U. S. BUREAU OF MINES

HYDROGENATION DEMONSTRATION PLANT DIVISION

The following drawings were omitted from T-137 - Abstract of Report on High Pressure Vessels for Hydrogenation.

PLEASE ATTACH DRAWINGS TO YOUR COPIES.

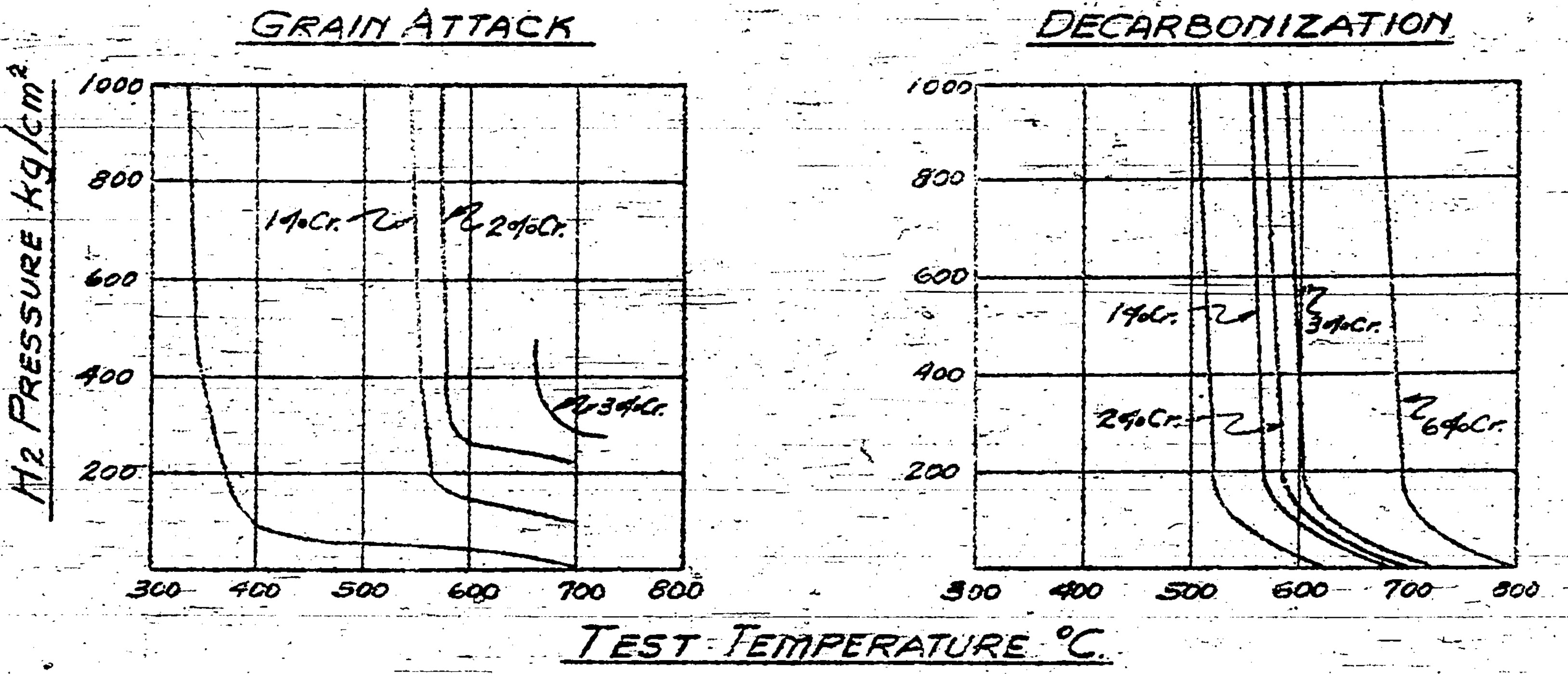


FIG. 2 RESISTANCE LIMITS AGAINST H_2 ATTACK OF Cr-Mo STEELS

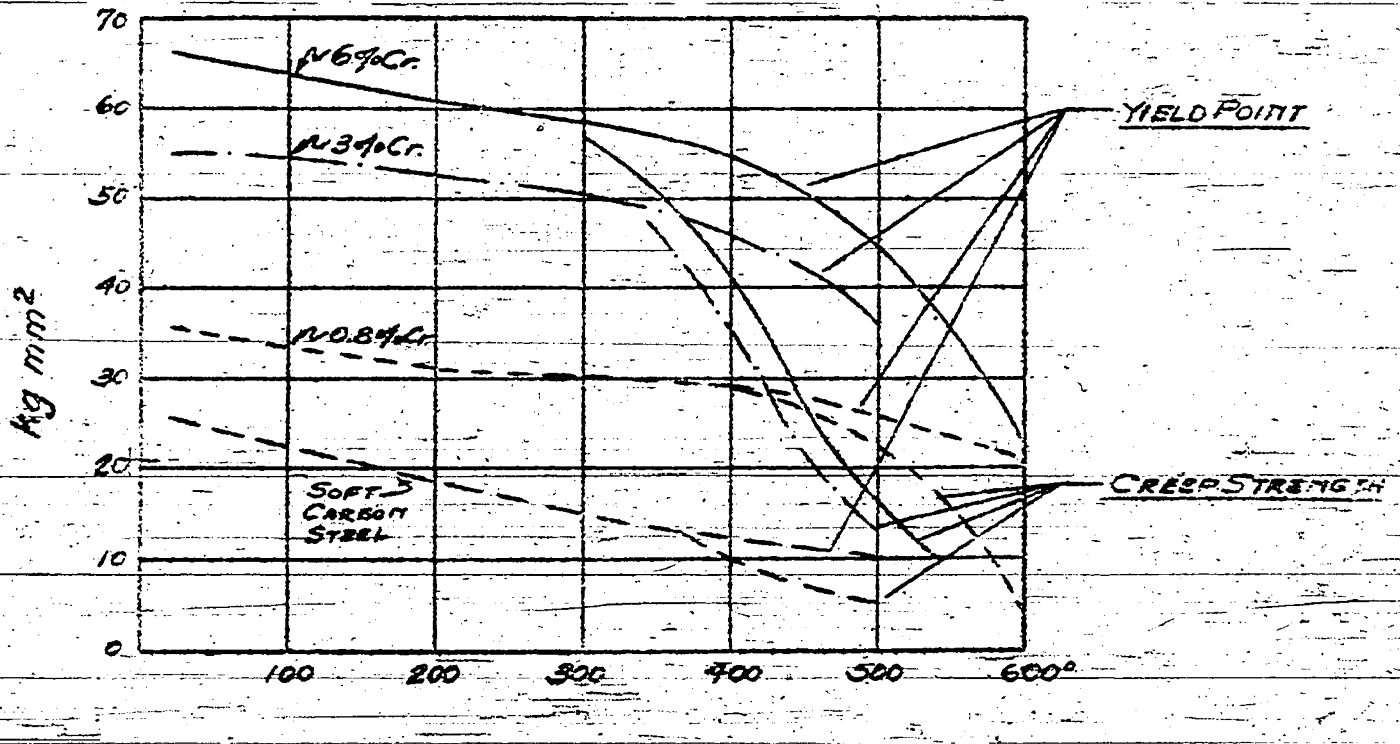
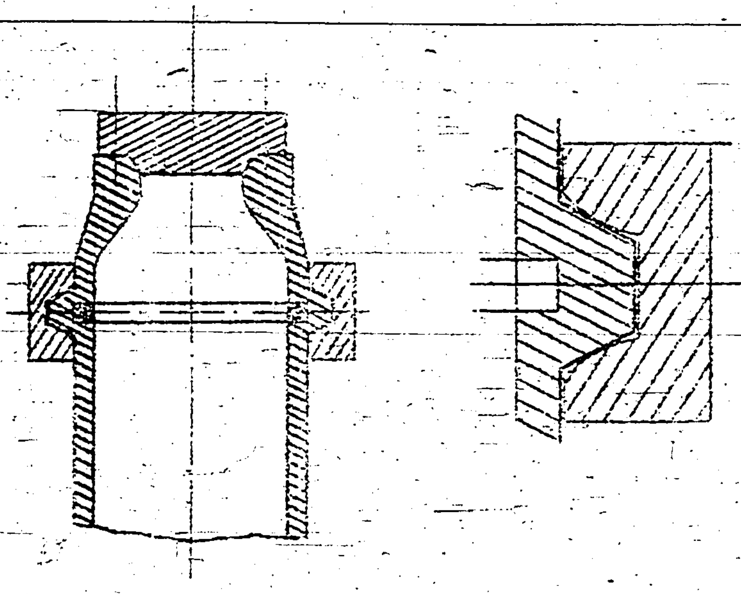


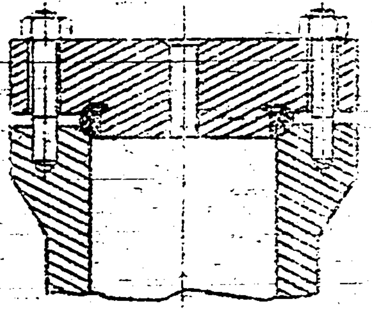
FIG. 4 YIELD POINTS AND CREEP STRENGTHS OF ALLOYED STEELS

T-137



Connection of High Pressure Jacket Parts with Clamp Ring & Elastic Metal Seal

Fig. 12



Cover Closures with Stud Bolts and Elastic Conical Sealing Ring

Fig. 18

Newman

T. O. M. Reel 181
Pgs. 447-456

U. S. Bureau of Mines

Hydro. Demon. Plant Div.

KCBraun
11/1/46
T-138

Comparative Efficiencies of Synthetic Liquid Fuels Production

INDEX

TABLE I
High Pressure Hydrogenation and Synthesis

	(A) PROCESS	(B) PRODUCTS	(C) Coal Consumption St= Bitumin. Coal, Hu= 7000 Kcal/Kg. Br= Brown Coal Hu=2300 Kcal/Kg	(D) Coal Req'd	(E) Efficiency of Fuel Production, based on Coal Consumption	(F) Efficiency of Power Generated at the Motor Shaft based on Coal Consumption	(G) Coal Consumption	(H) Coal Consumption
			T/T Product	T/T Product	%	%	Kg/HPh	Kcal/HPh
1)	Bitumin. Coal Hydrogenation	High Performance Gasoline Aviation Gasoline Auto Gasoline Marine Fuel Oil Liquid Gas	4.5-6 St	4.5-6 St	36-40	9-10	0.9-1.05 St	6300/7400
2)	Brown Coal Hydrogenation	Aviation Gasoline Auto Gasoline Diesel Oil Lubricating Oil Liquid Gas	12-14 Br	12-14 Br	36	9	2.9-3.1 Br	6700/7200
3)	Brown Coal Synthesis	Auto Gasoline Diesel Oil Paraffin Liquid Gas	12.5 Br	12.5 Br	36.2	9.9	2.78 Br	6400
4)	Bitumin. Coal Synthesis	Auto Gasoline Diesel Oil Paraffin Liquid Gas (Benzol)	4.0 St	5.6 St	37.4	10.1	0.91 St	6350

(Note:-HP=Metric Horsepower)

TABLE 2

Combined Hydrogenation and Synthesis Process

(See Table I for Column Headings)

	(A)	(B)	(C)	(D)	(E)	(F)	(G)	(H)
1)	Bitum. coal kiln operation with kiln tar hydrogenation.	High Performance Gasoline Aviation Gasoline Auto Gasoline Marine Fuel Oil Liquid Gas	3.0 St	10.5 St	50	12.5	0.73 St	5100
2)	Brown coal kiln operation with kiln tar hydrogenation.	Auto Gasoline Diesel Oil Liquid Gas	9 Br	16 Br	50	12.5	2.24 Br	5200
3)	Bitum. coal kiln operation with kiln tar hydrogenation & kiln coke synthesis	High Performance Gasoline) Aviation Gasoline) from Auto Gasoline) hydro. Marine Fuel Oil) stage (1) Auto Gasoline) from Diesel Oil) synthesis Paraffin) stage. Liquid Gas	3.8 St	3.8 St	40.1	10.4	0.87 St	6090

(Continued on next page)

TABLE 2 - Continued

(See Table 1 for column headings.)

	(A)	(B)	(C)	(D)	(E)	(F)	(G)	(H)
4)	Brown coal kiln operation with kiln tar hydrogenation & kiln coke synthesis.	Auto Gasoline) from hy- Diesel Oil) dro.stage (2) Auto Gasoline) from the Diesel Oil) synthesis Paraffin) stage Liquid Gas	10.5 Br	10.5 Br	43.6	11.1	2.48 Br	5720
5)	Bitum. coal coking with tar hydrogenation and high temp. coke synthesis.	High Performance Gasol) from Aviation Gasoline) hydro. Auto Gasoline) stage Marine Fuel Oil) (3) Auto Gasoline) from Diesel Oil) synthesis Paraffin) stage Benzol Liquid Gas 250 atm. bottle gas	2.5 St	2.5 St	44.6	11.0	0.78	5460

(Note:-Kiln operation (Schwelung)=low temp. carbonization.)

(Proportion of Products from the hydrogenation stage to those from the synthesis stage (1) 1 : 1, (2) 2 : 1, (3) 1 : 4.

TABLE 3

Process for Production of 250 Atm. Bottle Gas.

(See Table I for Column Headings)

(A)	(B)	(C)	(E)	(F)	(G)	(H)
1) Brown coal high pressure gasification	55 kg Gasoline 955 m^3 L.P.G. 250 atm.	4350 kg Br	46.2	11.5	2.4 Br	5520
2) Bitum. Coal coking	45 kg Benzol 1480 m^3 L.P.G. 250 atm.	1430 kg St	67.1	16.8	0.54 St	3780

L. P. G. (Treibgas) = Liquified Petroleum Gas

TABLE 5

Comparison with other Vehicles

(See Table I for Column Headings)

(A)	(B)	(C)	(E)	(F)	(G)	(H)
Steam Locomotive			17			

TABLE 4

Fuel Process for Use in Vehicle Generator
(See Table I for Column Headings)

	(A)	(B)	(C)	(E)	(F)	(G)	(H)
1)	Bitum. coal kiln operation with kiln tar hy'genation and kiln coke processing in the vehicle generator.	114 kg Gasoline 23 kg L.P.G. 4120 m^3 Generator-gas	1430 kg St.	55.8	12.6	0.72 St	5040
2)	Brown coal kiln operation with kiln tar hy'genation and kiln coke processing in the vehicle generator.	244 kg Gasoline 24 kg L.P.G. 2520 m^3 Generator-gas	4350 kg Br	55.3	12.4	2.22 Br	5110
3)	Vehicle generator with brown coal kiln coke	6360 m^3 Generator-gas	4350 kg Br.	63.6	13.9	1.98 Br	4560
	with bitum. coal kiln coke	6400 m^3 Generator-gas	1430 kg St	64.0	14.1	0.65 St	4550
	with Anthracite	7500 m^3 Generator-gas	1350 kg Anthracite	75.0	16.5	0.52 Anthracite	3850
	With wood	7500 m^3 Generator-gas	2590 kg Wood	25.0	16.5	1 wood	3350

REMARKS

TABLE I

Item-1.)	Used for production of high performance and aviation gasolines and marine fuel oil.
Item-2.)	Used for production of aviation and auto gasolines and diesel oil.
Item-3.) & Item-4.)	Used primarily for production of paraffin and diesel oil.

TABLE 2

Item-1.)	Under construction in Upper Silesia at present. Could not be used before, because kiln operation had not been sufficiently developed and the sale as well as the utilization of kiln coke had not been fully clarified.
Item-2.)	This process combination is used very extensively. The quality of the gasoline comes within the lower limit of aviation fuel. The sales problem of the surplus crude has been eliminated since this is used in trucks.
Item-3.)	A small plant is operating in the Ruhr, though with this difference, that the kiln tar is not further processed in a hydrogenation stage, but is used directly as marine fuel oil. The preceding applies to the products from the synthesis stage.
Item-4.)	This combination can not be used for the production of aviation gasoline. The kiln operation with the following tar hydrogenation is used very extensively. In Ruhland, instead of using crude as raw material, the synthesis stage processes brown coal briquettes.
Item-5.)	This combination is used in several Ruhr plants; practical for small plants, too. In further development of this process combination different aspects must be tried out; good coking coal is scarce. Coincidental production of the various products must agree with the demand for them.

REMARKS--Continued

TABLE 3

Item-1.) & Item-2.) In these processes, gases with a heat value of 4250 WE/m^3 are bottled @ 250 atm. The utilization of these processes is limited by the use for the product. Its range of action is small and the heavy bottles reduce the useful load.

TABLE 4

Item-1.) Disadvantages are the high HP weight of the motor and generator and the high heat value of the fuel carried. Can not be used for military purposes.

Item-3.) Anthracite must first be de-ashed. Wood can not be used in Germany because of the great demand for it for other uses.

T-139

TOM Reel 57
Pgs. 211-233

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

KCBraun
10/31/46

The Calorific Efficiency of Bituminous Coal Hydrogenation.

By Dr. Wilde and Diple Ing. Schappert.

I. G. Farbenindustrie, A. G.
Ludwigshafen/Rhein, 20 Feb. 1942.

I. Calorific Efficiency of Bituminous Coal Hydrogenation.

The calorific efficiency of coal hydrogenation is the relation between the calories contained in the desired final product, that is, gasoline and liquified petroleum gas (L.P.G.), and the calories contained in the coal required to produce the desired final product.

The calorific efficency of bituminous coal is, therefore:

$$E = \frac{\text{Gasoline} + \text{L.P.G.} \quad (\text{in Kcal})}{\text{Coal for (Hydrog.} + H_2 + \text{Power)} - \text{Hygas Surplus} \quad (\text{in Kcal})}$$

The 3 figures in the denominator represent the 3 most important operations:

		Final Product
Hydrog. Coal --	Hydrogenation --	Gasoline + L.P.G.
H_2 Coal --	H_2 Production --	Pure Hydrogen
Power Coal --	Power Plant --	KW + Steam

It is, therefore, proper to determine the calorific efficiency for these 3 operations separately, to enable us to get a better picture of the influence of the H_2 production on the efficiency of the total hydrogenation.

Efficiency of the Power Plant

		Back-Pressure Operation	Condensation Operation
Pressure	atm.	100/5	100/0.03
Adiabatic Gradient	Kcal/Kg	190	320
Turbine Shaft Efficiency	%	75	75
Specific Steam Consumption	Kg/KWh	5.70	3.40
Boiler House Efficiency	%	85	85
Power House Efficiency	%	60	25

		Auto Gasoline		Aviation Gasoline	
Gasoline Production (tons)	t	1		1	
H_2 Consumption	m^3	2800		3200	
Power Requirements:					
Steam for Hydrogenation	t	4.035	7.285	4.490	8.190
Steam for H_2 Production	t	3.250		3.700	
Elec. Energy for Hy'genation	KW	1000	2680	1360	3280
Elec. Energy for H_2 Prod.	KW	1680		1920	
Electrical Energy Production:					
Back Pressure Operation	KW	1275		1430	
Condensation Operation	KW	1405		1850	
Power Plant Efficiency	%	42		40	

The power plant efficiency varies only slightly with a change in the proportion of steam: El. Energy.

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		Auto Gasoline		Aviation Gasoline	
Gasoline Production (tons)	t	1		1	
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Power Requirements:					
Steam for Hydrogenation	t	4.035	7.285	4.490	8.190
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II. Calorific Efficiency of H_2 Production.

1.) Fig. 1. shows the heat-flow diagram for H_2 production, based on a final product of 1000 m^3 pure H_2. Table 1. contains coke plant data, Table 2. contains data on the water gas plant, as well as gas cleaning and compression, based on the report of Dr. Witte of 27 November 1941 on the investigation of the H_2 requirements at Gelsenberg, while the other energy figures originated at Scholven and Nordstern.

The following efficiencies, based on different assumptions, may now be derived:

$$E_1 = \frac{\text{Pure } H_2}{\text{Hy'genation Coal} + \text{Energy Coal} - (\text{Restwatergas} + \text{Benzol} + \text{tar})} = 26.3\%$$

$$E_2 = \frac{\text{Pure } H_2 + \text{Benzol} + \text{Gasoline (from tar} = 50\%)}{\text{Hydgenation Coal} + \text{Energy Coal} - \text{Restwatergas}} = 30.4\%$$

In further calculations, however, only $E_1 = 26.3\%$ was used, because it is not always possible for the hydrogenation plant to dispose of the coke by-products, tar and benzol, at will.

2.) By the introduction of gas decomposition by Linde the calorific efficiency was considerably increased. Following is a heat balance of the Scholven Linde plant:

Incoming		Outgoing	
Gas	149.7×10^6 Kcal/h	H_2	70.0×10^6 Kcal/h
Energy Coal	40.1×10^6 Kcal/h	Rest-gas	70.7×10^6 Kcal/h
Total	189.8×10^6 Kcal/h	Steam	9.6×10^6 Kcal/h
		Energy Loss	39.5×10^6 Kcal/h
		Total	189.8×10^6 Kcal/h

$$E(H_2) = \frac{H_2}{\text{Total Input} - (\text{Restgas} + \text{Steam})} = \frac{70}{189.8 - 80.3} = 64\%$$

Even though it is seldom possible to produce 100 % of the H_2 requirements by Linde decomposition, such as from coke oven gas, the possibility always exists of covering 1/3 of the total H_2 requirements in this manner, from:

a.) The hy-poor gases, including let-down & kiln-gas.

b.) The restwatergas, which is properly converted.

c.) The coke oven gas, which is equivalent to the amount of coke used for making water gas.

Considering that the hy-poor gas is largely produced at the Linde plant pressure of 25 atm, the efficiency of a combined Linde and water gas plant is

$$E(\text{Linde} + \text{W.G.}) = 39\%$$

A further increase in efficiency could be gained if the rest gas produced in the Linde plant were further processed in a hy-gas splitting plant. This possibility was, however, not considered in the following.

III. Calorific Efficiency of Hydrogenation Proper.

1.) The heat-flow diagram of the hydrogenation proper at $E(H_2\text{-Production}) = 100\%$ is shown for the production of aviation gasoline in Fig. 2. for the data below, in which the corresponding values for auto gasoline are also given:

	Aviation Gasoline	Auto Gasoline
H_2 requirement, m^3/t gasol.	3200	2800
Input Coal, t/t	2.15	1.95
Hy-gas production, 10^6 Kcal/t gasol.	8.3	6.2
L.P.G. therefrom, 10^5 Kcal/t gasol.	2.64	1.98

The corresponding energy values may be seen in Table 3.

The following efficiencies apply to the production of gasoline:

	Aviation Gasoline	Auto Gasoline
E(hydrogenation proper)	49.0	51.1

However, with the actual efficiency of H_2 production from water gas of $E(H_2) = 26.3\%$, the efficiency of total hydrogenation drops to:

	Aviation Gasoline	Auto Gasoline
E(hydrogenation)	27.2%	28.9%

The heat-flow diagram of the total hydrogenation for aviation gasoline shown in Fig. 3 is based on these relations of actual H_2 production, in which the quantity of coal required for the production of 1 ton aviation gasoline was subdivided as follows:

a.) Input coal for hydrogenation = 15.5 Kcal x 10^6

b.) Input coal for H_2 production = 24.5 Kcal x 10^6

c.) Energy coal for power plant = 7.78 Kcal x 10^6

Total 47.78 Kcal x 10^6

The hy-gas surplus is added to the energy coal. The rest-watergas also goes to the power plant, see Fig. 1. Fig. 3 does not properly show this, because it was offset by a corresponding reduction in energy coal.

The heat value given above of 47.78 x 10^6 Kcal is the total calorific input for the production of 1 ton aviation gasoline and 0.24 ton L.P.G.

This quantity of heat is then subdivided percentagewise into:

Products, Energies and Losses.

Since the hy-gas surplus represents a closed circuit in a certain sense, and is, therefor, not included in the input of 47.78 x 10^6 Kcal, its percentage share is shown in apprentices in Fig. 3.

The "hydrogenation loss" in the amount of 2.05 x 10^6 Kcal = 4.4% represents the remainder and contains the losses which have not been subdivided, such as:

d). Product losses in distillation, in tank storage, etc.

e). Circulating gas losses.

f). H_2 consumption for the formation of H_2O, NH_3 and H_2S.

Table 4 shows the influence of $E(H_2)$ on the total efficiency of hydrogenation, which is shown graphically in Fig. 4 for both aviation and auto gasoline. The operating point used for making Fig. 3 is shown encircled on the corresponding curve in Fig. 4.

As these curves indicate, the efficiency of hydrogenation drops very little with rising H_2 requirements. A greater H_2 requirement increases the hy-gas production correspondingly and increases the cost of power. The relation is about the following:

100 m^3 increased H_2 consumption changes the E(hydrogenation) about 0.45% @ $E(H_2)$ = 26.3% and about 0.2% @ $E(H_2)$ = 50%.

IV Possibilities of Increasing the Calorific Efficiency of Total Hydrogenation.

1.) Introduction of a Linde process.

As shown in II, $E(H_2)$ may be increased from 26.3% to 39%, where for H_2 production:

a.) The hy-poor gas, including kiln and let-down gas,

b.) The rest water gas,

c.) The coke gas corresponding to the coke used in the water gas plant,

is decomposed. (c.) is possible in some plants.

The E(hydrogenation) is thereby increased to 31.9% for aviation gasoline and 33.7% for auto gasoline.

2.) Utilization of Kiln Residues.

Kiln residue with Hu= 5900 Kcal/Kg.
Hu= 5780 Kcal/Kg.

can profitably be burned in the boiler house, which is to be done in the future both in Gladbeck and Nordstern.

With a decomposition of 94% and a kiln loss of 6.5%, based on the oil yield, the heat contained in the kiln residue = 1.3 x 10^6 Kcal/ton gasoline.

The E(hydrogenation) is thereby increased to 33.1% for aviation gasoline and 35.1% for auto gasoline.

3.) Introduction of Paste Heat Exchange.

Heating the paste requires,

Without heat exchange　1.8 x 10^6 Kcal/ton gasoline.

With heat exchange　0.8 x 10^6 Kcal/ton gasoline.

Paste heat exchange therefore, produces a saving in heat of 1.0 x 10^6 Kcal/ton gasoline, wherein the lower power requirement of the hot gas blowers is included.

Paste heat exchange has been successful in Stettin and Ludwigshafen.

The E(hydrogenation) is thereby increased to 34.1% for aviation gasoline and 36.2% for auto gasoline.

4.) Recovering the Waste Heat from the Hot Let-Down and Products.

In the let-down saturated steam cooler and in the hottest part of the product cooler approximately 0.5 x 10^5 Kcal/ton gasoline may be recovered.

Upper Silesia and Gladbeck are being supplied with these coolers. The steam may be used for heating the copper liquor in the gas cleaning process to about 50° C.

The E(hydrogenation) is thereby increased to 34.6% for aviation gasoline and 36.8% for auto gasoline.

Fig. 5 shows the relation between E(hydrogenation) and H_2 requirement for aviation and auto gasoline production, again taking into consideration the above 4 possibilities for increasing the E(hydrogenation).

Summary

	Aviation Gasoline	Auto Gasoline
The calorific efficiency of the power plant = %	40	42
The calorific efficiency of the H_2 production from water gas = %	26.3	
The calorific efficiency of hydrogenation proper = %	49	51
With an H_2 requirement of m^3/ton gasoline	3200	2300
The calorific efficiency of total hydrogenation is, therefore, = %	27.2	28.9

It has been shown that it is possible to increase the E(hydrogenation), however, to about 34.6% for aviation gasoline and 36.8% for auto gasoline by various measures, such as,

1.) Introduction of a Linde Process,

2.) Utilization of Kiln Residues,

3.) Introduction of Paste Heat Exchange, and

4.) Recovering the Waste Heat from the Hot Let-Down and Product,

which measures have largely been introduced in Upper Silesia.

There are undoubtedly other means by which to increase the calorific efficiency, but since this is not the only factor in the total economy of a hydrogenation plant, capital expenditures, steel requirements, etc. are often of greater importance, the above values of 34.6% and 36.8% probably represent the calorific efficiencies for aviation and auto gasoline, respectively, possible today.

TABLE 1

Yield in Gas, Coke and Tar from Ruhr Coal

The figures were taken from the Ruhr Coal Handbook, year 1932, and are still valid today.

Coking temperature	900° C
Input product, washed fine coal,	8-12% water, Hu = 6900-7200 WE/kg.
Gas yield/100 Kg. fine coal,	27-29 m^3/100 Kg, 0°C, 760 mm Hg, Hu = 5100-5600 WE/m^3
Coke yield/100 Kg. fine coal,	63-73 Kg/100 Kg.
Tar yield	3-5 Kg/100 Kg.

The benzol produced in washing = (28-X)4500 + 35000 X=28 x 5350
X=0.786
=0.8 Nm^3 benzol
=2.7 Kg. benzol

The residual gas = 28-0.8 = 27.2 Nm^3, of which 13.6 m^3 is used for heating the coke ovens and 13.6 m^3 is used for other purposes.

The Heat balance of a coke plant then looks like this:

Input

Coal	100 kg	7050 WE/kg	705000 WE	100%
TOTAL Input			705000 WE	100%

Output

Coke	68 Kg.	7300 WE/Kg	496000 WE	70.4%
Gas	27.7 Nm^3	4500 WE/m^3	122000 WE	17.3%
Tar	4 Kg.	10000 WE/Kg.	40000 WE	5.7%
Benzol	2.7 Kg.	10200 WE/Kg.	28000 WE	4.0%
Loss			19000 WE	2.6%
TOTAL Output			705000 WE	100.0%

Coke Plant = 100- (8.65 + 2.6) = 88.75%

Table 2

Data on Coke and Gas Plants

	Coal Requirement & Production				Power Requirement				
					Electric Energy		Steam		
	Jan. - Sept. 41	pr 1000 m^3 H_2	Energy Content Kcal	%	KW	Kcal	Tons	Kcal	%
Coke Plant									
Coal			8450						
Raw Coke	415×10^6 kg (1)	940 Kg	6550 (1)	80.1					
Benzol			375	3.6					
Tar			530	5.05					
Gas Plant					36.6 (2)	78	--	--	0.74
θ-Gas	528.2×10^6 m^3 (1)	1195 m^3	2790 (1)	26.3					
Rest-Gas	148.3×10^6 m^3 (1)	335 m^3	670 (1)	6.3					
Conversion									
CO_2 & CO-Wash					560 (2)	1210	1.163 (2)	750	18.6
H_2 (100 %)	441.8×10^6 m^3 (1)	1000 m^3	2370	22.4					

(1) Report of Dr. Witte of 27. Nov. '41

(2) Nordstern Figures

Energy Data for Hydrogenation

Table 3

I. Aviation Gasoline

		Mill	High Pressure	Distillation	Residue Preparation
Power Requirement					
Electr Energy	KWh	129	1023	151	58
	Kcal	277	2200	324	125
Steam 2.5 atm	kg	66.5	800	2200	711
	Kcal	43	520		461
Steam 18 atm	kg	66.5	222	315	107
	Kcal	47	156		75
Fuel gas (Hu=3000 WE)	m^3	71	600	233	67
	Kcal	213	2040		201
	Kcal		9031		
Coal Consumption	tons		2.15		
	Kcal		15500		
H_2 Consumption	m^3		3200		
	Kcal		7590		
Gasoline Production	t		1		
	Kcal		10300		
L.P.G. Production	t		0.24		
	Kcal		2640		
Hy-Gas Excess	Kcal		5660		
Efficiency	%		49.0		

II. Auto Gasoline

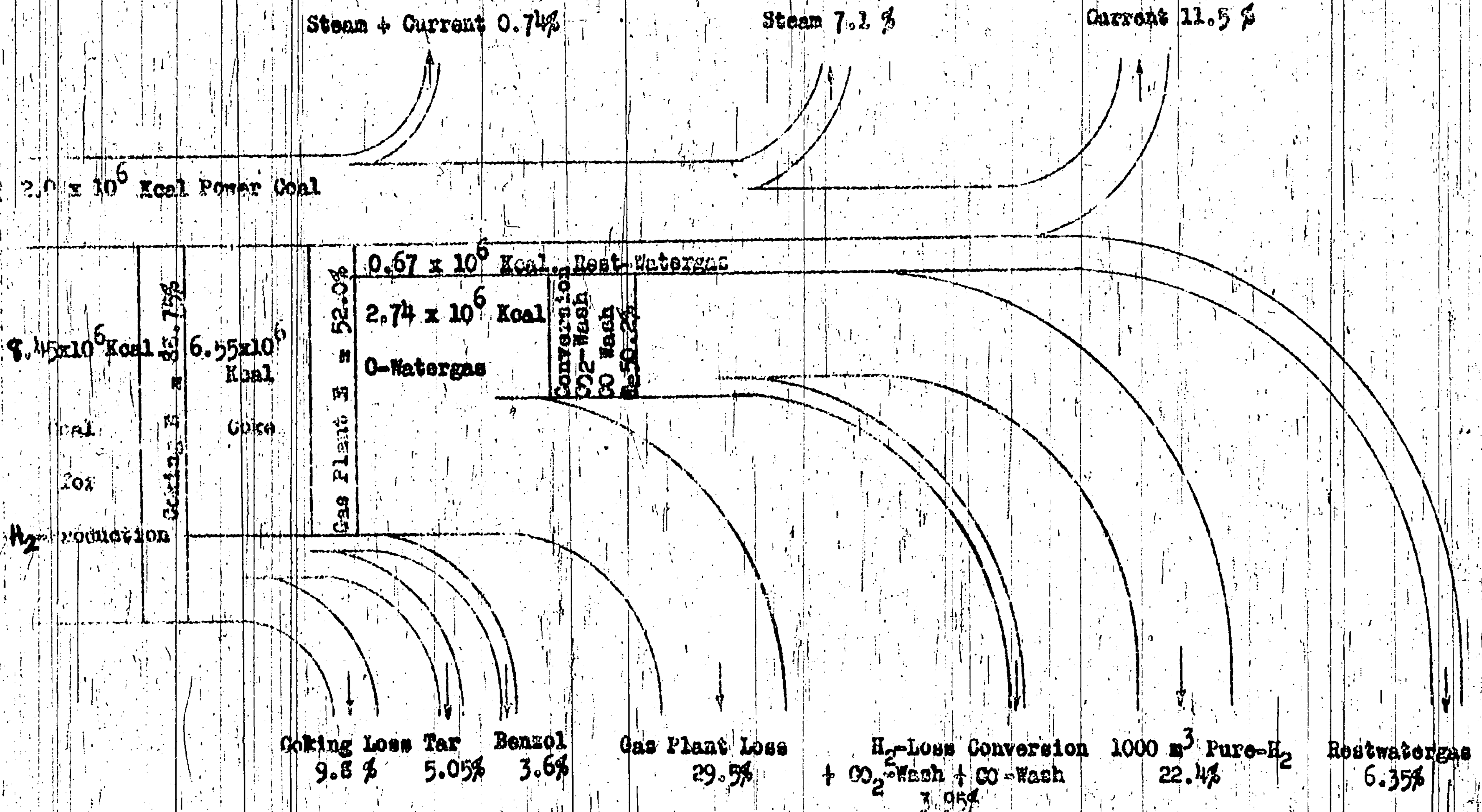
		Mill	High Pressure	Distillation	Residue Preparation
Power Requirement					
Electr. Energy	KWh	116	695	136	52
	Kcal	238	1425	279	107
Steam 2.5 atm.	kg	60	720	1980	640
	Kcal	39	468	1285	415
Steam 18 atm.	kg	60	200	283	96
	Kcal	42	140	198	68
Fuel gas	m^3	64	612	210	60
	Kcal	192	1835	629	136
	Kcal		7541		
Coal Consumption	tons		1.95		
	Kcal		14050		
H_2 Consumption	m^3		2800		
	Kcal		6640		
Gasoline Production	t		1		
	Kcal		10300		
L.P.G. Production	t		0.18		
	Kcal		1980		
Hy-Gas Excess	Kcal		4220		
Efficiency	%		51.1		

$$\text{Efficiency:} = \frac{\text{Gasoline} + \text{L.P.G.}}{\text{Coal} + H_2 + \text{Power} - (\text{Hy-Gas Excess})}$$

TABLE-4

Total Hydrogenation Data

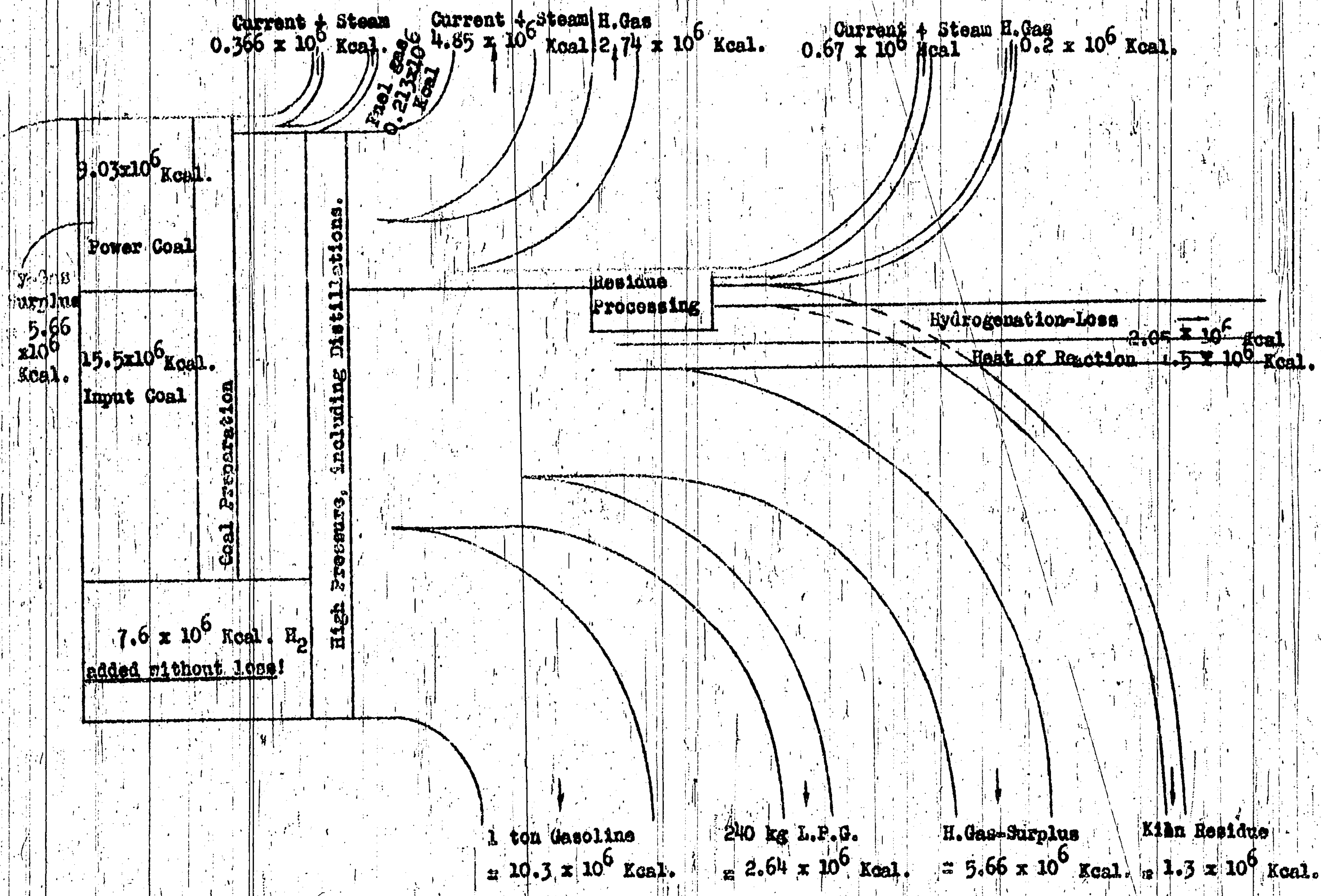
		Aviation Gasoline				**Auto Gasoline**			
Coal Consumption	tons Kcal	2.15 15500				1.95 14050			
H_2 Consumption Eff. H_2 = 100 %	m^3 Kcal	3000 7110	3200 7590	3400 8060	3600 8530	2600 6160	2800 1640	3000 7110	3200 7590
Gasoline Production	tons Kcal	1 10300				1 10300			
Hy-Gas Produced of which L.P.G. Rest Fuel Gas	Kcal Kcal Kcal	7820 (0.24 5180	8300 t) = 5660	8770 2640 6130	9240 6600	5720 3740	6200 (0.18 4220	6670 t) = 1980 4690	7150 5170
Fuel Gas Consumption	Kcal			3153				2837	
Fuel Gas to Power Plant	Kcal	2027	2507	2977	3447	903	1383	1853	2333
Power Requirement									
Steam Electr. Energy Power Requirement	tons KW Kcal Kcal		 1360 2920 5872	4487 (Pow. Pl. Eff = 40%)			4035 1000 2050 4705	 (Pow.Pl.Eff=42%)	
Efficiency of Hydrogenation %									
for H_2-Prod. =	26.3 %	28.0	27.2	26.4	25.8	29.9	28.9	28.0	27.2
" " =	25 %	27.1	26.4	25.6	25.0	28.9	28.0	27.1	26.2
" " =	30 %	30.1	29.4	28.7	28.0	32.1	31.2	30.3	29.1
" " =	40 %	34.9	34.3	33.6	33.1	37.0	36.2	35.4	34.7
" " =	50 %	38.8	38.2	37.7	37.0	40.7	40.1	39.5	38.9
" " =	70 %	43.9	43.6	43.3	43.0	46.1	45.7	45.4	45.1
" " =	100 %			49.0				51.1	



H_2 Yield 26.3%

(Rest-W.G. + Tar + Benzol as Credit)

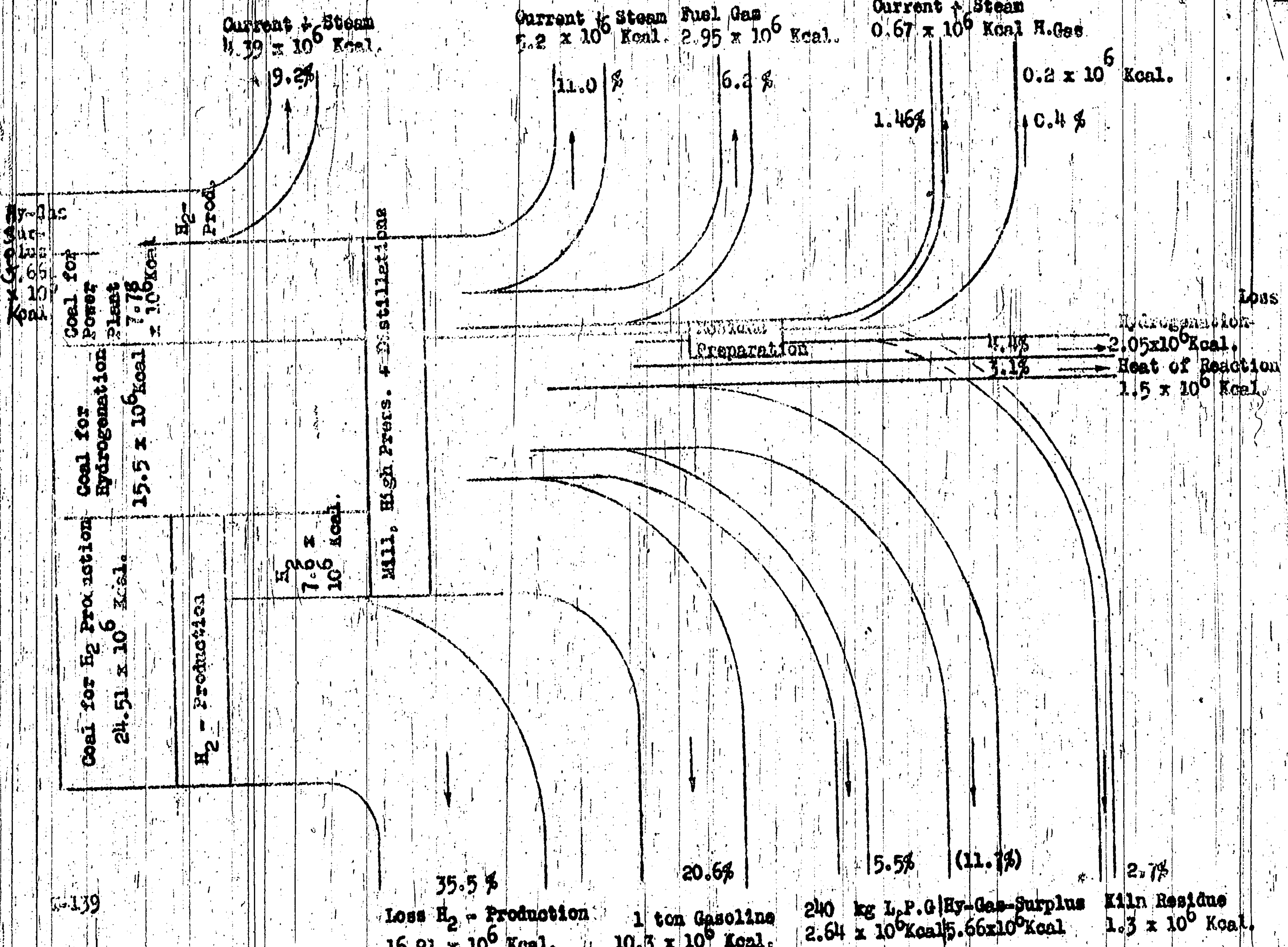
Fig. 1



$$E\text{-}Hyd. = \frac{\text{Gasoline} + \text{L.P.G.}}{\text{Power Coal} + \text{Input Coal} + H_2 - \text{H.Gas-Surplus}} = \frac{10.3 + 2.64}{9.03 + 15.5 + 7.6 - 5.66} = \frac{12.94}{26.47} = 49\%$$

Efficiency of Hydrogenation Process.

Fig. 2



$$\text{Hyd.} = \frac{\text{Gasoline} + \text{L.P.G.}}{\text{Coal for } H_2 + \text{Coal for Hydr.} + \text{Power Coal}} = \frac{(10.3 + 2.64)\ 10^6}{24.51 + 15.5 + 7.78} = 27.0\%$$

Efficiency of Hydrogenation, including H_2 Production

TOM 57 Fig. 3

Efficiency of Hydrogenation (E-Hyd)

depending on H_2 Consumption

at Efficiencies in H_2 Production of 25-100%.

