FIG. 88

to follow R

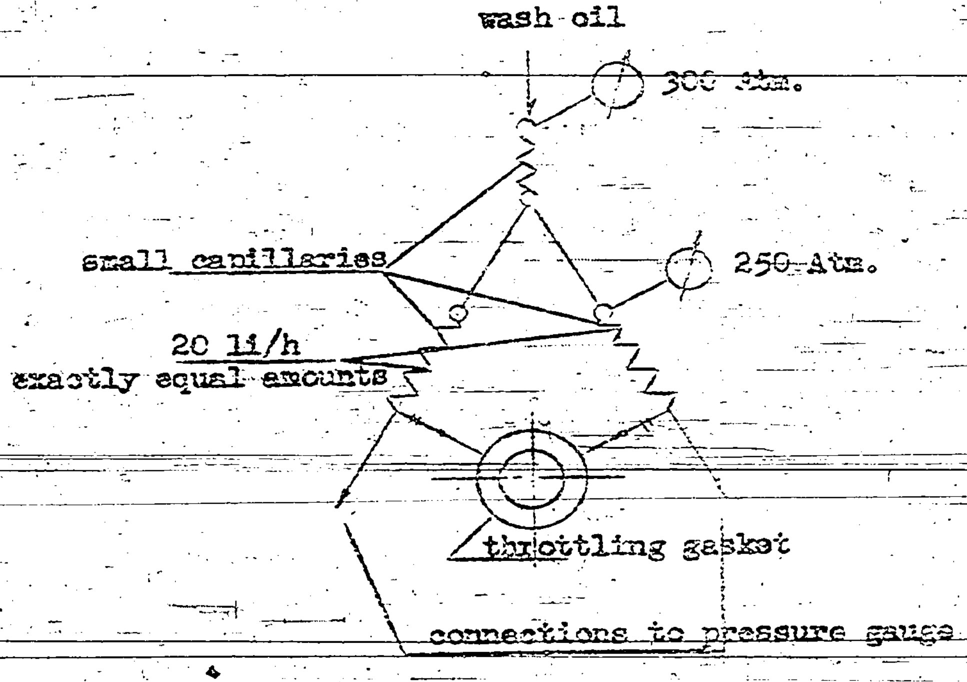
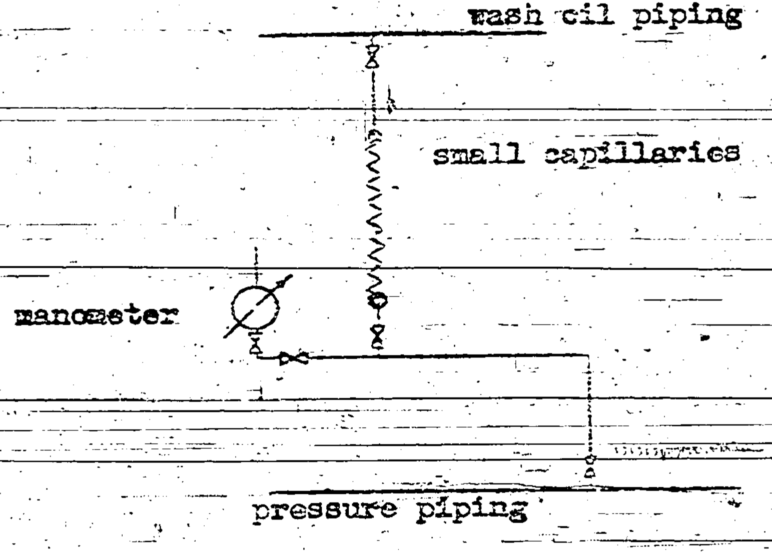


FIG. 88a

manometer with wash oil connections



T-46

CHARACTERISTICS OF HYDROGENATION WORKS
Ludwigshafen, 1942

Reference # h. T.O.M. Reel No. 129 pp.654-674

I. LEUNA:

Operations first started in 1927.

Production:

100,000/yr te of automobile gasoline from brown coal, brown coal tar, and petroleum.

In 1935 production increased to 350,000/yr te.

In 1940 and subsequent years our production further increased to 600,000/yr te.

Operations at present almost exclusively on brown coal.

At present aviation gasoline and diesel oil are produced in equal quantities as well as about 10% fuel gas.

Production Method

Number of High Pressure Stalls:
Coal Stalls: 9 Stalls
Prehydrogenation: 6 Stalls
Bensination: 3 Stalls

Pressure: 225 Atmosphere

Raw Material: Middle German brown coal which led to a high hydrogenation paraffin products.

Difficulties caused by the high ash content (sand and chalk) can now be overcome.

Hydrogen Production:

Winkler Processes with lignite coke and oxygen as well as hydrogen from coke.

A total hydrogen consumption of about 170,000 m^3/te.

The hydrogenation unit is coupled with the ammonia and methanol synthesis. Gas production for both.

II. Scholven

The first coal hydrogenation work in Germany was put in operation there in 1936.

Production:

Originally 125,000 year te of automobile gasoline.
Soon increased to 200,000 year te.
Can be converted to aviation gasoline. Produces at present aviation gasoline only, and about 10% fuel gas. September production 210,000 year te of aviation gasoline plus 25,000 year te of fuel gas.

Production Method:

Number of high pressure chambers:

Coal Stalls:	5 Stalls
Prehydrogenation:	2 Stalls
Benzination:	2 Stalls

Pressure: 300 Atmospheres

Raw Material: Coal with 83% C.

In addition, 10-15% of the production from high temperature coal tars and oils.

Coal as a raw material yields low-hydrogen, a high-anti-knock aviation gasoline.

Utilization of coal only 90%. The installation at present being changed to 700 atm. pressure.

Scholven is the only works in which chlorine is added in the coal chambers as a catalyst. The chlorine corrosion on the cooling side is averted by neutralizing with soda in a second hot catch pot.

At first considerable difficulties experienced in the pre-heater (in the return bends) which resulted from chlorine corrosion and erosion and were overcome by improvement of material (enamelling of the preheater return elbows) and by lowering the chlorine concentration. Half the chlorine is added behind the preheater directly into the first converter.

Hydrogen Production:

Total hydrogen production 90,000 m^3/h.
Hydrogen production in coal generator (50%)
Linde process (30%)
Buna (20%)
Hydrogen gas has been found to be less economical in Ruhr than the Linde decomposition of coking gases and the Hydrogenation gases.

Fuel Gas Production:

The fuel requirements are met by the Linde residual gas, Hydrogenation off-gas, and residual water gas.

Power Generation:

Power requirements about 90,000 KW.
The current is delivered by the power station at Hibernia which was erected for the hydrogenation but is not part of the hydrogenation works.

Production of By-Products:

Alkylate: Upon completion of installation 73,000 a year/te half of it from their own gas, balance from the butane delivered from Gelsenberg.
Production of Sulfur: About 8,000 a year/te is possible.
Production of Nitrogen: Of 5500 year/te of N as 20% of ammonia water.

Deliveries at Leuna.
Deliveries of ethane to Zweckel for production of ethylene.
Deliveries of 30,000 year/te of HOLD as binder in the production of briquettes. The production should be extended to 60,000 year/te.

In combination with the coking:
Delivery of coke oven gas, 28,000 m^3/hour.
Hydrogen and return delivery of fuel gas.
Combination with Buna-Hüls:
The delivery of gases, hydrocarbons, methane and propane, with an average carbon content of 1.6 to Buna and return delivery of 18,000 m^3 of hydrogen to Scholven.
Combination with Gelsenberg:
Delivery of 41,000 year te of normal and isobutane from Gelsenberg to Scholven for the alkylating installation.

III. Böhlen and Magdeburg:

Subsidiary of the BrK-Industrie, I.G., DEA and the Electrowerke.

Production:

Bohlen	Magdeburg
180,000 year/te of aviation gasoline	120,000 year/te automobile gasoline
	120,000 year/te diesel oil
	240,000 year/te
About 15% fuel gas	About 3% fuel gas

Operations started in 1937.

Production Methods: Number of High Pressure Stalls:

	Böhlen	Magdeburg
Liquid Phase	2	2
Vapor Phase	3	3

The two steps of vapor phase contain different catalysts in the hydrogenation plants, but the thinned out catalyst is sufficient during the hydrogenation of the brown coal--tar middle oils (However, also performed in two steps).

Pressure: 300 atm.
Raw Material: Low temperature carbonization tar from central German brown coal, which results in the production in high-hydrogen and paraffinic products.

No difficulties encountered, Coronen deposition behind the hot catch pot.

Hydrogen Production:

Winkler process with lignite coke and oxygen.
Total hydrogen about 25,000 m^3/hr.

Fuel Gas Requirements:

Are covered by the residual hydrogen gases.

Power Requirements: About 30,000 KW. The current is supplied in Bohlen from the neighboring low temperature carbonization works of A.S.W., which produces it from lignite coke.

The hydrogenation works in Magdeburg use current from the electric power station in Magdeburg which formerly used brown coal and is at present using lignite coke.

The Böhlen plant immediately adjoins the low temperature carbonization plant at Espenhein of the A.S.W.; they are however, separate plants.

Carbonization and hydrogenation considered together, indicate that the lignite coke produced in coking central German lignite coal with a water content of 55% and a tar content of 6% (equal 15% tar referred to the pure coal), is entirely used up in the hydrogen and electric power production. This fact was of importance at one time, because the disposal of larger amounts of excess lignite coke would have caused difficulties. Because of the large amount of steam drying, the coking plant is a large steam producer. 100,000 te gasoline from lignite coke tar requires about two million te of raw lignite, and a small excess of lignite coke of about 200,000 year te is produced, and in large coking plants the excess of current is about 8,000 KW.

By-Products:

Production of 58,000 year te of alkylate is foreseen by using 15,000 year te of butane from the Fischer plant at Schwarzheide as well as 4300 year te of gas from Zeitz.

IV. Welheim

Operations started in November, 1937.

The first plant to operate with 700 atm. pressure.

The plant was erected for the hydrogenation of the coal extract from the Stinneskohle plant obtained by the Pott-Broche process. The extraction installation was not ready and coal tar extract was hydrogenated instead. The extraction installation was planned for 50,000 year te extraction production.

It is at present intended to use the extraction for the production of electrode carbon.

Production: 120,000 year te, of which 30% is aviation gasoline and 70% fuel oil, with an additional production of about 10% of fuel gas.

Method of Production:

Number of high pressure stalls:

Liquid Phase	3 stalls
Vapor Phase	1 stall

Pressure: 650 atm. total pressure in the liquid and vapor phase.

The vapor Phase is also operated at a pressure of 600 atm. In this case part of the middle oil from the liquid phase is used for aromatisation with a special catalyst with a simultaneous refining of the liquid phase gasoline.

Combined gas circulation for the liquid and vapor phase.

Oil Washing is not in use. This special case of omission of the circulating washing in the liquid phase is a special case of hydrogenation of pitch to fuel oil.

The residue is not worked in Welheim. The HOLD drawn off is removed and distilled in the still in the working up of the coking tar.

The extraordinarily small production of iso butane is characteristic. No provision is therefore made for an alkylation installation.

Raw Material:

170,000 te of pitch, as well as the oils from high temperature tar which are particularly well adapted for the production of low-hydrogen, high quality aviation gasoline.

Hydrogen Production:

Linde installation for coke oven and residual hydrogenation gases and H_2 production from coke in the Pintsch-Hildebrand generator.

Total hydrogen production about 22,000 m^3 per hour.

Fuel Gas Production:

The hydrogenation off-gases as well as residual gases of the Linde decomposition installation are used for heating. The excess fuel gas is returned to the coke oven gas.

Power Station:

Energy requirements about 40,000 kw/h.
The power station is incorporated in the concern.

There is an affiliation of the Stinnes concern with a coking plant through the use of coke oven gases.

Affiliation with the coking tar installation in the utilization of the hydrogenation residue.

Affiliation with the Buna works Hüls for delivery of 20 to 30,000/year to of gases, hydrocarbons methane - propane with an average G value of 1.6

V. **GELSENBERG:**

The first coal hydrogenation works at 700 atm. were put in operation in the beginning of 1939.

Capacity:

3 construction steps:

150,000/year te, 200,000 and 280,000/year te of motor gasoline which may be converted to aviation gasoline. At present only aviation gasoline is produced.

Capacity 340,000/year te of aviation gasoline and 70,000/year te of fuel gas (≈ 20%).

Process:

Number of high pressure stalls:

Sump phase:	6 stalls
Prehydrogenation:	4 stalls
Benzination:	5 stalls

Pressure: 300 atm. in the liquid phase, 300 atm. in the vapor phase.

Hydrogenation coal: Young brown coal from Ruhr with 83% C.

The coarse coal is de-ashed by the Sophia Jakoba process, the fine coal in the settling machines. In addition, 10 to 15% of the production is obtained from high temperature coal tars and oils.

The coal produces low-H_2 aviation gasoline with a high anti-knock.

There were originally difficulties when using the N10 material at 700 atm.

H_2 Production:

Total H_2 production 130,000 m^3/h.
H_2 is produced in automatic demag generators from coke: 10,000 m^3/h, or 75%.

H_2 from the Linde installation by decomposition of coking gases and residual hydrogenation gases: 15,000 m^3/h, or 10%. H_2 delivers from Buna works Huls: 6,500 m^3/h, or 5%.

H_2 recompression: 10,000 m^3/h, or 10%.

Fuel Gas Production:

The requirements in fuel gas are completely covered by the residual water gas of the coking furnaces, the hydrogenation off-gases and the residual gases at the Linde decomposition installation. The excess is delivered as a fuel gas to the Gelsenberg Mining Stock Society.

Power Production:

The power requirements are 135,000 KW.
The hydrogenation works erected a powerhouse of their own on their land.

Production of By-Products:

No alkylation installation of their own has been constructed. Gelsenberg delivers to the Scholven alkylating installation 41,000/year te of C_4.

Production of Sulphur:

3,000/year te of sulphur

Production of Nitrogen:

The total ammonia water is sent to the coking plant and utilized there.

Hydrogenation residue is sent to the smelter because of the high iron content.

Affiliation of the Nordstern coking plant of this concern. Delivery of coke oven gas for 15,000 m^3 H_2/h and return delivery of the fuel gas.

Affiliation with Buna Hüls.

Delivery of 50,000/year te of gaseous hydrocarbons of an average C content of 1.6 to Buna Hüls and return delivery of 6,500 m^3 H_2/h.

VI. ZEITZ:

The only installation for the utilisation of the low temperature carbonisation tar of brown coal, using the TTH process.

Start of operations: End of 1939.

Capacity:

Present	Projected
100,000/year to motor gasoline	75,000/year to motor gasoline
170,000/year to diesel oil	123,000/year to diesel oil
25,000/year to paraffine	53,000/year to paraffine
15,000/year to lubricating oil	40,000/year to lubricating oil
310,000/year to	291,000/year to

Raw Materials:

Tar from low temperature carbonisation of brown coal:

From Neukieritzsch	30%
From Bohlen	20%
From Espenhain	30%

Process:

Pressure: 300 atm.

Preliminary preparation of the tar over fixed catalyst (5058) in one step with low but strongly staggered temperatures (inlet temperatures 300°C to 400°C). Practically no cracking, only refining with production of viscous lubricating oils and paraffine.

Number of high pressure stalls:

5 for the TTH step
1 for the vapor phase step

The paraffine is produced by the propane method developed in America for the de-paraffining of petroleum products. Hydrogenation may be performed at somewhat higher temperatures (MTH) and will produce in this case Diesel oil and gasoline. The de-paraffining installation was late in being made ready for operations, and difficulties have been experienced in putting it in operation, and as a result the MTH process was used during the first two years.

Yield is very good: 90% final products, referred to tar.

Hydrogen Production:

The H_2 consumption is very low, only 500 m^3/te of product.
Total H_2 consumption is around 20,000 m^3/h.
H_2 produced in a Winkler generator with lignite coke and oxygen.

Fuel Gas Production:

No production of fuel gas has been anticipated. Requirements are met with the hydrogenation off-gases. However the gasification with TTH is very low and barely sufficient for the fuel gas requirements and a reserve is provided in the hydrogen generator.

Power Requirements:

About 30,000 KW. They have their own power station.

By-Products:

None
No excess gasification.

VII. LUTZKENDORF:

is owned by Wintershall concern.
Operations started in 1940.

Capacity:

15,000/year to, of which 10,000 year/to of Diesel oil.
The expected production 30,000/year to.

Raw Materials:

Coal tar and petroleum residues ("paraffingatsch" and petrolatum).

Process:

Number of high pressure stalls:

Liquid Phase - 1
Vapor Phase - 1

Pressure: 700 atm.

H_2 production capacity about 5,000 m^3/h from 2 H_2-sources from the synthesis installation: 3,000 m^3/h for the hydrogenation proper, 2,000 m^3/h from the off-gas from the catalyst regeneration.

Fuel Gas Production:

The fuel gas requirements are met by the hydrogenation offgas. The deficiency is met from the fuel gases from the adjoining synthesis installation.

Power Requirements:

2,500 kW delivered by the synthesis installation.

The unit was originally planned for the joint operations with tar oil and "kogasin" from the neighboring Fischer installation of Uhde. It was the intention to utilize the H_2 of the "kogasin" for the hydrogenation of the coal tar. However, high pressure experiments in Ludwigshafen have shown that there are no addition effects. The pure octane number of the Fischer gasoline was supposed to be improved by the hydrogenation gasoline and the specific gravity somewhat raised.

Affiliated with the Fischer synthesis installation and the petroleum refinery installation.

VIII. PÖLITZ

Started operation in the middle of 1940.
Founded by foreign petroleum societies and the I.G.

Capacity:

200,000 year/te of aviation gasoline
150,000/year te of high test gasoline (DHD)
About 10% fuel gas

Raw Materials:

Young brown coals from Upper Silesia, pitch, brown coal tar and petroleum.

Method:

Number of high pressure stalls:

Liquid phase: 4 for brown coal
2 for pitch and oil

Prehydrogenation: 5
Benzination: 3
Dehydrogenation: 2

Pressure: 700 atm. in the liquid phase
300 atm. in the vapor phase
50 atm. dehydrogenation

The hydrogenation works receive hydrogenation coal, coke and fuel coal from Upper Silesia. The de-ashing installation is not at the hydrogenation works but at the mines in Upper Silesia.

In the liquid phase hydrogenation of the tar and petroleum residues, the residues are distilled in a vacuum distillation unit and the residue utilized for the oil firing in the power-house.

The hydrogenation residue from the brown coal is used up as an addition to the less valuable fuels.

H_2 Production:

H_2 requirements: 120,000 m^3/h from brown coal coke from Upper Silesia obtained in 8 Pintsch generators of a capacity of 80,000 m^3 H_2/h.

From their own hydrogenation off-gases treated in 12 cracking furnaces of 106,000 m^3 of H_2/h capacity.

Because of the high freight on coke, the hydrogenation off-gas cracking installation is very economical in Pölitz.

Power Production:

About 10,000 kW.

Fuel Gas Requirements:

Around 45×10^6 heat units/h.

The hydrogenation off-gases are worked up for H_2 production in the hydrogenation off-gas cracking installation, and only the residual water gas of the Pintsch coke generators is available. The hydrogenation off-gas cracking furnaces in turn make large demands on fuel gas. There was therefore a relatively large fuel gas installation erected at Pölitz. At present there are 32 fuel gas generators in operation with a gas production each of 6.5×10^6 heat units/h.

By-Products:

Iso-octane from hydrogenation off-gases:
12,000/year te under construction.
59,000/year te alkylate installation under construction.

Affiliated with the neighboring lubricating oil plant of the Norddeutsche Mineralölwerke.

The plant does not depend entirely on coal. It must be shipped by waterways or by railroad from Upper Silesia (or Ruhr).

Originally Pölitz was planned purely as an oil installation. 450,000/year te of gasoline + Diesel oil were to be produced from the cracking residues from Venezuela and Aruba (port of importation?). 100,000/year te coal gasoline was later added, and the plant was laid out finally in such a way that of the 6 available liquid phase stalls 2 were to be used for oil and tar operations, and 4 stalls for coal.

Pölitz is constructed on a particularly large scale with respect to the tankage for crude and finished products of the oil installation as well as for the distillation, and is a model of elasticity of the hydrogenation installation for the conversion of different raw materials and finished products.

IX. BRUX:

Hydrogenation plant of the Reichswerke, Hermann Goering A.G.
Put in operation October, 1942.

Capacity:

Expected production of construction step 1:

150,000/year to automobile gasoline
150,000/year to aviation gasoline

of the construction step 2:

150,000/year to automobile gasoline
150,000/year to aviation gasoline

therefore total: 600,000/year to of products + 90,000/year to of fuel gas.

Raw Materials:

750,000 year to of tar from low temperature carbonization of brown coal. Very high phenol and relatively low-H_2 tar. As a result the quality of the output is high and is intermediate between that from coal and the middle German brown coal. Similar to the Rhein brown coal.

Process:

Number of high pressure stalls:

Liquid phase: four 4-phase stalls
Prehydrogenation: three 4-phase stalls
Benzination: three 4-phase stalls

Pressure: 300 atm. in the liquid and vapor phases.

No peculiarities in the tar hydrogenation. DHD installation for 300,000/year to DHD gasoline under construction.

H_2 Production:

M. Winkler generator. Total H_2 consumption and capacity 80,000 m^3/h.
The first application of pressure conversion.

Fuel Gas Production:

Pressure gas method of the Lurgi. Hydrogenation and coke oven off-gases. The excess fuel gas is piped out through a gas main (19 atm.)

Power Production:

Power production 120,000 kW, produced at their own power station, in addition a connection with a 100 K-volt transmission line.

By-Products:

Phenols:

12,000 te/yr	by dephenolisation of the coke oven and hydrogenation water
18,000 te/yr	from coking gasoline
30,000 te/yr	

Alkylates:

Under construction is an alkylate installation of capacity of 59,000/year te of alkylates.

Unlike Brabag, the coking and hydrogenation are in the same hands.

8 million tons of crude brown coal (30% H_2O, 12% tar) are shipped in; 1 million ton of sifted dust is returned to the syndicate, 7 million ton are coked in 80 Lurgi gas furnaces and produce 700,000/year te of tar and 3.5 million ton as low temperature coke; of these 0.6 million ton are used up in gas production in the Winkler generator and 0.9 million ton in the power station, which means that 2 million ton are obtained from the syndicate. In the second construction stage the coal from Upper Flos will be carbonised, a coal strongly layered with clay. One has to make a decision during the designing of the works between direct brown coal hydrogenation or carbonization followed by tar hydrogenation. The comparison caused a decision in favor of the hydrogenation of tar at 300 atm. against the coal hydrogenation at 600 atm. because the energy utilisation of tar hydrogenation is far more profitable in Brux than direct hydrogenation of coal. The total fuel consumption/te of gasoline is the same, but an excess of low temperature coke is obtained during the hydrogenation of tar which amounts to nearly one-half of the consumption.

Affiliation with gas distributing system and delivery of 40-50 million N m^3 of illuminating gas (19 atm.)

X. RHEINBRAUN:

The foundation of the Rhine brown coal industry started operations August, 1941.

Capacity:

Present	Theoretical Production
45,000/year te motor gasoline	105,000/year te motor gasoline
60,000/year te Diesel oil	120,000/year te Diesel oil
105,000/year te	225,000/year te

or 195,000/year te aviation gasoline.

Raw Materials:

Rhenish brown coal from open cut works with 55% water, 5% ash. Coal very high in oxygen.

With respect to quality of the products obtained, this coal is intermediate between the Ruhr coal and the middle German brown coal.

The products are similar to those from middle German coal or from the tars obtained by their carbonisation.

In addition 30,000 to 45,000/year te of tars and pitch.

Pintsch generator	15,000/year te
Dotternhausen tar	10,000/year te
Wolfersheim tar	2,000/year te
Strassen tar	15,000/year te

Process:

Number of high pressure stalls:

Liquid phase:	four 4-phase stalls
Prehydrogenation:	three 2-phase stalls
Bensination:	one 2-phase stall

Pressure: 700 atm. in the liquid phase
300 atm. in the vapor phase

Residue utilisation: one-half of it in conveyor furnace (Schneckenofen) (as in Leuna) and the other one-half in spherical furnaces: (Kugelofen)(as in Scholven).

The coal is dried at the mine.

Difficulties: Great difficulties with formation of caviar (C_aCO_3 + bitumen), spherical chonchoidal structures, and also from salt deposition. These phenomena may be traced to alkalinity and are elimiated chiefly by neutralisation. They have at present been overcome by sanding operations and chemico-physically by injection of oil.

H_2 Production:

H_2 requirements about 75,000 m^3/h.

Produced from brown coal briquettes in Pintsch-Hildebrand generators, 18 in number, as well as from hydrogenation off-gases in splitting converters, 4 in number.

Fuel Gas Consumption:

About 50,000 N m^3/h with a heating value of 1,700 heating units/N m^3.

Requirements met from excess hydrogenation off-gas and from fuel gas from rotary generators.

Power Requirements: About 70,000 kW, produced at their own station.

Production of By-Products:

DHD installation for 200,000/year te DHD gasoline has been foreseen and is under construction.

Because of its location on the Rhine, Rheinbraun is intended to be enlarged for a production of 700,000/year te of oil and 540,000/year te of gasoline.

XI. UPPER SILESIA:

Founded by the Upper Silesia brown coal group, Hermann Goering works and the state.

The start of operation set for 1943.

Production Capacity:

Theoretical production: 240,000/year te of aviation gasoline
260,000/year te of fuel oil
About 60,000/year te of fuel gas

Considerable expansion is foreseen. In construction steps 2 and 3 the same production capacity is again intended.

Raw Materials:

Young Upper Silesia brown coal.

Nut and calted coal lumps, 5-6% ash.

No de-ashing at the hydrogenation works.

45,000 te hot tar from neighboring coke ovens.

4,000/year te of generator tar from own furnaces.

Process:

Number of high pressure stalls:

Liquid phase:	four 4-phase stalls
Prehydrogenation:	two 3-phase stalls
Benzination:	one 3-phase stall and one 2-phase stall

DHD installation for 210,000/year te is under construction.

Pressure: 700 atm. in the liquid phase
300 atm. in the vapor phase
50 atm. dehydrogenation

The first installation for fuel oil from brown coal, grinding of coal with middle oil, with increased thru-put in the coal stall, paste heat exchanger. Thru-put 67 te/h of coal paste or three times as high as in Scholven.

Total decomposition of the hydrogenation off-gases (pure and rich gases) in a Linde installation.

The second construction program foresees 700 atm. pressure in the vapor phase for mild aromatization to be followed by special combination possibilities with 300 and 700 atm. vapor phase in combination with the DHD installation. Eventually also utilisation of the liquid phase fuel oil in the 700 atm. gas phase.

H_2 Production:

Total H_2 requirements: 135 m^3/h (135,000)
The intended production in the Demag generators using the coke from their own coke ovens.

15,000 m^3 of H_2 from the decompsoition of the hydrogenation off-gases in the Linde installation.

Return of H_2 from Heydebreck (?).

Pressure conversion at 11 atm. is foreseen.

Fuel Gas Production:

The residual water gas from the Demag generators will be used as fuel gas.

There is in addition fuel gas production in their own Bamag - gas generators, with theoretical production of 44 million heat units/h in 10 units.

No gaseous hydrocarbons will be used for fuel gas.

Power Production:

130,000 kW at own generators.
17,000 kW of outside power is foreseen.
Steam requirements: 870 te/h

By-Product Production:

Alkylate:	47,000/year te
Phenol	1,700/year te from hydrogenation off-water
	4,000/year te phenol from the coke oven off-water.

Affiliations of the hydrogenation works: with coke oven plant; hot tar and low temperature coke oven gasoline of the coke oven plant is hydrogenated. The coke is used up in the production of H_2, fuel gas and for power generation. Eventually special combination possibilities of the coke oven plant with the residue utilization (briquetting of the coal with hydrogenation residues).

Affiliation with the I. G. works Heydebreck.
Delivery of 59,000/year te of methane and 36,000/year te ethane for chemical conversion.

Low Temperature Coking of the Coal:

Erected because of the shortage in coking coal and delays in the construction of coke oven plant; the higher tar yield in low temperature coking. The Upper Silesia coal for coking is particularly well adapted because of its low coking temperature, while Blechhammer is to deliver coke for automobile generators.

Process:

Lurgi converters for lump coal (nut or briquettes), 10 units, with a provision of a briquetting plant.

Production:

Low temperature coking gasoline	8,500/year te
Hot tar	45,500/year te
Fuel Oil	50,000/year te
Total	104,000/year te of tar
Coke for the market	270,000/year te
Coke for generators & power station	550,000/year te
Total	820,000/year te

Sternberg/mc/pkl
7-3-1946

T.O.M. Reel No. 181
pp. 537-539

April 20, 1942

[illegible] COAL HYDROGENATION

The table below contains the ultimate analysis of the raw materials and products of coal hydrogenation and the molecular weights of the gross formula estimated from them.

	COAL	HOLD	ORGANIC SOLIDS FROM HOLD	MIDDLE OIL	GASOLINE	GASIFICATION ($C_{1.8}H_{5.6}$)
C	81.97	91.95	88.1	87.13	84.62	79.4
H	5.31	6.13	3.1	9.43	13.73	20.6
O	9.20	0.91	6.7	2.45	1.20	--
N	1.73	0.93	1.0	0.92	0.42	--
S	1.07	0.08	1.1	0.07	0.06	--
Estimated mol. wt.	1,000	400	1,000	180	100	27.2
Gross Formula	$C_{68.3}$	$C_{30.7}$	$C_{73.5}$	$C_{13.1}$	$C_{7.1}$	$C_{1.8}$
	$H_{53.1}$	$H_{24.5}$	$H_{31.0}$	$H_{17.0}$	$H_{13.7}$	$H_{5.6}$
	$O_{6.2}$	$O_{0.2}$	$O_{4.2}$	$O_{0.3}$	--	--
	$N_{1.2}$	$N_{0.3}$	$N_{0.7}$	$N_{0.1}$	--	--
	$S_{0.3}$	--	$S_{0.3}$	--	--	--

We may imagine the following reaction equations using these gross formulae. The heats of the reactions were estimated from the H_2 consumption similarly to analogous reactions recorded in [illegible], February [illegible], 193[illegible].

1) Coal ⟶ organ. solids + 65% middle oil + 10% gasoline + 20% gasification

$C_{68.3}H_{53.1}O_{6.2}N_{1.2}S_{0.3} + 36.6\ H_2 \rightarrow 0.046\ C_{73.5}H_{31}O_{4.2}$
$N_{0.7}S_{0.3} + 3.4\ C_{13.1}H_{17}O_{0.3}N_{0.1} + 0.95\ C_{7.1}H_{13.1} + 7.6$
$C_{1.8}H_{5.6} + 5.2\ H_2O + 0.2\ NH_3 + 0.3\ H_2S + 470$ cal.

2) Coal ⟶ HOLD oil

$C_{68.3}H_{53.1}O_{6.2}N_{1.2}S_{0.3} + 7.6\ H_2 \rightarrow 2.23\ C_{30.7}H_{24.5}O_{0.2}$
$N_{0.3} + 5.8\ H_2O + 0.5\ NH_3 + 0.3\ H_2S + 107$ Cal.

3) Coal ⟶ organ. solids from HOLD

$C_{68.3}H_{53.1}O_{6.2}N_{1.2}S_{0.3} \longrightarrow 0.93\ C_{73.5}H_{31}O_{4.2}N_{0.7}S_{0.3} +$
$9.0\ H_2 + 2.3\ H_2O + 0.5\ NH_3 - 115$ Cal.

4) HOLD oil ⟶ middle oil

$C_{30.7}H_{24.5}O_{0.2}N_{0.3} + 0.5\ H_2O + 7.4\ H_2 \rightarrow 235\ C_{13.1}$
$H_{17.0}O_{0.3}N_{0.1} + 0.1\ NH_3 + 95$ Cal.

5) Organ. solids ⟶ middle oil

$C_{73.5}H_{31}O_{4.2}N_{0.7}S_{0.3} + 35.2\ H_2 \rightarrow 5.61\ C_{13.1}H_{17.0}$
$O_{0.3}N_{0.3}N_{0.1} + 2.5\ H_2O + 0.1\ NH_3 + 0.3\ H_2S + 450$ Cal.

6) Middle oil ⟶ gasoline

$C_{13.1}H_{17.0}O_{0.3}N_{0.1} + 4.6\ H_2 \rightarrow 1.84\ C_{7.1}H_{13.7} + 0.3\ H_2O$
$+ 0.1\ NH_3 + 60$ Cal.

7) Gasoline ⟶ gasification

$C_{7.1}H_{13.7} + 4.2\ H_2 \longrightarrow 3.95\ C_{1.8}H_{5.5} + 55$ Cal.

We cannot discuss these equations on theoretical grounds. An exact computation of equilibria are impossible because of the absence of any foundation. Strictly speaking, Nernst' so-called approximation formula is not dependable because of the large molecular weight changes. (The equilibria for equations 1 and 7 have been calculated and the calculations shown in the appendix as an illustration of the nature of the results obtained with the approximation formula.)

