

Hydrogenation of Coal Extract at 600 atm. in Chamber 17

November 1 to 26, 1938

Bottrop, December 15, 1938.

I. Introduction

Up to the end of October 1938 the raw product for hydrogenation at Welheim was, with the exception of a few days, a mixture of pitch, pitch-distillate, and anthracene oil, since the continuous production of extract did not yet proceed so satisfactorily as to enable an uninterrupted operation for several days of hydrogenation with extract.

The first of the longer operating periods began on November 1, 1938 as experiment 12 in chamber 17, and ended on November 26, 1938; owing to the shutting-down of the extract chamber. During this period it was possible to obtain a rich and satisfactory collection of statistical material on all of the questions hitherto remaining unexplained, which will be evaluated in this report.

II. Equipment of Chamber 17

- 2 regenerators: 12,000 mm. in length and 500 mm. in diameter.
- 1 preheater, 27 hairpin tubes (14,800 mm. in length); 70 mm. in diameter; total volume, 31.7 cubic meters.
- 4 furnaces, 12,000 mm. in length, 900 mm. in diameter; total reaction space, 15.3 cubic meters.
- 1 heat exchanger: 8,000 mm. in length, 500 mm. in diameter.

III. Experiments on Hydrogenation of Extract

A. Hydrogenation of a Mixture of Extract and Fuel Oil in a Ratio of 1 : 1.

1. Method of Operation

The mixture of extract and fuel oil in a ratio of 1:1 (residue from building 14) was mixed in building 29 with such a quantity of desludging residuum that the injection product for chamber 17 had a composition of: 33.4% extract, 33.1% fuel oil, and 33.5% desludging residuum.

The addition of purification mass 11002 amounted to 0.041% of the injection material; the addition of soda, 0.057% of the injection product.

All of the numerical values are averages from November 10-13, 1938, since on these days the chamber operated very uniformly and the necessary equilibrium had been established.

The content of purification mass 11002 was kept very low in comparison with the experiments with pitch-pitch distillate because:

- (a) The low level of the desludging residuum at the beginning of the

experiment (November 1, 1936) had risen to 1% through a large addition of contact to the injection product.

Since the extract was, according to the opinion at that time, free of solids, the solids necessary for the hydrogenation should be provided solely through Mo char; that is, when the operation was effected with 30% of solid material; this 30% should be pure catalyst with the exception of the added soda.

(b) According to the opinion at that time, there should be withdrawn from the cycle in 24 hours only as much desludging-solids, and therewith also active contact, as the quantity of soda introduced to counteract the chlorine.

According to this calculation, the solid-material level would, once it had been established, remain constant at equal amounts of injection material and desludging residuum, in the case of a daily yield of about 500-700 kg. of desludging residuum. Actually, at a charge of 85 metric tons of extract per day, 14 metric tons of desludging residuum, amounting to 4.5 metric tons of solids, were yielded; in other words, solids were introduced into the chamber through the extract which contained about 5.5-6% of solid material. (According to laboratory analyses, the mixture of extract and heating oil in a ratio of 1:1 contained 2.8-3.5% of material insoluble in tetraline and cresol.)

According to this finding, the method of operation with only 0.04% purification mass 11002 (calculated on the injection material) is impossible over a long period, since, just as in the case of experiments with pitch, a part of the contact introduced is always withdrawn from the circuit with the desludging residuum and must be replenished with fresh purification mass if the Mo level is not to drop below a definite value.

The experiment further showed that, through the continuous recirculation of the desludging residuum (as was done from November 1-8, 1936 for the purpose of reaching a 25% concentration of solid material), the product always becomes harder, and the reactivity of the total mixture is thereby considerably reduced. From this it follows that:

(1) Recirculation of desludging residuum without the removal of a definite quantity of solid material (and hence with treatment of the residua) is impossible, since about 5-6% of solid material is introduced into the circuit through the extract itself.

Assuming a charge of 100 metric tons of extract per day, and a 40% production of solid material, 15 tons of desludging residuum would be yielded daily; that is, in order to maintain a Mo level of about 0.20%, which is necessary for good hydrogenation, the addition of contact must be exactly, as in the case of charging pitch, at 0.4% of the fresh feed.

(2) A contact concentration of about 25-30% is therefore impossible for economic reasons according to (1).

(3) Even if the extract does not contain any solid material, a very hard, inert product would be formed by the possible recirculation of the desludging residuum which unfavorably influences the reactivity of the injection product.

