption for the decomposition of the catalyst seems to be on the basis of disintegration occurring during change from the liquid to the gas phase. Dr. Schmitt stated that fluids and oils which change over to the gas phase should not be brought in contact with 5058 because this causes the catalyst pills to disintegrate to dust.

From the above assumption that change from the liquid to the gaseous state causes catalyst decomposition, the appearance of catalyst decomposition

(continued)

in chambers 6 and 7 during the experiments may be explained either by (1) rise of pressure difference during operations in the last oven of chambers 6 and 7, or (2) rise in pressure difference in the first oven of chambers 6 and 7 after release of heat.

A second reason for catalyst decomposition is given in defectiveness (lack of solidity) of the fresh catalyst. Tests were made and results are presented in tabular form. In tests on the resistance of the catalyst to pressure it was observed that: (1) Ludwigshafen catalyst (which had been stored a year in Wolfen) had lower resistance to pressure than Leuna catalyst, and (2) decrease occurs in resistance during operation. It was concluded, in summary, that some decomposition might be attributed to this cause but not nearly all of it.

Suggestions were made for the removal of catalyst decomposition. Conditions were listed for experiments to be carried out along this line.

DEVELOPMENT OF COAL CATALYSTS

Frames 902-912

Abstract

A discussion is presented on the development of coal catalysts, including a review of the history of these catalysts, the men who worked toward their development, the experiments performed to accomplish this development. A review is given of the work performed to date on the important iron catalysts: FeS, $FeSO_4 \cdot 7H_2O$, Sulfigran, Luxamasse, Lautamasse, and Bayermasse. Their properties are set forth, showing the adaptability of each as a coal catalyst. Suggestions are given for their use and further development in the future.

EFFECT OF TEMPERATURE ON CATALYTIC DEHYDROGENATION OF MIDDLE OILS

Frames 913-916

Translation

SUMMARY

(1) Scholvener thinning middle oils were passed over Catalysts 5058, 7846, and 8376 at temperatures between 7.5 mV (175°C) and 27.5 mV (518°C) at extensively comparable conditions. (1) The dependence on the temperature of the

(1) 5058 was effective with additions of 0.1 kg./l./h., the thinned catalyst with additions of 0.8 kg/l/h. It is seen from these same addition experiments that one gets about the same results with 0.8 additions of 5058 as with 1.0 with about 0.3 MV higher temperature.

important reactions (hydrogenation, measured by the aniline point; refining, measured by the phenol content and the base number; cracking, measured by the formation of gasoline) is represented graphically in Illustrations IV-VI.

(2) Up to 14.5 mV (295°C) has been referred to under the applied condi~~tions with no hydrogenation reaction to the three catalysts. (2) 8376 is added~~ as the first catalyst, 5058 sometime later. Catalyst 8376 is sufficiently prepared at 21-21.5 mV (about 415°C) with an aniline point of 49, a broad hydrogenation maximum, while Catalyst 7846 has a sharp maximum with an aniline point of 42. Also, with Catalyst 5058 the hydrogenation maximum is at an aniline point of about 51 and a relatively high temperature and is very broad. Strong cleavage exists with Catalyst 5058 in the area of the maximum aniline point, which is not the case with Catalysts 8376 and 7846.

(3) Phenol and base reduction occurs at about the same temperature as the hydrogenation reaction with all three catalysts. At the following temperatures technically useful refining-results were obtained with the individual catalysts under the conditions applied:

Catalyst	5058	7846	8376
Addition	1.0	0.8	0.8
O-Refining (Phenols)	20.0	19.3	18.8
N-Refining (Bases)	19.8	(22.1)	19.9

In phenol reduction the thinned catalysts, especially 8376, are definitely superior to 5058. Practically no difference exists between 5058 and 8376. The values of 7846 are not comparable because of use of other determination methods and will require further testing in an emergency. 914

(4) The gasoline formation occurs at the same temperature as the refining reaction, since the gasoline is formed at first exclusively by reduction of the phenol and base. The results obtained may be summed up as follows: with the thinned catalysts 7846 and 8376 the gasoline formation increases so long as the phenol and base reduction is practically complete (the order of magni-

(2) In the absence of substances harmful to the catalysts, the catalyst olefins (for example, diisobutylene) hydrogenate completely at temperatures under 12 MV (under about 250°C). The hydrogenation of pure aromatics occurs first with an 0.8 addition (in the absence of harmful substances) at about 15 MV (about 300°C).

tude of 99 per cent exchange). This was attained with Catalyst 7846 at 21 mV, with Catalyst 8376 at 18.5 mV. After higher temperatures, there followed with both catalysts a period of constant gasoline formation. (additional gasoline formation only by phenol and base reduction!. The area extends to 23.7 mV with 7846 and to 21.5 mV with 8376. At these temperatures, the gasoline formation begins to increase anew. The gasoline produced beyond the phenol and base reduction originates by true C-C-cracking. (Crack-gasoline). With 5058 the proportion is otherwise. Phenol and base reduction is practically complete at about 20.0 MV. However, true cleavage begins as early as 17.1 MV. Consequently, constant gasoline formation is the case in no sphere and the curves for gasoline formation follow an ascending course up to the highest temperatures.

(5) The vaporization is very slight (practically nill) as long as no true cracking takes place, calculated both on the injection as well as on the gasoline plus vaporization. With the beginning of cracking, the vaporization rises upon injection, and, since the content of total gasoline increases in cracked gasoline, the vaporization of gasoline plus vapors rises also.

(6) The distillation end points of the stripper of the 7846 and 8376 experiments indicate that these catalysts polymerize in the range of 13-15 MV. Catalyst 5058 was not investigated in the temperature range of these experiments.

(7) It is shown that the hydrogenation activity of Catalysts 5058, 7846, and 8376 is not harmed in the manufacture of coal thinning agent middle oils at the highest temperatures, except with operation at extremely high temperatures. With Catalysts 7846 and 8376 a plain decrease can be established at 27.5 MV in a few days. In 1941-43 experiments were conducted to establish the influence of the temperature upon the results of the prehydrogenation of Scholvener thinned middle oils with Catalysts 5058, 7846 and 8376.