Reactions 1, 2, 4, 5, 6 and 7 proceed exothermally from left to right that is, low molecular weight hydrogen-rich cracking products are formed in equilibrium. The organic solids formation according to equation 3 should properly speaking proceed from right to left. The fact that "organic solids" are nevertheless formed is explained by two causes. They either are formed from particular hydrogen-poor parts of the initial coal, or else by hydrogenation or polymerisation as a result of an insufficient supply of H_2. It is obvious from the fact that much H_2 enters reaction everywhere except in reaction 3 that the H_2 pressure will favor the reactions.

Appendix

Nernst' approximation formula to the empirical reaction equations 1 and 7 is schematically applied below. We must add to the normal inaccuracies of the approximation formula the fact that the so-called conventional chemical contents are unknown for middle oil, gasoline, etc. (they were assumed to be + 3.5) and the heats of the reaction could only be estimated. There is in addition a doubt in the principle of the computation of equilibria of mixtures. It is entirely probable that, for instance, the formation of gases or light benzene fractions takes place from other parts of the raw materials, then, for instance, the formation of the organic solids, so that this kind of partial reaction will proceed with a different H_2 consumption and heat of reaction. Such breaking down of the reaction however cannot be done at will, and, it has been assumed as a first simplification of the computation, that the substances mentioned in the first table are uniform.

The following equilibria are then obtained at 450°C:

1) Coal -> 5% organ. solids + 65% middle oil + 10% gasoline + 20% gasification

Since coal and organic solids are principal substances, the equation is:

$$\log K_p = \frac{P_{H_2}}{3.4 \quad 0.95 \quad 7.6 \quad 5.2 \quad 0.2 \quad 0.3} = \frac{470{,}000}{4.57 \times 723}$$

$$+ 18.95 \times 1.75 \log T\ 23 + 36.6 \times 1.6 - 3.4 \times 3.5 - 0.95$$

$$\times 3.5 - 7.6 \times 3.5 - 5.2 \times 3.6 - 0.2 \times 3.3 - 0.3 \times 3.0$$

$$\log K_p = -142 + 95 - 3.6 = -50.6$$

$$K_p = \frac{p_{H_2}^{36.6}}{p_{M.O}^{3.4}\ p_{gasoline}^{0.95}\ p_{gas}^{7.6}\ p_{H_2O}^{5.2}\ p_{NH_3}^{0.2}\ p_{H_2S}^{0.3}} = 10^{-51}$$

This shows that for 5 atm. the partial pressure of gasoline + middle oil requires a H_2 partial pressure of only 0.1 atm.

2) Gasoline ⟶ Gasification

$$\log K_p = \frac{p\ \text{gasoline}^{3.15} \times p\ H_2^{4.2}}{p\ \text{gasification}} = \frac{-55{,}000}{4.57 \times 723} + 1.25 \times 1.75 \times \log T - 3.6 = 13.9$$

At $p\ H_2 = 200$ atm,

$$\frac{p\ \text{gasoline}^{3.95}}{p\ \text{gasification}} = \frac{10^{13.9}}{200^{4.2}} = \frac{10^{-13.9}}{10^{9.7}} = 10^{-23}$$

that is, gasoline is completely cracked to gas at an equilibria point, with an H_2 - partial pressure of 250 atm.

Sternberg/mc/pkl
7-15-1945

T.O.M. Reel No. 181
pp 507-510

HEAT EFFICIENCY OF THE SOFT COAL HYDROGENATION

The accompanying table shows the heat and energy changes in the amount of coal used in hydrogenation, necessary for the production of 1 to of aviation gasoline. We shall call "heat efficiency" the proportion of the amount of energy contained in gasoline and in the fuel gas to the energy in the coal. As calculated here from energy proportions, it amounts for the high test product of the aviation gasoline to 27.2%

For the production of 1 to of aviation gasoline + 240 kg of fuel gas the total amount of soft coal required is 6.7 to and the heat values are:

Gasoline	10,300 kcal.kg
Fuel Gas	11,000 " "
Coal	7,200 " "

There are 2 energy streams in addition to the 2 non-shaded streams for gasoline + fuel gas:

The excess of hydrogenating gas which is returned as a fuel gas and is therefore returned into the diagram in the upper left hand corner (the percentage value is therefore enclosed in parentheses).

The coking residue which may be burned under the boiler.

The remaining streams laid out are loss streams. The total loss amounts to the difference of 100 - 27.2 72.8%

Of this amount the proportion for the H_2 production	35.5%
For the compression of H_2	9.2%
That is, total for H_2	44.7%

The remaining losses are distributed on the operation of the actual hydrogenation. The unincluded losses are repsented horizontally in the picture as well as losses formed by the heat of the reaction, and amount to a total of 7.5%

The diagram shows therefore that losses incurred in the production of H_2 form the major part, which explains the fact that the heat efficiency of fuel requiring less H_2 is greater.

Under the same conditions it already rises in automobile gasoline to 28.9%

In the production of aviation gasoline + fuel oil in the proportion of the two products of about 1 : 1 it even rises to 35.5%

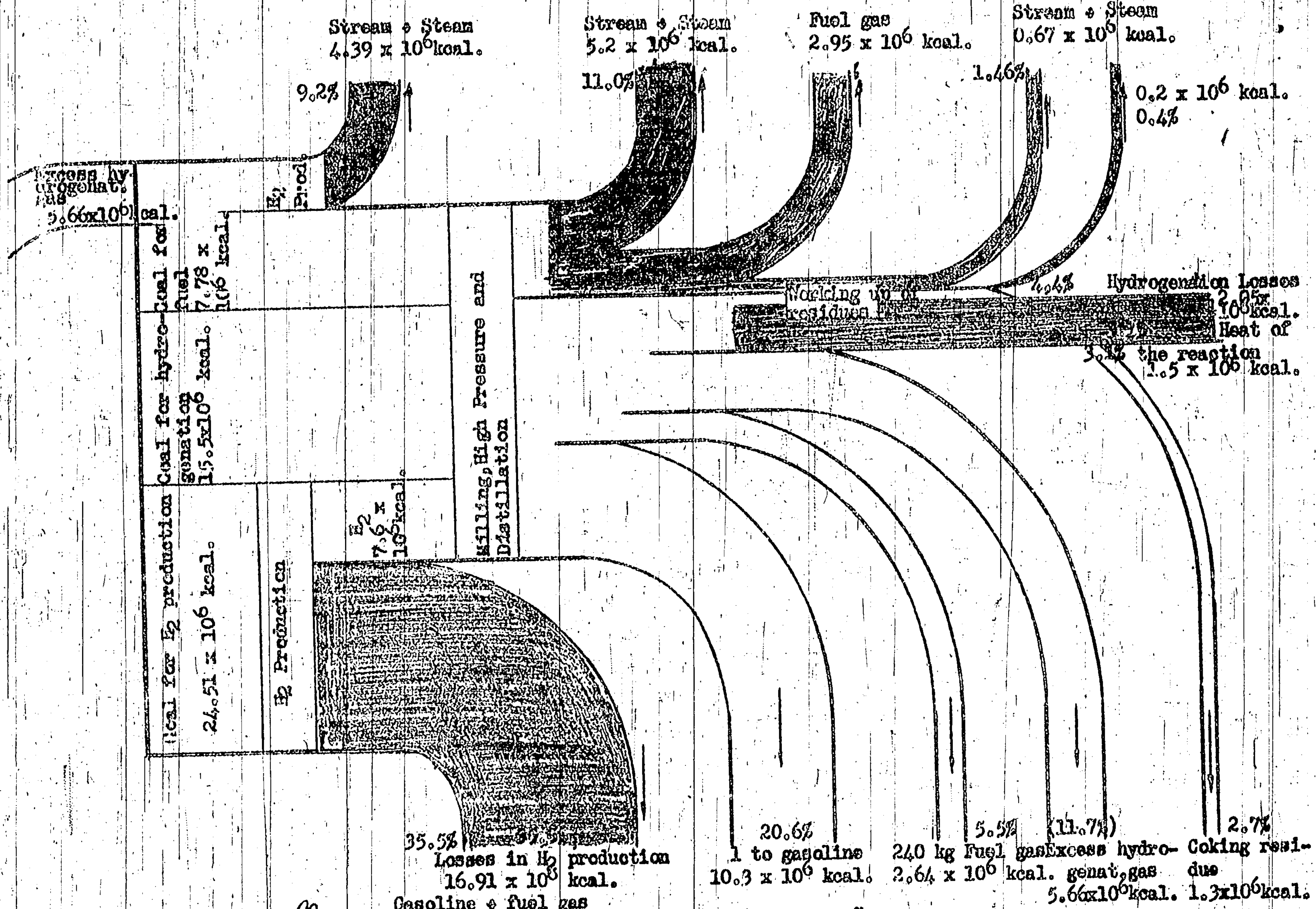
The H_2 production, taken by itself, has a heat efficiency in the water gas process of 26.3%

These low heat efficiencies of the H_2 production are however materially improved when H_2 is obtained by the Linde decomposition process, either entirely, when there is sufficient coke oven gas available, or partially, when the available hydrogenation gases are decomposed.

When hydrogenation is considered by itself, that is, with H_2 production entailing no losses, the thermal efficiency is 49 per cent. This would be the thermal efficiency of soft coal hydrogenation when H_2 is bought under 1 atm pressure.

However, when the heat efficiency of soft coal hydrogenation is around 30 per cent, we are dealing with a great increase in the worth of the coal, and the final production must not even as a first step be judged from the heat efficiency.

Sternberg/mc/pkl



$$\eta_{Hyd.} = \frac{\text{Gasoline} + \text{fuel gas}}{\text{Coal for } H_2 + \text{Coal for Hydrogen} + \text{Power Coal}} = \frac{(10.3 + 2.64) \times 10^6}{24.51 + 15.5 + 7.78} = \underline{27.2\%}$$

Heat Efficiency of Hydrogenation, incl. H_2 Production

T-49

T.O.M. Reel 11
Target 30/4.09
Bag No. 2247 - Item 12

Scholven, Jan 10, 1939.

Sulfur balance (on hydrogenation side) at the Scholven III installation (S/t gasoline and S/h at 250,000 Year-tonne)

	S/t gasoline	S/h
Sulfur intake:		
a). 2 t. dry coal/t gasoline, with 1.05% S	21 kg.	
b). Lump sulfur during hydrogenation over 5058 and 6434/t gasoline	10.5 kg.	
Total S intake	31.5 "	
With 250,000 year-tonne and 330 x 24 = 7920 hours operation, 31.6 t gasol./h		995 kg.
S contents:		
1): Washwaters:		
a) liquid phase catch pot liquid 12.5 m^3/h, with about 20 g S/h		250 "
b) vapor phase catch pot liquid 6 m^3/h, with about 40 g S/h		240 "
c) hydrogenation gas washer 4 m^3/h, w. abt. 15 g S/h		60 "
	Total	550 "
2) Wash alkali, 2 kg/t gasol.,		60 "
3) Kiln residue 210 kg/t gasol., 1.5% S	3.6 kg.	114 "
4) Low H_2S off gases:		
a) 869 m^3/h coal stall off gas, I-st step 2.8 g H_2S/m^3		2.3 "
b) 7097 m^3/h circulating gas wash I step 2.6 g H_2S/m^3		15.0 "
c) 1577 m^3/h vapor phase stall off gas, I step 6.0 g H_2S/m^3		9.5 "
d) 830 m^3/h kiln gas 2.3 g H_2S/m^3		2.0 "
e) 1138 m^3/h HOID gas 4.5 g H_2S/m^3		4.9 "
		757.7 "
5) High H_2S gas with over 8 g H_2S/m^3:		
a) 7655 m^3/h coal hydrogenation gas (coal stall off gas, step 50/1) (Circulating gas, " 50/1) (" " " vacuum) (A distillation with 20.4 g H_2S/m^3, average	156 kg H_2S/ h	147.0 kg
	156 "	904.7 "

b) 1967 m^3/h vapor phase hydrogen gas (vapor phase stall, step 30/1) (B distillation, stabilization with 36.1 g H_2S/m^3	71 kg.	67.0 kg
Total	227 kg.	971.7 kg.

This equals within 2.9% the sulfur intake of 995 kg. About 214 kg. H_2S/h were available from the high H_2S gases. With a 95% conversion into 65.9° Be sulfuric acid, 620 kg/h, or 14,900 kg/day of the acid have been produced.

Signed Moller

SGMiller/ /pkl
7-17-1946

Gelsenberg Benzin A.G.
Hydrogenation Div./Dr. At.

Gelsenkirchen - Horst, 9/2/43
T-50

Translation of Ditto Copy in Dr. L. L. Hirst's Possession

Report of the Dismantling of Stall 306 After the Third Operation Period

Prior to rebuilding the stall into three stage operation, converters 1 and 2 were blown out with nitrogen for the removal of catalyst dust because of indications they gave of increased resistance. This proceedure appeared at first to be successful (judging from the measurements made at atmospheric pressure, column 1 on the table below), but difficulties were met immediately upon the resumption of operations. Increased pressure differences (12.7 atm) were found in converter No. 2. The values in converters 1 and 3, 1.3 and 1 atm. respectively, were normal. The total difference in the stall amounted to 20 atm. with 13 m^3 injection. The resistance kept steadily increasing during the operation period. After a failure of the current on 3/22/43 the pressure increased 5 atm. more, and the total pressure difference in the stall amounted to 30 atm. It had, therefore, been decided to discontinue operations of the stall and to rebuild the converter because of the resistances obtained.

The control division thereupon measured all the converters under atmospheric pressure:

Converter	At the start of the Operation Period	After suspending Operations.
	mm. water column	mm. water column
1	120	140
2	96	190
3	72	80

The resistance of converters 1 and 2 was too high, and both were emptied. When the converter operations were suspended the catalyst in converters 1 and 2 was 511 days old, in converter 3-- 205 days old. 1 & 2 were emptied on July 31 and August 6 without any difficulties and refilled on August 4 and 8th with the same kind, but well sifted catalyst.

During the emptying of converter 1, much dust has been found in the upper part. The dust contained:

73.41% FeS
46.91% Iron
26.80% Sulfide S
14.92% WO_3
12.47% L.O.I

Inside converter 1 (102) a larger iron-sulfur deposit has been found in the catalyst tube and upon the charge. In converter 2, there was a heavy caked layer of catalyst, as well as much dust. The decomposition of the catalyst, and the formation of the layer connected with it may have been caused by a temporary infiltration of air in February 1942.

No dirt was found in dismantling the thermocouple openings.

The control division determined the resistance of converter 1 (1017) to be 83 mm after refilling and of converter 2 (1020) to be 80 mm water column.

Heat exchangers 602 and 601 were replaced with 611 and 623, because the tube bundles either had an increased resistance, or were not tight. The preheaters and the pipe line were cleaned. The FeS deposit of the pipes and hair pins amounted to 0.5 - 1.0 mm. In addition, a system of 70 mm valves was added at the inlet of the gas cooler to detect acid formation during operations.

Signature not decipherable.

F.O.S. REEL No. [illegible]
pp. 499-501

SUMMARY OF RESULTS OF ROUND TABLE DISCUSSIONS ON THE SULFURIZATION OF MIDDLE OIL

	GELSENBERG		NESSELING		SCHOLVEN	
	Prehydrog.	Benzination	Prehydrog.	Benzination	Prehydrog.	Benzination
% S in fresh feed	abt 0.1	insignific.	0.1	insignific.	0.1	0.1-0.05
" in inject. feed	abt 0.1	"	0.1	"	0.1	-
" in circ. gas	0.017-0.0425	0.019-0.071	0.08	0.08	0	0.54
" addit. as H_2S	0	0	0	0.5-0.6	0	0
" " " element. S	— 0.2	0.4-0.5	0	0	0.1	0.3-0.35
Total S in react. space, referred to injection feed	0.317-0.3425	0.419-0.571	0.18	0.58-0.68	0.2	0.94-0.94
% phenols in fresh feed	13	-	15-20	-	13	-
" inject. "	13	0.02-0.04	15-20	0.05	13	0.05
$\%O_2$ in fresh feed	-	-	2.5	-	-	-
Special observations at the preheater	Electr. preh used only when stopp. or starting No disturb. incrustat.	A single case of plugging-up of preh. hairpins w sulfide crust	-	No experience as yet available	Incrustation in preheater, not growing	Very slight deposition of soluble salts in preheater
Special observations in heat exchangers	At start oft plugging-up of h.exch. behind converter - cracking of sulfide crust. Not caused by sulfuris but by increased Cl content with increase in tar oil (50%)	No stops from cracked-off sulfide crust. K value deteriorated with 6434, occasionally very quickly and strongly. Catalyst long in use showed no change upon dismantl	-		Deterioration of heat transfer, esp. in cold h.exch. by iron sulfide, org. matter and much sol salts (NaCl, Na_2SO_4 $(NH_4)_2SO_4$) - They come in part from sulfur, rest from moist middle oil.	Deposition of water soluble salts in h.exchanger is very slight.
Special observations in catchpot	-	Occasionally contaminated with small amounts of black FeS slime.	-			
Other observations on sulfurizing injection feed.		Occasionally when over-sulfurizing the B middle oil with el. S (up to 1%) some S deposited in			The injection feed is filtered through filter candles charged with 1 cm. thick pills.	

ELHEIM aromat.	LUTZKEND. Benzinat.	BRUX Prehydr.	BRUX Benzinat.	BÖHLEN Prehydr.	BÖHLEN Benzination	PÖLITZ Prehydration	PÖLITZ Benzination
-	-	0.34	-	1.0-1.35	0.18-0.40	0.05 -0.10	0.00
0.1-0.2	0.47	0.34	0.01	1.0-1.35	-	0.05 -0.10	0.00
?	traces	abt 0.78	abt. 0.9	0.53-2.46	0.53-2.46	0.005-0.0125	0.16-0.34
0	0	0.22	0.48	yes	yes	(0.2-0.3 at earl. date)	0.2 -0.3
				present in circul. gas			
0	0	0	0	0	0	0.2 -0.3	0
	abt 0.47	1.26	1.33	1.53-3.81	0.71-2.86	0.255-0.4125	0.36-0.64
-	-	abt 15.5	traces	5 - 7	-	12-15	-
-	0.811	abt 15.5	"	5 - 7	1-1.4	12-15	0.01
-	-	6 - 7	0	2 -3.63	0.17-0.90	2%	0.00
-	none	none yet	none yet	formation of a layer of FeS		A stronger reduction of heat transfer in the total operations than in benzination.	No reduction in K values
	none	uniform reduction in K value	none yet	formation of a layer of iron sulfide		This happens with and without sulfurization by deposition FeS caused by the phenol content. The deposition is favored by addition of sulfurization agent, in particular H_2S.	No reduction of K values. (According to older observations, S deposited when sulfurizing with elem. S when the injection feed was too light.)
-				-	-	Action of H_2S in presence of phenol at 40° even at low pressure. Part of the phenolates are destroyed and FeS is deposited in a finely divided form. (filtration difficult). Use of lump S temporarily introduces some contamination,	
		none	none	none	none		

		cold catchpot with resultant large pressure differences.				
Results when sulfurizing with H_2S with the concentration of H_2S in the gas below 80%	-	-	-	-	-	-
How is sulfurizing done:	Element. S	Element. S		80-90% H_2S Best w. CO_2	Elementary S	Elementary S
Temper., Press.				25-30° C, 1 m. water column	45°C, 200 mm water column	45°C, 200 mm water column

Remarks: Sulfurization with the elementary S is done using their own S (Claus unit). Sulfurization with H_2S is foreseen and kept ready for use but not as yet used, because the experience of sulfurizing with the elementary sulfur has been satisfactory.

						filtration also difficult.	
				-	-	Sulfurized only at concentrations below 80%. No relationship has been found between this concentration and damage to catalyst.	
	See below	79.4% H_2S + 20% CO_2	79.4% H_2S + 20% CO_2	62-67% H_2S balance CO_2		50 - 60% H_2S 50 - 40% CO_2	50 - 60% H_2S 50 - 40 CO_2
		43°C and 600 mm w.col.	39°C 600 mm w.col.	H_2S added to the rich gas compression and thus directly to circ gas.		40°C, 400 mm (elem. S at 40-50°C, no press.)	40°C, 400 mm

*The circulation connections show that some H_2S reach prehydration with the outlet gases, and their quantitative estimation is impossible. The same is true of benzination.

In Lutzkendorf: Addition of sulfur is made occasionally when low sulfur injection feed is used; this is done by conducting the sulfur-rich circulation gases from the pressure side of the liquid phase to the suction side of the circulation gases of the vapor phase.

The sulfur values in the vapor phase circuit have recently been greatly increased by the use of a high-sulfur vapor phase injection feed. No definite information is as yet available.

Sternberg/kp

7/17/46

Newman

T.O.M. Reel No. 130
Pages 246 to 320
Table & graphs, Reel 181
Pages 215, 217, 218 & 219

T-52

August 4, 1943

LARGE SCALE FUEL OIL PRODUCTION EXPERIMENTS

Ludwigshafen 1941

SUMMARY

The effect of the coal throughput and of the composition of the vehicle oil, in particular the effect of different middle oil contents in the vehicle oil has been tested in small scale experiments of hydrogenation of coal. In the course of that work possibilities have been shown of increasing the throughput through the converter when producing heavy oil together with middle oil. The proper industrial detail for the production of heavy oil had to be developed first on a semi-commercial plant.

The Upper Silesian coal lent itself readily to the study of production of gasoline, middle oil and heavy oil at 700 atm. in a 1.6 m^3 converter and to relate the results with the small scale experiments (10 liter converter).

In addition, paste heat exchange was carried out successfully.

The following experiments, among others, were carried out: Mixed coal (Beuthen and Heinitz collieries, 1:1) were hydrogenated to 100% gasoline plus middle oil, to 65% gasoline plus middle oil, + 35% heavy oil, as well as to 50% gasoline + middle oil + 50% heavy oil, in part with the usual iron catalyst combination, in part with sulfurated Bayer mass together with iron sulfate and sulfigran. The Heinitz coal has been hydrogenated with the usual iron catalyst to 65% gasoline + middle oil + 35% heavy oil.

The most important resulting balances are summarized in the following table:

Coal	Beuthen + Heinitz mines 1 : 1				Heinitz coal, alone
Method	100% gasol. + middle-oil	65% gasol. + m.o., + 35% heavy oil	50% gasol. + m.o. + 50% heavy oil	50% gasol. + m.o. + 50% heavy oil	65% gas. + m.o. + 35% heavy oil
Catalysts:	1.2% $FeSO_4.7H_2O$ + 1.5% Bayer mass + 0.3% sulfigran (Na_2S)			1.2% $FeSO_4.7H_2O$ + 1.5% sulfided Bayer mass + 0.3 % sulfigran	1.2% $FeSO_4.H_2O$ + 1.5% Bayer mass + 0.3% sulfigran
Pure coal throughput	0.45	0.61	0.65	0.62	0.60
Utilisation	97.0	96.6	96.2	96.4	95.5
Total yield	0.26	0.38	0.41	0.41	0.38
Gasoline + m.o. yield	0.25	0.25	0.19	0.20	0.25
% Asphalt in M.O.L.D.	14.6	15.6	17.1	16.3	18.2
% gasification/actual oil production + gas	23.4	20.5	19.0	18.0	(18.6)
Composition of actual oil produced:					
% Gasoline	21.8	23.5	23.5	22.2	23.7
% Middle Oil	74.0	41.7	22.2	27.4	43.3
% Heavy Oil	4.2	34.8	54.3	50.4	33.0

The figures show, that in the process for 65% gasoline + middle oil, + heavy oil the same gasoline + middle oil production is obtained (0.25) as in the process for 100% gasoline + middle oil, and in addition, heavy oil with a throughput of 0.13 is produced. In the process for 50% gasoline + middle oil + 50% heavy oil the capacity for heavy oil is increased at the cost of gasoline and middle oil production.

No advantage has been found in the substitution of sulfurized Bayer mass as a catalyst for the usual Bayer mass.

The Heinitz coal alone was distinctly poorer than the 1:1 mixture of Beuthen and Heinitz coals. 1/

1/ Gasification values are somewhat uncertain.

Curves, sheet 1, show the results obtained in the different processes with the 1:1 mixture of Beuthen and Heinitz coals. Comparisons were based on the proportions in the oil production. In the processes for heavy oil production the utilization is somewhat lower than in the process for 100% gasoline + middle oil. The higher throughput in the process for heavy oil increases the asphalt content in the H.O.L.D. A limit to the throughput is given in the normal methods of utilization of residues, in centrifuging and kiln operations. The maxima of 17 - 18% asphalt in H.O.L.D. obtained in the semi-commercial tests could still be recovered by the residue utilization methods without much trouble. Gasification referred to the oil produced + gasification is naturally lower in the processes for heavy oil production than in the complete break down of the oils to middle oil.

The change of the gasoline and middle oil process to the heavy oil is done by using either a mixture of middle and heavy oil, or else of middle oil alone, in the dilution during centrifuging, against the use of heavy oil alone in the process for gasoline + middle oil. The maximum converter temperature is practically the same in all the processes.

The tests gave the following principal results:

The high throughputs used in the heavy oil process could be mastered with no difficulties nor has the presence of middle oil resulted in any complications in the vehicle oil.

About 60% of purified coal in the form of 42% paste (32-33% pure coal) was heated by heat exchange through the temperature interval 250-410° using the hot mixture of the products and gases behind the hot catch pot. The heat transfer numbers remained practically constant in the heat exchanger with this concentration of solids during 7 weeks of uninterrupted operations. The concentration of solids in the paste was raised for a short time to 47%. This concentration of solids has caused a disproportionate reduction in the heat transfer and introduced the additional danger of formation of excessive pressure differences. Dismantling of the heat exchanger has shown no formation of deposits either in the forward or in the return pass.

The dismantled apparatus has shown moreover the following: the incrustation in the hairpins of the hot and the cold passes were normal when 1 - 2 mm. thick. Contrarywise even in the relatively short period of use, the third pass (thick pass component) had a fairly heavy incrustation. It appears that the low velocity (0.7 m/sec) at that viscosity is the primary cause of incrustation.

The lower third of converter No. 1 was filled with fine coal, converter No. 2 was clean. The inside of the hot catch pot was found upon dismantling to be in perfect condition, nor were there any coke deposits in the funnel.

No important operating difficulties have been encountered in the grinding of coal, preparation of paste or working up of the residues. When puremiddle oil was used as a thinning oil in centrifuging a slight asphalt precipitation was observed in the centrifuging.

INTRODUCTION

The large scale fuel oil tests were made in the 1.6 m^3 converter 804 at 700 atmospheres from July 15, 1941 to September 21, 1941.

The purpose of the tests was:

1) To adapt in pilot plant operations the process developed in small scale tests to the production of gasoline and fuel oil from coal; creating the foundation for designing the hydrogenation works at Blechhammer by utilizing the Upper Silesian coals as the raw materials.

2) To determine the optimum conditions for heat exchange heating of the coal paste.

DIVISION OF THE REPORT

1) Experimental Procedure
2) Raw Materials
3) Running of the Tests
4) Experimental Results:
    A) Balances and Yields
    B) Analyses of the products obtained
    C) Paste Heat Exchange
    D) Observations on Dismantling

1. EXPERIMENTAL PROCEDURE

A. Stall Design

Preheating

1. Gas preheater: Jacketed coil, 20 atm. steam, N_2, 24 mm. dia., 120 m. long, [illegible] m^2 heating surface, routing of the intake gases to the passes for the thick and the thin paste.

2. Gas heated preheater: 3 ring burners, fire box of the Ludwigshafen system, 3 heat passes.

Heat pass I: Preheating of the thin paste, 4 hairpins, N8, 45 mm. dia., 7.5 m. length of leg, $Fa = 224\ m^2$, $Fi = 8.6\ m^2$.

Heat pass II: Preheating of the thick paste, 6 hairpins, N10, 45 mm. dia., 7.5 m. long, $Fa = 336\ m^2$, $Fi = 12.7\ m^2$.

Heat pass III: Final preheating of the mixture, 3½ hairpins, N10, 34 mm. dia., 7.5 m. long, $Fa = 130\ m^2$, $Fi = 5.5\ m^2$.

3. Heat exchangers: 3 heat exchangers for heating of thin paste.

Exchanger III: 600/850 dia., 12 m. long, 30 tubes 18 mm. dia. 4 x 11 m. long, cross section of stream $Fi = 23.5\ cm^2$, $Fa = 65\ cm^2$, average heating surface $15.3\ m^2$.

Exchangers II and I: 240/160 dia. x 8 m. long, 22 tubes 18 mm. x 4 x 7080 mm. long, average heating surface $7.3\ m^2$, cross section of stream $Fi = 17.3\ cm^2$, $Fa = 46\ cm^2$.

4. Converters: 2 Converters

Converter I: 600/850 mm. dia., 8 m. long, reaction space 330 mm. dia., 955 liters.

Converter II: 600/850 mm. dia., 12 m. long, reaction space 330 mm. dia., 955 liters.

Combined reaction volume: $1.6\ m^3$

5. Neutralization tube: 120 mm. dia., 13.2 m. long.
(The neutralization tube was provided for the unperformed tests with chlorine as a catalyst.)

6. Hot catch pot: Normal construction, 600/850 mm. dia., 7 m. long. Total volume 540 li., capacity of funnel 57 li., funnel cooled with gas; hot circulation, the H.O.L.D. introduced over the edge of the funnel.

7. Cooler: 2 Air Coolers, connected in parallel both with an I.D. of 24 mm., 35 m. long, 71 $mm.^2$ ribbed surface.

2 Stage Coolers connected in parallel, each with 10 stages, total cooling surface $43\ m^2$, the three hottest stages made of N8, the balance of N2.

8. Catch pot:	Horizontal 700 atm. product catch pot of 400 liter capacity. A horizontal 50 atm. intermediate pressure-reduction vessel 400 liters capacity.
	Group of valves arranged as usual.
9. Gas circuit:	Circulating gas scrubber: A double jacketed tube inclined upwards, N 2, 34 mm. dia., 50 m. long.
	Water catchpot, vertical, 400 liters capacity.
10. Circulation gas oil scrubber:	1 scrubber 640/900 mm. dia., 5850 mm. long, filled with Raschig rings.
	Catch pot: 145 mm. I.D. x 3500 mm. long, 55 li. capacity.
11. H.O.L.D. line:	1 Air Cooler, 24 mm. I.D. 29 m. long, 30 m^2 ribbed surface.
	2 variously operated groups of valves; throttling valves with 3 mm. nozzles; 2 H.O.L.D. containers, each 1 m^3 capacity.
12. Cold gas connections:	In front of heat exchanger III, in front and in the converter, in front of the neutralization tube and in front of the H.O.L.D.
13. Cooling gas:	In the funnel of the hot catchpot; gas was returned to the outlet gas behind the heat exchanger.
14. Fresh gas:	Addition behind the pressure flask of the circulation pump.
15. Cold oil connections:	In front of converters I and II.
16. Preparation of thin paste:	The thin paste was prepared from the thick paste and the centrifuge oil on the high pressure side behind the pumps with no special mixing installations. The mixing distance to the gas inlet was about 60 m. long.

B. Coal Grinding Installation and Pasting.

The crude coal delivered from the coal pile was carried on a conveyor over a magnetic separator of the impact mill with no screen to a breaker. The crushed coal was then brought to the two Fuller mills from two supply bins.

The finely ground coal, still moist, passed through a Hilburg scale and was then sprayed in a pugmill (Hauerschnecke) with 50% iron sulfate solution (catalyst I) heated to 60 - 80° and mixed in a screw conveyer with the solution. Coal containing about 10% H_2O was next dried in a tubular drier heated in an atmosphere of N_2 as a circulation gas with 4 atm. steam to a water content of 0.5 - 1%. The dew point of N_2 circulated by a fan is lowered by spraying water in a Horden washer, after depositing the entrained coal dust in a cyclone. Dried Bayer mass ("red mud") and sulfigran (catalysts II and III) are added to the dried coal by way of a proportioning feed into the front part of a second pugmill.

The pasting with oil follows the addition of the catalysts in a rod mill, with the coal added to the continuously re-pumped oil until the desired coal concentration has been obtained. The paste is sent to the pumps from the coal paste storage installation through an annular pipe line. Screens (pressure filters) are built-in in front of the storage vessels and of the supply pipe line. The double screening of the paste was made to exclude the difficulties resulting from the presence of coarser particles during the heat exchange.

C. Catchpot Distillation

The kiln oil is added to the catchpot feed prior to distillation which is made in a column 22 m. high and 1,200 mm. cross section (the diameter of the lower narrower part is 650 mm.), to which was joined a side column 6.7 m. high and 600 mm. wide for the separation of gasoline. The column was subdivided by division plates which were filled with Raschig rings of 50 mm. diameter. About 320 kg. of steam was injected with a 1,200 kg. hourly injection.