2. Operating Conditions and Values

Injection products: 9200 liters per hour: 33.4% extract, 33.1% fuel

-3-

oil, 33.5% desludging residuum containing 0.041% purification mass
11002 and 0.057% soda.

Solids: 91.4%; solidification point, +85° C; MoO₃, 0.77%.

Furnace Temperatures: 25.6, 25.6, 25.5, 25.3 Mw.

Gas inlet: 11,000 cubic meters per hour; specific gravity = 0.280
0.300.

Differential pressure: 14 atm.

Fresh gas to chamber: 5900 cubic meters per hour; cold gas, 6800 cubic
meters per hour.

Recycle expansion gas: 2250 cubic meters per hour.

Fresh gas consumption: 549 cubic meters per metric ton of injection
material and 1685 cubic meters per metric ton of fresh feed.

If it is taken into consideration that the hydrogen of the
recycle expansion gas (64.5%) can be recovered in a Linde plant, then the
hydrogen consumption is decreased to 423 cubic meters per metric ton of
injection product and 1266 cubic meters per metric ton of extract.

Yield of catch-pot product: 6000 liters per hour, specific gravity
1.010 at 20° C.

New catch-pot product: 2350 liters per hour.

Water for regeneration: 400 liters per hour.

Cl content of water from catch-pot: 48 mg. per liter.

Desludging residuum: 3300 liters per hour; solids, 25%; solidification
point 96° C. MoO₃, 0.84%.

Heat conductivity: Regenerator I 82) kg. cal./m²/hr.
Regenerator II 132)

Heat tone:	<u>Kg. Cal./kg. injection prod.</u>	<u>Kg. Cal./Kg. Extract</u>
Furnace I	35	101
Furnace II	21	60
Furnace III	14	40
Furnace IV	19	55
Total	<u>89</u>	<u>256</u>

The heat tone of the pure extract is therefore fairly considerable.
Through the recirculation of a large quantity of fuel oil, however, it is
decreased for the injection product by 2/3 to 89 Cal./kg. A mixture of
pitch and pitch distillate in a ratio of 53 to 47 has, in comparison, a
heat tone of 141 cal./kg.

-4-

3. Product Balance

A complete and very good picture of the products obtained from hydrogenation of extract is shown in Table 1, giving averages of 4 days of operation. All of the yields are calculated on the fresh feed, that is, extract plus hydrogen charged.

Table 1

Product Balance

For the Period Operating with Extract and Fuel Oil (1:1) - Nov. 10-13, 1938.
Operating hours: 96

Injection material: 10.92 tons/hr. (33.4% extract, 33.1% fuel oil, 33.5% desludging residuum)

Fresh feed: 3.56 tons/hour (100% extract).

Consumption per ton fresh feed (extract): 3.07 tons injection product + 0.1795 tons hydrogen.

Product obtained from 1179.5 kg. fresh feed:

	Kg.	% calc. on Extract + H ₂	
<u>Catch-pot product (gas-free):</u>	616.80	52.275	
Benzine Bldg. 20	186.200	15.791	
Heavy benzine	102.400	8.680	
Diesel oil	293.600	24.872	
Fuel oil	34.600	2.932	
Desludging residuum	112.80	9.564	
Reaction water (pure)	33.57	2.845	
Solutes in injection- and reaction water:			
NH ₃	8.59	0.728	
H ₂ S	4.37	0.370	
CO ₂	0.97	0.082	
Phenol	0.79	0.067	
<u>Gases:</u>	418.95	35.515	
Free H ₂ (Expansion gas I + III)	37.450	3.177	
" " (" " II + VI)	0.238	0.020	
CH ₄	100.600	8.530	
C ₂ H ₆	97.750	8.288	
C ₃ H ₈	107.700	9.137	
C ₄ H ₁₀	56.050	4.755	
C ₅ H ₁₂	14.330	1.215	
CO ₂	0.453	0.038	
H ₂ S	1.019	0.086	
NH ₃	3.360	0.285	
	1196.84	101.446	

Characteristic of the balance and of the period operating with extract in general is the comparatively low yield of new catch-pot product of 52.3% and the extremely high amount of gasification of 35.5% as compared with 73% and 12% in the experiments with pitch plus pitch distillate.

However, it must be taken into consideration that:

(1) The specific gravity of the gas inlet was kept very low (0.280-0.300), that is, the quantity of expansion gas from the circuit, which appears in the gasification as "free hydrogen" from expansion gas I + III + recycle expansion gas, cannot be directly utilised as a value of comparison. At a normal specific gravity of 0.320-0.340 the percentage would be 1.5 instead of 3; the total gasification would, therefore drop from 35.5% to 34.0%.