Catalyst	5058	7846	8376
Pressure (at)	250	250	250
Addition (kg/l/h)	1.0	0.8	0.8
Temperature, MV	16.0-25.5	75.-27.5	7.5-27.5
Gas, B-oil (kg/cbm)	3.0	3.0	3.0
C S_2 Addition to oil (%)	0.4	0.4	0.4
Oven Volume (ccm)	200	200	200
Initial Product P 1271 on	Mar. 15, 1943	June 16, 1941	April 29, 1943
Beginning Dist. Point, °C	200	185	186
%-225, °C	28	27	30
Ending Dist. Point, °C	330	328	330
A. P. °C	-22.0	-21	-27
% Phenol	14.2	20.6	13.0
% N(Dumas)	0.64	0.80	0.63

The conditions admit a rather extensive comparison to the experimental results. A higher addition was chosen with 5058 than with either of the thinned catalysts, since those must also be used in the large-scale experiments with higher additions. The difference in phenol and nitrogen content of the initial product has the effect that with Catalyst 7846 at low temperatures the phenol and nitrogen content of the accumulated product is somewhat higher and at high temperatures (with extensive refining) more gasoline will be obtained in the amount collected than would be the case if the initial product had a similar analysis to the initial product for the 5058 and 8376 experiments.

While the results were obtained with Catalyst 7846 in a single experiment, while two experiments were necessary with Catalyst 8376 and three with 5058. The results show that the experiments were very reproducible.

Altogether, experiments were run that were begun at usual temperatures (5058, 20.0 MV; 7846, 20.5 MV; 8376, 22.5 MV). Then the temperature was lowered gradually and then run again for a time at the initial temperature. The temperature was next raised gradually to 25.5 and 27.5 and once again run for a time at the initial temperature. The following results were obtained with the various periods held at the initial temperature:

Catalyst	5058		7846	8376	
Temperature (MV)	20.0	20.5	22.5	22.5	22.5
A. P. at Beginning of Experiment	47.0	47.0	40.0	48.0	47.0
A. P. after Running at Lower Temp.	47.5	-	42.0	47.0	-
A. P. after Running at Higher Temp.	-	39.0	28.0	-	40.0

It is seen that running at lower temperatures (with 5058, up to 16 MV; with 7846, and 8376 up to 7.5 MV) does not harm the catalyst, while high temperatures (with 5058 up to 25.5 MV; with 7846 and 8376, up to 27.5 MV) have an injurious effect on the hydrogenation activity of all three catalysts. The same applys to the refining activity.

Results of the experiments are summarized in Tables 1, 11, and 111 and are graphically represented in Curves 1, 11, and 111.

CATALYST COMPOSITIONS

Frame 967

Translation

Catalyst	Composition
Hydrogenation, Gas Phase	
5058	100% WS_2
7846-W-250 (substitution for 5058)	10% WO_3 + 3% NiO + active alumina
6434	10% WS_2 + Terrana
Hydrogenation, Dehydrogenation	
7360 (1. Refining Catalyst)	6% "M O_3" + alumina
6108 (2. Refining Catalyst)	alumina
Hydrogenation, CV 2 b (Aromatic) 7019	15% CrO_3 + 5% V_2O_3 + A-coal
Hydrogenation T 52 - Ka/a	
3076	2 Molecular wts. Ni S + 1 Molecular wts. WS_2
Hydrogenation, Sump Phase (Tar)	
11002 Me - Lu 5475	2% M O_3 (hot ashes)
6612	10% $FeSO_4$
10927 Me	10% $FeSO_4$ + 5% NaOH
Hydrogenation, Sump Phase (coal)	
Iron Sulfate	$FeSO_4 \cdot 7H_2O$
Sulfigran	Na_2S
Bayermasse, Dried unformed	SiO_2 - 7.6% CaO - 17.6% TiO_2 - 7.7% MgO - 0.7% Fe_2O_3 46.0% SO_3 - 0.9% Al_2O_3 12.7% Na_2O 6.5%
T 52 Arrangement	
5063 (Dehydrogenation)	Al_2O_3 + CrO_3
4821 (Polymerization)	———
Cracking Catalysts	
2041 - BZ Catalyst	Ni ore
Brown Oxide Catalyst (Conversion)	
Zinc Oxide (org. S purification	
Bayermasse in Pills (org. S purification)	

(continued)

Luxmasse (Tower purification)
~~Lautamasse was installed in its place with about 45% H_2O.~~

53.7% Fe_2O_3 (u. Ti)
10.1% Sand u. Ton
74.0% Hydrated Material
22.2% Alkali

16-Claus Catalysts (Claus installation) alumina
(2 Types: soft and hard, Ratio 1:1)

Only a portion of this frame was translated, the part containing the actual percentage composition of the catalysts. The remainder of the chart gives other data such as: the plants where each catalyst was prepared, amount prepared, storage time, cost of preparation, etc.

DATA ON GAS HYDRATES
STOPPAGES DUE TO GAS HYDRATES 972

TABLE OF CONTENTS

Frames 973-986

Translation

I. After the introductory greeting by Dr. Pier, Dr. Wiesel reported on the case of the "shut down" of coal chambers 13 and 15 in Pölitz. 973

Pölitz has four coal chambers, two of which are joined in a circuit. The pressure differentials in this circuit amounts to 60 atm. The pressure differentials in the coal chambers amounts to 30 atm. in each chamber. On the pressure side the gas circulation is charged through the coal chambers in two parallel streams, separated from the gas circulation of the two tar chambers; on the suction side the gas from the tar chambers is no longer charged separately, so that three parallel streams are present. The fresh hydrogen is added on the pressure side of the circulation pump.

About eight days before the accident, a sharp reduction in throughput became necessary because of the deficiency of the water-works due to ice formation in the canal, whereby a greater abnormality occurred in the chamber operation. On the date of the accident itself, the trouble had not been observed. At 11 o'clock there suddenly occurred at coal chambers 13 and 15 a reduction of the gas pressure and of the total cold gas. The amount of gas at the outset which goes through the chambers, amounts to 25,000 m^3, the amount of cold gas in one chamber is about 30,000 m^3, in the other, slightly less. All initial precautions, like valve-cracking on the fresh gas-and cold gas-feed, brought no improvement. It would immediately reverse on the rubbing oil; and the circulation differential would go up. Pölitz had the possibility of operating the pressure differential at 100 atm; while it normally would stay at 60 atm. Within the next six minutes the temperature in the furnace increased about 27.2 or 27.8 M.V. Although the reversal had worked out on the rubbing oil in the first furnace, it had still not been changed enough, in order to completely lower the temperature. After another six minutes, the gas surge suddenly started again, went through the chamber, after 2-3 minutes burst into the first section of the tube from the second to the third furnace , and 1/2 minute later burst into the second section of the transfer line from the third to the fourth furnace. The emergency sludge removal was put into operation; and no further interference with flow was encountered.