D. Working of Residue

Centrifuging

The H.O.L.D. was collected and hourly weighed after its pressure was brought down. It was mixed with a diluting oil by weight in the Wiege mixing tank. The ready centrifuge mixture was then pumped in storage containers of the centrifuge unit. From these tanks the mixture was sent through a jacketed pipe line where it was heated by steam at 20 atm., then fed into the baskets of the centrifuges. At the outlet of the overflow tank replaceable screens (0.25 mm.) were placed. The centrifuging was done on Laval centrifuges of the most recent construction (maximum r.p.m. 3,200). The mixture was kept at 160 - 170° when operating for gasoline + middle oil (heavy oil dilution), while when operating for gasoline + fuel oil (with middle oil in the dilution oil) it was heated to 140 - 145°C. In either method the amount of centrifuged material did not equal to the hourly production of the H.O.L.D., and periodic operations were necessitated. The thru-put through the centrifuges was 3 - 3.3 te/h.

Kilns

The distillation of the residue from the centrifuge was done in a semi-industrial ball kiln (Kugelofen). The kiln drum was 9 m. long and 1.5 m. in diameter. The separating wall was extended to within about 1 m. from the outlet end. In operations for fuel oil, products containing middle oil were distilled and the kiln was fitted out with an electrical pressure preheater of the same kind as used in the vapor phase preheating. It consisted of 11 vertical №6 tubes 30 x 48 x 10,000 mm. long. A de-compression valve group was located between the preheater and the still inlet (the usual throttling valves, Patronenventils). The still was filled with 98 sharp-edged impacting pieces (eight weighing 24 kg.) and 103 balls (13 kg. each). Normally the still was operated with 12 revolutions, with a chance to double the number of revolutions. N_2 preheated in a muffle was used as the flushing gas and was blown in at the outlet end. The preheater was operated at 20 atm., the feed was heated to 290 - 350°. With 800 - 1,000 kg. hourly injection 20 m^3 N_2 were added for the preheating and for the removal of the gases.*

At the end of the test the outlet shute of the still was replaced by a simple outlet tube. The product through an oil trap and a perforated tube (vertical tube with 4 holes 11 - 14 mm.) in a thin stream into a granulating trough with a worm drive, filled with water. To avoid an excessively long stay of the material in the ball kiln holes were placed near the lower edge of the closing plate of the outlet head, which helped keep the level in the kiln as low as possible.

E. Preparation of the Vehicle Oil

The vehicle oil was obtained by proper mixing of the centrifuge oil and of the preheater kiln oil to which H.O.L.D. was added in the process for heavy oil production.

The thin paste was thinned to the desired solid contents by the addition of pure centrifuge oil.

2. RAW MATERIALS

A. Coals

The tests were made with coals of the Upper Silesian coal bed, using coals of the Beuthen and Heinitz collieries. The selection of coals and their grading was done at the Upper Silesian Hydrogenation Works in conjunction with the Upper Silesian Coal Syndicate, under consideration of subsequent requirements for use of these coals in the hydrogenation works at Blechhammer. High pressure experiments on a small scale have been run at Ludwigshafen with these coals.

The coals used in these experiments were washed to the required ash content in the cascade washer of the Hohenzollern mine (Upper Silesia) industrial unit. It was economically impossible to bring down the ash content to the desired amount of 4% to the weight of the dry coal, and the mixed coal used had an average ash content of 5%. The washed coal was delivered in covered railroad cars and was then kept in bins according to grades.

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*This high thru-put could not be maintained at the beginning of the production for gasoline + middle oil, because the output of the stall was lower.

Coals Received, By Their Origin, Grade and Amount

Origin	Grade	Amount in Tons	% H_2O	% Ash/Dry Coal
Beuthen	Fines 0 - 8 mm.	885	8.8	4.0
Beuthen	Nut 8 - 25 mm.	1,092	9.5	6.1
Heinitz	Nut 8 - 30 mm.	2,010	7.6	5.0
Heinitz	Fines	85	11.0	3.8
Heinitz	Nut + Fines	13	10.0	4.8
	Total	4,085	Av. 9.5	Av. 5.0

The mixing of the coals (coals from the Beuthen and Heinitz collieries in proportion of the delivery of the grades of coal) was done by weight during transportation to the coal grinding unit.

The analysis of the coal is shown in table 1. All coal contained 81.6 - 82% carbon in the pure coal. The coal contained 4% of available H_2 and 11.0 - 11.7% of oxygen referred to pure coal. The volatiles in the pure coal were 37 - 38%.

B. Catalysts

The usual iron catalysts and sulfigran were used with the sulfurized Bayer mass used in the test Period 4. Table 2 gives the analyses of the catalysts. The Bayer mass was added dry. The sulfurization of the Bayer mass was made in a worm driven furnace at 400°, using gaseous H_2S. When so treated, the Bayer mass contained 19% of sulfur.

C. Vehicle Oil

The test was started with vehicle oil and H.O.L.D. oil from the Gelsenberg Benzin A. G. Table 3 shows that the Gelsenberg vehicle oil contained 10.5% solids and 7.8% s-asphalt in the oil.

3. DESCRIPTION OF EXPERIMENTS

According to program the tests were made under 700 atm. total pressure in 5 test periods.

Test Period 1: Mixed coal (Beuthen and Heinitz coals, 1:1) operated for gasoline and middle oil, July 15 to August 5, 1941.

Test Period 2: Mixed coal (Beuthen + Heinitz coal 1:1) worked for gasoline + 35% heavy oil, August 6 to August 18, 1941.

Test Period 3: Mixed coal, 1:1, for gasoline + 50% excess heavy oil, August 19 to September 2, 1941.

Test Period 4: Mixed coal, 1:1, gasoline + 50% excess heavy oil, using sulfurized Bayer mass, September 3 to September 10, 1941.

Test Period 5: Heinitz coal operated for gasoline + 35% excess heavy oil, September 11 to September 21, 1941.

TEST PERIOD 1

July 15-16, 1941, filling of the stalls with gas thru-put (1,000 cub. m/h through the thick paste pass, 800 cub. m/h through the thin paste pass), 700 atm. beginning with 114° 500 kg/h of Gelsenberg liquid phase middle oil through both passes of the paste, 30 li. of water behind catchpot.

July 17, 1941, the hot circuit (400 kg/h) and the washer started at 288° converter temperature, volume of gas through the thick paste pass reduced to 900 cub. m/h. The gas through the thin paste pass was divided; 1,200 cub. m. through the pass 1 of the preheater, 500 cub. m. first through exchanger III because the outlet of the preheater pass I indicated a higher temperature than the heat exchanger return.

July 18, 1941, change of the paste to 70% of the Gelsenberg vehicle oil + 30% of the Gelsenberg catchpot heavy oil, temperature raised, injection into the thin paste pass increased to 800 kg/h, hot circuit operated at 800 kg, inlet gas reduced to 700 and 1,400 cub. m.

July 19, 1941, the thick paste pass was started on 40%-solids coal paste at 408 converter temperature, and the injection set at 700 kg. The paste consisted of mixed coal (Beuthen and Heinitz, 1:1) with 1.2% $FeSO_4 \cdot 7 H_2O$, 1.5% Bayer mass, 0.3% sulfigran and Gelsenberg vehicle oil. The thin paste pass (the heat exchanger pass) was at first operated with a mixture of 5 parts coal paste and 3 parts vehicle oil (24% solids in mixture), 30 kg/h of water injected into the return of heat exchanger III.

July 20, 1941, condition of converters. Converter 1, maximum 466°. The paste injection through the thin paste pass was increased to 1,000 kg/h, the solids increased to 30% by reducing the amount of addition of vehicle oil. The amount of flushing water was increased by 50 kg. each in the heat exchanger III return and behind the catchpot.

July 21, 1941, the maximum converter temperatures in I and II - 471°. Concentration of thick paste 41% solids, and thick paste 31% solids.

July 22 to 24, 1941, 40% paste run through both paste passes.

July 25, 1941, the thick paste concentration increased to 43%, while the 40% paste passed through heat exchangers.

July 26 to August 5, 1941, the concentration of the thick paste was increased during that time to 50 - 51% solids, while the thin paste was retained at 40 - 41% solids. The following amounts of injection and gas were finally set for gasoline and middle oil production: 1,000 lbs. thick paste and 900 cub. m. gas in the thick paste pass, 900 kg of thin paste (700 kg thick paste + 200 kg vehicle oil) and 1,500 cub. m gas in the thin paste pass corresponding to concentration of 45 - 46% of solids in the converter intake.

The average temperature in the converters was 476°, the maximum temperature 480°, and the intended operating conditions have been reached, when about 1,200 cub. m. of cold gas was used. The upper outlet temperature of the hot catchpot was 426°.

There have been no important breaks in this operating period of the high pressure unit.

TEST PERIOD 2

August 6 to 9, 1941. Change-over to the fuel oil production. The centrifuge thinning oil was changed from 100% heavy oil to 50% heavy oil and 50% middle oil, and the H.O.L.D. was re-used (about 16% calculated to the vehicle oil). The amounts of gas were kept at 2,600 cub. m. as previously, the thru-put in the thin paste pass was raised to 1,800 kg. (1,400 kg. thick paste + 400 kg. vehicle oil), and 1,000 kg. of 50% thick paste were run through the thick paste pass while the hot circulation remained 800 kg. as before. Increasing the thru-put caused considerably better heat exchange action in the cold heat exchanger.

August 11 to 12, 1941, the average converter temperature was 478°, the total cold gas 1,500 cub. m.

August 14 to 16, 1941, the concentration of the thick paste was raised to 52% solids. As a result the H.O.L.D. solids increased slowly to 26% which was attributed to poor operators of the hot circuit and was equalized by reducing the outlet temperature of the catchpot from 426° to 417°.

The stall difference rose to 28 atm. which forced the lowering of the thru-put of thick paste to 800 cub. m., and of the thin paste to 1,400 cub. m., while simultaneously temporarily increasing the water injection. In this way the stall pressure difference was reduced to 23 atm. As a safety measure the step cooler and the outlet pipe lines were heated.

August 17, 1941, the injection was finally raised to 1,050 kg. thick paste and 1,930 kg. thin paste (1,450 + 480).

The heat exchanger III now operated so well that the cold gas addition in front of the heat exchanger could be omitted.

The flushing water behind the catchpot was raised to 100 kg.

August 18, 1941. No change.

TEST PERIOD 3

August 19 to 22, 1941, the operations were changed to over 50% heavy oil excess. The H.O.L.D. return was set at 24 - 25% of the vehicle oil. A correct composition of the centrifuge thinning oil was determined to be 85% middle oil + 15% heavy oil when operating in this way, and this proportion was retained.

August 23, 1941. Trouble: The vehicle oil pumps which feed oil necessary for the preparation of the thin paste were stopped for about 1 hour. This caused the 52% paste to pass during that time through the heat exchangers. There was as a result a rapid drop in the K-value of the heat exchangers from 300 to about 90 with a simultaneous increase in the pressure difference of the heat exchangers equal to 5 - 6 atm. (for details see report on exchanger operations). When the disturbance was overcome all interrupted operations were resumed in a normal way, and the K value and the pressure difference reached their former values.

August 24 to 26, 1941. The hot circuit was interrupted with 26% solids in H.O.L.D. The solid content was lowered by lowering the upper outlet of the hot catchpot to 408° and in addition injecting heavy oil into the neutralization tube between converter II and the H.O.L.D. (up to 250 kg./h). The hot circuit operations were improved with a lower H.O.L.D. solid concentration.

August 25, 1941. No change.

August 26-27, 1941, the hot circuit increased to 1,200 m/h. The heavy oil addition in front of the hot catchpot was replaced with a thinning oil consisting of 100% middle oil.

August 28, 1941. A lower temperature in the preheater and converter because of the drop in heating value of the fuel gas.

August 29, 1941. The average converter temperature again raised to 478°.

August 30, 1941. The H.O.L.D. was thinned with pure middle oil and light asphalt depositions were observed in the screen in front of the centrifuge and occasionally also during centrifuging proper. In spite of repeated flushing there were breaks in centrifuge operations. Operations were again changed to a thinning oil consisting of 85% middle oil and 15% heavy oil. The solid content in the H.O.L.D. (19 - 20%) again permitted the injection of the heavy oil into the hot catchpot. The upper outlet of the hot catchpot was kept at 408°.

September 1, 1941. Trouble: The last units of converter 2 became too hot at 540°; the converter conditions could be brought back by the addition of cold oil in the converter inlet.

September 2, 1941. Converter operations: Converter 1, average temperature 480°, Converter 2, average temperature 475°.

TEST PERIOD 4

September 3, 1941. A change to 1.5% sulfur in the Bayer mass.

September 4 to 7, 1941. No changes. The change in the catalyst has caused no directly observable changes in the converter operations.

September 8-9, 1941. From July 15 to September 7 the thin paste contained 40 - 42% of solids and was successfully heated with gas in the heat exchanger through the temperature interval of 250 to 410° with a rather constant K value of 300 and over through the whole heat exchanger. An attempt has been made now to further increase the concentration of solids in the thin paste. In this way the limiting solid concentration was found at which heat exchange remained possible with a permissible heat transfer value. For this reason starting with September 8 the vehicle oil addition in the thin paste pass was step-wise reduced.

September 10, 1941. Changing over to unsulfurized Bayer mass. Trouble: A short time rise of temperature in the converter 2 to 570°. The converter was sprayed with cold vehicle oil.

TEST PERIOD 5

Operations with Heinitz coal, with the unsulfurized Bayer mass as catalyst II.

September 11, 1941. Changing over to the Heinitz coal. Trouble: Low heat value of the heating gases caused a drop in temperatures. A brief interruption in the hot circuit.

September 12, 1941. Trouble: A rise of the stall pressure difference, presumably as a result of the plugging up of the pipe line between the water catchpot and the washer through formation of propane hydrate caused by the lower outside temperature. Changed over to vehicle oil.

September 13, 1941. Improvement in the heating of the outlet pipe line.

September 14-15, 1941. Change of the coal paste to former concentration, with a return of the stall pressure difference to 18 - 20 atm.

The solids in the H.O.L.D. were reduced by injecting 100 kg. of the catchpot heavy oil into the neutralization tube and the corresponding selection of centrifuge thinner.

September 16-17, 1941. The solids in the thin paste were slowly raised since September 8 to a total of 47.5%. There was a rapid lowering of the K value to 120-130 and less.

Beginning with September 17 the thin paste was again set at 42% solids. There resulted a marked improvement of the heat transfer to a K value differing but little from the normal.

September 18, 1941. No change.

September 19 to 21, 1941. Conclusion of the experiments, change to vehicle oil, reduction of temperature and pressure, change to centrifuge oil, catchpot heavy oil, and finally middle oil. The H.O.L.D. of the converters, etc. were normal at 250 atm.

4. RESULTS OF TESTS

A. Product Balance and Yields. (cf. table, taken from Reel 181, page 215)

Test Period 1: The throughput of pure coal was 0.45 te/h (of which 43% were heated in heat exchangers). A pure coal utilization of 97% with the production of 0.25 gasoline + middle oil. The actual oil production consisted of 21.8% gasoline, 74% middle oil and 4.2% heavy oil. Gasification referred to gasoline + middle oil + gasification was 23.4%. The H.O.L.D. 14.6% asphalt.

The catchpot heavy oil was used for centrifuge thinning oil. The amount of gas left amounted to 3.4 cub. m/kg. of pure coal, 1.7 cub. m/kg. of pure coal of cold gas was consumed.

Test Period 2: The centrifuge thinning oil consisted of 50% middle oil and 50% heavy oil and the actual oil production was composed of 23.5% gasoline, 41.7% middle oil, 34.8% heavy oil. The pure coal throughput reached 0.61 and 59% of it was heated in heat exchangers. The maximum converter temperature was 481°. The coal utilization was 96.6% and 20.5% gasification (referred to practical oil production plus gasification) the actual oil throughput was 0.38, the gasoline and middle oil throughput 0.25 te/h. The H.O.L.D. contained 15.6% asphalt. The gas used amounted to 2.5 cub. m/kg. of pure coal intake gas and 1.5 cub. m/kg. of pure coal of cold gas.

Test Period 3: The centrifuge thinning oil in this method of procedure consisted of 85% to 92.5% of middle oil and 15% to 7.5% heavy oil. At 481° the pure coal throughput was 0.65 te/h (59% through heat exchanger) and 96.2% utilization with a practical oil production of 0.41 te/h. The practical oil production consisted of 23.5% gasoline, 22.2% middle oil and 54.3% heavy oil, which corresponded to gasoline + middle oil production of 0.19 te/h. The gasification amounted to 19%, referred to practical oil production + gasification. The H.O.L.D. contained 17.1% asphalt. 2.1 cub. m. inlet gas/kg. of pure coal, and 1.9 cub m. cold gas/kg. of pure coal have been used.

Test Period 4: Operating conditions during this test were essentially the same as in test period 3. The pure coal throughput was only 4.6% less than in the test period 3, amounting to 0.62 te/h. The gasification with sulfurized Bayer mass was somewhat better compared to the runs with unsulfurized Bayer mass (18% against 19%) with somewhat lower asphalt (16.5 against 17.1%) and somewhat more gasoline and middle oil in the practical oil production (22.2% gasoline, 27.4% middle oil, 50.4% heavy oil). However these small improvements were close to the limit of errors of the balance computations, because the test period 4 lasted only 7 days.

Test Period 5: This test, lasting 8 days, is to be compared with the test period 2. The pure coal throughput was practically the same (0.60) and the temperature the same (481°). The utilization of the Heinitz coal (95.5) was somewhat poorer than of the mixed coal (96.6) with however practically the same production and cracking. The lower gasification (18.6%) appears to be somewhat uncertain considering the short test period. The asphalt value of the Heinitz coal is distinctly poorer (18.2% against 15.6% in the HOLD).

The eventual explanation will be found in the somewhat higher oxygen content of the Heinitz coal and its somewhat higher ash proportion, as one may see by comparing analyses of coal (table 1).

GENERAL CONSIDERATIONS

The results of the semi-industrial scale tests confirm in general the results obtained with 10 li. converters. The utilization and gasification were somewhat better on the semi-industrial scale.

The somewhat low water formation, 6 - 6.5% during the test periods 4 and 5, was probably the result of errors of measurements.

The higher amounts of asphalt in the heavy oil process caused no operational difficulties in the kilns. In spite of the higher asphalts the kiln yields (83%) in the test periods 3, 4 and 5, when compared to 1 and 2, are to be explained by the higher thru-put (degree of filling) and by the breaking-in of the converters.

The principal chemical results of the process for heavy oil production may be formulated as follows:

The change of the gasoline and middle oil process to the heavy oil or fuel oil process was brought about by the introduction of the mixture of middle oil and heavy oil or of pure middle oil as the thinning oil in centrifuging, instead of pure heavy oil as used in the process for gasoline + middle oil, which resulted in an increase in the thru-put. The total amount of gas used in the heavy oil process was about 25% lower than in the gasoline + middle oil process (3.5 - 3.9 cub. m/kg of pure coal against 5.0 cub. m/kg of pure coal).

Curve 1 shows the distribution of practical oil production into gasoline, middle oil and heavy oil in the thru-puts and temperatures closed in large scale tests with mixed coal (Buethen and Heinitz) and the effect of thinning oil used. The production of gasoline in the total oil has changed very little, but the heavy oil content increased with increasing amount of middle oil in the thinning oil, and the middle oil content is also increased.

The increase in thru-put within the same temperature limits is determined by the simultaneously increasing asphalt level, which remains yet to be overcome in kiln operations. 17 - 18% asphalt in HOLD corresponds to 11 - 12% asphalt in the oil of the centrifuge residue and could be readily worked in the kilns, as mentioned previously.

With about 35% heavy oil content in the actual oil production the pure coal thru-put of 0.61 could be maintained. During operation for 50%

heavy oil in the total oil. [illegible] raised to [illegible] with the production [illegible] 92.5% [illegible] in the [illegible] by the use of pure middle oil as a thinning oil [illegible] a further increase in the proportion of heavy oil in the total oil.

[illegible] gasoline middle oil [illegible] heavy oil [illegible] the same [illegible]

[illegible] additional [illegible] heavy oil.

[illegible] vaporization space [illegible] gasoline and middle oil [illegible] heavy oil [illegible] vaporization space, the gasoline [illegible] middle oil process.

[illegible]

[illegible] of centrifuge residue [illegible]

[illegible] operated [illegible] pressure of [illegible] Upon [illegible] pressure [illegible] and [illegible] continually.

[illegible] of Coking

[illegible]

Brucol. Schiebler [illegible]

ANALYSIS OF BALANCE

Test Period:	1	2	3	4	5
Coal	Bouthen + Heinitz Coal 1:1				Heinitz coal
% Ash / P.C.	4.9				5.3
% C / P.C.	81.96				81.61
Pretreatment of coal & addition of catalyst	Coal crushed, ground, moistened w. 1.2% $FeSO_4 \cdot 7 H_2O$, dried, mixed with 1.5% Bayer mass and 0.3% Sulfigran.			Like 1-3, but with sulfurized Bayer mass.	Like 1-3
Thinning oil for fuges	100% H.O.	50% H.O. 50% M.O.	7.5% H.O. 92.5% M.O.	15% H.O. 85% M.O.	27% H.O. 75% M.O.
HOLD: Thinning Oil	1:0.52	1:0.41	1:0.44	1:0.45	1:0.37
Compos. of vehicle oil	Fuge Oil + Preheater Oil	Fuge Oil + Preheater Oil + HOLD Ret.	Fuge Oil + Preheater Oil + HOLD Ret.	Fuge Oil Preheater Oil + HOLD Ret.	Fuge Oil + Preheater Oil + HOLD Ret.
% Solids	8.2	13.7	11.6	12.8	13.4
% Asphalt in Oil	9.2	9.8	12.6	11.0	11.5
% -325° in Oil	2.8	15.4	27.6	28.0	25.6
T Coal + Cat : Return	-	1:0.21	1:0.28	1:0.30	1:0.37
Total Paste: % Total solids	46.0	44.8	45.8	44.3	44.9
% Pure coal	38.4	34.2	36.5	34.2	34.2
Thin Paste: % Tot. solids	41.0	41.6	41.8	40.5	42.5
% Pure coal	33.3	30.7	32.6	30.5	31.1
Operating Cond:					
Temps: Preh. outlet (144)	452°	455°	446°	442°	444°
Conv. I, max.	480°	481°	481°	481°	481°
Conv. II, max.	480°	481°	481°	481°	481°
Catchpot	434°	425°	412°	412°	416°
Press, atm.	700	700	700	700	700

Thru put: Tot. Paste	1.17	1.79	1.79	1.82	1.75
Thin Paste	0.58	1.18	1.18	1.19	1.14
Tot. Pure Coal	0.449	0.612	0.653	0.622	0.595
Pure coal in thin paste	0.193	0.362	0.386	0.363	0.355
Intake gases: m3/kg p.c.	3.35	2.46	2.11	2.18	2.10
Cold gas: m3/kg p.c.	1.72	1.47	1.34	1.42	1.40
Total gas: m3/kg p.c.	5.07	3.93	3.45	3.60	3.50
H_2 partial pressure, at inlet atm	590	587	590	570	550
Exper. Results:					
Rough balance	98.2	97.8	97.9	98.6	97.5
Ash balance	98.3	100.2	99.5	104.3	101
Utilization	97.0	96.6	96.2	96.4	95.5
Oil Prod. at high press	69.5	71.6	71.2	73.0	71.4
% off water / P.C.	9.2	8.5	8.6	6.5	6.0
Practical Oil Prod.	0.264	0.383	0.410	0.410	0.377
Composition of oil produced:					
% gasoline - 200°	21.8	23.5	23.5	22.2	23.7
% m.o. 200-325°	74.0	41.7	22.2	27.4	43.3
% H.O.	4.2	34.8	54.3	50.4	33.0
Product: gasol. + m.o.	0.253	0.250	0.187	0.204	0.248
H.O.	0.011	0.133	0.223	0.206	0.129
Gasific./oil + gasif.	22.6	20.5	19.0	18.0	18.6
Gasific./gas + oil + gasif.	23.4	28.2	34.0	31.0	25.5
% gasified C in P.C.	18.1	16.6	15.3	15.3	15.1
of this % as CO	1.8	2.0	2.2	2.2	1.3
% as CO_2	3.3	3.1	3.4	3.9	5.1
% New HOLD/total coal+cat	106	124	102.5	114.5	111.2
Properties of HOLD: % Solids	19.7	21.9	20.8	21.6	22.3
% Ash in Solids	53.	49.4	51.0	51.9	51.0
In Oil: % Solid asphalt	14.6	15.6	17.1	16.3	16.2
% M.O.	4.5	4.5	10.5	9.9	11.1
% Solids in/fuge res.	38.5	40.0	38.5	39.0	41.1
% Kiln Yield	80	7[illegible]	82.7	83	83

	Fuge Residue	Topping Oil	Topping Residue (Anhydrous)
Oils			
% - 325	25.6	53	1.5
- 350	35.6	-	6.6
% s-asphalt	10.9	-	21.5
Krä-Sar	-	-	85°

The balance shows a small formation of solids amounting to 11% of the organic solids in the fuge residue. This formation is to be attributed to the long stay of the material in the kiln (in excess of 1 hour). The granulation arrangement favors the occasional production of foaming and water-containing residues. The new experiment in Scholven permitted improving the process.

B. ANALYSIS OF INTERMEDIATE AND FINAL PRODUCTS.

Coals:

The analyses of coals have been discussed in the section on raw materials. Table 4 tells the fineness of grinding. The coal was first crushed to 69% on Din 400 (50 mesh). The ground dry coal left 38% on the Din 400 with 30% through Din 10,000 (250 mesh). With this degree of fineness no difficulties were experienced, neither on the high nor on the low pressure sides.

Vehicle Oil or Film Oil:

The vehicle oil was a mixture of fuge oil, kiln heavy oil and HOLD. The film oil used in the preparation of the thin paste was the centrifuge oil alone.

Table 3 contains a results of analyses obtained in the individual test periods. During test period 1 (gasoline + middle oil without return of HOLD) the vehicle oil contained 8% of solids, its middle oil content was 2.5%, s-asphalt content was 9%. The vehicle oil in the processes for excess or heavy oil had 11.6 - 14.4% of solids, 10-13% of asphalt. Depending on the fuge thinning oil, there was 16.7 - 29.8% middle oil in the oils.

The film oils contained 8.3 - 11.5% solids, 10.9 - 11.7% asphalt and 16.9 - 35% of middle oil. In the gasoline + heavy oil process oils were somewhat richer in hydrogen than in the gasoline + middle oil process.

Coal Paste:

Table 5 gives viscosities of the thick and the thin paste. The determinations were made in an Engler viscosimeter with 6 mm orifice at 90°

and correlated with the solids contents of the paste. Such correlation is not strictly accurate in view of the fact that the quality of oil itself has an appreciable effect upon viscosity, however the table shows a great increase in the flow time when the solid concentrations of 46 - 47% were exceeded, which can be made to agree well with experience in the heat exchangers (see report on heat exchange).

HOLD (Table 6):

The solids in the HOLD varied between 20 and 22%. The greater the fuge oil excess used the higher also was the concentration of asphalt. The HOLD from the Heinitz coal (5) had distinctly higher asphalt content than that produced from the mixed coal. The middle oil content in the method for production of an excess or heavy oil had an 11% maximum and was therefore higher than in the process for gasoline + middle oil.

Table 5 contains the HOLD viscosities which were measured at 100° with the Leuna viscosimeter. One may observe a distinct effect of the higher solids and asphalt contents upon viscosity. A somewhat higher middle oil content (test periods 3-5) results in a slight lowering of viscosity.

Screen analysis of the HOLD solids (table 4) gave 0.1% residue on Din 900 (75 mesh) and 97.5 through Din 16,000 (325 mesh).

Filtration experiments with the HOLD in stone pressure-filters (5 atm. 160°) after dilution of the HOLD with 2 parts of coal phase middle oil gave the following filtration productions, in kg/sq. m/h:

HOLD	Filtered Mixture	Original Feed (Calculated)
1 Gasoline + middle oil	49	16
3 Gasoline + excess of heavy oil	77	26

These filtration results indicate the impossibility of industrial filtration as previous experience had already shown. The HOLD obtained in the method for heavy oil is more readily filterable than the gasoline + middle oil HOLD.

Fuge Oil, Fuge Residue (Tables 7 and 8):

The solids contents of the fuge oil varied between 8.8 and 12.6%, with 29 - 35% ash. The solids content of the fuge residue were satisfactory at 37.8 - 40%.

The ash enrichment in the solids amounted to 53.8 - 55.4%.

The fuge oils contained somewhat more asphalt than the centrifuge residues left behind. The middle oil content of the corresponding oils was the same. The solids and the ash contents of oils of the different test period were practically the same. Nor was there any marked selective action of the fuging with regard to chemical composition of the individual components.

A comparison of the screen analysis of the solids from the fuge oil and from the fuge oil residue (table 4) indicates somewhat coarser solids in the fuge residue, as was to be expected.

Kiln Oils:

Tables 9, 10, 11 and 12, reproduce the analyses of the kiln oil. About 50% of the kiln oil was obtained as kiln heavy oil, about 20% as the light oil. The heavy oil was used in paste, the light oil in the catchpot distillation.

The middle oil content of the fuge residue can be distinctly recognized in the kiln oils of the heavy oil production. The middle oil content of the kiln heavy oil (table 9) was approximately equal to the middle oil content of the oil entering the kiln.

The gasoline content of the light kiln oil (table 10) increased with increasing middle oil return.

Kiln Residue:

The oil content of the kiln residue was at least 0.5%.

Table 13 gives the analysis and heat value of the kiln residue. The ignition residue of the kiln residue was about 42-46% and the heat value varied between 4,500 and 4,950 heat units.

Catchpot:

For analysis see Table 14. Comparison of the boiling point curves and the column distillation of the catchpot product from the different test periods show that the heavy oil process results in hardly any increase in the amount of gasoline in comparison with the gasoline + middle oil process, but there is an increase of middle oil content which corresponds to the middle oil added into the circuit (40.5 to 45% against 30%). As a result of the milder hydrogenation, the phenol content in the heavy oil process is apparently higher.

A comparison of the ultimate analyses shows higher oxygen and nitrogen contents in the heavy oil process.

The gasoline, middle oil and heavy oil from the catchpot were analyzed after a laboratory column distillation as well as after industrial distillation. In the laboratory decomposition, no kiln light oil was added, as is usually done in the industrial installations. In balance computations, the weight analyses of the catchpot (and of the kiln oils) were used.

Liquid Phase Gasoline:

The crude gasoline and gasoline dephenolated with sodium hydroxide have been studied (tables 15-20). The gasolines used were obtained from the laboratory catchpot distillation (tables 15 and 17), the gasoline from the industrial distillation (16-18) and the gasoline of the industrial distillation to which the corresponding low boiling fraction (gasbenzin) (35) have been added (tables 19 and 20).

There are differences in the cuts of the laboratory and industrial gasolines. The laboratory gasoline is cut higher than the industrial (end point of the former up to 220°). This causes a difference in the specific gravity, the boiling point curve, in the phenol content, etc.

There is moreover a difference between the gasoline and middle oil process and the process for heavy oil production in that the sump gasoline has a much higher content of aromatics in the gasolines of the latter process, than in the former (tables 15, 16, 17, 18). There is a corresponding rise in the octane number from 69 to 71 - 73.8.

Catchpot Middle Oils (Tables 21 and 22):

In this case again, both laboratory and technical products, have been studied. The middle oils of the heavy oil production method are somewhat higher in oxygen and in nitrogen than from the gasoline - middle oil process.

Catchpot Heavy Oils:

Tables 23, 24 and 25 show the results of tests undertaken in the laboratory catchpot heavy oil as well as with the technical oil. The catchpot heavy oils of the heavy oil process have a higher specific gravity (1.052 as against 1.042) than the catchpot heavy oil of the gasoline - middle oil process; the boiling point curves are somewhat changed, there is somewhat more phenols, correspondingly higher oxygen value and somewhat higher nitrogen value. Tests on the properties of fuel oils indicate a somewhat higher pour point, asphalt value, coke test and viscosity for heavy oil production method. The catchpot heavy oils produced by any method fail to meet the Navy fuel oil requirements with respect to viscosity and pour point.

Fuel Oils (Tables 25 and 26):

For the production of fuel oils which would meet the requirements of the Navy from catchpot oils, mixtures were prepared with catchpot middle oil in such a way that the mixed oils had a viscosity of 35° E/20° with a satisfactory specific gravity.

The catchpot heavy oil of the gasoline - middle oil method required a mixing proportion of about 74 parts heavy oil and 26 parts middle oil. The resultant fuel oil has then a specific gravity of 1.044 - 1.046/20°, pour test -3 to -5°, coke test 0.55 - 0.58, and a viscosity of 35.5° E/20° or 3.8° E/50°.

All oils met the settling specifications.

At the end of the tests, fuel oils were prepared and sent to the chemical-physical testing laboratory of Kiel-Dietrichsdorf, to the Deschimag Bremen and to the Navy Yard at Kiel.

Table 28 contains a description and analytical results of these tests and their comparison with the specifications of the Navy.