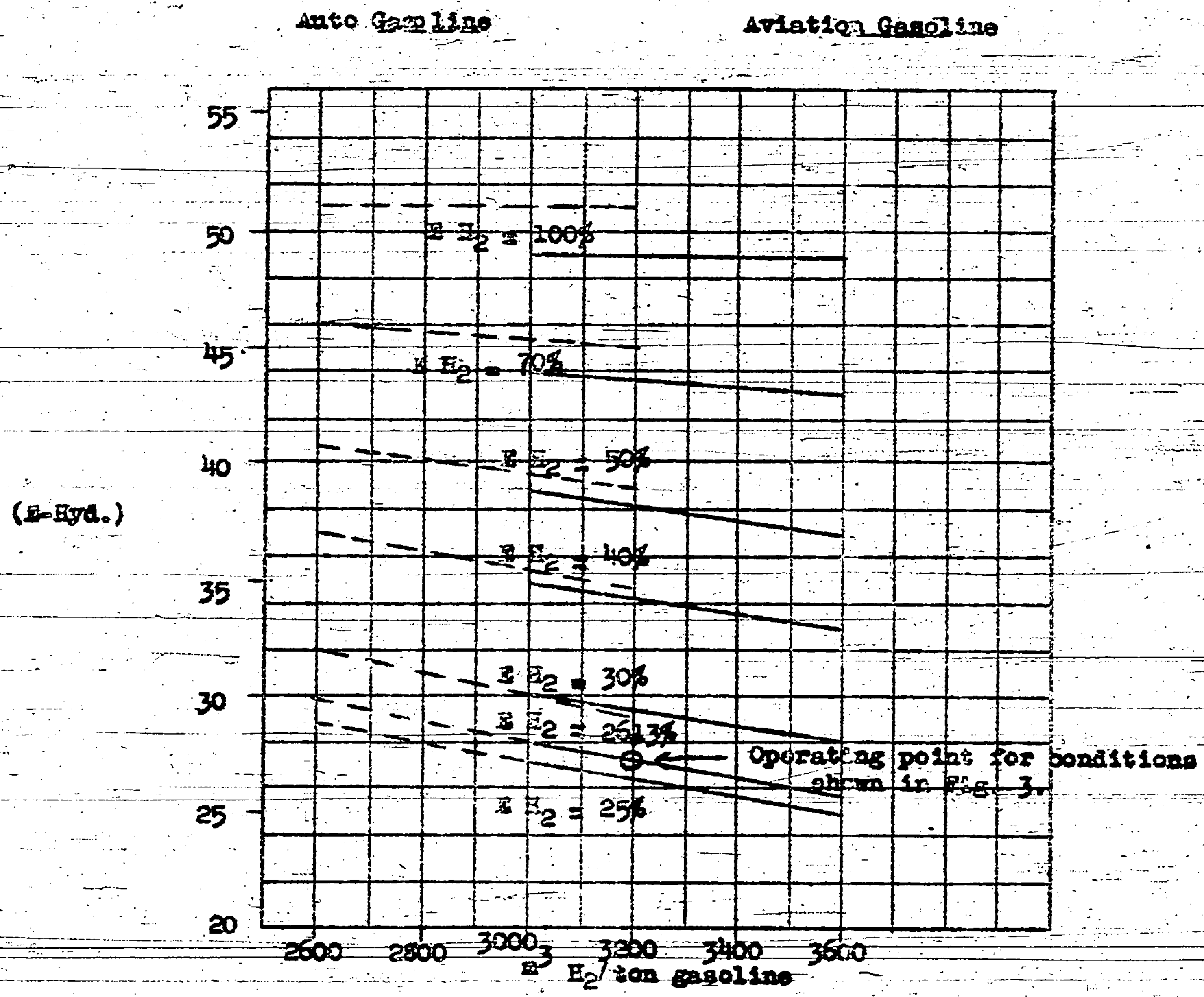


Fig. 4

Efficiency of Hydrogenation,

Depending on H_2 Consumption,
in combination H_2 production: Water gas-Linde (E=39%)
in paste heat exchange
in utilization of kiln residue
in Recovery of Waste Heat in Let-Down Cooler

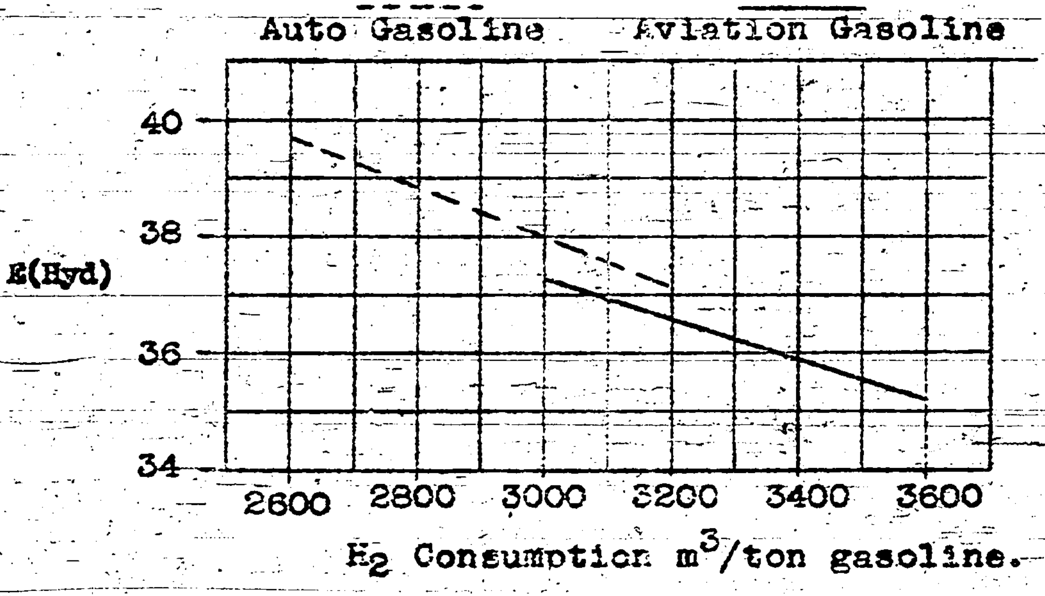


Fig. 5.TOM 57

Newman

T.O.M. Reel 7
Bag 2732
Target 0/4.11
(near end of roll)

U. S. Bureau of Mines
Hydro. Demon Plant Div.

T-140
June 28, 1944

Comparison of Different Catalysts for Hydrogenation of Coal Coke Oven Tar in the Liquid Phase.

An abstract.

Experiments carried out for one year, June 1943 - June 1944 for comparison of I. G. Catalyst 11002 (2% MoO_3 on grude) with the "Ersatz" catalyst 10927 (abt 10% Fe on grude).

Tests have formerly been made in large scale operations only. Results were poorly comparable because of the difference in reaction conditions and in the injection feed.

In the present tests these factors remained constant (but different from large scale operation conditions)

Injection feed	70% coke oven tar 30% fuel oil
Catalysts	1-4% of injection
Thruput	1 Kg/l/hr.
Temper.	24.0 mv.
Pressure	abt 700 atm
Gas:	made-up gas.
Feed: Gas ratio	1: 4500

11002 was found to be much superior to any other catalysts tested. 10927 was next best, 3% addition of it being equivalent to 1% 11002.

Attempts to prepare still more active catalysts had no positive results. Terrana or Kilselguhr were used as the carriers, and either very small amounts of Mo or Cr or high concentration of iron was deposited on them.

W. M. Sternberg

Newman

TOM Reel 70
Frames 000656 - 7.

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-141
Leuna, 12/14/1944

Leuna Methods of Rapid Emptying and Pressure Release of Operating Equipment filled with Combustible Liquids and Gases, as Means of Prevention of Spreading of Fires.

The, at times, considerable amounts of combustible liquids in the equipment during distillation, gas separation scrubbing, etc. require special precautionary measures even under normal conditions. Obviously, such amounts of combustibles carry the danger of spreading fires from any disturbances, such as breaks in the piping of the slides, explosion, small fires, etc. The precautions against this danger taken in the past were limited at first to wide use of fire extinguishers, construction of localizing compartments, spray systems, reduction of the amount of liquid, dispersing spreading out of operations, etc. However, such measures are of little worth in case of air attacks.

Leuna has decided for a long time because of these reasons to provide facilities in such units, which would permit a rapid emptying, with a simultaneous rapid exhausting of all equipment under pressure in case of danger (threatened air attack). Releasing the pressure will prevent the formation of sharp pointed flames with their devastating consequences.

The installation consists essentially in handily located groups of valves, sufficiently long pipe lines and in storage farms conveniently located for receiving the liquids; also a discharge into the air. Provisions are always made for returning the materials under atmospheric pressure, thus preventing losses.

The figures show:

1. Rapid emptying of scrubbers for circulation gas under high pressure.
2. Rapid emptying of gasoline catchpot still.
3. Rapid emptying of tar catchpot still.
4. Rapid emptying and pressure release of gas separation unit.

These installations have repeatedly proven their worth, several have already repeatedly prevented a total destruction of the protected units, and we can no longer think of doing without them in operations.

T-141

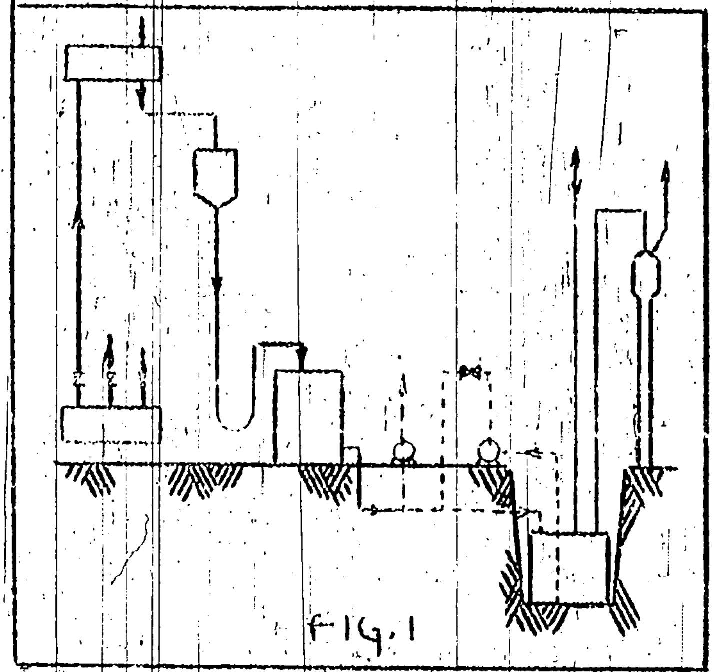


FIG. 1

Emergency let-down of circul. gas scrubbing

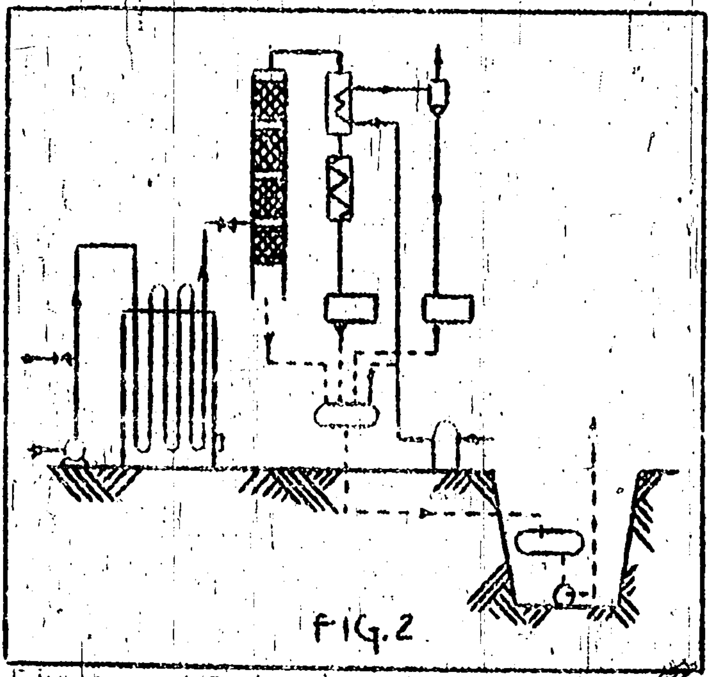


FIG. 2

Emergency let-down Gasoline Catchpot Distill

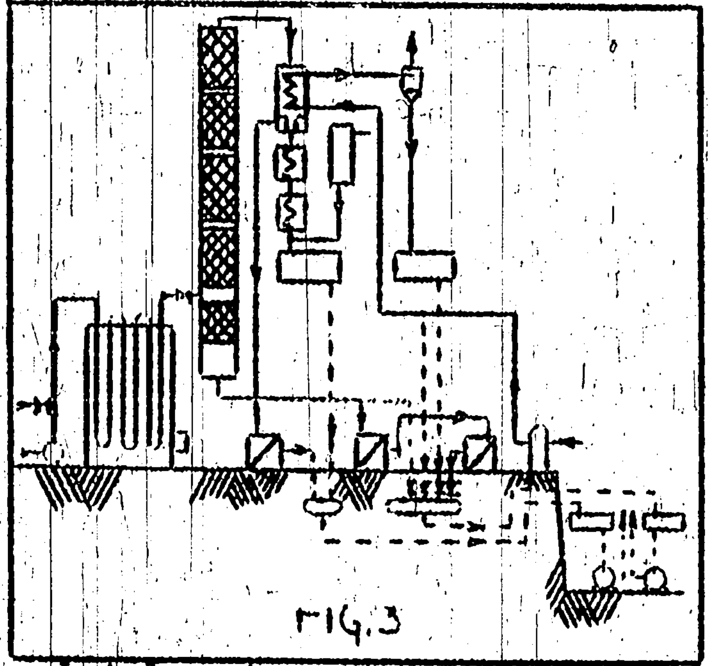


FIG. 3

Emerg. Let-down tar catchpot distillation

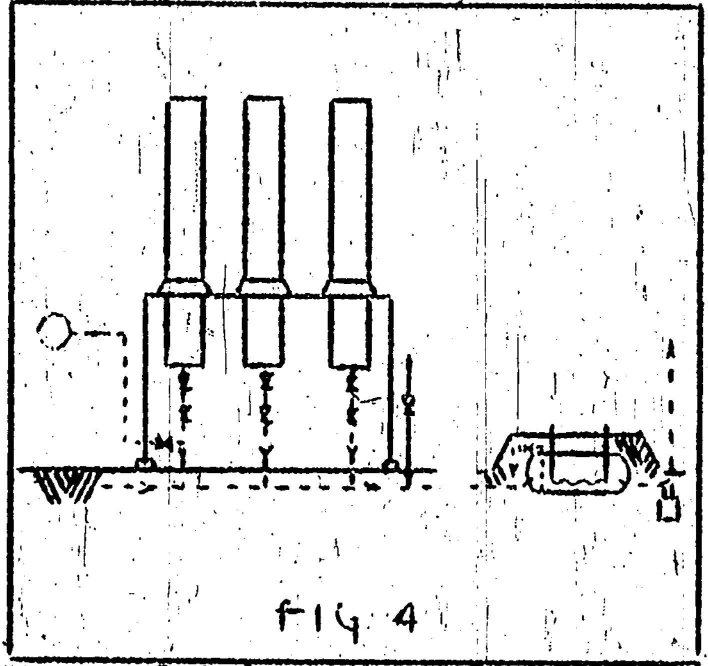


FIG. 4

Emergency let-down of gasoline and gasol separation installation

TOM 181 U. S. Bureau of Mines T-142
Frames 06589 - 6594 Hydro. Demon. Plant Div.

(Duplicated Reel 145
Item 10)

Relationship Between the Composition of Coal and Its Behavior During Hydrogenation

The generally observed regularities in the hydrogenation of coal have been redetermined from the results of experiments for the production of gasoline and middle oil in a 10 liter converter, with coal under a pressure of 600 atm. and an iron catalyst. It appeared particularly desirable to reexamine the relationship between the composition of coal and its hydrogenating ability in order to find out the extent to which analytical results may be used in evaluating coals for hydrogenation.

Hydrogenation results of twelve coals were available, and they were arranged according to the coal basins, and inside of these groups in the order of increasing carbon content of the pure coal. For the purpose of comparison, two brown coals have been added.

Nature of Coal	Coal Basin	Coal Sample		Mine	% C in pure coal
Soft coal	Upper Silesia	1.	1262	Janina	78.26
		2.	1118	Gräfin Johanna	79.61
		3.	1263	Myslowitz mine	
		4.	1197	Heinitz mine	81.27
		5.	1180	Beuthen mine	81.80
		6.	1160	Preussen mine	83.47
		7.	1174	Castellango	83.48
	Ruhr	8.	1216	Zweckel	82.11
		9.	1090	Gelsenberg, young	83.94
		10.	1086	Gelsenberg, old	86.76
	Saar	11.	1060	Vikt. Puttlingen	82.24
		12.	1152	Luisenthal	84.69
Brown coal	Rhineland	13.	229	Union Rhein	68.73
	Mid-German	14.	143	Böhlen	71.79

We may summarize the results by saying, that the predominating influence of the carbon content is again confirmed in the experiments with 600 atm.

We must attribute this to the steady changes in the properties of coal, especially soft coal, with the carbon content. In the coals here investigated, the increase in the carbon content was accompanied by: an increase in the amount of available hydrogen, the degree of coalification, the yield in low temperature tar, and in the Upper Silesian coals the amount of chlorine. The following properties in generally decreased with increasing carbon content: the oxygen content, the volatile matter, iron and alkalinity. There was no connection between the carbon content, sulfur, and, naturally ash on the one side, and the yield of benzol extract (bitumen) on the other.

The following regularities have been found with regard to hydrogenation behavior:

1). All coal required about the same hydrogenation temperature, of about 468 - 474° to reach trouble-free operations, by which is meant the start of the liquefaction operation with a sufficient utilization of asphalt and a sufficient mastery of the converter operations.

2). The utilization of the coal is evidently a function of its fusain content. In general, differences among the different coals are not great in this respect. On the other hand, one may observe a slight deterioration of utilization with increasing carbon content.

3). The yield in gasoline and middle oil is primarily determined by the carbon content, as may be seen in curve 1. The yield of the two Saar coal was below normal, and these coals differ from the rest by a higher ash content (6.81 and 6.7%) of a different composition (much magnesium and little calcium).

4). Contrary to the frequently expressed opinion, the utilization of asphalt is lower with a higher oxygen content of the coal, i.e. it becomes poorer with increasing carbon content (cf fig. 2). The experience with brown coal of an increasing asphalt level with increasing oxygen content, and independent of the carbon content, may therefore not be applied to soft coal.

In this respect also the Saar coals behave less favorably, which must be attributed to their higher ash content.

The young Gelsenberg coal occupies a position all its own because of its especially low asphalt value; it differs even from the behavior of other coal from the same deposit. It may not therefore be taken as a prototype of the Ruhr coal. Its exceptional favorable behavior is probably connected with its peculiarly low alkalinity, its CaO and MgO content is the lowest amongst all the coals, and possibly with its high sulfur content.

The relatively favorable asphalt values of coal in experiments 5 and 6 may possibly be attributed to the relatively high reaction temperatures.

5). Gasification is probably the most complicated (fig. 3). There is no simple relationship between gasification and carbon content. The gasification of brown coal is the same as of soft coal with 8 - 14 % more carbon. Among soft coals, gasification does greatly increase with the carbon content, but there are appreciable difference from one coal basin to another. Gasification of Silesian coal is the same as of a Ruhr coal with 3 - 4 % more carbon, or, to put it differently Silesian coal gasification differs by 6 - 7 % from that of Ruhr coal of the same carbon content. Values for the Saar coals are scattered between the other two.

These differences in the gasification from different coal basins is presumably the result of the action of many factors.

A working hypothesis has been created that the relatively high available hydrogen content and volatile matter of the Ruhr coal is mostly connected with a relatively low oxygen content and the presence of relatively long paraffin chain hydrocarbons (side chains in the coal itself), which produce upon splitting molecules which form oils. Other coals have such short side chains that they form gaseous hydrocarbons chiefly in the thermal splitting in the converter. This hypothesis is also confirmed by a consideration of Saar coals, in the case of which deviations of gasification from the average are parallel to an increase or decrease of available hydrogen.

The low CaO content of the relatively low ash, or the low alkalinity, may however be favorable to gasification.

The effect of ash is possibly insufficiently understood from its determination in the ignition residue and its composition. It seems important to learn the mineralogic composition of inorganic constituents of coal, since the action, say, of calcium would surely be different when present as a silicate, dolomite, aragonite or vaterite, as happens when calcium is first set free from its organic combination during hydrogenation in the converter.

/s/ Hupfer.

W.M.Sternberg.

Figure 1.

Gasol + M.O. Yield

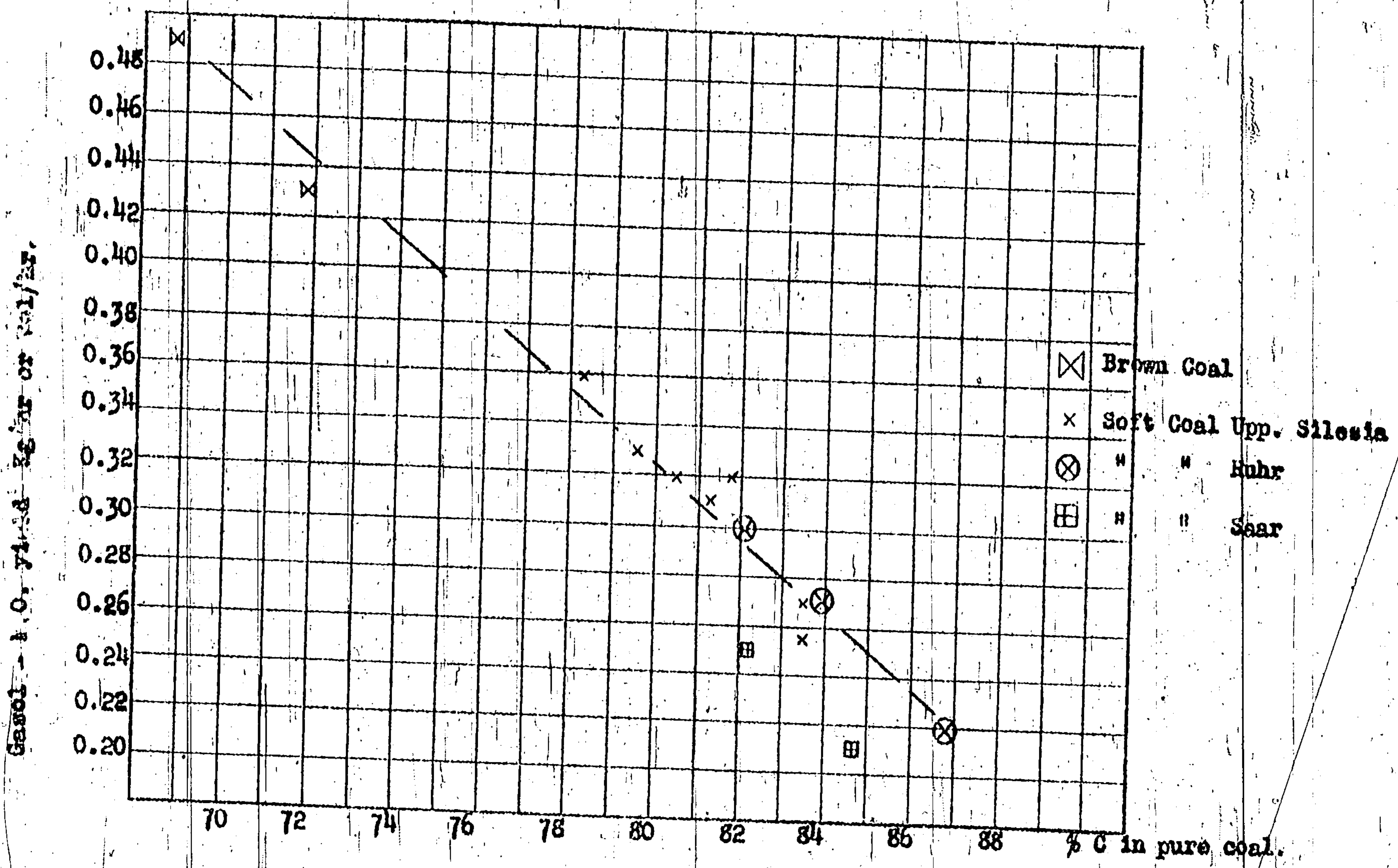
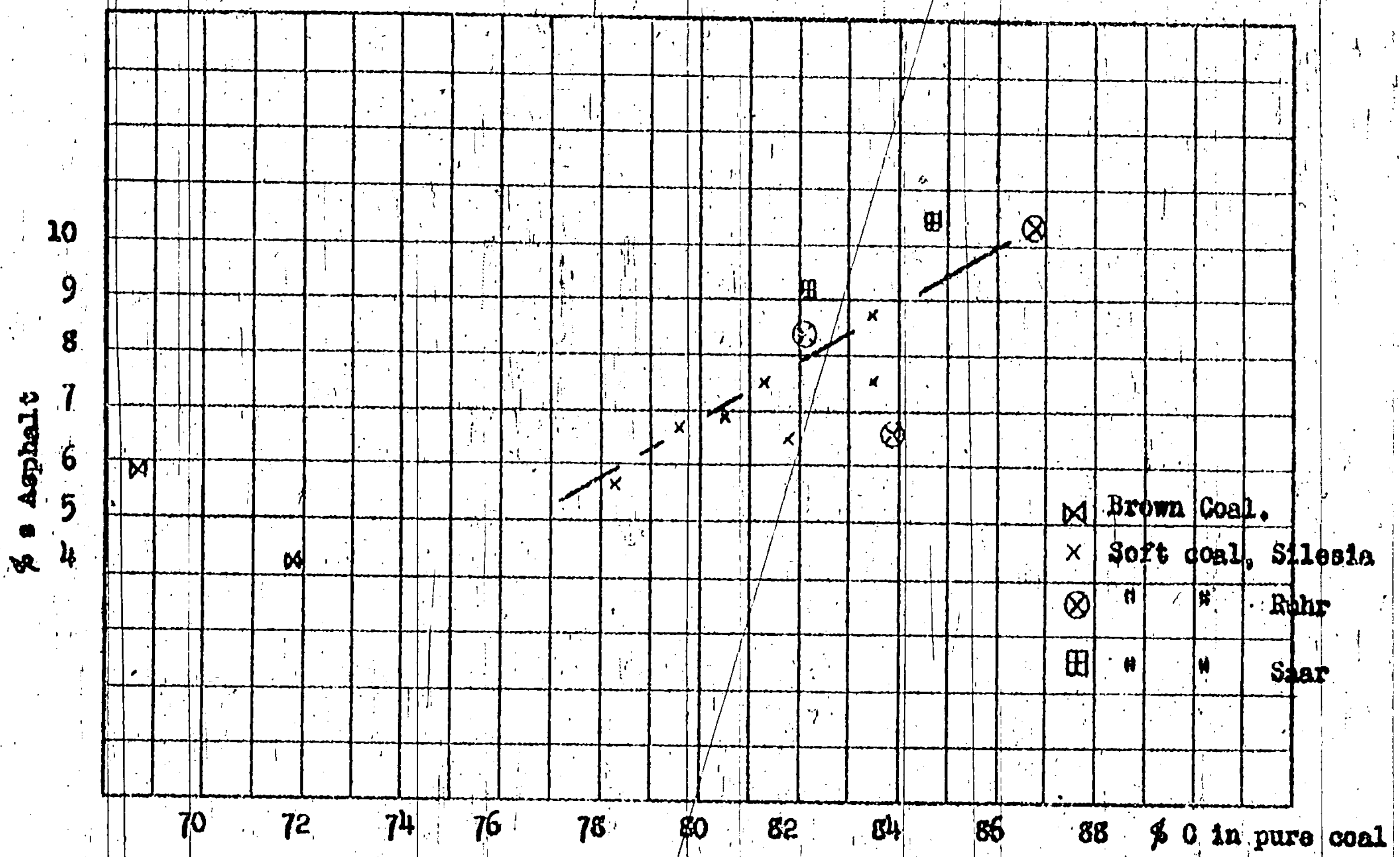


Figure 2.

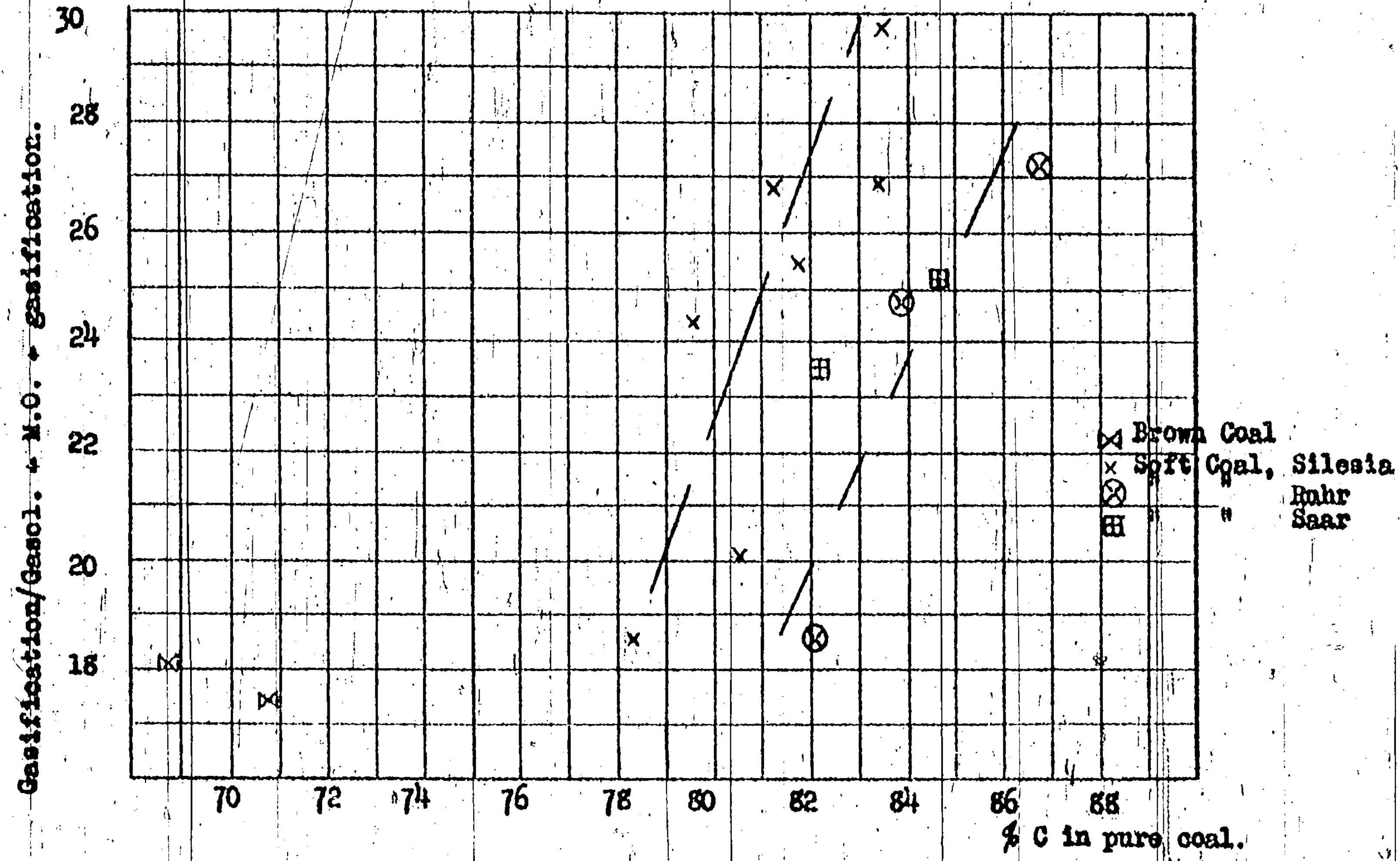
% a Asphalt in relation to % C in pure coal.



TOM Reel No. 184

Figure 3

Gasification



Newman

TOM Reel 162
Frames 877-878

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-143

Industrial Production of Catalyst 5058.

Leuna, May 3, 1943.

1. Production from Tungstic Acid.

Tungstic acid, $WO_3.H_2O$ produced in Bitterfeld from the ore concentrates (65 - 70% concentrate of the wolframite), is used as the raw material. It contains about 92.8% WO_3. The ore is dissolved in NaOH, the sodium tungstate formed decomposed with acid, the tungstic acid washed, dissolved in ammonia, the solution again decomposed with acid, and the pure hydrate filtered, washed and dried in a drying oven. The filtrate still containing some tungstic acid is freed from the remaining WO_3 with lime, and the $CaWO_4$ decomposed with hydrochloric acid.

The Bitterfeld tungstic acid is very pure and contains, in addition to water, only 0.2% impurities (about 0.03% Fe, traces of molybdenum, small amounts of Ca and Na (about 0.01%).

WS_2 is formed as a solid phase from an intermediate compound, the ammonium sulfotungstate, upon heating in the presence of hydrogen to temperatures reaching 400°, according to the following equations:

1). $WO_3 + 2\ NH_4OH \longrightarrow (NH_4)_2WO_4 + H_2O$

2). $(NH_4)_2WO_4 + 4\ H_2S \longrightarrow (NH_4)_2WS_4 + 4\ H_2O$

3). $(NH_4)_2WS_4 + \text{(purely thermally)} \rightarrow WS_2 + S + (NH_4)_2S$ (a)

$+ H_2 \text{ at } 400^\circ \longrightarrow WS_2 + H_2S + (NH_4)_2S$ (b)

The solid $(NH_4)_2WS_4$ is prepared by solution of tungstic acid at 65-70° either in 12% NH_3 or in the mother liquor obtained after filtering off the thiotungstate and containing 12-13% NH_3 and about 10% H_2S. The insolubles are settled on standing, filtered off (55 - 60°), the filtrate saturated with H_2S in a saturator provided with a stirrer, with the temperature rising to 70° (excess pressure of H_2S is 250 mm); the temperature is next slowly lowered 20-25° while maintaining the pressure. Monoclinic crystals of ammonium sulfotungstate separate out as beautiful orange red powder.

Saturation with H_2S must be done at elevated temperatures (60 - 70°) because of the high concentration of tungstic acid (about 20%); in the cold there is a danger of coprecipitation of tungsten oxysulfate with the sulfotungstate. The oxysulfate is rather little soluble in the cold, and should it form, it will not be converted rapidly with H_2S into the desired sulfotungstate.

That salt is formed according to the following equation:

$$(NH_4)_2WO_4 + 2\ H_2S \rightarrow (NH_4)_2WO_2S_2 + 2\ H_2S$$

The oxysulfate cyrstallizes as lemon-yellow needles and produces a much less active catalysts upon thermal decomposition, than 5058.

Higher temperature during saturation has the additional advantage of accelerating the formation of the sulfotungstate which would precipitate as a crystalline deposit in the cold only after several days standing even at sufficiently high concentrations of H_2S.

The crystallized sulfosalt is separated from the mother liquor in a covered suction filter. The filtrate is used for the solution of fresh amounts of WO_3. The precipitate is dried in hydrogen in a steam heated vessel provided with a stirrer.

<u>Decomposition to WS_2.</u>

<u>The dry solid ammonium sulfotungstate</u> is decomposed in a continuous furnace with a screw drive, in a stream of hydrogen, at high temperatures (max. temperature 21 - 22 mv.) and forms in this way WS_2 which retains to outward appearance its monoclinic structure, but which otherwise would crystallize in the hexagonal system. This apparent pseudomorphy results in a porous structure of the crystal lattice which produces an active catalyst.

The WS_2 powder cooled in the rear end of the furnace in a stream of hydrogen is finely powdered to a definite mesh and compressed in Kilian presses under a maximum pressure of 10 - 15 tons into cylindrical tablets, 10 mm in diameter and height. Such tablets can resist a pressure of 300 - 350 kg/cm^2.

The operation life of the catalyst is 1 - 2 years, depending upon operation conditions, and it is then regenerated in the catalyst factory.

The <u>regeneration</u> is very simple. The used catalyst is roasted in rotating furnaces with air heated from the outside the roasted product dissolved in the mother liquor from ammonium sulfotungstate, and the solution treated in exactly the same way as in the production of tungstic acid.

The solution residue contains much iron and consists of FeS and undissolved WO_3 (or WS_2), is reroasted and leached. The second leaching residue contains over 50% FeS is then converted into pure WO_3 by treating it in the same way as ore.

/s/ v. Füner

W. M. Sternberg

Newman

TOM Reel 75
Frames 909-912

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-144

11/14/46

Blechhammer, Febr. 11, 1944

Dr. Kornig's Letter Regarding Incrustation of Preheaters, and the Action of Catalysts in Causing Them.

Dear Dr. Wissel,

I wish to thank you for your letter of the 3rd of Feb. from which I learned with regret, that the formation of crusts has not yet been overcome. As you correctly assume, my opinion in this matter is based on the close connection between the formation of the crust and the coal catalyst. I may therefore be excused if I followed your suggestion and I briefly discussed here the develoment of the modern coal catalysts, as I remember it. I have carried over no data from the days in Ludwigshafen, I must depend entirely on memory. It may therefore readily happen that my recollections here and there were somewhat in error. I would therefore request you to treat the information as given to the best of my knowledge and recollections. Should you find discrepancies with the Ludwigshafen records, I would be grateful for your telling me about them, to permit me to review thoroughly my memory on these points.

Hydrogenation was begun in Scholven in 1936, using tin oxide - Cl as a catalyst, and a pressure of 300 atm. We believed to have sufficiently mastered the very aggressively acting chlorine to expect no large difficulties, in spite of the experience of JCJ which cautioned us to use special precautions. There was chlorine corrosion of the preheater, as you remember, the neutralization in the catchpot and in the neutralization unit did not proceed as smoothly, as we may have wished it. True, these difficulties were later entirely overcome, but we still retained the impressions that it would be advantageous to eliminate entirely the use of chlorine.

Experiments were then started on replacing SnO - Cl-catalyst at 300 atm. pressure with larger amounts of iron catalysts, omitting of chlorine. We have found, however, that the activity of the catalyst was by no means sufficient, especially with respect to the utilization of asphalt.

Use was then made of observations made in the vapor phase, that highly active catalysts at intermediate pressure can be readily replaced with less active catalysts at higher pressures. It was decided to investigate, whether coal hydrogenation could be successfully performed at 700 atm. pressure with the non-aggressive iron catalysts. When operating details and equipment has been developed for operations at 700 atm., experiments with iron catalysts (Bayer mass, lux mass, bog iron ore) were resumed.

These catalysts were found to work satisfactorily at 700 atm. An additional argument in favor of the iron catalysts was their successful use in Leuna in the hydrogenation of brown coal.

De-ashing is carried out very extensively with soft coal, and it was desirable to hold the increase in ash by the catalyst within the narrowest possible limits. It was found therefore at which concentration of the catalyst the advantages of greater catalytic activity ceased to counterbalance the disadvantage of higher ash content. This was found to be with 1.5% (This figure is still used today in our designing; I have read with interest that you have increased it in the mean time. I would welcome your informing me the reasons for this increase).

A neutralization of the coal ash has been found to be of prime importance for the improvement of the action of the catalyst, as has already been found in tests with 300 atm. pressure. These experiments were repeated and confirmed for 700 atm. Working with sulfuric acid was, however, not particularly pleasant and we selected a neutralizing agent which was easier to handle, namely ferrous sulfate. We found that in addition to neutralization, there was also in this case the beneficial action of the ferrous sulfate, and that the ferrous sulfate was in effect preferable to ;sulfuric acid. Nor did we see any objection for using it in large scale operations, because Leuna has occasionally used ferrous sulfate in regular operations with brown coal. It had already been previously found with brown coal, that as long as it was not run with molybdenum, the coal paste had to be only partially neutralized, say 25 - 50%, and the same has been found true here. We have found that 1.1% iron sulfate was sufficient; an increase resulted in no improvement of the catalytic action. The method used with ferrous sulfate from the start was the same as used with sulfuric acid, namely the coal was sprinkled with it. We have found no radical difference in the action of these two neutralizing agents because of the large amount of hydrolysis of ferrous sulfate (the required water being supplied by the water of crystallization of the ferrous sulfate, when added to the perfectly dry coal) which could therefore actually be considered sulfuric acid. However, should this sulfuric acid have failed to interact with the lime of the coal ash, which was by no means assured when the addition was made in the form of a powder, it will react with the pasting oil, and the primary effect would in this case be polymerization. We knew from our tests with sulfurous acid, - and the sulfuric acid anhydride would act still more unfavorably, - that SO_2 polymerizes oils, harmless in themselves, to colloidal asphalt (Klebesphalt) to such an extent, as to cause a rise in temperature during preheating. Pilot plant experience showed the advantage of sprinkling a solution of ferrous sulfate upon moist coal, which would give the greatest assurance of neutralization of the lime in the ash and provisions have been made for it in the plant here, even if it was connected with considerable inconveniences. We kept telling ourselves that as far as possible the correct theoretical way should be selected, in preference to a simpler way, which may have not been found in practice to be undesirable, but disagreed with the theory. You and Gelsenberg were more greatly influenced by simplicity of operation, than we, and had decided to make the addition in the dry way.

In spite of advantages resulting from the use of iron sulfate, there also was one disadvantage: sulfuric acid neutralized part of the alkalinity of the coal ash, and the remainder is no longer sufficient to combine with chlorine in the coal. Slight corrosion is found back of the catchpot, which naturally is particularly disagreeable at 700 atm. We were however determined to avoid in Scholven at all costs the inconveniences connected with the chlorine neutralization back of the converter. We were looking, accordingly, for a substance which would combine with chlorine right after it was added, and would not affect catalysis. We found such an agent in "sulfigran"; an addition of 0.5% completely stopped corrosion at the outlet, and it even promoted an increased catalytic activity.

I hope to have clearly presented the genesis of to-day's ideas on catalysts, in so far as I could do it from memory.

Now to your question about the "guilty party" in corrosion.

It has been stated at the meeting in Frankfurt A.M. on 10/22/1943, that Ludwigshafen went intensively into the analyses of crusts, and had in particular attempted to attribute the guilt for the formation of the crust to the Fe:Ti ratio. However, as far as I can remember, these analyses were insufficient to produce a definite answer. I do not know if additional information had in the meantime been collected; you would however surely be familiar with it, and I would naturally be interested to hear more about it.

I agree with you, that iron sulfate is primarily responsible; I would like to be told whether sulfigran is also responsible, and to what extent. I would imagine, that iron sulfate acted first, as a result of hydrolysis, upon oil, causing polymerization with the formation of colloidal asphalts, to which iron sulfide, formed by the interaction with hydrogen sulfide or hydrogen in the circulation gas would adhere. The crust formed is acted upon by hydrogen under high pressure in presence of iron sulfide, which acts as a catalyst, and the organic constituents of the crust (the colloidal asphalts) are largely hydrogenated away, leaving a crust consisting finally of almost pure iron sulfide with small occlusions of coke-like organic binder.

We may differ in our understanding of the course of the reaction, but the final results obtained with either view points will be the same, and you should therefore expect the crust formation to diminish should you introduce ferrous sulfate and sulfigran directly into the first converter. I agree with your expectation, that you will have to accept a reduction in the utilization of coal. It has however been repeatedly found, that it is the catalytic activity in the preheater, i.e. during the initial depolymerization of the coal molecules, which has the decided effect upon the utilization of coal. This may be the exact reason for the pronounced effect of ferrous sulfate upon the coal utilization. In any case, I would recommend, that iron sulfate (and, as far as possible the sodium sulfide as well) be ground with the catchpot heavy oil, this oil being relatively resistant to the polymerizing action of the iron sulfate. The addition of the asphalt-containing oil should be done as close to the high pressure converter, as possible.

We entertain the hopes that with our method of addition, iron sulfate interacts quantitatively with the ash in the coal, and has therefore lost its terror as a plymerizer before ever coming in contact with the pasting oil. Should this hope be justified, and also the already expressed opinion on the course of the reaction, it may be possible to take advantage of the action of the iron sulfate dispersed the coal upon the utilization of the coal, with no formation of crust in the preheater.

These, however, are speculations. Should we be lucky, we would know by the end of this year, whether our reasoning was right or wrong.

We thank you for your kindly wishes upon our starting hydrogenation. We could well use luck, because the task really is not simple. We were troubled with leaks even at our start of hydrogenation of hot tar, which probably were caused by thermal strains, and which resulted from the bridging of the hairpins. However, we hope soon to overcome these difficulties of start.

/s/ W. Kronig

W. M. Sternberg

Newman

TOM Reel 75,
Frame 904-908.

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-145

COAL CATALYSTS.

(Re: Dr. Krönig's letter on "The Genesis of Coal Catalysts, 2/11/44, T-144).

Dr. Kronig, in his statement that 1.5% is the point at which a further increase in the amount of catalyst is no longer counterbalanced by an increase in coal utilization, overlooks the fact, that in his days (1936-37) the catalyst consisted of FeS or $FeSO_4$, rather than Bayer, Lux or Lauta masses.* The replacement of part of the 1.5% FeS with other iron catalysts has been tested only subsequently (end of 1937), for reason of supply; this was the way the by-product, Lux masse, came up for consideration, and ultimately preferentially into use (in pilot plant). What amount of Lux mass was used to replace $FeSO_4$ we do not at present know; the proportion of 1.2% $FeSO_4 \cdot 7H_2O$ to 1.5% Lux mass is based entirely on an attempt at price equality with 3% $FeSO_4 \cdot 7H_2O$ (abt. RM 90.00 per te of motor gasoline). The possibility of using the other masses, Lauta and Bayer, were only considered after the question of catalysts for Gelsenberg came up. The preference of the Lauta (or Bayer) mass in Gelsenberg came as a result of advantages in availability and freight. The amount used was increased at the advice of Leuna (1.5% to 2%, referred to coal), because a lower efficiency than of the Lux mass was anticipated.

Speaking chronologically, the "discovery" of $FeSO_4$ as a neutralization agent which followed the use of the purification agent was not very accurate; $FeSO_4$ was the original material tested in experiments on replacement of SnO + Cl at 300 as well as at 700 atm. It is true that the discovery of the need of neutralizing only to the extent of 25 - 50% was made at that time; also the possibility of replacing sulfuric acid with ferrous sulfate as a neutralization agent. This has nothing to

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* The Bayer, Lux and Lauta masses are apparently all similar to the "red mud" product in the purification of alumina by the Bayer process. The bayer mass seems to be lower in iron and higher in alumina than the other two, but the analyses of all three vary in different reports. Representative composition of the three may be considered:

Bayer mass:	Fe_2O_3	41%,	Al_2O_3	23%,	Na_2O	7.84%
Lux mass, Reel 34, item 54:	"	60%,	"	6%,	"	2%
Lauta mass, Reel 33, item 19:	"	50%,	"	11-12%	"	3-4%

(all on a dry basis.) (Translator)

do, however, with determining the amount of $FeSO_4$ (1.1%), nor with the amount being fixed with respect to increased catalystic action. The alkalinity of the mass used in conjunction with coal is known to be far in excess of that of coal (over 260 g, against 10 g $FeSO_4$/kg), i.e. the consumption of acid by the Bayer mass naturally affects very considerably the catalytic action of ferrous sulfate. One must logically increase the amount of $FeSO_4$ with any increase in the amount of the Bayer mass to get the same catalytic action if the coal is strongly alkaline (as the Upper Silesian coal actually is). Dr. Krönig's 1.1% point for ferrous sulfate is therefore as fallible as the 1.5% point for the Bayer mass. Neither of these two points are fixed, but simply fictitious assumptions, even if based on experimental data.