(2) The fresh feed of 33.4% is very low as compared with the injection material. The gasification, as the greatest loss item, would be considerably less if it were possible to increase the fresh feed of extract, since, of course, the gasification losses of the products passing through the circuit are also calculated on the comparatively small quantity of extract (33.4% of the injection product). The same thing is also true of the consumption of H_2 , which is very high at 1685 cubic meters per metric ton of extract.

An increase in the charge of extract in this experiment was, however, already impossible because of limitations in apparatus.

The residue of building 14 has, at a mixture ratio of extract to fuel oil of 1 to 1, a solidification point of $+96^\circ C$; by the addition of desludging residuum the latter drops to about $+85^\circ C$. Owing to the high solidification point the inlet temperature at the paste presses must be maintained at at least $175^\circ C$.

When mixing the recycle gas of about $40^\circ C$ with the hot paste of $175^\circ C$ there occurs a temperature drop of about $75^\circ C$; that is, to a temperature below that of the solidification point of the injection product. This cooling brought about difficult results at noon of November 11, 1938.

The chamber pressure suddenly increased 8 atm. at 2:00 P.M. and the resistance at regenerator II was present on the pressure side, where the injection product solidified as a result of cooling by recycle gas. By increasing the temperature at the paste presses about $10^\circ C$ to $185^\circ C$, and simultaneously decreasing the amount of gas from 13,000 to 11,000 m^3 , it was possible to eliminate the danger. In the design of new chambers it will therefore be necessary to provide for a gas regeneration (desludging residuum regenerators), in order to make it possible, if necessary, to operate at a high concentration of extract; in doing this it is, however, doubtful whether a temperature of more than $100-120^\circ C$ would be tolerable because of technical reasons (paste presses, etc.).

(a) Liquid Products

Up to the present time it has been believed that by the hydrogenation of extract there was principally formed, in addition to bensine, a very good fuel oil. Table 2 shows that with the present method of operation there is obtained bensine and middle oil and only very small quantities of fuel oil.

Table 2:

-6-

Conversion of Extract into Gaseous Benzine, Benzine, Heavy Benzine, Middle Oil, and Fuel Oil

From 1 metric ton of extract there was obtained (according to Table 1):

	<u>Propane</u>	<u>Butane</u>	<u>Pentane</u>	<u>Benzine</u>	<u>Heavy Benzine</u>	<u>Middle Oil</u>	<u>Fuel Oil</u>
% by Wt.	10.77	5.61	1.43	18.62	10.24	29.36	3.46
Gaseous benzine + Benzine:	36.43% by Wt.						

Hydrogenation of extract can produce benzine and middle oil almost exclusively, even in the sump phase, whereas the production of fuel oil is impossible by this method of operation.

As shown in Table 1 the reaction water is about 1% higher than in experiments with pitch, a value which had previously been found for extract at Leuna.

The amount of phenol in the catch-pot product from extract is considerably greater than in the product from pitch plus pitch distillate. There was an average of 3.0% phenol in the total catch-pot product of the operating period. If this value is recalculated on the basis of the new catch-pot product obtained from the extract, then there is present in the catch-pot product from extract 8.1% phenol; the average for catch-pot product from pitch plus pitch distillate is 1.8%.

(b) Gases:

Table 3 (see appendix) shows the exact analyses of the individual expansion gases from hydrogenation of extract, and, for each of the gases, the specific values (cubic meters per metric ton of injection product and cubic meters per metric ton of extract). The numerical values served as the basis of the calculation of both the gasification in the product balance and of the C-gasification in Table 5.

Characteristic of the hydrogenation of extract is particularly the large quantities of methane formed as compared with previous experiments with pitch, from which it is necessary to conclude that extract has a stronger tendency than pitch to gasification into lower hydrocarbons, or, in other words, extract behaves substantially like the coal from which it is produced (about 25% gasification in the hydrogenation of coal). The percentage of gasification in the hydrogenation of extract as compared with that of pitch is shown in Table 4.

The percentage of C₃, C₄, and C₅ is similar to that of the hydrogenation of pitch plus pitch distillate, so that, by the high gasification in the hydrogenation of extract, the yield of benzine and Calergas amounts to 36.43% of the extract, as shown in Table 2.