The investigations which afterwards were instigated have shown that 974
hydrocarbon hydrates are probably responsible for the percussion-like reversal. With the expansion of the gas circulation in the next few days, the lumps of gas hydrate were found. After the circulation was at a lesser pressure the hydrocarbons, whose presence was traced back to the disintegration of the gas hydrates were determined by gas analysis. Even a few days later, gas hydrates were still being blown out. The circulation in Pölitz is operated with water injection. The water is separated out of the circulation before the oil wash.

It is especially to be regretted that in the neighboring chamber 14, in which people were working, two persons were fatally burned. It was decided that for people who work in a chamber near a running chamber, warning signals would be provided to give indications of possible danger.

Fitting in with the report, photographs are attached, which show the two damaged chambers.

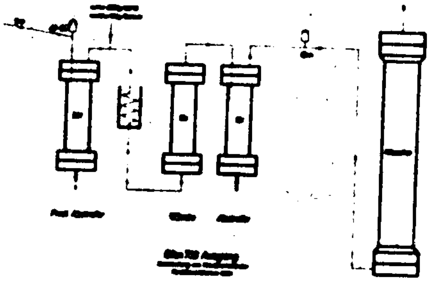
Dr. Pier: The official report concerning the accident has been sent to all participants in the conference. For explanation one has commented in the first two lines on the clogging of the gas circulation system, regarding which, actual investigations have shown the presence of gas hydrates.

Dr. Pier then proposed that the representatives of individual plants report on their knowledge of gas hydrates. Likewise it would be important to speak on the removal of ammonia and the formation of ammonium carbonate. It is known that above a certain temperature 35-40°C, gas hydrates are no longer stable, immaterial of what water concentration and what carbon concentration is present. Out of the literature and experience of the American oil industry, it is known that the different gas hydrates are influenced mutually. In the presence of ammonia, oil, or alcohol the dangers of gas-hydrate formation are less. Also by extraordinarily large amounts of water the gas hydrates are removed. However, it is practical to employ as little water as possible. With the raising of the temperature, the water vapor concentration increases in the circulation, thereby forming a higher partial pressure. If, then, 975
in the case of a long circulation, refrigeration sets in somewhere, there occurs the condensation of the water vapor and therefore the possibility of gas-hydrate formation is given.

Dr. Simon reported on the trouble which has occurred in the large and small apparatus at Ludwigshafen because of the propane-hydrate formation. The first trouble which could be traced back to the propane hydrates occurred at the 100-liter furnace in June, 1938. The furnace would operate at 700 atm. with cracking residue, and with a gas circulation amount of 185 m^3/hr. The gas circulation system contained about 18 per cent hydrocarbons; the carbon average was 1.4. There appeared a pressure difference, which at first could not be localized. One assumed that the trouble might be traced back to the formation of ammonium salts, and increased the wash-water behind the separator and regenerator up to 1.5 kg/hr. of water behind the separator, 1.5 kg./hr. behind regenerator mouth, and 2.5 kg./hr. behind the product stripper. However, these precautions remained ineffective. One had to operate the furnace cold and shut it off. The examination of passage did not reveal the locality of the contamination. The consideration led to the conclusion that gas hydrates are probably responsible for the trouble. Similarly after the start, pressure difference occurs anew. For retarding the pressure from 700 to 200 atm. reducing the pressure difference still further, the gas hydrates cease forming. With a normal, annexed, flask manometer 976
only the pressure difference as between product stripper and the initial supply lying behind the water wash could be determined. Therefore, this supply was heated; moreover the water washing behind the stripper retracted from 2.5 kg/hr. to 1 x 100 g./layer. Accordingly there occurred no more pressure difference.

der Temperatur steigt die Wasserdampfkonzentration im Kreislauf, dadurch entsteht höherer Partialdruck. Wenn dann bei einem langen Kreislauf irgendwo eine Kühlung eintritt, kommt es zur Kondensation des Wasserdampfes und damit ist die Möglichkeit zur Gashydratbildung gegeben.

Dr. Simon berichtet über die Störungen, die in der Gross- und Kleinapparatur Ludwigshafen durch Propanhydratbildung aufgetreten sind. Die erste Störung die auf Propanhydrat zurückgeführt wurde, trat ein beim 100 Liter-Ofen im Juni 1938. Der Ofen wurde bei 700 atm mit Krackrückstanden gefahren, Kreislaufgasmenge 185 cbm/Std. Das Kreislaufgas enthielt ungefähr 18 % Kohlenwasserstoffe, das mittlere C war 1,4. Es trat eine Druckdifferenz auf, die zunächst nicht lokalisiert werden konnte. Man nahm an, dass die Störungen auf die Bildung von Ammonsalzen zurückzuführen sei und erhöhte die Wasserspülung hinter dem Abscheider und Regenerator auf 1,5 kg/Std. Wasser hinter dem Abscheider, 1,5 kg/Std hinter dem Regeneratoreingang und 2,5 kg/Std hinter dem Produktabstreifer. Aber diese Massnahme blieb erfolglos. Man musste den Ofen kalt fahren und abstellen. Die Durchgangsprüfungen liessen den Ort der Verstopfungen nicht erkennen. Die Überlegungen führten dazu, dass es voraussichtlich Gashydrate sind, die die Störungen verursachen. Gleich nach dem Anfahren trat erneut Druckdifferenz auf. Beim Zurücknehmen des Druckes von 700 auf 200 atm verschwand die Druckdifferenz jedesmal wieder

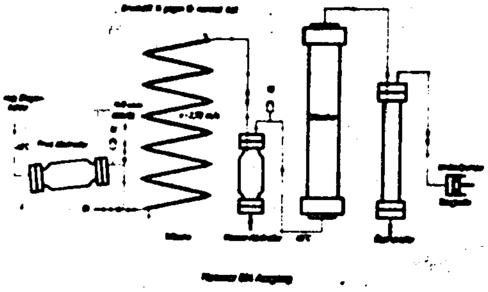


Das liess eindeutig auf Gashydrate schliessen. Mit einem normalen angeschlossenen Kolbendifferenzmanometer konnten nur die Druckdifferenzen als zwischen Produktabstreifer und Ausgangleitung hinter der Wasserwäsche liegend bestimmt werden. Es wurden deshalb diese Leitungen beheizt, ausserdem wurde die Wasserspülung hinter dem Abstreifer von 2,5 kg/Std. auf 1 x 100 g/Schicht zurückgenommen. Danach traten keine Druckdifferenzen mehr auf.

Es wurden zur Verhinderung solcher Störungen zwei Massnahmen getroffen:

1) die gesamten Leitungen werden auf 40°C geheizt,
2) die Wassereinspritzung wird möglichst weit zurückgenommen.