The table below gives the dates in the development of the catalysts:

Amounts of Catalyst in the Liquid Phase.
(Amounts in % of pure Coal)

Source and Date of Proposal	Recommended By	$FeSO_4 \cdot 7H_2O$	Lux mass (L) Bayer (B) Lauta (La)	Sulfigran
Calcul., 6/17/37	Donath	(1.5% FeS)	-	-
7/1/37	Donath	3.0	-	-
Report 12/15/37	Krönig	1.2	1.5 L (10% H_2O)	0.8
Discussion, 9/12/38	Krönig Anthes v. Füner	2.4	2.0 L (50% H_2O)	0.8
Conference 9/19/38	v. Füner (Hermann)	2.4	2.2 L (50% H_2O)	0.3
Discuss. 12/17/38	Simon v. Füner	(2.2)	2.2 B (50% H_2O)	(0.3)
Calcult. 11/24/39	Berger	1.1	1.3 L	0.7
Present Consumption,	Pölitz	2.0	3.0 B (10% H_2O)	0.3
Gelsenberg, Jan-April 1942		1.3	2.0 (La) B	0.33
Test, July 1943		(0.0)	(1.8 (La) B)	(0.3)
Aug. 1943		1.2	1.8 (La) B	0.3

The table shows that values arrived by calculation are in every case lower than the amounts for consumption of catalyst assumed in the discussions; the data for $FeSO_4$ differ by 100%. When designing the Pölitz plant the final values selected were the same as in Gelsenberg (1.2 or 1.5% and 0.3%), as discussed on 7/1/37 and 12/15/37;

to what extent these figures are based on larger experiments (cf communications of later dates) is shown by the fact that they were originally set on the basis of price of $FeSO_4$, as may be seen from the table. A further doubt is created by the Bayer mass which is purily oxidic when compared with the hydroxidic Lux and Lauta masses, and is less well suited to hydrogenation, according to Dr. Simon. The Bayer mass has however been used for years by the I.G., also in the heavy oil production in Leuna, while the dried Lauta mass manufactured in Leuna is actually identical with our Bayer mass; it should be advisable therefore to carry out experiments with the hydroxidic masses (Lux, Lauta, also bog iron ore*). It is not impossible, that Upper Silesian coal behaves differently in this respect from Ruhr coal, even aside from the fact, that Leuna as well, according to Ludwigshafen, used at first not the pure Bayer mass, but a mixture of it with 40% bog iron ore.

We may mention briefly the impregnation of coal with the catalyst ($FeSO_4$), according to available data. We are as yet unaware of any experiments which prove definitely and unequivocally the advantages of impregnating the coal; the perfect absence of such proofs may be the reason why Gelsenberg and Pölitz have changed over to the simpler mechanical method of addition.

(The next two paragraphs are omitted, being polemical with Dr. K.'s preference for using a solution of the sulfate on theoretical grounds, and claiming that polymerization is prevented by the addition of sulfigran, rather than by adding the catalyst in solution.)

Sulfigran may in general be considered as a regulator, and the amount of sulfigran used is determined in practice by the amount of corrosion (intermediate layer in the coal catchpot), i.e. by shifting the reaction into the acid or alkaline range. It is further important in this connection to point out that on theoretical grounds it would be correct to add $FeSO_4$ to the upper limit, because the real catalytic action is to be attributed to the ferrous salt, while the Bayer mass, even more than the Lux mass is oxidic, i.e. contains the iron in the ferric state.

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*The original plans for using the Lauta mass had to be changed after large scale tests because of its high water content (50%): no proportioning of it above the bunkers was possible (plugging up and lump formation). This may also have been the reason why Gelsenberg uses the dried Bayer mass.

These considerations were the principal reasons for expecting success with the hydroxidic masses; the systematic supervision of the mixing of the coal with the catalyst and of the alkalinity and the ferrous sulfate content seem to be valuable aids. There are some interesting investigations available about alkalinity.

	Alkalinity (g H_2SO_4/kg dry Substance)
Ruhr Coal	10
Upper Silesian soft coal	40
Bayer of Lux mass	263-278
Upper Silesian Coal + 1.2% $FeSO_4$ + 1.5% Bayer mass	42
do. + 0.3% Na_2S	43

Dr. K. mentions the beneficial effect of $FeSO_4$ on hydrogenation in spite of the "polymerization terror". However, should we suspect ferrous sulfate as the "guilty party" in the formation of the crust inside the preheater, one may ask oneself why, should the addition of ferrous sulfate cause it, its addition to the first converter, would be beneficial as means of preventing the formation of the crust.

Luckily, the formation of the crust is connected less with the $FeSO_4$ than with the Bayer mass, which has apparently a great tendency to settle, especially in the presence of high-sulfur products, with a reduction to FeS. Dr. K's other assumptions will not be discussed here because of absence of exact investigations.

W. M. Sternberg

Newman

TOM Reel 177
Pps. 1355-56

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-146

KCBraun

Production Costs of Auto Gasoline and DHD-Feed from Bituminous Coal

Lu, 4 November 1942

700 Atm. Liquid Phase. Capacity 146,000 tons/ann. Gasoline + Middle Oil.

	RM/ton Gasol. + Mi-Oil		
1.73 t. hydr. coal, 10% water, 4% ash @ 23.00	40.00		
1900 m³ H₂ @ 0.044 (Pintsch-Brassert-Gen. 24.00/ton coke)	83.50		
Liquid Petrol. Gas Credit 141 Kg. @ 0.20	21.20		
Hy-Gas Credit 2.95 x 10^6 WE @ 6.00	17.10	85.20	
Catalyst and Chemicals	2.40	2.40	
Power (HP-steam 4.50/t, LP-steam 3.70/t, current 0.015 KWh, water 0.01/m^3, Hy-gas 6.00/10^6 WE)	32.00	32.00	
Wages & Salaries, Operating materials & Laboratory costs (salaries 30% of wages, oper. materials and laboratory costs 20% of wages)	19.60	19.60	
Repairs, 6.5% of 34,200,000	15.20		
Fire protection and taxes (fire protection 0.6%)	7.50		
Amortization, 10% of 34,200,000	23.40	46.10	185.30
General, 10% of 185.30	18.50		
Interest, 3% of 34,200,000	7.00		
Interest, 5% of 2,300,000	0.80	26.30	
	Total		211.60

Vapor Phase, 300 Atm.

	RM/t Auto-Gasoline		RM/t 170° E.P. Gasol. (Based on Gasol. free of C_4)	
Liq. Ph. Gasoline + Mi-Oil	1.06 t	= 222.50	1.11 t	= 227.25
Hydrogen	800 m³	33.60	665 m³	37.20
L.P.G. (Liq. Petrol. Gas) (Credit)	66 Kg	13.20	121 Kg	24.20
[illegible]-Gas @ 1000 [illegible] (Credit)	245 m³	2.10	475 m³	[illegible]
Catalyst & Chemicals		4.80		5.15
Power		11.80		12.35
Wages, Salaries, Operating Matls. and Laboratory Costs		9.20		8.95
Repairs, 6.5% (based on estimate of cost of plant for 170° C E.P. Gasoline)	12,700,000	6.00	13,800,000	6.85
Fire Protection & Taxes		3.45		3.40
Amortization	(310.70)	19.35	(311.65)	10.50
General, 10% of total less raw materials		6.30		5.75
Interest, 3% of	12,700,000	3.10	13,800,000	3.15
Interest, 5% of	820,000	0.30	900,000	6.35
		320.40		326.90
			Stabilization	2.50
			Wash	0.90
			Unexplained (40 x .20)	8.00
				338.30

Vapor Phase, 700 Atm.

		RM/t Auto Gasol.	RM/t	170° E.P. Gasoline
Liq. Ph. Gasoline + Mi.-Oil	1.11 t =	227.25	1.14 t =	233.50
Hydrogen	675 m³	28.40	695 m³	29.20
L.P.G. (Liq. Petrol. Gas)(Cred)	58 Kg	11.60	100 Kg	21.60
Hy-Gas @ 1000 WE (Credit)	340 m³	3.29	610 m³	3.65
Catalyst & Chemicals		1.35		1.45
Power		13.70		14.55
Wages, Salaries, Operating Matls. and Laboratory Costs		9.60		9.40
Repairs, 6.5% (based on estimate of cost of plant for 170° C.E.P. Gasoline)	16,600,000	8.25	16,600,000	8.45
Fire Protection & Taxes		3.95		4.00
Amortization	(305.15)	12.65	(313.50)	12.95
General, 10% of total less raw materials		6.30		5.50
Interest, 3% of	16,600,000	3.80	16,600,000	3.90
Interest, 5% of	1,880,000	0.40	1,080,000	0.40
		315.65		348.50
			Stabilization	2.50
			Wash	0.90
			Unexplained (40 x .20)	8.00
				359.90

KCBraun

TOM Reel 177
Pps. 1357-58 & -65

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-147
KCBraun

Flow Sheet at Lu-Op Plant for Auto Gasoline or 170° C E.P. Gasoline for DHD from Bituminous Coal.

Lu, 2 November 1942

Running: Gasoline + Middle Oil		Running: 32% Heavy Oil Excess	
Liquid Phase 700 Atm. L = 0.245		Liquid Phase 700 Atm. L = 0.4 →	
Primary Crushing,	31.6 t/h	Primary Crushing	50.5 t/h
Drying,	32.0 t/h	Drying	51.5 t/h
Paste Milling & Pressing	74.0 t/h	Paste Milling & Pressing	111.4 t/h
Gas Circulation,	123,000 m^3/h	Gas Circulation	197,600 m^3/h
Oil Washing,	110 m^3/h	Oil Washing	150 m^3/h
Compression,	35,000 m^3/h	Compression	50,000 m^3/h
Stalls, 2 (4 converters, 18 m. lg. x 1000 mm. dia.)		Stalls, 2 (4 converters, 18 m. lg. x 1000 mm. dia.)	
Centrifuge Plant,	45.0 t/h	Centrifuge Plant	73.3 t/h
Kiln Plant, (Low temp. carboniz.)	8.1 t/h	Kiln Plant	14.2 t/h
Cold Catch Pot Product Distillation	41.7 t/h	Cold Catch Pot Prod. Distillation	75.5 t/h
Yield from Column, 3.31 t/h Gasol. @ 200° C. & 14.9 t/h Mi-Oil @ 325° C.		Yield from Column, 5.55 t/h Gasol. @ 200° C. & 27.85 t/h Mi-Oil @ 325° C.	
L.P.G. (Liqu.Petrol.Gas) and V.R.G. (Vapor Recovery Gasoline 0.55 t/h C_5 + 2.58 t/h C_3 & C_4		L.P.G. & V.R.G., 0.9 t/h C_5 + 3.58 t/h C_3 & C_4	
		Fuel Oil	14.75 t/h

	Vapor Phase 300 Atm.		Vapor Phase 700 Atm.	
	170° Gasoline	Auto Gasoline	170° Gasoline	Auto Gasoline
Injection Pumps	18.95 + 19.6 t/h	18.3 + 16.3 t/h	34.7 t/h	33.0 t/h
Gas Circulation	76000 + 39000 m³/h	73000 + 32600 m³/h	104000 m³/h	99400 m³/h
Compression	—	—	11300 m³/h	11100 m³/h
Stalls	(1 (3 Convert.) + (2 (3 Convert.)	(1 (3 Convert.) + (1 (2 Convert.)	(1 (3 Convert.) + (1 (2 Convert.)	(2 (3 Convert.) + (2 (2 Convert.)
Cold Catch Pot Dist.	18.2 + 16.9 t/h	17.6 + 16.3 t/h	33.2 t/h	[illegible] t/h
Yield Fr. Column	6.0 + 10.4 t/h	6.1 + 10.2 t/h	16.6 t/h	16.45 t/h
Gasol. Stabilization	—	16.6 t/h	—	16.45 t/h
Gasol. Wash	—	16.6 t/h	—	16.45 t/h
(Debenzination and (L. P. G. Production	(0.33 C_5 + 2.0 (C_3 + C_4 t/h	(0.3 C_5 + 1.16 (C_3 + C_4 t/h	0.24 + 1.73 t/h	0.24 + 0.97 t/h
Hy-Gas Prod. - 95% Recovery	111.7 x 10^6 WE	100 x 10^6 WE	110.5 x 10^6 WE	101.5 x 10^6 WE
Aromatics, Recovery %	Abt. 10	Abt. 9	32	31
Total H_2 Consumption	(13600 m³/h (35000 m³/h 48600 m³/h	(13450 m³/h (35000 m³/h 48450 m³/h	(11100 m³/h (35000 m³/h 46100 m³/h	(11100 m³/h (35000 m³/h 46100 m³/h

	Vapor Phase 300 Atm.		Vapor Phase 700 Atm.	
	170° Gasoline	Auto Gasoline	170° Gasoline	Auto Gasoline
Injection Pumps	15.2 + 10.3 t/h	15.05 + 9.5 t/h	25.0 t/h	24.1 t/h
Gas Circulation	61000 + 20600 m^3/h	60000 + 19000 m^3/h	75000 m^3/h	72000 m^3/h
Compression	—	—	7180 m^3/h	7250 m^3/h
Stalls	1 (3 Conv.) + 1 (2 Conv.)	1 (3 Conv.) + 1 (2 Conv.)	1 (4 Conv.)	1 (4 Conv.)
Cold Catch Pot Dist.	14.3 + 9.9 t/h	14.2 + 9.15 t/h	23.2 t/h	24.0 t/h
Yield fr. Column	7.9 + 5.65 t/h	7.8 + 5.9 t/h	13.45 t/h	13.7 t/h
Gasol. Stabilization	—	13.7 t/h	—	13.7 t/h
Gasol. Wash	—	13.7 t/h	—	13.7 t/h
(Debenzinization and ((L.P.G. Production	0.3 C_5 + 1.45) C_3 + C_4 t/h)	0.28 C_5 + 0.84) C_3 + C_4 t/h)	(0.27 C_5 + 1.18 ((C_3 + C_4 t/h	(0.27 C_5 + 0.67 ((C_3 + C_4 t/h
Hy-Gas Prod.-95% Recovery	—	—	—	—
Aromatics, Recovery %	Abt. 10	Abt. 9	31	30
Total H_2 Consumption	(10400 m^3/h ((50000 m^3/h 60400 m^3/h	9600 m^3/h 50000 m^3/h 53600 m^3/h	(7180 m^3/h ((50000 m^3/h 57180 m^3/h	(7250 m^3/h ((50000 m^3/h 57250 m^3/h

H_2 Production

For Lu-Op Plant for Bituminous Coal

I. From Coke:

a). DHD-Gasoline, 700 Atm. Vapor Phase (including liquid phase).

Hy-Gas (95%)	110,000,000 WE	
DHD-Gas (95%)	13,000,000 WE	
RWG (Restwatergas)	29,000,000 WE	152,000,000
L.P.G. from Hy-& DHD-Gas	53,000,000 WE	
Fuel Gas Requirement for Hydr. & DHD	46,000,000 WE	- 99,000,000
	Excess	53,000,000

b). DHD-Gasoline, 300 Atm. Vapor Phase (including liquid phase).

Hy-Gas	112,000,000 WE	
DHD-Gas	29,000,000 WE	
RWG	31,000,000 WE	172,000,000
L.P.G. from Hy-& DHD-Gas	64,000,000 WE	
Fuel Gas Requirement for Hydro. & DHD	52,000,000 WE	-116,000,000
	Excess	56,000,000

II. Hy-Gas Splitting:

a). DHD-Gasoline, 700 Atm. Vapor Phase (including liquid phase).

Fuel Gas Requirement for Hydr., DHD & Splitting	92,000,000 WE (Foreign)	
Hy-Gas + DHD - Gas	125,000,000 WE	
L.P.G.	-53,000,000 WE	70,000,000
Hy-Gas Requirement for splitting	115,000,000 WE	

Accordingly, only abt. 60% of the H_2 can be produced from Hy-Gas at full L.P.G. recovery.

b). DHD-Gasoline, 300 Atm. Vapor Phase (Including liquid phase).

(Fuel Gas Requirement for Hydr.,		
(DHD & Splitting	102,000,000 WE (Foreign)	
Hy-Gas + DHD-Gas	141,000,000 WE	
L.P.G.	-64,000,000 WE	77,000,000
Hy-Gas Requirement for Splitting	125,000,000 WE	

Accordingly, only abt. 60% of the H_2 can be produced from Hy-gas at full L.P.G. Recovery.

Power Requirement

I. 170° E.P. Gasoline & 300 Atm, incl. Liq. Phase & H_2 Production

	HP.Steam	L.P.Steam	Gas	Water	Current
Hydrogenation, Vap. Ph.	0.1	6.0	9000	1110	2245
H_2	35.0	70.0	----	4450	26500
Hydrogenation, Liq. Ph.	7.3	27.5	34000	1830	13500
	42.6	103.5	43000	7390	42245

2. 170° E.P. Gasoline & 700 Atm.

Hydrogenation, Liq. Ph.	7.3	27.5	34000	1830	13500
H_2	33.0	65.0	-----	4100	24600
Hydrogenation, Vap. Ph.	0.7	6.0	6200	1150	4545
	41.0	98.5	40200	7080	42645

b). DHD-Gasoline, 300 Atm. Vapor Phase (Including liquid phase).

(Fuel Gas Requirement for Hydr.,
(DHD & Splitting 102,000,000 WE (Foreign)

Hy-Gas + DHD-Gas	141,000,000 WE	
L.P.G.	-64,000,000 WE	77,000,000
Hy-Gas Requirement for Splitting	125,000,000 WE	

Accordingly, only abt. 60% of the H_2 can be produced from Hy-gas at full L.P.G. Recovery.

Power Requirement

I. 170° E.P. Gasoline & 300 Atm. incl. Liq. Phase & H_2 Production

	HP. Steam	L.P. Steam	Gas	Water	Current
Hydrogenation, Vap. Ph.	0.1	6.0	9000	1110	2245
H_2	35.0	70.0	----	4450	26500
Hydrogenation, Liq. Ph.	7.5	27.5	34000	1830	13500
	42.6	103.5	43000	7390	42245

2. 170° E.P. Gasoline @ 700 Atm.

Hydrogenation, Liq. Ph.	7.3	27.5	34000	1830	13500
H_2	33.0	65.0	-----	4100	24600
Hydrogenation, Vap. Ph.	0.7	6.0	6200	1150	4545
	41.0	98.5	40200	7080	42645

Newman

T 148

TOM Reel 177
Pps. 1359-64

U.S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

KCBraun/jw
11/6/46

Estimate of Cost of Plant Producing 125,000 t/ann. DHD-Feed for 100,000 t/ann. High Performance Gasoline from Bituminous Coal, at Ludwigshafen-Oppau.

Lu, 3 November 1942

Summary

1.	H_2 Production (Gas Plant Expansion at Oppau)	= 24.5%	= RM 20,700,000 -
2.	Hydrogenation Proper	= 47.3%	= RM 40,000,000 -
3.	Hygas Cleaning		= 1,400,000 -
4.	Power Production (Expansion of Power Plant and Water works)	= 8.3%	= 7,000,000 -
5.	Power Distribution } + (3)	= 19.9%	= 4,000,000 -
6.	Auxiliary Plants }	100.0%	= 11,000,000 -
7.	Miscellaneous	6.5%	= 5,500,000 -

Total Cost= RM 90,000,000.00

1. H_2 Production

50,000 m^3/h from Coke

This part of the plant will be built at Oppau as an expansion of the existing gas plant.

Coke Storage and Conveying Equipment	RM	300,000.00
Water Gas Plant for 50,000 m^3/h H_2, 9 + 1 Generators		6,500,000.00
Plant for H_2S Removal by "F-coal".		1,400,000.00
Catalytic H_2 Production		2,300,000.00
Gas Tanks		400,000.00
CO_2 Cleaning		2,000,000.00
Compression to 325 atm.		5,000,000.00
Pipe Lines		800,000.00
Total RM		20,700,000.00

2. Hydrogenation Proper

Flow Sheet of 2. November 1942.

This part of the plant and (3) will be built on the terrain between Ludwigshafen and Oppau.

a). Liquid Phase, 700 atm.:

Coal storage, grinding, drying and mashing	RM 2,300,000.00
Paste Press Division	2,400,000.00
Gas Circulating Pump Division	800,000.00
Coal Stalls, 2 x 4-stage	10,000,000.00
Circulating Gas Wash	2,000,000.00
Residue Centrifuging	1,300,000.00
Residue Low Temp. Carbonization (Kilns)	1,400,000.00
Tank Storage for Heavy Oils	700,000.00
Coal Catch Pot Distillation	1,300,000.00
Compression, 300 to 700 atm.	1,000,000.00
High and Low-Pressure Lines, incl. Recirculating Lines	1,500,000.00
Total RM	25,300,000.00

b). Vapor Phase:	700 Atm.	300 Atm.
Injection Pumps and Gas Circulating Pumps	RM 1,600,000	RM 1,600,000
Gasoline Stalls, 1 x 3-stage, 1 x 2-stage & 2 x 3-stage, respectively	7,300,000	5,400,000
Tank Storage for Light Oils	700,000	700,000
Gasoline Catch Pot Distillation	1,000,000	1,200,000
High and Low Pressure Lines	1,200,000	1,000,000
Compression, 300 to 700 atm.	400,000	--
Total	RM 12,200,000.00	RM 9,900,000.00

2. c). **Common Installations:**

Distributing Tank Storage Filling Station	RM	1,500,000.00
L.P.G. Plant & Debenzination Filling Station		2,000,000.00
Insulation Scaffolding, Emergency Pressure Release		300,000.00
Pipe Lines		1,000,000.00
Total RM		4,800,000.00

3. **Hygas Cleaning:**

Alkacid Plant	RM	400,000.00
Claus Plant (Oxygen Burning-?) Acid-Proof Stack		600,000.00
Blower House for H_2S Oils		150,000.00
Gas Tank		200,000.00
Miscellaneous		50,000.00
Total RM		4,000,000.00

4. **Power Production:**

Expanding the Power Plant, 1 Boiler & 1 Turbine	RM	5,000,000.00
Expanding the Water Works and build Cooling Towers		2,000,000.00
Fuel Gas Requirement is covered by the Residual Water Gas (Restwassergas) and Hygas without C_3, C_4, & C_5		---
Total RM	RM	7,000,000.00

5. **Power-Distribution:**

Steam Lines	RM	1,000,000.00
Water Lines		500,000.00
Elect. Power Lines, incl. Transformer Station		2,000,000.00
Hygas Lines		400,000.00
Miscellaneous		500,000.00
	Total RM	4,400,000.00

6. **Auxiliary Plants:**

Work Shops incl. Instrument Repair Shop, Forge Shop, Electrical Repair Shop and Stores	RM	1,200,000.00
Laboratories		1,000,000.00
Factory Buildings		800,000.00
Wash Rooms		400,000.00
Air Raid Shelters		1,000,000.00
Preparing the Site		1,500,000.00
Railroad Tracks and Roads		1,500,000.00
Sewers		1,000,000.00
Phenol Water Purification		500,000.00
Electric Wiring & Water Supply in Buildings		200,000.00
Construction Buildings		100,000.00
Railway Vehicles		1,000,000.00
Non-Rail Vehicles		200,000.00
Fencing		50,000.00
Miscellaneous		550,000.00
	Total RM	11,000,000.00

7. Miscellaneous:

Catalysts	RM	1,000,000.00
Design		1,000,000.00
Construction Expenses & Supervision		2,000,000.00
Participation in Building of Homes		1,000,000.00
Purchase of Site		On hand
Workers Shelter		On hand
Miscellaneous		500,000.00
	Total RM	5,500,000.00

Newman

~~TOM Reel 75~~
Frames 638-639

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-149

DEVELOPMENT OF LIQUID PHASE CATALYSTS FOR BROWN COAL

Pölitz, March 7, 1944

Dr. Döllein, with Dr. Becker, have told about the development of the catalyst additions. In 1927-1929 brown coal was hydrogenated in Leuna without the addition of any catalysts. It had however been found that when proceeding in this way, the utilization of brown coal amounted to but about 60%, and the asphalt content of the HOLD was so high, that any subsequent operations with the HOLD was made difficult. Hydrogenation on a large scale was partially interrupted during the economic crisis of 1929, but small scale operations were continued in Leuna, and hydrogenation has been tried with the addition of catalysts to the liquid phase. The addition of 2% of Mo, calculated as MoO_3, added as the ammonium molybdate, produced very good results. Utilization suddenly proceeded to values which were economically permissible. Subsequent experiments showed, that if the alkalinity of the brown coal was neutralized with sulfuric acid to within 25% of the total neutralization value, 0.05% Mo, calculated as MoO_3, produced the same amount of utilization, as larger amount of it to the not neutralized coal. (These tests gave probably rise to the theory, that hydrogenation and splitting of coal proceeded better in an acid medium.) Further tests were made with nickel salts which also produced very good results. However, the above mentioned compounds were not available in sufficient quantity, nor did they offer many advantages from an economic view point, and the addition of iron salts was also found to give gratifying results. Moreover, coal neutralized with sulfuric acid before the addition of iron salts acted less well than iron salts used as such. It had even been found, that the addition of iron salts with alkalies produced the best utilization of the coal, which has led to the development of catalyst 10927, alkalized iron sulfate upon grude. The coal was moistened in Leuna with a solution of iron sulfate, the amount of the latter being 0.8%, calculated as Fe. This iron sulfate was next neutralized with an equivalent amount of alkali. The alkalies used were, in addition to the hydroxide, also sodium carbonate and sulfigran. It has been assumed that the ferrous hydroxide first formed interacted with the sulfur of the coal to form ferrous sulfide, which was especially active as a catalyst. Iron sulfate itself was not available in sufficient amounts, and lauta mass, lux mass, and finally the Bayer mass have been tried. Patent difficulties had at first to be overcome because Bergius had claimed the lux mass as an active catalyst in his original patent. Tests have shown that while these products acted catalytically, their activity was less than that of ferrous hydroxide produced be precipitation from ferrous sulfate and sodium hydroxide. We may say that, as a rule, that about double the amount of the Bayer mass, in terms of its iron content, was necessary to produce the same results upon coal, as ferrous sulfate. At present, Leuna is using the Bayer mass, and has found the addition of 1.8% of it, in terms of iron, as being the proper amount. This represents about 5% of the Bayer mass. No particular effects are produced by using more. Sulfigran and iron sulfate are no longer added in Leuna.

Ludwigshafen has used in hydrogenation tests in 1939: 1.2% $FeSO_4.7H_2O$ + 1.5% Bayer mass + 0.3% sulfigran, all computed to dry coal. This, recalculated, gives 0.2% Fe as ferrous sulfate, 0.4% as the Bayer mass, or a total addition of 0.6% Fe. The 0.3% sulfigran is approximately equivalent to the iron of the iron sulfate. These amounts of catalyst for soft coal are appreciably below those calculated for brown coal. The amount of iron sulfate was reduced presumably because the additional drying requirements of the water added with the sulfate to the coal made such reduction advisable. Dr. Dölein and Dr. Becker could add no further information on the addition of iron catalysts to soft coal.

signature illegible

W. M. Sternberg
11/5/46

TOM Reel 152
Frames 657-658

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-150

Distillation of 8 to/h of Hydrogenation Residues in Pölitz

Recklinghausen
Oct. 16, 1945

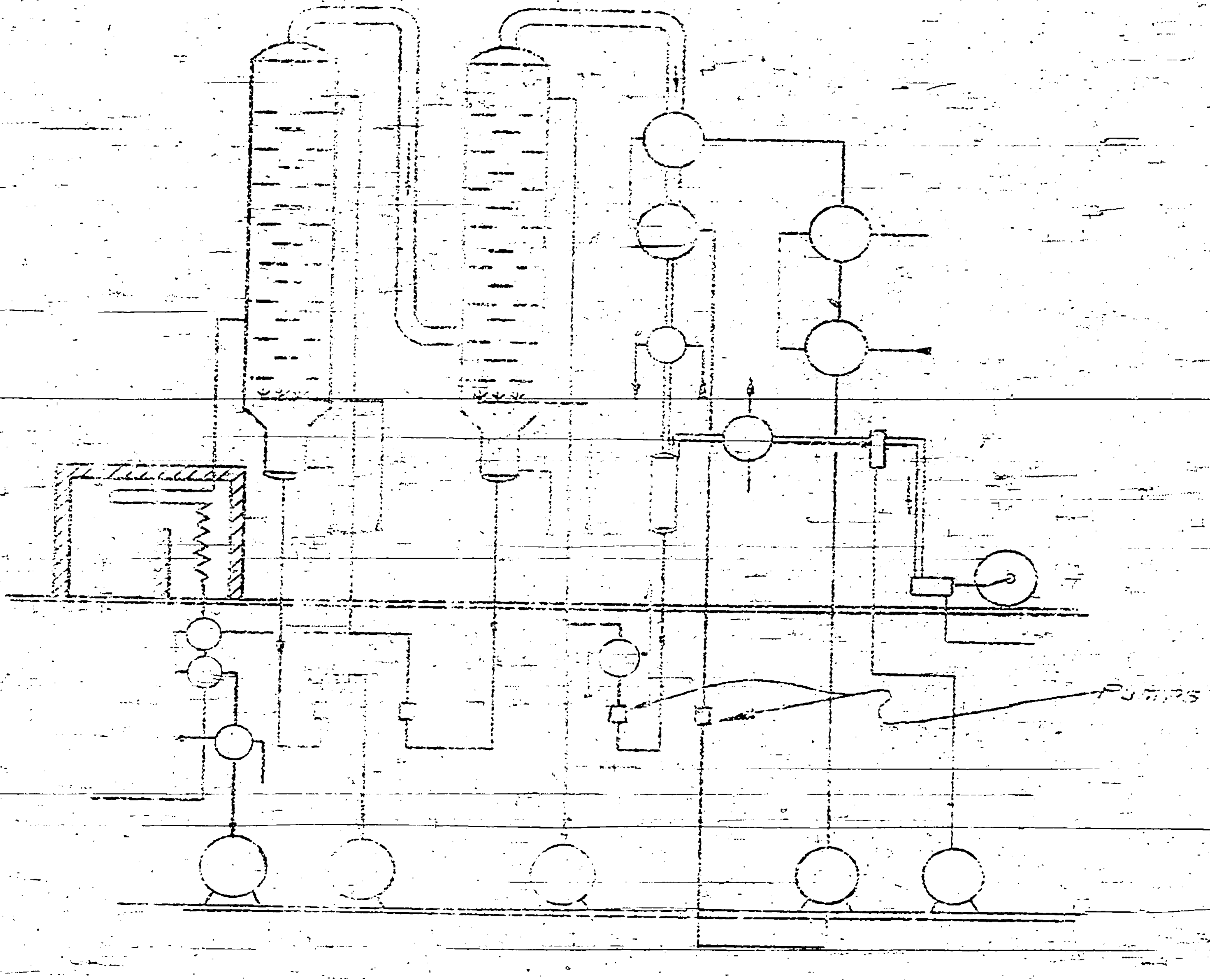
The material to be treated is the so-called hydrogenation sludge consisting of 80% oil and tar particles and 20% of powdered ash and of catalyst constituents.

The material is distilled in vacuum. It is transmitted by a pump through different heat exchangers in which it is preheated by the foots of the vacuum distillation column, then through a tubular furnace of the usual construction and made to enter the distillation column. The residue boiling above about 450° (at 760 mm Hg) is drawn off from the bottom using a constant level regulator, and returned to a suitable place for use in the hydrogenation plant. The oil vapors leave the column at its head, and enter a vacuum fractionating column still kept at about 50 mm Hg, where they are separated into two fractions, up to 350° and between 350 and 450° (referred to 760 mm Hg pressure). Some of the middle oil is drained off at the bottom of the vacuum still and returned to the head, while the rest of the output is pumped through heat exchangers and coolers to the proper storage tanks. Oils with a boiling range of up to 350° leave at the head of the column, pass through two oil coolers and a water cooler into tanks which adjoin the stills. Some of the output from this tank is pumped back to the head of the vacuum fractionating still, while the balance of the production from this pump is sent to final cooling and into the proper tank. Provisions are made for temperature regulators for the heads of the two columns as well as level regulators of the bottom of the vacuum fractionating column and for the distillate from the first unit. The vacuum pump can maintain a vacuum of 50 mm Hg abs.

The plugging-up with the solids of the HOLD sludge is avoided by not building the fractionating columns with the usual bell-jar bottoms but with mutually connected packed screen bottoms, as shown in the accompanying sketch.

W. M. Sternberg
11/19/46

T-150



HYDROGENATION RESIDUE DISTILLATION SCHEME

Lab. S 916

TOM Reel 43
Frame 209

U. S. BUREAU OF MINES
HYDRO DEMON. PLANT DIV.

T-151

Schwarzheide, 2/20/43

A NOTE ON KOPPERS GAS PRODUCER

Re: Powdered coal gasification.

Obermeister Gebhard of Brabag has made tests for a long time to gasify powdered coal in the producer furnished by us. The producer has operated for three weeks with a throughput of about 17 te coke, of which 6 - 7 te was powdered, introduced in layers. The producer was operating with no need to poke the fire and with no flames at the edge. The heating value of the gas is 1220-1230 heat units. A second steam inlet has been introduced above the grate.

Dr. Wagner is very much interested in these experiments because of the great saving possible if all producers operate in the same way.

We are interested in this improvement, and it would be proper for us to get in touch with Mr. Gebhard.

/s/ Häberle

W.M. Sternberg

TOM Reel 43
Frames 000210-214

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-151-a

Schwarzheide, 5/17/43

DISCUSSION OF KOPPERS GAS PRODUCERS

1. Production of Fuel Gas.

Mr. Kaden has stated about fuel gas production, that it had been decided to introduce no changes in the Schmalfeldt installation because any changes decided upon will merely be compromises; the gas producers must be designed for powdered fuel gasification. We have accordingly been asked to submit bids for a usual design. An engineer's report will then be handed us on the design of this installation. No approval by the Government is here required, as has been found in a discussion with Director Altpeter.

A gas of high heating value is needed for the heating of the Cowper unit, and efforts must be made to operate the Schmalfeldt unit as originally intended, i e., by gasifying powdered grude with a mixture of rinsing gas and steam. Getting a gas with a heating value of about 2000 heat units is expected, and it is to be used as a fuel gas.

We advised, that such a construction would be attempting no worthwhile objective, and that

1) such a gas would consume much more heat than it would produce, and

2) carbon will be but partially gasified; but this did not interfere with the program of these gentlemen. This experiment will have to be run at all costs. However, we already are in position today to forecast the results on the strength of our experience. We can and shall prove in our Rheinpreussen installation to the gentlemen involved the impossibility of the method.

Schmalfeldt in Lützkendorf is the originator of these tests. The fact, that the producer is fed with the Lützkenford brown coal, and that the solid carbon is there utterly unattacked, is kept completely out of consideration. The Schmalfeldt installation is nothing more than a distillation and cracking unit. Solid carbon is carried out practically unattacked into the dump. In Ruhland, powdered grude i.e. a product dumped in Lützkendorf is to be substituted for powdered brown coal. These arguments can not be seriously contradicted, and it was decided to have the practical tests run regardless of the results, and the statement modified by saying, that the producer will not be used for gas production, but for reforming the fuel gas.

It pays therefore to discuss the whole question, because we would prefer to build a normal unit of our system, instead of rebuilding the Schmalfeldt installation.

We shall accordingly expect a request for that kind of an installation.

2). Production of Synthesis Gas.

Brabag has received the commission to design a Fischer-Tropsch installation. It is to be located in Upper Silesia.

Raw material:	Upper Silesian soft coal, including grude coke.
capacity/hour	100,000 m^3 $CO + H_2$ (8,000 hours/year)
$CO : H_2$	1.65 : 1

The gas is to be used for intermediate pressure synthesis with iron catalyst, very likely to produce Katsch (?). The latter is an assumption based on the very great interest taken in this installation by Dr. Keppler, whose special field we know to be the production of edible fats.

The gentlemen at the Ingenieurtechnischen Zentrale are fully conversant with our process as a result of discussions carried out for the Zeitz addition, and we need not include here many detail. We have on our part stated, that no special requirements need be made with regard to the coal. The coal must be readily ground, i.e., abt. 75% must pass through 4900 mesh (= 175 mesh, U.S.). The required gas composition with this ratio of $CO + H_2$ has been repreatedly obtained in our Rheinpreussen pilot plant. The Gas Company has made complementary tests on condensable hydrocarbons as well as on the sulfur content. This experience has enabled us to give accurate information on which to base subsequent design and to claim the ability to produce gas of the following quality:

Raw Powdered Coal:	Water	1.95%
	Ash	8.75%
	V.M.	22.3%
	Fixed Carb	77.7 %
	Heat units	7650 H.U./kg
Ratio $CO : H_2$ in gas produced		1.6 : 1

Composition of the gas:	CO_2	11.0%
	CO	54.0%
	H_2	34.0%
	N_2	1.0%
Concentration of CO + H_2		88.6%

Minimum heating value	per nm^3 Sy. Gas	2505 H.U.
Amount of gas	per kg raw powd. coal	2.10 nm^3
O_2 consumption:	per kg raw powd. coal	0.57 "
	per nm^3 synth. gas	0.27 "
CO_2 consumption	per kg raw powd. coal	0.14 "
undergrate firing	" " " " "	350 H.U.
Steam production, 16 atm, 350° C	" " " " "	0.71 kg
Steam consumpt. 3 atm, sat.	" " " " "	0.40 "

This represents our normal construction. However, the heater unit is too large, and we ordinarily use one heater for two gasifiers and coolers as well as desintegrators. Such a unit would produce 20,000 m^3 of a gas of the above character.

The discussion of the number of aggregates to be ordered has assumed great latitude. We have agreed that a total of six aggregates should be built, one of which is to be used as reserve and for emergency. The price quoted was 2.7 mill. RM per aggregate. This means 16.2 mill. for the six aggregates. The cost of gas given by us is 1.7 Pfg, based on the following:

1)	1 te ground powdered coal, at the bin	RM. 14.00
2)	1 nm^3 95% oxygen	" 0.025
3)	1 8-hour labor shift	" 10.00
4)	1 kwh power	" 0.02
5)	1 m^3 clarified and cooled circulating water	" 0.025
6)	1 m^3 make-up water	" 0.025
7)	1 m^3 boiler water	" 0.25
8)	10^6 kcal heat gas	" 4.75
9)	1 te steam, 16 atm	" 3.00
10)	1 t steam, 3 atm	" 1.50
11)	Amortization and interest	12%
12)	Maintenance and repair	2%

The Ingenieurtechnische Zentrale is in position to start on the preliminary designs. We have been asked to present a written bid in greatest detail, and if possible to supplement it at least with data on coal preparation and water treatment (the data on which will be communicated by telephone). We are also asked to give information on the sulfur content and the composition of the sulfur compounds in the gas, based on tests made at Rheinpreussen.

/s/ Sect.I/O.S.

W.M. Sternberg (signature undecipherable)

TOM Reel 43
Fr. 000219-224

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-152

April 13, 1943

BRABAG-ZEITZ POWDERED COAL GASIFICATION

Discussions on the above subject have taken place in Böhlen on April 8 and 9, 1943. The participants included representatives of Brabag-Böhlen, Brabag-Zeitz and the Koppers Company.

Schwarzheide was discussed on April 8 and the discussions reported in a separate report.

The consideration of the gas producer unit at Schwarzheide was to be postponed in accordance with Speer's decision not to start any construction work which could not be put in operation prior to the end of 1943. Opportunity has presented itself, however, to thoroughly discuss the basic questions.

We based our discussions on a comparison of the coal and oxygen requirements of our process with the coal and oxygen requirements of the Winkler process and used this comparison in the decision on economy of our process in comparison to that of the Winkler process.

During these dicussions Dr. Mott furnished us in confidence for comparison with our figures the operating results of the Winkler process during the gasification of brown coal dust from his report to the management of Brabag. It must be emphasized here, that in so far as the Winkler process is concerned, the comparison is only of theoretical interest, because no powdered brown coal can be gasified in the Winkler producer because of the low gasification temperatures necessitated by the process.

Mr. Mott suggested never-the-less to base the comparison on powdered brown coal gasification, because this was the only fuel for which we had operating data by our process. We have given in the appendix to this report the operating costs of the raw powdered coal gasification from Dr. Mott's report. According to them the production costs per nm^3 of CO + H_2 are,

for the Winkler process	2.11 Pfg
for the Koppers process	1.82 "

The saving by our process will amount therefore to 0.29 Pfg per nm^3 of CO + H_2, which will figure, on the strength of the daily production of 646,000 nm^3 CO - H_2 to a daily saving of RM 1870.-, and a yearly savings of RM 682,000.-.

Dr. Mott points out in his report, that there are, in addition to the greater economy of production of the Koppers process, also the following advantages in the use of the gas:

1) The $CO + H_2$ concentration in the Koppers process is 85%, against 72.5% by the Winkler process, requiring lower compression costs in the preparation of the gas.

2) The Koppers installation produces gas free from hydrocarbons, while the Winkler gas contains 1.2-1.5% methane, which results in considerable savings by using our gas in hydrogenation in terms of hydrogen consumption per te of hydrogenation product.

Dr. Mott said in his summary that the Koppers process is superior in economy and technically to all the other known powdered coal gas producers.

During the discussion on April 9, Dr. Willie and Dipl. Ing. Lackner stated first, that the Zeitz construction was being decided upon, and nothing definite about it could be said for two weeks, while permission was being sought for it in view of the fact that 50 million Reichsmark have already been spent for the Zeitz plant. Dr. Willie emphasized however, that when the construction work in Zeitz is resumed, our system will be used, and one unit will at first be installed capable of being enlarged to the four aggregates as designed by us. He emphasized the extreme importance of testing our process for the production of water gas in Brabag, because of the impossibility of doing so in Schwarzheide because of the absence there of an oxygen unit.

We have submitted our bid of the project B for a powdered coal gasification unit at Zeitz with one producer aggregate of a capacity corresponding to one Cowper unit, for a price of RM 1,480,000.

Dr. Willie is of the opinion that the preliminary construction of a small Cowper unit can not be avoided, and suggests breaking down the four aggregates provided for the large Cowper group into aggregates for two smaller Cowper groups, to have the installation consist of two gasifier aggregates with a capacity corresponding to the Cowper unit. In this way, a single aggregate would be built for the Cowper group, which will be tested, and if approved, a second aggregate put in, and if needed a second Cowper group added with two producer aggregates. He requests that this proposition be tested and a price bid made.

Economy was discussed next without reference to Dr. Mott's confidential information. It has been stated by the gentlemen, that it would be difficult to make a comparison of our process with the Winkler process, because we had limited ourselves to operating data on powdered brown coal gasification, which can not be gasified in a Winkler producer. A correct comparison only becomes possible after we give operating data on the gasification of grude coke. Dr. Lockner has given the approximate composition of grude coke as:

Water,	about	3%
Ash,	"	20
C	"	71
H	"	2
O + N + S	"	4

Dr. Lackner called attention to the fact, that in the Winkler process about 250 g multiclone coal dust with 50% C is collected per nm^3 of gas. The heating value of the dust is 4200 kcal per kg and it can be used as boiler fuel, and must therefore be evaluated RM 5.- per te in the economic balance.

We have also been requested to prepare a design and bids for an installation for one producer aggregate, composed of two aggregates, with two aggregates per Cowper group, and to give operating data for gasification of powdered grude in this installation in Zeitz. Dr. Mott gave us in confidence the operating data of Winkler producer operating on powdered grude.

The composition of the powdered grude is:

Water	2.09%
Ash	24.68%
C	69.63%
H	2.52%
O + N + S	1.08%
	100.00%

(I have only subsequently found that the sum of O + N + S could not actually add up 1.08%).

Composition of the gas:

CO_2	24.39
CO	28.81
H_2	44.42
CH_4	1.25
N_2	0.54
H_2S	0.59

Minimum heating value	2152 kcal/nm^3
Grude consumption/nm^3 gas	0.561 kg
C yield	68.58%
Efficiency of gasification	58.05%
Oxygen consumption	0.237 nm^3/nm^3 of raw gas
Steam consumption	0.608 kg/ " " "
Ash produced	12.1 kg/nm^3 raw gas, contains 54.1% C
Multiclone dust	180-200 g per nm^3 gas, " 43.1 "

Heating value 4041 kcal/kg

Steam production 0.622 kg/nm^3 raw gas

Power consumption 28.44 kwh/1000 nm^3 raw gas

Water consumption (circulating cooling water) 17 liters/nm^3 raw gas

We must count on RM 1.50/million kcal as operating cost with outside gas. This is the off- gas to be furnished us from the heating of the Cowpers.

In conclusion of our discussion, Dr. Mott has given us an opportunity to visit the Winkler plant.

There are three producers in Böhlen with a theoretical capacity of 30,000 m^3 of raw water gas. The unit can not, however, be used to capacity, and is usually operated only to 25,000 m^3 capacity. The impression created by the examination of the plant was very good. Anyway it is clear why the Winkler installation costs but one half the cost of ours per m^3 water gas. One may remark the following:

The oxygen-steam mixture is supplied underneath the stationary grate, set up of different pieces. A rotating stirrer revolves at a relatively high speed (abt. 2 rpm) above the grate. The fuel is supplied by two worm drives to the outside of the lining of the shaft. There is therefore no mechanical distribution of the powdered fuel in the shaft proper. The fuel is introduced about 700 mm above the grate. The secondary oxygen, with some steam is introduced about 500 mm above the fuel supply. (The steam is used for cooling of the tuyeres). The temperature in the fuel bed underneath the secondary oxygen addition is around 800° C. The temperature is carefully maintained, as slag formation is otherwise likely to occur. The temperature in the cupola of the gasifier, i.e. above the inlet of the secondary oxygen, is around 900°, and the water gas produced leaves the producer at that temperature. The ash formed is forced by the stirrers into two chutes located between the grate and the casing. The ash is carried out from these dropping chutes, by two worm conveyors located at the opposite sides, and is carried out into the dump from which it is removed periodically in cars at a temperature of 800°. This ash does not burn in the car, in spite of the 50% C it contains. The grain size of the ash is around 2 mm, and rather uniform.