The C-gasification has also been calculated from the yields of the individual hydrocarbons per hour (Table 3), which, under the assumption that the B-product (fuel oil and desludging residuum) present in the circuit no longer reacts, amounts of 33.33% of the extract; that is,

from 1000 kg. of charged extract-carbon, 333 kg. of carbon were gasified (Table 5). If this is compared with the values for the charging of pitch plus pitch distillate, there is found a C-gasification of 12.83%.

Table 4

Distribution of Gasification in Extract- and Pitch-Hydrogenation

	<u>Hydrogenation of Extract</u>	<u>Hydrogenation of Pitch + Pitch Distillate (53:47)</u>
Free H ₂ (Expansion gas I + III + Recycle gas expansion)	8.648	6.205
Free H ₂ (Exp. gas II + VI)	0.054	0.919
CH ₄	23.240	15.710
C ₂ H ₆	22.570	26.220
C ₃ H ₈	24.880	26.900
C ₄ H ₁₀	12.950	13.100
C ₅ H ₁₂	3.310	2.398
CO ₂	0.327	1.533
H ₂ S	1.243	3.830
NH ₃	2.763	3.296

Table 5

C-Gasification for Charging Extract as Compared with that of Pitch
Plus Pitch Distillate (53:47)

Extract: 88.5% C
Charge: 3.56 tons or 3.150
tons C/hr.

Pitch + Pitch Distillate (53:47): 91.5% C
Charge: 7.45 tons or 6.820 tons C/hr.

Hydrocarbon	Hydrogenation of Extract		Hydrogenation of Pitch + Pitch Distillate	
	% C	Yield of C, kg/hr.	% C	Yield of C, kg/hr.
C ₁	8.52	268.2	2.89	196.8
C ₂	8.69	273.8	4.26	290.2
C ₃	9.68	305.0	3.77	257.3
C ₄	5.10	160.4	1.64	112.0
C ₅	1.34	42.4	0.27	18.6
	33.33	1049.8	12.83	874.9

The kind and amount of gasification agree with the values obtained at Leuna for hydrogenation of extract. (Dr. Winkler and Dr. Frese, Large-Scale Experiment of the Hydrogenation of Coal-Extract at 300 atm. (heavy-oil Experiment), Report III, page 17, and supplement 7.)

4. Charge, Yield, and Conversion of Chamber 17 in Hydrogenation of Extract Plus Fuel Oil in Ratio of 1:1

During the entire period operating with extract and fuel oil in a ratio of 1:1 the figures for the charge, yield, and conversion were considerably lower than those in the experiments with pitch plus pitch distillate. The present values for extract are similar to those for coal, but this is not surprising in view of the fact that in both cases the concentration of B-product in the injection material is very high.

Table 6

Charge, Yield, and Conversion of Chamber 17 in the Hydrogenation of Extract and Fuel Oil in a Ratio of 1:1

	<u>11-10</u>	<u>11-11</u>	<u>11-12</u>	<u>11-13</u>	<u>Comparative Values for Pitch + Pitch Distillate</u>
Charge: Injection product/furnace volume - t/h/m ³	0.73	0.74	0.71	0.71	0.85
Charge: Fresh feed/furnace vol. t/h/m ³	0.24	0.24	0.23	0.23	0.61
Yield: New catch-pot/furnace volume - t/h/m ³	0.16	0.15	0.15	0.15	0.52
Conversion: New catch-pot/fresh feed - t/t/h	0.66	0.625	0.64	0.64	0.85

5. Absorption Coefficients of Catch-pot Product and Desludging Residuum

The absorption coefficients are listed in Table 7, in which connection it should be noted that the catch-pot product consists of extract and new catch-pot product plus recycle fuel oil.

Table 7

Absorption Coefficients of the Catch-Pot Product and the Desludging Residuum for the Period Operating with Extract-Fuel Oil November 10-13, 1938

(m³ Gas/m³ Catch-pot product (Desludging res.) /Atm.)

Yield of Catch-pot product per hour = 5.98 tons = 2.20 tons new catch-pot product + 3.78 tons recycle fuel oil (specific gravity = 1.007/20°C)
 Yield of Desludging Residue per hour = 3.98 tons.