It shows that mixtures of catchpot heavy oils with middle oils satisfy the Navy specifications.

Phenols in the Oils and Off-Water (Table 29):

The catchpot of the heavy oil method had 0.9 - 1.1% carbolic acid and 1.7% cresols. The phenols of the sump gasoline consisted of 65% carbolic acid, 54.7% pure; the phenols of the sump middle oil contained 44.7% carbolic acid and 21% cresols. The determination of phenol in the off-water varied so strongly that no conclusions could be drawn from them.

1 tank car of the off-water was sent to the experimental works of I. G. Leverkusen.

Flushing Oil (Table 30):

A coal sump middle oil of specific gravity 0.950 at 20° was available for use as a flushing oil; about 92% of it distilled below 350°C.

Hydrocarbon Gasification:

Table 31 gives the distribution of the hydrocarbon gasification in per cent by weight of hydrocarbons as well as in per cent by weights of carbon, also the amounts of unsaturated and of iso compounds.

A comparison of the methods for heavy oil and for gasoline - middle oil shows an increase in the amount of methane in the process for heavy oil. The content of unsaturated and of iso butane in the total butane is about the same as in aromatics.

Solubilities (Table 32):

With respect to solubilities of H_2, CO, N_2 and CH_4 in flushing oils and the catchpot products obtained by different methods, there were practically no differences exceeding the limits of accuracy of measurement and the analytical errors.

The differences of solubility in HOLD in the gasoline - middle oil process and the heavy oil process are considerable, and the products of the heavy oil process have a lower solubility. In the heavy oil process the total heavy oil is considerably higher (as a result of return) than in the gasoline - middle oil process. In spite of that the amount of HOLD was not increased to the same extent. This leads to the conclusion that no saturation has been reached or that in the gasoline - middle oil process some gas is entrained.

CO_2, H_2S, NH_3:

Table 33 gives the distribution of the CO_2, H_2S and NH_3 in gases and off-water.

Organic Sulfur:

The results of determinations of organic sulfur in the off-gases are given in table 34. The strong discrepancies of the figures obtained do not permit recognizing characteristic properties of the individual production methods. The organic sulfur content is less in the HOLD off gas.

Hydrogen Consumption:

The hydrogen consumption found from material balance is given in table 38. The lower H_2 consumption in the fuel oil process against the gasoline - middle oil process is quite striking. 66 - 71% of H_2 is used up chemically.

C. HEAT EXCHANGE

The results of paste heat exchange are given in the adjoining report of Schappert "Das Ergebnis des Grossversuches KS04 hinsichtlich der Breiregeneration" of October 8, 1941. The information on trouble in the heat exchanging and the dismantling of the heat exchangers is shown in chapters B on "The Course of the Test" and D "The Dismantling Results" and in "The Preliminary Dismantling Report".

D. RESULTS OF DISMANTLING

The accompanying Preliminary Reports on the Results of Dismantling of the High Pressure Stalls used in fuel oil tests by Eichle and Hamacher (T-54 and T-55) October 13, 1941, give details of the results found upon dismantling.

The report does not contain the results on dismantling of heat exchanger III (the pictures however are included), of the converter 1, nor the analytical results of the investigation of the incrustation during heating.

Heat Exchanger III:

Measurements of resistance to flow in the forward and return passes indicated no changes from original values. The subsequent dismantling showed that the forward pass was slightly coated with soft oily masses, while the return pass was completely clean.

Converter I:

The HOLD of this converter contained about 20 kg of abraded pieces of different size (up to about 4 mm in size). A thru-put test made at the converter gave good results. At subsequent openings of the converter the following picture was observed:

The funnel of the converter, as well as the bottom and the middle converter parts, were filled out to a level of 2 m from below with granules of the above kind as have been found in the HOLD. Above that there was a soft layer (50 cm) in which the same granules were mixed with somewhat more oily constituents. The upper part of the converter was clean. The total settled solids amount to about 330 kg or 0.025% referred to the dry coal introduced, or to 0.5% of the ash.

In contradiction to the caviar formation with brown coal, these grains were not spherical. Moreover sections of these granules showed no well developed layer formation. They have a thin outer layer which forms about 1/10 of the grain and which evidently holds the grains together. The analysis of the grains was interesting:

Origin of the Sample	Bottom of the Converter	Middle of the Converter (the soft part)	Comparison with Coal Ash (mixed coal)
% Benzol Insolubles	37.0	32.6	
% Ash in Solids	96.1	90.3	
% Asphalt in Oil	2.8	3.4	
Analysis of Ash			
% SiO_2	16.72		32.65
% Fe_2O_3	21.80		10.36
% Al_2O_3	30.80		28.11
% CaO	4.23		9.47
% MgO	0.49		4.94
% Na_2O	9.43		1.71
% Cl	Trace		-
% SO_4	13.70		9.59
In total prod.			
% Sulfide S	4.93		
% Total S	5.72		
% Cl[1]	6.55		

[1] In extract with dilute HNO_3

The granules consisted therefore practically entirely of inorganic constituents. The principal constituents of the residue after ignition were SiO_2, Fe_2O_3, Na_2O. Sulfide sulfur and chlorine were present in fairly large amounts.

The SiO_2, Fe_2O_3 and Al_2O_3 probably were produced from the inorganic constituents of the coal. The strong enrichment with alkalis and chlorine lead to the assumption that the above coal constituents are essentially held together by the alkali chloride and possibly also by iron sulfide (possibly from catalysts). With 0.014% of chlorine in the coal about 10% of the chlorine referred to coal is present in the deposits.

At the hydrogenation works Pölitz which were operating with practically the same coal, similar deposits have been observed (Pölitz works with unwashed coal containing 7% ash). Such phenomena have not as yet been observed with the Ruhr coal in large scale operations at 700 atm. using iron catalysts, which leads one to assume that this is a peculiar property of the Upper Silesian coals. There may possibly be a close connection with the high alkalinity of these coals.

The deposits occupy reaction space, and may possibly require cleaning of the converters.

Incrustation in Heating:

In addition, examinations were made of a number of crusts and deposits found upon dismantling. In spite of the fact that no complete analyses are available we may recognize the following:

The greater part of depositions is formed by iron sulfide. In addition, some large chlorine and alkali amounts have been ascertained. This leads one to assume a certain relationship between deposition during the preheating and the formation of "breeze" ("Griess") in the first converter. Whether the chlorides are formed during preheating from the organically combined chlorine of the coal, or whether this only takes place in the converter and the chlorides are returned with the vehicle oil into the system and are deposited there, can as yet not be decided on the strength of the available material.

Sternberg/MC/jw

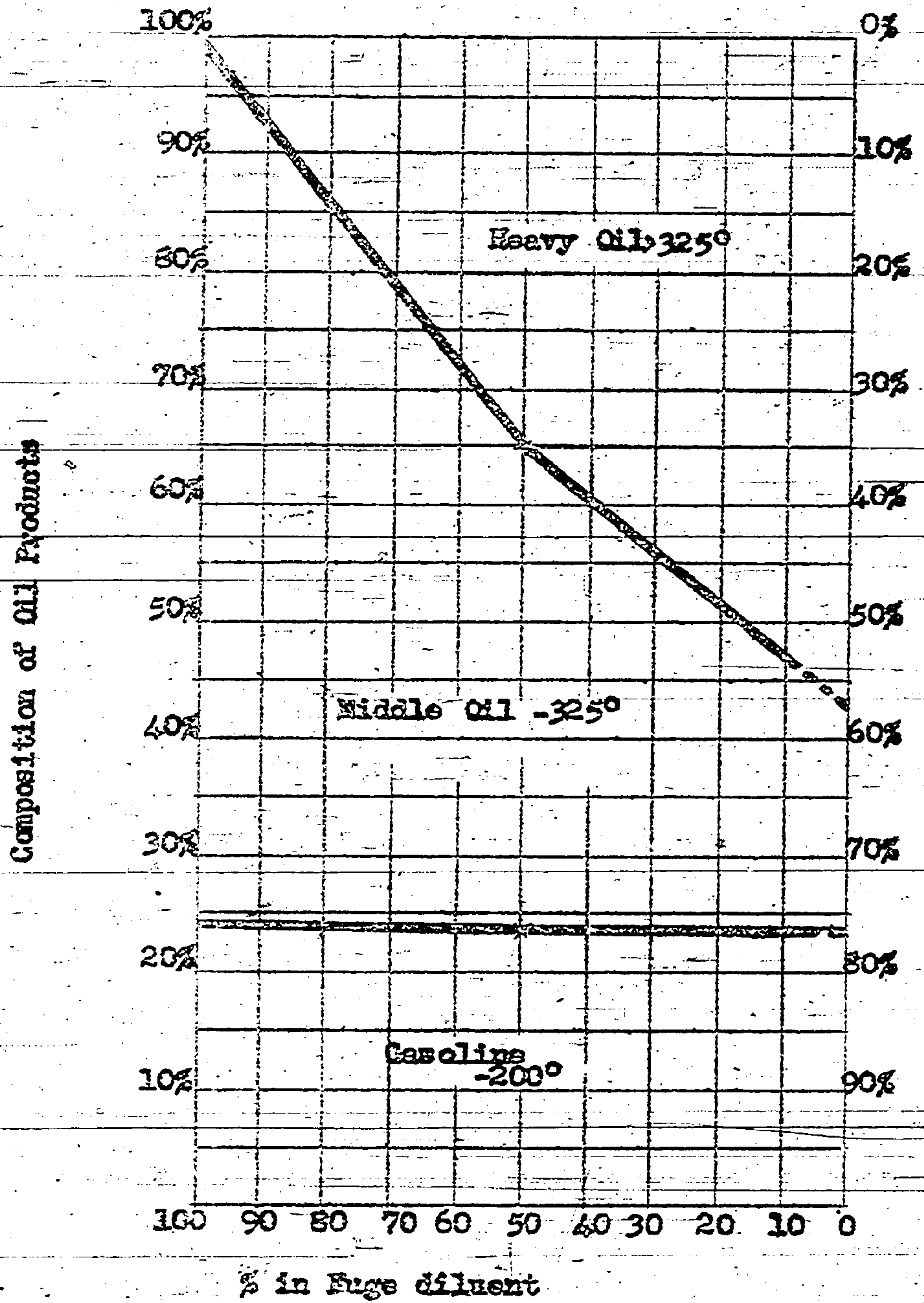
7-19-46

Analysis of Incrustation in Preheater

Location of Crust	Thickness	% Benzol Insoluble	% Ash i. Solid	% s-Asphalt i. Oil	In Benzol - Insolubles:						
					%SiO_2	%Fe	%Ca	%Na	%S	%CO_2	%Cl
Last Hairpin, Pass II,	4 mm	86.5	96.5	-	1.3	34.7	2.2		20.4		8.3
Last ascend. tube Pass II, before entering Pass III	4 mm	72.5	98.1	-	3.4	19.0	2.1		11.5	2.2	
Connecting Elbow Pass II to pass III (soft deposit)	-	67.5	55.0	7.4	7.6	17.9	1.5		9.9		8.0
Connecting Tube Heat Exch. I, Pass III	1.7 mm	75.0	98.3	-	7.2	33.3	4.6		11.5		1.5
Last Descend. tube, pass III	3-4 mm	100	98.0	-	1.7	26.5	0.8		10.4	0.4	
Connection Preheater to Convert. I. (hard const. of crust)	-	82.3	93.9	-		40.0		7.7	17.9		
Connection Preheater to converter I (soft part of crust)	-	78.0	98.0	5.4							

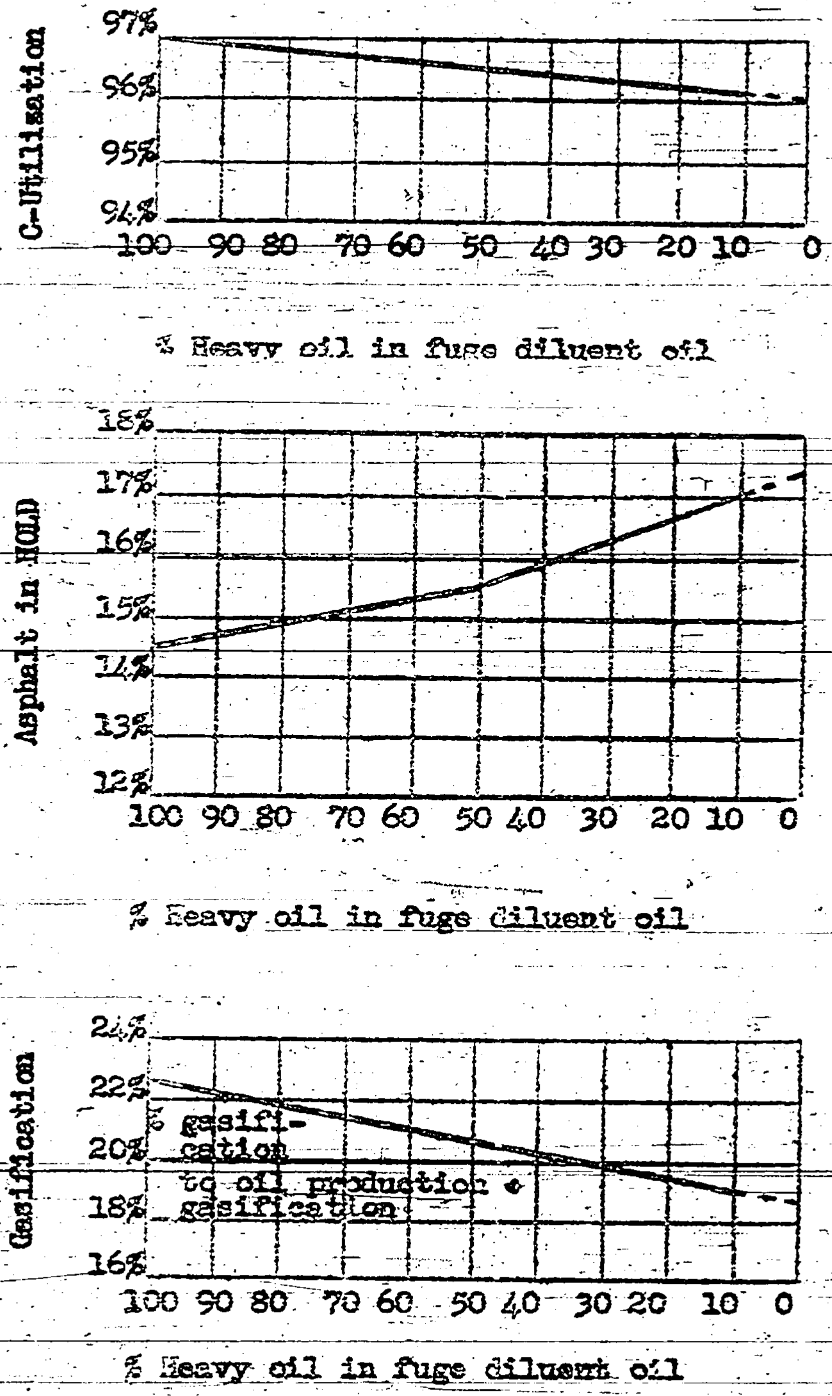
CURVE 1

EFFECT OF COMPOSITION OF FUGE OIL UPON THE DISTRIBUTION OF FRACTIONS IN TOTAL OIL PRODUCED



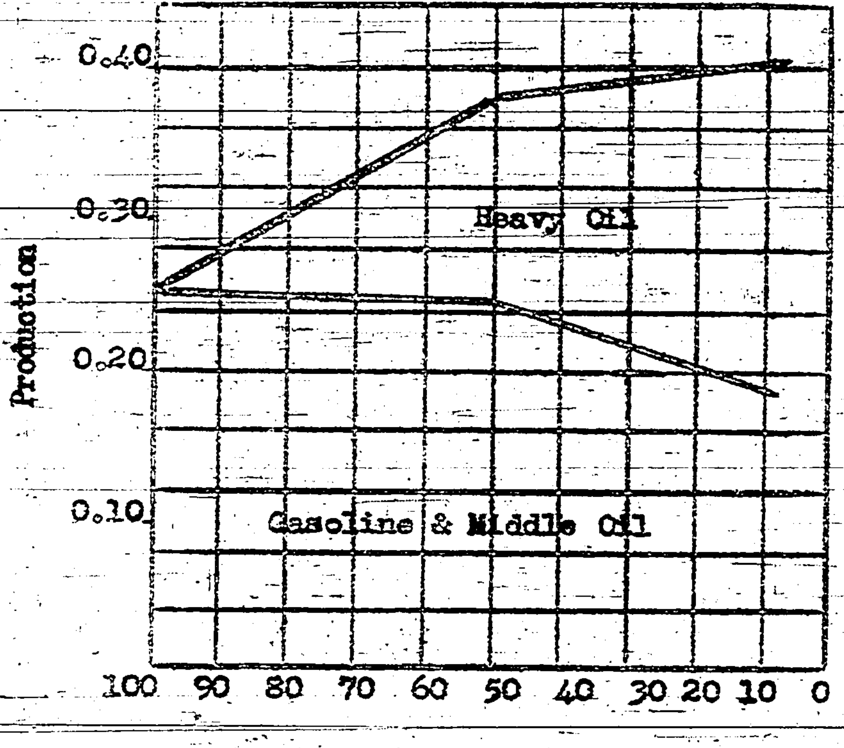
CURVE 2

EFFECT OF COMPOSITION OF FUGE DILUENT OIL UPON COAL UTILIZATION, ASPHALT CONTENTS OF HOLD AND GASIFICATION



CURVE 3

OIL PRODUCTION WITH FUGE DILUENT OIL OF DIFFERENT COMPOSITION



% Heavy oil in fuge diluent oil

Table 2
Catalysts

Catalyst	$FeSO_4 \cdot 7 H_2O$	Bayer mass dry	Bayer mass Sulfurized	Sulfigran
% Fe	19.69			
% SO_4	34.77			
% Na				56.03
% S			19.2	38.52
% H_2O	45.24	2.01		
% SiO_2	0.03	8.57		0.16
% Fe_2O_3		40.38		0.47
% Al_2O_3		22.71		0.10
% CaO	0.004	1.68		0.44
% MgO	0.59	0.70		0.19
% Na_2O		7.84		
% SO_3		0.18		
% CO_2		2.74		0.59
% P_2O_5		0.50		
% TiO_2	0.35	5.80		
% Cl	Trace	Trace		trace

/pkl

TABLE I

Experimental coals

* Data recalculated from Cal. to BTU.

COAL	BEUTHEN-MINE	HEINITZ-MINE	PROPORTION IN COAL MIXTURE AS DELIVERED
SIZE	Proportion of Nut and Fines as Delivered	Nut Coal 8-30 mm	Nut Coal and Fines
%Water	7,94	8,88	7,65
%Ash in dry coal	4,78	5,29	4,88
Coal on ash less base: %C	81,69	81,61	81,96
H	5,04	5,02	5,04
O	11,24	11,73	11,03
N	1,62	1,18	1,67
S volatile	0,37	0,42	0,29
S fixed	0,65	0,64	0,66
C_1	0,04	0,045	0,014
H disp.	3,99	4,01	3,97
Volatiles	37,17	37,55	37,59
Alkalinity g H_2SO_4/kg dry coal	20,0	19,35	17,7
carbonization analysis of pure coal:			
%Coke	78,4	76,69	77,8
%Tar	10,3	10,21	10,1
%Water	5,1	5,45	5,2
%Gas + Loss	6,2	7,65	6,9
Content of pure coal	0,30	0,30	0,30
Dry Coal:			
* Heat of combustion BTU	13,480	13,230	13,420
Heating Value	13,000	12,730	12,980
Composition of Ash:			
$\%SiO_2$	33,02	31,63	32,65
Fe_2O_3	9,96	11,88	10,36
Al_2O_3	28,31	26,59	28,11
CaO	9,76	9,88	9,47
MgO	5,10	5,07	4,94
K_2O			
Na_2O	} 1,75	} 1,83	} 1,71
SO_3	8,73	12,09	10,25
P_2O_5	0,98	0,73	0,89
Cl	–	–	–
TiO_2	1,27	1,20	1,27
M.P. °C	1253°	1237°	1245°

Table 3
Pasting Oil and Film Oil (Preparation of Thin Paste)

A	B	C	D	E	F	G	H	I	J	K
Product	Pasting Oil Gelsenberg	Pasting Oil	Pasting Oil	Film Oil	Pasting Oil	Film Oil	Pasting Oil	Film Oil	Pasting Oil	Film Oil
Test Period		1	2	2	3	3	4	4	5	5
Sp. Gr.		1,0[illegible]7/100°	1,116/100°	1,08[illegible]/100°	1,102/100°	1,076/100°	1,0[illegible]9/100°	1,07[illegible]/100°	1,104/100°	1,094/100°
% H₂O	2.1	0.3	0.3	0	0.1	0	0	0	0	0
% Solids	10.5	[illegible]	14.4	11.4	11.0	8.8	14.7	9.4	13.3	11.5
% Ash in solids	58.3	57.7	44.7	42.1	43.0	37.6	[illegible]	36.4	42.6	42.5
Oil Sp. Gr.	1,063/ 70°	1,063/100°	1,071/100°	1,067/100°	1,062/100°	1,044/100°	1,06/100°	1,04[illegible]/100°	1,068/100°	1,047/100°
[illegible] 325°	6.5	2.9	16.7	16.9	27.6	35.0	29.8	31.5	22.2	30.2
[illegible]	7.0	12.9	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	11.3	11.9
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	28
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	60.0	67.3	[illegible]	[illegible].6	[illegible]	63.4	[illegible]	[illegible]	[illegible]	64.8
Pasting Fraction										
[illegible]	[illegible]	1,043/40°	1,081/46°	1,091/40°	0,989/40°	0,978/40°	0,98[illegible]/40°	0,978/40°	0,997/40°	0,980/40°
[illegible]	[illegible]	1,038/60°	1,061/60°	1,047/60°	1,036/60°	1,048/60°	1,05[illegible]/60°	1,03[illegible]/60°	[illegible]/60°	1,016/60°
375-32[illegible]°	1,061/7[illegible]°	1,057/90°	1,064/90°	1,063/90°	1,066/90°	1,063/90°	1,075/90°	1,06[illegible]/90°	1,0[illegible]1/90°	1,043/90°
Elementary Analysis of Oil										
% C	89.88	90.56	89.93	89.91	89.66	89.60	90.18	89.71	90.08	89.98
% H	7.46	7.39	7.37	7.51	7.41	7.67	7.46	7.62	7.58	7.68
% O	[illegible].74	1.33	1.90	1.65	2.09	1.86	1.36	1.67	1.50	1.70
% N	0.83	0.69	0.90	0.89	0.93	0.99	0.45	0.92	0.97	0.97
% S	0.09	0.03	0.04	0.01	0.05	0.04	0.04	0.04	0.06	0.04
% Cl	<0.01		<0.01		<0.01	<0.01	<0.01	<0.01	<0.01	0.01
H/100 C	8.3	8.16	8.35	8.36	8.4[illegible]	8.49	8.26	8.53	8.10	8.57
Net Value, Cal.	9136	9187	9004	9226	9114	9229	9103	9123	9049	9114
Heat of Comb.	9541	9536	9487	9532	9524	9544	9536	9536	9448	9529
From Solids: Dry Sample (Ash) Ash free extraction										
% Ash	[illegible].25	39.70	43.77	40.06	43.56	37.30	44.25	34.00	43.69	40.92
% C	[illegible].58	54.75	53.40	53.90	50.10	57.75	50.05	60.83	51.62	52.70
% H	[illegible].47	2.20	2.17	2.27	2.17	2.46	2.13	2.45	1.95	2.25
% O										
% N	[illegible].42	0.53	0.77	0.73	0.53	0.56	0.40	0.62	0.49	0.49
% S Vol.	[illegible].38	2.34	2.17	2.49	2.37	2.06	2.96	2.45	2.50	2.93
% S [illegible]	[illegible].90	3.42	3.68	3.42	3.96	2.98	4.60	3.56	4.38	4.25
% Cl	[illegible].19	[illegible]	0.18	0.15	0.11	0.19	0.17	0.12	0.21	0.18
H/100 C	[illegible]	4.0[illegible]	4.07	4.21	4.[illegible]	4.26	4.26	4.03	3.78	4.27

Table 3 (continued)

Pasting Oil and Film Oil (Preparation of Thin Paste)

A	B	C	D	E	F	G	H	I	J	K
Product	Pasting Oil Gelsenberg	Pasting Oil	Pasting Oil	Film Oil	Pasting Oil	Film Oil	Pasting Oil	Film Oil	Pasting Oil	Film Oil
Test Period		1	2	2	3	3	4	4	5	5
Heat Value	3810	4960	4879	4965	4700	5605	4628	5590	4753	4883
Heat Comb.	3909	5072	5009	5089	4818	5748	4755	5735	4858	5004
Ash Analyses:										
% SiO_2		27.39	26.86	26.80	27.39	26.45	24.44	25.13	23.78	23.81
Fe_2O_3		19.67	20.46	19.51	20.78	20.21	25.07	28.19	22.48	23.25
Al_2O_3		29.72	27.82	30.23	26.13	27.24	21.62	20.11	23.30	25.22
Ca O		5.15	5.10	4.80	5.71	5.18	6.11	6.17	5.82	5.35
Mg O		2.93	3.03	2.98	3.32	3.03	3.23	3.11	3.31	2.96
K_2O		6.37	7.24	7.57	6.51	6.89	6.56	6.40	6.22	6.79
Na_2O										
SO_3		5.16	5.84	4.85	6.21	5.30	8.79	6.57	11.34	9.30
P_2O_5		1.10	0.70	1.15	0.91	1.11	0.78	1.01	0.80	0.82
TiO_2		2.37	2.36	3.01	2.23	3.47	3.32	2.94	2.32	2.49

T-52

TABLE 4

Screen Analysis (Averages)

Products Tested	% Residue on								Thruput
	100	400	900	2500	4900	10000	12000	16000	16000
Crude coal (crushed)	42.7	26.2	10.6	6.8	3.1	2.1	0.8	0.5	7.2
Dried Coal	23.5	14.7	9.7	9.7	5.9	6.0	3.1	1.7	25.7
Solids in Paste	1.9	5.5	7.9	11.3	6.8	6.8	3.3	2.4	54.1
Solids in HOLD	0	Tr.	0.1	0.4	0.7	0.7	0.4	0.4	97.3
Solids in Fuge Oil	0	0	Tr.	0.4	0.2	0.4	0.2	0.9	97.0
Solids in Fuge Residue	0	0	0.1	0.5	0.8	1.1	0.3	0.8	96.4

Remarks: The screens are rated (Din.) in mesh per cm^2. To convert into mesh per linear inch use $2.54 \sqrt{\text{Din.}}$

100 Din mesh = 25.4/lin inch
400 = 51 " "
900 = 76 " "
2500 = 127 " "
4900 = 178 " "
10000 = 254 " "
12000 = 279 " "
16000 = 323 " "

/pkl

TABLE 5

VISCOSITY

COAL PASTE.

Flow Time for 100 ml. at 167°F in Engler-Viscosimeter with 6 mm. orifice

Nature of Paste	Run Number	% Solids in Paste	Flow Time
Thick Paste	4	50.4	66.8
"	5	48.3	64.6
Thin Paste	5	46.6	29.1
"	5	45.8	40.8
"	5	45.6	28.7
"	5	45.4	23.5
"	4	45.2	19.2
"	4	43	20.1
"	4	42.2	13.1
"	5	41.8	18.9
"	5	41.0	16.7

TABLE 5a

Viscosity

H.O.L.D.: Leuna Viscosity Meter at 100°: Centistokes

Operation Period	1) in H.O.L.D. % Solids	in H.O.L.D. % Asphalt in Oil	in H.O.L.D. % Middle Oil in Oil	Viscosity Centistokes
1	19.7	14.6	4.5	900-950
2	21.9	15.6	4.5	900-930
Maximum	26.8	19.1	5.8	2300-3700
3-5	22.0	17.3	11.1	700-900

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1) Average.

T.O.M. Reel No. 130

H.O.H.D. INVESTIGATION

PERIOD	1	2	3	4	5
S. Gr./100°C	1.143	1.175	1.206	1.197	1.192
% Benzol Insol.	19.7	21.9	20.8	21.6	22.3
% Ash solids	53.6	49.4	51.0	51.9	51.0
IN OIL:					
Sp. Gr./100°C	1.096	1.112	1.113	1.112	-
Soluble Asphalt	14.6	15.6	17.1	16.3	18.2
% - 325°C	4.5	4.5	10.5	9.9	11.1
Vacuum distill. at 20 mm Hg					
Fractions: to 225°C: %	14.9	14.5	15.5	13.1	-
Sp.gr./40°C	1.004	1.014	0.995	1.002	-
225-275°C.%	23.3	18.3	15.0	14.2	-
Sp.gr./60°C	1.043	1.052	1.048	1.054	-
275-325°C:%	14.4	14.4	14.4	13.7	-
Sp.gr./90°C	1.061	1.068	1.064	1.066	-
Ultimate Analysis: % C	-	-	-	90.88	-
H	-	-	-	6.87	-
O	-	-	-	1.56	-
N	-	-	-	0.64	-
S	-	-	-	0.04	-
Cl	-	-	-	<0.01	-
% H / 100 C	-	-	-	7.6	-
Heat value BTU	-	-	-	16,300	-
Heat of Comb. BTU	-	-	-	16,970	-
Solids: Solid sample					
% Ash (Res. on Ignition)	52.62	49.67	48.25	52.00	44.85
% C	42.21	46.05	45.79	42.35	44.82
% H	1.73	1.82	1.98	1.73	1.75
% O	-	-	-	-	-
% N	0.57	0.55	0.45	0.39	0.48
% S volatile	2.95	2.31	1.94	3.54	2.43
% S fixed	5.01	4.61	4.55	5.47	4.86
% Cl	-	0.16	0.18	0.11	0.23
% H / 100 C	4.1	4.0	4.3	4.1	3.9
Heat value, BTU	7,000	7,640	7,560	7,900	7,350
Heat of combustion BTU	7,160	7,840	7,760	7,180	7,510
Comp. of Ash: % SiO_2	24.3	25.2	26.3	24.9	24.2
% Fe_2O_3	21.7	21.4	21.3	20.6	22.6
% Al_2O_3	24.8	25.6	25.4	25.8	24.3
% CaO	5.8	5.5	6.6	6.4	6.5
% MgO	3.1	3.3	3.3	3.7	3.3
% K_2O / Na_2O	6.8	6.6	6.4	6.3	6.6
% SO_3	10.1	8.0	6.3	8.6	9.7
% P_2O_5	0.8	0.8	0.8	0.9	0.8
% Cl	-	-	-	-	-
% TiO_2	2.3	2.4	2.3	2.6	[illegible].9

TABLE 7

Centrifuge Oils

Period	1	2	3	4
Sp. Gravity/100°	1.082	1.092	1.100	1.095
% Benz. Insoluble	8.9	12.6	8.8	11.5
% Ash in Solids	33.1	35.0	31.5	28.9
In Oil: Sp. Gr./100°	1.062	1.064	1.059	1.061
%-Asphalt	9.9	12.2	12.4	13.4
% - 325°	2.8	27.3	33.8	30.2
Vacuum Distill. at 18 mm				
Fraction: - 225° : %	22.0	30.7	38.8	36.8
Sp.Gr./40°	1.012	0.991	0.975	0.983
225 - 275° : %	33.7	20.7	12.7	13.8
Sp.Gr./60°	1.038	1.047	1.045	1.055
275 - 325° : %	12.2	11.7	10.0	10.6
Sp. Gr./90°	1.057	1.065	1.061	1.065
Ultimate Analysis: % C	90.21	89.85	89.16	89.72
H	7.41	7.45	7.54	7.39
O	1.47	1.78	2.25	1.91
N	0.83	0.86	1.00	0.95
S	0.03	0.05	0.04	0.04
Cl	< 0.01	< 0.01	< 0.01	0.01
%H/ 100 C	8.2	8.3	8.5	8.2
Heating Value	9130	9150	9084	9154
Heat of Combust.	9530	9550	9491	9553
In Solids: Dry Sample:				
% Ash (Res. on Ignition)	41.10	40.85	32.69	35.30
% C	53.72	54.95	61.24	58.90
% H	2.15	2.16	2.65	2.50
% O	-	-	-	-
% N	0.63	0.68	0.80	0.67
% S Volatile	2.09	1.77	2.10	2.73
% S Fixed	3.41	3.40	3.07	3.88
% Cl	0.13	0.10	0.16	0.13
% H / 100 C	3.8	3.9	4.3	4.2
Heating Value	4970	5051	5600	5390
Heat of Combustion	5086	5155	5742	5530
Ash: % SiO_2	27.2	27.7	26.1	23.2
% Fe_2O_3	20.6	19.3	22.6	27.1
% Al_2O_3	28.0	29.5	25.6	17.7
% CaO	5.5	4.8	5.3	6.0
% MgO	2.9	3.0	3.1	3.1
% K_2O + Na_2O	5.9	6.8	6.7	5.9
% SO_3	5.9	4.6	7.6	13.0
% P_2O_5	0.4	1.0	1.1	1.0
% Cr	-	-	-	-
% TiO_2	3.2	2.5	2.0	3.0

TABLE 8

Centrifuge Residue

Period	1	2	3	4
Sp. Gravity/100°	1.266	1.263	1.255	1.264
% Benz. Insoluble	38.6	37.8	38.5	40
% Ash in Solids	53.5	53.8	55.4	55.4
In Oil: Sp. Gr./100°	1.058	1.061	1.050	1.059
s-Asphalt	8.2	11.3	11.5	11.6
% - 325°	3.4	18.5	32.2	30.2
Vacuum Distill. at 18 mm				
Fraction: - 225° : %	23.7	29.4	40.3	37.1
Sp. Gr./40°	1.014	0.991	0.976	0.985
225 - 275° : %	31.5	21.8	12.5	13.8
Sp.Gr./60°	1.038	1.045	1.047	1.052
275 - 325° : %	15.4	12.0	9.7	9.5
Sp. Gr./90°	1.058	1.061	1.063	1.067
Ultimate Analysis:				
% C	90.31	89.90	89.26	90.03
H	7.45	7.58	7.56	7.52
O	1.40	1.78	2.28	1.38
N	0.81	0.88	0.85	1.02
S	0.03	0.05	0.05	0.04
Cl	-	<0.01	<0.01	0.01
% H/100 C	8.2	8.2	8.5	8.4
Heating Value	9188	9172	9118	9115
Heat of Combust.	9590	9571	9526	9521
In Solids: Dry Sample				
% Ash (Res. on Ignition)	57.40	54.85	56.15	57.10
% C	37.85	40.50	38.95	36.42
% H	1.62	1.80	1.71	1.56
% O	-	-	-	-
% N	0.50	0.54	0.39	0.43
% S Volatile	3.48	2.83	2.08	2.80
% S Fixed	5.76	5.34	5.15	5.59
% Cl	-	-	-	0.12
% H/100 C	4.3	4.4	4.4	4.3
Heating Value	3325	3775	3550	3337
Heat of Combust.	3320	3875	3646	3414
Ash: % SiO_2	24.1	25.5	26.2	25.2
% Fe_2O_3	25.8	23.7	23.8	21.1
% Al_2O_3	22.6	26.2	23.2	22.1
% CaO	6.7	6.7	7.4	8.0
% MgO	3.4	3.6	3.6	3.9
% K_2O + Na_2O	4.9	7.4	7.1	5.8
% SO_3	10.8	4.3	7.5	10.8
% P_2O_5	0.4	0.8	0.6	0.7
% Cl	-	-	-	-
% TiO_2	3.5	2.2	1.5	2.6

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TABLE 9

KILN OILS

Heavy Kiln Oil.