The water gas produced enters a double pass vertical tubular boiler from above, goes downwards, enters through a partition wall into the second pass, leaves it above at a temperature of 400°, enters the steam superheater and finally the heat exchanger. Dust collecting bins are placed underneath the boiler and the heat exchanger from which the dust is withdrawn a temperature of 350° to 300° C. From the heat exchanger the gas enters the multiclone dust separator. 80% of the dust still remaining in the gas is separated here. The temperature in the multiclone is around 250°. From the multiclone the gas enters a gasholder with hydraulic seal, and from there through a collector main for all three producers into the final cooler with wooden baffles, where the gas is cooled to about 30° C. The gas is finally purified in Theisen scrubbers.

The pressures in the unit are as follows:

Underneath the producer grate - 2000 to 3000 mm water column; in the producer shaft - about 1500 mm water column.

The loss in pressure in the subsequent treatment is around 500 mm, in the collector main the pressure is reduced by further 200 mm, in the final cooling - 30 mm, and the gas pressure in front of the desintegrator (sic) is about 600 mm, i.e. the unit operates with no blowers ~~from~~ to the desintegrator directly from the water gasholder.

The fuel is blown with carbon dioxide to the operating bins of the producers. The operating bins of each producer hold 200 te grude. Attention is paid to the contents of fines in the grude, by having it contain the least possible amount of sizes under 0.5 mm, which do not gasify and are carried over by the gas into the rest of the equipment. The dust content of the gas varies between 200 and 300 g/nm^3, depending on the dust content of the fuel. 80% of this dust, with a C content of 50% is used as fuel in the boiler installation. The balance goes into the washwater of the final cooler and into the desintegrator.

The construction of the producer is very compact, without making supervision difficult, i.e. with a relatively large gasification capacity per unit area. The shut-off and control equipment for all the three units are housed in a common room about 5 m above the floor.

The service of the installation, aside from the dust and ash removal, is limited to the supervision of control and measuring instruments. As soon as the instruments indicate irregularities in the fuel, oxygen, or steam supply, the producer is disconnected from the producer gas main and a blow-off valve opened until the difficulty is overcome. The amount of labor for the whole installation is extraordinarily small.

It must be admitted, that the process and the construction of the installation represent a real accomplishment.

We later went on a trip with Dr. Mott through a distillation installation erected by us in Böhlen and through the Linde-Fränkel air separation plant. The latter, which supplies oxygen only for the Winkler installation, occupies practically the same ground space, as the Winkler installation. One may judge from the size of the equipment and machines, that a 98% oxygen must cost at least 3 Pfg/nm^3, when the price of power is 1.5 pfg/kwh.

Section I/designs

/s/Undecipherable.

W.M. Sternberg

Essen, April 13, 1942.

COST COMPARISON OF THE WINKLER AND THE KOPPERS POWDERED COAL GASIFICATION PROCESSES.

(Per nm^3 of raw water gas.)

Construction costs, for the production of 645,000 nm^3 ($CO + H_2$) in 24 hours, corresponding to 760,000 nm^3 water gas from Koppers producer, and 840,000 nm^3 water gas from Winkler.

Koppers installation, incl. reserve Cowper	RM 5,300,000
Winkler installation	3,000,000

Koppers Installation:

1.	Amortization and interest, 15%	Pfg 0.2870/nm^3
2.	0.16 nm^3 oxygen @, pfg 3.1	0.4960 "
3.	0.539 kg coal, RM 9.-/te	0.4770 "
4.	24 man-hour labor, a day @ RM 2.-/hr, incl. superv.	0.0560 "
5.	0.120 kg outside steam, @ RM 1.50/te	0.0180 "
6.	0.035 kwh power, 1.5 pfg	0.0525 "
7.	20 li. circulating water, @ pfg 2.0/m^3	0.0400 "
8.	0.53 li. feed water, @ pfg 20.0/m^3	0.0106 "
9.	650 kcal outside heat, @ RM 1.-/1 million H.U.	0.0650 "
10.	2.5% cost of repairs, referred to construction costs	0.0477 "
	Total	Pfg 1.5497/nm^3

= pfg. 1.82/nm^3 $CO + H_2$

Winkler Installation:

1.	Amortization and interest, 15%	Pfg 0.1470/nm^3
2.	0.237 nm^3 oxygen, @ pfg 3.1	0.7350 "
3.	0.561 kg coal, @ RM 9.-/te	0.5050 "
4.	18 man-hour labor a day, @ RM 2.-/hr, incl superv.	0.0343 "
5.	0.028 kwh power, @ pfg 1.5	0.0420/nm^3
6.	17 li. circulation water, @ pfg 2.0/m^3	0.0340 "
7.	0.622 li. feed water, @ pfg 20.0/m^3	0.0125 "
8.	3% repair costs on construction costs	0.0294 "
	Total production costs	1.5392/nm^3

= 2.11 Pfg/nm^3 $CO + H_2$

We have been told in the discussion with Mr. Mott that a comparison of operating costs by the Koppers and Winkler processes had only theoretical significance, because the brown coal gasification is predicated for both processes, although it is impossible to run a Winkler generator with it. For this reason, Dr. Mott, when compiling data for the Winkler generator and deriving the cost per m^3 water gas or per m^3 $CO + H_2$ based them on grude coke utilization, while the Koppers data were compiled on the basis of use of brown coal fines. The cost of the raw material, whether powdered brown coal or grude coke was set the same in the computations, or RM 9.-/te.

The comparison is therefore not only theoretically inaccurate, but actually wrong, being based on different fuels.

W. M. Sternberg.

TOM Reel 43
Frame 000260 -
000267

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-154

Essen, July 16, 1942.

OPERATION COSTS IN THE SYNTHESIS GAS PRODUCTION IN RHEINPREUSSEN

The operating costs were computed on the strength of the heat flow sheet of the Treibstoffwerk Rheinpreussen of 7-6-42 and the computations made from it by Mr. Schürhoff on 7-13-42.

1. Computation of operation costs of a water gas installation with 10 producers of 6000 m^3 hourly capacity = 60,000 nm^3 water gas/hour, or 1,440,000 nm^3 water gas/day.

<u>Expense per day:</u>

1.	Coke, $\frac{1,440,000}{1440}$ = 1000 te, @ RM 20.-	RM	20,000.-
2.	75 labor shifts, @ RM 12.-	RM	900.-
3.	Power $\frac{25 \times 1,440,000}{1000}$ = 36,000 kwh @ RM 0.02	RM	720.-
4.	Circulating water $\frac{17 \times 1,440,000}{1000}$ = 25,000 m^3 @ RM 0.02	RM	500.-
5.	Make-up water, 10% of circulating water = 2,500 m^3 @ RM 0.02	RM	50.-
6.	Feed water, 1,800 m^3, @ RM 0.20	RM	360.-
7.	Steam, 3 atm, 1,700 te @ RM 1.50	RM	2,550.-
8.	Amortization, interest and maintenance 12 + 5 = 17% on 12,150,000	RM	5,750
	Total expense, per day	RM	30,830.-

<u>Receipts per day:</u>

1.	Steam, 16 atm, 1,440 te,	@ RM 3.- =	4,320
2.	Steam, 3 atm, 325 te,	@ RM 1.50 =	487

Total receipts per day = RM. 4,807.-

Operating costs, incl. capital service and maintenance RM 26,023.-

Operating costs/nm^3 water gas $\frac{2,602,300}{1,440,000}$ = 1.818 pfg

Operating costs/nm^3 $CO + H_2$: $\frac{1.818}{87.22}$ = 2.075 Pfg

Rheinpreussen uses RM 17.50 as the price per te of coke. This reduces the operating costs per day to RM 23,523, or

per nm^3 water gas to Pfg 1.635, and
per nm^3 $CO + H_2$ Pfg 1.875

II. Estimation of operating costs of a gasification plant for fines with 5 producer units of 12,000 nm^3 hourly capacity = 60,000 nm^3 water gas per hour, or 1,440,000 nm^3 water gas per day.

Expenses per day:

1.	Coal $\frac{1,440,000}{2,700}$ = 533 te,	@ RM 14.-	RM 7,460.-
2.	Oxygen, 1,440,000 x 0.17 = 245,000 nm^3	@ 0.025	6,125.-
3.	51 labor days, @	@ RM 12.-	612.-
4.	Power $\frac{25 \times 1,440,000}{1000}$ = 36,000 kwh	@ 0.02	720.-
5.	Circulating water $\frac{17 \times 1,440,000}{1000}$ = 25,000 m^3	@ 0.02	500.-
6.	Make-up water, 10% of circ. water	@ 0.02	50.-
7.	Feed water, 650 m^3	@ 0.20	130.-
8.	Fuel gas, 485 x 10^6 kcal	@ 4.50	2,183.-
9.	Steam, 3 atm, 576 te	@ 1.50	865.-
10.	Amortization, interest and maintenance, 12 + 2% = 14% on RM 9,000,000		3,450.-
	Total expense, per day		RM 22,095.-

<u>Receipt per day:</u> (No. carried from Pg.1) 22,095.-

Steam, 16 atm, 625 te	@ RM 3.-	1.875.-
Operating costs, includ. capital services and maintenance.		20,220.-

Operating costs per nm^3 water gas $\frac{2.022.000}{1,440,000}$ = Pfg 1,405

Operating costs per nm^3 $CO + H_2$ $\frac{1.405}{83}$ = Pfg 1.69

III. Estimate of operating costs of a gas reforming plant for 10,000 nm^3 coke oven gas = 16,000 nm^3 reformed gas per hour = 356,000 nm^3 reformed gas per day.

<u>Expenses per day</u>

1.	Coke oven gas, 240,000 nm^3	@ RM 0.017	RM 4,080
2.	Fuel gas, 232×10^6 kcal	@ 4.50	1,045
3.	9 man-days	@ 12.-	108
4.	Power, $\frac{15 \times 386.500}{1000}$ = 5800 kwh	@ 0.02	116
5.	Circulating water, $\frac{18 \times 386.500}{1000}$ = 7000 m^3	@ 0.02	140
6.	Make-up water = 10% of circ. water	@ 0.02	14
7.	Steam, 3 atm. 175 te	@ 1.50	263.
8.	Amortization, interest and maintenance 12 + 2% on 2,000,000 =		767.-
	Operating costs, including capital services and maintenance		6,533.-

Operating costs per nm^3 reformed gas, $\frac{653.300}{386,500}$ = Pfg. 1.69

Operating costs per nm^3 $CO + H_2$ $\frac{1.690}{85.36} \times 100$ = 1.98

Rheinpreussen figures the price of coke oven gas of 1.3 Pfg per nm^3, corresponding to 0.336 Pfg per 1000 kcal.

Should this value be used in the estimate of operating costs, for coke oven gas or fuel gas, the operating costs per day, including capital services and maintenance will amount to RM 5,308.-.

$$\text{or, per } nm^3 \text{ of reformed gas } \frac{530{,}800}{385{,}500} = 1.375, \text{ Pfg.}$$

$$\text{per } nm^3 \text{ of } CO - H_2 \quad \frac{1.375}{83.56} = \text{Pfg } 1.61$$

The operating costs in the above computations were determined with the inclusion of capital services and maintenance, and were:

per nm^3 water gas from coke
per nm^3 water gas from powdered coal
per nm^3 reformed coke oven gas.

Below are given the costs of synthesis gas obtained by mixing of water gas obtained from coke or from powdered coal with the reformed gas from coke oven gas.

A). <u>Synthesis gas with water gas from coke:</u>

	65% water gas +	35% reformed gas =	100% synthesis gas
CO_2	7.39%	5.12%	6.50%
O_2	0.17	0.12	0.15
CO	36.69	14.60	29.00
H_2	50.53	70.76	58.00
CH_4	0.37	1.90	1.00
N_2	4.85	7.50	5.35

With a coke price of RM 20.- per te and a coke oven gas price of Pfg 1.7 per nm^3, the cost of synthesis gas will figure to:

$$0.65 \times 1.81 + 0.35 \times 1.69 = \text{Pfg } 1.769 \text{ per } nm^3 = \text{Pfg } 2.035 \text{ per } nm^3\ CO + H_2$$

With a coke price of RM 17.50/te and a coke oven gas price of Pfg. 1.3/nm^3, the cost of the synthesis gas will figure to:

$$0.65 \times 1.635 + 0.35 \times 1.375 = \text{Pfg. } 1.543/nm^3 = 1.775/nm^3\ CO + H_2$$

B). Synthesis gas from powdered bituminous coal.

	70% water gas +	30% reformed gas =	100% synthesis gas
CO_2	16.0%	5.12%	12.70%
O_2	-	0.12	0.04
CO	34.0	14.60	28.00
H_2	48.0	70.76	56.00
CH_4	-	1.90	0.66
N_2	1.0	7.50	2.70

With the price of coke oven gas of Pfg. 1.7 nm^3, the cost of synthesis gas will figure to

$0.70 \times 1.405 + 0.30 \times 1.690 =$ Pfg $1.487/nm^3 =$
" $1.770/nm^3$ per nm^3 $CO+H_2$

With the coke oven gas at Pfg $1.3/nm^3$, the synthesis gas figures to

$0.70 \times 1.405 + 0.30 \times 1.375 =$ Pfg $1.392/nm^3 =$
" $1.660/nm^3$ per nm^3 $CO+H_2$

IV. Estimation of powdered coal producer installation with 5 producer units of 12,000 nm^3 hourly capacity for <u>the direct production of synthesis gas</u> with the proportion of CO : H_2 = 1.2, total 60,000 nm^3 synthesis gas per hour, or 1,440,000 nm^3 synthesis gas per day.

<u>Expense per day:</u>

1.	Coal $\frac{1.440.000}{2800} = 514$ te	@ RM 14.-	RM 7,200
2.	Oxygen 1,440,000 x 0.16 = 230,000 nm^3	@ 0.025	5,750.-
3.	51 man-days	@12.-	612.-
4.	Power $\frac{25 \times 1.440.000}{1000} = 36{,}000$ kwh	@ 0.02	720.-
5.	Circulating water $\frac{17 \times 1.440.000}{1000} = 25{,}000\ m^3$	@ 0.02	500.-
6.	Make up water, 10% of circul. water	@ 0.02	50.-
7.	Feed water, 720 m^3	@ 0.20	144.-
8.	Fuel gas, 547×10^6 kcal	@ 4.50	2,460.-
9.	Steam, 3 atm, 662 te	@ 1.50	994.-
10.	Amortization, interest, maintenance, 12 + 2 = 14% on RM 9,000,000		3,450.-
	Total expense per day		RM 21,880.-

<u>Receipts per day:</u>

Steam, 16 atm., 700 te	@ 3.00	2,100.-

Operating costs, including capital services and maintenance 19,780.-

Operating costs per nm^3 synthesis gas $\frac{1,978,000}{1,440,000}$ = Pfg. 1.370

Composition of synthesis gas:

CO_2	18%
CO	27%
H_2	54%
N_2	1%

Operating costs per nm^3 $CO + H_2 = \frac{1.370}{81}$ = Pfg. 1.690

W. M. Sternberg

Newman

TOM Reel 126
Frame 000843

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-155

11/14/46

August 12, 1941

WORKING UP OF OIL CONTAINING RESIDUES
(KILN OPERATIONS)

We informed you in our letter of July 11, 1941, that the operating procedure suggested to you by the Brownkohle-Benzin A.G. is not unknown to us. We have worked for many years on the utilization of oil-containing residues, such as obtained in the pressure hydrogenation, and have acquired some patent rights in our country and abroad. The patent application, German, J 61,317 VI/10a (18/01), has been published, in which the hydrogenation residue is mixed with carbonaceous materials and coked at a low temperature. We believe your powdered brown or bituminous coal addition to fall within our specifications for carbonaceous materials. This application mentions specifically that coal fines, grude residue, etc., may be added. In three supplementary applications, (J 62,603 VI/10a, J 63,353 VI/10a and J 64,386 VI/10a) specific operating conditions have been described for the above application.

We wish to inform you as follows with regard to your other questions:

to 1): Oil residues are not destroyed in our experimental operations. The larger quantities of oil residues obtained in our hydrogenation experiments are worked up on an experimental scale. The different residues obtained in varying amounts and of varying composition in test runs are also worked up experimentally.

to 2). In general, hydrogenation residues and other residues are centrifuged, and the fuge residues are kilned, with practically the recovery of all the original oil. The kiln residues actually contain only up to 3% benzol soluble constituents. The working up of the residues is protected by special means, e.g. the DRP 550,157; 630,965; 656,364; 675,957; 699,707.

I.G. Farbenindustrie Aktiengesellschaft.

W M Sternberg

Newman

TOM Reel 162 U. S. Bureau of Mines T-156

Pps. 898-900 Hydro. Demon. Plant Div. KCBraun

Data for Comparing Hydrogenation with Low Temperature Carbonization (L.T.C.) of Bituminous Coal.

By Dr. Fahr, Lu. 24 May 1943

(A)	(B)	(C)	(D)
Coal, 7000 WE/Kg, 8% H_2O	Lurgi Rinsing Gas Carbonis. For Upper Silesia	B.T. Carbonization For Upper Silesia	Coke Plant For Upper Silesia
Coke Yield 7000 WE, 5% H_2O			
Coke, 0% H_2O, based on TKO	72.5%	75.0%	71.0%
Coke, 5% H_2O, based on TKS	70.2%	72.5%	68.7%
Particle Size, 0-5 mm	4.7%	2.8%	5.0%
5-20 mm	13.0%	25.6%	10.0%
20 mm	82.3%	71.6%	85.0%
Volatile Constituents	7.0%	9.0%	7.0%
Tar Yield			
Tar } based on TKO	9.2%	8.5%	4.0%
L.T.C. Gasoline } based on TKO	0.9%	0.75%	1.0%
C_3+C_4 } based on TKO	– (1)	0.55%	0.0%
	10.1%	9.8%	5.0%
Tar } based on TKS	8.5%	7.8%	3.7%
L.T.C. Gasoline } based on TKS	0.8%	0.69%	0.92%
C_3+C_4 } based on TKS	– (1)	0.51%	0.0%
	9.3%	9.0%	4.62%
L.T.C. Gas			
Nm^3/ton TKO	495	81	320
WE/Nm^3	2000	6200	1400
WE/Mill.t. TKO	1000×10^9	500×10^9	1410×10^9
WE/Mill.t. TKS	920×10^9	460×10^9	1300×10^9

(1) 0.4% not recoverable, based on TKO

(1)	(2)	(3)	(4)
Required to Heat:			
Kiln Drum			
WE/Mill. t. TKO	455 x 10⁹	500 x 10⁹	700 x 10⁹
WE/Mill. t. TKS	420 x 10⁹	450 x 10⁹	645 x 10⁹
Retorts		The rich gas is used as long distance gas and the weak gas is used to heat the kiln drum.	
WE/Mill. t. TKO	345 x 10⁹		
WE/Mill. t. TKS	370 x 10⁹		
Coal for [illegible]			
Production			
[illegible] 1 Mill. t. TKO		100,000 t/ann.	240,000 t/ann.
Based on 1 Mill. t. TKS		98,000 t/ann.	190,000 t/ann.
		+ 8% tar based on coal	+ 8% tar based on coal
Power Requirement			
KWh/h for 1 Mill. t. TKO	2100	3200	5000
Steam Requirement			
t/h for 1 Mill. t. TKO	7.8 L.P.	10 L.P.	19 L.P.
	3.5 H.P.	0 H.P.	0 H.P.
Coal for Current & Steam			
for 1 Mill. t. TKO	12,900 t/ann.	12,000 t/ann.	20000 t/ann.

(A)	(B)	(C)	(D)
Tar Processing		**L.T.C. Gasoline = 95%**	
		Refined auto gasoline	
		L.T.C. Tar = 72.5% Fuel Oil	Pitch = 58.8%
		7.0% Phenols	Oil = 35.0%
		15.0% Pitch	H_2O-Loss = 6.2%
		5.5% Loss	
		100.0%	100.0%
Power for Tar Processing)) 1 Mill. t.		1720 KW))30000 tons	100 KW
Steam for Tar Processing) TKO		29 tons)	2 t/h
Power for Tar Processing) 1 Mill. t.) TKS		1582 KW	92.0 KW
Steam for Tar Processing)		17.5 tons	1.8 t/h
Fuel Gas Requirement, 1 Mill. t. TKO			1×10^6 WE/h
Coal for Fuel Gas Production			1740 tons

(A)	(B)		(C)		(D)	
	1 Mil.t.TKO	1 Mil.t.TKS	1 Mil.t.TKO	1 Mil.t.TKS	1 Mil.t.TKO	1 Mil.t.TKS
Iron Required:						
for L.T.C.	18,800 t	17,300 t	24,000 t	22,000 t	21,000 t	19,300 t
for Power Plant (Proportion)	760 t	700 t	810 t	750 t	540 t	500 t
for Fuel Gas Prod.			2,500 t	2,300 t	4,130 t	3,800 t
for Phenol Extraction from Waste Waters			330 t	300 t		
for C_3 & C_4 Extraction and for Power for C_3 & C_4 Extraction			2,340 t	2,150 t		
	19,560 t	18,000 t	29,980 t	27,500 t	25,670 t	23,600 t
for Tar Conversion			3,820 t	3,510 t	1,000 t	900 t
for Power Plant (Proportion)			540 t	500 t	100 t	90 t
			34,340 t	31,510 t	26,770 t	24,590 t

TOM Reel 69
Pps. 53-56
Pgs & 1122-26
Tom Reel 164

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-157
Trld KCBraun/jw
11/8/46

Comparison of Coal and Iron Requirements Between Bituminous Coal Hydrogenation and Low Temperature Carbonization (L.T.C.) followed by Hydrogenation

Berlin, 21 April 1943

Plants Producing:

100,000 tons/ann Aviation Gasoline) by hydrogenation
25,000 tons/ann L.P.G. (Liq. Petrol. Gas))

and

100,000 tons/ann Aviation Gasoline) by L.T.C. followed
15,000 tons/ann L.P.G. by hydrogenation of
912,000 tons/ann Excess L.T.C. Coke) the L.T.C. tar.

1.) Specific Data on L.T.C. (Lurgi - Rinsing Gas Process)
(Lurgi - (Spülgasverfahren)

L.T.C. coke yield	72.5%
Tar Yield, based on coal input	10.0%
Aviation gasol. yield, based on L.T.C. tar	67.5%
Fuel gas required/kg. coal feed	200 WE
Surplus gas/kg. coal feed	400 WE
Power coal required for 1 Mill. t. coal	25,000 t
Coal heat value/kg.	7,000 WE
Coke required/1000 m^3 H_2	0.75 t

2.) Specific Data for Hydrogenation of L.T.C. Tar

H_2 Consumption	2100 m^3/t av. gasol.
Power Coal	0.95 t/t av. gasol.
H_2 required (based on hydro. coal)	3200 m^3/t av. gasol.
Power coal for coal hydrogenation	1.0 t/t av. gasol.

3.) Total Coal Requirement for L.T.C. of Coal and Hydrogenation of the L.T.C. Tar

From the above figures for H_2 requirement, tar yield and L.T.C. coke yield, we derive an input of 1,480,000 tons of L.T.C. coal. The power coal requirement for L T.C. = 37,000 tons and 95,000 tons for hydrogenation, or a combined coal requirement for L.T.C. and hydrogenation of 1,612,000 tons. From these 1,612,000 tons converted to L.T.C. and hydrogenation corresponding to the coal equivalent of the various final products, we derive a coal requirement of 482,000 tons for tar hydrogenation and 1,130,000 tons for surplus coke and gas, which are available to other consumers. Therefore, about 30% of the total coal is used for aviation gasoline and L.P.G. and about 70% for surplus coke and gas.

Subdividing the 482,000 tons of coal for tar hydrogenation, we get the following coal equivalents:

for tar	202,000 t	
for coke and H_2	174,000 t	
for power coal	106,000 t =	482,000 t

Subdividing the residual coal we get these results.

for surplus coke	1,034,500 t	
for surplus gas	95,500 t =	1,130,000 t
	Total	1,612,000 t

These figures contain 26,000 tons of power coal proportionally charged to the last two products.

From these residual coal figures, we get 912,000 tons surplus L T.C. coal and 592 x 10^9 WE surplus gas for other consumers.

The conversion of the coal input quantities to the L.T.C. products, tar, coke and surplus gas, was based on the calory content.

If the sum of the calories equals 100, we get the following proportion of the coal input.

for tar	13.7%
for coke	80.0%
for surplus gas	6.3%
Total	100.0%

which corresponds to a basic coal value

for tar	208,000 t
for L.T.C. coke	1,213,500 t
for long distance gas	95,500 t
Total	1,517,000 t

From the coal value of 1,213,500 t for L.T.C. coke, 179,000 t go into hydrogenation for H_2 (coal value). The coal value for the surplus coke is, therefore, 1,034,500 t.

Since 11,000 t power coal from L.T.C. are contained in hydrogenation, these were subtracted proportionally from tar and coke and added to the power coal.

4.) Hydrogenation of Bituminous Coal

Due to the higher coke yield compared to the assumption of March 24, the L.T.C. coal is reduced from 320,000 t to 306,000 t, while the tar yield is increased from 29,000 t to 30,600 t, so that the H_2 is reduced from 300 million m^3 to 297 million m^3. The coke is proportionally reduced from 225,000 to 222,000 t. The heat contained in the surplus gas, with a specific value of 400 WE/kg coal, = 122.2 x 10^9 WE. The power coal requirement for the small L.T.C. was at first calculated proportionally, but, because of the smaller plant, was increased from 7,700 t to 10,000 t. This results in an increase in the total power coal requirement for hydrogenation to 110,000 t, and the bituminous coal requirement is reduced to 584,000 t. Subdividing the coal in proportion to the calorific value results in the following values

for fuel	554,000 t	(444,000 for av.gasol.) (110,000 for fuel gas.)
for surplus gas	30,000 t	
Total	584,000 t	

5.) Iron Requirement

The iron requirement for tar hydrogenation was increased to 60,000 t, while that for coal hydrogenation remained unchanged at 100,000 t. The railroad installations were eliminated in both cases. The iron requirement for the complete Lurgi rinsing gas L.T.C. plant has been set at 15,000 t/million t of coal thruput in combined agreement with the Mineralölbaugesellschaft and Dr. Oetken. Consequently, 22,200 t of iron are required for the large L.T.C. plant and 4,600 t for the small plant, wherein 50% of the proportional requirement was added to the latter. To this must be added 4200 t of iron for the power plant for the large L.T.C. plant and 900 t for the smaller.

As far as the iron requirement in mining is concerned, it was assumed that the combined L.T.C. and hydrogenation would require only 30% of the total coal, while the remaining 70% are available for general use, for which this quantity is required, regardless of whether it is used directly or in bituminous coal hydrogenation as a coal conversion process. On the other hand, bituminous coal hydrogenation is a new consumer, who must also be supplied with coal and who must, therefore, be charged with the iron required by the mines in providing additional production facilities. A specific iron requirement of 25,000 t/million t of additional coal production was assumed. This results, in the case of bituminous coal hydrogenation, in 14,600 t of iron, and in the case of combined L.T.C. and hydrogenation, in 12,100 t.

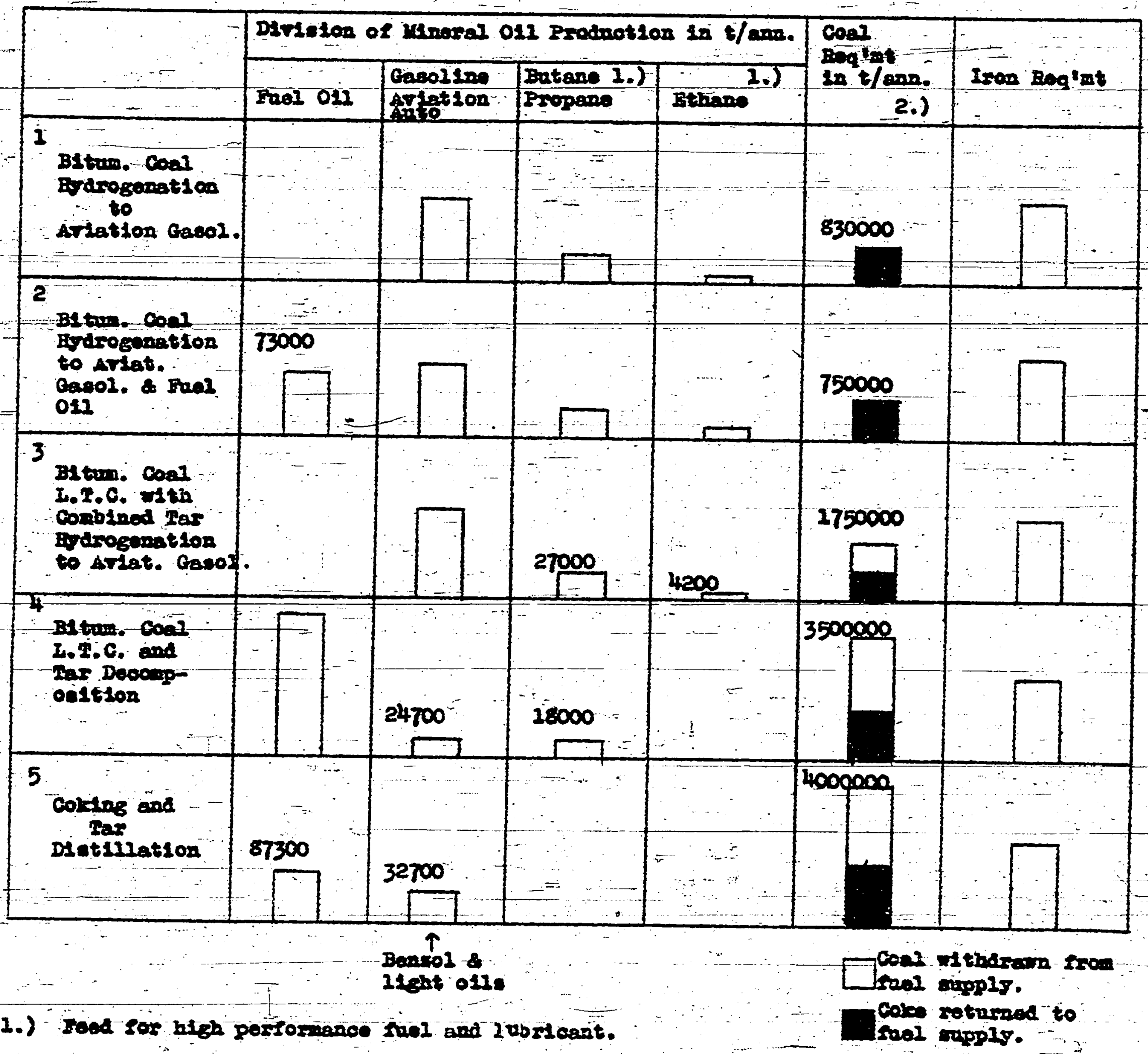
IRON REQUIREMENT

L.T.C. & Tar Hydrogenation		
Mining (Proportion)	12,100 t	
L.T.C.	26,400 t	of which 30% for av. gasol. and ~~fuel gas~~ L.P.G. 6660 t 1260 t 7920 t and 70% for surplus coke and gas 15540 t 2940 t 18480 t
Hydrogenation	62,000 t	
Total	100,500 t	

Bitum. Coal Hydrogenation	
Mining	14,600 t
L.T.C.	7,500 t
Power Plant	1,200 t
Hydrogenation	100,000 t
Total	123,300 t

Differences Between Various Bitum. Coal Conversion Processes.

Mineral Oil Production Per 100,000 t Iron Requirement.
(The height of columns corresponds to the heat content of the products)
(Quantities in tons)



1.) Feed for high performance fuel and lubricant.

2.) Diagram reduced in ratio 1:125 compared to mineral oil production.

Diagram 1.

Differences Between Various Bitum. Coal Conversion Processes.

Iron & Coal Requirement for a Mineral Oil Production of 100,000 t/ann.
(The height of columns corresponds to the heat content of the products)
(Quantities in tons)

	Division of Mineral Oil Production of 100,000 t/ann.				Coal Req'mt for the prod. of 100000 t/ann. min. oil, in t/ann.	Iron Req'mt for a plant producing 100,000 t/ann mineral oil.
	Fuel Oil	Gasoline Aviation Auto	Butane 1) Propane	Ethane 1)		
1 Bitum. Coal Hydrogenation to Aviation Gasol.		72400	22400	1200	450000	72900
2 Bitum. Coal Hydrogenation to Aviation Gasoline & Fuel Oil	34100	41800	15000	1000	400000	52000
3 Bitum. Coal L.T.C. with Combined Tar Hydrogenation to Aviation Gasoline		77300	19900	1000	1248000	71000
4 Bitum. Coal L.T.C. and Tar Decomposition	84200	8100	8000		1530000	43000
5 Coking and Tar Distillation	67300	32700			4000000	100000

1) Feed for high performance fuel and lubricant.

2) Diagram reduced in ratio 1:125 compared to mineral oil production.

☐ Coal withdrawn from fuel supply.

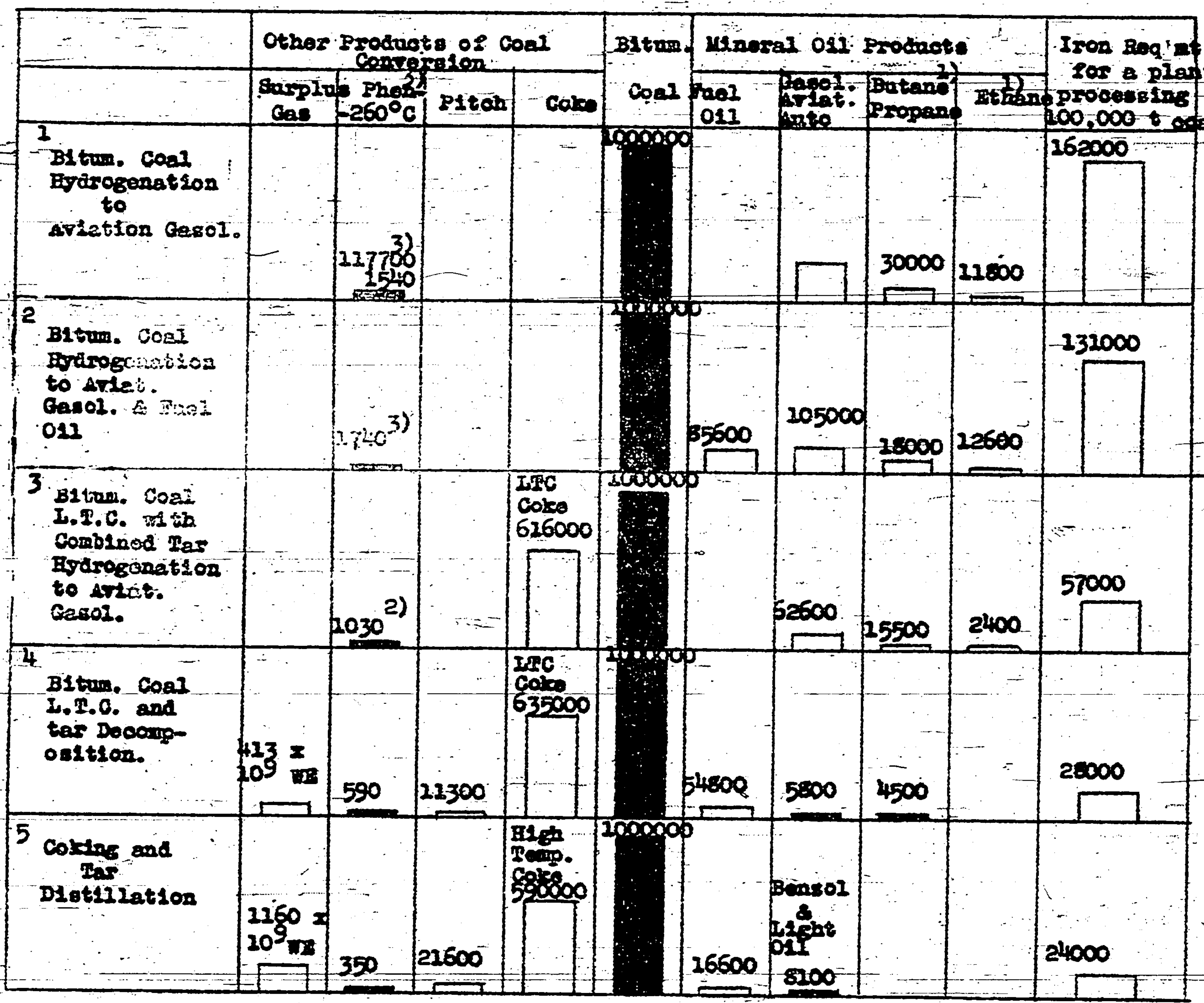
■ Coke returned to fuel supply.

Diagram 2.

Differences Between Various Bitum. Coal Conversion Process

(The height of columns corresponds to the heat content of the products)

(Quantities in tons)



	Other Products of Coal Conversion				Bitum.	Mineral Oil Products				Iron Req'mt for a plant processing 100,000 t coal/a
	Surplus Gas	Phen. 2) -260°C	Pitch	Coke	Coal	Fuel Oil	Gasol. Aviat. Auto	Butane 1) Propane	Ethane 1)	
1 Bitum. Coal Hydrogenation to Aviation Gasol.		3) 117700 1540			1000000			30000	11800	162000
2 Bitum. Coal Hydrogenation to Aviat. Gasol. & Fuel Oil		1740 3)			1000000	55600	105000	18000	12600	131000
3 Bitum. Coal L.T.C. with Combined Tar Hydrogenation to Aviat. Gasol.		1030 2)		LTC Coke 616000	1000000		52600	15500	2400	57000
4 Bitum. Coal L.T.C. and tar Decomposition.	413 x 10^9 WE	590	11300	LTC Coke 635000	1000000	54800	5800	4500		28000
5 Coking and Tar Distillation	1160 x 10^9 WE	350	21600	High Temp. Coke 590000	1000000	16600	Bensol & Light Oil 8100			24000

1) Feed for high performance fuel and lubricant.

2) Phenol prod. may be increased at the expense of min oil prod.

3) When abstracting higher phenols quantities increase abt. 2-3 times.

Diagram 3.

Newman

TOM Reel 126
Pgs. 425-27

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-158

KCBraun

VELOCITY IN PREHEATERS @ 600 ATM.

By Oettinger, Lu, 22 Nov 1938

The velocities of flow for gas compiled in the report of 10 November 1938 are valid for a preheater temperature of 24.5 MV = 450° C, without considering the space taken up by the coal paste.

For an average temperature of 14 MV = 270° C in the preheater, the velocities of flow for gas are reduced 25%. See table.

		Linear Velocity in Preheater cm/sec 3)		
Converter Size	Coal	Preheater Temperature		Paste
		270° C = 14 MV Gas 1)	450°C = 24.5 MV Gas 1)	
1.5 Liter	Rhenish Brown Coal	6.7	9	2
10 "	"	33	44	6.1
1 4-Stage Stall at Rheinbraun	"	975 2)	1300 2)	195
1.5 Liter	Bitum. Coal	7.5	10	1.5
10 "	" "	23	31	4.2
100 "	" "	73 2)	97 2)	13
1 3-Stage Stall at Nordstern	" "	580 2)	770 2)	96
1 4-Stage Stall at Nordstern	" "	770 2)	1030 2)	128

1) Without considering the space taken up by the coal paste.

2) Assumption: All gas passes thru preheater.
For example, if only half the gas is run thru the preheater the given values are reduced 50%.

3) The original shows these same figures as representing velocities in cm sec $^{-1}$,-?

Essen, June 15, 1942

DIRECT PRODUCTION OF SYNTHESIS GAS FROM POWDERED FUEL.

I wish to make the following additions to your letter of 12-th ult.

I have further discussed with the Generaldirektor Kost the topics covered in our conference.

We agreed, that a powdered coal fuel gas installation on a bituminous coal basis as a first unit is less desirable to build than a powdered coal synthesis gas producer, i.e. with oxygen gasification. The operating experience at the Rheinpreussen pilot plant installations have repeatedly confirmed the ease and certainty of gasification with oxygen, and the risk of putting up such a unit is small and no difficulties need be expected.

Mr. Kellner (of the "Hermann Göring" works) has told me in the presence of Mr. Kost that the construction of powdered gas producer installation is very important to him, in particular in Watenstedt, because of the insufficient amount of gas available for heating the converters after the completion of the second addition. He had no intention, however, of building an air gas unit, and would prefer the 70 - 80% oxygen, to produce higher heating values. If needed, the gas then could also be sent to the steel works.

I told Mr. Kost in this connection that the proposed installation for the Pattberg mines should be reconsidered to decide, whether the 100 to powdered coal producer installation would not be better located at the gas works instead of the Pattberg mines, and whether a correspondingly large oxygen installation should be built at the same time. Computations have shown that the operating costs of such an installation for the production of synthesis gas, including interest and amortization, are considerably more favorable than the costs of his present water gas installation, especially because of the ever increasing break-downs and costs of repairs. According to his statement, the operating costs per m^3 of water gas amounted to but 2.2 Pfg at the gas works, and the increased costs of repairs to be expected could not have been included there. Mixed synthesis gas cost of 2.5 Pfg would not be too high.

In reply to Mr. Kost's question on the operating costs of a synthesis gas installation with an oxygen plant, I have quoted him the price of 1.6 - 1.7 Pfg, but without obligating myself, and that the cost of erection of the required oxygen unit would be between RM 500,000 and 600,000. Mr. K. appeared interested. He suggested, however, that in such a case we would be unable to operate with air-gas, but I replied that we would include a second air gasifier in this installation to permit testing out both processes in one installation. There would be an incentive for Rhenish Prussia, since he always informed us that he could get but 1.2 Pfg/m^3 for Ruhr coke oven gas, and that the powdered coal air gasification unit at the Pattberg mines would always remain an auxiliary operation, while a synthesis gas producer built at the gas company would be a profitable business. The latter might also be considered as a reserve water gas producer, if the synthesis gas production be limited to 8000 m^3 per hour. As long as operations were carried on with air gas, this fuel gas could be supplied to the coke ovens of the Mine V (Rinselmann ovens) instead of the new Pattberg mines ovens. Mr. Kost thought it plausible. A pipe line is available, no additional costs would be incurred, and the Hinselmann ovens had been built as association ovens.

It would be advisable to meet again within the next weeks. Mr. Kost will by then have informed us, whether your letter of the 12th ult. for a powdered coal unit at the Pattberg mines be considered, or whether it be altered in the above sense. Mr. K. requests data on the costs of installation and operation and the space requirements, because the available space at the gas works is very limited, and Mr. K. would like to keep the construction of the water gas producers as a safety measure.

W. M. Sternberg.

Newman

TOM Reel 163
Pps. 125-27

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T 160
Trld. KCBraun/JW
11/12/46

Residue Processing

By Gieg & Rank. Lu. 15 Oct. 1942

In the first stage of coal hydrogenation, the liquid phase, light and heavy oils are produced, the latter containing the non-liquefied parts of the coal, the coal ash and catalyst substances. It is the problem of residue processing to extract from these so-called let-downs oils free of insolubles, which can be used as pasting oils for the coal. The object is to obtain a maximum oil extraction and a complete removal of the solids, because, if the latter are returned to the process, they needlessly burden the reaction space. The difficulties in residue processing are due to the high molecular oil particles, the so-called asphalts and resins, which, under certain conditions, produce a sticky, plastic hydrogenation residue. These substances represent the transition between oil and solid coal and, on their return to the hydrogenation process, are transformed into light oil particles.

For the separation of solids in residue processing, various processes may be used, such as filtration, centrifuging, extraction, distillation, or low temperature carbonization (L.T.C.) The apparently simplest method of L.T.C. of the let-down causes too great oil losses, because the asphalts and resins contained in the oil produce too low a yield, while greater oil yields are obtained by returning them to the hydrogenation process. Mechanical processing of the let-down, such as filtration or centrifuging is, therefore, the most suitable, since a maximum oil yield may be expected from it, because only a small portion of the let-down oil contained in the filtration or centrifuge residue must be thermally treated. Filtering the let-down would yield pasting oils entirely free of solids, if it were not for the low filter performances to date.

Centrifuges are, therefore, used in mass production for separating the solids at the present time, foregoing return oils entirely free of solids. The most satisfactory has been the Laval, which delivers liquid centrifuge residue and centrifuge oil continuously. By comparison, the semi-continuous centrifuges, like the Haubold and the Reine, deliver plastic residues difficult to handle.

Various apparatus have been developed for low temperature carbonization of centrifuge residues. Of these, two types have been accepted by industry, the spiral screw kiln, primarily for brown coal, and the ball kiln for bituminous coal hydrogenation. Both types are based on the idea of carbonization in thin layers, which assures the greatest oil yield.

Recently, efforts have also been made to produce electrode carbon and briquette binder as by-products of the liquid coal phase.

To produce briquette binders, the let-down or centrifuge residue is distilled to the desired consistency, for which distillation columns or the L.T.C. apparatus proper may be used.

Filtrates from hydrogenation let down are used for making electrode coke.

In the liquid phase hydrogenation of tar and petroleum residues the residue processing is simplified considerably by the fact that the solid substances to be removed are primarily inorganic, largely used catalyst, and small in quantity. The small let-down masses are generally carbonized (L.T.C.) directly and produce a good yield because of the low asphalt content. The residues may also be used in concentrated form for coal briquette binder. In Pölitz, tar and petroleum let-down is concentrated in vacuum and the residue is burned in the boiler house.

The L.T.C. residue contains much ash, including, in some cases, volatile substances. It can, therefore, be used as fuel only with a specially constructed combustion chamber and as an addition to inferior fuels. At times it contains so much iron that it can also be sold to blast furnace plants.

Newman

TOM Reel 5 U. S. Bureau of Mines T-161

Pps. 189-96 Hydro. Demon. Plant Div. KCBraun

Experiments to Increase the Centrifugal Effect in Centrifuging Let-Down.

By Dr. V. Dorp. Gelsenkirchen - Horst,
14 October 1942.

It is very difficult to centrifuge the letdown in the form produced in the coal stalls. It is, therefore, diluted to about 16.5% solids content with a thinning oil and this mixture is then centrifuged in a continuous process in Laval Centrifuges. Our plant produces an oil with about 10-12% solids and a residue with 35-39% solids by this method. Since the oil is again used as pasting oil, the solid substance is ballast for the hydrogenation and the object is to keep the solids content of the oil as low as possible. On the other hand, the solids content of the residue should be as high as possible, in the most favorable case at the limit of its pumpability, in order to keep the oil losses in carbonization (L.T.C.) as low as possible. The difficulties in separating the letdown into oil and solids by centrifuging are based on the fact that the letdown is not simply a suspension of solids in an oily medium but that it represents a colloidal system. This colloidal letdown is further complicated by the fact that a component of this system, namely, the carrier substance "asphalt", is itself a colloid. In order, therefore, to obtain a maximum solids content in the centrifuge residue and an oil with a minimum of solids, it is necessary to destroy the colloidal structure. This may be done in two ways:

1). by dilution with oils acting as solvents, by reducing the viscosity of the centrifuge mixture, by dissolving the carrier substance "asphalt", by increasing the difference in specific gravity of solids and oil, and possibly in the case of selective solvents, by causing the colloidal system to flocculate.