Ein zweiter Fall von Gashydratbildung wurde beobachtet beim Heizöl-Grossversuch an der 700 atü-Kammer 804 im September 1941.



Um die Bildung von Gashydrat zu vermeiden, waren die Leitungen vom Produktabstreifer bis zum Eingang der Ölwäsche auf der ganzen Strecke auf 40°C geheizt; ausserdem wurde warmes Wasser hinter dem Produktabstreifer eingespritzt in einer Menge von 100 Liter/Std., um die Ammonkarbonatbildung zu vermeiden.

Two modes of action were provided for the hinderence of such trouble:

(1) The entire supply is heated to 40°C.
(2) The water injection is retarded as much as possible.

A second case of the formation of gas hydrates was observed in the case of the hot oil experiment in the 700 atm.-chamber 804 in September 1941.

In order to avoid the formation of gas hydrates, the whole distance from the product stripper to the entrance of the oil wash had been heated to 40°C; moreover warm water was injected in the amount of 100 l/hr. behind the product stripper, in order to avoid the formation of ammonium carbonate. 977

During the operation of the chamber (hot-oil-experiment), a pressure difference was adjusted in common in the portion of the supply between the product stripper and oil wash of about 4 atmosphere. The oil wash itself showed no measurable pressure difference. These pressure ratios in the chamber outlets were held approximately constant during the duration of the whole ten-week experiment with one exception: the pressure difference of the chamber increased from about 23 atm. to about 29 atm. The gas-entrance balance showed irregularity, so that the danger existed that the gas entrance might not be held constant. The chamber was surrounded in rubbing oil. The investigation showed, that the pressure difference lay in the portion of the supply between the product stripper and the oil wash. This portion of the supply had been cold through failure of a condensing pot. After the removal of this trouble and the re-heating of the supply, the pressure difference returned to its normal position and the operation could continue without difficulty. The chamber was operated with a circulation amount of 4.350 m^3/hr. The circulation gas was composed as follows:

10%	methane
2%	ethane
1%	propane
0.5%	butane

A third case of the formation of propane hydrates occurred at the DHD chamber with the filling of the furnace with expansion gas. The external temperature was very low, and the pressure was 50 atm.

Dr. K. Winkler reported concerning the trouble in Welheim regarding propane hydrates. With the starting of the sump-phase at 650 atm. in the autumn of 1937, the gas circulation was not heated. Water was injected into the gas-cooler and the circulation system for the avoidance of ammonium carbonate-clogging. Shortly after the start, there occurred, without an especial lowering of the external temperature, in the circulation of the 70th supply, the formation of propane hydrates. The propane hydrates were extracted in bars out of the supply stream. Ever since the whole circulation is kept heated at 30-35°C, and no more water is injected. It is furthermore seen to, that no liquid water is in the circulation system, under the assumption that gas hydrates formed only when water in the liquid form is present. After this precaution no more propane-hydrate trouble occurred. 978

In order to avoid ammonium carbonate formation, Welheim now has no carbonic acid in the circulation, only ammonia. The condition is thereby reached in which the CO_2-containing fresh-gas is added shortly before the pressure bottle of the circulation or directly before the chamber entrance and all of the CO_2 is washed out before the stripper.

A second case of trouble through gas hydrate formation occurred in December, 1940, in the gas phase. Welheim operates with relatively dense gas (0.340-0.450) and has no oil wash. The hydrogen concentration amounts to 53-55 per cent, the pressure to 650 atm. The gas cooler is inserted doubly parallel. One explained the trouble in such a manner, that the stream failed to appear through the cooling system, and thereby the supply became cold. The propane hydrate grew within the supply. It could be removed by leading vapor through the cooler.

A third case occurred at the end of December 1940 and the beginning of January 1941 at the newly mounted sump-chamber. On account of the gas scarcity the chamber was not filled with hydrogen but with circulation gas. All possibilities for propane hydrate formation were given. The pressure difference of the chamber at the start increased suddenly from 3 to 41 atm. In the regenerator, in the furnace, in the supply, everywhere one found propane hydrates, which had formed out of the isolation of hydrocarbons and water. The thawing of the supply continued about 14 days. Ever since, the rule has been followed that one should not fill a new, still moist chamber with circulation gas.

In the United States, one has essentially completely freed the hydrocarbon gas from water, even though it cost much money.

<u>Dr. Frese</u> completed the data of Dr. Winkler on the basis of the data of widely dispersed reports, in which the existing knowledge is collected. 979

<u>Dr. Kuppinger</u> reported on the trouble through gas hydrate formation in 1936 in the gas-phase at Leuna in the case of the operation with catalyst 5058 in the case of pre-hydrogenation. In 1938 there occurred the same phenomenon in Bari, and indeed, for the most part, also in the case of pre-hydrogenation. In both cases the gas hydrate formation occurred on the suction side of the gas circulation supply, when the chamber was only slightly charged and many gaseous hydrocarbons were formed. They could be avoided, if the water injection was shut off in the regenerator.

<u>Dr. Urban</u>: In Scholven no formation of gas hydrates was observed. For the avoidance of gas hydrates the ratio at 300 atm. was more favorable than at 700 atm. Scholven had trouble in the parallel-inserted gas cooler before the oil wash, which, however, was plainly traced back to ammonium carbonate. Now the rule is observed, that every quarter of an hour a man feels the cooler, to see that it is not too cold.

The measured supply is heated continuously in Scholven. The stripper products are maintained at 55-65°C, by throttling of the product cooler, and then usually enter the foam stage.

The circulation is not heated and also not isolated. Scholven has, however, on account of the earlier building methods at the first stone coal hydrogenation plant, a short circulation (only 100-150 m.), while today at the erection of new plants one demands a circulation system that is as long as possible. The chambers in Scholven lie near the relatively hot circulation pump house. The circulation lies rather protected between the buildings.

Into the sump-phase chambers about 800 liters/hr. of water are injected, and the same amount into the gas-phase chamber. Scholven must inject water into the circulation, since usually ammonium salts do not sufficiently pass out. The fresh gas is added before the suction flask.

Also, as yet, no gas hydrates have been observed in the gas-phase at Scholven. The stripper temperature amounts to 25-35°C, on account of the lack of cooling. The residue cooling plants, which are laid out only for two chambers, are insufficient for four chambers. 980

Dr. Urban mentioned further, that at the great apparatus of chamber 803 in Ludwigshaven stoppage often occurred in the parallel tubes of the cooler.