A. Absorption Coefficients of Catch-Pot Product:

	H ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	C ₄ H ₁₀	C ₅ H ₁₂	CO ₂	H ₂ S	NH ₃	CO	N ₂
Exp. gas I - m ³ /h	126.80	77.17	39.52	18.11	4.88	0.47	0.00	0.11	0.72	1.11	8.70
Exp. gas II "	10.18	27.46	72.32	81.90	39.68	8.12	0.66	2.34	12.82	0.32	0.68
Exp. gas VI "	0.09	0.19	1.93	4.11	3.08	0.69	0.016	0.00	0.09	0.00	0.01
Exp. gas I + II + VI - m ³ /h	137.07	104.32	113.77	104.72	47.64	9.28	0.676	2.45	13.63	1.43	9.39
m ³ /m ³ Catch-pot product	23.08	17.64	19.16	17.62	8.02	1.563	0.114	0.412	2.295	0.241	1.582
Partial pressure - gas outlet (580 atm.)	365.80	107.20	41.90	26.58	9.15	1.855	0.02437	0.09055	0.780	1.785	25.33
Absorption Coefficient	0.0631	0.1645	0.4570	0.6630	0.8767	0.8430	4.680	4.550	2.942	0.1351	0.0625

B. Absorption Coefficients of Desludging Residuum:

Exp. gas III - m ³ /h	70.00	33.49	17.42	12.46	4.71	2.02	0.21	0.12	2.29	0.59	6.58
Desludging residuum	21.12	10.10	5.26	3.76	1.42	0.61	0.063	0.036	0.69	0.178	1.985
Partial pressure - gas outlet (580 atm.)	364.40	107.30	42.13	26.92	9.22	1.91	0.0348	0.0928	0.864	1.786	25.03
Absorption Coefficient	0.0580	0.0942	0.1248	0.1396	0.1540	0.3195	1.812	0.388	0.799	0.0996	0.0785

B. Hydrogenation of a Mixture of Extract, Heating Oil, and Pitch Distillate (boiling above 300° C) in Ratio of 2:1:1.

1. Method of Operation

As already explained in the description of Experiment A, it is possible in the hydrogenation of extract to increase the yield and decrease the gasification if it is possible to increase the fresh feed. Since at the present time it is impossible for technical reasons to increase the concentration of extract, the only other possibility for increasing the output consists of replacing the B-product heating-oil in the extract paste with fresh product.

In Experiment B, 50% of the paste plus heating oil was replaced with pitch distillate distilling over above 300° C; the pitch distillate could not contain any constituents boiling below 300° C since it is added to the pure filtrate, the same as the fuel oil from Bldg. 14 (solvent separation). For the purpose of further increasing the fresh feed, another 16% normal pitch distillate (calculated on the injection product) was added in building 29.

The values used for the calculation are the average values for 48 hours of November 18 and 19, 1938.

2. Operating Conditions and Values

Injection material: 9500 liters per hr. 27.2% extract, 13.7% fuel-oil, 13.4% pitch distillate (above 300° C) 15.7% pitch distillate, 30% desludging residuum containing 0.45% Purification Mass 11002 and 0.051% soda.

Solids: 9.5%; solidification point +50° C; MoO_3 0.68%.

Furnace temperatures: 25.6, 25.6, 25.5, 25.3 Mv.

Chamber pressure variation: 14 atm.

Gas Inlet: 12,000 cubic meters per hour.

Fresh gas to chamber: 5600 cubic meters per hour; cold gas 6000 cubic meters per hour; recycle expansion gas, 1750 cubic meters per hour.

Fresh gas consumption: 500 cubic meters per metric ton of injection. 908 cubic meters per ton of fresh feed.

If the hydrogen of the recycle expansion gas is recovered in a Linde plant, the consumption of hydrogen is reduced to 400 cubic meters per ton of injection, and 726 cubic meters per ton of fresh feed.

Amount of catch-pot product: 7150 liters per hour; specific gravity = 1.027 at 20° C.

New catch-pot product: 4850 liters per hour.

Water for regeneration: 500 liters per hour.

Cl content of catch-pot water: 25 milligrams per liter

Desludging residuum: 3100 liters per hr; solids: 24.7%, solidification point +77° C; MoO₃ 0.73%.

Heat conductivity: Regenerator I 94 Cal/m²/hr.
Regenerator II 129 cal/m²/hr.

<u>Heat Tone</u>	<u>Cal./kg. Injection</u>	<u>Cal/kg. fresh feed</u>
Furnace I	47	83
II	9	16
III	6	11
IV	9	16
	71	126

3. Product Balance

The product balance for this operating period (Table 8) presents a much more favorable picture than that of Table 1 for the charge of extract and fuel oil in ratio of 1:1. The formation of catch-pot product is greater, and the gasification has decreased by half to 17%, which shows how important it is to have the concentration of the fresh feed as high as possible.