2). by adding electrolytes and the consequent change in the P_H-value, the colloidal structure is destroyed, because even the most stable colloidal solutions are stable only in a very definite, narrowly limited P_H range.

Making the Experiments

In the following experiments, we first tried to determine and to increase the centrifugal effect by the use of different thinning oils, according to (1.), using an intermittent, electrically driven, laboratory centrifuge. Several lengthy trial experiments at room temperature indicated little or no success. In order to provide actual operating conditions, the experiments would, therefore, have to be conducted at higher temperatures. Because it was impossible to heat the whole centrifuge, the experiments described herein were conducted in the following manner:

The centrifuge mixture was heated to 120° C. in the centrifuge vessels, then centrifuged for five minutes at 3500 RPM. The temperature of the product had then dropped to 60° C. From the uppermost layer of oil one gram of oil was skimmed off in order to determine its solids content. The solids values so determined are compiled in the following tables. A centrifuge mixture containing 16.5% solids, corresponding to actual operating conditions, from a letdown containing 23% solids and 1% asphalt, was used. Only products available in large quantities in the plant were used as thinning oils, such as:

1. A-heavy oil (normal thinning oil), start boil 338° C., over 360° C.: 73%, aniline point - 28.7° C.
2. A-middle oil, start boil 188° C., end boil 345° C., aniline point - 23.4° C.
3. B-middle oil, start boil 164° C., end boil 305° C., aniline point + 47.6° C.
4. L.T.C. oil I, start boil 354° C., below 360° C: 98%, aniline point - 15.6° C.
5. L.T.C. oil II, start boil 214° C., below 360° C: 8%, aniline point - 12.1° C.
6. Tar oil, start boil 203° C., below 360° C: 11%, aniline point - 29.7° C.
7. A-gasoline, start boil 47° C., end boil 160° C., aniline point + 54.1° C.

With these solvents alone, as well as with mixtures of these individual solvents and A-heavy oil in the proportion 1:1, the letdown was now diluted to a centrifuge mixture containing 16.5% solids.

Results of Experiments

The results obtained in these experiments are compiled in Table I, below.

TABLE I

Centrifuge Mixture of Letdown and:	Solids before Centrifuging, %	Solids after Centrifuging, %
A-heavy oil	16.5	11.8
A-middle oil	16.4	8.6
A-middle oil 50% + A-heavy oil 50%	16.6	10.9
B-middle oil	16.2	1.7
B-middle oil 50% + A-heavy oil 50%	16.6	7.5
L.T.C. Oil I	16.0	13.5
L.T.C. Oil I 50% + A-heavy oil 50%	16.2	14.0
L.T.C. Oil II	16.4	2.3
L.T.C. Oil II 50 + A-heavy oil 50%	16.6	9.5
Tar Oil	16.7	2.8
Tar Oil 50% + A-heavy oil 50%	16.6	6.8
X) A-gasoline	16.3	11.2
X) A-gasoline 50% + A-heavy oil 50%	16.7	9.2

The column "Solids after centrifuging" represents the solids content of the top layer of the centrifuge oil. Due to the nature of the centrifuge, the oil can, unfortunately, not be separated from the solids. The given values are averages of at least two experiments. Because of the lowboiling point of the A-gasoline, the A-gasoline centrifuge mixtures could not be heated to 120° C., see X). These 2 experiments were made at 70° C. and can, therefore, not be directly compared to the other experiments.

In looking at the results, it is apparent that B-middle oil, A-gasoline, L.T.C. Oil II, Tar oil, A-middle oil, A-heavy oil and

L.T.C. oil I are, in the order named, progressively less effective. The most effective is B-middle oil, with 1.7 to 16.2. It is significant also, that the aniline points of the solvents used, with the exception of the tar oil, drop in the same order as the centrifugal effects deteriorate. It may be seen, therefore, that the centrifugal effects improve with a rising aniline point, that is, with increasing paraffin content, of the solvent. To show this fact more clearly, mixtures with purely paraffinic or purely aromatic solvents were now tried, such as:

1. Paraffin oil, aniline point + 106.5° C.
2. Benzol, start boil 80° C., below 100° C.: 5% aniline point -17.5° C.
3. Heptane, start boil 97° C., end boil 105° C., aniline point + 71.4° C.

The results of experiments with these solvents are compiled in Table II.

TABLE II

Centrifuge mixture of letdown and:	Solids before Centrifuging,%	Solids after Centrifuging,%
A-heavy oil	16.7	12.0
Paraffin oil	16.6	0.4
Paraffin oil 50% + A-heavy oil 50%	16.6	12.6
Benzol	16.4	3.3
Benzol 50% + A-heavy oil 50%	16.6	3.8
Heptane	16.5	1.6
Heptane 50% + A-heavy oil 50%	16.4	3.8

As these results indicate, the previous observations are fully confirmed. Paraffin oil with 0.4: 16.6% shows by far the best centrifugal effect, followed by heptane. The worst thinning agent is the aromatic benzol. These experiments indicate that even the viscosity is of secondary importance compared to the paraffinic structure, because paraffin oil with a viscosity of 17.5° Engler at 20° C. and 3.7° Engler at 50° C. is considerably more viscous than benzol with a viscosity of 0.93° Engler at 20° C. and 0.85° Engler at 50° C., yet the paraffin oil is superior in its centrifugal effect.

Heptane is a precipitating agent for asphalts. It is, therefore, to be expected that an increase in the asphalt content of the residue and a decrease in the oil should occur with heptane. Actually, an asphalt content of 10.1% was found in the oil from the mixture of letdown and A-heavy oil after centrifuging, 7.3% from the mixture of paraffin oil, and only 3% in the oil from the heptane mixture. The good centrifugal effect of the paraffinic thinning oils seem, therefore, to be based partly on a flocculation of the asphalts and consequent destruction of the colloidal structure.

The experimental temperature chosen was 120° C., because below 120° C. the viscosity of the centrifuge mixture greatly depends on the temperature, due to the steep slope of the viscosity curve, so that each degree temperature rise causes a considerable change in viscosity. At 120° C. the mixture is quite thin and has a viscosity of about 2° Engler. Above 120° C. the viscosity drops very slowly with a rise in temperature. In spite of this an increase in centrifugal effect may be obtained by merely increasing the temperature, as the following experiments show. The same mixture was centrifuged at various temperatures and the solids content of the oil determined. The results are shown in Table III.

TABLE III

Centrifuge Mixture:	Centrifuging Temperature	Solids
Letdown and A-heavy oil, Solids content 16.3%	60° C.	13.0
	80	12.8
	100	12.3
	120	11.6
	140	10.2
	160	7.5

As the figures show, the centrifugal effect may still be increased by raising the temperature from 120 to 160° C. The experimental temperature of 120° C. was, however, retained, because the boiling points of several of the solvents used as thinning oils are fairly low, while on the other hand, A-heavy oil, for example, has a flash point of 170° C. In comparing results of tables II and III it may again be seen that the viscosity is of secondary importance, since a rise in the centrifuging temperature merely decreases the viscosity. Even with the great temperature difference of 100° C. only a decrease to 75% could be obtained. The viscosity, therefore, is of less importance in the centrifugal effect than the chemical structure of the thinning agent, as already shown in the paraffin oil-benzol example.

When experimenting with residue samples at Pölitz, it was found that Pölitz obtained a better centrifugal effect than we did. We, therefore, decided to conclude our experiments by using letdown and thinning oil from Politz and letdown from Scholven.

Politz letdown, 25% solids, 26% asphalt.

Scholven letdown, 21% solids, 25% asphalt.

Gelsenberg letdown, 23% solids, 18% asphalt.

Gelsenberg thinning oil, start boil 343° C., below 360° C.: 85%, aniline point + 3.3° C.

Pölitz thinning oil, start boil 338° C., below 360° C.: 73%, aniline point = 38.7° C.

The mixtures indicated in Table IV were centrifued as before, with results as shown.

TABLE IV

Centrifuge Mixture:	Solids before Centrifuging, %	Solids after Centrifuging, %
Politz letdown + Politz thinning oil	16.3	9.3
Scholven letdown + Politz thinning oil	16.7	13.6
Gelsenberg letdown + Politz thinning oil	16.3	12.4
Pölitz letdown + Gelsenberg thinning oil	16.6	12.7
Scholven letdown + Gelsenberg thinning oil	16.4	13.5
Gelsenberg letdown + Gelsenberg thinning oil	16.7	12.0

The results seemed incredible at first, and the experiments were, therefore, repeated several times. The results, however, were always the same.

Letdown is, therefore, not simply letdown, but, in order to obtain the best centrifugal effect, a special thinning oil must be used for each let-down. This may also explain why our centrifuges operate satisfactorily, at times, with a higher asphalt content in the letdown, while at other times it operates unsatisfactorily with a lower asphalt content, although the centrifugal effect normally decreases with rising asphalt content in the letdown. This probably also explains the difficulties in centrifuging when starting up and shutting down the coal stalls.

Since these experiments were made in an intermittently operating laboratory centrifuge, the results can not be applied directly to the continuous Laval Centrifuges, operating on a different principle.

Summary

We have tried to determine and, if possible, to improve the centrifugal effect in centrifuging mixtures of letdown and various thinning agents. The solvents used were mainly such products as were available at the plant in large quantities. These experiments showed that by the addition of B-middle oil, L.T.C. oil II, A-gasoline, and of tar oil a considerable improvement in the centrifugal effect, compared to the thinning oil consisting of A-heavy oil, could be obtained. They showed that the centrifugal effect improves directly with a rising aniline point of the thinning oils, and that paraffin oil with 0.4: 16.6 is by far the best thinning oil for our letdown. They further showed that increased temperature and changes in viscosity are only of secondary importance in the centrifugal effect compared to the chemical composition of the thinning oil. When using paraffinic thinning oils, the colloidal character is largely destroyed by flocculation of the asphalt with a consequent increase in the asphalt content of the residue and a decrease in the asphalt content of the oil.

The experiments with letdown from Pölitz, Scholven and Gelsenberg showed that not all letdown reacts equally to the same thinning oil. For the best centrifugal effect, a special thinning oil must be used for each letdown. The colloidal structure of the letdown is, therefore, not effectively destroyed by the addition of thinning oil. In the experiments to follow, we shall try to obtain still better results by the addition of electrolytes and consequent change in the P_H value.

Further experiments in Laval Centrifuges alone can show how far the results obtained in the preceding experiments are applicable to them.

Newman

TOM Reel 75
pp. 388-389

U.S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-162

REPLACING COLD GAS WITH COAL PASTE
(Letter by Schappert)

Leuna, 10/9/41

In coal stalls which operate with no heat exchange the amount of heat supplied in the preheaters is roughly equal to the heat of the reaction, since 1 Kg of converted pure coal will set free 440 KCal, while the amount of heat required to heat 2.5 Kg coal paste (corresponding to 100 Kg pure coal) from 100° to 425° is also equal to about 440 KCal.

It has repeatedly been recommended, in particular by Dr. Pier, to replace cold gas with coal paste, with the shape of the paste intake being a slit pipe about 1 m in length. This would permit a considerable reduction in the heat consumption of the preheaters, regardless of whether the operations are run with or without heat exchangers. The suggestion has not as yet been actually adopted in Upper Silesia and in Gladbach.

In the meantime Dr. Urban has actually introduced in Scholven the addition of cold paste. The test has been run for about a week, with 4 te of coal paste being added by means of a temporarily idle pump to the bottom of converter II of a three converter stall. The converter has been started with the present load, and the heating of these additional 4 te paste may be considered a net gain. Up to now, no changes in the thruput of the stall or of its products could be observed; however complete analyses are as yet unavailable. In the near future the paste will be regulated by means of a throttling valve and added to the top of the 1st converter.

Dr. Urban will in addition attempt to reduce the preheater load and increase the stall thruput in the following way:

The mixture of the distillate and gas boils off at the hot catchpot is at a relatively low temperature of 22.5 mv. A different catchpot construction (suggested by Dr. Meyer of the Berger Construction Company) will result in a higher catchpot temperature which will be available for the gas heat exchange. It is hoped, that this will permit reducing the number of gas hairpins and replacing them with paste hairpins. It must be emphasized, however, that the gas hairpins do not merely assume the peak temperature of the circulating gas, but will also compensate for the deteriorating heat exchange. Following a suggestion from Leuna, the heavy oil from the catchpot will be added to the sump. This will also help in keeping the sump cool.

Similar suggestions have been previously adapted in Upper Silesia were the greatest possible utilization of heat exchange is of still greater importance.

/s/ Schappert

W.M. Sternberg 11/19/46

TOM Reel 181 U. S. BUREAU OF MINES D-163
Frames 06611-06614 HYDRO. DEMON. PLANT DIV.

High Pressure Experiments, Jan. 31, 1944.
Leuna, 1.

To the Theory of the Liquid Phase

Summary.

Regularities in the behavior of the liquid phase are illustrated with a few examples, and a method of testing of hydrogenation behavior of coals is outlined.

The table contains examples of the behavior of different coals in the liquid phase for the purpose of production of oils of different composition. All tests have been run at 600 atm with iron catalysts, a normal gas throughput and with about a 50% paste. The only remaining independent variables were the temperature, the trough-put, and the composition of the fuge oil (pasting oils). When these three requirements are fixed, the conversion of a given coal, yield, composition of the oils produced, gasification and asphalt production will be perfectly definite for a fixed method of the working up of the residue. Results will vary from coal to coal, and the converse is also true, that for different coals the three above mentioned requirements must differ when the same results are desired.

Should a definite oil composition be required, e.g., a process intended for the production of middle oil and gasoline, i.e. with the production of no heavy oil, or e.g., for the production of 50% heavy oil, the number of independent variables will be reduced by setting these requirements.

Should we make the stipulation, that the thinning oil in the process for 100% gasoline and middle oil be 100% heavy oil, and the temperature be likewise fixed, there will be but a single throughput for a given coal to operate the converters with the production of oil of this fixed composition. All results, such as conversion, throughput, gasification and asphalt are fixed.

Should an additional requirement be made in the above case, such as a definite asphalt content, or a definite amount of gasification, then with a fixed composition of the thinning oil, there can be but a single throughput and temperature.

Different methods for the production for gasoline, middle oil or heavy oil could be similarly outlined.

It may, for instance, be possible for these reasons, to obtain from a given coal the same distribution of the oil composition with respect to the proportion of gasoline + middle oil to heavy oil by different procedures, which will differ from each other by different temperatures, throughput and composition of the thinning oil on the one hand, on the other by different throughputs, gasification, asphalts and conversion.

There are certain limitations to these conclusions, in that small temperature changes will not have equally great results upon all the factors (say upon the percent of conversion, of the solids, or the asphalt), and on the other hand by the composition of the coal which will affect the definite proportion of gas, middle oil, heavy oil and asphalt simultaneously produced in the destructive hydrogenation. Any strong deviation from these simultaneously predetermined proportions may cause, e.g., a larger gasification, etc. In the process for the production of gasoline and middle oil, the process may be imagined to consist in a simultaneous formation of part of the liquid phase gasoline and middle oil (together with gas, heavy oil and asphalt) directly from coal, while the rest is produced by varying gasification from the heavy oil and asphalt produced at the same time, which enter the return cycle. This may explain why in, say the process for gasoline + middle oil + 25 or 50% heavy oil, in spite of shortened time of contact, the same high pressure reaction volume and temperature will produce considerable amounts of heavy oil in addition to the gasoline and middle oil, compared with the process for gasoline and middle oil. The explanation would lead to the requirement, that each coal be operated for a certain kind of production, best suited to it. One may see in the second line, representing the Heinitz coal hydrogenation, that a process for gasoline and middle oil could hardly be considered suited because of the 27% gasification by this process against the low losses through gasification in the process for the production of heavy oil. Conditions for the production of heavy oil as used with the Zweckel coal do not seem to be the proper ones for this coal because of no advantages against the gasoline + middle oil process produced by the modification.

The observation has already been recorded, that Ruhr and Upper Silesian coals have about the same carbon content but have very different hydrogenation characteristics, when hydrogenated for gasoline and middle oil, and the same applies also to a process for gasoline + fuel oil. With the same proportion of oil, Upper Silesian coals showed no increased throughput even at a higher temperature.

As a general rule, the Ruhr coal seems to be better suited to gasoline + middle oil production than the Upper Silesian coal.

The recycling of oils in a closed cycle may not be the best means to determine the particular method best suited to a given coal, because of the possibility of superimposed effects, which will obscure the issue. The use of B bombs with a standard pasting oil is basically more correct, in spite of the disadvantages of using a non-continuous process with errors resulting from it.

It may well be that a continuous test in a 1 to 2 liter converter, using some standard pasting oil and a standard procedure would produce the best foundation for the evaluation of coal and for finding the relationship between oil production and gasification.

/s/ Rank

W.M. Sternberg

11/21/46

onversion of Different Coals into Gasoline + Middle Oil, or Gasoline+Heavy Oil

uel Oil Experiments in 10 Li. Converters, at 600 Atm, with Iron Cat.

	Temper.	Pure coal throughput	Centrifuge thinning oil	% conversion	total yield	Gasoli + m.o. yield	% gasific in oil prod.	%Asphalt in Liquid
)	480	0.45	100% heavy oil	97.0	0.26	0.25	22.6	13.5
+	481	0.61	50% heavy oil 50% midd. oil	96.6	0.38	0.25	20.5	16.4
oil +	481	0.65	7.5% h. oil	96.2	0.41	0.19	19.0	15.1
il			92.5% m. oil				27.1	7.7
.o. 5%	472	0.57	100% heavy oil	95.0	0.32	0.32	21.9	7.3
50%	475	0.72	50% heavy oil 50% midd. oil	95.3	0.48	0.35	21.6	8.6
	476	0.76	100% midd. oil	94.8	0.50	0.30		
m.o. 50%	480	0.47	100% heavy oil	94.6	0.30	0.30	18.5	11.5
	476	0.61	80% midd. oil 20% heavy oil	96.3	0.41	0.21	18.5	11.7
m.o. 50%	468	0.44	100% heavy oil	96.1	0.27	0.27	24.7	7.4
	477	0.81	100% midd. oil	96.4	0.55		20.5	11.5

[illegible]

[illegible]

[illegible] Fuel Oil Experiments in 10 Li. [illegible]

	Coal	% C in pure coal	Process	Temper.	Pure coal throughput	[illegible] thinning oil	% conversion	Total yield	[illegible] + m.o. yield	[illegible] in oil prod.	[illegible] in Liquid
1	Beuthen - Heinitz, 1 : 1 (production test)	81.96	Gasol.+m.o. gasoline + 35% s. oil gasoline + 50% s. oil	480 481 481	0.45 0.61 0.65	100% heavy oil 50% heavy oil 50% midd. oil 7.5% h. oil, 92.5% m. oil	97.0 96.6 96.2	0.26 0.38 0.41	0.25 0.25 0.19	22.6 20.5 19.0 27.1	13.5 16.4 15.1 7.7
2	Heinitz coal	84.0	gasol.+ m.o. Gasol.+ 25% s.oil Gasol. + 50% s.oil	472 475 476	0.57 0.72 0.76	100% heavy oil 50% heavy oil 50% midd. oil 100% midd. oil	95.0 95.3 94.8	0.32 0.48 0.50	0.32 0.35 0.30	21.9 21.6	7.3 8.6
3	Zweckel coal Scholven (10 li.conv.)	82.5	Gasol. + m.o. Gasol. + 50% f. oil	480 476	0.47 0.61	100% heavy oil 80% midd. oil 20% heavy oil	94.6 96.3	0.30 0.41	0.30 0.21	18.5 18.5	11.5 11.7
4	Gelsenberg coal (young) (10 li. conv.)	83.9	Gasol. + m.o. Gasol. + 50% h. oil	468 477	0.44 0.81	100% heavy oil 100% midd. oil	96.1 96.4	0.27 0.55	0.27	24.7 20.5	7.4 11.5

Newman

T-134

TOM Reel 130 U. S. Bureau of Mines
Pps. 112-134 Hydro. Demon. Plant Div. KGBraun

Starting Up and Shutting Down Stalls

Note: (This article is full of summary statements and ambiguous terms so that its translation is subject to possible misinterpretations.)

Starting Up Stall 3. (Tar)

26 March, (see Table I):

Stall heated only with gas to 15 mv.

Starting Catalyst Pumps

1. Suction line flushed out from suction vessel.

2. Brought up to pressure (220 atm.) with flushing oil pump.

3. Start pump with catalyst paste. First release pressure back to suction vessel, then open valve to stall. Line to stall previously flushed with N_2. The valve on the stall wall first remained shut so that the line could first be filled and brought up to pressure of 270 atm. Drive oil closed. The valve on the stall wall was then opened and the pressure on the pump fell to 220 atm. The pump was again started with a thruput of 500 L/h, which was raised to 4000 L/h within an hour. The catalyst paste went only to the preheater.

Valve Groups in Catalyst Lines on Stall Wall

(See Figure 1)

After the catalyst pumps were started the hot catch pot liquid level was tested. A little gas was injected through the long tube, nothing through the short tube; the level gauge must then register.

Starting Hot Circulating Pump

(See Figure 2)

1. Lines LR & LI (Pendelleitungen) were flushed by the pump with N_2.

(Pressure releases (Entspannungen) on valve box open).

2. Lines LR & LL flushed by pump with flushing oil. Flushing oil circulating through both lines LR & LL. Pressure releases on valve box open. (To save time, flushing oil was run through both lines LR & LL, pressure releases on valve box alternately tested). Then a leak in a 120 mm flange connection on the preheater outlet was discovered. The stall was again shut down. The catalyst paste was shut off and flushing oil (tar) was pumped through the catalyst lines instead. The stall was cooled off, (See Table I). The cause of the leak was probably this, that after the stall was rebuilt the connections in the lines to the preheater were not tightened. 2 bolts were loose.

Stall 3 after Rebuilding

4 Converters, 800 mm dia. x 13 m lg.

2 heat exch., 500 mm dia. x 13 m lg.

1 hot catch pot, 800 mm dia. x 9 m lg.

2 hot catch pot double coils

The intermediate catch pot with its double coils and the small heat exchanger were removed.

Starting Up Stall 3

28 March, (See Table I)

Stall was heated with gas and catalyst paste was injected to the preheater.

Starting Hot Circulating Pumps

(See Figure 2, 3, & 4)

1. Lines LR & LL were flushed with N_2 and flushing oil successively as on March 25th, oil drained from valve box pressure releases of lines LR & LL.

2. Pressure release on pressure side of the valve box open, so that the valve box is flushed out to the pressure valves.

3. Lines LR & LL brought up to pressure with flushing oil to 250 atm. = pressure of shock absorber (Puffer) in the injection header. Consequently, the valve box above the suction

valve was also brought up to pressure, the part below the suction valve had been flushed before with N_2.

4. Hot circulating pump started.

First open valve in by-pass of large drive oil valve (to regulate speed), then open valve to emergency control (to regulate stroke, close, then lengthen stroke, open, then shorten stroke). Pump adjusted to 2000 L/h (by number of strokes), when by-pass is closed and pump connected to stall.

5. Connect valve box:

2 suction valves open,

2 pressure valves open,

by-pass closed (open between pressure releases).

6. Connect stuffing box oil. This oil flows to stall. Wait for level in hot catch pot. Then it was suddenly discovered that the catch pot was full.

The catch pot oil could not be released and the stall had to be shut down again. It must first be determined where clogging occurred. The pressure release group (Entspannungsgruppe) was brought up to pressure with a small hand pump, see Fig. 4. Although a little oil could be pumped into the catch pot, nothing could be released in the opposite direction from the catch pot. Pumps were then [illegible] connected to the level indicator, with the same result. Then a larger electric driven pump was used, but the clogging could not be freed. The main pressure release line, as well as the level indicator connection, was clogged. The stall was again cooled off. (See Table I). It was found that a large quantity of cleaning wool was left in the catch pot.

Shutting Down a Liquid Phase Stall

1. Reduce temperature @ 0.8-1 mv/h. The quantity of fresh tar is then reduced. Regulation with valves in fresh oil injection line because the two tar stalls have shock absorbers. Men at injection pumps watch pressure on shock absorber flask. Level in catch pot must be held. The catalyst paste thru-put remains the same for the time being.

2. When converter inlet has been reduced to 22 mv, the catalyst paste is shut off within 20 minutes. (Shut-off regulated at the catalyst pump).

3. The cold catch pot temperature should not drop. Leuna runs at 60-80° C. in the liquid phase.

4. When temperature reaches 12-10 mv, shut off fuel gas.

~~5. Shut off blowers.~~

6. Shut off fresh tar and hot circulation at 7-8 mv.

7. Shut off gas thruput in 10 minutes.

8. De-sludging, start at the back, (1) catch-pot, (2) Converter IV, (3) Converter III, etc. Clean up first to reduce danger of clogging.

9. 2 hours gas flow.

10. Disconnect and release pressure into atmosphere ("Über Dach") and again desludge.

Starting Up Vapor Phase Stall II

(January 1938). (Fig. 5&6)

1. Flush with N_2.

   Analyze N_2 for O_2 & H_2.

   a). Flush separator flask in gas inlet line with N_2 and bring to 30 atm. pressure. (Connect N_2 to drain of flask).

   b). Flush gas inlet line back to pressure circulating header by pressure release between the two ratchet valves. (Close inner ratchet valve again).

   c). All cold gas lines to the converter are then flushed first opening valves 15 (oil operated), 17 and 8. (Fig. 5). The shut off valve (ahead of the main valve) and the by-pass valve around the main valve in each cold gas line are opened for about two minutes and then again closed.

   d). Stall is flushed. Valve 13 is opened for half an hour. Finally, the gas outlet line to the suction circulating line is flushed by releasing the pressure between the two ratchet valves. (Valve 16 is opened and closed again after flushing).

Analyses are made on the pressure release line to atmosphere and on the catch pot. The O_2 content must be less than 5%.

The N_2 absolute pressure is held to about 1 atm. (Connect manometer). All valves must be closed while analyses are made.

2. N_2 Pressure Test

a). In a new stall, or if major repairs have been made, the stall is brought up to operating pressure. (Speed of compression same as for H_2-?).

b). In normal shut downs the N_2 pressure test is made only at 30 atm. The whole stall is tested for leaks.

The N_2 pressure is again released to 1 atm. absolute. (Low press. manometer). In starting the first stall of a plant, the circulating pumps can be started with N_2 at about 50 atm. and the pressure gages can be checked.

3. H_2 Pressure Test

The stall is now compressed with H_2. The ratchet valves and oil operated valve 15 are opened, so that the gas inlet line is on circulating pressure to valve 13. The stall is first flushed with H_2 to keep the N_2 in the stall out of the circulating system. Even fresh gas is no good. The by-pass valve around 13 is carefully opened and also valve 9. Watch low pressure manometer on suction side. After about five minutes valve 9 is again closed. Then the H_2 pressure in the stall is increased in the following manner. To avoid damage to the catalyst the pressure must be increased very slowly at the start. Care must also be taken that the catalyst temperature does not exceed 3 mv.

Filling with H_2

(5058 and 6434)

0-1 atm.	30 min.
1-10 atm.	90 min.
10-20 atm.	60 min.
20-100 atm.	45 min.
100-200 atm.	45 min.
Total	270 min. = 4½ hrs.

If the by-pass valve around 13 is too coarse, the filling may be started with a 10 mm by-pass valve of the cold gas supply lines.

When the stall pressure equals that of the circulating suction line, valve 13 is closed and the main shut-off valve to the circulating suction line is opened.

[illegible] Increasing the Pressure

The by-pass valve around 13 is opened and the inlet gas volume is adjusted to 20,000 m^3/h by means of the inlet gas pressure gage. All other pressure gages are brought under pressure.

The power is turned on in the preheater and the temperature of the highest element in the first converter is increased about 0.5-0.6 mv/h.

At about 10-12 mv cold gas is run into each of the cold gas lines to check the thermoelements and cold gas tubes, starting with the last cold gas tube in the last converter. If there is any doubt about the connection of any element or tube, this check is repeated at [illegible] mv.

When the temperature at the inlet to the first converter (first element in the converter) has reached 19-20 mv, injection may begin. All cold gas valves are tested and the shut-off valves (ahead of them) are opened. The cold gas volumes are regulated by the small by-pass valves.

The starting injection volume is dependent upon the reactivity of the oil and the activity of the catalyst, 2000-3000 L/h can generally be used at the start. The volume is regulated by the small by-pass valve and the small throttling disk (orifice plate -?).

The inlet temperature at the first converter is held constant. At the first injection the power to the preheater must be slightly increased. If the temperatures rise above the predetermined, they are regulated with cold gas. If the temperature in the last converter increases due to the heat of reaction, the power to the preheater may be reduced. In the case of Stall II, which was filled with old catalyst and was run with 5058 saturated oil, all thermoelements in the first converter were held to 20 mv.

When the temperatures in the last converter, begin to rise, the injection is gradually increased to perhaps 1000 L/h. This increase of the injection is dependent upon the heat of reaction of the product. When the temperatures in the last converter have also reached 20 mv, the first converter may be somewhat reduced. The temperatures are regulated according to the desired spec. gravity of the catch pot product, (Stall Δ S.G. = 0.07).

After starting injection, water is injected into the cooler and the cold heat exchanger. In Leuna water is continuously injected in both places, in winter about 1000 L/h/stall.

Starting Stall 11

18 January

12.45 - 17.30, fill with H_2.

18.00, started, 20000 m^3/h inlet gas.

18.30, preheater started and temp. raised.

20 January

3.00, element #1 @ 20.0 mv.

9.30, injection started, 3000-4000 L/h,

element #44 @ 18.6 mv,

element #1 @ 20.0 mv.

Temp. dropped back somewhat and preheater power was increased from 780 to 840 KW.

Element #1 was held @ 20.0 mv so that converter could pull faster.

11.00 - 14.00, injection increased from 4000 to 6000 L/h,

16.00, injection 8000 L/h.

Preheater power reduced.

Injection gradually increased further @ max. converter temp. 20.0 mv and Δ S.G. about 0.07.

21 January

14.00, injection 22400 L/h.

S. G. injection 0.826/26° C. = 0.830/20° C.

A. P. 62.7° C. 0.070

S. G. catch pot prod. should be 0.760/20° C.

Shutting Down Vapor Phase Stall

Reduce injection 1000 to 3000 L/h and keep S.G. injection and S.G. catch pot product constant. Keep temp. high. Then shut off injection and run another two hours with preheater power to keep the same converter temperature to dry out stall. Then reduce temperature 0.3-0.4 mv/h. When temperature has dropped to 16 mv it can be reduced at the rate of 0.6-0.8 mv/h. To keep up the temp. in the last converter the volume of inlet gas should be increased.

Releasing the pressure at the shut down of a vapor phase stall should be done at the same speed as in filling with H_2, see "Starting Up Stall II":

200-100 atm.	45 min.
100 -20 atm.	45 min.
20 -10 atm.	60 min.
10 - 1 atm.	90 min.
1 - 0 atm.	30 min.
Total	270 min. = 4½ hrs.

1. Reduce injection gradually by about 4000 L/h, keeping converter temperatures high in order to vaporize all oil residue. If the temp. in the last converter drops while reducing injection, the gas thruput should be increased. A larger volume of gas is of this advantage later in cooling off, that the heat is carried off faster. In general, the power should be reduced together with the injection.

2. When the injection has been reduced to 3000-4000 L/h it may be cut off entirely. The converter temperatures should then remain high another two hours after no more oil is produced, if need be, increase the gas thruput. Power to the preheater should be correspondingly increased. Keeping the temperature high for two hours is absolutely necessary, if the temperatures have previously dropped below 20.0 mv.

3. Reduce temperature at the inlet to converter #1 by 0.5-0.7 mv/h to about 3-4 mv. If the catalyst is to be decomposed, the temperature should be reduced to 1 2 mv, otherwise to 4 mv.

4. Cut off stall from circulating system, after the gas thruput has been reduced in the course of 10 min., valve 13, because of circulating pumps (keeping pressure differential in circulating system), close main shut-off valves at the stall inlet and outlet.

5. Release pressure, valve 9, keeping valve 14 closed. This keeps the flow in normal direction. The speed of the pressure release has already been given. Release to 0 atm. During pressure release the cold heat exchanger is again deslugged. It is very important that the speed of the pressure release in the lower pressure range, previously indicated herein, be not exceeded, in order not to damage the catalyst.

If the pressure is released in this manner, valve 14 closed, care must be taken that the stall pressure difference does not exceed 15 atm. If this should actually happen, valve 14 is opened very carefully, so that no back flow occurs. This pressure difference is practically never exceeded in the vapor phase.

6. Flushing out with N_2 (cold gas lines).

Analysis at the outlet should show not more than 5% combustibles (H_2 + hydrocarbons).

7. Stick blind disks into inlet and outlet of ratchet valves; (if only one ratchet valve is used, then inside of oil operated valve).

If the stall is cooled off only for keeping in reserve, it must be kept under pressure.

High Pressure Gas Wash (See Fig. 7).

Available in Leuna, 9 washers 1300 mm dia. x 15 m lg, 6 for liquid phase and 3 for vapor phase. No washing is done any more in the vapor phase and the washers serve only as separators.

Of the six liquid phase washers, five were in operation.

Total gas production - 225,000 m^3/h

Total oil production - 370 m^3/h

Normal load on washers

gas - 50,000 m^3/h

oil - 70-80 m^3/h

Maximum load

gas - 100,000 m^3/h

oil - 100 m^3/h

Expansion machines (Entspannungsmaschinen)

3 available; 1 old and 2 new in the new building.

3 high pressure wash oil pumps with steam engines.

Capacity: exp. machines, to 300 m^3/h

pumps 40 m^3/h

A small amount is released directly to the 20 atm. expansion vessel for regulating the level fluctuations in the washer. Approximately 10% of the wash oil is supplied by the supplementary pumps.

Determination of foam break-down time

25 cc of regenerated wash oil is shaken vigorously for 5 sec. The time interval to the moment when the surface first becomes visible is then determined. (At room temperature; bottle must be clean; always use same bottle). After 60 seconds test another suction vessel.

Various tests are taken from a note book:

Seconds	Room Temp.	Density
24	20° C.	0.830
35	18	0.835
40	18	0.840
50	18	0.845
59	20	0.860
60	20	0.855

TABLE I

STARTING UP STALL 3

Time (A)	Temperatures 60 (B)	61 (C)	Conv. I 8 (D)	Conv. IV 101 (E)	Gas Input, m^3/h Main (F)	Double Coil (G)	Hot Catch Pot Coil (H)	Fuel Gas m^3/h (I)	Catalyst L/h (J)
24 March									
9.55	Started Stall				13000	4000	1000		
10.55	Fired Preheater. Started Blowers				"	"	"		
12.00	-	1.8	1.8		"	"	"	550	
13.00	-	3.3	3.3	0.3	"	"	"	580	
15.00	1.5	5.4	5.2	2.4	"	"	"	480	
17.00	2.8	6.9	6.5	3.7	"	"	"	420	
19.00	3.5	8.0	7.6	4.3	"	"	"	420	
21.00	4.4	9.2	8.7	5.1	"	"	"	480	
23.00	5.0	10.6	9.9	6.0	"	"	"	520	
25 March									
1.00	6.0	13.0	11.8	7.2	"	"	"	500	
3.00	7.1	14.5	13.0	8.5	"	"	"	500	
5.00	7.8	15.7	14.3	9.4	"	"	"	520	
7.00	8.8	17.5	15.3	10.3	"	"	"	500	
9.00	9.4	17.5	15.3	10.9	"	"	"	520	
9.40	Started catalyst paste to preheater, increased to "								4000
10.00	9.4	16.9	15.2	11.5	"	"	"	900	
10.30	Shut off catalyst paste, stall cooled off. Flushing oil instead of catalyst paste. 120 mm flg. at preheater outlet leaked.								

TABLE I (Cont.)

	Temperatures				Gas Input. m^3/h				
(A)	(B)	(C)	(D)	(E)	(F)	(G)	(H)	(I)	(J)
11.00	9.8	17.3	16.3	11.7	13000	4000	1000	520	
12.00	10.1	16.0	14.9	11.8	"	"	"	350	
14.00	9.6	14.4	13.4	11.2	"	"	"	320	
16.00	9.0	12.6	11.7	10.2	"	"	"	100	
18.00	7.5	10.3	9.7	8.4	"	"	"	100	
20.00	6.4	9.1	8.2	7.2	"	"	"	100	
20.00	Shut off blowers. Inject CO_2				"	"	"		
20.10	Shut off flushing oil.				"	"	"		
21.00	Shut down converters & release pressure. Converters 1-4 & H.O.P. desludged.								
26 March									
1.30	Shut off CO_2				"	"	"		
23.15	Started stalls				"	"	"		
23.45	Fired preheater				"	"	"		
24.00	0.2	6.5	6.2	0.3	"	"	"	460	
27 March									
2.00	3.4	8.8	7.7	4.4	"	"	"	440	
6.00	5.2	10.0	9.2	6.2	"	"	"	350	
10.00	6.3	11.6	10.8	7.3	"	"	"	430	

TABLE I (Cont.)

(A)	(B)	(C)	(D)	(E)	(F)	(G)	(H)	(I)	(J)
27 March (Cont.)									
14.00	7.0	12.9	12.0 -	8.3	13000	4000	1000	460	
18.00	7.9	13.9	12.9	9.3	"	"	"	500	
22.00	8.7	15.5	14.2	10.1	"	"	"	430	
28 March									
2.00	9.1	15.6	14.7	10.5	"	"	"	340	
7.00	9.2	16.0	15.0	10.7	"	"	"	350	
7.55	Started catalyst paste to preheater, increased to"								4000
10.00	9.3	16.9	16.9	12.1	"	"	"	1070	4000
10.15	Connected valve box. Started hot circ. pump								2000
11.00	9.3	18.0	17.9	12.9	"	"	"	1350	4000
	Catch pot clogged, pressure released, temperature somewhat reduced.								
12.20	Determined level in hot catch pot (total of 15 m^3 injected).								
	Still cooled off. Catch pot clogged.								
15.00	10.6	15.7	15.2	13.8	"	"	"	420	2500
	Shut off catalyst paste, started flushing oil through stuffing boxes of catalyst pumps to preheater, (with pumps down).								

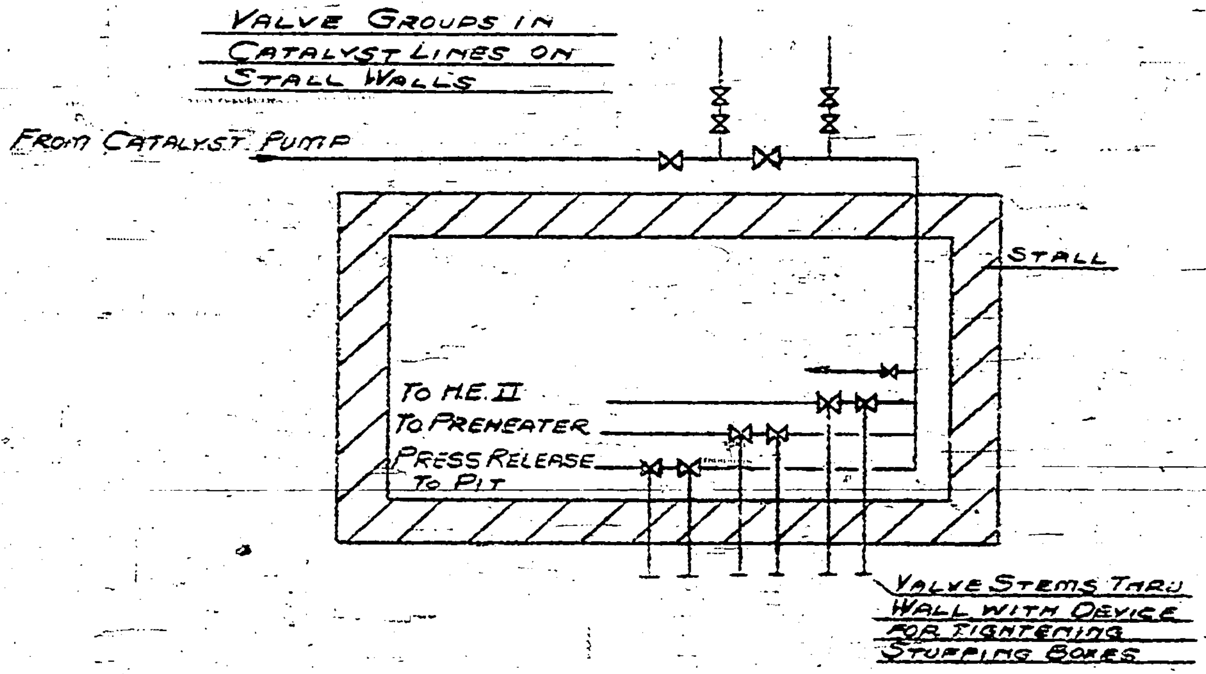

VALVE GROUPS IN
CATALYST LINES ON
STALL WALLS
FROM CATALYST PUMP
STALL
TO H.E. II
TO PREHEATER
PRESS RELEASE
TO PIT
VALVE STEMS THRU
WALL WITH DEVICE
FOR TIGHTENING
STUFFING BOXES

FIG. 1

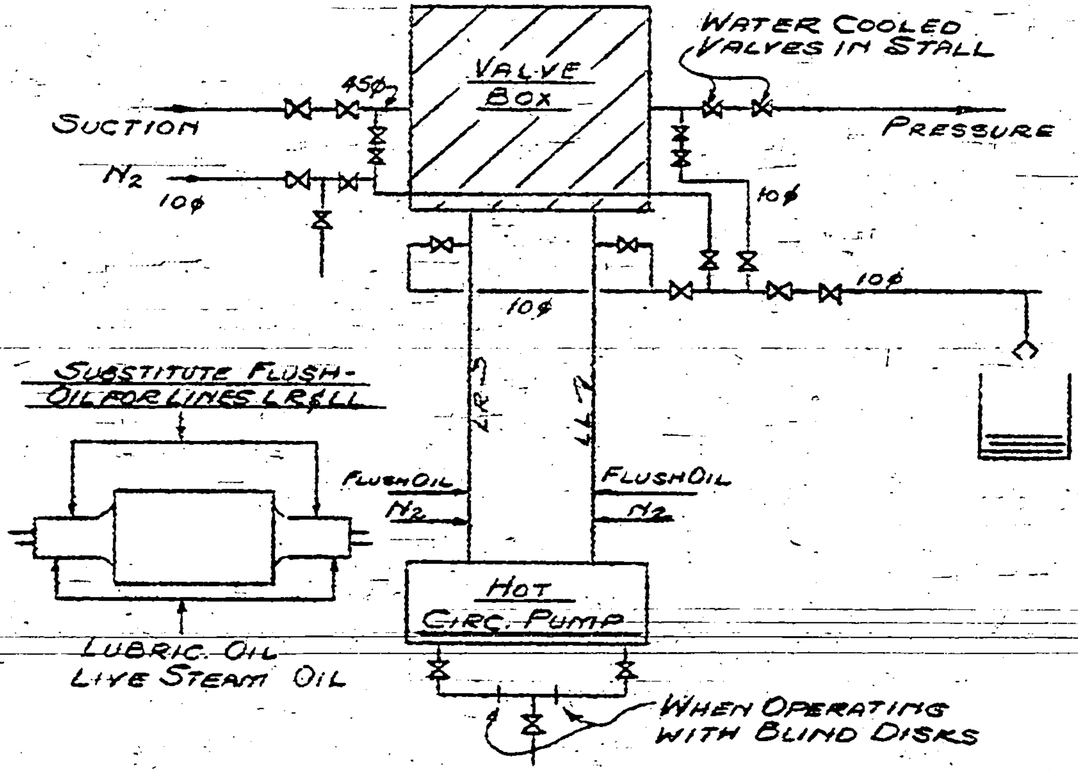

VALVE
BOX
WATER COOLED
VALVES IN STALL
SUCTION
45φ
N_2
10φ
PRESSURE
10φ
10φ
10φ
LR
LL
SUBSTITUTE FLUSH-
OIL FOR LINES LR & LL
FLUSH OIL
N_2
FLUSH OIL
N_2
HOT
CIRC. PUMP
LUBRIC. OIL
LIVE STEAM OIL
WHEN OPERATING
WITH BLIND DISKS