Dr. Jacob reported concerning the trouble in Gelsenberg. Gelsenberg was operated for two years, in which period no water was injected into the chamber, but there occurred in the gas cooler trouble which could be removed by means of the injection of water. It was designated as the formation of ammonium carbonate.

In November, 1939, the then single operating chamber suddenly had no more cold gas. The chamber was immediately freed from mud. After this necessary task the chamber was once more filled with gas. The circulation supply was disconnected, but neither ammonium carbonate nor propane hydrate were established. An explanation was not found. Also in the wash, trouble occurred on account of the pressure difference. In the investigation it was found, that the filter rings had completely pressed together Here, also, neither ammonium carbonate nor propane hydrate were found. Perhaps a short distance in front of the washer propane hydrate had precipitated and then had been conveyed through.

In the gas phase nothing of that kind has occurred.

Since for some time no trouble in the circulation has occurred, perhaps therefore, the gas cooler was so operated that before the wash plant the temperature was held at 30-35°C. The product left the stripper with a temperature of 60°C and in the gas cooler was cooled down only to 35°C. The feed between the stripper and the circulation is heated. Likewise the branches and circuit of the circulation system are heated. Gelsenberg injected into its circulation 500 liters of water per hour per chamber. Before the chamber cooler 1000 liters of water are injected each hour. At the exit of the chamber and the entrance of the gas circulation 0.5 per cent ammonia and 2.1 per cent carbonic acid are present.

Dr. Rosec reported concerning the occurrence in Zeitz in January, 1940. 981
A chamber held in reserve for TTH had been repaired during the cold period. As it was then filled with gas (16,000 m^3), there occurred a strong pressure difference. The pressure difference could be lowered in the course of a few hours, but was not completely removed when the chamber was opened again. The bend of the entrance to furnace 2 showed strong contraction of passage. The stoppage consisted of a snowlike mass and left after its thaw an amount of gas. The gas was investigated and yielded the following analysis;

20 %	hydrogen sulfide
23 %	methane
4.7 %	ethane
27 %	propane
20 %	butane
3.7 %	higher hydrocarbons

Water was not injected. The circulation has a temperature of 30°C. In operation no trouble has been established yet.

Dr. Paukert: The gas circulation at Rheinbraun had in the cooler eight parallel streams; the pressure was 500 atm. In the circulation gas there was 15 per cent hydrocarbons, with a carbon count of 1.3. The stripper on account of the foam must be maintained at a very high temperature, i.e., at 70°C. Into the chamber water was injected at the rate of 1000 kg/hr. The circulation gas had been saturated behind the stripper with water. In the circulation gas cooler, it was cooled to 25°C. Therefore in various parts of the circulation water precipitation was observed. One of the days it was observed, that a few of the eight streams of the gas cooler received no feed. In the stopped up places water was added and vapor introduced; with the result that all the streams were free again. Ever since, the temperature at the exit to the cooler has been kept at 30°C, the clogging up has occurred no more. The content of the circulation gas in ammonia amounts only to a few mg., the carbonic acid to 2-3 per cent.

In the gas phase no trouble has been noted at 30 atm.

Dr. Hahn reported concerning the trouble in the measuring instruments. 982
This was removed by heating the supply stream and using fresh gas as rinsing gas.

Fr. Schmitt: In January 1941 at chamber 6434, which was operated with a deficient charge without lowering of temperature, the pressure difference in the product cooler suddenly increased. The occurrence of the pressure difference, however, was not compelling grounds for shutting down the chamber. As after a short time through lack of products the opportunity was offered to investigate the chamber, and gas hydrates were then found. The gas hydrates occurred at a pressure of 300 atm and a temperature of 15°C. The water injection amounted to 500 l/hr in the last regenerator, 1000 l/hr behind the product cooler and 500 l/hr in each chamber in the circulation. This high water-injection was sent through, in order to lower the ammonium carbonate in the circulation.

In the tar-sump-chamber at 700 atm there occurred, at the beginning of the operation of tar generation, a contamination in the neighborhood of the circulation. The stripper temperature was 30-35°C. At low pressure it was again possible to obtain flow of the gas. It was closed upon the presence of hydrocarbon hydrates. The stripper was then operated at 50-60°C, and no more contaminations were observed.

II. Dr. Nonnenmacher gave a literary summary concerning gas hydrates. The reference is attached as appendix 1.

III. Dipl. Ing. Schappert reported concerning the gas circulation at 983
700 atm., especially from the standpoint of technical measurement. The reference is entered as appendix 2.

Dr. Urban: Scholven has a pressure-difference for the complete chamber; there is measured the pressure difference of the oil wash inclusive of the circulation gas cooler, where the formation of gas hydrates is possible. The circuit around the oil wash can be operated in case of trouble through the quick-closing valve. These valves are on a small space of 2 cm., so that it is possible to operate them within a few seconds by hand. The heating of this small space has been provided for and is easily carried out.

Scholven has on the coal-side no electric valves, but oil-controlled valves. The oil-controlled valves have proved better on the coal-side.

Dr. Kuppinger showed a schematic drawing of the Leuna-circulation. The circulation is about 1200 m long, and operates at a pressure of 250 atm. The pressure difference is measured in Leuna with the usual manometers of the Eckardt firm. The pressure difference in Leuna amounts only to 27-28 atm. In each chamber is an indicator-manometer. At 200 atm. this manometer indicates sufficiently accurate, other measures are not installed. Dr. Kuppinger mentioned that Leuna had installed in the gas circulation supply quantitative measures recommended by Dipl. Ing. Schappert.

Brabag: At Brabag there is essentially the same measuring arrangement as in Leuna. The measurement of pressure difference in the circulation was accomplished according to the Leuna system.

Dr. Frese: In Welheim circulation pressure difference was measured twice. 984'
Welheim has two recording, air-controlled manometers. On these manometers were, however, many apparent deficiencies. They proved all right at a starting pressure difference, but, for example, in the case of a 50 atm.-pressure difference, the manometer required 2-3 minutes to reach the end of its deflection.

Obering. Egli reported concerning the deficiency of the air-controlled manometers. Systematic measurements showed in the case of a great pressure difference a lag of 2-4 minutes until the reaching of the end of deflection, while the flask-difference manometer had a lag of only a few seconds. Herr Egli suggested, therefore, to install, in the important positions of the circulation-pressure differences, an air-controlled and a flask-difference manometer in parallel.

The operations director regulates simply according to the air-controlled manometer. He is, however, in the position to detect abnormal pressure points in the flask-difference manometer. Gelsenberg has wondered, if one gave the man who operates the pressure-difference position the liberty to raise the pressure difference of the circulation pump whether trouble would occur. One came, however, to the conclusion, that the man through independent changes of the circulation-pump pressure differential could do more harm than good.