(a) Liquid Products

The percentage of the individual catch-pot fractions was quite different as compared with Experiment A. While the charge of a pure mixture of extract and fuel oil gave, besides a small amount of fuel oil, mostly benzene and middle oil; the total quantity of components distilling over at 300° C was considerably decreased in favor of fuel oil by charging pitch distillate; about 80% of the pitch distillate was converted into fuel oil.

The reaction water and the phenol content of the new catch-pot product were somewhat less.

(b) Gases

In Table 9 there are listed the analyses of the expansion gases as well as the specific gas values. Since no stock analyses were available for this operating period, it has been possible to calculate in each case only the total hydrocarbons and its C value. On the basis of this C value (about 2) it can be stated that the percentage of the hydrocarbons is approximately the same as in Experiment A and in the experiments with pitch plus pitch distillate; through the fact that the gasification is 50% less than in the hydrogenation of extract and fuel oil in a ratio of 1:1, the percentage of Calorgas and gaseous benzene is naturally reduced by half.

Table 6

Product Balance for the Period Operating with Extract, Fuel Oil, Pitch Distillate, Pitch Distillate. Nov. 18 and 19, 1938

Operating hours: 48

Injection: 11.30 tons/hr. (27.2% Extract, 13.7% fuel oil, 13.4% pitch distillate (above 300° C), 15.7% pitch distillate, 30.0% desludging residuum).

Fresh feed: 6.27 tons/hr. (48.3% extract, 23.8% pitch distillate (above 300°C) 27.9% pitch distillate).

Consumption per ton fresh feed: 1.803 tons injection + 0.099 t H₂.

Product obtained from 1099 kg. newly charged product:

<u>Product:</u>	<u>kg.</u>	<u>% calc. on fresh feed + H₂</u>
Catch-pot product (gas free)	796.00	72.440
Benzine - Bldg. 20	96.30	8.760
Heavy benzine	48.45	4.410
Diesel oil	190.25	17.320
Fuel oil	461.00	41.950
Desludging residuum obtained	64.80	5.895
Reaction water (pure)	26.98	2.455
Solutes in reaction and injection water:		
NH ₃	8.09	0.736
H ₂ S	6.76	0.615
CO ₂	0.62	0.056
Phenols	0.80	0.073
Gases:	188.49	17.155
Free H ₂ (Exp. gas I + III + recycle exp. gas)	17.47	1.592
Free H ₂ (Exp. gas II + VI)	0.09	0.008
Hydrocarbons (C = 2)	169.45	15.420
CO ₂	0.00	0.000
H ₂ S	0.83	0.075
NH ₃	0.65	0.060
	1092.54	99.425

4. Charge, Yield, and Conversion of Chamber 17 in the Hydrogenation of a Mixture of Extract and Fuel Oil Plus Pitch Distillate (above 300° C) in Ratio of 2:1:1.

As already mentioned several times, the amount to which the fresh feed can be increased is decisive in judging the economy of a hydrogenation process. In Experiment A the charge and yield must have been extremely low, since 66% of the injection product was introduced in the circuit in the form of inert product. By the replacement of a part of the paste plus fuel oil with pitch distillate and an additional amount of pitch distillate, which does not require any B-product, it was possible to obtain a considerable increase in the charges and yields as shown in Table 10 (compare Table 6).

Table 10

Charge, Yield, and Conversion in the Hydrogenation of a Mixture of Extract, Heating Oil, and Pitch Distillate in a Ratio of 2:1:1 Plus 16% of Pitch Distillate

	<u>11/18/38</u>	<u>11/19/38</u>
Charge (injection/furnace-volume) ton/hr./m ³	0.76	0.76
Charge (fresh feed/furnace-volume) ton/hr./m ³	0.39	0.39
Yield (catch-pot product/furnace volume) t/hr/m ³	0.34	0.34
Conversion (catch-pot prod./fresh feed) ton/ton/hr.	0.875	0.875

C. Hydrogenation of a Mixture of Extract and Fuel Oil in a Ratio of 1:1 in "Straight passage."

Since the charge, yield, and conversions of Chamber 17 were not satisfactory in the charging of extract and fuel oil in ratio of 1:1 (Table 6), an experiment was made to hydrogenate this mixture in "straight passage." In this experiment the injection should be considerably increased, and fresh oil centrifuged or distilled out of the large amounts of desludging residuum. Since in this method of operation experience shows that the quantity of catch-pot product is less, hydrogenation in a "straight passage" is only able to show an advantage in comparison with the method of operation at Welheim in that the total amount of catch-pot product plus new oil from the desludging residuum is greater through the increased charge than the yield of catch-pot product at a lower charge and less desludging residuum containing 25-30% solids.