FIG. 2

T-164

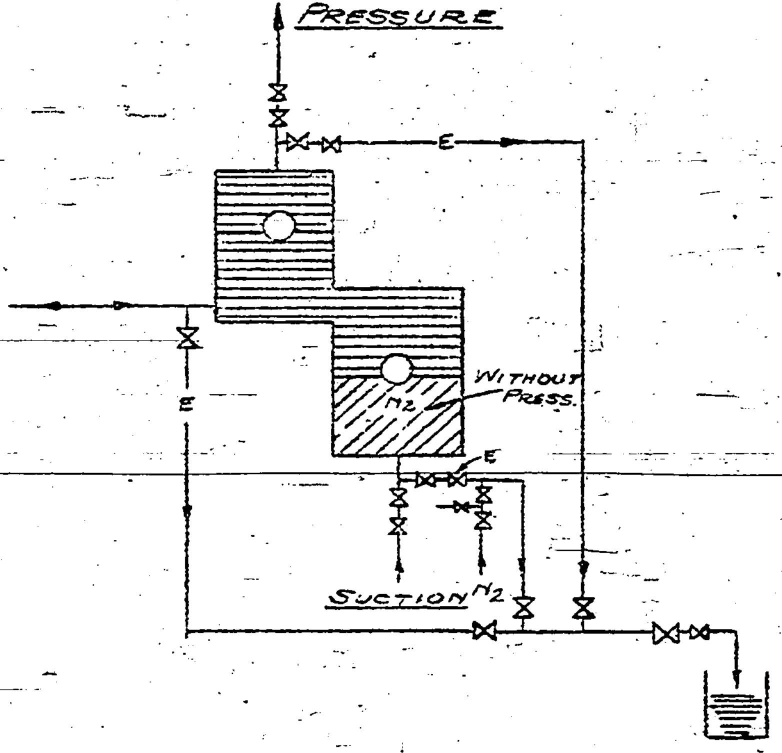


FIG. 3

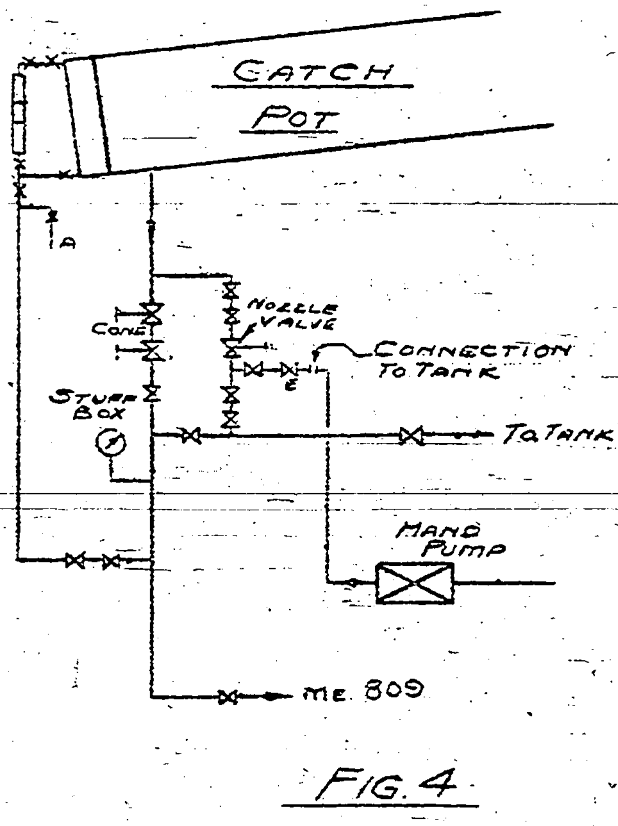
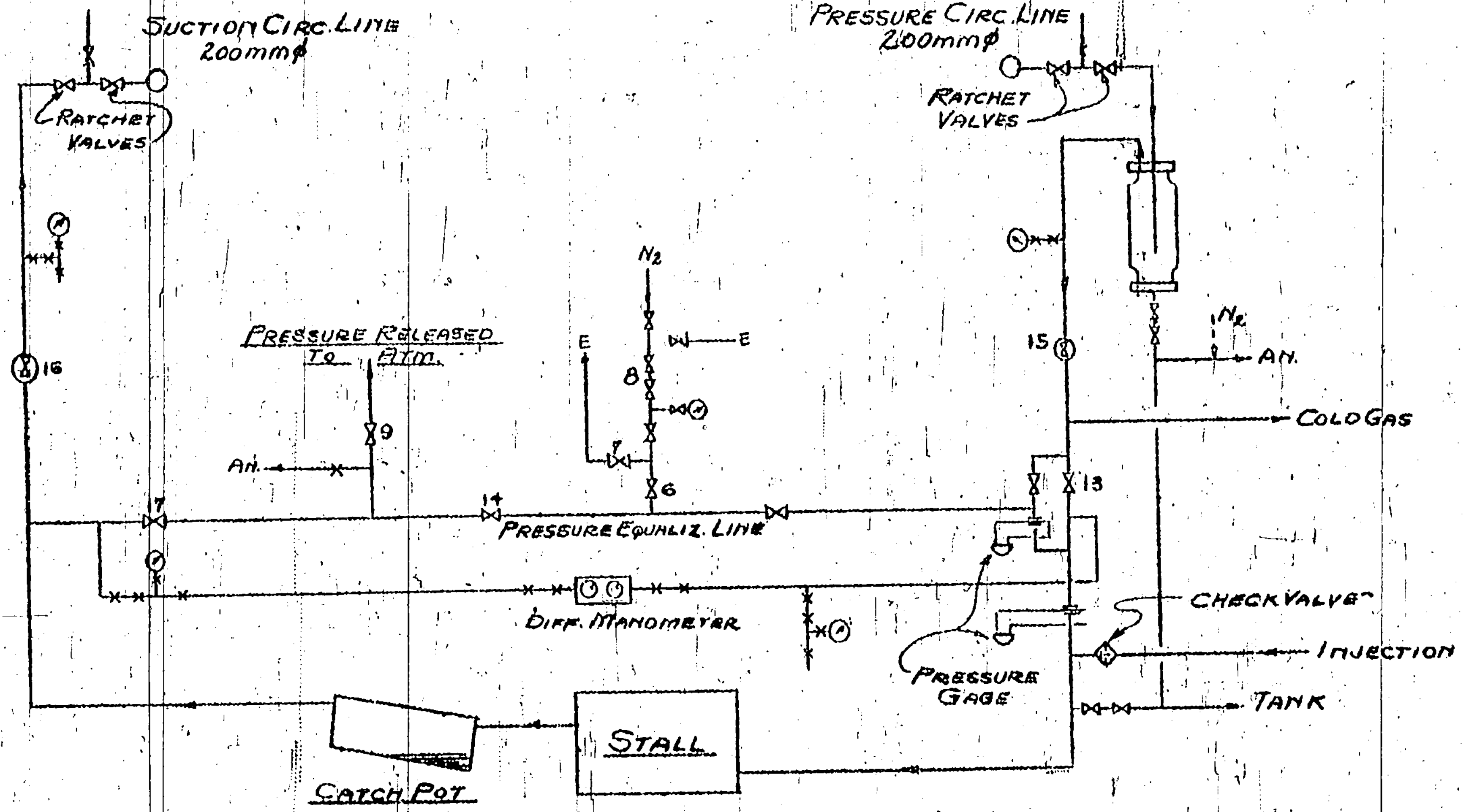


FIG. 4

T-164



Gas Inlet Valve & Outlet Valve Arrangement, Stall 11

Fig. 5

VALVE DESIGNATION IN LEUNA

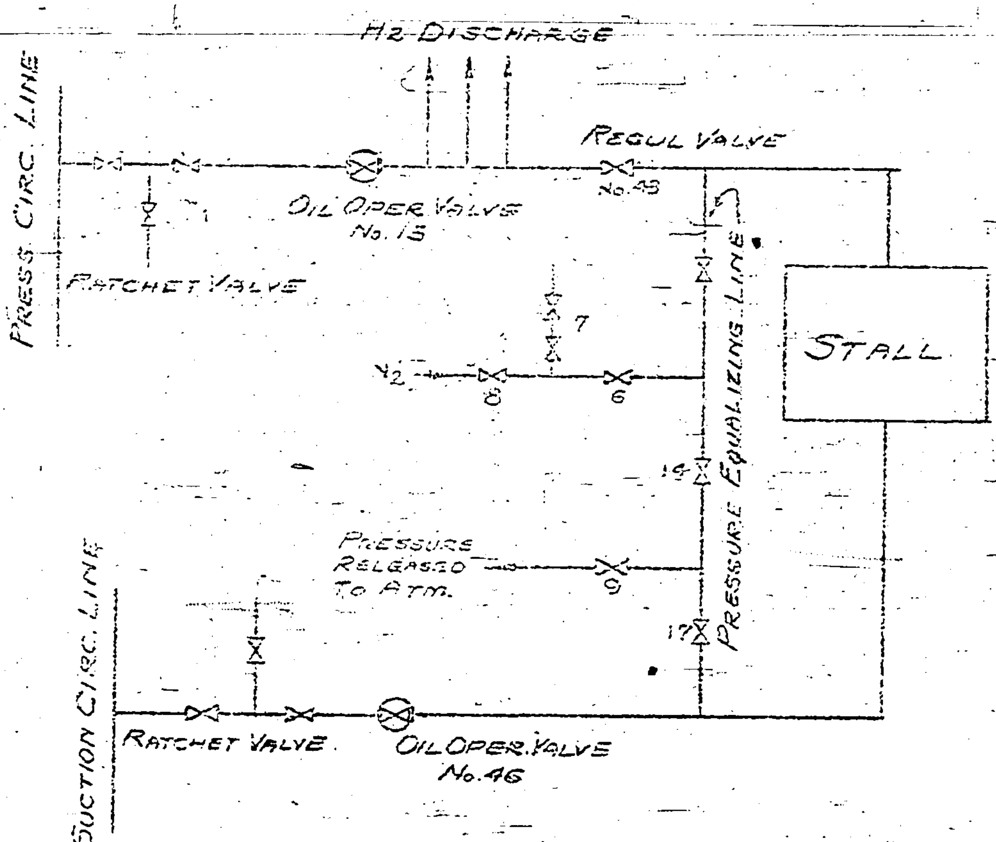


FIG. 6

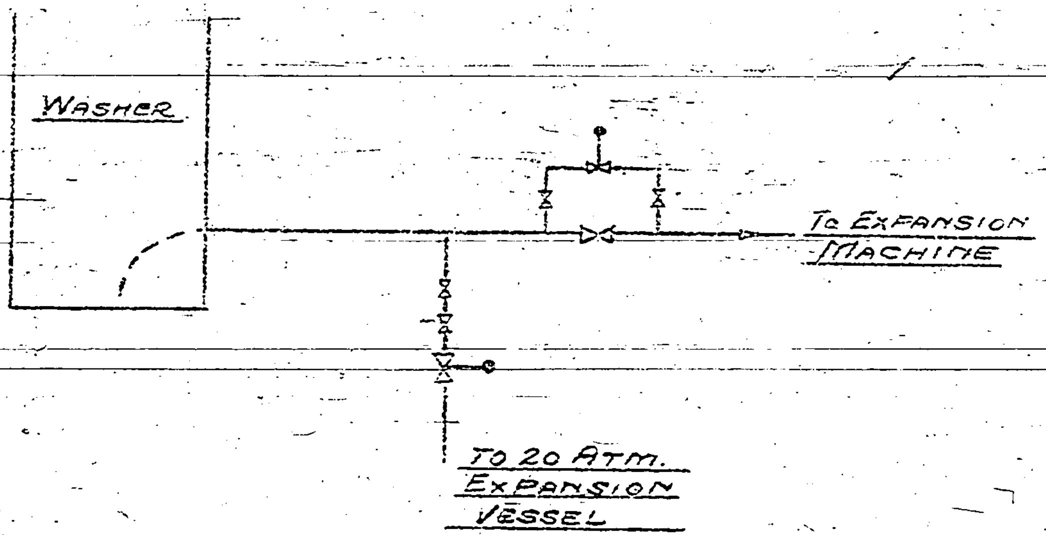


FIG. 7

TOM Reel 130
Pps. 230-245
Ref#0

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-165

Report on Dismantling of Stell #5, March 29, 1941

Leuna, April 29, 1941

Date of shutdown: March 29, 1941
Time in operation: 269 days
Reason for dismantling: The blower rotor flew apart, destroying the blower housing. Because of the length of operation of the stall, the decision for a major overhaul has been made.

The different parts of equipment were shut down, emptied and dismantled.

Converter #1, 2564/599, with Converter #1 de-sanding (cold gas pipe not insulated). The operating time of the catalyst tube was 269 days. It contained some loose scales in the narrower part of the bottom funnel. The catalyst tube was in good condition and had but a few blisters. The thermocouple protecting rod and the cold gas tube had an asphalt-like deposit 1 mm thick, but were not appreciably harmed. The de-sanding nipple was unscrewed and considerable wear was discovered in the inner part of the nipple for a length of 65 mm, and at the outlet of the inclined de-sanding canals in the bottom converter cover. The bottom converter cover was taken down and a wear discovered at the canal inlet into the cover (see Sketch #1).

Converter #2, 225/546. Operating time of the catalyst tube 927 days (cold gas tube not insulated), was free from residue. The catalyst tube had a deposit of iron sulfide 1-1.5 mm thick. The thread of the bottom funnel was torn at the pointed end. The fishplate of the bottom circular thread, two of which had been previously repaired, had become loose. The 16,000 mm long thermocouple tube had a flat ground spot about 120 mm in length at its bottom end. The 14,000 mm long thermocouple protecting tube was in good condition. The 14,000 mm long cold gas tube was broken at a distance of 2200 mm from the upper edge of the mantle flange; 2800 mm below this spot the tube was cut through for a distance of about 60 mm. A supporting bracket was at that level, and we may assume that the tube was worn out by this bracket. The cold gas tube, 10,000 mm long, was in good condition. The wall of the 2,000 mm long cold gas tube was very thin and eroded at the bottom end. The cold gas tubes had an asphalt-like deposit 1-2.5 mm thick. The cover seal ring was strongly damaged.

Two-thirds of the sheet metal of the shell had fallen off and the insulation of the ring was laid bare. The remaining sheet metal had an iron sulfide deposit 0.5 mm thick. The funnel of the seal ring was in good condition.

Converter #3, 229/554. 923 days in operation (cold gas tube not insulated) was free from residue. The inside surface of the catalyst tube was completely covered with blisters the largest of which were approximately 80 mm in diameter. Two round threads of the catalyst-cold gas tube and the thermocouple protecting tubes were in good condition.

Converter #4, 220/564. The operation time of the catalyst tube was 581 days (cold gas tube not insulated); in good condition. It was subsequently used in the stall as a fourth converter.

HOLD Catchpot, 331/E606. Operating time 81 days. Free from residue. The surface of the catalyst tube was somewhat roughened. The inlet and outlet of the tank had a slight deposit. The tank was left in the stall.

Intermediate Catchpot, 351/E509. In operation 1423 days. Contained some oil in the bottom funnel which could be removed. The catchpot was left in the stall.

Heat Exchanger #1, 826/E61, with long V2A chest. The operating time of the insert - 346 days, had a high resistance in the outer pass. The bundle was clean. The gradual bend #1585M, 120 mm inside diameter, made of N6, showed the well known small tongue. No other erosion had been observed. The heat exchanger was steamed. Tests for thruput and tightness were good. It was left in the stall.

Heat Exchanger #2, 848/E85, 81 days in operation, had a high resistance in the outer pass. The exchanger was steamed. The thruput and tightness were found good. Left in the stall.

Cooling Columns #15 and #16 were newly installed on December 15, 1940. Column #16 occupied the second position and was shut down after 46 days of operation. 250 mm below the tip was a leak close to one of the level indicators and a second leak about 60 mm long along the welded seam 250 mm below the first leak. The column was welded together at this place out of V2A sheet.

Column #15 was tight and was returned to operation.

The double coils #542, at I/1, 81 days use in stall 5
503, at I/2, 81 days use in stall 5
530, at II, 269 days use in stall 5
were dismantled for x-ray analysis.

Of the two N6 Zig-zag coolers, #5 and 21, used 269 days cooler #5 was dismantled from the HOLD catchpot pipe line and taken apart. No noticeable abrasion was observed. Cooler #21 was kept in the stall.

The check valve #2250 for the hydrogenation gas in the paste pipe line, i.d. 58 mm, made of N6 was dismantled for checking. The lower part (seat) and ball were strongly worn after 81 days of use. These two parts were scrapped.

The S_2 coil cooler I #343 and II #315 were remeasured. Results have not been yet reported.

The gas preheater was shut off and the piping examined for deposits and abrasion. The 120 tube section of the mixer part had an asphaltic deposit up to 1.3 mm thick.

The 70 tube section of the gas part had an iron sulfide deposit up to 0.7 mm thick. All the tubes were removed, drilled and x-rayed, which disclosed a reduction of 0.7-4 mm in the wall thickness of the return bends of the 120 tube section. (See sketch #2).

The upper return bends were sent to Me 823 for measuring the wall thickness. Decreases of wall thickness of 1.8 to 4.8 mm were observed.

Corrosion, attributed to the formation of sulfuric acid from SO_2, and water-containing flue gases, caused an erosion of mineral insulation and of the surrounding shell, with the formation of holes in the shell the size of one's hand. The underlying insulation stones (Kieselguhr) became cemented 20-35 mm deep to the side of the shell and broken down. The outside walls in these places was full of cracks. The insulation was repaired and the preheater shell put back in condition by welding of sheet metal to the damaged spots. The shell had to be patched in this way in 36 places. In addition, the explosion dampers were gone over. The blower, which had been eliminated because of the breaking-off of the rotor blades at the rotor #103 and the destruction of the housing, was torn down. See report of 4/18/41 on the destruction of rotor and housing, which is added as an appendix.

The blower foundation was strongly damaged by cracks. It had to be completely replaced. The foundation pit was clad with sheet metal as prtoection against radiation.

The two-neck flask (upright dewatering vessel) #1992 was examined by the district office, compressed under 350 atm. and removed.

The gas cooler was turned off and the tubes examined for residue. The cooler tubes were clean.

The outlet tube of the feed cooler had a solid residue up to 15 mm thick. All the rest of the tubes had a soft residue. The tubes were steamed anddrilled and rejoined. The thruput test was good.

Piping in the stall.
The piping was tested for depositions and abrasion. The deposition was very slight, with the largest amount of it in the line between converter IV and the HOLD catchpot, with up to 1.2 mm in thickness.

Noticeable abrasion was found only in the line between heat exchanger I and the preheater. The long radius bend 1585 M, of N6, with a wall thickness 21.5 mm and the pipe bend #306 SB of N8, wall thickness 15 mm, have been scrapped (v. sketch 3). There was a grooving in the latter. The original wall thickness of the bend 306 SB was 21 mm. The wall thickness was therefore reduced by 6 mm.

No standards of comparison were available for the long radius bend 1585 M. The pipe-line was reassembled without drilling.

The HOLD pipe line was taken down and tested for abrasion.

No abrasion was noted in the 45 mm high pressure section. The N6 bend, 45 mm i.d., #64, was slightly worn.

On the low pressure side, the following parts were scrapped because of wear:

90 mm injection tube,	#146	(see sketch 4a)
90 mm injection tube,	#160	"
90 mm bend tube,	#156	"

The following special shapes were sent to Me 823 for measurement and found in good condition:

90 mm T-piece,	#147	Sketch 4a.
90 mm bend	#151	
90 mm T-piece	#203	
45 mm T-piece	#197	
45 mm bend	64	

The 30 mm N6 bends, #411, 414 and 334 in the HOLD pipe line from converters I and II were measured and found in good condition (sketches 4b & 4c).

<u>The de-sanding pipe line of converter I</u> was taken apart and examined for deposition and abrasion.

No abrasion was observed other than that in the de-sanding nipple of N8 and the inclined de-sanding channel of the bottom converter cover. The de-sanding nipple can not be reused.

The following shapes were sent for measurement to Me 823 and found in good condition:

45 mm bends of N6: #478
480
482
483 } Sketch 4c.
489
490
494

The amount of salt like deposition in the pipe line of the de-sander of converter I was small. The largest deposit (2mm) was in the horizontal arm of the 45 mm N8 bend #469 infront of the water cooled valve. No salt was found in the pipe line behind the oil activated valve (sketch 4c). Both valves were free from abrasions.

<u>The stall was equipped with:</u>

Conv. I, #2570/574, 1200 mm diam., 12m. long catal. tube 580 days in use.

Conv. II, #101/595, 1000 mm diam, 18 m long, cat. tube 299 days in use.

Conv. III, #210/551, 800 " " 18 " " , " " 882 " " "

Conv. IV, #220/564, 800 " " 18 " " " " 581 " " ".
(Kept in use)

Hot Catchpot #331/606, Insert 81 days in use (Kept in use)

Intermed. catchpot #351/509 Insert 1423 days in use (Kept in use)

Heat Exch. I, #826/61 " 346 " " " " " "

Heat Exch. II #848/85 " 81 " " " " " "

Cooling Column #15. 81 " " " " " "

N6 double coil I/1 #550 - new .

N8 " " I/2 536 - 540 days in use.

N5 " " II/1, 521 1491 days in use.

N6 Zigzag cooler #21, retained from preceding test.

N8 Coil cooler #402, i.d. 45 mm - new .

S_2 " " 354, " " 45 " "

Check valve #2535 for hydrogenation gas.

The gas preheater was equipped on the mixture and the gas passes with five 120 mm and four 70 mm resp., hairpins from stock. A new blower replaced the one destroyed. The rotor 114 was built into the blower and a new surface cooled motor put in.

Principal Changes

1. Converter I received two inlet thermocouples #44 and 44a (44-a a glass resistance thermometer).
2. Converter II 1000 mm diam. x 18,000 mm long with an elastic ring packing (double cone) replacing the converter 8000 mm diam. x 18,000 mm long.
3. 2 intake thermocouples in converter III, ## 244 and 244 a. (244a - glass resistance thermometer).
4. The first cooling column behind the hot catchpot was eliminated, together with the gas inlet valve and the water cooled gas outlet valve. Only one single cooling column is kept in frontof the double coils.
5. The gas inlet valve on the stall wall to the only cooling column left in the stall has been eliminated.
6. The Weis automatic attachment to the converter I de-sander needle valve has been removed and the valve rebuilt for manual operation.
7. The double coil I/2 was connected in reverse.
8. The zigzag cooler behind the hot catchpot has been removed.
9. W_8 and S_2 coolers in the catchpot I HOLD pipe line, thermocouple #87 between the coolers.
10. De-watering tank with tangential inlet pipe.
11. H_2-pressure pipe line 90 mm with 45 mm valve with no by-pass.

12. At the service panel of the cold gas group the 24 mm valve with the by-pass omitted in the cold gas preheater of converter I. Bubblegas (?) inlet pipe line to the converter cold gas is omitted.

13. Horizontal catchpot introduced for the gas level indicator of the HOLD.

14. H_2 connection, 10 mm, in the cold gas main.

15. 6 mm N_2 connection to the circulation pressure manometer eliminated.

16. Differential manometer connection and analysis of the by-pass were eliminated.

17. Differential manometer connection and analysis introduced in the 90 mm section side.

18. Differential manometer connection and analysis introduced in the 90 mm pressure side.

19. Analysis of the circuit suction manometer was eliminated.

20. Analysis eliminated in the N_2 pipe line.

21. Cold gas by-passes for converters III and IV widened from 6 to 10 mm.

22. The gas cooler had a new water distributor added with two separate supply lines and 2 direct steam connections.

23. Flushing oil connections for the atmospheric pressure pipe line in the converter I de-sander between the valves.

24. Removal of East fuel gas line from the net with two slide valves. with intermediate exhausting.

25. Fuel gas pressure release at the burner increased from 50 mm to 80 mm. diameter.

26. The blower foundation protected from radiation by sheet metal lining of the pit.

27. On the regulating valves in the circulating gas intake and outlet lines guadrants (Anschlagringe) have been mounted.

28. On the south-end needle valve of the HOLD catchpot a Wiegram chilled cast bearing has been experimentally installed.

The stall was started May 2 at 6:50 A.M.

/s/ undecipherable.

W. M. Sternberg.

Leuna, April 18, 1941

The stall 5 at Leuna had to be shut down at 1 P.M. in March 29, 1941 because the rotor of the blower No. 103 flew apart, and in doing so destroyed the blower. The total operating time of the rotor was 1480 days, of which time it had been used in stall 5 for 270 days prior to its destruction. The rotor was of an older construction type, with no cast steel cross reinforcement.

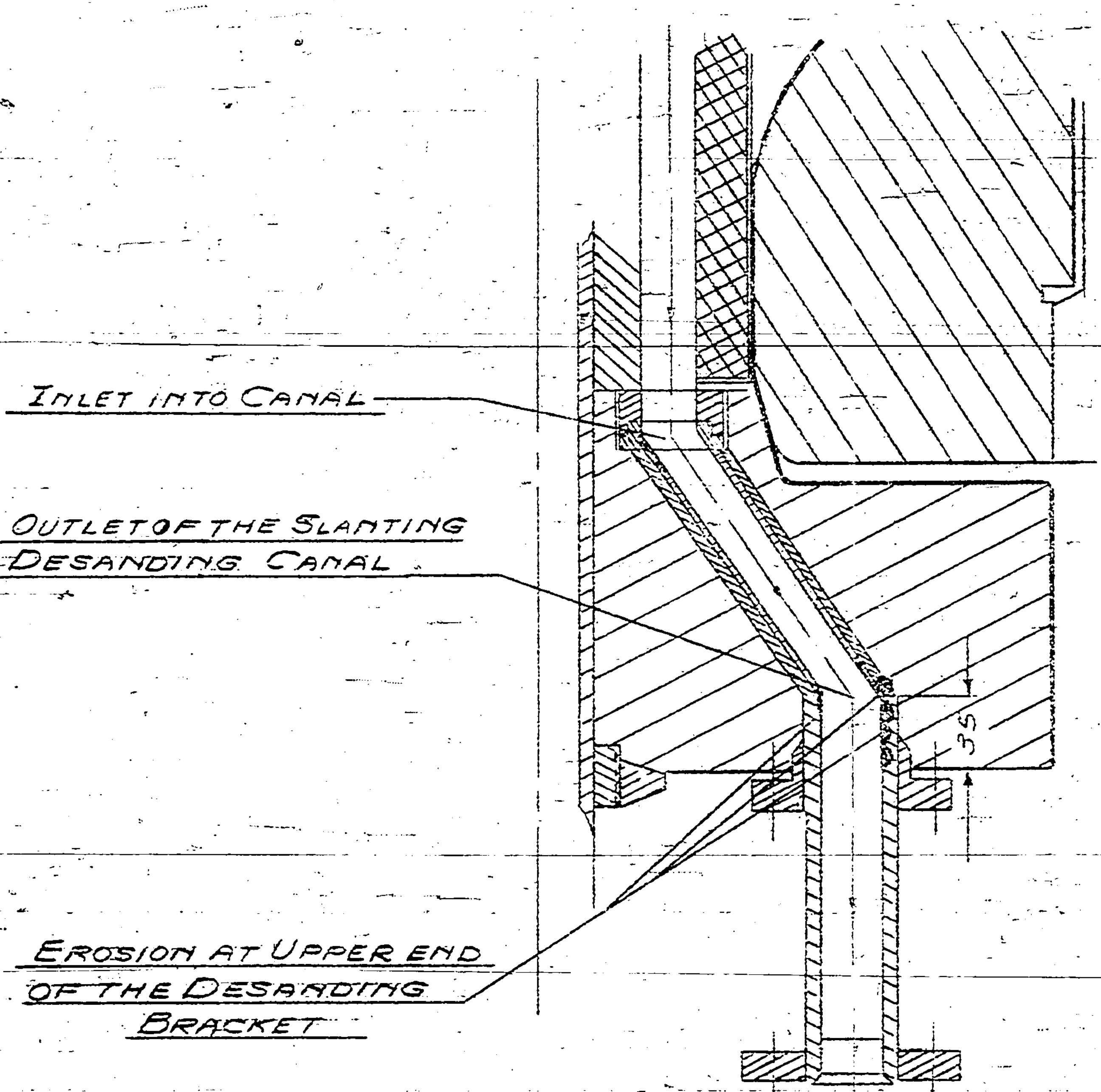
Definite reasons for the accident have not been discovered even after a thorough investigation. The break may have started at the sheet metal ring at the blades opposite to the rotor disk. Two sheared off seams have been found, which showed plainly, that they have been previously repaired; however, the place at which they were installed has not been determined. All the blades of the rotor have been stripped, which caused the whole circumference of the disk to become distorted. During the destruction, the rotor was thrown with such a force towards the suction side, that the coupling bearings were destroyed. The sudden braking of the rotor caused all the tongs on the motor side of the coupling to be sheared off, while the leather disks held.

This accident called for a testing of the whole accident prevention system, in order to avoid repetition of the damage in the future. This system consisted in that the blowers are overhauled in each stall overhauling, with the rotor replaced every time. The latter is then send to the materials testing office which issues a certificate of approval from the main testing office at Me 15. The testing is done magnetically and in part with X-rays.

It is however absolutely necessary that the main testing office, after making all the necessary repairs, should make a dynamic evaluation of the rotor. Such testing has now been arranged, for with the proper authorities. The rotor is to receive a stamp of approval only after the rotor has been adjudged perfect, and this is to be mentioned in the delivery certificate sent to us. This will give us assurance that the rotor can be unconditionally reinstalled. The rotor will be installed by us only after the receipt of such a certificate.

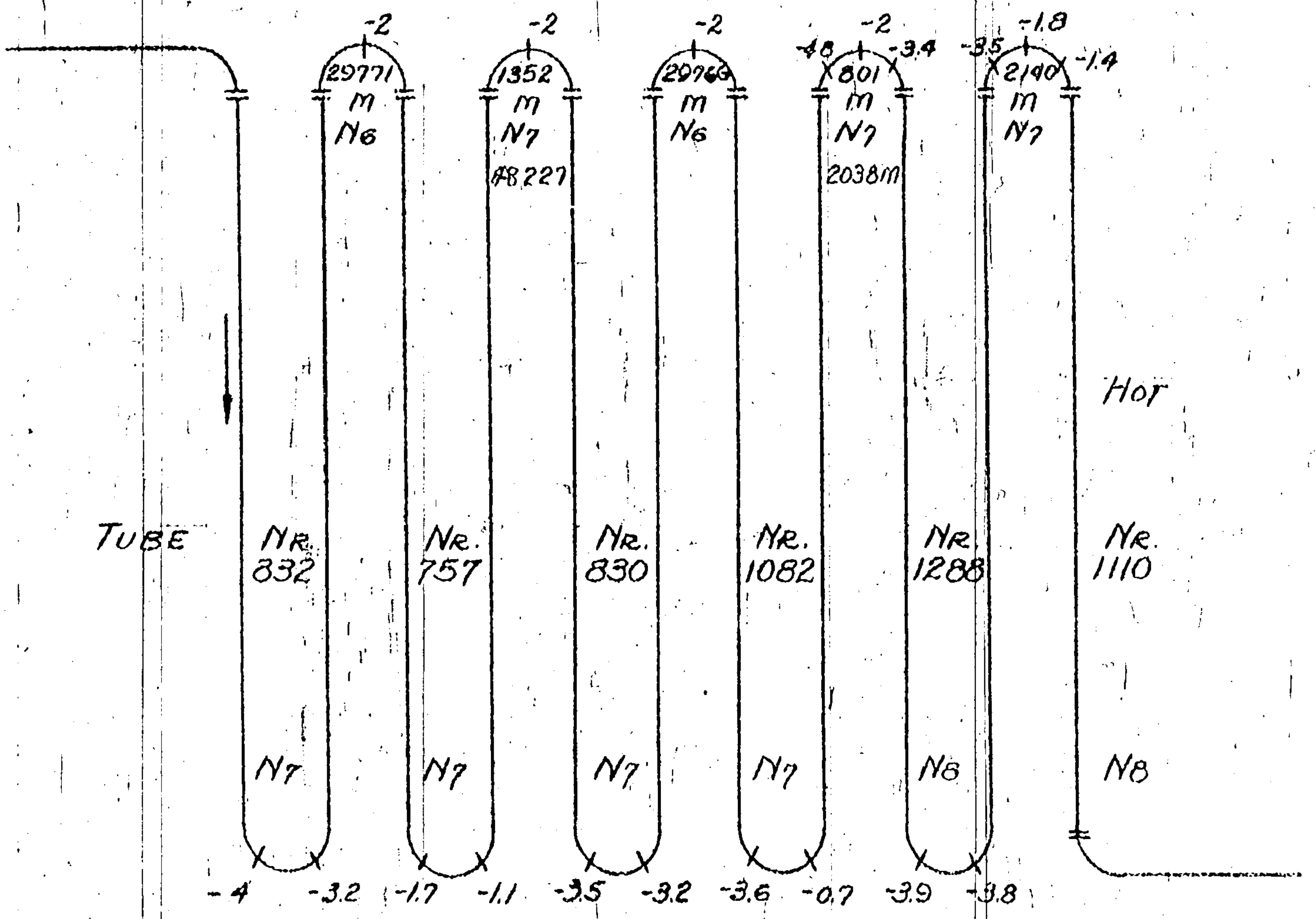
/s/ Schwab, Gassner, Cron.

T-165
Sketch-1

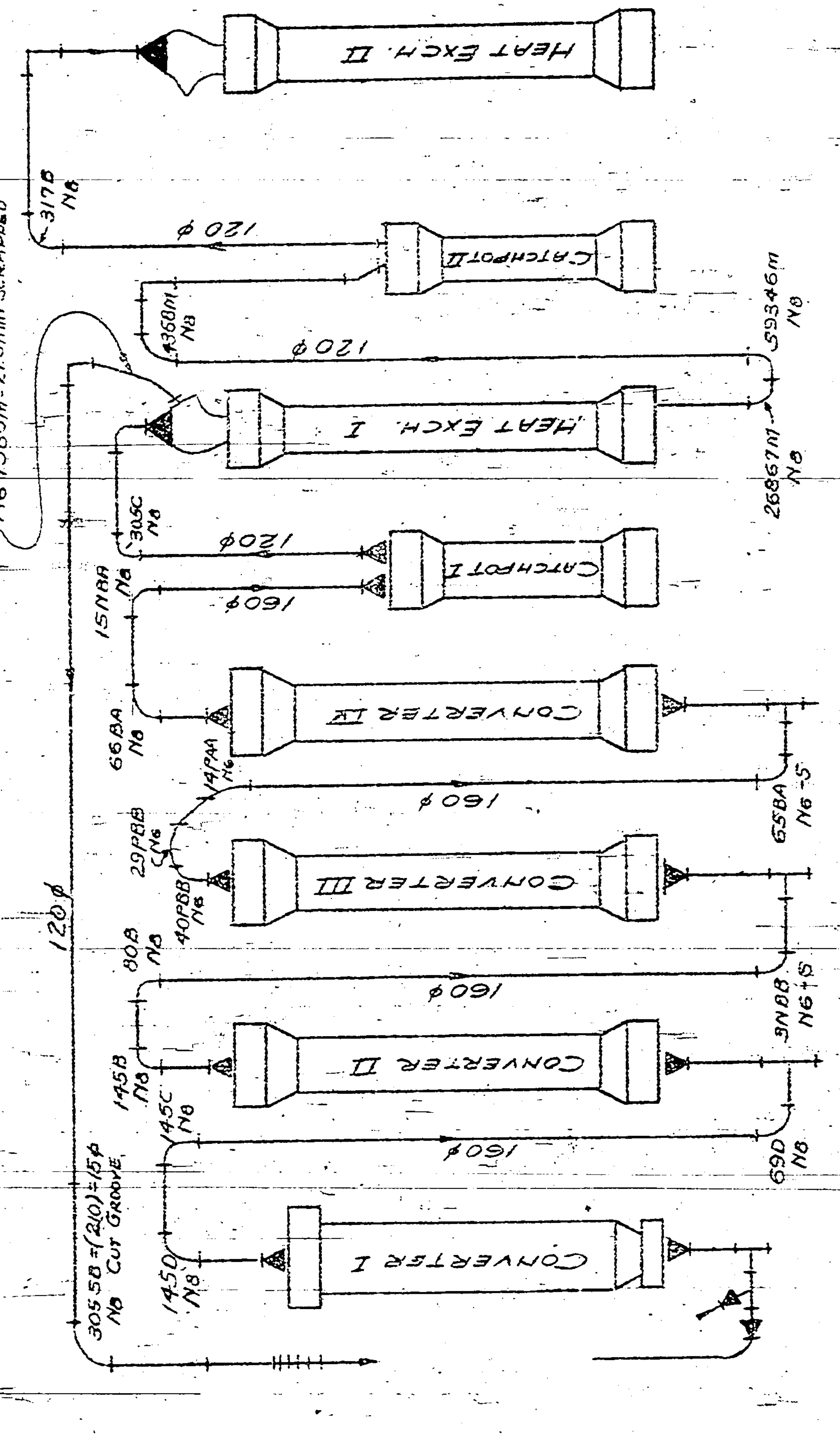


Desanding of Converter 1,
(Nr. 2564/599), of Stall 5, 3-29-41.

MILLIMETER REDUCTION IN WALL THICKNESS IN THE MIXTURE PASS (120 PINS & RETURN ELBOW) OF THE PREHEATER OF STALL 5 FROM 6/6/40 TO 3/30/41, (269 DAYS)



I-165
SKETCH -2



STALL 5

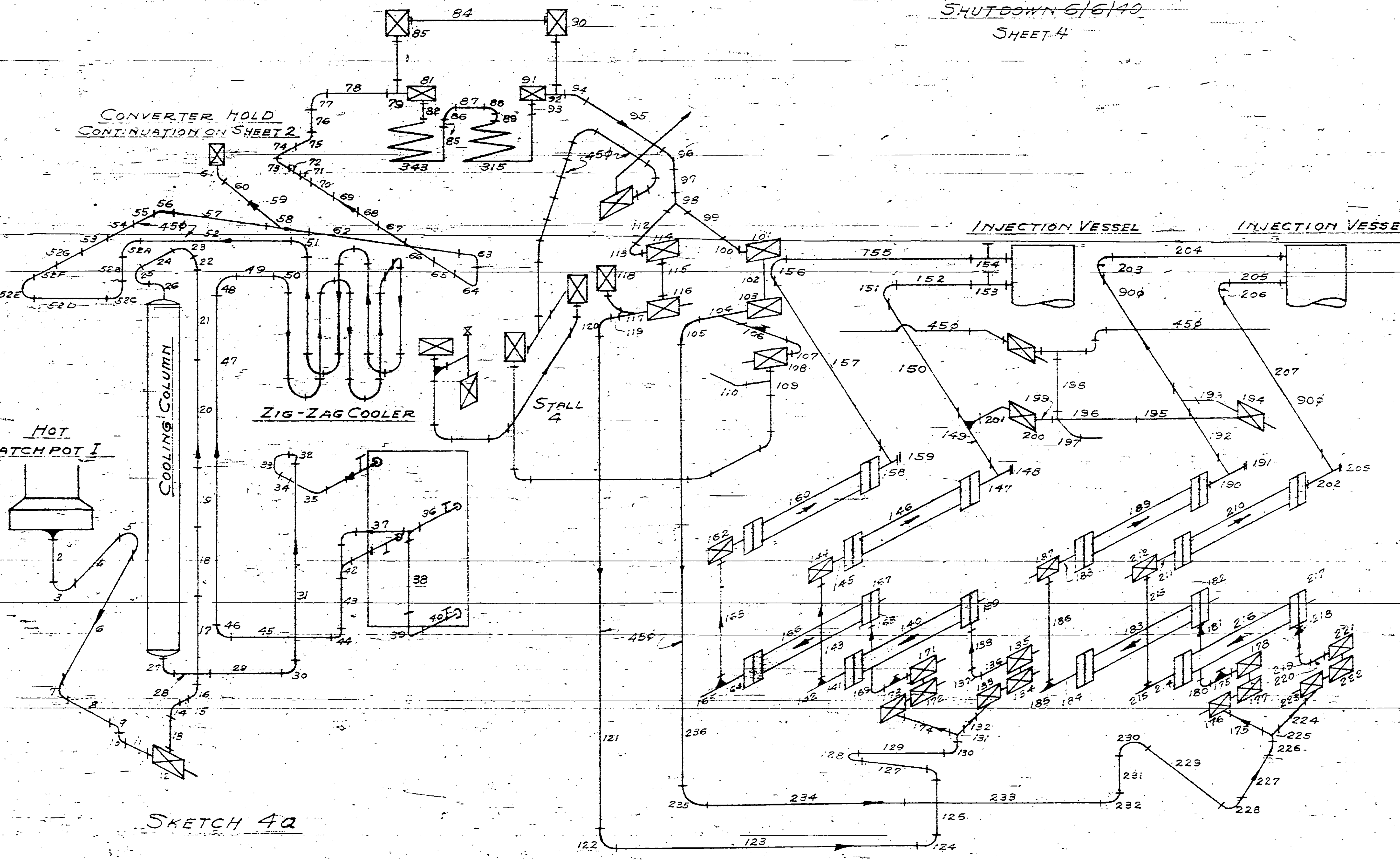
EQUIPMENT AND PIPE LINES (OPERATING PERIOD 6-6-40 — 30-3-41)

SKETCH 3

HOLD CATCHPOT STALL 5

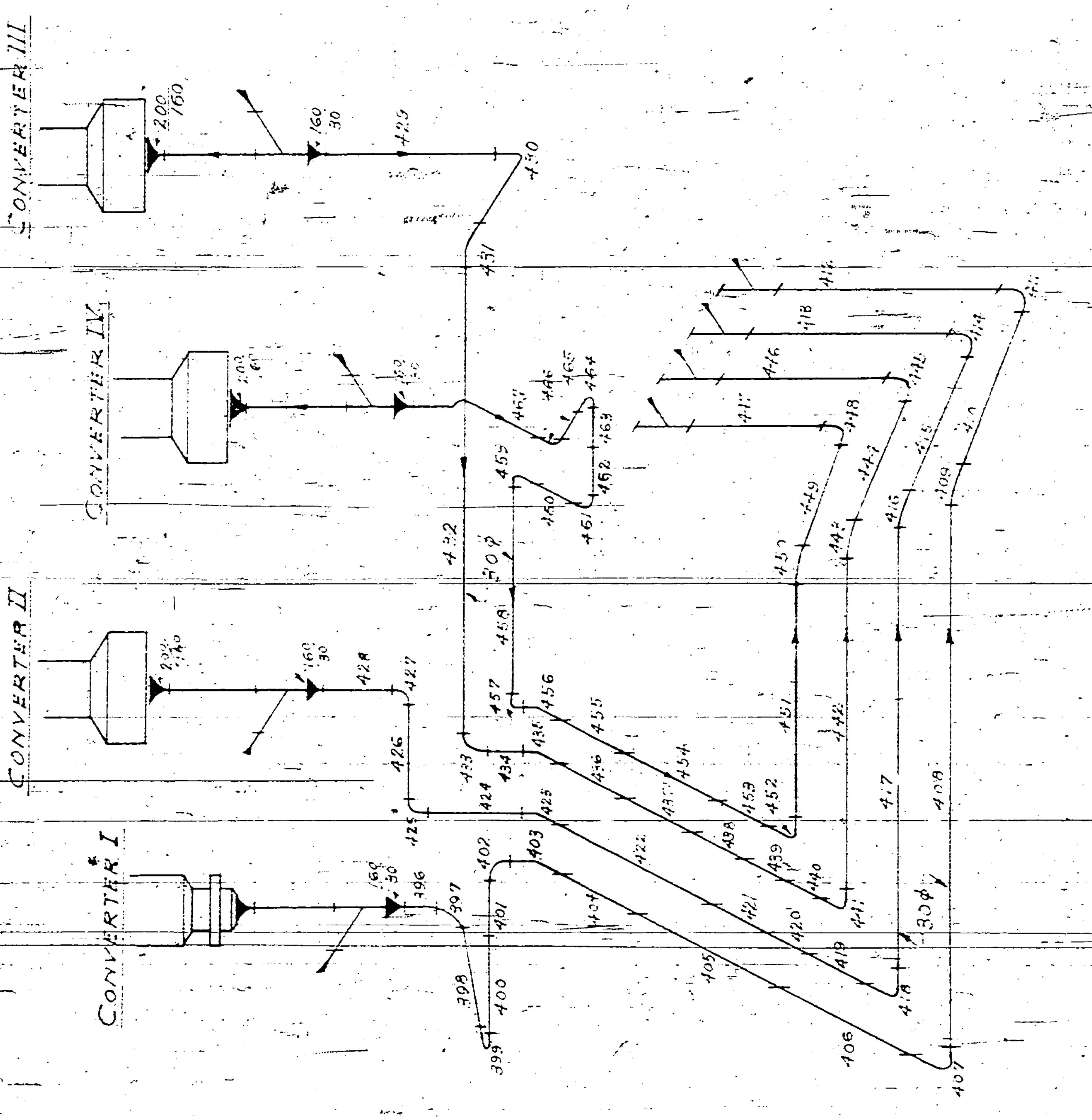
SHUTDOWN 6/6/40

SHEET 4



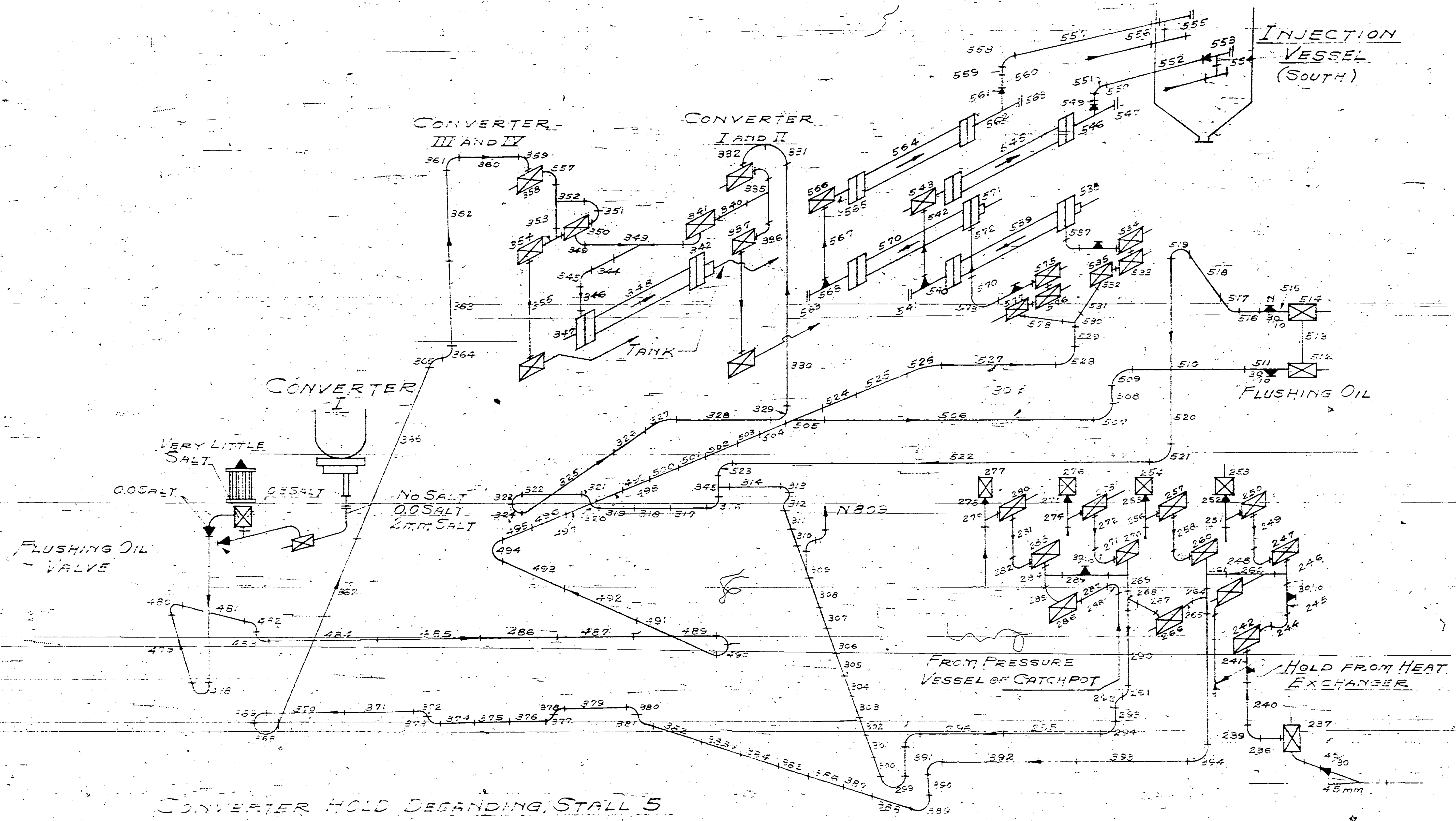
SKETCH 4a

T-165
SKETCH 4b



CONVERTER NO. 1, STALL 5, SHUTDOWN 6/6/40

T-165



CONVERTER HOLD DESANDING, STALL 5

SHUT-DOWN 6-6-40

SHEET 2

SKETCH 4C

	Kind of equipment	Delivered by	Operating Data			Temp. of Feed	Length Stroke	Dimensions of Plunger or Piston Rod			Diameter of rod at stuffing box	Length of Piston rod mm
			Pressure Atm.	Strokes	Feed			Plunger mm. diam. mm	Length of Plunger, mm	Piston Diam. mm.		
1.	Circulation gas pump, 30,000 m3/hour	Holberg at Ludwigs.	650-701	145	Impure circulation gas with hydrocarbons. Ammonia	35-80	320	--	--	140	60	2580
2.	Injection presses 10,000 li/hour	Schwarzkopf	700	6 double strokes	Injection feed of the liquid phase (tar, tar distill. catal., HOL)	110-130	600	170	1825	--	--	--
3.	Injection presses 5,000 li/hour	Esslingen	700	12 double strokes	Middle oil, vapor phase	35	600	88	1380	--	--	--
4.	Injection presses 16,000 li/hour	do	175	12 double strokes	coal paste	80-90	600	155	1380	--	--	--
5.	3 plunger pumps for water inject. 2,000 li/hour	Balcke, Rankelthal	700	95	condensate	30-40	200	30	625	--	--	--
6.	6 Stage gas compressor	Demag	700	125	pure hydrogen	ab. 160	5th, 6th steps =	4-th stage 200 5-th stage 150	1625	--	--	--
	4-6-th stages						500	6-th stage 65	1275	--	--	--
	6 stage gas condensor								1676	--	--	--
	4-th - 6-th stages	Standard equipment	325	125	"		1000	--	--	4 stage 255	165	--
7.	Booster compressor	Demag/Sultzer (standard machine)	700	145	"		500	90	1630	--	--	--

PUMP AND PRESS CONSTRUCTION MATERIALS - Continued

Delivered by	Cooling	Hardening	Nature	Stuffing Material	Box Oiling	Rinsing	Operating Experience
1. Krupp	Not cooled	Material FP 12 Bearing surface at stuffing box nitrided. Depth of hardening 0.5 mm	Ring Packing	Special bronze	Renania Ossag Ed 12	Stuffing box with machine oil	Foliation of the nitride sheet by too high temperature,/ stuffing box. Normal temper. 40-60°. Probably caused by presence of catchpot bottoms in circulating gas, with solution of machine oil and operating dry. To be started carefully!
2. Krupp	Cooled	Material: St C 60/61 Heat hardened FP 12, nitrided	Ring Packing	Zink Alloy			Temperature to be raised slowly, first with nitrogen, then with circulating gas. Experiments still in process
Mannesmann	Cooled	Material, Mannesmann	Götze packing	Tego bronze a. termite			No damage. Material satisfactory
3. Krupp	Cooled	FP 12, nitrided	Götze Packing	Termite	Fuel oil		No damage, have been found very satisfactory.
Mannesmann	Cooled	Mannesmann, Verbandstahl (1937, case hard					All broken! No longer in use.
Esslingen	Cooled	Electric cast iron, flanged.					No damage. Service satisfactory. Require grinding.
4. Mannesmann	Cooled	Mannesmann, Verbandstahl, case hardened	Götze Packing	Termite	Fuel oil		No damage. Have been found satisfactory.
5. Balcke	Not cooled	CV steel, hardened	Mouth rings	Leather and cast iron.	Fuel oil		Satisfactory in service.
6. Demag/ Krupp	Not cooled	St 70.11, not hardened	Ring Packing	Bronze	Compress. oil.	Compress. oil.	Good. No damage.
----	Cooled	FPE23, nitrided					
----	Cooled	FPE23, nitrided					
Demag	Not cooled	St 55, not hardened	Ring Packing	Bronze	Compress. oil.	--	Good. No damage.
7. Demag/ Krupp	Not Cooled	FPE 23, nitrided	Ring Packing	Bronze	Compress. oil.	Compress oil.	Good. Difficulties with oil.