Period	1	2	3	4	5
Sp. Gr./100°	1.138	1.117	1.088	1.073	-
% H_2O	0.1	0.2	0.1	0.1	-
% Solids	4.0	4.5	3.0	6.1	-
% Ash in Solids	21.3	22.5	20.6	16.3	
In Oil: Sp. Gr./100°	1.142	1.076	[illegible]	1.063	-
% s-Asphalt	11.6	7.8	9.1	8.7	-
% - 325°	3.8	15.2	36.7	32.4	-
Vacuum Distill. at 18 mm Hg					
Fractions - 225° %	14.4	32.6	39.5	38.5	-
Sp. Gr./40°	1.043	1.021	1.023	1.013	-
225 - 275° %	36.4	28.0	22.8	22.3	-
Sp. Gr./50°	1.092	1.081	1.094	1.090	-
275 - 325°	16.0	12.4	12.7	12.3	-
Sp. Gr./90°	1.112	1.109	1.105	1.101	-
Ultimate Analysis: % C	90.22	91.09	90.24	90.13	-
H	5.93	6.77	6.69	7.00	-
O	2.80	1.38	2.11	2.14	-
N	0.95	0.75	0.90	0.67	-
S	0.10	0.06	0.05	0.05	-
Cl	<0.01	0.01	<0.01	0.01	-
% H/212°F	6.6	7.4	7.4	7.8	-
Heating Value	8901	9050	9026	8996	-
Heat of comb.	9220	9415	938[illegible]	9374	-

Light Kiln Oil.

	1	2	3	4	5
Sp. Gr.	1.037 15°	1.042 20°	0.985 20°	1.012 20°	-
% H_2O	2.5	0.3	1.5	-	-
% Solids	0.4	0.4	0.3	0.6	-
% Ash in Oil	0.2	0.02	Spur	Spur	-
% Asphalt	2.5	1.4	0.5	0.6	-
Phenols	-	-	20.2	23.8	-
Ultimate Analysis: % C	88.52	89.25	[illegible]	84.82	-
H	7.22	7.44	7.99	7.94	-
O	3.85	2.82	5.26	6.34	-
N	0.34	0.42	0.69	0.86	-
S	0.05	0.05	0.04	0.03	-
Cl	<0.01	0.01	0	<0.01	-
% H/212°F	8.2	8.3	9.3	9.4	-
Heating Value	9091	9101	9017	8799	-
Heat of Comb.	9481	9503	9448	9228	-

TABLE 10

RAW GASOLINE FROM LIGHT KILN OIL (SCHWELOFEN) LABORATORY ANALYSIS

Test Period	1	2	3
% of Light Kiln Oil	9.6	16.0	25.8
Sp. Gr./15°	0.957	0.940	0.936
A.P.I.	26.1	35.0	26.5
Initial boil. pt. °C °F	133° 273	106° 208	80° 176
% - 100° - 212		—	3.5
- 150° - 302	3.0	3.0	12.2
- 180° - 356	47.0	51.5	57.6
- 200° - 392	81.8	85.0	92.3
- 220° - 428	93.0	95.0	—
End point °C / % °F	222°/95.5 432	239°/97.9 462	208°/98.2 406
Residue	3.0	1.5	1.4
Loss	1.5	0.6	0.4
Element Analysis:			
% C	84.94	85.55	82.93
H	8.45	8.68	8.63
O	6.10	4.15	7.65
N	0.47	0.57	0.72
S	0.03	0.05	0.02
Cl	<0.01	0.01	—
% H / 100 C	10.0	10.0	10.5
Heating value	9586	9461	8727
Heat of comb.	9459	9451	9196
% Phenols	—	—	16.5
% Paraffins 1)	—	—	32
% Naphthenes	—	—	29.5
% Aromatics	—	—	29
% Unsaturates	—	—	9.5

1) De-phenolated

TABLE 11

MIDDLE OIL FROM LIGHT COOLER OIL (SCHWELOFEN) LABORATORY DISTILLATION

Test Period	1	2	3
Crude M.O.:			
% of Light Kiln Oil	45.2	42.5	62.9
Sp. Gr./15°	1.023	1.016	0.990
Phenols	15.7	11.6	26.2
A.P.I	-35.0	-35.3	-33.3
Engler: Initial B.P., °C.	200°	200°	200°
% - 225°C	13.7	15.7	50.7
- 250°	38.2	43.9	81.8
- 275°	60.4	63.1	93.6
- 300°	79.5	80.0	
- 325°	93.2	92.5	
End point / %	340°/97.4	329°/95.6	290°/97.3
	644	624	553
Residue	2.1	4.0	2.5
Loss	0.5	0.4	0.2
De-phenolated Middle Oil:			
Sp. Gr./15°	0.975	[illegible]	0.971
A.P. I	-45.8	-40.7	-35.2
Unsaturated	21.6	22.7	18.3
Fractions			
- 225°: Sp. Gr./15°	0.965	0.967	0.956
A.P. I	-47.9	-46.8	[illegible]
225 - 250°: Sp. Gr./15°	0.976	0.980	0.965
A.P. I	-45.4	-45.3	-40.1
250 - 275°: Sp. Gr./15°	0.994	0.995	0.978
A.P. I	-43.0	-47.2	-35.8
275 - 300°: Sp. Gr./15°	1.012	1.013	0.997
A.P. I	-38.8	-40.1	-32.4
300 - 325°: Sp. Gr./15°	1.022	1.036	
A.P. I	-27.9	-34.7	

TABLE 12

HEAVY OIL FR[illegible] OIL (SCHOLVEN) LABOR. DISTILLATION

Test Period		1	2	3
% Light Ends [illegible]		33.2	41.4	11.3
Sp. Gr./20°		1.107	1.116	1.081
Vacuum Distill. 22 mm Hg				
Fractions:	- 200° %			
	Sp. Gr.			
	- 225° %	10.[illegible]	9.5	19.2
	Sp. Gr.	1.[illegible]/80°	1.070/40°	1.0[illegible]3/40°
	225 - 250° %	3[illegible]	30.2	17.4
	Sp. Gr.	1.095/80°	1.075/80°	1.043/40°
	250 - 275° %	19.[illegible]	19.3	19.2
	Sp. Gr.	[illegible]/80°	1.102/80°	1.057/80°
	275 - 300° %	9.1	5.1	22.6
	Sp. Gr.	1.114/80°	1.107/80°	1.079/80°
	300 - 325° %	6.4	6.9	20.9
	> 325° %	22.3	26.4	[illegible]
	[illegible] %	[illegible]	[illegible]	[illegible]

TABLE 13

KILN RESIDUE

Test Period	1	2	3	4
Solids in dry Sample:				
% Ash (Ignition Residue)	42.05	46.10	43.92	44.35
% C	55.68	52.21	53.28	53.45
% H	1.30	1.14	1.42	1.57
% O	-	-	-	-
% N	0.73	0.38	0.44	0.35
% S vol.	1.85	1.99	1.84	1.89
% S fixed	4.11	4.51	4.02	4.00
% Cl	-	0.12	0.21	0.14
% H /100 C	2.3	2.2	2.7	2.9
Heat of combustion	5030	4810	4865	4539
Heating value	4956	4740	4787	4492
Analysis of Ash % SiO_2	-	23.7	25.3	-
% Fe_2O_3	-	25.0	23.8	-
% Al_2O_3	-	23.3	22.6	-
% CaO	-	6.5	6.1	-
% MgO	-	3.4	3.1	-
% $K_2O + Na_2O$	-	6.7	5.7	-
% SO_3	-	7.8	9.9	-
% P_2O_5	-	0.7	0.7	-
% TiO_2	-	2.4	1.5	-

TABLE 14

COLD CATCH POT BOTTOMS

Test Period	1	2	3	4	5
Sp. Gr./15°	0.999	0.935	0.998	0.984	0.985
% H_2O	2.0	2.4	2.3	1.4	1.8
% Phenols	9.3	10.2	10.9	14.2	16.5
Weight Engler:					
Initial boiling point –	65°	59°	65°	65°	57°
% – 100°	1.7	2.3	1.8	2.3	2.8
– 125°	3.0	3.5	2.6	3.0	3.4
– 150°	4.8	5.2	3.8	3.9	4.5
– 175°	7.2	8.4	7.6	7.7	7.[illegible]
– 200°	9.8	11.[illegible]	11.5	11.3	11.5
– 225°	13.4	18.2	21.0	19.0	20.4
– 250°	19.5	27.[illegible]	31.2	31.3	34.4
– 275°	25.2	35.[illegible]	44.1	42.1	44.6
– 300°	33.0	45.[illegible]	54.7	55.7	56.0
– 325°	45.6	57.[illegible]	64.8	66.5	63.1
– 350°	53.[illegible]	71.3	75.9	75.1	75.0
Residue	[illegible]	27.[illegible]	23.4	23.7	24.0
Loss	0.8	0.8	0.7	1.2	1.0
Laboratory Column Distillation:					
– 200°	14.[illegible]	16.7	16.9	17.0	14.7
200 – 325°	55.[illegible]	40.[illegible]	48.1	48.0°	48.2
325°	32.1	41.[illegible]	34.0	33.3	35.9
% H_2O	0.7	0.6	0.8	0.5	0.7
% Loss	1.1	0.3	0.2	1.0	0.5
Ultimate Analysis – % C	87.33	86.85	85.76	87.22	86.53
H	9.09	9.2[illegible]	9.44	9.33	9.35
O	2.73	3.[illegible]	3.15	2.59	3.23
N	0.59	0.54	0.51	0.75	0.55
S	0.03	0.08	0.03	0.04	0.03
Cl	0.01	<0.01	<0.01	<0.01	<0.01
H / 100 C	10.4	10.[illegible]	10.9	10.73	10.80
Heating Value	9[illegible]	9234	9316	9257	9302
Heat of Combustion	96[illegible]	97[illegible]	9826	9754	9307

Table 15

Raw Gasoline Invest. (Lab Analysis of C.C.P. Bottoms with no Lt. Kiln Oil).

Test Period	1	2	3	4	5
Specific Gravity/15°	0.835	0.853	0.851	0.840	0.834
Phenols %	18.0	26.6	24.5	16.7	22.4
A.P. I	+15.2	+10.2	+16.0	+14.4	+15.5
A.P. Ia	+30.5	+ 7.1	+ 7.3	+14.7	+17.0
A.P. II	+54.1	+53.5	+53.9	+51.0	+48.0
Iodine Number		31.5	19.4	17.5	13.7
ASTM: Init. b.p. °C	46	43	46	38	37
% - 50°	0.5	1.3	0.2	1.5	2.0
- 70°	6.4	10.3	6.6	6.5	8.0
-100°	20.0	20.8	19.4	20.9	23.4
-150°	51.3	46.0	45.9	50.7	53.4
-180°	78.7	70.9	72.3	78.7	79.8
- 200°	92.1	92.4	92.3	93.9	93.8
E.P. °C / %	214°/	214°/	218°/	208°/	205°/
	96.2	97.9	97.9	96.5	97.4
Residue	1.2	1.2	1.3	1.6	1.5
Loss	2.6	0.9	0.8	1.9	1.1
Fractions:					
90 - 100°:%	9.6 1)	3.9	4.7	6.4	6.4
Specific Gravity/15°	0.750	0.739	0.739	0.743	0.740
A.P.I	+36.4	+33.8	+33.7	+37.5	+33.0
120 - 140°:%	13.3	10.8	10.4	6.6 2)	12.2
Specific Gravity/15°	0.795	0.796	0.794	0.798	0.795
A.P.I	+21.1	+22.5	+22.3	+23.6	+23.0
160 - 180°:%	21.1	19.3	19.7	21.6	20.8
Specific Gravity/15°	0.886	0.856	0.885	0.896	0.895
A.P. I	+ 4.5	+ 3.0	+ 3.8	+ 3.5	+ 4.0
190 - to		210°	209°	208°	205°
%		10.0	10.2	6.8	8.2
Specific Gravity/15°		0.954	0.954	0.960	0.955
A.P.I		8.5	- 5.9	- 7.0	-3.1
Composition: % Paraff.	30.5	21.2	22.5	21.5	17.5
% Naphthene	33.5	25.0	25.8	36.0	45.0
% Aromatics	23.0	40.8	43.5	35.5	32.0
% Unsaturated	13.0	13.0	8.2	7.0	5.5
Ultimate Analysis % C	83.51	82.89	82.92	83.81	83.40
H	11.85	11.56	11.33	11.71	12.01
O	4.11	4.81	5.16	3.81	3.97
N	0.50	0.73	0.58	0.64	0.60
S	0.02	0.01	0.01	0.02	0.01
Cl	0.01	<0.01	<0.01	<0.01	<0.01
% H / 100 C	14.2	13.9	13.7	14.0	14.4
Heating Value	9512	9423	9366	9568	9453
Heat of Comb.	10152	10022	9977	10200	10102

1) 80 - 100°
2) 120 - 130°

Table 16

Crude Gasoline Invest. (Tech. Distillation of C.C.P. Bottoms with no Lt. Kiln Oil)

Test Period	1	2	3	4	5
Specific Gravity/15°	0.792	0.810	0.798	0.806	0.794
Phenols %	3.6	6.9	4.3	ca 3	3.8
A.P. I	+ 19.6	+ 16.5	+ 17.3	+ 15.5	+ 17.8
A.P. Ia	+ 31.1	+ 14.4	+ 16.6	+ 16.6	+ 17.1
A.P. II	+ 53.5	+ 54.1	+ 53.3	+ 52.4	+ 52.7
Iodine Number		29.0	21.7	18.5	14.3
ASTM: Init. b.p. °C	44	54	42	43	38
% - 50°	0.6	-	2.0	-	2.0
-70°	6.6	4.5	7.7	7.0	9.0
-100°	26.8	18.4	25.8	23.0	27.0
-150°	69.8	61.9	63.8	62.0	64.4
-180°	89.8	88.6	86.1	85.4	84.0
-200°	96.3	96.3	94.9	94.8	92.2
E.P. °C / %	204°/ 98.5	207°/ 97.9	210°/ 98.1	210°/ 97.8	210°/ 95.4
Residue	0.8	1.4	1.5	1.2	1.6
Loss	0.9	0.7	0.4	1.0	3.0
Fractions:					
90 - 100°: %	6.2 1)	5.5	7.4	7.4	8.0
Specific Gravity/15°	0.742	0.737	0.736	0.742	0.740
A.P.I	+ 31.6	+ 28.7	+ 30.1	+ 30.1	+ 30.5
120 - 140°: %	17.5 2)	15.8	14.5	16.2	13.8
Specific Gravity/15°	0.790	0.783	0.789	0.794	0.796
A.P.I	+ 15.8	+ 20.0	+ 18.8	+ 19.7	+ 19.0
160 - 180°: %	13.3 3)	17.6	14.8	16.2	15.2
Specific Gravity/15°	0.849	0.850	0.855	0.858	0.853
A.P. I	+ 7.2	+ 7.2	+ 3.2	+ 4.2	+ 4.4
190 - to		207°	209°	205°	210°
%		4.0	5.5	7.0	6.4
Specific Gravity/15°	—	0.875	0.888	0.894	0.887
A.P.I		+ 1.6	- 6.2	- 5.2	- 8.8
Composition:					
% Paraff.	30.0	24.5	28.0	25.0	25.5
% Naphthene	36.0	27.2	27.8	33.5	33.0
% Aromatics	23.0	36.5	35.5	35.0	36.0
% Unsaturated	11.0	11.8	8.7	6.5	5.5
Ultimate Analysis % C	85.31	85.18	85.60	86.23	85.70
H	12.88	12.59	12.76	12.42	12.97
O	1.45	1.80	1.43	0.92	0.97
N	0.34	0.41	0.18	0.41	0.33
S	<0.01	0.01	0.02	0.02	0.02
C	<0.01	<0.01	<0.01	<0.01	<0.01
% H / 100 C	15.1	14.8	14.9	14.4	15.1
Heating Value	9982	9917	10002	9944	9987
Heat of Comb.	10678	10597	10690	10614	10687

1) 80-90° 2) 110-130° 3) 150-170°

Table 17

Invest. of Dephenolated Raw Gasoline
(Lab. Distillation of Catchpot Bottoms with no Kiln Oil added)

Test Period	1	2	3	4	5
Specific Gravity/15°	0.795	0.803	0.800	0.794	0.788
Unsaturated %	3.5	7.4	8.8	7.0	6.5
A.P. I	+17.5	+15.2	+15.2	+17.4	+19.8
A.P. Ia	+32.3	+13.4	+20.2	+22.3	+24.0
A.P. II	+54.3	+54.2	+54.3	+52.3	+52.6
Iodine Number		18.7	14.0	14.1	9.5
ASTM: Init. b.p. °C	43°	45°	49°	45°	45°
% - 50°	-	0.9	-	0.2	0.8
- 70°	4.0	5.7	3.2	5.0	6.0
-100°	23.0	21.0	20.4	22.4	27.9
-150°	62.0	54.4	57.1	62.0	63.5
-180°	82.6	73.8	76.9	80.6	82.7
-200°	91.6	88.9	90.2	92.2	95.5
E.P. °C / %	214°/	220°/	221°/	212°/	212°/
	96.6	98.0	98.1	97.0	97.5
Residue	1.2	1.2	1.4	1.8	1.2
Loss	2.2	0.8	0.5	1.2	1.3
Fractions:					
90--100°: %	6.0[1]	6.4	6.8	8.0	11.0
Specific Gravity/15°	0.737	0.725	0.733	0.737	0.741
A.P.I	+33.5	29.5	+33.6	+33.5	+33.3
120 - 140°: %	16.4[2]	15.7	13.2	19.1	14.8
Specific Gravity/15°	0.787	0.767	0.783	0.778	0.785
A.P.I	+19.0	+23.7	+21.1	+20.0	+21.4
160 - 180°: %	12.8[3]	13.5	13.8	10.2	13.0
Specific Gravity/15°	0.837	0.831	0.834	0.835	0.836
A.P.I	+ 6.8	+ 4.4	+ 4.6	+ 6.0	+ 7.5
190 -		210°	211°	212°	212°
%		10.6	11.3	9.4	7.8
Specific Gravity/15°		0.867	0.870	0.870	0.870
A.P.I		- 2.5	- 1.7	- 1.2	+ 0.3
Composition:					
% Paraffine	32.5	27.8	29.5	26.5	28.0
% Naphthene	35.0	38.0	30.8	37.0	38.0
% Aromatics	22.5	34.7	34.5	31.5	30.0
% Unsaturated	10.0	7.5	5.8	5.5	4.0
Ultimate Analysis % C	86.73	86.72	86.16	86.55	86.21
H	12.90	12.74	13.04	13.02	13.08
O	0	0.10	0.38	0.00	0.35
N	0.35	0.42	0.40	0.41	0.34
S	0.02	<0.01	0.01	0.02	0.01
Cl	<0.01	<0.01	<0.01	<0.01	<0.01
% H / 100 C	14.9	14.7	15.1	15.0	15.2
Heating Value	10142	10092	10126	10179	10115
Heat of Comb.	10839	10780	10830	10882	10821
Octane Number Research	80.0	76.2	80.5	77.0	77.0
Motor	69.2	72.0	73.8	72.0	71.0

1) [illegible] 2) [illegible] 3) 150-170°

Table 18

Investigation of the De Phenolated Raw Gasoline (Technical Distillation of the Let-Down with Kiln Oil Addition)

Test Period	1	2	3	4	5
Specific Gravity/15°C	0.787	0.788	0.789	0.796	0.786
Unsaturated %	6.9	7.3	8.5	8.5	7.5
A.P. I	+20.4	+19.5	+18.3	+16.2	+17.7
A.P. Ia	+30.8	+24.0	+23.0	+21.3	+21.0
A.P. II	+53.3	+53.8	+52.0	+52.4	+52.8
Iodine Number	21.8	22.9	18.6	16.0	11.5
ASTM: Init. b.p. °C	47°	39°	42°	47°	40°
% - 50°	-	5.4	0.7	1.5	1.0
-70°	4.2	9.0	6.4	8.4	8.0
-100°	26.2	26.6	26.9	30.8	29.7
-150°	69.5	66.8	66.4	70.0	66.9
-180°	88.9	86.5	84.8	89.4	83.5
-200°	96.4	96.2	93.7	96.2	93.5
E.P. °C / %	205°	206°	208°	213°	211°
	98.4	97.8	96.8	97.2	96.5
Residue	1.0	1.2	1.5	1.6	1.8
Loss	0.6	1.0	1.7	1.2	1.7
Fractions:					
90 - 100°; %	7.0 1)	8.4	8.1	9.6	9.7
Specific Gravity/15°	0.740	0.733	0.739	0.741	0.741
A.P.I	+32.4	+33.9	+30.3	+32.0	+30.4
120 - 140°; %	17.2 2)	14.4	14.6	15.2	15.7
Specific Gravity/15°	0.785	0.782	0.784	0.785	0.791
A.P.I.	+19.7	+18.9	+18.4	+18.6	+17.5
160 - 180°; %	13.2 3)	12.2	11.7	13.4	11.5
Specific Gravity/15°	0.832	0.836	0.836	0.840	0.838
A.P.I.	+ 9.1	+ 7.8	+ 5.2	+ 4.5	+ 4.5
190° -			208°	213°	211°
%			6.2	3.6	7.0
Specific Gravity/15°			0.870	0.874	0.873
A.P.I.			- 3.6	- 3.0	- 1.2
Composition:					
% Paraffine	30.0	29.0	25.8	26.0	27.0
% Naphthene	37.5	32.0	37.5	35.5	35.0
% Aromatics	24.0	30.0	29.5	32.0	33.0
% Unsaturated	8.5	9.0	7.2	6.5	5.0
Ultimate Analysis % C	86.26	86.22	86.32	86.67	86.92
H	13.28	13.18	13.09	12.87	12.89
O	0.19	0.31	0.44	0.09	0.90
N	0.25	0.26	0.13	0.34	0.28
S	<0.01	0.02	0.02	0.02	<0.01
Cl	<0.01	<0.01	<0.01	<0.01	trace
% H / 100 C	15.4	15.3	15.2	14.9	14.8
Heating Value	10113	10140	10133	10092	10074
Heat of Comb.	10830	10852	10840	10780	10770
O.N. Research	-	-	79.3	77.0	79.5
Motor	69	ca. 70	72.3		72.0

Table 19

Gasoline from Industr. Distillation With Gasol
Raw Gasoline

Test Period	1	2	3	4	5
Gasol. in the mixture	11.8	7.1	11.7	12.2	14.5
Specific Gravity/15°	0.793	0.794	0.787	0.806	0.786
Phenols %	6.8	4.8	2.3	2.7	3.2
A.P. I	+22.0	+20.1	+20.5	+16.7	-
A.P. Ia	+18.3	+17.7	+21.2	+17.7	+18.0
A.P. II	+53.7	+54.1	+51.8	+52.7	+ 52.2
Iodine Number	21.9	24.2	21.7	15.8	11.9
ASTM: Init. b.p. °C	39°	40°	35°	49°	38°
% - 50°	3.6	2.6	3.5	-	2.0
% - 70°	13.4	10.4	12.8	3.5	11.0
% - 100°	34.0	29.5	34.2	21.2	36.0
% - 150°	65.8	65.1	68.2	62.8	70.2
% - 180°	88.8	88.1	87.1	87.4	88.2
% - 200°	95.4	94.9	94.8	95.4	95.2
E.P. °C / %	210°/	207°/	209°/	205°/	206°/
	97.8	96.7	96.7	97.4	97.2
Residue	1.2	2.4	1.6	1.6	1.4
Loss	1.0	0.9	1.7	1.0	1.4
Fractions:					
90 - 100° : %	7.0[1]	7.3	7.9	7.2	9.5
Specific Gravity/15°	0.720	0.735	0.738	0.740	0.741
A.P.I.	+35.4	+32.8	+33.3	+34.3	+31.8
120 - 140° : %	12.6[2]	13.6	13.1	16.6	13.2
Specific Gravity/15°	0.783	0.790	0.794	0.790	0.792
A.P.I	+21.6	+19.6	+18.5	+20.5	+19.3
160 - 180°	15.0[3]	15.8	12.7	15.8	13.0
Specific Gravity/15°	0.860	0.857	0.859	0.852	0.850
A.P.I	+ 8.8	+ 6.4	+ 3.4	+ 4.2	+ 4.4
190 -	210°		209°	205°	
%	4.0		5.4	4.4	
Specific Gravity/15°	0.895		0.889	0.889	
A.P.I	- 1.4		- 4.9	- 3.1	
Composition:					
% Paraffine	25.5	21.5	24.3	25.0	24.5
% Naphthene	31.5	35.0	36.2	33.5	35.0
% Aromatics	34.0	34.0	30.8	35.0	35.5
% Unsaturated	9.0	9.5	8.7	6.5	5.0
Ultimate Analysis % C	84.67	85.05	85.59	85.80	85.33
H	12.85	12.78	12.93	12.52	13.15
O	2.24	1.74	1.00	1.52	1.25
N	0.21	0.40	0.44	0.11	0.23
S	0.02	0.02	0.03	0.04	0.06
Cl	<0.01	<0.01	<0.01	<0.01	<0.01
% H./ 100 C	15.2	15.0	15.1	14.6	
Heating Value	9961	9873	10048	9914	9863
Heat of Comb.	10655	10563	10747		10573

1) 80-90° 2) 110-130° 3) 150-170°

Table 20

Industrially Distilled Gasoline + Gasol
De-phenolated Crude Gasoline

Test Period	1	2	3	4	5
% Gasol. in the mixture	11.8	7.1	11.7	12.2	14.5
Specific Gravity/15°	0.776	0.748	0.781	0.797	0.779
Unsaturated %	5.6	8.5	7.2	7.0	5.5
A.P. I	+23.7	+20.9	+20.7	+17.3	-
A.P. Ia	+25.0	+25.4	+24.8	+21.5	+27.0
A.P. II	+54.2	+54.5	+52.5	+52.5	+52.4
Iodine Number	20.2	22.7	17.0	14.2	12.4
ASTM: Init. b.p. °C	44°	42°	40°	54°	42°
% - 50°	0.4	0.8	1.5	-	1.0
- 70°	9.0	7.2	10.5	2.0	9.0
-100°	33.2	28.6	31.8	19.4	35.2
-150°	69.4	67.8	66.9	63.6	72.8
-180°	87.4	87.4	84.4	86.2	88.2
-200°	96.4	96.4	92.9	94.6	95.2
E.P. °C / %	208°/ 98.2	206°/ 98.0	209°/ 96.0	207°/ 97.4	205°/ 97.4
Residue	1.2	1.2	1.8	1.5	1.4
Loss	0.0	0.8	2.2	1.1	1.2
Fractions:					
90 - 100°: %	8.2 1)	8.4	7.3	8.6	10.4
Specific Gravity/15°	0.721	0.773	0.739	0.734	0.735
A.P.I	+34.7	+33.9	+32.7	+34.8	+33.8
120 - 140°: %	15.0 2)	14.4	13.1	17.8	13.2
Specific Gravity/15°	0.781	0.782	0.792	0.785	0.783
A.P.I	+21.1	+18.9	+17.0	+20.2	+20.5
160 - 180°: %	12.0 2)	12.2	12.1	15.2	10.2
Specific Gravity/15°	0.833	0.836	0.852	0.836	0.835
A.P.I	+ 8.2	+ 7.8	+ 1.2	+ 5.5	+ 6.2
~~190~~	208°		209°	207°	205°
%	5.6		7.0	6.2	5.7
Specific Gravity/15°	0.861		0.885	0.870	0.871
A.P.I	+ 6.8°		- 5.3°	- 2.3	+ 1.0
Composition:					
%Paraffine	30.5	31.0	28.0	26.5	29.0
%Naphthene	33.0	31.0	37.5	36.0	39.5
%Aromatics	28.5	29.0	27.8	32.0	26.5
%Unsaturated	8.0	9.0	6.7	5.5	5.0
Ultimate Analysis % C	-	86.22	86.36	86.70	86.38
H	-	13.18	13.23	12.81	13.25
O	-	0.31	0.12	0.32	0.17
N	-	0.26	0.26	0.13	0.12
S	-	0.02	0.03	0.03	0.07
Cl	-	<0.01	<0.01	<0.01	<0.01
% C / 100 C	-	15.3	15.3		
Heating Value	-	10140	10173	10015	10157
Heat of Comb.	-	10852	10888	10707	10873
O.N. (Research	-	74	-	-	-
(Motor	69.7	69.7	72.4	70.2	

1) 80-90° 2) 110-130° 3) 150-170°

TABLE 21

Study of Middle Oil (Laboratory distillation of catch pot bottoms with no addition of kiln oil)

Crude Middle Oil

Test Period		1	2	3	4	5
Sp. Gr./15°		0.979	0.981	0.964	0.976	0.971
A.P. I °C		-31.0	-30.1	-30.2	-29.4	-23.3
% Phenols		12.9	15.0	15.5	22.1	24.1
Weight-Engler:						
Initial b.p.		221°	222°	191°	205°	201°
% - 225°		1.3	0.4	7.0	12.2	18.3
- 250°		17.7	24.8	40.1	43.5	50.6
- 275°		53.9	56.9	60.0	68.7	74.2
- 300°		80.7	83.2	89.6	90.2	91.4
- 325°		95.9	-	-	-	-
E.P. °C/ %		327°/ 97.5	324°/ 96.4	319°/ 97.9	319°/ 97.6	316°/ 97.2
Resid.		1.9	3.0	1.9	2.2	2.7
Loss		0.6	0.6	0.2	0.2	0.1
Ultimate-Analyses:	% C	87.08	86.68	86.54	86.24	86.32
	H	9.66	9.42	9.56	9.64	9.35
	O	2.58	3.36	3.28	3.56	3.45
	N	0.64	0.51	0.60	0.53	0.84
	S	0.03	0.02	0.01	0.02	0.03
	Cl	<0.01	<0.01	<0.01	<0.01	<0.01
% H / 100 C		11.1	10.9	11.0	11.2	10.8
Heating Val.		9343	9357	[illegible]	9317	9267
Heat of comb.		9865	9865	9844	9839	9772