With the increase of injection material and the yield of desludging residuum, there is a decrease in the concentration of solid material which automatically brings about such a great decrease in the quantity of catch-pot product, that the total amount of catch-pot product + oil from the increased desludging residuum becomes constantly less; the concentration of solid material in the furnace constantly decreases so rapidly that a maintenance of, not to mention an increase in, the injection material is impossible.

In Experiment C which was terminated after 12 hours, the quantity of new catch-pot product decreased by about 40% (from 5300 to 3000 liters per hour) at a decrease in the concentration of solid material from 25% to 15%, through the removal of larger quantities of desludging residuum. This quantity could not be compensated for by distillation of marketable oil from the double quantity of desludging residuum; that is, the output of this chamber

-14-

which is already low, was still further reduced by this method of operation. At a further drop in the concentration of solid material of the desludging residuum to 4%, the newly formed catch-pot product dropped to practically zero.

This experimental finding confirms the necessity already mentioned above of the highest possible content of solid material.

Table 3

Composition and Amount of Hydrocarbon Gas, Chamber 17 Using Extract and Fuel Oil (1:1) from November 10 to 13, 1938.

Hours of Operation: 96

Injected Material: 10.92 tons/hour = 33.4% Extract, 33.1% Fuel oil, 33.5% desludging recycle with 0.041% R.M. (Purification mass ?) No. 11002 + 0.056% Soda.

Fresh Feed: 3.56 tons/hour = 100% Extract with 0.123% R.M. 11002 + 0.173% Soda.

Yield: Expanded gas 1 (60 atm. - 60 atm.): 26,600 m³ = 277.2 m³/hr. Recycle gas expansion: 211,800 m³ = 2206 m³/hr.

Expanded gas 2 (60 atm. - 0 atm.): 23,400 m³ = 243.7 m³/hr. Expanded gas 6 (Dist. waste gas): 1,000 m³ = 10.42 m³/hr.

Expanded gas 3 (Desludging waste gas): 14,200 m³ = 147.8 m³/hr.

Average Analyses

(Ca and higher: saturated and traces of unsaturated hydrocarbons)

	<u>E. G. 1</u>	<u>E. G. 2</u>	<u>E. G. 3</u>	<u>Recycle Gas</u>	<u>E. G. 6</u>
H ₂	45.78	4.18	47.35	64.46	0.86
CH ₄	27.81	11.27	22.67	18.46	1.79
C ₂ H ₆	14.25	29.68	11.78	6.75	18.52
C ₃ H ₈	6.64	33.61	8.43	4.07	42.37
C ₄ H ₁₀	1.76	16.28	3.19	1.32	29.55
C ₅ H ₁₂	0.17	3.33	1.37	0.27	6.65
CO ₂	0.00	0.27	0.14	0.00	0.15
H ₂ S	0.04	0.96	0.08	0.00	0.00
N ₂ + CO + O ₂	3.55	0.42	4.99	4.67	0.11
	<u>100.00</u>	<u>100.00</u>	<u>100.00</u>	<u>100.00</u>	<u>100.00</u>

Table 3 (continued)

	Expanded gas 1		Expanded gas 2		Expanded gas 3	
	m ³ /hour	m ³ /ton Inject	m ³ /hour	m ³ /ton Inject	m ³ /hour	m ³ /ton Inject
H ₂	126.80	11.610	10.18	0.932	70.00	6.414
CH ₄	77.17	7.060	27.46	2.515	33.49	3.067
C ₂ H ₆	39.52	3.620	72.32	6.620	17.42	1.596
C ₃ H ₈	18.41	1.660	81.90	7.500	12.46	1.142
C ₄ H ₁₀	4.88	0.447	39.68	3.634	4.71	0.432
C ₅ H ₁₂	0.47	0.043	6.12	0.744	2.02	0.185
CO ₂	0.00	0.000	0.66	0.060	0.21	0.019
H ₂ S	0.11	0.011	2.34	0.214	0.12	0.011
N ₂ +CO+O ₂	9.84	0.901	1.02	0.093	7.38	0.676
	<u>277.20</u>	<u>25.352</u>	<u>243.68</u>	<u>22.312</u>	<u>147.81</u>	<u>13.542</u>
		m ³ /ton Fresh Feed		m ³ /ton Fresh Feed		m ³ /ton Fresh Feed
		35.600		2.859		19.670
		21.670		7.714		9.410
		11.100		20.310		4.894
		5.094		23.020		3.199
		1.371		11.140		1.323
		0.132		2.282		0.568
		0.000		0.185		0.059
		0.031		0.658		0.034
		2.766		0.287		2.073
		<u>77.764</u>		<u>68.455</u>		<u>41.530</u>