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U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-167

Nitride Hardening of Piston Rods & Plungers

October 10, 1940

We are enclosing a report of October 5, 1940 on the experiences gained with nitrided plungers. We are sending you these with the express hope that you will place in our hands any replies you may get from the other works on this subject.

We wish to make some additional remarks:

A discussion has taken place on that subject between Krupp, the Material Testing office at Berlin-Dahlen, a few representatives of other firms, and ourselves, in which the great importance of strength and proper hardening of the base materials and the gradual transition of the diffusion layer was emphasized for the permanency of the nitriding. We have stipulated in this discussion the preparation and the experimental introduction of test rods. We understand the report was sent to you to deal with these test rods, so you may use them as examples of the most recent conditions of nitriding at that time. We have further observed that the frictional forces on the nitriding surface should not exceed some definite values because the layer would otherwise rapidly peel. This was the reason why plungers with the natural densest packing have at once been found satisfactory. Our experience cannot be directly transferred to other operation conditions and the stuffing box construction must be used in our considerations. In this respect, the Mannesmann rods are less sensitive. Communications to you cover our experience with nitrided steels of old composition. Such steels at present may be used only to a limited extent. It might, therefore, be recommended that the recently introduced nickel and molybdenum-free steels should be tested prior to your introducing them into larger operation tests.

/s/ Wolfram, Maier

WM Sternberg

11/19/46

Newman

TOM Reel 181
Fr. 06701 - 06702
Me 803/5

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-168

EXPERIENCE WITH NITRIDING PLUNGERS

Leuna Works, Oct. 5, 1940

The data for the middle oil pump 803/5 in Me are:

Capacity: 17 m^3/h
Stroke : 450 mm
R.P.M. : 100
Plunger : 46 mm, diam.

Two additional nitrided plungers have been obtained from Krupp, Nos. K1 and K2.

1. Operating period with K1 and K2, 46 mm diam., October 1937 to March 1939. Both plungers were installed with WM 10% stamped packing. After some 150 in 50 days, the hardened layer became loose and the surface became so rough that the plunger had to be resurfaced.

2. Operating period with K1 and K2:

K1 - 8/20/38 to 10/13/38
K2 - 8/1/38 to 9/12/38

The plungers were reground until a perfect, clean surface was obtained; the new diameters produced were:

K1 - 45.96 mm
K2 - 45.72 mm

The plungers were again installed with a new WM 10% stamped packing. Results:

Plunger	Diameter When Built	Days in Operation	Diameter When dismantled	Abrasion mm		Reason for dismantling
				Absolute	Per Day	
K1	45.96	15	45.78	$\frac{18}{100}$	$\frac{1.2}{100}$	Plungers roughened. Stuffing box leaked.
K2	45.72	40	45.60	$\frac{12}{100}$	$\frac{0.3}{100}$	Same as K1

During the resurfacing the hardened layer was lost and the plunger was returned to Krupp for re-nitriding.

3. Operating period, K1: 11/28/39 - still running
K2: 11/28/39 - 9/28/40

The re-nitrided plungers were installed with the Merkel self-tightening, soft packing. Results:

Plunger	Diameter When Built	Days in Operation	Diameter When dismantled	Abrasion mm Absolute	Abrasion mm Per Day	Reason for dismantling
K2	44.42	272	44.3	$\frac{12}{100}$	$\frac{0.04}{100}$	Packing used up. The plunger could be re-used.

The plunger has no roughness worth mentioning.

The second nitrided plunger has also been running for 270 days with the Merkel packing, is still in use and has so far operated perfectly.

Results obtained with the second nitriding must be called very good when compared with measurements, of which we have made a large number, on normal case hardened and hardened steel plungers. Abrasion with the last named plungers is on the average about $\frac{0.20}{100}$ per day, if self-tightening Merckel packing is used to protect the plunger.

A critical examination of the tests confirms the frequently made experience that perfect nitrided plungers possess excellent operating qualities, but the assurance of the production of perfect nitrided bearing surfaces remains yet to be desired. Case hardened plungers have been found so far more dependable in the preparation and produce better results than the nitrided plungers and we have so far given the case hardened plungers preference. Nevertheless, additional tests will be made with nitrided plungers.

/s/ Underwriters

W.M. Sternberg

11/20/46

Newman

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Fr. 06703-4

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-169

Nitrided Plungers & Rods

Ludwigshafen, July 4, 1941.

We return to your questionnaire of 5/26/41 and take the following stand regarding our experience with piston rods and plungers of different nature.

a. Nitrided Plungers:

Our tests with several nitrided plungers delivered by Krupp/Zapp for our middle oil pumps are shown in our report of 10/5/40. We have used no additional nitrided plungers since.

Nitrided Rods

Nitride hardened piston rods have not been used in our large circulating gas pumps for 100,000 m^3/h at 220/250 atm. Our experiences with nitride hardened piston rods are principally those of the double acting steam driven circulating gas pump made by Esslinger of 30,000 m^3/h capacity with the rods 65 mm in diameter. This machine was equipped with nitride hardened rods and been in operation since 1937. A total of three piston rods have been used. Two of these rods had been discarded at an early date because the surface was partly eroded and cracks appeared which were not overcome by regrinding within the tolerance of the diameter. The third rod has been found satisfactory. The wear of the rods, when equipped with ring packing, was about 5-10/100 mm after ten months' operation. The hardness had dropped by about 10% after regrinding to 64.5 mm diameter. The rod was kept in operation for about 13 months longer and was removed during general overhauling of the machine. The rod now had normal slight ridges. It was reinstalled after regrinding. We may mention that other piston rods ordered from our shops were supplied of S2 steel. They show satisfactory operating life.

b. Hardened Flanges. We have had no experience with hardened flanges.

c. Case Hardened Rods

1. Mannesmann case hardening method.
2. The usual case hardening methods.

We use plungers prepared by the Mannesmann process as well as some which have been delivered from our shops case hardened in the usual way. They have been successfully used in all our middle oil pumps, scrubbing oil pumps, flushing oil pumps as well as in the paste process. With our operating conditions and experience we must consider both processes as being of practically equally good. Of greater importance than the surface properties of the rods is the quality of the packing used and the properties of the feed with respect to quantity and nature of contaminations. Plungers made in Merseburg were hardened and tempered to Brinell hardness of 400-500.

Plungers of Chilled Casting

We may add that some of our paste processes have for years operated with chilled cast plungers. The results were good. We also have used 46 mm diameter plungers for middle oil injection pumps made of chilled casting. They have been found very satisfactory with metal packing as well as with self-tightening soft packing.

/s/ Ammiakwerk
Merseburg
Schmidt
Other signature illegible

W.M. Sternberg

11/20/46

Newman

TOM Reel 181
Fr. 06705

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-170

I G Ludwigshafen

Leuna, Apr. 30, 1942

Hardened Plungers & Piston Rods for High Pressure Machines

In reply to your letter of April 11, 1942 and to supplement our letter of April 7, 1941, we wish to inform you as follows:

We have not used in our gas compressor units rods of materials mentioned in points 1 - 4 of our letter. We have, however, made experiments with rods of chrome steel, hardenable carbon steel and nickel steel. Only the hardened rods made of the relatively soft Krupp material, E42 ONN, with about 1% nickel and iron and bare traces of chromium have been found satisfactory. However, such rods have not been ordered because of the long delivery time.

Otherwise we have completely departed from the use of hardened rods in our compressors because of their generally greater tendency to break especially in the threaded parts (larger surface sensitivity) than of the commonly used SM rods of the Guthoffnungshütte or Dortsmund-Hoerder-Hüttenverein made of union machine construction steel, a material of lower limit of elongation than has been used previously. The following summary will give you the information desired. The booster compressors for 325 atm. and the circulation pumps are fitted with nitrided piston rods of the Krupp special steel F.P.e 23 and F.P. 12, stroke 450 mm, n = 60-70, 80 mm diameter.

Piston rods are all fitted with ring packing lubricated with T.D.A.A. oil (compressor oil). The pressure is 325 and 700 atm. Our experience with them was very satisfactory. In operation for two years. Since 1936 two piston rods of Mannesmann Ferbundstahl had been used in the hardened state in the pressure release machines, diameter 120 mm, stroke 950 mm, 120 strokes per hour, (stuffing boxes, leather sleeves). The rods and the stuffing boxes are very satisfactory. The rods were not at all worn off. The leather sleeves also operate on the average one year and longer (middle oil). All the plungers of the injection pumps for methanol, 500, 1,000, 1,500, 2,000, and 3,000 liter capacity are case hardened (2-3 mm deep). They operate very well. For sometime two plungers in the 10 m^3 compression pumps, 85 mm diameter, 550 mm stroke, n = 60-70 have been made of material SII, case hardened.

All the stuffing boxes have special lip packing. We have as yet no data on the life of these stuffing boxes.

Ammoniakwerk, Merseburg

Two signatures (illegible)

W.M. Sternberg 11/20/46

TOM Reel 181
Fr. 06707-06[redacted]0
72

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T-171

Hardened Plungers & Piston Rods for High Pressure Compressors

I.G. Farbenindustrie A. G.

Gelsenkirchen,
July 7, 1942

Below is a summary of information on dimensions, materials and operating conditions, as well as our experience in the use of the piston rods and plungers in our works. The surface hardening of these parts and their resulting life and wear are of prime importance.

We give the life of parts which were in normal operation in uninterrupted use on May 1, 1942, that is, such which required no additional work to be done to them up to that time. Computation of averages from the large number of simultaneously operating parts would not alter the picture because uniform conditions make operations during equal and longer times probable.

We have not, however, omitted giving the data of the shortest and longest life of parts subjected to particularly great operational strains. Here average values were only computed when a sufficiently large number of average results were available. In all cases, the time given is for an uninterrupted use of a part. During that time no re-machining of the parts had been done.

Data on the wear are almost without exception for the longest life. Conditions are shown in the column marked "Remarks". Operational conditions effecting wear which differed for each type of machine could not be given in sufficient detail in the table. They will be discussed in the following paragraphs.

Our discussion is in the order of the questions in your letter.

1. Nitriding Hardening: Results obtained with nitriding hardening are in general dependable. However, one occasionally meets with wear resulting from peeling or from insufficient hardness. Factors effecting the adaptation of the process are the price, availability and the small number of nitriding establishments, the loss of material during rehardening and the soft body of the part. The layer is only 0.15 mm thick, can be readily pushed through, in regrinding usually loses very much hardness, and is broken through in rods which are not entirely straight. When renitriding, the diameter of the rod is reduced at least 1.5 mm, which frequently requires the putting in of new stuffing boxes. Nitriding offers most protection against corrosion. B-middle oil causes compact corrosion within the stuffing box.

2. Autogeneous Hardening: Autogeneous hardening offers the advantages of cheapness, the possibility of performing it in our own installations by a simple and readily mastered process, and a greater depth of hardening and corresponding life. Depth of hardening of 2-3 mm may be readily achieved and repeated regrinding may be done. Depending on the choice of steel sufficient hardness can be produced and peeling and cracking off of the surface may be avoided by proper treatment. A comparison of plungers and piston rods when nitrided and autogeneous hardened shows that the wear of the nitrided parts is mostly greater. Thus, nitrided plungers and rods of the circulation pumps for 700 and 300 atm. showed wear a few hours after the beginning of operations, which is not the case with hardened parts.

Autogeneous hardened parts possess a very uniform surface hardness in comparison with case hardened materials. It is free from inner stresses caused by quenching and these stresses have resulted repeatedly in breaks starting from the cooling water drilling holes.

It is frequently desirable to apply a more durable surface to the usual steel 5011. The improvement obtained here may be quite considerable as proven by the hardening of driving piston rod of paste press in which the hardness was increased from 150 to 410 Brinell. This increase is sufficient to eliminate the effect of the soft packing upon the piston rod. The process is equally well adapted for a foundation coating under a hard chromium coating. The compressor pump plungers shown in the table have failed because of outside influences such as the placing of the piston rings, displacement, or because of attack by corrosive liquids such as phenol water and B middle oil.

3. Case Hardening. - Case hardened plungers and rods have not been used by us.

4. Deep Case Hardening. (Mannesmann process). The results by this process are known. It is difficult to produce a uniform distribution of hardness. Parts drilled for cooling water are in danger of cracking. This results from the method of production and therefore inside drillings are best omitted. The operating properties and life are sufficiently good.

5. Chilled Casting, Alloy Steel, Hard Chromium Plated Steel, Hardened, Chromed and Naturally Hard Materials:

The experience with chilled cast plungers on paste presses of Maschinenfabrik Esslingen were very satisfactory. They are considerably cheaper than the Mannesmann plungers to buy, stand up well in operation and are delivered without delays.

The best results for phenol water has so far been obtained with alloy steel sonnit made by the Rheinmetall-Düsseldorf. Their life remains yet to be determined. Remainit plungers of the DEW in Krefeld of the composition so far selected have not as yet been found to be very corrosion-resistant and also are too soft. Tests with it are still continued.

The best properties for phenol water pumps have been exhibited so far by the recently developed hard chromium plated upon heat hardened metal. Their test in larger numbers and over longer operating times is as yet unavailable. A hard chromium plated Mannesmann plunger of a paste press has given exceptionally good service. However, this part made of chilled casting is also entirely satisfactory.

A test is being made at present of chrome steel with a hard chromium coating of plungers for phenol water. This material is made with the idea that a chromium layer desired for operating reasons, but not completely tight, would coat a base already containing chromium, which would result in reducing corrosion of such material. This type of plunger has at present been intended for the B middle oil.

Piston rods of all the compressors, the paste presses and pressure release machines are of hard steel. We may expect no important improvement in the operating properties in the compressors, but one may hope a great reduction of stuffing box repairs from heat hardening of the paste press piston rods and especially of the pressure release machine piston rods.

We may summarize as follows:

Nitriding hardening is one of the best processes for the production of wearproof surfaces. Case hardening and autogeneous hardening have been found satisfactory. Heat hardening in particular has been found a cheap process in numerous applications. Surfaces can be obtained by hard chrome plating which have the same or higher wear resistance as nitriding and excel in the depth of hardness. However, the heat hardening alone produces hard layers which have sufficiently good properties for plungers and piston rods of the booster compressors, gas circulation pumps, paste presses, compressors and possibly pressure release machines.

The possession of a hardening installation offers the advantage of production of most of the required equipment right at the works, and avoids delays in delivery from manufacturers. This is particularly important if a grinding machine is available. On assumption of rental from friendly works, the economy of such a combination is always assured. This arrangement must be supplemented with a shaft furnace in which parts could be heated to remove stresses before and after the machining.

To avoid distortion during hardening, a small stock of forged steels of VC135 and StC 4561 and similar water quenching steels must be kept. Experience gained with heat hardened plungers is that this kind of installation will be found sufficient.

W.M. Sternberg

11/20/46

Machine	Part	Material	Delivered By	Harden. Treatm.	Harden. By	Feed	Press. Atm.	Temper. °C	Stroke mm	Diam. of Rods mm	Speed of rods m/sec.	Surface	Brinell hardness	Penetr. of hardn. mm	shortest oper. time hrs.	Longest oper. time hrs.	Aver. oper. time	Hrs. oper. on 5/1/41	Wear mm	Pack.	Lubr.	Flush.	Remarks
Six-Stage Compressor	Piston Rods 1st stage	St.30.11	Halberg	Natural	-	Raw H2	3.5	100-130	1000	220	4.16	ground	150	thruout	-	-	-	16624	0.13*	Pazi-fik Ring	comp. oil	none	* 1664 operat. hours
	2nd "	" "	Essling	"	-	" "	11	"	"	220	"	"	"	"	-	-	-	"	0.12*			"	* " " "
	3rd "	" "	Borsig	"	-	" "	28	"	"	220	"	"	"	"	4481	-	-	"	0.3*		"	"	" " "
	4th, 5th & 6th	" "	Schwartzk.	"	-	Pure H2	90	"	"	160	"	"	"	"	6787	-	-	"	0.4*	"	"	"	" " "
Booster Compressor	Flanges	F.P.E.23	Krupp	Nitride harden.	Krupp	Pure H2	700	90	500	90	2.45	ground	730	0.15	2700**	-	-	11385	0.03*	"	"	comp. oil	* 6300 operat. hours **packing damaged by entrainm.of Cu-Soln.
Nitrogen Compressor	Piston Rod 3./4.St.	St.50.11		-	-	Nitrogen	245	115	400	80/120	2.66	"	150	thruout	1400	5600	3400	-	0.2-0.3*	"	"	none	* 5600 operat. hours
"	Plunger 5.St.	F.P.E.23	Krupp	Nitride	Krupp	"	900	Ab. 125	400	46	2.66	polished	730	0.15	1900	2600	2200	-	0.2-0.3*	"	"	"	* 2600 operat. hours
"	"	StC 6061	Ruhrstahl	Autogen.	Peddingh.	"	900	" 125	400	46	2.66	"	670	2	2200**	2535**	-	3100	0.08*	"	"	"	* 3100 " " **change in packing
Blending	Piston Rods 1.St.	W40 extra	Ruhrstahl	Natural Hardness	-	Gasoline blend	5.5	95	750	140	3.68	ground	180	thruout	-	-	-	13000	0.08-0.13*	"	motor oil or emulsion		* 13000 operat. hours
"	Piston Rods 2.St.	" "	"	" "	-	" "	35	116	750	140	3.68	"	180	"	4600	7600	5500	-	0.16-0.25*	"	"	"	* 7600 " "
Gas Circulation Pump 300 atm.	Piston Rod	StC 1061	Mannesmann	Case Hard.	Mannesmann	Circulation gas	325	25	500	110	2.03	"	620-780	2-5	4413	11000	8500	-	0.14*	"	saturtd. steam oil	none	*11000 " " trouble with packing
		W60 extra	Ruhrstahl	Autogen.	Grisogen	" "	325	25	500	110	2.03	"	620-670	2-5	-	-	-	4500	0.1*	"	"	"	*4500 operat. hours
" "	" "	Mn.5 V extra	Hermig	Natural Hardness	-	" "	325	25	500	110	2.03	"	230	thruout	159**	-	-	-	-	"	"	"	**removed because of ridging
Gas Circulation Pump 700 atm.	Piston Rod	F.P.E.23	Krupp	Nitride	Krupp	Circ. Gas	700	40	500	100	2.03	"	730	0.15	7571	8650	8500	9900	0.26*	"	"	mach. oil	*8650 operat.hrs. Removed because of chg. packing
	Plunger	FPE 23	"	"	"	"	700	40	500	170	1.73	"	730	0.15	974	7940	5000	-	0.22*	"	"	"	*7940 op.hrs. packing changed
		StC 6061	Rheinmet.	Autogen.	Peddingh.	"	700	40	500	170	1.73	"	620-670	2-3	-	-	-	4800	0.08*	"	"	"	*4800 op. hrs.
Pumps for Copper Solution	Plunger	F.P.E.23	Krupp	Nitrided	Krupp	Copper solution	325	30	310	80	1.03	ground	730	0.15	600**	4017	2000	-	0.8*	chrome leather	none	drip. of ammon.water	*Aft.4017 hrs use **Because of chg. of packing
	"	StC 6061	Boch.Ver.	Autogen.	Peddingh.	" "	325	30	310	80	1.03	"	670	3-4	400	1400	1200	-	0.6*	"	"	"	*1400 hrs.use
A middle oil pumps	"	F.P.E.23	Krupp	Nitrided	Krupp	A mid.oil	325	40	310	80	1.03	"	730	0.15	4635**	-	-	19000	1.2*	Burgmann-Sola G6	"	drip.of wtr.	*19000 hrs.use **Temp.f/B mid. oil use
B middle oil pumps	"	"	"	"	"	B " "	325	40	310	80	1.03	"	730	0.15	350	1100	700	-	0.6*	"	"	"	*Replaced after 1100 use because of wear
"	"	StC 6061	D.H.H.V.	Autogen.	Peddingh.	" "	325	40	310	80	1.03	"	620-670	2-3	-	350	-	-	0.4*	"	"	"	*After 350 hrs. use repl.for wear
Washing Oil [illegible]	"	F.P.E.23	Krupp	Nitride	Krupp	Wash.oil	700	30	310	58	1.03	"	730	0.15	700**	2200	1600	-	0.4*	",G	"	"	*2200 hrs. use **Because of chg. of packing
Pump for Condensate	"	Sonnit hard	Rheinmet.	Heat hardened	Rheinmet.	Condensate	325	60	220	55	0.6	"	500-550	thruout	5052	6000	-	-	0.05*	" "	"	none	*Taken out after 6000 hrs.use(pac
	"	Remanit 1540	D.E.W.	" "	D.E.W.	"	325	60	220	55	0.6	"	315	"	332**	-	-	430	0.07*	" "	"	"	*430 hrs.use **Taken out because of grooving
	"	StC 6061	Boch.Ver.	Autogen.	Peddingh.	"	325	60	220	55	0.6	"	620-670	2-3	-	-	-	25	-	" "	"	"	

o.	Machine	Final press. atm.	(Stroke) R.P.M.	Stroke mm	Rod speed m/sec.	Diam. of plunger or rod	Capac. (IG) m^3/hr.	Stuffing Boxes Feed	Matl. or Kind	Lubric.	Flush.	Material piston rod of plunger	Deliv. or Manuf. by	Results
1	Unit compressor 1st stage	2.5	128	1000	4.28	220	13000	Contact gas	Pacific	Michalk. oiler	-	St55	Schwartzkopff #1,2,3,9, 10,11,12	Satisfactory
	2nd stage	11.0	128	1000	4.28	220	13000	"	"	"	-	St55	Borsig #4, 5	"
	3rd stage	28	128	1000	4.28	220	13000	"	Ring packing	"		-	Esslingen #6,7,8,13,	"
	4th stage	80	128	1000	4.28	122	10000	"	-	-	-	St50	14 Ehrhardt & Schmeer #15	Life is shortened when the temperature rises as a result of drop in pressure from 6th stage
	evener stage	80	128	1000	4.28	160	10000	"	"	"	-	St50		
	5th stage	195	128	1000	4.28	263	10000	"	-	-	-	St50		
	6th stage	320	128	1000	4.28	122	10000	"	-	-	-	Piston G.E. 2291 with 14 piston rings		
2	Nitrogen compressor made by Sürth 1st stage	2.6	108/ 160	500	1.8/ 2.67	-	1200	80% N_2 20% CO_2	-	-	-	St60.12	Sürth	
	2nd stage	20	"	"	"	-	1200	"	-	-	-	"	"	
	3rd stage	90	"	"	"	100	1000	N_2	Ring	Michalk. oiler	-	"	"	Normal life
	4th stage	320	"	"	"	60	1000	"	"	"	-	"	"	Life limited
	5th stage	750	"	"	"	35	1000	"	"	"	-	"	"	"

									Stuffing Boxes					
No.	Machine	Final press. atm.	(Stroke) R.P.M.	Stroke mm	Rod speed m/sec.	Diam. of plunger or rod	Capac. (IG) m^3/hr.	Feed	Matl. or Kind	Lubric.	Flush.	Material piston rod of plunger	Deliv. or Manuf. by	Results
3	Booster compressor 700 atm. (unit comp.)	725	147	500	2.45	90	10250	H_2 35-40 °C	Ring	Steam saturat. cyl.oil	Oil 4 °E, 50 °C	Nitride steel	Krupp	Max. 6580 hrs. (Still in use) Av.5000 hrs.*
4	Gas Circulation pumps (Schwartzk.)	315	122	360	1.45	248	75000	25 °C Aver. Cir. gas	"	" "	-	a)Mannesmann Verbundst.	Mannes.	Max.4730 hrs. (still in use) Av.3000 hrs.*
												b)St 60.11 not hardened	Schwartzk.	Max.2870 hrs. (still in use) Av.2200 hrs.*
5	Gas Circulation pump unit	730	104	500	1.73	170	57000	Circ- 50-60°	Ring packing 82,80, 75,70, 60 & 75,75,75 70, 60	" "	4 °E, 50 °C	a)Plunger nitride steel	Krupp	Max.4705 hrs. (still in use) Av.2000 hrs.*
	Halberg model	730	122	500	2.035	210	69300	"	82,80, 75/70 70/60 70/60	" "	"	b)Piston rod of nitride steel	Krupp	3321 hrs. (still in use)
	Rebuilt unit compressor	730	104	500	1.73	190	72300	"	"	" "	"	c)Piston rods Mannesmann Verbundst.	Mannesmann	The Mannesmann piston rods in use since only recently
6	Tripple compression pump Esslingen 350 atm.	350	100	350	1.17	73	25	Mid.oil 6434 25-35 °C 5058	Goetze-6 rgs. Weissnetall w/comp. rings	-	-	a)Mannesmann Verbund.	Mannesm.	Max.4200 hrs. (damaged by cor Now 3000 hrs.*
												b)Nitride steel	Krupp	As with Mannesmann plunger, hardness unimp. at 325 atm.

No.	Machine	Final press. atm.	(Stroke) R.P.M.	Stroke mm	Rod speed m/sec.	Diam. of plunger or rod	Capac. (IG) m^3/hr.	Feed	Stuffing Boxes			Material piston rod of plunger	Deliv. or Manuf. by	Results
									Matl. or Kind	Lubric.	Flush.			
												c) Chilled casting	Essling.	The wear of packing is greater because chilled cast plunger is more attacked by feed
												d) Hard chromium coated chilled cast pl.	"	Results yet unknown, 3 pcs. in use since early July 1942
7	Triple compression pump Balcke 350 atm.	350	100	210	0.7	48	6	mid.oil 6434 25-35°C 5058 50°C, both with H_2S	(Goetze)	steam sat. cyl.oil	-	Mannesmann Verbund.	Mannes.	Max. 5397 hrs. (still in use) Av. life unknown machines little used
8	Triple comp.pump Balcke 350 atm.	"	"	"	"	"	"	Condensate 60-70°C	"	"	-	"	"	Max. 8725 hrs. (still in use) Av. 5000 hrs.*
9	Triple comp.pump Esslingen 350 atm.	"	"	350	1.17	73	25	Copper soln. 28-30°C	Chrome leather sleeves	NH_3 water	-	A) nitride pl.	Krupp	
												b) Mannesmann Verbund.	Mannes.	Plunger life 8000-10000 hrs.
												c) Chilled cast pl.	Esslingen	

o.	Machine	Final press. atm.	(Stroke) R.P.M.	Stroke mm	Rod speed m/sec.	Diam.of plunger or rod	Capac. (IG) m^3/hr.	Feed	Stuffing Boxes Matl. or Kind	Stuffing Boxes Lubric.	Stuffing Boxes Flush.	Material piston rod of plunger	Deliv. or Manuf. by	Results
0	Triple comp.pump Esslingen 750 atm.	750	100	350	1.17	52	12.5	Wash oil 30-40°C	Burgmann - Sola Rope	-	-	Mannesmann Verbund.	Mannes.	Packing max. 500 hrs. Plunger removed for machining after 2500 hrs. with 0.3-0.5 mm wear *
									Goetze's with steel press.rings	Steam sat. cyl.oil	-			Av.packing 600 hrs. Tests not completed
1	Triple comp.pump Esslingen 750 atm.	"	"	"	"	"	"	Tar min. oil resid. 70°C	Goetze - Tego III 6 rings with steel press.rings	"	-	a) Mannes. Verbund.	"	Max. 22300 hrs. normal 1500-2000hrs. experience limited because of many mach. & little injec.; mach.frequently interchanged.
												b) Chilled casting Esslingen	Essling.	No results as yet available
2	Triple comp.pump Balcke 750 atm.	"	"	210	0.7	33	3	Phenol water 60-70°C				a) Mannes. Verbund.	Mannes.	Max.5300 hrs. (still in use) Av.2600 hrs.* wear through corrosion
												b) Remanit c) Sonnit		No results as yet because no deliveries have been made
3	Paste press 600 mm stroke 750 atm.	700	6 Dbl. strokes per min.	600	0.12	170	10	Thick & thin cold paste, pasting oil, producer tar	Tego sl. rings	Rinsing oil only		Mannes. Verbund.	Mannes.	Max.7800 hrs. (still in use)

o.	Machine	Final press. atm.	(Stroke) R.P.M.	Stroke mm	Rod speed m/sec.	Diam. of plunger or rod	Capac. (IG) m^3/hr.	Feed	Stuffing Boxes			Material piston rod of plunger	Deliv. or Manuf. by	Results
									Matl. or Kind	Lubric.	Flush.			
4	Paste presses 400 mm stroke 750 atm.	700	9 dbl. strokes per min.	400	0.12	110	4	Flush. oil HOLD & Catal.	Tego sleeves soft packing lip rings only	None with flush. oil		Mannes. Verbund.	Mannes.	Max.6400 (still in use) Max.7400 hrs. (still in use) Av.4750 hrs.*) Strong wear by solids in feed & by crusts & foreign substances, material improvement since introduction of sieves.)
5	Expansion machines 500 mm 700 atm.	700	70	2650	0.62	500	65	Wash. oil	Leather sleeves	Sat. steam cyl.oil-	-	Nitride steel	Krupp	Packing Av. 2000 hrs. Rods 7000 hrs. So far no stop
6	Expansion machines 650 mm 300 atm.	325	65	2800	0.61	650	120	Copper solu. 30°C	Chrome leather sleeves	-	-	"	"	Packing 1200-1400 hrs. Rods 8400 hrs. (still in use), so far no trouble

*) The numbers represent average running time up to first remachining.

TOM Reel 130
Pages 612-622

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-172

Results of Tests of N10 - Leuna, 1943

Leuna - March 8, 1943

The composition of N10 is:-

C	Si	Mn	Cr	Mo	W	V
0.19-0.25	0.4	0.25-0.50	2.5-2.7	0.35-0.45	0.30-0.45	0.70-0.85%

The amounts of Ni and Cu are not fixed, but they should not exceed the usual limits of about 0.4% Ni and 0.3% Cu.

The heat treatment consists of quenching from 1020-1050°C either in oil or in air followed by annealing to the required hardness at temperatures of 690-730°C. The standard for hardness after annealing with air hardening is 220-260 Brinell, and with oil quenching 240-280 Brinell.

A sufficiently rapid cooling is necessary during the quenching of the material since otherwise a sufficient hardness of the material is not obtained in quenching and subsequent annealing produces no ductility. Brittleness measured at room temperature of a poorly hardened piece of N10 is no longer found, however, at the operating temperature of the tube, around 500°C.

The creep strength of the test pieces found with different annealing stages is shown in Figure 1, (not available; tr.) The values plotted were found by the usual method of determining the creep strength, that is, the limiting strength at which the creep velocity is exactly equal to 1/2% per hour on the 1000th hour or later. This will result in smaller values in comparison with the more usual D.V.M. definition (10 x 10^{4}% per hour on 25-35th hours).

The large difference between the oil and air quenched materials is to be noticed. The creep strength of the air quenched material at 500-600° is about double that of the oil quenched. The special creep strength of the N10 in test rods is therefore an air hardening effect. The diagram in general shows that at 500-600° we are working in the steeply descending part of the curve, that is, consideration of strength alone indicate that we are operating in a boundary field.

The resistance of N10 to hydrogen is another one of its important properties. The earlier 200 hour tests with test rods, 5.5 mm diameter, exposing the rod to hydrogen under

700 atm. at 560°C, showed no changes, that is, the contraction which is the most sensitive indicator for attack by hydrogen was either not changed or else so little changed as to justify considering the changes within the limits of accuracy of the measurements. In agreement with this, the microscopic structure disclosed no effects of an attack by hydrogen. Bending tests in which samples were bent over 180° disclosed no changes in the surface.

This knowledge of N10 left us no suspicion that this material for hydrogenation at high pressure and at temperatures in excess of 500°C was trusted altogether too much.

All the earlier damage found in tubes sent for determining the causes of failure of the N10 material could be explained by faulty hardening, or local soft spots. In April 1942 and in the beginning of 1943 tubes have began to come which broke in operation, and which had some grain separation manifestations which had never been observed before.

One tube shows on the inside wall first a very narrow zone with partially decarbonized crystallites, next a zone with grain boundary cracks, which penetrated up to 9 mm into the material. Mechanical properties measured in the wall of that tubing are shown in fig. 3. We may see from the table, that the properties of the tubes are perfectly normal outside of the affected inner zone, and that even the material so thoroughly attacted in the inner zone could be improved to a surprising extent by rehardening.

An other tube showed on the inside a thin layer formed during use; then a zone about 0.2 mm wide of partly decarbonized steel with evidence of grain separation, then some grain separation cracks, which penetrated however in this case only about 1 mm into the material.

Tests on that tube showed in agreement with metallographic observations that the zone embrittled in use extended barely 1 mm into the material. The mechanical properties of all the other material back of that zone was unaffected by use. The bending tests of samples from the outer and middle zones were somewhat poorer than of samples at a distance 1 mm from the inner edge, but this could not be attributed to the effects of operations, because good ductility was found only in samples 1 mm from the inner edge. It must have been some irregularity present in the original tube.

This damage produced in the tubes during use has caused subjecting the N10 material to more exhaustive tests, in part using the earlier results in order to determine the causes of the grain separation.

These tests have been extended to 1000 hours, and they showed rather surprisingly that some of the samples treated under no tension atm. with hydrogen may suffer a distinct reduction in contraction, even if the samples have not changed any after the first 200 hours. Never the less, examinations of structure of samples which had an appreciable reduction in contraction indicated no effects of attack by hydrogen.

This effect of the hydrogen undetectable in structure changes may be considered a physical effect, in contradiction to the normal effect which consists in a destruction of the carbides; the diffusion of hydrogen must have accelerated the changes of spatial locations of the atoms.

It is not possible to draw conclusions from the creep tests on the suitability of the material for tubes (after 1000 hours) and the behavior of pieces under no load in an atmosphere of hydrogen, which has forced us to supplement the former tests on rods with tests on tubes filled with high pressure gas in order to establish the permissible load on the tubes. Tests were carried out to the breaking of the tubes. The length of time they stood up gives then a measure of the worth of the tubes. Tubes tested with nitrogen gave the true resistance capacity of the tube material under the combined effects of temperature and pressure, those tested with hydrogen, were affected by all the factors which affect the tubes in use.

Because of the large amount of time required, the tests are by no means concluded, nor can the tests be in any way shortened when wishing to obtain accurate information on the behavior of the tubes under operating conditions. Most of the air quenched tubes were naturally run with hydrogen. Oil quenched material and tubes were at first only occasionally tested with nitrogen. The principal conclusions to be drawn so far from the tests are;

1.) operating life of the tubes tested with nitrogen is much higher than of the tubes tested under hydrogen.

2.) shorter life of tubes tested in hydrogen must be attributed to grain separation cracks.

These grain separation cracks must be considered as general phenomena. They start at the inside of the tubes, where the hydrogen pressure and the mechanical stresses are the greatest.

The results of all the tube tests were plotted on fig. 8 to permit drawing the proper conclusions. The logarithms of the operating times were plotted on the abscissa, on the ordinates a value Z calculated from the tube dimensions, the inside pressure and the creep resistance after 1000 hours test. The value was selected to give comparable values for all the tubes run at different temperatures, having different hardness, and of different dimensions. The value Z refers to the incipient flow on the more strongly affected inner fibers, and if all the assumptions of the calculations were correct, all tubes with a value of Z equal to or greater than 1 should last forever, while all the tubes with Z values less than 1 should creep or flow. If Z is but little less than 1, the creep of the tubes would be but slow, and breaks would only occur after much time. With a smaller Z, say equal to 0.4, there must be a pronounced flow of the tube, which would have resulted in a break after a relatively short time and with strong deformation.

Should a break during the test occur in a tube with a Z value greater than 1, some other damage, unrepresented in the calculation, must have taken place. Such a damage may be the result of physical effect of hydrogen upon premature grain separation cracks.

Tubes with Z values less than 1, and which have not broken during the test mean that the creep strength used in the computations is no proper unit to be used in computations, or else that the stiffening effect of the many axed strains of the tubular form has been effective.

The meaning of the individual signs and numbers will clear from the table proper. The actual time of operation is referred to 1/17/1943 as the zero day. One of the most important things in the diagram is that the air quenched N10 tubes operated and broken under the internal pressure of hydrogen are located surprisingly accurately on a straight line slanting upwards, which is the more significant, as tubes of different batches of steel were tested. As is stated above the value Z tells that:

3.) N10 will stand up under hydrogen the longer, the higher the annealing hardness and the lower the operating temperature.

The effect of the annealed hardness is shown on a tube with a hardness of 302 which had been kept in operation for 3000 hours and still had not broken. If the straight line were applicable to times in excess of 10,000 hours, it would last some 30,000 hours. One may also see on a tube with a hardness of 223, which is at present considered still permissible, that one may expect a break after 1,500 hours at 560°C. The nitrogen tube with a hardness of 241 has been run at 600° C and is still being tested.

There appears to exist an improvement of hydrogen durability in the oil quenched tubes in comparison with the air quenched tubes tested with 700 atm. inside pressure. This would contradict the data on 1000 hours creep strength. The few experiments obtained with tubes should be confirmed by additional experiments. Should the above statement be confirmed, oil quenching would have to be preferred to air quenching. There can however be as yet no question of oil quenching of the bulky welded tubes without some basic changes in shop practices, and air quenching must still remain in use for N10.

No changes in alloys should be made without first subjecting the tubes to long time tests. To make such changes on the strength of tests with rods only would be assuming great risks.

Summary. Experience with tubes broken in operation and with tubes tested with an inside hydrogen pressure tell us that under operating conditions tubes are used so near their limiting operating properties, that after sometime which will depend on the quenching hardness, grain separation will occur at the inner zone. Tests have further shown that the tubes can to a great extent be reconditioned when not subjected too long (not over 10.000 hours) to the maximum strains.

/s/ Ziegler, v.Fehner.

W.M.S.