De-phenolated Middle Oil

Sp. Gr./15°		0.968	0.963	0.956	0.957	0.950
A.P. I °C		-29	-25.6	-24.0	-26.6	-23.7
Unsatur.		13.0	14.0	9.8	10.0	11.0
Fractions:	Initial b.p.	227°	223°	[illegible]	200°	210°
to 225°	Sp. Gr./15°	-	-	0.930	0.903	0.922
	A.P. I °C	-	-	-22.4	-21.2	-25.1
225-250°	Sp.Gr./15°	0.939	0.936	0.933	0.932	0.932
	A.P. I °C	-25.0	-24.8	-25.0	-26.0	-25.1
250-275°	Sp.Gr./15°	0.955	0.946	0.947	0.950	0.950
	A.P. I °C	-27.4	-25.3	-25.7	-27.2	-22.1
275-300°	Sp.Gr./15°	0.975	0.959	0.969	0.970	0.970
	A.P. I °C	-29.6	-25.2	-26.1	-27.4	-23.1
300°-to		323°	325°	324°	309°	
	Sp.Gr./15°	0.991	0.987	0.986	0.984	-
	A.P. I	-30.7	-25.9	23.3	-24.8	

Table 22

Middle Oil Studies (Ind. Distillation of Catchpot Bottoms with Addition of Kiln Oil)

Crude Middle Oil

Test Period	1	2	3	4	5
Sp. Gr./15°	0.979	0.981	0.982	0.982	0.978
A.P.I °C	-25.7	-28.7	-27.8	-27.3	-27.8
% Phenols	21.6	15.9	19.5	22.1	21.4
Weight-Engler:					
Initial b.p.	161°	191°	197°	194°	196°
% 200°	2.5	1.3	1.1	0.9	1.4
% 225°	12.9	15.4	17.5	20.1	19.8
% 250°	32.4	35.5	40.4	42.8	43.9
% 275°	55.5	56.6	58.7	65.2	65.4
% 300°	80.5	79.3	77.9	83.1	84.9
% 325°	-	-	92.6	94.5	
E.P. °C/%	324°/	326°/	331°/	327°/	322°/
	96.5	97.2	96.8	96.3	96.8
Resid.	3.2	2.7	2.9	2.8	2.9
Loss	0.3	0.1	0.3	0.9	0.3
Ultimate-Analyses: %C	86.18	85.69	85.62	85.96	85.73
H	9.43	9.45	9.42	9.30	9.30
O	3.6	4.12	4.13	3.87	4.03
N	0.67	0.71	0.80	0.84	0.85
S	0.02	0.02	0.02	0.02	0.02
C_1	<0.01	<0.01	<0.01	<0.01	<0.01
% H / 100 C	10.9	11.0	11.0	10.8	10.8
Heating Val.	9197	9249	9219	9175	9139
Heat of Comb.	9706	9760	9728	9677	9642

De-phenolsted Middle Oil

	1	2	3	4	5
Sp. Gr./15°	0.965	0.965	0.961	0.956	0.956
A.P.I °C	-16.5	-28.4	-26.4	-26.1	-24.7
Unsatur.	16.5	14.4	11.9	9.5	12.6
Fractions: Initial b.p.	195°	216°	204°	207°	197°
to 225° Sp.Gr./15°	0.910	0.924	0.917	0.919	0.914
A.P. I °C	-19.5	-21.2	-24.0	-24.1	-23.7
225-250° Sp.Gr./15°	0.925	0.929	0.929	0.932	0.937
A.P.I. °C	-24.5	-25.4	-25.2	-24.5	-26.5
250-275° Sp. Gr./15°	0.950	0.950	0.948	0.950	0.950
A.P. I °C	-28.5	-27.3	-25.7	-25.7	-26.0
275-300° Sp. Gr./15°	0.974	0.974	0.979	0.970	0.977
A.P. I °C	-31.9	-29.4	-26.3	-26.5	-28.3
300° to	325°	325°	325°	320°	322°
Sp.Gr./15°	0.993	0.995	0.992	0.990	0.998
A.P. I °C	-30.3	-30.7	-27.1	-25.3	-27.3

Table 23

Study of Catchpot Heavy Oil (Lab. distillation of catchpot, no kiln oil added)

Test Per.		1	2	3	4	5
Specific Gravity/50°		1.042	1.052	1.052	1.054	1.052
% Phenols		1.0	5.2	4.4	6.3	7.2
Weight Engler:		322°	340°	330°	338°	334°
% - 350°		21.2	12.8	18.0	19.2	19.0
Weight Vac. Distill. (22 mm Hg)						
Initial b.p.		195°	204°	204°	200°	194°
Fractions:						
- 225°: %		25.7	19.1	32.2	28.8	30.8
Spec. Gravity/40°		1.010	1.024	1.024	1.022	1.022
225-250°: %		35.5	34.4	32.2	29.8	29.8
Spec. Gravity/40°		1.034	1.040	1.045	1.046	1.048
250-275°: %		17.0	18.6	17.7	16.6	16.2
Spec. Gravity/80°		1.032	1.040	1.041	1.045	1.042
275-300°: %		9.2	10.5	9.0	10.5	17.8
Spec. Gravity/80°		1.053	1.051	1.049	1.058	1.056
300-325°: %		5.7	6.7	7.6	4.9	6.9
Residue 325°: %		6.3	10.1	9.7	9.0	3.1
Loss		0.6	0.6	0.6	0.4	0.4
Ultimate Analysis: %	C	89.58	89.37	89.01	89.14	88.65
	H	8.22	7.92	8.20	8.00	8.08
	O	1.45	2.09	2.33	1.90	2.25
	N	0.71	0.59	0.42	0.92	0.99
	S	0.03	0.02	0.03	0.03	0.04
	Cl	<0.01	<0.01	<0.01	<0.01	<0.01
% H/ 100 C		9.2	8.9	9.2	9.0	9.1
Heating Valve		9275	9283	9227	9295	9265
Heat of Comb.		9718	9705	9666	9727	9701

Table 24

Catchpot Heavy Oil. (Industrial distillation of Catchpot, with Kiln Oil added)

Test Per.	1	2	3	4	5
Specific Gravity/50°	1.045	1.053	1.056	1.060	1.049
% Phenols	1.2	2.2	2.6	6.5	8.5
Weight Engler:	333	337	351	351	338
% - 350°	20.5	9.4	-	-	11.4
Weight Vac. Distill. (22 mm Hg)					
Initial b.p.	208°	208°	214°	219°	--
Fractions:					
-225° : %	24.2	12.7	15.4	8.1	29.2
Spec. Gravity/40°	1.021	1.038	1.031	1.029	1.022
225-250° : %	32.0	37.8	39.0	39.6	29.5
Spec. Gravity/40°	1.045	1.046	1.045	1.048	1.044
250-275° : %	21.0	20.2	18.1	20.5	17.9
Spec. Gravity/80°	1.034	1.042	1.042	1.042	1.041
275-300° : %	9.6	10.9	12.4	13.3	7.5
Spec. Gravity/80°	1.050	1.055	1.057	1.060	1.059
300-325° : %	6.4	7.1	5.8	6.5	6.9
Residue 325°: %	6.1	9.8	8.2	10.7	8.1
Loss	0.7	1.5	1.1	1.3	0.9
Ultimate Analysis: % C	89.48	89.27	88.63	88.88	89.39
H	8.19	8.08	8.08	7.87	8.02
O	1.53	2.16	2.25	2.68	1.68
N	0.76	0.46	0.99	0.52	0.85
S	0.03	0.02	0.04	0.04	0.05
C_1	<0.01	<0.01	<0.01	<0.01	<0.01
% H/ 100 C	9.1	9.1	9.1	8.8	9.0
Heating Valve	9274	9260	9265	9168	9237
Heat of Comb.	9717	9697	9701	9593	9670

Table 25

Fuel Oil Properties of Catchpot H.O.
Laboratory Distillation, No Kiln Oil Added

Test Periods	1	2	3	4	5
Color	d.green	d.green	d.green	d.green	
Specific Gravity/50°	1.046	1.055	1.049	1.056	-
Pour Point	+ 5°	+ 7°	+ 12°	+ 8°	-
Flash Point	183°	183°	182°	180°	-
Kindling Temp.	236°	238°	233°	233°	-
Coking Test %	0.4	1.7	1.0	1.5	-
s-Asphalt %	0.9	1.8	2.2	1.7	
Ash %	traces	traces	traces	traces	
Phenols %	1.2	5.2	2.8	6.3	-
Viscosity °E/20°	-	-	-	-	-
" " /50°	12.4	19.7	17.6	21.9	
" " /80°	2.7	3.4	3.2	3.5	-
Heating Value	9275	9283	9227	9295	
Heat of Comb.	9718	9705	9666	9727	-

Fuel Oil Properties of Catchpot H.O.
(Industrial Distillation of Catchpot Bottoms with Kiln Oil Added)

Test Periods	1	2	3	4	5
Color	d.green	d.green	d.green	-	d.green
Specific Gravity/50°	1.048	1.057	1.056	-	1.054
Pour Point	+ 7°	+ 8°	+ 8°	-	7°
Flash Point	184°	189°	188°	-	185°
Kindling Temp.	238°	250°	241°	-	236°
Coking Test %	0.7	1.1	1.1	-	1.3
s-Asphalt %	1.0	1.9	2.0	-	2.0
Ash %	traces	traces	traces	-	traces
Phenols %	1.0	4.4	2.6	-	8.5
Paraffine %		1.1	1.3	-	-
Viscosity °E/20°	-	-	-	-	-
" " /50°	12.2	18.5	16.8	-	21.9
" " /80°	2.7	3.3	3.1	-	3.5
Heating Value	9274	9260	9168	-	9237
Heat of Comb.	9717	9697	9593	-	9670

Table 26

Properties of Fuel Oil from Mixtures of H.O. and M.O.

Heavy and Middle Oil from lab. distillation of Catchpot bottoms with no addition of kiln oil.

Test Period	1	2	3	4
Mixing Proport. H.O. : M.O.	74.2 : 25.8	69.5 : 30.5	72:28	71:29
Specific Gravity/20°	1.044	1.046	1.044	1.048
Color	d.green	d. green	d. green	d. green
Pour Point	-3°	-10°	-1.5°	-10°
Flash Point	132°	131°	124°	112°
Kindl. Temp.	170°	163°	159°	150°
Coking Test %	0.55	0.80	0.62	1.3
S - Asphalt %	0.6	0.8	1.5	1.4
Ash	trace	trace	trace	trace
% Phenols in M.O.	12.9	15.0	15.5	22.1
% Paraffines	0.9	-	-	
Viscosities °E/20°	35.6	35.3	35.3	35.2
°E/50°	3.8	3.7	3.7	3.7
Sedimentation Test	12000 Mesh	sieve	no sed	at 0°
Weight Engler: % - 250°			2.0	4.9
% - 275°			9.1	12.1
% - 300°	11.5	16.3	15.1	20.5
% - 325°	22.8	27.6	24.1	29.5
% - 350°	42.1	42.7	38.3	42.0
Ultimate Analysis:% C	87.10		86.64	86.24
H	9.63		9.54	9.64
O	2.45		3.49	3.56
N.	0.79		0.31	0.53
S	0.02		0.01	0.02
Cl	0.01		0.01	0.01
% H / 100 C	11.1		11.0	11.2
Heating Value	9333		9354	9317
Heat of Comb.	9853		9869	9839

Table 27

Properties of Fuel Oil from H.O. - M.O. Mixture

H.O. and M.O. from industrial distillation of catchpot bottoms with addition of kiln oil

Test Period	1	2	3	4	5
Mixing Proport. H.O. : M.O.	74:26	71.8:28.2	65:35	66:34	69:31
Specific Gravity/20°	1.046	1.050	1.045	1.050	1.046
Color	d.green	d.green	d.green	d. green	d.green
Pour Point	-5°	-7°	-12°	-13°	-8°
Flash Point	122°	122°	114°	114°	114°
Kindl. Temp.	159°	154°	148°	145°	142°
Coking Test %	0.58	0.84	0.9	1.4	0.85
S - Asphalt %	0.7	1.0	1.4	1.8	1.0
Ash	trace	trace	trace	trace	trace
% Phenols in M.O.	21.3	14.8	16.0	22.1	21.4
% Paraffines	0.9	0.9	0.7		
Viscosities °E/20°	35.4	35.4	29.4	35.4	35.0
°E/50°	3.8	3.8	3.3	3.7	3.6
Freedom From Sedimentation	12000Mesh Screen at 32°F - Sed. Free				
Weight Engler: % - 250°			8.0	7.9	7.2
% - 275°			15.8	16.4	13.6
% - 300°	14.1		22.2	23.3	19.5
- 325°	23.2	26.3	30.2	29.1	28.3
- 350°	40.7	40.6	43.4	38.1	41.5
Ultimate Analysis % C	86.11			85.96	86.04
H	9.41			9.30	9.35
O	3.51			3.87	3.67
N	0.94			0.84	0.91
S	0.02			0.02	0.02
C	0.01			0.01	0.01
% H / 100 C	10.9			10.8	10.9

Table 28
Fuel Oil Samples for Military Uses

Destination	Kiel-Dietrichsdorf			Deschimag Bremen		Navy Wharf	For Comparison: Navy Specifications			
Shipping Date	X-1-41	X-1-41	XI-29-41	X-18-41	X-18-41	I-14-12				
Amount	10 Ltr.	10 Ltr.	7 Ltr.	48 to	42 to	120 to				
Designation of sample	VIII	IX	X	Qual.A	Qual.B					
Composition % Catchpot H.O.	100	65 1)	65 2)	100	65 1)	75 1)	Old	New 3)	For Delivery	For the Navy
" M.O.	–	35	35	–	35	25				
Analysis: Sp.Gr/20°	1.081	1.043	1.047	1.079	1.041	1.050	>1.030	>1.040		
Viscos. °E/20°	"	22.0	35.5	"	15.5	31.5			No viscosity limits, depending on [illegible]	Under >35°E/20° Under 300 E/0° [illegible] at 0°
" °E/50°	23.5	3.1	3.0	19.1	2.6	4.59				
[illegible] Heat Value Cal/kg	9227	9267	9253	9189	9199	9312	As high as possible (≈9000)			
Low. Heat Val. Cal/C	9980	9650	9700	9900	9600	9800				
% C	89.37	87.79	88.66	89.50	87.91	88.47				
% H	[illegible]	8.73	8.59	8.03	8.47	8.38			No deposits at 0°C if viscosity is below 200° Engler at 0° C	
% S	0.05	0.03	0.05	0.04	0.03	0.04	Not >1.0	Not >0.5		
Pour Point °C	+8	+16	-12	+6	-14	-9				
% Creosote	1.9	11.0	5.5	1.9	12.0	9.6				
% H₂O	trace	0.4	0.1	0.5	0.5	0.2	Not >1.0			
% Ash	trace	trace	trace	trace	trace	trace	Not >0.05		Thicker oils must be free from sediment at that temperature at which their viscosity is equal to 200°	
% N-Gasoline Insol (H-Asphalt)	2.3	1.3	1.1	2.3	1.1	1.7	Below 3.0			
% Xylol Insol.	0.01	<0.01	0.01	0.01	<0.01	0.02	Not >0.2			
Coke, Acc. Conradson %	1.0	0.7	0.73	1.3	0.8	0.85	Below 6.0	Below 2.0		
Flashpoint (PM) °C	184	101	133	180	95	105	>90°	>100°		
Flashpoint (DVM) °C	193	119	146	192	113	123				

1) Normal M.O. 200-325°
2) M.O. 250-325°
3) Not yet in force

Table 29

Phenol Fractionation

Product. Methods		2		3
Product Studied		Gasoline 1)	M.O. 1)	CCP Bott.
% Phenols		7.1	14.8	12.7
Fractions:	% Under	3.9	2.8	3.2
	% Phenol	64.9 2)	4.7	8.9
	% o-Kresol	11.0	8.5	2.6
	% m+p Kresol	11.8	12.5	11.0
	% Xylenol	-	16.6	14.6
	% Higher	4.8	54.9	59.7
	% Loss	3.6	-	-
From Catchpot	% Phenol	0.85		1.1
	% Kresols	1.75		1.7

Phenol Content of Off-Water

Test Period	1	2	3	4	5
g Phenols/Ltr.	0.2-2.9	3.6-4.8	0.9-4.8	0.5	0.4-1.1

Industrial

1) Dist. of catchpot butts: 10.8% Gasoline, 50% M.O.
2) 84.7% Purity, 31.6° Solidific point

Table 20

Analysis of Fresh Oil

Sp. Gr.		0.950/15°
% Phenols		9.9 %
[illegible] Engler	Start. b.p.	83°
	% - 100°	0.8
	- 150°	2.7
	- 180°	5.8
	- 200°	8.3
	- 250°	39.2
	- 300°	74.2
	- 325°	85.9
	- 350°	91.5
Ultimate Analysis	% C	86.68
	H	10.15
	O	2.56
	N	0.53
	S	0.03
	Cl	0
	% H / 100 C	11.7
Heat. Value		9445
Heat of Combust.		9993

/pkl

Table 31
Distribution of Hydrocarbon Gasification (Weight %)

Test Period	1	2	3	4	5
% CH_4	27.1	30.8	33.1	31.6	31.9
% $C_2H_6 + C_2H_4$	24.2	23.8	22.9	22.7	22.9
% $C_3H_8 + C_3H_6$	30.4	26.6	27.0	28.1	29.4
% $C_4H_{10} + C_4H_8$	18.3	18.8	17.0	17.6	15.8
Unsatur.					
% of C_2	1.8	1.4	1.8	1.5	1.4
% of C_3	2.1	2.2	2.3	2.4	2.3
% of C_4	4.2	3.1	4.2	3.8	4.2
% Isobutane in Butane	11	10	11	10-11	11

C Distribution in Hydrocarbon Gasification

Test Period	1	2	3	4	5
C in CH_4 %	25.5	28.8	31.3	29.8	30.1
C in $C_2H_6+C_2H_4$ %	24.2	23.8	23.0	22.8	23.1
C in $C_3H_8+C_3H_6$ %	31.2	27.2	27.8	29.0	30.4
C in $C_4H_{10}+C_4H_8$ %	19.1	20.2	17.9	18.4	16.4

Table 32
Solubilities
Flushing Oil $m^3/m^3/atm$

Test Periods	1	2-5
Sp. Gr. Oil Temperature[1]	0.950/15° 18°	0.950/15° 18°
Solubility: H_2	0.067	0.066
CO	0.097	0.112
N_2	0.074	0.075
CH_4	0.216	0.185
C_2H_6	0.525	0.432
C_3H_8	0.801	0.540

Catchpot Butt m^3/te Anhydrous Catchpot Butts/atm

Test Periods	1	2-5
Sp. Gr. Oil Temperature	0.999/15° 40°	0.991/15° 40°
Solubility: H_2	0.060	0.059
CO	0.104	0.090
N_2	0.059	0.067
CH_4	0.184	0.180
C_2H_6	0.607	0.567
C_3H_8	1.18	1.02

Hold m^3/te HOLD Oil/atm

Test Periods	1	2-5
Sp. Gr. Oil Temperature[2]	1.096/100° 430°	1.112/100° 430°
Solubility: H_2	0.199	0.135
CO	0.246	0.121
N_2	0.170	0.127
CH_4	0.296	0.170
C_2H_6	0.459	0.239
C_3H_8	0.718	0.309

1) Intake Temp. of oil in scrubber
2) HOLD Intake

Table 33

Proportion of CO_2 and NH_3

Off Gas and Off Water 1)

(In Wt. % of processed AMF coal)

Test Period		1	2	3	4	5
CO_2	Off Gas	0.83	0.77	0.90	1.20	1.59
	Off Water	0.92	0.77	0.65	0.68	0.71
	Total	1.75	1.54	1.55	1.88	2.30
H_2S	Off Gas	0.06	0.06	0.08	0.12	0.10
	Off Water	0.41	0.29	0.37	0.31	0.26
	Total	0.47	0.35	0.45	0.43	0.36
NH_3	Off Gas	0.06	0.10	0.06	0.11	0.08
	Off Water	0.75	0.63	0.64	0.62	0.63
	Total	0.81	0.73	0.70	0.73	0.71

---

1) Distillation and Kiln Off Gases were not considered.

/pkl

Table 34

Content of Organically Bonded Sulfur in the Gases

mg S/m^3 Gas

Gas	Period			
	1	2	3	4
Letdown I	19.4	8.3	9.9	
Letdown II	11.5	8.3	20.8	
Washer Letdown I	15.8	7.5	6.5	8.6
Washer Letdown II	11.9	17.8	25.1	10.3
HOLD Gas	7.8	4.7	5.7	

/pkl

Table 35

H_2 Consumption

Test Period	1	2	3
m^3 consumed / To MAF Coal	1190	1020	983
of which % chemically	66.0	69.1	70.7
% solution	27.5	28.0	26.0
% lost	6.5	2.9	3.3

/gkl

BUREAU OF MINES

T.O.M. Reel # 129 T-53
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ABSTRACT OF REPORT ON TECHNICAL EXPERIENCES
IN THE OPERATION OF A CONVERTER STALL
IN THE SECOND HALF OF YEAR, 1936

Report By -- Dipl. Ing. Amon

Dated L.-W., 19 January 1937

1 - LEAKING OF HIGH PRESSURE PIPE LINES

There are many causes of leaks in flange connections of high pressure lines. In spite of this, leaks have been rare in hydrogenation in large size high temperature lines. For example, lines of 160 mm I.D. or finned tubes of 120 mm I.D. in the gas preheaters, have been laid with safety. This success is not attributable only to careful handling and inspection of the sealing surfaces of flanges as well as extreme care in installation, but is mainly due to proper laying of lines in regards to anchors, expansion and initial tension. Improperly laid lines may be assembled with the greatest care, yet will leak in temperature fluctuations and cause operating interruptions.

To keep step with the development of pipe lines the following three (3) rules should be observed:

a) Fitting in a length of sprung pipe

b) Fitting in one or more right angle bends

c) Allowing for initial tension

In the operation of converter stalls, it has been shown that for lines of an I.D. of 120 - 160 mm the sprung length should not be less than three meters. If this length cannot be held it is advisable to use articulated joints of elbows or bends. To lessen the possibility of buckling of the sprung pipe, allowing for initial tension in the laying of hot lines has proved very successful. The initial tension should be taken as one half the expansion in any straight run of pipe.

A further cause of hot flange connections are the temperature fluctuations in the products passing through the lines. If the bolts no longer have the necessary initial tension, due to expansion of the lines, leaks will occur. It is, therefore, of importance to keep the bolts cold wherever possible in order to avoid lowering the initial tension given the bolts when they were installed. Therefore, no insulation should be applied to any hot flange connections.

A further insurance against leaks caused by temperature fluctuations is offered by the use of bellows sealing rings. Contrary to the normal sealing ring, the bellows sealing ring seals through elastic change of form and will spring enough to assure tightness under extreme temperature fluctuations. This type of flange has been successfully used on lines of 45 - 160 mm I.D. under maximum temperature changes in a stall. However, the bellows seal is somewhat higher in cost than the normal seal, but its advantages are so great that we intend to use it in the future for all hot lines. (See Sketches # 1 and # 2)

(over)

To avoid bending moments in the flange connections, so-called flange props were recommended for use with the bellows seal. This prop requires considerable skill in installation and this type of flange protection has not been proved in practice.

2 -- PRESSURE RELIEF VALVES WITH HARD METAL INSERTS

(See Sketch # 3)

The HOLD is purged in two (2) steps: First, from 200 to about 100 at. and second, from 100 - 0 at. The most favorable nozzle diameter has been shown in practice to be 10 mm., with a HOLD volume of four to six thousand liters per hour. In the purging, the first step is regulated, while in step two, the pressure relief, or purge, valve remains fully open. As a general rule, as little as possible regulation should be done in order to increase the life of the nozzle. For this reason an automatic regulating valve is now being developed by means of which the valves can be much more uniformly regulated than when operated by hand.

3 -- HOT GAS FANS

A sensitive part of our gas heated preheater is the fan, which must circulate flue gas at a temperature of 400 - 500°C. Various bearing difficulties have been solved by the use of water-cooled self-aligning roller bearings and the use of a suitable lubricant. (A part of this report is missing here, but it seems to deal with difficulties in fan operation.) One of these difficulties seems to have been the loosening of the fan wheel due to high operating temperatures. In one case this was caused by the shaft key being loosened by vibration. In the future both keys will be welded together in the middle and provided with lugs on the ends. The welds must be made with dispatch in order to prevent distortion of the keys and their being welded to the shaft. (Illustration showing this is missing).

On December 25, 1936 the fan in stall #6 exploded, so that the wheel and housing were completely destroyed. The fan had been satisfactorily operating for about two days and suddenly burst without any warning. The cause of this was determined to be the breaking of a welding seam on the wheel side plate on the suction side. We have, therefore, adopted these regulations:

1.) If possible, avoid all welds on the wheels and rivet them instead,

2.) If welding cannot be avoided, the welds should be x-rayed before the fan is put into operation,

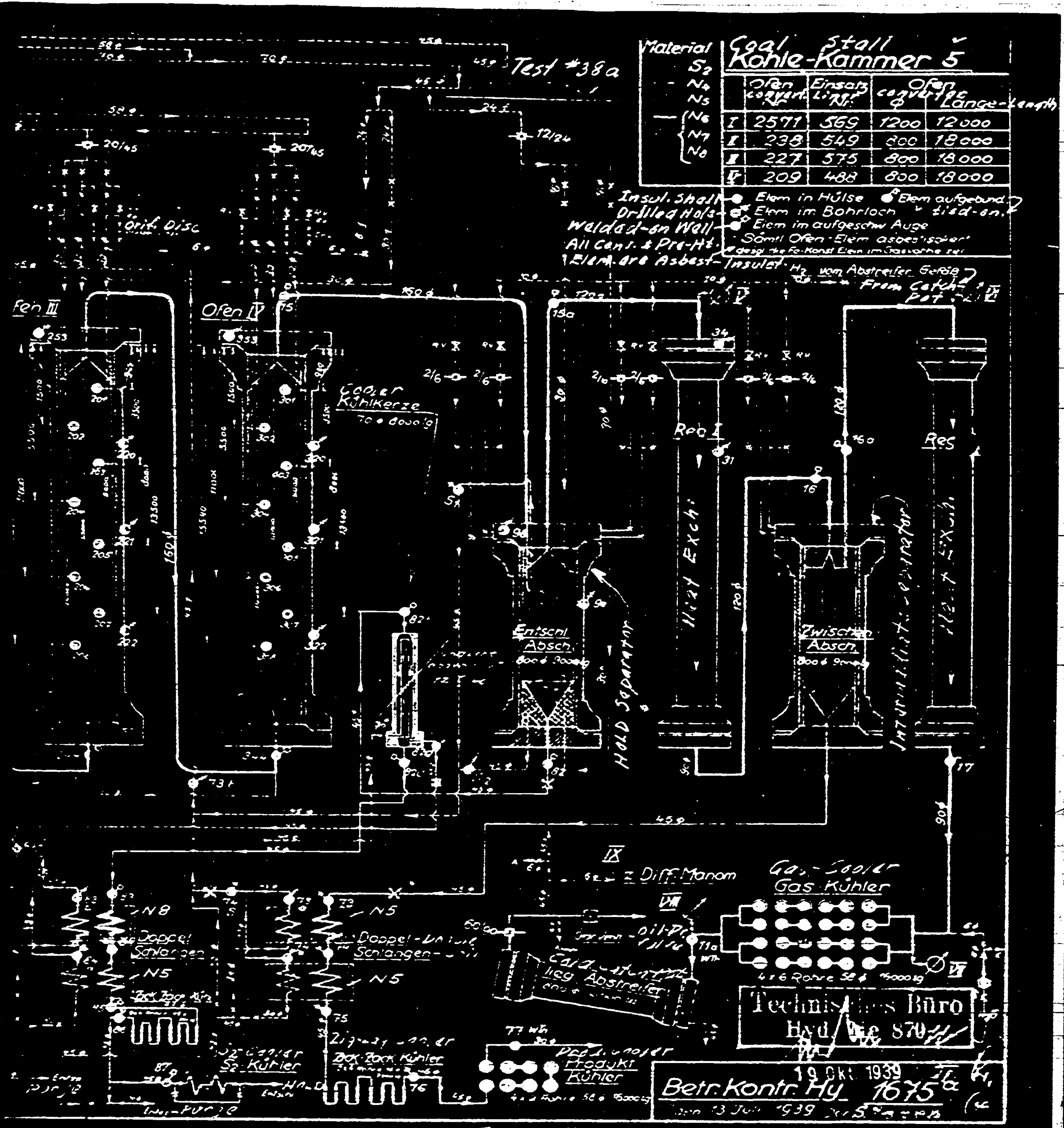
3.) The welds should be protected by cover rings or splice plates.

Furthermore, in expectation an eventual lowered efficiency, carefully balanced wheels should be chosen, in which any stresses caused by unsteady running are taken by spiders on the suction side instead of the blades. (Sketch #4 shows the construction of the old and new wheels.)

#4 --

Sketches # 5 and #6 are not discussed in this report. However, they seem to indicate a diagrammatic arrangement of pipe lines and equipment and the location of thermo elements for a certain test (#38A of Coal Stall #5.)

KCBraun/mc/pkl



Material	Coal Stall Kohle-Kammer 5			
S_2				
- N_4				
N_5				
— N_6 N_7 N_8				

	Ofen convert. Nr.	Einsatz Liner Nr.	Ofen converter ø	Länge-Length
I	2571	569	1200	12000
II	238	549	800	18000
III	227	575	800	18000
IV	209	488	800	18000

● Elem in Hülse
● Elem im Bohrloch
● Elem im aufgeschw Auge
● Elem aufgebund.
" " lösd-en.
Sämtl Ofen-Elem asbestisiert
desgl. die Fe-Konst Elem im Gaswege sei

Technisches Büro
Hyd/Lie 870

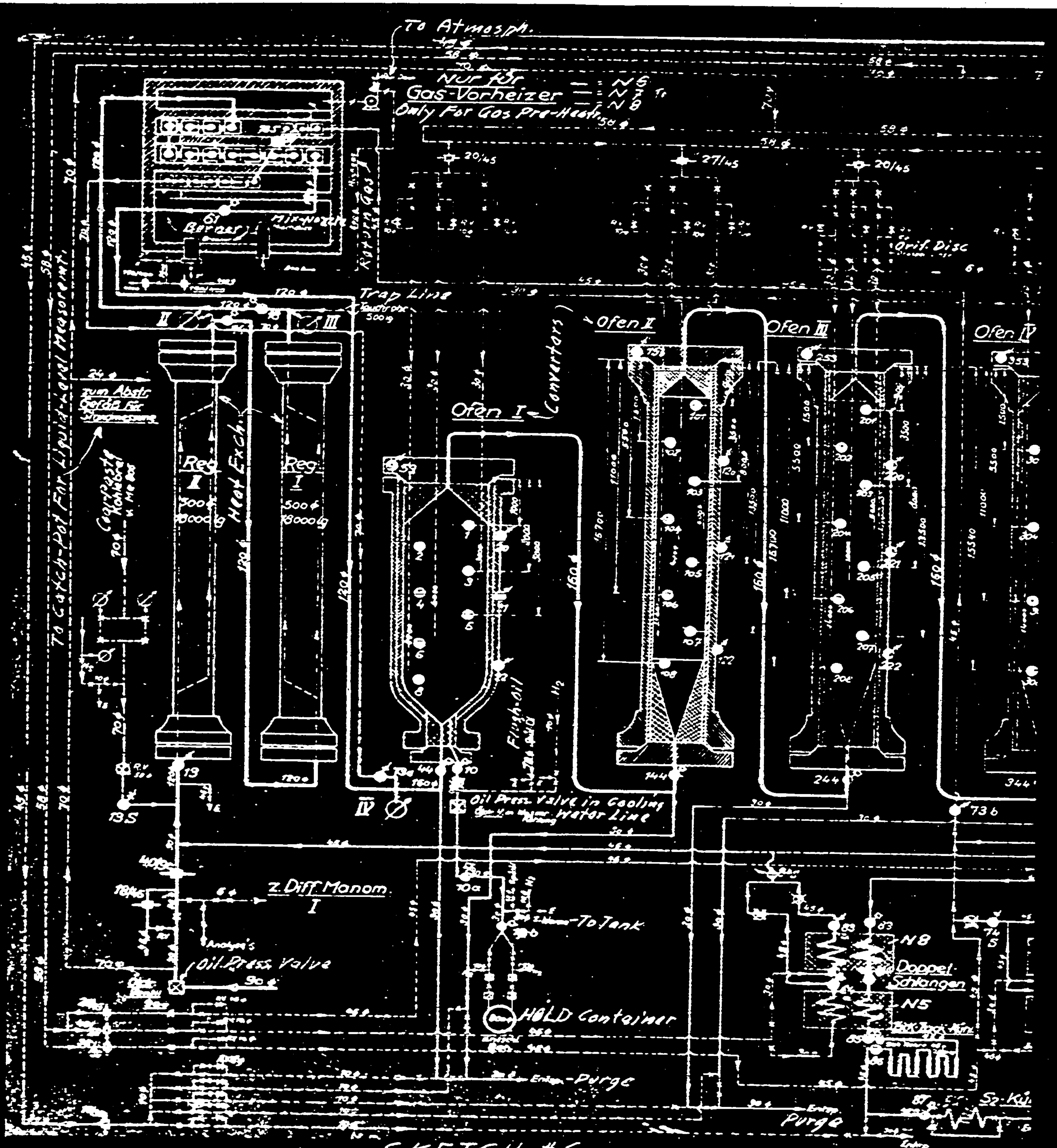
19 Okt. 1939

Betr. Kontr. Hy 1675a

den 13 Juli 1939

SKETCH #5.