	Recycle gas expansion		Expanded gas 6	
	m ³ /hour	m ³ /ton Inject	m ³ /hour	m ³ /ton Inject
H ₂	1422.00	130.250	0.0897	0.00822
CH ₄	407.00	37.270	0.1864	0.01707
C ₂ H ₆	148.80	13.630	1.9290	0.17670
C ₃ H ₈	89.72	8.215	4.4140	0.40440
C ₄ H ₁₀	29.10	2.665	3.0800	0.28230
C ₅ H ₁₂	5.96	0.546	0.6930	0.06347
CO ₂	0.00	0.000	0.0156	0.00143
H ₂ S	0.00	0.000	0.0000	0.00000
N ₂ +CO+O ₂	103.00	9.438	0.0115	0.00105
	<u>2205.58</u>	<u>202.014</u>	<u>10.4192</u>	<u>0.95464</u>
		m ³ /ton Fresh Feed		m ³ /ton Fresh Feed
		399.400		0.0252
		114.350		0.0524
		41.820		0.5420
		25.020		1.2410
		8.174		0.8650
		1.674		0.1947
		0.000		0.0014
		0.000		0.0000
		28.940		0.0032
		<u>619.378</u>		<u>2.9279</u>

Table 9

Composition and Amount of Hydrocarbon Gas Chamber 17 Using Extract/Fuel Oil/Pitch Distillate - Pitch Distillate
December 18 and 19, 1938

Hours of Operation: 48

Injected material: 11.30 t/hr. = 27.2% Extract/13.7% Fuel Oil/13.4% Pitch Distillate (above 300°C) - 15.7% Pitch Distillate - 30.0% Desludging Recycle with 0.45% R.M. 11002 (Purification mass ?) + 0.051% Soda.

Fresh Feed: 6.27 t/hr. = 48.3% Extract/33.8% Pitch Distillate (above 300°C) - 27.9% Pitch Distillate with 0.80% R.M. 11002 + 0.091% Soda.

Field: Expanded gas 1 (600 atm. - 60 atm.) = 13,100 m³ = 272.8 m³/h
 " 2 (60 atm. - 0 atm.) = 10,300 m³ = 214.7 m³/h.

Exp. gas 3 (Desludging waste gas): 6,300 m³ = 131.3 m³/hr.
 Recycle gas expansion: 84,200 m³ = 1754 m³/hr.

Average Analyses:

	E. G. 1		E. G. 2		E. G. 3		Recycle gas			
	m ³ /hr.	m ³ /ton Fr. feed	m ³ /hr.	m ³ /ton Fr. feed	m ³ /hr.	m ³ /ton Fr. feed	m ³ /hr.	m ³ /ton Fr. Feed		
H ₂	124.35	11.02	19.84	1.09	57.85	5.12	9.22	1145.50	101.35	182.70
Hydrocarbons	136.40	12.07	21.76	31.68	64.65	5.73	10.31	504.00	44.60	80.40
CO ₂	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
H ₂ S	0.49	0.05	0.08	0.31	0.70	0.06	0.11	0.53	0.05	0.08
NH ₃	0.46	0.04	0.07	0.51	1.38	0.14	0.22	0.70	0.06	0.11
N ₂ +CO+O ₂	11.10	0.98	1.77	0.58	6.70	0.59	1.07	103.40	9.15	16.50
G =	272.80	24.16	43.52	34.17	131.28	11.64	20.93	1754.13	155.21	279.79

	Expansion Gas 1		Expansion gas 2		Expansion gas 3	
	m ³ /hr.	m ³ /ton Fr. feed	m ³ /hr.	m ³ /ton Fr. feed	m ³ /hr.	m ³ /ton Fr. Feed
H ₂	45.61	3.20	44.08	1.97	65.30	1.64
Hydrocarbons	49.97	92.56	49.24	1.97	28.73	1.64
CO ₂	0.00	0.00	0.00	0.00	0.00	0.00
H ₂ S	0.18	0.92	0.53	0.03	0.03	0.03
NH ₃	0.17	1.48	1.05	0.04	0.04	0.04
N ₂ +CO+O ₂	4.07	1.84	5.10	5.90	5.90	5.90
G =	100.00	100.00	100.00	100.00	100.00	100.00