IV. Dr. Jacob: In the case of trouble with the flow of gas there is an immediate emergency mud-cleaning. First is the No. 1 furnace freed from mud, then furnace 2, 3 and 4.

Dr. K. Winkler- Dr. Frese: In Welheim one likewise cleaned the first furnace first. Welheim has no electric-controlled valves. The valves must be operated by hand. For the emergency mud-cleaning of the first furnace, two valves must be opened. Accordingly the furnaces 2, 3 and 4 are released.

Dr. Urban: Scholven always put into operation, when temperature in- 985
creases occurred in the furnace, the emergency mud cleaning. All furnaces were cleaned simultaneously. For the most part one could operate again in six hours with complete paste amounts.

Dr. Pier suggested to consider, in connection with the trouble in Pölitz, whether one does not provide further places of ignition in the chambers. Also he pointed out the knowledge that in most cases an ignition came about with a stream of gas or combustible liquids. On this account a long discussion was released, in which among others Dr. K. Winkler explained, that the higher the pressure is, the easier do the issuing gases ignite. In one case, however, the gas occurring in a 700 atm. chamber in Leuna ingnited too late. The ignition of the mixed gas cloud had a very great action.

Dr. Pier: Out of the formation which Dr. Wissel showed it resulted, that one must consider how one could either secure vertical piping in the chamber or how one could connect the furnaces overhead with each other in the shortest way. One should avoid the vertical connectionsif possible.

Obering. Berger sketched a proposal, to avoid the long connection tubing, that one lay the ascending tubing in the furnaces in the middle of the dipping tubes and connect the furnaces only through short connection tubing overhead The length of the connection pieces amounts to only 1/5 of the present ascending tubing.

Constructively the conduction is directly possible. Especial care must be laid in this case on the thermal expansion of the hot conduction tubes.

The dipping tubes must be operated for the purpose of preventing coking in the margin of the furnace, similarly as in the case of the hot separator.

The proposal was also made to intentionally weaken the dipping tubes 986
issuing out of the furnace, in order to produce an explosion section in the case of the "running away of the furnace."

Such a weakening is possible and feasible, if only for the above sector (half the circuit) a suitable tube of the length of about 1 m. is inserted in the wall thickness of the tube collar.

High Pressure Experience
LU 558

GAS CIRCULATION CONNECTED WITH HYDROGENATION CHAMBERS AT 700 AT. FROM THE STANDPOINT OF MEASURING TECHNIQUE

Frames 987-994

(Report of Dysloma Engineer Schappert
presented on the 4th of December 1941
in Ludwigshafen at a meeting in which
plant experiences were exchanged)

Translation

The gas circulation diagram is given in simple schematic form in Figure 1, and is based on the Stettiner design =.

Gas circulating pumps
Chambers including the strippers
Scrubbers

Most often in gas circulation several chambers are assembled in parallel with each other, as is also true for most scrubbers which are also assembled in several units. In order that one does not go beyond a certain pipe dimension, for example 12 cm pipe on the 700 at. plant, the piping is laid in parallel, ring connections being preferred. The gas circulating pumps are connected into the bypass, the main purpose of the bypass is to take up the excess gas from the vessels which results from improper control of outlet valves. In principle, however, the following simplified flow diagram always holds:

The most important pressures in the system are always observed while in flow and most always recorded. The absolute pressures P_1 and P_4 are interesting for being above all, the highest and lowest pressures in the entire system.

The pressure difference $P_1-P_4 = P_4$ is obtained from the indicator of a recording differential gage. This differential pressure, whose upper limits are determined by the capacity of the gas circulation pumps, adjusts itself to the chamber with the highest pressure difference, so as to conserve electrical energy.

It has been mentioned that the gas circulation pumps for the 700 atmosphere plant were first designed for a differential pressure of 100 atmospheres, later however, for 70 atmospheres and it is now to be decided if, on the basis of this discussion the differential pressure should be decreased to 75 or 80 atmospheres.

The difference in pressure of each vessel is of importance, depending mainly on the throughput, if no irregularities such as plugging or incrustration cause increased pressure differences. This pressure difference can be determined either as a direct pressure difference or by determining absolute pressures at the inlet and outlet of the vessel.

The inlet valve to the vessel has a variable resistance and is throttled the least, the higher the pressure differential of the vessel. The various pressure differences of the vessels themselves can vary, while the pressures P_3 at the outlet of each vessel are practically equal since the scrubber represents a constant resistance and the suction side of the circulating pump also works at a constant pressure.

It is therefore possible to calculate two pressure differences which are of definite interest:

1.) ΔP_1; next to the pressure side of the piping system with its fittings and flanges the most important part of the resistance must be in the inlet valve. It is a general rule that this pressure difference across the inlet valve should be at least 10-15 atmospheres in order that the cold gas can leave the oven with sufficient pressure. In a few cases this difference in pressure can however be lower, if for example more regenerators than usual are built in or if the preheater represents a large resistance. The cold gas at the entrance to the first oven is under a pressure of around 20-25 atmospheres. If this pressure because of any reason should suddenly be cut in half, it is clear that the mass of gas flowing through the oven will reverse itself, until sufficient cold gas is brought in through the cold gas valve to reverse the flow. It is understandable therefore to want to be able to observe this pressure differential of the inlet valve. Pölitz has for sometime planned to install at this point on each vessel a differential manometer.

2.) The other and almost as important pressure difference ΔP_3 consists of the suction side of the piping system, in particular the oil scrubber which consists of several units. The varying resistances of the scrubbers is compensated through the inlet valves to each scrubber and it is evident that this manner of regulation is limited.

Even though it isn't of immediate importance to register this pressure difference, it is expedient to measure continuously this pressure difference at least by means of the absolute pressures P_3 and P_4 in order to be able to recognize gradually and in proper time any changes that may have set in. Thus one had already been able to measure in Pölitz weeks before the disturbance, the pressure difference of all the scrubbers including the gas cooler and it was observed that the value is higher than one theoretically could expect, as was anticipated due to large temperature changes in the gas circulation cooler causing irregularities in stream relationships. This increased resistance, however, did not cause any production difficulties, since one could increase considerably (to 100 atm) the total pressure difference of the circulating pumps. This fixed increase of the regular pressure difference would perhaps indicate the presence of small deposits. It is best then to perhaps recommend that these temporarily installed instruments in Stettin should at least be installed universally where there is evidence of clogging with gas hydrates or ammonium carbonate.