T.O.M. Reel No. 123



SKETCH #6.

T-53

T. O. M. REEL NO. 129

T.O.M. Reel No. 182 (Also Reel 130, pp. 340-348) T-54
pp. 949-958

October 13, 1941

LARGE PILOT PLANT AT LUDWIGSHAFEN: THE RESULTS FOUND ON DISMANTLING THE HIGH PRESSURE STALL AFTER THE LARGE SCALE FUEL OIL TESTS, AUGUST - OCTOBER, 1941 (See T-52)

SUMMARY.

The original dismantling of the stall was limited to the parts of particular interest in designing a production unit, and was primarily concerned with the finned preheater and heat exchanger. The complete dismantling was postponed because of the intention to return the stall as rapidly as possible to large scale tests on the production of electrode coal from the hydrogenated coal.

1. Heat Exchange:

In addition to the testing of the production method for fuel oil, the large scale test was intended to bring information on the coal paste heat exchange. The dismantling of the 3 heat exchangers showed that both in the forward and in the return pass no deposits were present as was to be expected with a good K value which remained at around 300 throughout the whole test.

2. Finned Preheaters:

The preheater was first run for 24 days with coke-oven tar and then after a long rest for 67 days with coal. The hairpins 11 and 12 have only been installed during operations with coal.

The inclosures 2 and 3 show that incrustation of the hairpins was normal in the cold and the hot passes (1.2 mm), while the deposits in the middle pass were relatively large considering the short operating time.

The reasons for it are as follows:

1. The rate of flow in the middle pass was very small at the ordinary operation procedure (0.7 m/sec.).
2. As a result of using 2 partial streams the concentration of paste was higher in the middle pass (52%).
3. The heating in the middle pass was to 420° and therefore contained the whole critical range between 300 and 400°C in which according to our previous experience the coal swells and the heat transfer is particularly poor because of the great viscosity changes of the paste.
4. In the process for fuel oil the pasting of coal was done with 30% middle oil in the vehicle oil and we had to deal with a larger amount of volatilization of the oil constituent.

The relative importance of the above causes cannot as yet be definitely established; it seems however that the rate of flow was the most important.

It must be mentioned in this connection that with the same wall tube temperature (385°C) the crust in the intermediate piping between heat exchanger to preheater ($v \cong 1.3$ m/sec.) was 0.8 mm against 1.4 mm in the hairpin 6 of the middle pass ($v \cong 0.7$ m/sec.). The wall tube temperature is higher in the hot pass, but the crust is lower than in the last hairpins of the intermediate pass.

The very great narrowing down (up to 80%) in the horizontal part of the intermediate pipe between the middle and the hot passes is very probably to be explained by too low a rate of flow and by the strong pulsations, especially so in the intermediate pass. The same applies to the connecting pipe line between the preheater and converter 1 where there was a back flow of the thick converter contents because of too low a rate of flow, which has resulted in large depositions. These individual observations and measurements inside of the preheater show that in processes with different concentrations of paste and by the parallel connections necessitated by them in the preheater, the velocity in the preheater part in which the paste is heated directly, must be at least 5 - 6 m/sec., and the large temperature interval must be bridged over by mixing with coal paste coming from the heat exchange. Direct heating should be carried on at most to 320 - 330°, because the incrustation becomes greater at higher temperatures.

We may mention in conclusion that the plugging up in the lower bend of the hairpins observed formerly in the large scale tests at 300 atm., has not been present in the 700 atm. tests. The hairpin studied was made of N10 material 34/70 with a normal return bend.

3. Hot Catchpot:

The hot catchpot was discharged into a funnel in the hot circuit. The inner part of the catchpot has been found upon dismantling to be entirely clean, nor had any coke deposits been formed in the funnel.

Further details on the results of dismantling of the stall will be found in the following detailed report of dismantling.

/S/ Raichle

RESULTS OF DISMANTLING OF THE RIBBED PREHEATER

	Hair-pin Number	i.d. mm	Return of flow m/sec.	Concentration of paste %	Average tube wall temperature °C	Velocity of circulating gas m/sec.	Thickness of crust mm	Days of Operation	
								With coal	With coke oven tar
	1	45			260		—		
COLD PASS	2	"	1.12	41 (Fresh coal 34%)	295	96	—	67	24
	3	"			328		—		
	4	45			346		0.7		
	5	"			364		0.5		
INTER-MEDIATE PASS	6	"			382		1.4		
	7	"	0.70	51.6 (Fresh coal 45%)	400	10.30	20 -2.25	67	24
	8	"			418		2.25		
	9	"			436		3.15		
	10	"			455		4.0		
	11	34			465		3.9 - 3.8	67	—
	12	"			469		2.2	67	—
	13	"			473	9.25	2.6	67	24
HOT PASS	14	"	4.00	44.7 (Fresh coal 38%)	477		3.0	67	24
	15	"			481		3.0	67	24
	Descending tube	"			484		3.0-4	67	24

Circulating Gas: 12,500 m³/h (15°/735)

Sternberg/MC/fkp

T.O.M. Reel No. 182 (also Reel 130, pp. 349-372)
pp. 959-967

T-55

October 5, 1941

LARGE PILOT PLANT AT LUDWIGSHAFEN: DISMANTLING RECORD OF STALL 804 (See T-54)

I. Finned Preheater:

1. The intake piping a has not been dismantled.

2. Intake b, pass 1, south side.

a. Tubular piping i.d. 45 mm.
Temperature: Thermocouple 61: 4.1 - 4.4 mv. *
Results: The volume measured was 655 cm^3, corresponding to an average i.d. of 43.5 mm, and the deposit about 0.7 mm. In general clean and dry.

b. First hairpin in pass 1, descending tube, i.d. 45 mm.
Temperature: Thermocouple 61: 4.1 - 4.4 mv.
Results: Slightly narrowed down, crust less than 1 mm thick.

c. First hairpin in pass 1, ascending tube, no temperature measurements.
Results: Same as under b.

d. The connection from pass 1 to pass 2 has not been opened.

e. First hairpin in pass 2, ascending tube.
Temperature at the thermocouple 61 at the preceding descending tube, about 17.7 mv.
Results: Deposit about 1 mm thick.

f. First return bend in pass 2 between first and second hairpin.
Results: Volume measured 825 cm^3, corresponding to an average clear diameter of 43 mm; deposit therefore 1 mm.

g. Second hairpin in pass 2, descending tube, no temperature measurements.
Results: Deposit about 1 mm thick.
At a depth of 1,000 mm; 1.3 - 1.4 mm.

h. Third hairpin in pass 2, ascending tube. No temperature measurements.
Results: Deposits about 1.25 mm.
At a depth of 1,000 mm; 2.0 mm.

i. Return bend between third and fourth hairpin. No temperature measurements.
Results: Volume measured: 805 cm^3, corresponding to an average clear diameter of 42.5 mm; deposits therefore 1.3 mm.

---

* The conversion of millivolts to degrees centigrade is represented in the appendix; the cold junction is assumed to be at 40°C.

k. Fourth hairpin in pass 2, descending tube. No temperature measurements.
Results: Deposit on top 1.5 mm, at 1,000 mm depth 2.25 mm.

l. Return bend in the last hairpin in pass 2.
Results: Volume measured: 770 cm^3, corresponding to an average clear width of 41.6 mm; deposit 1.7 mm.

m. The last ascending tube in pass 2 to the connecting elbow to pass 3.
Temperature: Thermocouple 61; v = 24.7 - 25.2 mv.
Results: Deposit on top 3 mm, at 1,000 mm depth: 4 mm. The thickness of the deposit on the way from the first to the last hairpin increases therefore from 0.5 mm at 17.7 mv to 4 mm at 25 mv. The thickness increases rather uniformly.

n. Connection between pass 2 and pass 3, i.d. 45 mm.
Temperature: Thermocouple 61a, 22 - 22.8 mv.
Results: The horizontal pass has been narrowed down 80%. The deposit consists mostly of a soft granular mass which, peculiarly, fills the upper part of the whole length of the tube. The remaining cross section is surrounded by a hard crust about 3 mm thick. In the 2 vertical parts of the connecting pass the above hard crust had formed on the wall tube which resulted in a reduction of the clear width by about 8 mm.

3. Union of 35 and 65 T (the numbers refer to drawings):

a. Pipe line (65 T) from heat exchanger I to the finned preheater; i.d. 45.
Temperature: Thermocouple 62, 19.5 - 20.2 mv.
Results: Reduction of the clear width by 1.7 mm through solid deposit formation; in addition a slight contamination with the soft mass.

b. The T piece (union of 35 and 65 T).
Temperature: (35 T) Thermocouple 61a, 22 - 22.8 mv.
" : (65 T) " 62, 19.5 - 20.2 mv.
Temperature (after joining): Thermocouple 62a, 20.7 - 21.2 mv.
Results: Hard deposits about 4 mm thick in the vertical part corresponding to the vertical part of the connection piece. In the horizontal part (65 T) a slight deposit about 0.7 mm thick.

c. First hairpin pass 3 (descending tube) i.d. 34 mm.
Temperature: Thermocouple 62, 20.7 - 21.2 mv.
Thermocouple 62 n, 23.7 - 24.5 mv.
Results: Reduction in clear width,

at the beginning	1.7 mm
at a depth of 300 mm	2.7 mm
at a depth of 500 mm	3.8 mm

No measurement of clear diameter was possible at greater depth because of a slight bending of the tube.

d. First hairpin pass 3 (ascending tube) i.d. 34 mm.
No temperature measurements.
Results: Reduction of clear width: on top 0.6 mm, at a depth of 300 mm, 1.8 mm, at a depth of 1,000 mm, 3.9 mm.

e. Return bend between first and second hairpins.
Temperature at the nearest couple at the adjoining descending tube, thermocouple 62 v, 24.2 - 25.2 mv.
Results: Slight reduction in the clear width: 0.2 mm at the inlet 0.8 mm at the outlet.
Volume measured 385 cm^3, corresponding to an average clear width of 31.2 mm; deposit 1.4 mm.

f. Second hairpin in pass 3 (descending tube) i.d. 34 mm.
Temperature: Thermocouple 62 v: 24.2 - 25.2 mv.
Results: Reduction in clear diameter: on top 1 mm, at a depth of 300 mm 2.2 mm. Measurements at greater depth impossible because the tube had bent somewhat.

g. Hairpin penultimate return bend in pass 3, i.d. 34 mm.
No temperature measurements.
Results: Volume measured 360 cm^3, corresponding to an average clear width of 30.1 mm; deposit therefore about 2.0 mm thick.

h. Last return bend in pass 3, i.d. 34 mm, no temperature measurements.
Results: Volume measured 345 cm^3, corresponding to an average clear diameter of 29.5 mm; the deposit therefore about 2.2 mm thick.

i. Last hairpin in pass 3.
Temperature: Nearest thermocouple 62 v in the outlet descending tube: 24.2 - 25.2 mv. The hairpin was removed and the thickness of the wall of the bottom return bend was measured by radiation with mesothorium. The measurements are shown in the report for results - see the report (figure 18).
It was intended to X-ray the weld but it could not be done because of the overloading of the materials testing laboratory. The results will be recorded later.

k. Last return bend in pass 3.
Temperature: Thermocouple 62 v: 24.2 - 25.2 mv. Volume found: 355 cm^3, corresponding to an average clear width of 30 mm. The deposit 2 mm thick.

l. Outlet descending tube, pass 3, north side.
Temperature: Thermocouple 62 v: 24.2 - 25.2 and thermocouple 63: 25.1 - 23.6 mv.
Results: Hard deposits in the upper end 2 - 3 mm thick. At the bottom end 3 - 4 mm thick.

II. Pipe Line to Converter I:

1. Reducer, i.d. 45/34.
Temperature: Thermocouple 63: 23.1 - 23.6 mv.
Found: In addition to the same deposit as in the descending tube, the clear width reduced by 10 - 12 mm through formation of a soft, readily removable mass.

2. Elbow, i.d. 45 mm.
Temperature: Thermocouple 144: 23 - 24 mv.
Found: Narrowed down by a soft crust 5 mm thick.
Volume 285 cm^3, corresponding to an average clear diameter of 35.4 mm.

3. Nipple, 45 mm i.d., length 635 mm, horizontal.
Temperature: Thermocouple 144: 23 - 24 mv.
Found: Principally white crust about 5 mm thick.
Volume: 625 cm^3, calculated average clear width of 35.5 mm.
The above deposit filled the upper part of the opening, as in the connecting piece from pass 2 to pass 3 of the preheater.

4. Horizontal elbow, i.d. 45 mm.
Temperature: Thermocouple 144: 23 - 24 mv.
Found: Volume 250 cm^3 or an average clear width of about 33 mm; deposit therefore 6 mm.

5. Horizontal pipe, i.d. 45 mm, length 3,320 mm.
Temperature: Thermocouple 144: 23 - 24 mv.
Found: Strongly contaminated; the deposit is mostly soft, except for a few mm of hard crust.
Volume 2,710 cm^3 or average clear width 32.3 mm; deposit 6.3 mm.

6. The balance of the pipe line to converter I, consisting of T piece i.d. 45/24. Nipple (length = 450 mm), elbow (i.d. 45).
Temperature: Thermocouple 144: 23 - 24 mv.
Found: Horizontal part strongly contaminated; crust about 10 mm thick. The cold gas inlet at the T pass (i.d. 24) completely clean.
Volume, except for the 24 mm leg of the T piece: 485 - 57.5 = 427.5 cm^3, corresponding to an average clear width of about 24.3 mm.
As in all horizontal parts of the leaders to converter I, the deposit filled chiefly the upper part of the tube. It is remarkable that as indicated by measurements of volume, the thickness of deposit increases with increasing distance from the finned preheater.

III. Converter I has not as yet been opened:

IV. Pipe line from converter I to converter II:
Temperature: Thermocouple 244: 21.5 mv.
Found: Dry and clean.

V. Converter II, 330 mm i.d.; (12,000 mm long): The volume of the converter was measured by filling with oil.
Found: 871 li. The drawing indicates a volume of 904.5 li. net. There must accordingly be present some 25 li. of coke in the converter, but it is to be remembered that the calculated value of the volume is somewhat uncertain because of the unknown variations in measurements. As a result the amount of coke may differ from the above amount by a certain small quantity. It can however not be large enough to necessitate the dismantling and opening of the converter. Moreover some coke had been removed at the outlet opening.

VI. Pipe line from converter II to neutralizing converter:
Temperature: Thermocouple 344: 24.6 - 24.8 mv
Thermocouple 344a: 22.9 - 23.2 mv
Found: Dry and clean.

VII. Pipe line from the neutralizing converter to the hot catchpot:
Temperature: Thermocouple 20: 22.4 - 22.7 mv
Thermocouple 22a: 22.3 - 22.5 mv
Found: No solid deposits; oily and wet.

VIII. Pipe line from hot catchpot to heat exchanger:
Temperature: Thermocouple 20b: 22.0 - 22.1 mv
Thermocouple 17: 22.0 - 22.1 mv
Observations as under VII.

IX. Hot Catchpot: The hot catchpot was dismantled and opened. It was entirely clean nor was there a trace of coke deposits in the funnel.
The vertical sight glass indicated no increase in the resistance to flow against earlier measurements.

X. Heat exchangers:

1. Exchanger 1:

a. Forward pass, temperature: Thermocouple 13d: 18.4 - 18.5 mv
Thermocouple 14: 21.1 - 21.2 mv

b. Return pass, temperature: Thermocouple 17: 22 mv. The heat exchanger was dismantled and the tests for thru-put indicated no increase in flow resistance in either pass. The bundle of tubes was removed from the casing and the shell surrounding the bundle was removed.

Found: No deposits inside or outside the bundle of tubes. The volume measurements of the bundle of tubes gave 14.42 li. against the calculated value from the drawing of 14.26 li.

2. Heat exchanger 2:

a. Forward pass, temperature: Thermocouple 13b: 16.6 - 16.9 mv
Thermocouple 13c: 19.7 - 19.8 mv
Thermocouple 16c: 19.7 - 20.8 mv

b. Return pass, temperature: Thermocouple 16b: 18.0 - 18.1 mv

The heat exchanger 2 has also been dismantled. The flow resistance remained unchanged. The sheet metal shell surrounding the bundle of tubes was cut in one place and bent open.
Found: As in heat exchanger 1.

3. Heat exchanger 3. The dismantling of heat exchanger 3 was at first thought unnecessary because of the good results found with the two heat exchangers 1 and 2. Measurements of resistance to flow inside and outside the bundles of tubes showed no increased values in comparison with those obtained previously.

The measurement of the volume of the bundle of tubes gave 31 li. against 28.56 li. found from the drawing. One might draw the conclusion that heat exchanger 3 was free from contamination in both the forward and the return passes, but it nevertheless is going to be dismantled and opened up to get a complete picture. The report on the results found with heat exchanger III will come later.

XI. Air Cooler:

The resistance to flow of the two air coolers is practically unchanged against earlier values.

XII. Stage Cooler:

The western half of the stage cooler showed unchanged flow resistance, the resistance of the eastern half was 20 to 30% higher than formerly. One may expect that the former value will again be reached by flushing and steaming out.

XIII. The outlet of the feed catchpot, the gas scrubber, the water scrubber up to outlet into the air:

The flow resistance is 60 to 80% higher than at the former measurement. Presumably here again the resistance would be reduced by steaming out.

/S/ Hamacher

Sternberg/MC/fkp

7-23-1946

T-56

T.O.M. Reel No. 76
pp. 32-35

REPORT ON A ROUND TABLE DISCUSSION OF EXPERIENCES IN PREHEATER OPERATIONS, HELD AT LUDWIGSHAFEN, MARCH 22-23, 1943

Stettin-Pölitz, Apr. 1, 1943

The following points were discussed:

1. Measures taken to decrease the load on the preheaters particularly on the preheaters of the coal stalls in Blechhammer. These are smaller and therefore must be run at higher temperatures than, for instance, Pölitz. They also have a larger bore and consequently a thinner wall thickness. These effective measures for preheater load reduction were determined:

1.) To run a higher concentrated thin paste, since this will require less thick paste (Pölits runs a 40% concentration).

2.) To run a higher concentrated thick paste (Pölitz runs a 53% concentration).

3.) Gas heat-exchange.

4.) Injection of cold paste into Converter II. A volume of approximately 6 m3 of cold paste reduces the preheater temperature by about 20°C. By preheater temperature is meant the hairpin tube temperature and not the product temperature. Gelsenberg at the present time runs 4 tons of cold paste, making it necessary to measure the volume.

5.) The installation of a 5th converter as a pre-converter. The preheater is loaded only so that element 144 is held to 19 - 19.5 mv. Converter I is then put under draft. This measure reduces the preheater temperature by 50° C.

For comparison Leuna provides 80 to 90% of the necessary heat by heat exchange, Wesseling 65 to 70%, Welheim same, Gelsenberg 35%, Pölitz 55%, and Blechhammer tentatively 52%. Gelsenberg already runs a quadruple stall with the first converter as a preheat-converter, element 144 equals 19.5 mv and reaches reaction temperature only at the end of the first converter.

We were also informed that Scholven uses thinning oil in the hot catchpot. It is said that this has proved very successful in the centrifuge operation because the asphalts are said to be less harmful. They are more easily centrifuged and distilled. It is assumed that at high temperatures the oil aids the dissolution of the asphalts. This oil injection is properly made in the connecting line as provided in our #12 stall. In Pölitz they propose to start with catchpot heavy oil using about 2 m3/h.

2. Crust Formation. Dr. Rank reported on his investigation of crusts at Pölitz and Gelsenberg. He told us that chromium, molybdenum and vanadium were found in the samples at Gelsenberg. Pölitz could not confirm these findings. They even found more in the vapor stage than in the coal stage. Therefore Gelsenberg seems to have run at higher temperatures than Pölitz. Dr. Rank has investigated the relationship between iron and titanium and concluded that in Gelsenberg the following relations exist:

Ash	to 1 part titanium	51 parts of iron
Bayer-mass	" " " "	8.1 " " "
Iron sulfate	" " " "	90.0 " " ", and on the
investigated crusts on the tube	to 1 part titanium	66.0 parts of iron

His conclusion that, because of similar relations of iron and titanium in the crust and in the ash the crust formation is related to the coal ash, did not find general approval. On the contrary, it is believed that the preheater attack is not attributable so much to the ash as to the catalysts which we add ahead of the preheater. Dr. Pier and Dr. Klinkhardt were especially of the opinion that besides sodium sulfide (for the prevention of chlorine corrosion) the catalysts should not be added directly to the coal, but that the Bayer-mass and the iron sulfate be added in the first converter.

3. Preheater Tube Materials. Chief Engineer Koch reported on the latest developments in high pressure materials. Experience has shown that the ability to deform decreases with time, particularly of the N10 tubes, that is, the material becomes brittle in the course of a longer operating period. The factors bringing this about are time, hydrogen, temperature, pressure, eventual crust formation and nitrogen (by nitration). Which of these components is the most important has not been determined. In any case, however, the presence of hydrogen is required. The most important of these factors are temperature and pressure. According to investigations to this date the materials investigated possess an operating life of 2 years. After that it is necessary to heat-treat the tubes in order to put them again into a normal condition. It has been shown that a greater hardness is better, because the harder material is less readily influenced. In order to give a picture of the nature of these influences he states, that a temperature reduction of 10° and a pressure reduction of 10% would mean a doubling of its life. He indicates a limiting temperature of 520°C for the inner wall and of 530 to 540°C for the outer wall. There are various measures now under way to better the materials. For example, it is proposed to decrease the carbon content and increase the vanadium content. However there are certain dangers connected with this because the fatigue strength is reduced thereby. On the other hand, the hydrogen attack is reduced. The reduction of temperature and pressure also favorably affects cold hairpin tubes, according to Mr. Koch. The foregoing therefore points to the necessity of adopting the measures outlined in paragraph 1 preceding, particularly of reducing the temperature of the preheater by means of thin paste, cold paste, etc.. At Blechhammer the wall thickness of the preheater tubes is to be increased.

4. Paste Press Check Valves. It was determined that pressure and suction valves leaked, as a general rule, when flushed with oil. It is proposed to install a check valve behind each manometer. With the exception of Welheim no other plant has valves on the presses.

5. The cutting of middle oil for prehydrogenation. This point was particularly discussed in connection with the deterioration of 5058 converters in Pölitz.

Scholven has 2 prehydrogenation stalls with the following data:

1. Triple-prehydrogenation stall:

Converter 1, Catalyst 5058, Temperature 19-21 mv, Catalyst 345 days old
Converter 2, Catalyst 5058, Temperature 21 mv, Catalyst 127 days old
Converter 3, Catalyst 7846, Temperature 21.5 mv, Catalyst 100 days old

This chamber runs with a thru-put of 16 tons/h., 35,000 m3 inlet gas and 55,000 m3 (?) outlet gas. The aniline point of the middle oil rises from -12° C in the injection product to 45 -46°C in the catchpot.

2. Triple-prehydrogenation chamber:

Converter 1, Catalyst 5058, Temperature 19-21 mv, Catalyst 100 days old (?)
Converter 2, Catalyst 5058, Temperature 21-21.2 mv, Catalyst 356 days old
Converter 3, Catalyst 7846, Temperature 22.2 mv, Catalyst 356 days old

Both stalls run with an injection product of 350°C end point. The 95% point could not be determined. No deterioration of the catalysts has been shown in either of these stalls with this injection product. The 6434 stalls also operate at low temperatures and give good results. Scholven believes, however, that the basic octane number has deteriorated since the introduction of catalyst 7846. By using lead, the gasoline may, of course, be brought to the same octane number as before, but it has a poorer overload curve.

To this might be added, that while 7846 gasoline has fewer aromatics, it has 1 or 2 more naphtenes. But because less gasoline is made with catalyst 7846 than in the prehydrogenation with catalyst 5058, a possible octane deterioration would have to be equalized by a greater proportion of 6434 gasoline.

Scholven also informs us, that, in order to improve the doctor test, 1/6 of the last 6434 converter is filled with 7846 catalyst in order to avoid difficulties with the gasoline.

Gelsenberg has 3 prehydrogenation stalls, all of which are combination stalls.

Stall 303 has the following data:

Converter 1, Catalyst 5058, Av.Temperature 20.8 mv, Catalyst 394 days old
(Inlet Temperature) 19.5
Converter 2, Catalyst 7846, Av.Temperature 22.2 mv, Catalyst 149 days old
Converter 3, Catalyst 7846, Av.Temperature 22.4 mv, Catalyst 149 days old

Operations were conducted under the following conditions:

Thru-put 0.7, total gas 3,500 m3/ton injection, inlet gas 30,000 m3/h, aniline point-13°C. The injection product has the following data: 90% - 315°C; end point 335°C.

It has been decided that from now on the 95% point shall be uniformly introducted.

There are no doubts about the distillation, 1941 specifications are available. These specify that the 325° point is determined by the Engler-volume. By the way it was not generally known that there is a difference between Engler and ASTM.

Stall 306 has the following data:

Converter 1, Catalyst 5058, Av.Temperature 20.8 mv, Catalyst 420 days old
Converter 2, Catalyst 5058, Av.Temperature 21.5 mv, Catalyst 420 days old
Converter 3, Catalyst 7846, Av.Temperature 22.0 mv, Catalyst 114 days old

"B"-middle oil aniline point = 47 - 48°C, gasoline concentration in the catchpot at 155°C = 36%.

Chamber 302 has the following data:

Converter 1, Catalyst 5058, Av.Temperature 21.1 mv, Catalyst 792 days old
Converter 2, Catalyst 5058, Av.Temperature 21.8 mv, Catalyst 792 days old
Converter 3, Catalyst 7846, Av.Temperature 21.9 mv, Catalyst 40 days old

Aniline point of "B"-middle oil = 48 - 49°C, gasoline concentration in the catchpot at 155°C = 34%.

The injection product for the benzination over catalyst 6434 has a specific gravity of 0.848 at 20°, aniline point of 48.70, 0.03% phenol, and an end point of 305°C.

There seems to be no satisfactory explanation of the damages to the catalysts in the 5058 converters in Pölitz. The end point is high, but not so high that the catalyst should necessarily deteriorate because of it. It is thought that the damages are related to the distillation of the Brüx tars, possibly because phenols which have bypassed the liquid-phase, are introduced into the vapor-phase by the "A"-middle oil. Besides, the phenol content of 25% is very high. In this connection we are advised that Scholven will put an acid refining unit into operation in about 4 weeks, in which the 5058 injection product will be washed with 25% acid.

6. Sulfurization of prehydrogenation and benzination injection products. In Pölitz the sulfurization of prehydrogenation products is no longer done with hydrogen sulfide, but with granulated sulfur, because it was found that separations occur in the former method. The ash content of the injection product is, however, normal and equals about 2 - 4 mg/liter. The extraordinarily rapid drop of the K-values of the heat exchangers in the sulfurization with H_2S is appreciably slowed-up in the sulfurization with granulated sulfur.

The question arises here, if oxygen was contained in the hydrogen sulfide? In the opinion of Ludwigshafen this would be harmful.

The method of sulfurisation in Gelsenberg is at the present as follows:

5058 and 6434 are sulfurized with granulated sulfur, a sulfurization with hydrogen sulfide is being installed for both phases.

Scholven also sulfurizes the prehydrogenation injection product with granulated sulfur. It was intended to sulfurize with hydrogen sulfide but this method was dropped because of the experiences at Pölitz. No data are available on the sulfurization of 6434 injection products. In both plants the injection products are filtered after the sulfurization.

7. Operating times of coal stalls. In connection with the information on the operating times of the coal stalls at Pölitz, Dr. Urban informed us that Scholven has an average of 79%, equal to 289 production days, 5% for warming up and shutting down, and 16% for repairs.

8. Increased efficiency of kiln operations (Schwelerei). The effects determined by Dr. Horn and Dr. Hinz in our report on the tests for increasing the efficiency in the kiln operations were generally confirmed. The effects were reached but not in the same degree. Dr. Pross states that an increased addition of steam is more effective than an addition of sulfur.

9. The use of heavy oil on solid catalysts. Ludwigshafen used Zistersdorf petroleum and various other oils and found that such an operation would not do, if ash was present in the oil, because the ash would be deposited on the catalysts. If 0.001% ash was present no deposit was noticed, while at 0.1% ash a deposit was already noticed after 10 days. For TTH, 0.01% solids is considered permissible. An experiment to run crude oil without a catalyst resulted in coking up at Ludwigshafen.

In connection with the above experiments it may be said that it is necessary to put the oil first through a pressure distillation. This requires that a considerable proportion of the high boiling portion be split (weak cracking). These experiments were also partly made with the addition of crude oil. In Pölitz, however, it will be necessary to run the crude oil as before. (In spite of the negative results achieved so far in the mixing of pitch and crude oil, further mixing tests will be made.)

/S/ Wissel

KCBraun/MC/jw

7-25-1946

T.O.M. Reel No. 76
Bag # 2 and 3
Target 30/4.13
pp. 29 to 31 Incl.

An Abstract of a Report addressed to Hydrierwerke Pölitz dated Ludwigshafen/Rhein, 17 February 1943, on the strength of tube steel in 700 atmosphere preheaters.

The relationship between circulating gas temperature, tube wall temperature, and fatigue strength, as well as the dependence of tube wall temperature on the incrustation of the tubes in operation, has always been of the utmost importance in the construction of 700 at. gas preheaters.

While in 325 at. preheaters the circulating gas temperature could, in general, be limited to the temperature corresponding to the fatigue strength of the tube wall when fully incrusted, the conditions of the 700 at. stalls have forced us to set the maximum circulating gas temperature higher than the temperature corresponding to the fatigue strength of the incrusted tube wall because of the missing, or only partially possible, paste heat exchange, as well as the high thruput. In other words, to utilise to the utmost the fatigue strength values, even with a not fully incrusted preheater.

It has been generally agreed that the layout of the preheaters should be based on a maximum crust thickness of 10 mm. allowing a certain safety factor based on the fatigue strength. The maximum wall temperature of such an incrusted tube has been calculated to be 550°C. Later observation and investigation in materials testing have shown that the materials cannot properly be evaluated on the fatigue strength alone. Under the existing mechanical and chemical influences, at the high material temperature, the materials show a time dependence on the elastic strength of the materials.

It may be remarked here that this relationship has not been fully cleared up. In particular, the influence of the crust, or the lack of a protecting oil film because of the presence of this crust, is still to be investigated. Such laboratory investigations are now being made. Investigations are also being made to determine the factors responsible for the formation of this crust, with the object in view of minimising the crust formation. In any case, latest developments seem to indicate that increasing attention must be paid the crust thickness.

Tests so far made have shown that the maximum permissible inner wall temperature, to which N10 may be subjected to for a longer period at the full pressure of 700 ats., is for the time being limited to 520°C.

However, additional investigations must be made to determine if this temperature may be increased by proper heat treatment. It may already be said, however, that this temperature limit is strongly dependent on the mechanical stress of the tube wall, that is, on the operating pressure. If, for example, a stall is run at only 600 at. conditions become considerably more favorable. According to the latest tests, harder N10 tubes had a longer time-elastic-strength in hydrogen than softer tubes. If formerly soft N10 tubes were recommended, this was perfectly correct from general mechanical point of view. Soft N10 tubes have a greater toughness with still ample fatigue strength. As already mentioned, latest investigations have shown that the life duration of the tubes is not determined by the fatigue strength but by the time-elastic-strength and that this time-elastic-strength is insufficient for soft N10 tubes. Experience has further shown that no value need be placed on the greater toughness of the softer N10 tubes. It follows, therefore, that the tubes should be hardened to a greater degree by heat treatment. Since the wall temperature in the hot passes has exceeded 520°C, at least in some of our works, it is proper that all hair-pin tubes, which have been in service in the hot passes for about 10,000 hours, should be hardened by heat treatment. If such tubes are kept in service for a considerably longer period, loosening of the texture may be initiated, which we may not be able to correct by further heat treatment.

If in the future a wall temperature of 520°C on the inner surface is not exceeded in new installations, an after-treatment of the hairpin tubes after 10,000 hours should not be necessary. We believe that we can attain a similar condition in all old hair-pin tubes by means of the first after-treatment.

As already mentioned, a lowering of the circulating gas temperature is not possible without lowering the thru-put because an enlargement of the preheater would be necessary.

The little available data seems to indicate that the tube wall temperature of coal preheaters has risen to 520° after about a year. Based on calculations, this corresponds to a crust thickness of 5 mm. The preheater would, therefore, have to be shut down after about a year and the tubes cleaned of the crust. The operating time is naturally largely dependent on the product, on its tendency to crust formation, and on the operating method.