MECHANICAL PROPERTIES

Condition	Loc. of Sample	Yield Point	Tensile Strength	Elongat. %	Contraction. %
As delivered	a	63-71	74-83	13-40	34-58
	b	68-72	85-86	11-13	30-47
	c	64-74	85-87	11-13	55-57
	d	60-74	82-84	8-11	47-57
Hardened	a	55-60	68-72	12-16	47-62
	b	52-66	72-80	14-15	72-74
	c	54-63	69-73	16-18	72-74
	d	48-60	60-73	12-15	71-78

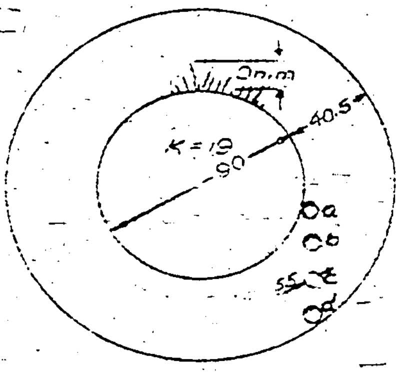


Figure 3

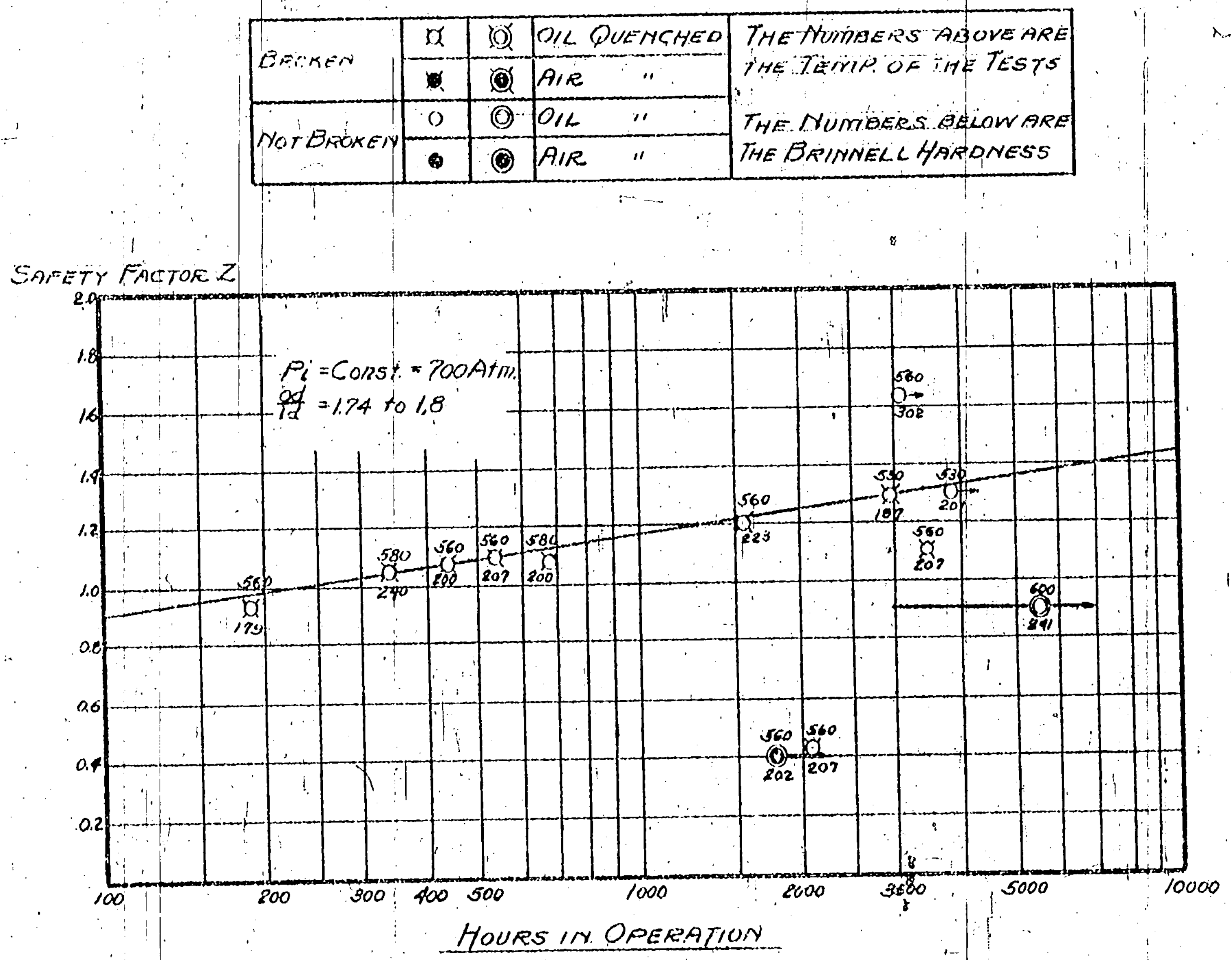

BROKEN
NOT BROKEN
OIL QUENCHED
AIR "
OIL "
AIR "
THE NUMBERS ABOVE ARE THE TEMP. OF THE TESTS
THE NUMBERS BELOW ARE THE BRINNELL HARDNESS
SAFETY FACTOR Z
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
100
200
300
400
500
1000
2000
3000
5000
10000
HOURS IN OPERATION
Pi = Const. = 700 Atm.
od/id = 1.74 to 1.8
560
179
580
240
560
200
560
207
580
200
560
223
530
197
530
201
560
207
560
302
600
241
560
202
560
207

FIG. 8

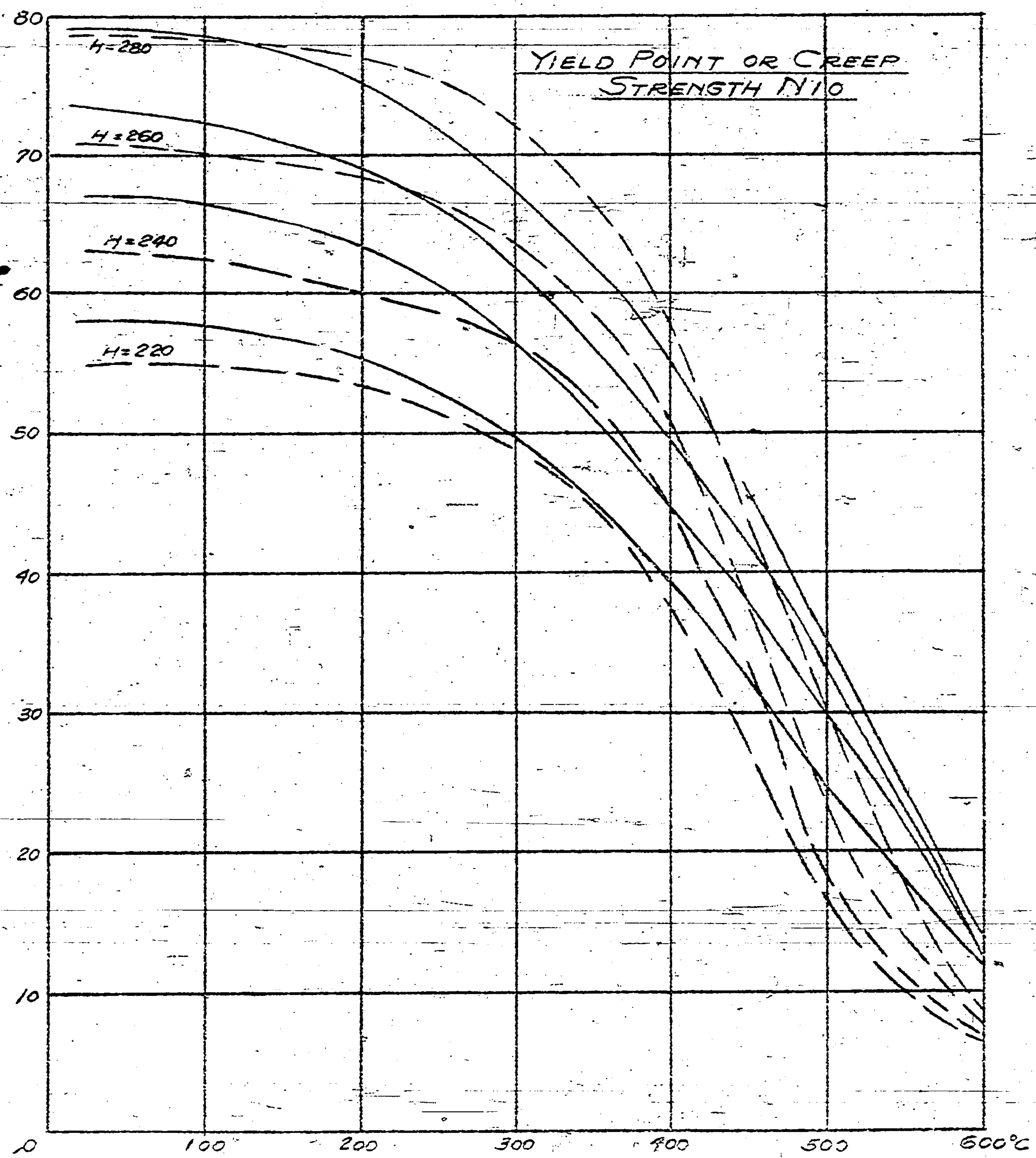
Figure 10

CONDITION:

BASED ON CREEP STRENGTH

——— AIR QUENCHED 1040-1080°C, COOLED IN AIR 25°/MIN. DIFFERENTLY ANNEALED DEPENDING ON HARDNESS

– – – – OIL QUENCHED 1000-1080°C, DIFFERENTLY ANNEALED TO HARDNESS



TOM Reel 126
Pps. 442-46

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-173

KCBraun

BITUMINOUS COAL STALLS FOR 700 ATM.

Lu, 3 November, 1937

A compilation of data on coal stalls and the evaluation of the results indicates that for bituminous coal at 700 atm. the best results will most probably be obtained with 2-stage converters. The arrangement of 4 converters in a stall, 2 each in pairs , has given good results according to reports by I.C.I. The longest operating time of such stalls has been 3-1/2 months.

It should be noted that the effective gas velocity, and consequent degree of filling, in a 700 atm. stall with 4 converters in series is just about as high as in the present 3-stage stalls in Scholven, so that such a converter arrangement appears quite possible for 700 atm. coal stalls. However, other experiences indicate that a smaller production may be expected from such stalls than from 2- or 3-stage stalls.

To decide whether the more economically favorable 4-stage coal stall at 700 atm. is feasible from the point of view of reaction, the following two problems must primarily be investigated:

1. Sufficient, but not too much settling of the catalyst.

2. Sufficient filling of the converters with liquid.

Attached Table I indicates the throughput, velocity, etc. of various coal stalls. It shows that the paste velocity, in t/m^2 converter cross-section per hour, which might be considered the controlling factor in the settling of the catalyst, is the same with two converters in series as in the 3-stage stalls in Scholven or the 4-stage stall in I.C.I. In 3- or 4-stage stalls the paste velocity is higher than in other bituminous coal stalls, nearly that in the 4-stage brown coal stalls in Leuna. Accordingly, the choice of 2-stage stalls for Gelsenberg appears correct from the standpoint of the most efficient use of the catalyst, even though no serious objection can be made against the arrangement of 3 or 4 converters in series, considering the good results obtained in Leuna and the high specific gravity of the catalyst proposed for Gelsenberg.

Other things being equal, the effective gas velocity under conditions of reaction is the controlling factor in the extent of filling. This velocity is about the same for a 2-converter stall in Gelsenberg as for a 2-converter stall in I.G.I. It is known that in I.C.I. a 2-stage stall has given better results than a 3- or 4- stage stall. With 4 converters in series the gas velocity is about the same as with 3 converters in Scholven or 4 converters in I.C.I. respectively. Since the extent of filling the converter is also dependent on the viscosity of the converter content, see note below, and in Gelsenberg this is nearer than in Scholven than in I.C.I., it is evident that a 700 atm. stall with 4 converters in series is possible for bituminous coal, but that a stall with 2 pairs of converters arranged in parallel will give better results.

For these reasons the installation of 700 atm. bituminous coal stalls, each with 2 pairs of converters in parallel, appears the most favorable.

Note on viscosity above:

The viscosity of the converter contents is dependent upon the asphalt content and the temperature, as well as the solids content. At 700 atm. the asphalt content is lower and the reaction temperature higher than at 300 atm. Both factors, therefore, tend towards better filling. The objection that the rising velocity of the gas bubbles must be smaller at 700 atm. because of greater density, with a consequent reduction in the extent of filling, is disproved by the following calculations:

Pressure Atm.	Effective Weight Under Conditions of Reaction, g/Ltr.		Uplift of 1 Liter Gas	
	Gas	Liquid	In g	In % of @ 300 Atm
300	25	abt. 600	575	100
700	47	abt. 600	553	96

which shows that the gas uplift at 700 atm. is only 4% lower than at 300 Atm.

Experiences with this arrangement in I.C.I. have, to our knowledge, been satisfactory. The longest operating time of such a twin stall in I.C.I. has been 108 days, according to Dr. Huebner, about as long as with a 2-stage stall in I.C.I, in which an operating time of 120 days was recently obtained.

Table I - Different Coal Stalls

	Merseburg		I.G.I.			Scholven	Gelsenberg		
Arrangement	2 x 800	4 x 800	2 x 1500	3 x 1500	4 x 1500	3 x 1200	2 x 1000	3 x 1000	4 x 1000
Conv. Lgth.-m	18	18	12.5	12.5	12.5	18	17	17	17
Inside Dia-mm	655	655	1000	1000	1000	1040	860	860	860
Cross-Sect.-cm^2	3370	3370	7830	7830	7830	8500	5800	5800	5800
Length, m	36	72	25	37.5	50	54	34	51	68
Stall Vol.-m^3	11	22	19	27.5	38	40.5	17.5	26.5	35
Pressure, atm.	230	230	250	250	250	300	700	700	700
Temp. °C.	460	460	460	460	460	460	475	475	475
Thruput, Kg/L/h	0.73	0.73	0.55	0.55	0.55	0.59	0.87	0.87	0.87
Thruput, t/h	8	16	10.4	15.1	20.9	24	15.3	23.1	30.5
Thruput, L/m^2/h	24	48	13.3	19.3	26.7	28	26.3	40	53
Gas m^3	12000	24000	16000	24000	32000	45000	27000	40000	54000
Reaction Gas Vol. @ Temp.-m^3	155	310	192	288	384	450	148	220	295
Velocity, cm/sec.	12.5	25	7	10	14	14.7	7	10.3	14
Reaction Gas Load, L/cm^2/h	46	92	24.5	37	49	53	25.5	38	51

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U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-174

KCBraun

Installation of Quadruple Instead of Triple Coal Stalls at Nordstern.
(See sketch attached)

Lu, 3 November, 1937

The gas is regulated in I.C.I. in the cold section. If only one heat exchanger will be provided for Nordstern, the gas should be regulated in the hot section, which, to be sure, means somewhat of a risk for a 700 atm. installation. However, some experience along that line is available at Scholven.

The twin arrangement could, however, be installed without difficulty with two heat exchangers, and six instead of four heat exchangers would be required for the coal phase at Nordstern. (Instead of four triple now three quadruple stalls).

I.C.I. has experienced no increased difficulties in the operation of the quadruple twin stalls as compared to the two converter stalls; the operating time is about 120 days in both cases. The production of the quadruple stall is equal to two 2-converter stalls, that is, the oil yield based on converter volume is higher than in a triple stall.

The following results in decreased production with the converters arranged in series have been obtained in experiments at I.C.I.

With a paste thruput of 0.55 Kg/L the oil yields were:
with 1 converter, 110% gasol. + Mi-Oil - 320° (Gasification abt. 29%)

2 converter
or 1, 2-stage,
4 converters
(twin arrangement, see sketch) } 100% gasol. + Ni-Oil-320° (Gasification abt. 26%)

3 converters, 92% Gasol. + Mi-Oil - 320° ? (Gasification abt. 24%)

4 converters, 70% Gasol. + Mi-Oil - 320° (Gasification abt. 17%)

With about 93% decomposition, the following approximate yields (gasoline + Mi-Oil -320°) would result:

1 converter, 0.150 Kg/L oil yield = 0.165 gasol. + Mi-Oil

2 converter
or 1, 2 stage, 4-converters (twin arrgmt). } 0.157 Kg/L oil yield = 0.157 gasol. + Mi-Oil

3 converters, 0.163 Kg/L oil yield = 0.150 gasol. + Mi-Oil

4 converters, 0.177 Kg/L oil yield = 0.125 gasol. + Mi-Oil

These figures indicate a slight increase in yield with a twin parallel arrangement in a quadruple stall as compared with a triple stall. The arrangement of four converters in series is apparently eliminated, considering the decrease in yield, although the last converter might still have sufficient heat of reaction.

The heat of reaction in Scholven:

Converter I, 212 cal = 49%

Converter II, 123 cal = 28%

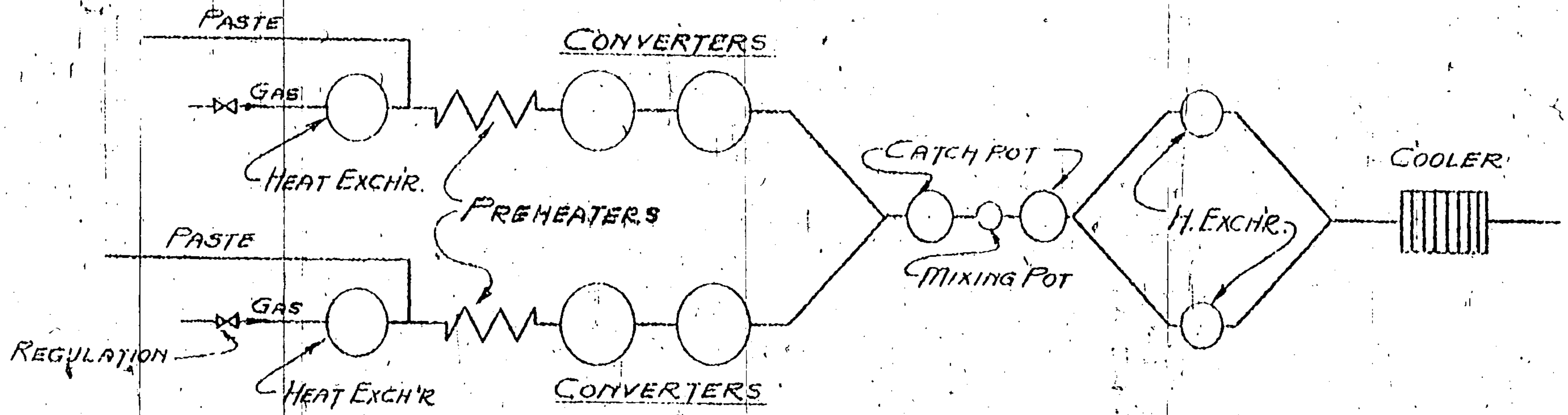
Converter III, 100 cal = 23%

Total 435 cal/Kg pure coal

<u>Summary:</u>

Experiences to date indicate no objection against the use of quadruple stalls. In particular, the parallel arrangement in the quadruple stalls at Nordstern should provide no particular difficulties, it may even result in a slight increase in yield.

T-174



ARRANGEMENT OF I.C.I TWIN STALLS

TOM Reel 126
Pps. 434-37

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-175

KCBraun

BASIC DATA FOR THE HYDROGENATION OF VARIOUS RAW MATERIALS TO DIESEL OIL AND GASOLINE IN A BITUMINOUS COAL HYDROGENATION PLANT.

Lu, 14 July, 1938

The following Table I contains data on the possible production of a plant for the hydrogenation of young bituminous coal to gasoline after conversion to diesel oil and gasoline (ratio about 1:1) and various raw materials.

In summary, the roughly estimated figures are:

No.	Raw Material	Production %	Remarks
1.	Bitum. Coal, 83% C	Gasoline 100% =	Basis of Comparison
		Conversion to Diesel Oil and Gasoline	
2	Bitum. Coal, 83% C	110	1/3 of vapor phase not utilized, liquid phase 5% overloaded.
2a.	Bitum. Coal, 87% C	110	Compare No. 2, also rust protection necessary.
3.	Bitum. Coal, 83% C	120	Compare No. 2a.
4.	Brown Coal	110	Distillation & Injection pumps in liquid phase barely equal to task, vapor phase ample capacity.
5.	Bitum. Coal Conversion a)	130	Without conversion from liquid to vapor phase, besides 100 L.Ph. Mi-oil or fuel oil after expansion of distill. and inj. pump facilities.
	b)	200	After conversion fr. liq. to vap. ph. and additions to distillation and injection pumps.
6.	Pitch, 75° E.P. from bitum. coal	145	Expansion of L.Ph. distill. necessary.
7.	Cracking Residue from crude oil	abt. 290	Expansion of distill. & inj. pumps in L. & V. Ph. necessary, V.Ph. barely equal.
8.	Pitch, 75° E.P. from crackg. residue	abt. 203	Expansion of distill., inj. pumps & gas circ. pumps in L. Ph. necessary.
9.	Petroleum Residue 325° (S.G.= 0.960)	abt. 400	Expansion of distill., inj. pumps & gas circ. pumps in L. & V. Ph. necessary.
10.	Pitch fr. Petroleum	abt. 245	Expansion of distillation & inj. pumps in L. Ph. necessary.

TABLE I

Production of a Bitum. Coal Plant for Gasoline After Conversion to Diesel Oil & Gasol. & Other Raw Materials

No.	1	2	3	4	5
	83% C, without Cl	83% C, without Cl, 87% C, with Cl (Bituminous	83% C, with Cl estimated Coal)	Rhenish Brown Coal	Bitum. Coal Crude Tar Ext.
Hourly Quantities for 100000 t/ann. End Product from Liq. & Vap. Phase					
% Diesel Oil	0	51	51	47	55
% Gasoline	100	49	49	53	45
Liq. Phase, 700 Atm.					
m^3 H_2	100	107	117	105	300
t Letdown	100	105	105	85	110
m^3 Converter Volume	100	108	145	140	240
t Injection	100	105	110	82	180
m^3 Circulating Gas	100	180	125	115	415
10^6 WE Hy-Off gas	100	105	130	120	240
Power Consumption KWh	100	125	130	115	295
Vapor Phase, 300 Atm.					
m^3 H_2	100	125	125	125	110
t Letdown	100	170	170	120	170
m^3 Converter Volume	100	145	145	115	145
t Injection	100	165	165	115	165
m^3 Circulating Gas	100	155	155	110	155
10^6 WE Hy-Offgas	100	175	175	140	175
Power Consumption KWh	100	135	135	110	120
Liq. & Vap. Phase, m^3 H_2	100	110	120	110	200

TABLE 1 - Continued

Production of a Bitum. Coal Plant for Gasoline After Conversion to Diesel Oil & Gasol. & Other Raw Materials

No.	6	7	8	9	10
	Pitch from Bit. Coal Crude Tar Soft Point 75° C.	Cracking Res. from Crude Oil	Pitch from Cracking Res. Soft. Point 75° C.	Petrol. Res. below 325° S.G.=0.960	Pitch fr. Petrol. Res. Soft. Point 75° C.
Hourly Quantities for 100000 t/ann. End Product from Liq. & Vap. Phase					
% Diesel Oil	51	53	51	60	55
% Gasoline	49	47	49	40	45
Liq. Phase, 700 Atm.					
m^3/H_2	195	280	180	410	210
t Letdown	90	125	125	135	125
m^3 Converter Volume	170	280	205	435	350
t Injection	160	185	155	170	165
m^3 Circulating Gas	300	255	130	190	175
10^6 WE Hy-Offgas	165	160	120	275	160
Power Consumption KWh	205	285	205	340	225
Vapor Phase, 300 Atm.					
m^3/H_2	115	300	300	400	400
t Letdown	170	180	180	250	250
m^3 Converter Volume	145	240	240	490	490
t Injection	165	170	170	220	220
m^3 Circulating Gas	155	210	210	370	370
10^6 WE Hy-Offgas	175	235	235	330	330
Power Consumption KWh	125	260	260	345	345
Liq. & Vap. Phase, m^3H_2	165	290	205	410	245

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Pps. 252-54

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-176

KC Braun/

Contributory Data on Rectification, Particularly of Mineral Oils.

28 October 1942

To separate liquid mixtures by distillation, i.e. socalled fractionation or rectification, packed or tray (bubble cap trays) columns are mainly used. Of these, the tray columns give a sharper separation, for the same size, and are, therefore, largely used for mass distillation, while in the laboratory or in small scale production columns packed with Raschig rings are often used, because of simpler construction and ease of cleaning, or where the use of an acid proof material is required.

The packing or the trays are for the purpose of bringing a rising vapor into intimate contact with a falling liquid. The heat required to vaporize the distillation feed is supplied in the lower part of the column, or in special preheaters, e.g. in continuous pipe stills, which consist of an enriching section in the upper, and a stripping section in their lower part, between which the distillation feed enters. The vapor leaving the head of the column is completely condensed. Part of the condensate is taken off as the desired end product and the rest, the socalled "return" or "reflux run", is returned to the column at the top. In large columns, in which the heat loss by radiation may be neglected, the heat balance for the exchange of matter between the rising vapor and the falling liquid is satisfied by the equation:

heat of vaporization = heat of condensation

on each tray or in each cross-section of the packing.

The sharpness of separation in a column processing oils is evaluated by the range in the boiling points in an Engler distillation of the resulting fractions, which is based on the low separating effect of the Engler distillation. For an exact characterization of the sharpness of separation, the "theoretical tray equivalent" of the column is specified, i.e. the number of trays which a theoretically ideal column would have to have in order to obtain the same separating effect, which will produce complete phase equilibrium between liquid and vapor on each tray. In packed columns, the corresponding determining factor is the "equivalent height" of a theoretical tray.

The "tray efficiency" of a tray column, i.e. the quotient of the theoretical tray equivalent and the actual number of trays depends not only on the construction but also on the speed of distillation and the quantity of the return run, as well as the composition of the outgoing mixture. It approaches unity the smaller the speed of distillation and the larger the return run, which also means the lower the production of the column is held. In practice an optimum production must be maintained because of operating and amortization costs. (For the "most favorable return run", the rule of thumb for the speed of distillation is velocity of vapor in seconds = the tray interval in mm?). Furthermore, there is a certain "smallest return run relation", which must be observed even with a simultaneous arbitrary increase in the number of trays, if the object of the distillation is to be achieved.

In large columns a mean tray efficiency of 0.6-0.8 is obtained, while in the laboratory this value is never reached because the vapors remain longer between individual trays, causing a poorer equilibrium relation. However, various very effective columns were proposed for the laboratory. Among tray columns may be mentioned that recommended by Schultze and Stage, in which an equivalent height of 6 cm. for a theoretical tray was obtained. The best packed columns are the Podbielniak with rotating insert and the State---with shaped cast wire packing elements. In packed columns a maximum tray equivalent of 2 cm. may be reached, in tray columns one of 3-4 cm. Conditions here are, therefore, the reverse of those with large columns in industry.

While distillation columns in the chemical industry in general are mostly used for the decomposition of a mixture of a few substances into comparatively pure, simple constituents, in the oil industry a raw material of inaccurately determined composition must be decomposed into fractions, which themselves are multi-substance mixtures with a wide boiling range. The theory of distillation primarily covers only the separation of two or at most three mixtures of chemically defined constituents, because only in such cases is it possible to calculate the theoretical number of trays required for a desired separating effect with the aid of an equilibrium diagram, which will give the relation between the composition of vapor and liquid in the corresponding binary or ternary system. The design of oil fractionating columns is, therefore, largely dependent upon empirical generalities, on multi-substance mixtures with constantly over-lapping boiling ranges of the constituents, as well as on values obtained in practical experience. The vapors occurring in the columns may largely be considered ideal gases and for their equilibrium with the liquids the approximate validity of the Raoult law may be assumed. In particular, values obtained in practice are always used for the allowable vapor velocity in the columns and the tray efficiency, and the molar heat of vaporization is considered constant over the whole height of the column. On the other hand, the Raoult law is no longer valid when it is not applied to the decomposition of a multi-substance mixture with a wide boiling range, but to the isolation of individual substances in a narrow oil fraction, because in such a case, e.g. with benzol-hexane-cyclohexane, azeotropes, i.e. constant boiling mixtures, may appear and offer unexpected difficulties to a separation.

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Pps. 485-507

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-177

KCBraun/jw
11/25/46

Abstract of Report on Filtration of Coal Hydrogenation Letdown

Leuna, 21 July 1938

Summary:

The problem was to split up the residues of brown coal hydrogenation (letdown) by filtration into oil free of solids, heavy oil, and solids free of asphalt.

It was first determined in laboratory experiments that the letdown from the same coal stall may fluctuate greatly in its filtration ability, that sluiced letdown can be filtered more readily than letdown released by needle valves, and that sluiced letdown also varies greatly in its ability to be filtered without any definite relation to other properties, such as solids- and asphalt- content, size of solid particles, etc. By storing for a longer period a good filtrable letdown becomes poorly filtrable. The speed of filtration increases with rising temperature, as well as with an admixture of hot oil, particularly one rich in hydrogen. On the contrary, the addition of such oil to the pasting oil of the coal paste has little beneficial effect on the filtration ability of the letdown.

Larger scale experiments indicated that the problem could be solved as posed. The average capacity of a Wolf cell filter at 140-150°C. was 95-100 Kg letdown/m^2 total filter surface per hour, the capacity of a Dorr filter, operating at 170-175° C. with a socalled "filter aid" was about half of that, and that of the column filter (Kerzenfilter) at 120-140° C. and 5-6 atm. absolute pressure about 130 Kg/m^2/h. Solids content of the filtrate, washing out of the filter coke and wash oil consumption were the most favorable with the column filters, which, however, require a more complicated apparatus and better trained operating personnel.

Laboratory Experiments:

The ability to be filtered depends very much upon whether the letdown is released from the high pressure stall by means of needle valves (patronenventile) or whether it is sluiced out through surge tanks (Pufferflaschen) or by expansion machines (Entspannungsmaschinen). Letdown released through needle valves is always much more difficult to filter than if it is sluiced out, assuming it is drawn from the same stall at the same time. The filter time relation is about 6:1.

But letdown withdrawn from the same stall by the same method but at different times often show varying filter times. For example, letdown released by needle valves varies in its filter time between 30 and 180 minutes, average 60 minutes, while sluiced letdown varies between 3 and 40 minutes, average 10 minutes.

If letdown is stored for an extended period in a heated vessel, constantly agitated and exposed to air, its ability to be filtered is very much reduced. Sluiced letdown, whose original filter time was 15 min. required 60 min. after two weeks storage, while the pour point of the filtrate rose to over 30° C. and the asphalt content from 10.58% to 15.56%.

Experiments to determine the suitability of various filter materials showed that cotton cloth, as used on tar filter presses, was resistant up to 150° C. and produced filtrate containing not over 1% solids at filter capacities of 150-250 Kg/m^2/h of sluiced letdown of average filtration ability. At temperatures above 150° C. the cloth became brittle and pervious in a short time. Some especially dense asbestos fabrics showed filter characteristics as good as cotton and were absolutely heat resistant. Metal filter cloth, including especially dense braided and twill fabrics with 1500 and 10000 mesh/cm^2 respectively, which had given good results in a previous experiment with another letdown, produced very impure filtrates when the letdown was introduced against the cloth from the bottom, but gave much better results, filtrates of 1% solids or less, when the letdown was introduced against the cloth from above. Apparently a denser filter cake was formed in the latter case, which acted as a filter layer.

Experiments were also made with the previously mentioned asbestos filter cloth to increase the speed of filtration by increasing the temperature. A rise in temperature from 150 to 250° C. doubled the filter capacity on the average. With pressures of 5-6 atm. the capacity was even trebled under the same condition, while above 300° C. the filter capacity increased roughly 8 fold compared to 150° C., although the capacity between 150 and 250° C. was comparatively much smaller on a pressure filter than on a suction filter. The purity of the filtrate was about the same for the suction filter as for the pressure filter. Besides an increase in capacity a decrease in the solids content of the filtrates was observed with rising temperatures.

By mixing letdown with A-middle oil at mixture temperature of 100° C. the filtration ability is affected materially. However, if the temperature of the mixture was increased to 150° C. a distinct improvement occurred, particularly when using a light oil rich in hydrogen, such as 5058 vapor phase B-product.

Larger Scale Experiments

A). Column Filters (Kerzenfilter).

Two 4-column pressure filters of 1 m^2 filter surface each were at first available for this purpose; later a 7-column filter with

3 m^2 filter surface was used. The columns (Kerzen) consisted of individual rings of ceramic material. The column filters were batch filters, taking 150-200 Kg/m^2 filter surface, when the filter cake reached a thickness of 25-30 mm, which was considered a maximum because the cake could not be readily removed otherwise, due to bridging between columns.

The solids content of the filtrate was about 0.5% when using filter rings with medium pores furnished by Schüler, Eisenberg, and 0.1% when using finer pore rings by Schumacher, Bietigheim.

The filter cake contained an average of 60% solids. By washing it out we succeeded in obtaining a filter cake practically free of heavy oil and asphalt without difficulty, which could be reduced to 0.1% oil content in a spiral kiln without coking. The first wash oil used was bitum. coal tar oil, fraction 200-275°, later coal catch pot middle oil, fraction 200-315°, with the same results.

In the washing out process, it was noticed that the letdown oil in the filter cake is uniformly displaced by the wash oil. The wash oil consumption is comparatively small and approaches the theoretical requirement, wash oil = letdown oil in filter cake.

The speed of filtration, not including the time required for washing out, change over, flushing, etc., at about 5-6 atm. filtration pressure and 130-150° C. filtration temperature, when using sluiced letdown of average filtration ability, was about 300 kg/m^2/h. If a pressure released letdown was processed the speed of filtration dropped to one third. The time required for washing was about the same as for filtration. Including washing, flushing, etc. the filter capacity with sluiced letdown of average filtration ability was 130 kg/m^2/h on the average.

b). Wolf Filters

Made by Wolf, Buckau, a continuous revolving cell suction filter with 0.6 m^2 filter surface. The cotton filter cloth lasted about 20 operating days at a filter temperature of 140-150° C. When the temperature was increased to 170° C. the cloth lasted less than half that time.

The filter capacity varied between 44 and 166.5 Kg/m^2/h of total filter surface (not per m^2 of immersed surface), or an average over 69 operating days of 96.5 Kg/m^2/h when filtering sluiced letdown.

When the letdown was diluted with 50% A-middle oil the filter capacity remained practically the same at 100° C., but about doubled when heated to 150° C.

The first wash oil used was bitum. coal tar light oil. This led to clogging of the lines to the vacuum pump because of its naphtaline content and was replaced by A-middle oil, fraction 200-275°, which is free of naphtaline.

Because the filter cake absorbs the washing medium with difficulty, it is necessary to apply the wash oil uniformly distributed over the length of the drum. Fig. 1 shows the arrangement of 3 distributing troughs, marked 1, 2, 3, with serrated overflow edges.

The filter cake cracked badly immediately after emerging from the filter trough. These cracks allowed the wash oil to seep through without washing the part of the cake between them. We, therefore, tried to transfer the point of crack formation to about point "A", Fig. 1, by trickling letdown from the overflow trough on the filter cake. This was only partly successful, inasmuch as the cake would crack shortly beyond this point and the wash effect was still imperfect.

An endless wash felt, a so-called paper machine wet felt of wool, was then applied, Fig. 2. This revolved with the filter drum and the wash oil was trickled on it. Funnel shaped troughs were installed below the bottom guide rolls to catch the surplus wash oil and carry it to a collecting vessel, from which it could be pumped back into the wash oil storage tank. This wash felt largely prevented the drying out of the filter cake and provided uniform distribution of the wash oil over the filter cake surface.

The wash oil was preheated to 130-140° C., as was the CO_2 protecting gas blown into the filter hubs.

When freshly sluiced letdown was used, the filter cake detached itself comparatively easily just above the scraper, aided by the injection of CO_2 into the cell nearest the scraper knife.

The filter cake contained roughly 50% solids without washing and about 60% with, i.e. it contained 60% solids, 30% wash oil, and 10% heavy oil.

The filtrates from the filter zone (pure filtrate) and the wash zone (wash filtrate) were drawn off separately into containers. In spite of this, the pure filtrate contained some wash oil. A considerable portion of the wash oil contained in the filtrates was vaporized in the containers. These vapors could be partly condensed in a cooler between the containers and the vacuum pump and collected in a catch pot. A considerable portion of these vapors, however, could not be condensed and reached the vacuum pump, causing lubricating oil dilution, or were discharged into the atmosphere. The vapors discharged could be condensed in a packed tower attached to the pump exhaust.

The wash oil condensate recovered in the coolers in the vacuum line was largely insoluble, but containing about 25% heavy oil from the "pure filtrate" and about 5% from the "wash filtrate". These condensates could not be added to the wash oil without re-distillation, but could be used for diluting the letdown. Such letdown would produce filter cake containing an average of 5%, in some cases 1%, heavy oil, without using any more fresh wash oil than with letdown not diluted.

When using easily filtrable letdown with good filter cloth the "pure filtrate" contained an average of 0.5% solids and the "wash filtrate" still less. Poorly filtrable letdown produced filtrates containing considerably more solids.

c) Dorr Filters

Also a continuous revolving cell suction filter with 1.8 m^2 filter surface. The letdown is filtered through a layer of so-called "filter aid", applied on the asbestos fabric covered filter drum ahead of the filtration proper. The "filter aid" consists of either Kieselgur or diatomaceous earth and is mixed to a sludge with a thin oil in the filter trough. On revolving the drum this sludge sticks to the filter cloth in a layer about 30-40 mm thick and the oil is filtered off. The filter trough is then filled with letdown and the filtration can begin. A knife, whose cutting edge slowly moves towards the filter surface, cuts off a thin layer of the "filter aid" together with the filter cake, so that the letdown to be filtered is always in contact with a fresh surface of the "filter aid".

In spite of higher filter temperatures, the capacity of this filter was considerably below that of the Wolf filter and the purity of the filtrate barely equal to it.

Continuation of Experiments

With respect to capacity, purity of filtrate and efficiency of washing, the column filter is superior to the revolving filter. It would be of interest to know what results could be obtained by using column filter material of greater porosity, quartz instead of refractory material. The use of higher temperatures, say up to 300° C., would also increase the yield without affecting the purity of the filtrate. Since the use of revolving suction filters involves considerable trouble in condensing and recovering the evaporated filtrate, emphasis should be placed on experiments with the Imperial pressure filter (Meissen), which operates under a pressure of 3-4 atmospheres.

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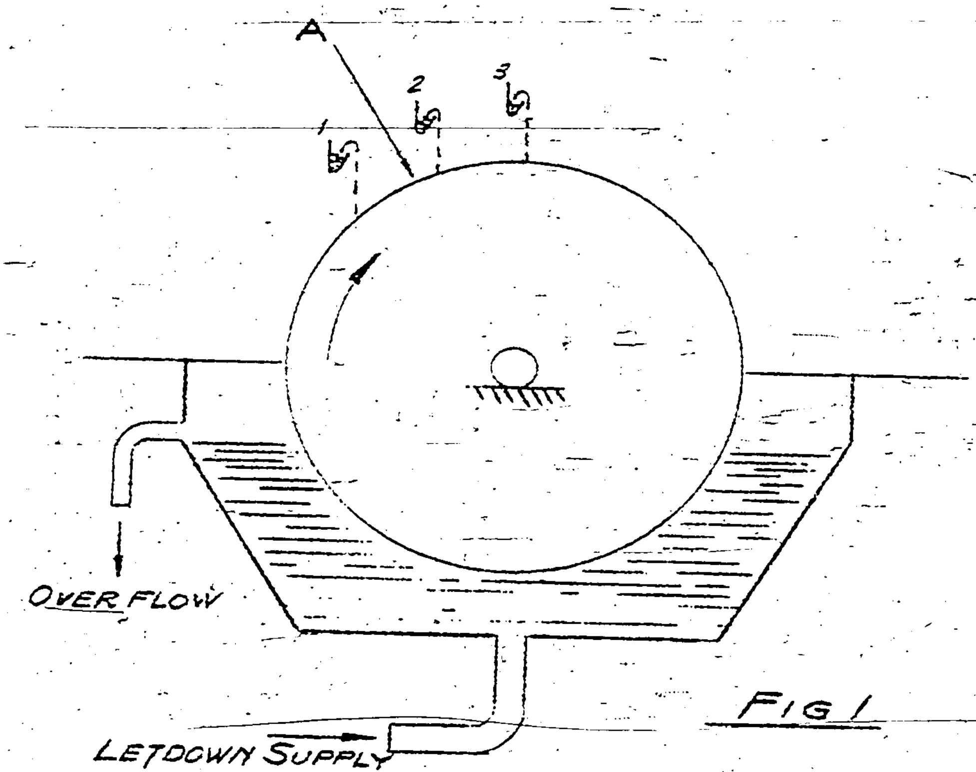

FIG 1

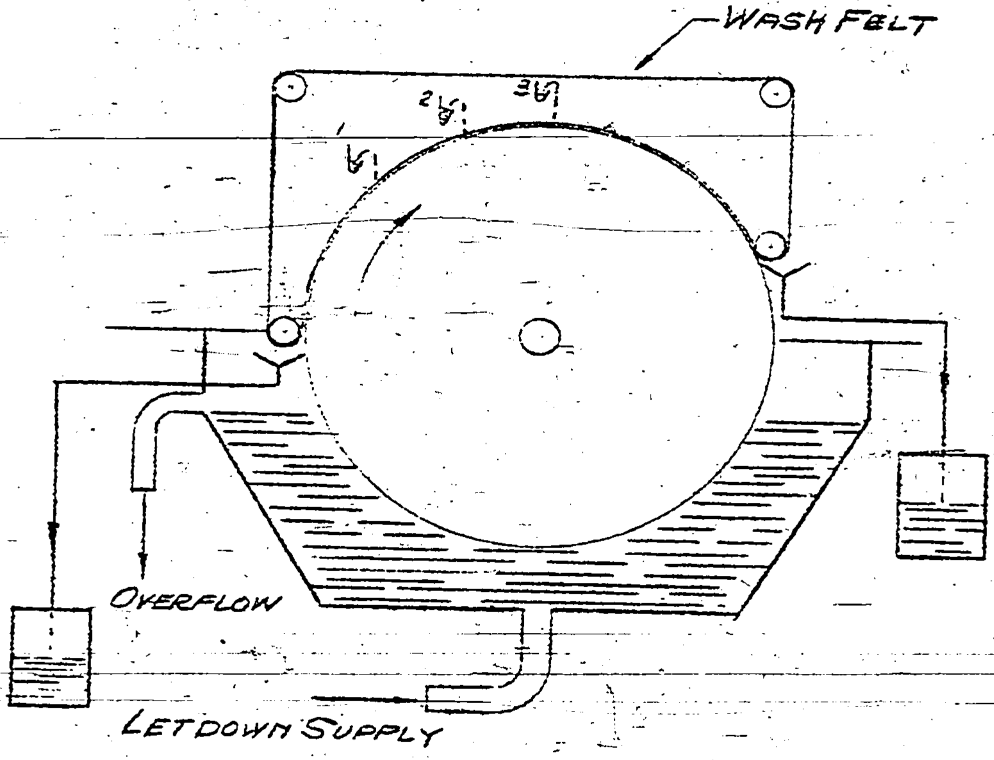

FIG. - 2

TOM Reel 130
Pps. 636-38

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ACCIDENTS AND DAMAGES IN HYDRO-WORKS

Ludwigshafen, 1942

I. Damages by Bombing Attacks.

Bomb damages to various plants up to March 1941 of no particular interest to our work. Generally no crippling damage inflicted, plants resumed operation shortly thereafter.

II. Damages by Failure of N10-Material.

Plant	Nature of Damage	Time
Nordstern	Fire in preheater. Crack in a double bend.	May 1940
Nordstern	Preheater explosion.	October 1940
	Plug in return bend blown out.	
Nordstern	Bursting of N10-piece in filling with N_2 at 680 atm.	January 1941
Pölitz	Cracks in hairpin coils in 2 electric preheaters.	September 1941

III. Accidents by "Runaway" of Converters.

Plant	Nature of Damage	Time	Remarks
Leuna	Stall destroyed by bursting of S2 tube installed in hot place.	1930	
Nordstern	Fire in coal stall when emergency expansion line burst.	Nov. 1940	Failure of check valve & emerg. expans. valve.
Pölitz	2 stalls down by explosion. Converters out of control through gas failure. Circ. line clogged by gas hydrates.	Nov. 1941	
Pölitz	2 stalls down by explosion. Flames spread 80 m. Converters out of control through gas failure. Gas inlet line clogged.	June 1942	7 dead in paste press bldg.

IV. Other Accidents and Damages.

Plant	Nature of Damage	Time	Cause	Remarks
Leuna	Damage to expansion machine.	Nov. 1935	Cylinder break	1 dead. Entire plant down 2 weeks. Full prod. in 3 mo.
Scholven	Explosion in coal drying plant.	1937	Dust Explosion	
Nordstern	Room explosion in CO_2 wash. Expansion line burst on expansion machine when thawing out line in extreme cold.	Jan. 1940	Failure of liq. level measurement on washer.	1 dead
Nordstern	Leaks in circulating line.	Nov. 1940	Floor sagged.	
Pölitz	Explosion in stall during repairs.	June 1940	Gas entered stall & exploded	3 dead

TOM Reel 130
Pps. 559-587

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KCBraun
11/26/46

I. G. CONSTRUCTION STEELS (Abstract)

IG-Norm 10-01. Plain Carbon Steels.

Covers designation, composition, mechanical properties and recommended use of:

Forging steel	DIN-1611
Shapes & rods	DIN-1612, 1613,
Sheets	DIN-1621, 1622, 1623
Heat treat steel	DIN-1661

IG-Norm 10-02. Low Alloy Steels.

Covers designation, composition, mechanical properties and recommended use of:

DIN-1663 & DIN-E-1665.

IG-Norm 10-03. Insert Steels.

Covers designation, composition, mechanical properties and recommended use of:

Plain insert steel DIN-1661,

Alloy insert steel DIN-1663 & E-1664

IG-Norm 10-04 Nitriding Steels.

Covers designation, composition, mechanical properties and recommended use of:

KM-Norm	Cr AlSt 65,
"	Cr AlMoSt80,
"	Cr AlNiSt80.

IG-Norm 10-09. Izett Steel.

Covers general specifications for delivery by maker, testing, stamping, etc. of various forms of Izett steels.

IG-Norm 10-20. Corrosion-and Acid-Proof Steels.

Covers designation, composition, mechanical properties and recommended use of martensitic Cr-and Cr-Mo Steels.

IG-Norm 10-21. Corrosion-and Acid-Proof Steels.

Covers designation, composition, mechanical properties and recommended use of ferritic and semi-ferritic Cr-and Cr-Mo Steels.

IG-Norm 10-22. Corrosion-and Acid-Proof Steels.

Covers designation, composition, mechanical properties and recommended use of austenitic Cr-Mn and Cr-Mn-Ni Steels.

IG-Norm 10-23. Corrosion and Acid-Proof Steels.

Covers designation, composition, mechanical properties and recommended use of austenitic Cr-Ni, Cr-Ni-W and Cr-Ni-Mo steels, particularly Krupp V2A, shown in the following tables. (Mechanical properties given are in heat treated condition).

Physical Properties of VA Steels:

Expansion Coeff: 0-100° C = 16.5×10^{-6}
0-300° C = 17.5×10^{-6}
0-500° C = 18.5×10^{-6}

Coeff. heat transm. = 0.03 - 0.04 cal/cm/sec/°C

Modul. of elasticity = 20000 Kg/mm^2

Austenitic steels are poorly magnetic.

Designation	Approx. Composition, %	Tensile Strength Kg/mm^2	Yield Point Kg/mm^2	Elongation at Break %	Notched Bar Impact Strength mKg/cm^2	Use
Krupp, V2A Supra	Cr = 18 Ni = 9 C = <0.07	55-70	22	55		Suitable to 350-450° C. Welding quality. Sheets, tubes, rods.
Krupp, V2A Normal	Cr = 18 Ni = 9 C = 0.1	55-75	>25	>50	18	Suitable for permanent heat exposure to 300-400° C. Requires heat treating after welding. Sheets tubes, rods, forgings.
Krupp, V2A Extra	Cr = 18 Ni = 9 C = 0.1 + Ti or Ta	55-75	>25	>40	>15	Greater heat resistance than preceding. Welding quality, practically free of tendency to grain decomposition. Sheets, tubes, rods.
Krupp, V2A Supra Special	Cr = 20 Ni = 6 C = <0.07 + N_2	>80	>50	>45		Substitute for V2A Supra (to save Ni) but considerably harder. Not pure austenite.
Krupp, V2A Extra Special	Cr = 22 Ni = 6 + Ti	>65	>40	>23	DVM >10	Subst. Ferritic structure. Incr. yield point without cold working in heat treated condition. Brittle above 300° C.
Krupp, V12A Supra	Cr = 12 Ni = 12 C <0.1	50-65	>20	>25	20	Chemical stability below V2A. High deep draw strength. Not resistant to grain decomposition, unsuitable for household use.
Krupp, V2A-Ed	Cr = 18 Ni = 10 W = 1.2	60-70	35	35	12	High heat resistance, particularly in 600° range. Hot working welding and heat treating influence heat resistance.

HEAT RESISTING PROPERTIES OF V2A

Designation	Yield Points, Kg/mm^2 Temperature, °C.								Creep Strength (Dauerstandfestigkeit) Kg/mm^2 Temperature, °C.					
	20	100	200	300	350	400	450	500	400	450	500	550	600	700
Krupp, V2A Supra		19	14	11		10								
Krupp, V2A Normal		18	13	11		10								
Krupp, V2A Extra	25	20	17	16		15	15	15			14	10		3
Krupp, V2A Supra Special	Values above V2A Supra. Not recommended for high temperatures because of brittleness.													
Krupp, V2A Extra Special	Brittle above 300°													
Krupp, V2A-KD Unannealed	35	34	32	31		30	29	27	28		25	16	12	

WELDING PROPERTIES OF V2A

Designation	Welding Method	Heat Treatment	Welding Material	
			Gas Welding	Arc Welding
Krupp, V2A Supra	Any	Not required, but desirable in limiting cases of high chemical stability.	Krupp, Zeus GV leg. V2A	Krupp, Zeus EV leg, V2A
Krupp, V2A Normal	Any	Absolutely necessary. Deficient heat treatment causes grain decomposition.	Krupp, Zeus GV leg. V2A	Krupp, Zeus EV leg, V2A
Krupp, V2A Extra	Any	Not required, even for high chemical stability.	Krupp, Zeus GV leg. V2A	Krupp, Zeus EV leg, V2A For temp. above 400° C. use Krupp Zeus EV 109. V2AX
Krupp, V2A Supra Special	Any	Same as V2A Supra	Krupp, Zeus GV leg. V2A	"
Krupp, V2A Extra Special	Not intended for welding			
Krupp, V12A Supra	Any	Not required for normal, low chemical stability.	DEW Thermanit J	DEW Thermanit J
Krupp, V2A- EG	Mostly arc-welding	Not required.		Krupp Zeus EV leg. V2AX

NOTE: In autogenous welding use neutral flame, carburizing must be especially avoided. Except for special cases, arc welding is preferable to autogenous welding. For good corrosion resistance use a flux on welding surfaces.