In order to be able to determine quickly and thoroughly which of the two streams is more or less clogged, one can measure the amount of gas going through each stream by means of an orifice plate or also by allowing the current in a parallel circuit to pass through each stream and measuring the electrical resistance of each stream. One must be careful when continually adding water to the stream. It is not important to measure the mass flowing exactly, but on the contrary, it is sufficient to obtain comparable deflections. One can also install the orifices in a by pass, so that they can occasionally be removed and inspected.

It would be practical to install the differential pressure manometer for ΔP_3, in the control room besides the orifice manometers on both streams. The operator is then in a position to distinguish between the normal pressure fluctuations which result from regulation of the mass of gas in the chambers and fluctuations which are due to clogging. The precautions which are to be taken in both these cases are opposite to one another: In the first case of a real pressure increase one would just open up the valve where in the case of pressure increases due to clogging, the pressure capacity of the circulation pumps must be used to try to dislodge the mass of material causing the clogging.

On Figure II are shown the pressure relationships as they exist for two gas flow streams in which 3 and 4 chambers are arranged together. Since the load on 4 chambers is 1/3 higher than on 3 chambers, the pressure difference is about 50 per cent higher, if one disregards the fact that the inlet gas mass also does not need to be 1/3 higher.

In this example the pressure difference on the gas circulation pump amounts to --55 atm.
with a pump capacity of --------------------------70 atm.
a pressure reserve of ----------------------------15 atm.
is available.

The 55 atm are divided as follows:

1.) Pressure side of flow path ------------------2 atm.
2.) Suction side of flow path and scrubbers------6 atm.

The pressure difference of the chambers may vary between
18---33 atm.

Accordingly the upstream pressure of the inlet valve varies between
29---15 atm.

The cross hatched surface represents respectively ΔP_2, that is, the pressure difference between the inlet and outlet of the chamber and this pressure difference is again divided up into:

(1) The upper single crosshatched surface represents the pressure difference of the regeneration plus preheating

(2) The middle, double crosshatched surface represents the pressure difference of the oven and

(3) The lower single crosshatched surface represents the return regenerators plus coolers ~~plus~~ stripper.

Two important points of views can be drawn:

1.) The upstream pressure of the chamber is supported by the pressure difference in front of the oven since the cold gas is short circuited to a certain degree to the oven.

2.) The chamber with the least resistance also the greatest upstream pressure, is in a less favorable position with regard to the partial vapor pressure of water resulting from the increased upstream pressure.

In conclusion one can say:

The normal equipment of the 700 atm gas circulation systems with regard to pressure measuring instruments, for example,
Absolute pressure of the circulation pump-suction side
Flow path pressure difference
Pressure difference of each chamber
are completely sufficient under normal working requirements

The additional following instrumentation is suggested for the Pölitz plant to prevent clogging should it occur.

1.) Measurement of the pressure difference across the inlet valve of each chamber.

2.) Pressure difference measurement of the entire scrubbing plant.

3.) Eventual installation of an orifice plate in each parallel stream on the suction side of the gas flow system.

4.) Measurement of the flow stream by two means with different instruments based on different operating principles, for example, on air driven differential manometer and a differential manometer based on a floating device.

/S/ Schappert

Frames 995-1010

*The information in these frames is essentially a report of American literature that is already available on gas hydrates.

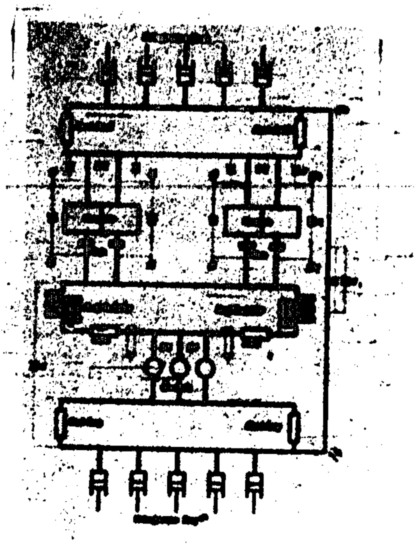


Abbildung I

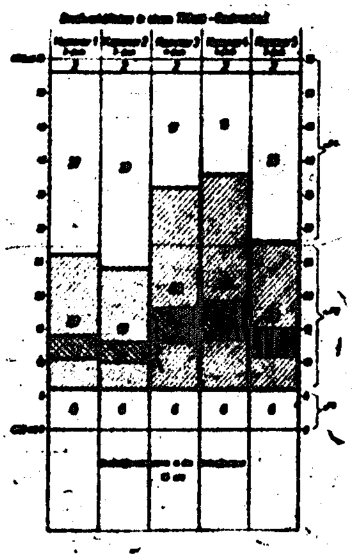


Abbildung II

USE OF LIGNITE AT POELITZ

Frame 1012

Abstract

Neither lignite coke nor dried lignite can be stored nor are cars of the right type for its transportation available. Raw lignite, which contains 50 per cent water, can be stored and could be used. Agglutination of the mills would be expected from its use because of its high moisture content. Transportation of this large amount of water may not be practical.

Uncoked lignite briquettes are quite suitable. These can be stored and transported in a manner similar to that used for coal. They contain 15 per cent water. The jaws of the powdering mills should be narrowed to prevent strike-back of the flames, and air below 100°C should be used. A conference with the furnace and boiler-manufacturing firm is recommended.

CRACKING OF LIGNITE TAR

Frames 1013-18

Abstract

Small-scale experiments on the cracking of lignite-tar to produce J_2-fuel were performed. Catalyst 7360, hydrogen at 50-80 atm., a temperature of 26 MV, and a feed rate of one vol tar/vol. catalyst/ hr. were used. At first, the product contained 50 vol per cent boiling below 330°C. When this fell to 40 per cent, the experiment was interrupted and the catalyst regenerated with air.

Unless the feed was free of middle-oil and of material boiling below 325°, hydrogen was consumed. Raising the temperature to 28 MV greatly increased production of asphalt, without increasing 325°-end point material.

CRACKING IN A COKING OVEN

Frames 1019-24

Abstract

Oven X was operated 338 hours for cracking. Petroleum residue, 0.8 m^3, and recycled primary-cooler oil, 2.2 m^3, were charged hourly and 0.6 m^3/hr (0.5 metric tons/hr: 350 metric tons/month) of secondary-cooler oil were produced. In order to attain a higher production, the drum temperature should be raised to 580° and the burner-jets should be bored out from 13 to 16 mm.

The use of round pebbles in furnace ~~X was~~ unsatisfactory. It delivered coke in large round pieces instead of as dust which was obtained when angular stones were used.

From 100 kg. of petroleum residue, containing 24 per cent boiling below 350°, was produced 21.9 kg gasoline, 50.8 kg middle oil, 18.3 kg gas, 9.0 kg. tar, coke, and loss.

The middle oil may fill the requirements of diesel fuel. Its pour point was below-10° and its cetane-number 38.

The gas contained 13 per cent ethylene, 11 per cent propylene and 5 per cent butylene. Its heating-value was 11,000 cal.

CRACKING IN A COKING OVEN

Frames 1025-33

Abstract

The oven was used for cracking for 16 days. Processed hourly was 2-3 m of petroleum residues at a drum temperature of 550-570°C. The yield per day was one tank car of secondary-cooler oil. This contained 40 per cent gasoline and had an end point of 350°C.

The gasoline produced is similar to gas-phase-cracking gasoline. It contains 55 vol. per cent unsaturates and aromatics.

The coke can be broken out, without interrupting the feed, by increasing the rate of revolution of the drum.

The end-point of the secondary-cooler oil can be lowered to 350°C by a corresponding operation of the (primary?) condenser, so that this product is yielded directly in the gas phase.

At a throughput of 3 m^3/hr of petroleum residue, at 570°C, was produced 0.73 m^3/hr or 0.62 metric tons/hr. of secondary-cooler oil.

PRODUCTION OF MOTOR FUEL BY CRACKING IN A BULB FURNACE

Frames 1034-9

Abstract

Only slight changes are necessary to convert a coking furnace for cracking. It would then be suitable for the production of motor fuel from asphaltic, hydrogen-poor petroleum residues and also, probably, from tars. The gasoline produced is rich in aromatic hydrocarbons and is similar in composition to gasoline produced by gas-phase cracking.

(Two illustrations, depicting the furnace before and after conversion for cracking, comprise frames 1038 and 1039).

CRACKING PROCESS (PATENT DISCLOSURE)

Frames 1040-3

Abstract

Asphaltic petroleum residues or tars are cracked at 350-570°C and near-atmospheric pressures in a rotating oven filled with pebbles. The pebbles serve to pulverize the coke which is then continuously removed by a screw.

From a throughput of 2 m^3/hr. were obtained, in a day, 25 m^3 primary-cooler oil, 19 m^3 secondary-cooler oil, 0.8 tons coke, some tar and about 1200 m^3 gas.

Frame 1043 is an illustration of the equipment.

USE OF STATIONARY APPARATUS FOR THE PRODUCTION OF MOTOR FUEL

Frames 1044-1047

Abstract

Apparatus is described consisting of a tube furnace, separators, coolers, heat exchangers, and fractionating columns. The furnace tubes have a diameter up to 100 mm and must permit pressures of 70 atm. and a temperature of 500°C. It is predicted that 14 tons recycle and 7 tons fresh feed will be processed per hour. An effluent containing 20 per cent gasoline and middle oil corresponds to a production of 4 tons per hour or 3000 tons per month of gasoline and diesel oil.

For cracking at near-atmospheric pressure, a rotating oven filled with pebbles was used. Petroleum residue containing 20 per cent boiling below 350°C was cracked at 550°C. At a throughput of 2 m^3/hr the following balance was obtained over a 24-hour period.

gasoline to 200°C	13.4	wt %
gas-oil 200-350°C	31.1	
product above 350°C	45.8	
coke	1.9	
gas	5.5	
tar and loss	2.3	

It was estimated that by recycling the product boiling above 350°C, the following yields would be attained:

70 %	gasoline and gas-oil
7-8 %	coke
20 %	gas
2 %	tar and loss

CRACKING WITH THE EXISTING APPARATUS AT THE HYDROGENATION WORKS

Frames 1048-56

Abstract

For cracking under pressure, a tube furnace is necessary, besides separators, coolers, heat exchangers, and fractionating columns. The tubes must have a diameter up to 100 mm and must permit pressures of 70 atm. and temperatures of 500°C. A flow sheet of proposed high-pressure cracking operation is presented (frame 1056) and described.

For cracking at atmospheric pressure a modified coking oven can be used. Illustrations of the normal and the modified coking oven are presented (frames 1053-1055). In the modified oven, a petroleum residue containing 20 per cent boiling below 350°C was cracked at 550°C. At a throughput of 2 m^3/hr, there were obtained, in 24 hours, 25 m^3 primary-cooler oil and 19 m^3 secondary-cooler-oil. Principal data are:

	Raw product	primary-cooler oil	secondary-cooler oil
d_{50}	0.90	0.92	0.84
first drop °C	263	275	58
Vol % to 200°C	0	0	30
Vol % to 350°C	22	24	80

Also produced was 50 m^3/hr of gas, small amounts of tar and 0.8 metric tons of coke (in 24 hours).

A day's balance follows:

Charged:	43.2 tons	= 100%

Produced:

gasoline (to 200°C)	5.8 tons	= 13.4 wt %
gas-oil 200-350°C	13.4 "	31.1 "
product above 350°C	19.8 "	45.8 "
coke	0.8 "	1.9 "
gas	2.4 "	5.5 "
tar and loss	1.0 "	2.3 "
	43.2 tons	= 100 wt %

POELITZ CRACKING PROCESS

Frames 1057-9.

Abstract

Cracking of asphaltic petroleum residues is accomplished in a rotating coking oven filled with pebbles, which serve to pulverize the coke which is formed. The coke is fed to a screw, by which it is continuously removed, as a dry dust, low in ash. (An illustration comprises frame 1059)

Report 578-4[illegible]

CRACKING EXPERIMENT IN A COKING OVEN

Frames 1060-1073

Abstract

Oven IX was modified for cracking petroleum residues to coke. The oven operated at 550°C with a charge of 2 m^3/hr. As a result of the experiments it seems worth while to introduce a settling zone between the screw and the condensers, in order to prevent carry-over of coke. Also it seems worth while to control the temperatures in the primary and secondary coolers, so that the kettle product from the secondary cooler will contain the least possible amount of material boiling above 350°C (ca. 95% to 350°C); and so that loss of gasoline overhead from the secondary cooler will be minimized, the head temperature should be about 20°C. The heavy product from the primary cooler would then be recycled.

Illustrations of the coking oven before and after modification, and a flow sheet are presented. (One page of the report is missing).

USE OF STATIONARY APPARATUS FOR THE PRODUCTION OF MOTOR FUEL

Frames 1074-77
(Same as Frames 1044-1047)

Frames 1078-80

Abstract

These are flow sheets of a quite conventional cracking plant.