In summary, the following rules for the increase in life duration of hairpin tubes should be followed:

1) Hairpin tubes with the greatest possible Brinnell hardness should be installed at the hottest points.

2) All hairpin tubes, which have been in service for about 10,000 hours in the hot pass at 700 ats., should receive an after-heat-treatment.

3) If an inner tube-wall temperature of 520° is not exceeded, a further after-treatment should not be necessary on new tubes and tubes which have received one after-treatment.

4) It is highly desirable that formed incrustations be removed at the latest after one year's operating time.

5) At lower pressure, for example, 650 or even 550 ats., used by some works at the present time, observations so far made seem to indicate that an after-heat-treatment after 10,000 hours of operating time is not required on normally hard hairpin tubes.

I. G. FARBENINDUSTRIE A. G.

Signed - Pier.

KCBraun/pkl

COMPOSITION AND PROPERTIES OF GERMAN STEELS

T-58

Designation	Carbon	Manganese	Silicon	Chromium	Molybdenum	Vanadium	Tungsten	Titanium	Mechanical Properties			
	C	Mn.	Si.	Cr.	Mo.	V.	W.	Ti.	Tensile Strength lbs/sq. in.	Elastic Limit lbs/sq. in.	Elongation %	Impact Value M kg/cm²
K3CV	about 0.25	> 0.45	—	about 1.00	—	about 0.25	—	—	100-114,000	61-71,000	16	7
K4MS	0.40	1.40	1.40	—	—	—	—	—	114-128,000	71-85,000	14-16	about 6
K7	about 0.30	1.00	—	1.50	—	1.20	—	—	114-135,000	85,000	about 15	5-8
FF30	0.05	—	0.60	2.80	—	—	—	—	71-78,000	>50,000	> 20	
N5A	0.12	0.25-0.50	<0.40	2.50-2.80	—	>0.10	—	—	71-85,000	43,000	> 20	8-12
N8A	0.20-0.25	0.30-0.50	0.25-0.40	2.35-2.50	—	0.25	—	—	107-128,000	> 71,000	> 16	4-8
N9	0.19-0.24	0.25-0.50	—	2.50-2.80	0.20-0.30	0.55-0.65	—	—	114-135,000	> 78,000	> 14	5-8
N10	0.18-0.24	0.40	—	3.00-3.60	≥ 0.70	≥ 0.75	≥ 0.30	—	114-135,000	> 78,000	> 14	5-8
S1	0.30-0.35	0.50-0.90	< 0.40	—	—	—	—	—	71-85,000	> 38,000	> 22	6
S2	0.15-0.25	about 0.60	< 0.40	—	—	—	—	—	53-68,000		26-28	10-12
S3	about 0.20	about 0.90	< 0.60	about 0.50	—	—	—	—	71-85,000	43,000	24	8
HM1	0.12	0.30-0.50	1.50	1.40-1.70	0.20-0.25	—	—	0.10	64-85,000	> 38,000	> 22	> 10
	<0.10	about 0.50	about 1.50	1.40-1.70	0.20-0.25	0.25-0.35	—	0.40-0.60	64-85,000	> 38,000	> 22	> 10
L2	about 0.10	about 0.50	< 0.40	1.00-1.20	—	—	—	—	57-71,000	> 37,000	24	4-6
K1MS	0.35	1.00	1.00	—	—	—	—	—	100-121,000	71,000	16	about 6
K2M	0.23-0.28	1.20-1.50	0.40	—	—	—	—	—	78-100,000	50-57,000	18	5-8
K3	about 0.30	0.50-0.80	about 0.30	> 0.90	0.15-0.20	—	—	—	100-114,000	> 71,000	16-17	8-12
K1	0.27-0.32	0.70	0.35	0.60-0.80	—	—	—	—		> 68,000	70	
K5	0.20-0.30	0.50	—	1.00	1.00	—	—	—		≥ 85,000	≥ 85	
K5V	0.25	—	0.80	—	about 0.50	about 0.40	—	—		≥ 85,000	≥ 85	
K6	0.30-0.40	0.70	0.35	—	0.30-0.40	0.20	—	—		92,000	90	
N5	about 0.12	0.50-0.80	—	—	≥ 0.20	—	—	—		≥ 43,000	≥ 50	
N8	0.15-0.20	< 0.80	—	—	≥ 0.50	—	≥ 0.50	—		73,000	≥ 70	
N8V	0.18-0.23	< 0.50	—	—	≥ 0.50	0.40	—	—		77-100,000	75-88	
K2	0.25	< 0.50	—	1.00	0.20	—	—	—		≥ 50,000	≥ 50	

Composition and Properties of German Steels Ref: Micro-film Reel 87 Bag 3379 Item 124	Bureau of Mines Coal Hydro Demon Plt. Div. Louisiana, Mo.	Engineering Data Sheet No. 58

Translated	W.M.S.	Checked	c.c.
Date	5/16/46	Date	5/20/46

APPLICATION SURVEY OF MATERIALS
MAXIMUM PRESSURE 700 ATMS.

Ref. I.G. 700 Atms. Standards - Sheet 22, dated December, 1942

BUREAU OF MINES
COAL HYDRO. DEMON. PLANT DIVISION
LOUISIANA, MISSOURI

Trs. W.M.S.	Ck'd.	Date 5/16/46	Date 5/17/46

ENG. DATA SHEET NO. 69

T-58

TEMP. STEP	PRODUCT TEMP. °F	mm I.D.	SUITABLE MATERIALS FOR: TUBES	CASTINGS	BLIND FLANGES	THREADED FLANGES	BOLTS	NUTS	GASKETS
I	0 - 390	6 - 16 24 - 160 O.D. 135 <135	$\boxed{\text{K2M}}$ K2	$\boxed{\text{S3}}$	$\boxed{\text{S3}}$ $\boxed{\text{K4MS}}$ K1 K1MS	$\boxed{\text{S1}}$ $\boxed{\text{K4MS}}$ K1 K1MS	$\boxed{\text{S3}}$ $\boxed{\text{K4MS}}$ K1 K1CV $\boxed{\text{K4MS}}$ K6	$\boxed{\text{S3}}$ $\boxed{\text{S3}}$ K1 K1V K1CV	$\boxed{\text{S2}}$ $\boxed{\text{S3}}$
II	390 - 750	6 - 45 58 - 100 O.D. 135 135	$\boxed{\text{N8A}}$	$\boxed{\text{N8A}}$	$\boxed{\text{N8A}}$	$\boxed{\text{K4MS}}$ K1 K1MS $\boxed{\text{K4MS}}$ K1 K3 K3CV	$\boxed{\text{K4MS}}$ K1 K1V K1MS $\boxed{\text{K4MS}}$ K1 K1V $\boxed{\text{K4MS}}$ K6	$\boxed{\text{S3}}$ $\boxed{\text{S3}}$ K1 K1V K1CV	$\boxed{\text{N5A}}$ N8
III New III Formerly	750 - 950 790 - 950	6 - 16 24 - 45 58 - 160 6 - 160	$\boxed{\text{N9}}$ $\boxed{\text{N10}}$ N8V N9 N10W	$\boxed{\text{N9}}$ N8V N10	$\boxed{\text{N9}}$ N8V N10	$\boxed{\text{K3CV}}$ K5 K5V $\boxed{\text{K7}}$ K5 K5V	$\boxed{\text{K3CV}}$ K5 K5V $\boxed{\text{K7}}$ K5 K5V	$\boxed{\text{S3}}$ K3 K1V K1CV $\boxed{\text{K1MS}}$ K3 K1CV	$\boxed{\text{N5A}}$ N8 N8A

Materials in a frame $\boxed{}$ are the replacement parts in present day use.

STEP	PRESENT	FORMER
I	0 - 390°F	0 - 390°F
II	390 - 750°F	390 - 790°F
III	750 - 950°F	790 - 950°F

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T.O.M. Reel No. 75
pp. 37-46

T-59

CONTINUOUS PROCESS FOR THE PRESSURE HYDROGENATION OF COALS, TARS AND MINERAL OILS IN LIQUID PHASE

Ludwigshafen, May 25, 1943

In the continuous pressure hydrogenation of coals, tars and mineral oils in liquid phase as, for example, of coal or oil pastes the liquid, together with the hydrogen required for the reaction, is at present preheated under pressure in a special preheater and brought to the reaction temperature. At this temperature the mixture then enters the reaction vessel. Here, due to the absorption of hydrogen by the hydrogenation feed, so much heat is generated, apart from the heat loss through the vessel walls, which is only a small part of the generated heat, that in practical operations cooling means must be provided as, for example, through built-in special cooling surfaces, through the introduction of cold gas or through the injection of cold oil or of coal paste.

These measures bring many disadvantages with them. In particular the introduction of cold substances brings about a decrease in the space available for the reaction and a change in the proportion between gas and reaction mass so that the course of the reaction is often influenced undesirably.

In accordance with the invention submitted herewith, these disadvantages are avoided and considerable advantages obtained. In this process the hydrogenation feed, together with hydrogen, enters at the bottom of one section of a reaction vessel divided by separating walls into two vertical sections, which are connected with each other at the top and bottom, so that the hydrogenation feed is given a circulating motion between the two sections of the vessel, whereby the greatest part of the hydrogen, together with the vaporous and as a rule also with a part of the liquid, mass is drawn off at the top.

The separating wall can advantageously consist of a centrally arranged tube in the reaction vessel, a so-called guide tube, so that the inner space of the tube forms one section and the outer space the other section of the reaction vessel.

The stream of hydrogenation feed and hydrogen gas entering at the bottom rises, for example, in the guide tube. At the upper end of the guide tube the main mass of the liquid portion, generally free of gas, then again flows downward outside of the tube, is again united at the bottom with the fresh incoming mixture and again passes upward in the guide tube. At the upper outlet of the reaction vessel a mass of gas and liquid (as such or as vapor) approximately equal to that entering at the bottom is carried off during operations.

While in the present process the gas bubbles rise to the top comparatively slowly and without order because they must overcome the viscosity of the liquid column above them and because they themselves retard the return flow of the liquid. This new process generates a lively circulating motion in the longitudinal direction of the guide tube, which can be considerably faster than the original rising velocity of the bubbles in the liquid.

Because of this an intermixture of the whole contents of the vessel is developed as well as a breaking up of the large gas bubbles into a foam of the smallest bubblets with a large surface area. A rapid intermixture of the contents of the vessel with the fresh incoming stream of the hydrogenation feed makes it possible to reduce the inlet temperature of the vapor-liquid mixture by an amount equal to the heat of reaction in the reaction vessel. This latter is directly used for further heating of the fresh incoming hydrogenation feed. This eliminates the necessity of adding cold gas in an amount at present necessary, if the inlet temperature is correspondingly low. The volume of these cooling substances may be so much reduced that the reaction may be readily controlled.

The circulating stream receives its start from the difference in uplift between the upward and downward flowing sections and from the stream energy of the mixture entering the vessel. The velocity of the stream is limited only by the resistance to the stream in its circulating path. Means for aiding this stream flow and increasing its velocity, such as a nozzle-shaped entrance opening with injector effect, rounding off the corners of the reaction vessel and the guide tube, and the largest possible diameter of reaction vessel and guide tube should properly be employed. Also the proportion of the stream cross sections should be such that the resistances in the upward and downward flow sections are as closely as possible equal to each other. Moving devices in the reaction vessel, such as power driven mixers, etc., are to be avoided by all means.

The passage time of the gas bubblets through the guide tube, and consequently their presence in the reaction vessel, is considerably shorter than in the present process because the gas volume in the reaction vessel is reduced with increased velocity of the circulating stream and because the gas is largely contained only in the rising stream, while only smallest bubblets, which are not separated from the stream at its change of direction at the top, are carried in the downward flowing stream.

Through this decrease in the gas volume present in the reaction vessel a corresponding space is freed for the liquid hydrogenation feed so that the feed remains in the vessel considerably longer, assuming equal volumes of reaction vessels, or that a greater volume of hydrogenation feed is processed, assuming equal time periods of its presence in the vessel.

The separation of the hydrogen and other gases in the upper part of the reaction vessels is aided considerably if the rising liquid-vapor stream is given a strong twist by properly arranged entrance nozzles or by the arrangement of guiding surfaces within the guide tube. Under the influence of this twist the liquid is easily separated at the return point at the top from the gas collecting in the center so that only little gas is carried downward. Small openings in the upper part of the guide tube will have the same effect because they reduce the velocity of the liquid at this point.

The guide tube can readily be constructed with a double wall and used as a very effective cooling or heating surface.

A particular advantage of the proposed means is, as already explained, the lower preheat temperature required. The manufacture and procurement of preheater tubes for high reaction temperatures offers many difficulties since the strength of alloyed steels for this purpose drops rapidly with rising temperature so that an increase of 10°C more or less in the wall temperature of the preheater tubes is of great significance. In addition, it is possible to attain the reaction temperature only by using considerably longer tubes, if the permissible temperature difference between the heating gases and the preheater wall temperature becomes small.

If, on the contrary, it is possible to introduce the feed into the reaction vessel at a temperature lower by about 50 or 100°C than at present, that is, within the reaction temperature, the preheater can be operated at lower wall temperature, and the previously mentioned difficulties disappear entirely. In addition, the preheater resistance and required pump energy drop considerably because of the shortening of the preheater tubes. Very often the preheater may even be entirely dispensed with in operation, if the now possible temperature difference between the outgoing and incoming stream is utilized in the heat exchanger so that the preheater is used only at the start of operations.

This provides a very considerable saving of fuel and energy and a great lessening of the danger to the reaction stalls caused by the accumulated heat in the preheater walls in a sudden shutdown.

Besides the saving in fuel a considerable saving in expensive alloyed metals is possible because of a lowered preheater temperature and a small preheater.

In connection with the lowering of the preheater temperature it is further possible to protect the reaction substance, prevent it from overheating and from coking on the preheater walls.

A further advantage is gained by the previously mentioned lowering of the cold gas volume required in the present process. The use of much cold gas requires a higher pressure in the circulating pumps and therefore considerably greater energy. Furthermore, in the present process the cold gas has to be added at various points in the reaction chamber to avoid local overheating. If it is desired to add cold gas at all in the new process, this may be done at one point. Since the entire content of the high reaction vessels is completely circulated within a fraction of a minute equal temperatures obtain in all parts of it. Furthermore, due to the decrease in cold gas volume it is possible to keep the proportion of gas to liquid volume in a chamber with several reaction vessels the same, so that the desired partial pressure can more easily be achieved.

A further advantage is gained in the decrease of the volume of the feed passing through the reaction chamber so that the connecting pipes and heat exchangers can be reduced in size or be used for larger capacities.

The fine distribution of the hydrogenation gases possible in this new process enables us to reach an equilibrium in the reaction, an effect, which otherwise is possible only through an increase in pressure, a longer time period required for the feed to remain in the reaction vessel, or the use of valuable catalysts. For the same reason it is possible to lower the average reaction temperature with the same capacity and hydrogenation effect.

The process is further clarified in the attached drawing.

In figure I, (1) is the inner wall of the reaction space of a hydrogenation converter, (3) is the inlet and (4) the outlet tube for the gas-liquid mixture (for example tar), (2) is the central guide tube which is arranged in converter I in such a manner that an unhindered circulating motion through the guide tube in a longitudinal direction of the converter is made possible. The mouth of tube (3) is nozzle-shaped in order to introduce the liquid mixture with increased velocity.

Figure II shows a cross section through converter 1 with guide tube (2). It is immaterial whether the flowing mass rises on the inside or the outside.

Figure III shows a cross section through another construction in which the separating wall is a simple cross wall (2) instead of a guide tube.

Figure IV shows a double walled guide tube to receive heating or cooling fluid or cooling gas or provide room for electrical heating. In order to decrease the stream resistances the guide tube is rounded off at (c) and (d), the converter at (e) and (f).

In order to achieve a better separation between vapor (foam) and liquid an additional turning motion of the circulating stream is superimposed. Helical shaped guide surfaces (5), tangentially arranged inlet nozzles (7) and guide surfaces (8) on the upper and lower end of guide tube (2) are provided, as shown in figure V. Under the influence of the twist produced in this manner the liquid moves preferably along the wall of tube (2) at (a) (figure IV) while the lighter foam flows towards the top mainly in the center. In this manner the gas volume in the converter is considerably decreased. In addition, openings (6) can be arranged at the upper end of the guide tube in order to decrease the stream velocity at the return point at the top and to make possible a better separation of liquid and gas.

Figure VI shows the guide tube (2) subdivided into several sections, so that several short circulating streams besides the main circulating stream, may be formed. These small circulating streams can also be completely separated from each other through cross walls so that so-called multiple converters are formed, which then may be further developed according to figures I to V.

PATENT CLAIMS

#1.-Continuous process for pressure hydrogenation of coals, tars or mineral oils in liquid phase, producing a circulation of the hydrogenation feed in the reaction vessel, so that; the hydrogenation mass, together with hydrogen, will enter one section of the reaction vessel (which is divided into two vertical sections, connected with each other at the top and bottom) at the bottom in such a manner that the hydrogenation mass is caused to circulate between the two sections, whereby the greatest part of the hydrogen entering at the bottom is taken off at the top, together with vaporous and fluid products of reaction.

#2.-Process according to Patent Claim 1,- characterized by a tube centrally arranged within the reaction vessel and possibly provided with side openings at the top, forms the dividing wall between the two sections of the reaction vessel, so that the space within the tube forms one of the sections and the space outside of the tube forms the other section.

#3.-Process according to Patent Claim 2,- characterized by the central tube, consisting of several pieces of a tube separated from each other by intervening spaces.

#4.-Process according to Patent Claims 1 to 3,- characterized by hydrogenation feed entering tangentially, so that it will flow towards the top in a spiral stream in one of the sections.

#5.-Process according to Patent Claims 1 to 4,- characterized by the section, in which the hydrogenation feed flows towards the top, provided with guiding surfaces giving the upward flowing mass a twisting motion.

#6.-Process according to Patent Claims 1 to 5,- characterized by the hydrogenation feed (possibly after being heated by heat exchange) entering the reaction vessel at a temperature considerably below the reaction temperature.

I.G. Farbin Industrie, A.G.

/S/ Klebba

KCBraun/MC/fkp

7-25-1946

FIG. I

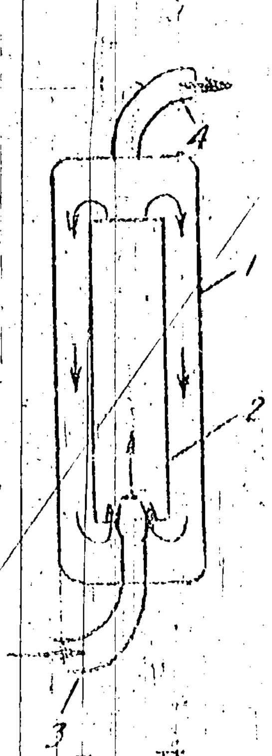


FIG. IX

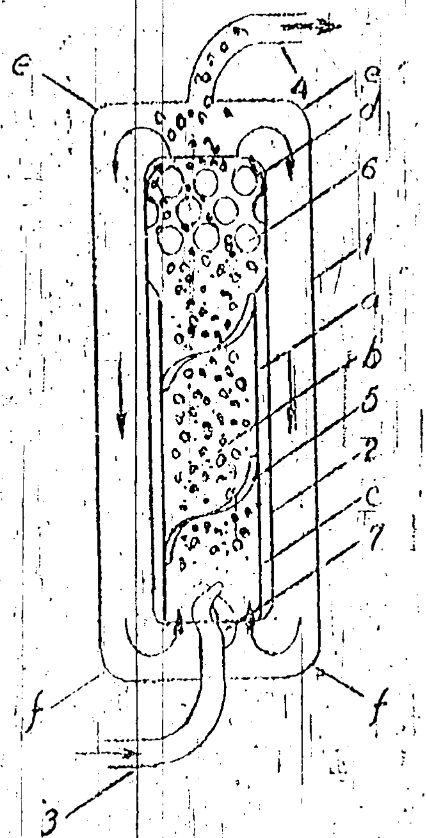


FIG. VI

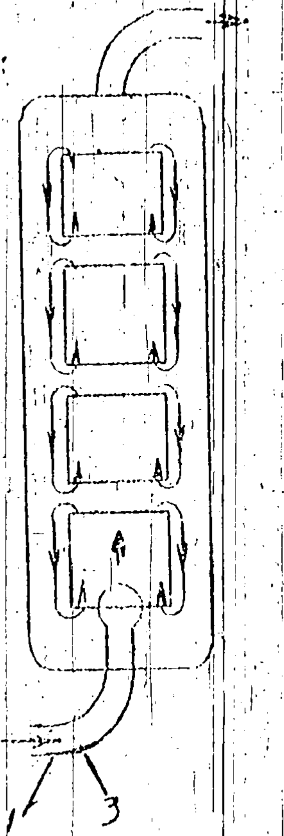


FIG. II

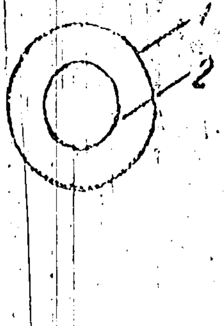


FIG. III

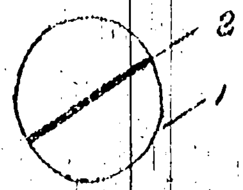
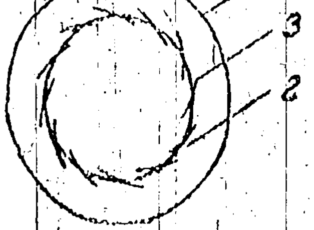


FIG. V



T.O.M. Reel No. 76
pp. 13-28

T-60

AN ABSTRACT OF A DISCUSSION OF HIGH PRESSURE VALVES AND FITTINGS

Ludwigshafen, Dec. 10 & 11, 1942

Participants in the discussion:

Chairman: Raeithel

Work	Böhlen:	Graichen, Hartmann
"	Brüx:	Kutter
"	Gelsenberg:	Beutner, Dr. Schmalter
"	Heydebreck:	Weber
"	Hüls:	Meyer
"	Linz:	Drechsler
"	Leuna:	Cron, Maier, Richter, von Rossum
"	Ludwigshafen:	Bahlinger, Bolbach, Berger, Bernhard, Dr. Class, Dr. Dinkler, Dr. Generlich, Hahn, Dr. Kümpfe, Dr. Kautz, Koch, Meyer, Muller, Rader, Raichle, Rick, Römer, Schanzenbächer, Schappert, Schaurer, Schmidt, Wagner, Weid
"	Magdeburg:	Bielek
"	Pölitz:	Dr. Benke, Zimmermann
"	Schkopau:	Riedl
"	Scholven:	Eisenbraun, Gehb, Koets
"	Wesseling:	Dr. Erdmann, Dr. Neubauer
"	Zeitz:	Nerge

NOTE: Much of this report is very difficult, and some of it impossible, to read.

Materials of Construction.

Rasithel: The high pressure materials may be subdivided into 4 main groups:

a) Materials subject to stress only, that is, which do not come in contact with the operating product. These are S1 and the K-materials. Basic color: white.

b) Materials coming in contact with the cold product and which must be leak-proof. These are S2 and S3. Basic color: green.

c) Materials coming in contact with the hot product and which must be leak-proof. These are the N- materials. Basic color: blue.

d) Materials, high temperature and corrosion-proof. These are the R- materials. Basic color: yellow.

The individual materials within these main groups again differentiate between operating pressure, temperature and dimensions. The necessity for saving insufficiently available alloyed constituents requires that we introduce new materials of construction. In W. Norm-Beiblatt 10-00 the new materials are compared with the old. The old materials are marked with simple longitudinal stripes on the basic color, the new with double stripes. The exact separation of the individual materials is extremely important.

(The author here discusses the marking and stamping of the various materials for identification in the warehouse.)

Bahlinger: All of the high pressure fitting parts of special materials, such as S3, as well as the K- and N- materials, are heat treated after forging or after delivery, if not already heat treated. The temperatures for the heating up and hardening as well as the desired degree of hardness are specified in specifications for heat treating. Changes in the composition of the steels must also be taken into consideration.

(The author here discusses heat treating temperatures and the control of heat treated materials in the workshops and the difficulties in obtaining suitable materials, government restrictions, etc., of no immediate interest to us).

Dr. Class: We try to control the use of materials in the simplest possible manner. Of particular importance is the assurance, for example, that a non-hydrogen proof steel is not used where a hydrogen-proof steel is essential. Since our hydrogen-proof steels are based on their chromium content and the chromium content can easily be checked by the spot test, the spot test for chromium must be considered the most important, and which can also be made without difficulty at the building site. Besides chromium content from about 1% on, molybdenum contents can also be recognized by the spot test. Vanadium contents however cannot be checked by a simple spot test.

The spot test, together with the hardness test, lends itself well for the individual testing of a large number of pieces. Ludwigshafen has largely followed this procedure.

. . . .

All parts of the chromium alloyed K- and N- steels are spot tested for chromium when they are not individually analyzed. Parts of the molybdenum alloyed N9 material are also spot tested for molybdenum content.

. . . .

Dr. Kümpfe discusses machine shop practices and high pressure testing, for example:

Pressure tests are made according to the following specifications:

	At 325 Atms.	At 700 Atms.
For all fittings pressure-tested with middle oil:		
a) Cold parts	430 atms. test pressure	910 atms. test pressure
b) Hot parts	500 atms. test pressure	910 atms. test pressure
For all assembled fittings such as valves, gates, liquid level gauges, also seal test of the entire housing as well as the shut-off parts, with nitrogen	325 atms. test pressure	700 atms. test pressure

In the nitrogen test, all parts are totally immersed in middle oil and held under pressure for a prescribed period of time, whereby any rising bubbles and a drop in the manometer pressure may be observed. In spite of this, it may happen that leaks may appear later in operation, because pores in the material often show themselves only after many hours.

(Raeithel here discusses identification of parts and the necessity for repeated tests.)

Valve Construction:

Raeithel: The former valves with inside thread have almost all been replaced by valves with outside thread. These have been repeatedly changed and improved in the course of years. The introduction of the 700 atm. stage forced us to adopt a new construction in order to avoid too heavy valves, very difficult to operate. This new construction has proved itself very successfully in operation. It was therefore advisable to reconstruct the 325 atm. valves in the same manner. The advantages of the new construction are the smaller space required, the lower weight and considerably smaller torque required to operate the valve stems. The changes consist above all in the decrease of the stem diameter, the changed location of the connection between valve seat and stem to the outside, the shortening of the stuffing boxes and the valve hubs, the drifting -(?) (Eindornen) of valve seats, and an increased hardness of the valve seat and disk. Of particular importance also is the saving in alloyed materials by welding on the valve stems and seats which were formerly made of solid alloyed materials.

Dr. Kautz: The weld~~ing~~ed on valve stems and seats must fulfill the following requirements:

1) The sealing surfaces on valve seat and disk must be corrosion-proof.

2) The hardness of the disk must be as high as possible.

3) The valve seat should be somewhat softer than the disk. The welded on layer must also be able to endure the deformation caused by the drifting (Eindornen). (Eindornen seems to imply a pressure fitting, possibly with a revolving plug (Dorn)).

(Parts of report impossible to read but seems to treat of welding procedures.)

The former valve seats of V2A were held in place by threaded rings. Later the smaller valve seats were drifted in (Eindornen), the larger were rolled in. Since 1939 valve seats to and including the 90 mm size are drifted in (Eindornen). Valve seats for the 120 mm size are rolled in, now as before. At the present time experiments are under way to weld in the valve seats. Extensive tests have shown that a better hold may be obtained if the valve seats to be drifted in (Eindornen) have protruding teeth on their outer jacket instead of the grooves formerly turned into the housing. Exact specifications for the drifting (Eindornen) exist, indicating drifting tool size (Dornzahl), drifting tool sequence (Dornstufung), and press pressures. The inspector will check the specified widening of the valve bore.

Packing of Stuffing Boxes: Precise specifications for this operation are available. At the present time only Burgmann-cord is used for packing. The rings for the individual valve sizes are cut to predetermined length, inserted into the box with a tap and pressed in on a press with predetermined pressure. The inner side of the rings is greased before insertion with a mixture of hot-steam cylinder oil and flaked graphite.

The subject of stuffing box packing was discussed at length, inasmuch as no more Burgmann-cord was available and had to be replaced by a substitute. In reports on experiences with metal packing of hard lead (for oxygen), aluminum, hydronalium and carbonyl-iron. Very good results have been obtained with aluminum packing in over-lapping (possibly V shaped) form. Valves packed in this manner were sent to a number of hydrogenation works for trial. It should be pointed out here that such stuffing boxes may be only lightly drawn. Sizes deemed suitable for tests were 24 mm for 700 atms. and 30 mm for 325 atms. Such packings of synthetic materials are unsuitable because of their inability to withstand high temperatures. Such packings of fiber have the disadvantage of shrinking when dry (this portion of the report is ~~very~~ impossible to read).

In the assembly operation of screw parts on the large valves a spring scale with predetermined turning moment is used to regulate the applied torque.

Special Valves:

Pressure Relief Valves:

Berger: The normal type of Pressure Relief valve (needle type - patronen ventil), that is with spring held cone-disc and tungsten-carbide nozzle, screen, eccentrically arranged sealing ring, which was adopted from 200 atms. for 700 atms., showed barely tolerable wear in Gelsenberg and very strong wear in Pölitz, particularly at the nozzle outlet. Protection of the wearing surfaces by Granitherm showed very little improvement.

Dr. Schnalter: Because of crushing (Auskolken) behind the nozzle, the nozzle soon breaks out. As a rule nozzles break out, rather than wear out. In Gelsenberg the nozzles are no longer soldered or shrunk in, but fitted in after copper plating. Copper plating does not eliminate any unevenness. Gelsenberg expects much from a short nozzle with contiguent widening, which was in operation for 1,037 hours instead of the usual 400 to 500 operating hours. Nozzles which are subdivided into individual rings will not do much good.

Zimmermann: Pölitz has achieved 1,400 operating hours with an elongated nozzle built up of 2 parts and widened at the outlet. In Pölitz also, the nozzles broke out after the surface material was gone.

Check Valves:

Berger: The spring check valves provided with mushroom disks have repeatedly stuck. This is due to the naturally low power of the springs, which weaken, particularly at higher temperatures, and to the closing pressure caused by the liquid stream, which loses effectiveness on the upper disk surface because of its characteristic construction. It is therefore proposed to build a springless mushroom disk check valve on which the closing pressure can freely act.

Regulating Valves:

Berger: While we use the present construction for the smaller sizes, we use the construction of the normal shutoff valve for sizes from 24 mm on up, in which a throttling pin or needle is attached to the disk. The throttling pin may be changed to suit operating requirements (gas, liquid, fine, or Coarse regulation). In larger sizes, remote control by means of electric drive is possible. The seating surface is so formed that it is not a throttling surface, that is, not subject to wear.

Weid: Regulating valves should be so installed that the flow of the fluid is in the direction of the stem, that is, the pressure is against the under side of the disk. If the flow is in the opposite direction, the regulating cone serves as nozzle, which makes regulation difficult.

Motor Operated Valves:

(Scherer discusses the advantages of motor driven valves built according to "Bauart 1940". This discussion discloses nothing new and is of no particular interest to us.)

Gates:

Baeithel: Gates are now so constructed that the inner pressure aids the opening. With the same direction of rotation of the hand wheel, the gate movement for closing is therefor opposite to that in the valves.

Beutner reports that leaks in gates are probably due to corrosion. Hahn points out that gates should only be used where no dirt or ~~sinter~~ scale is in the line. Maier reports that the nitride case has been crushed on individual gate disks.

Liquid Gauges:

Dr. Kämpfe: The sealing of large surfaces and the sensitivity of glass require a very careful construction. Many experiments have been made in the course of years to improve liquid level indicators. Experiments were particularly made on the means of producing initial tension. The initial tension must be high enough to produce an adequate seal, but must not be so high as to break the glass. The springing due to initial tension must be large enough to overcome unavoidable deviation in the glass thickness and the assembly dimensions. The following values indicate the recommended spring necessary to produce the required initial tension:

	Initial Tension:	Back Spring:
For Buna Frames:	About 1,000 kg.	About 2 mm
? For "Wellfedern"	About 8,300 kg.	About 1.6 mm
For Aluminum Wire: Compression		
From 4 to 2.5 mm	15,000 kg.)	About 0.2 - 0.4 mm
From 4 to 2. mm	35,000 kg.)	

Based on experiments, an initial tension of 5,000 to 6,000 kg. is sufficient. With the high initial tension produced by aluminum wire many glass-breaks occur. With Buna the frame is self-sealing, because it is put under higher tension only under operating pressure, but Buna cannot be used for 700 atms. and for various products. Therefor, most liquid gauges have, for a long time, been made with aluminum wire and aluminum foil for sealing. Since the glass thickness may vary as much as 0.1 mm, very accurate assembly is necessary. For special purposes, for which no aluminum can be used, liquid gauges of Buna frames with fiber cushions may also be used.

Fittings, Sealing Rings:

Raeithel: In the elbows the cutting edges on the inside are rounded off, in Ts they are broken. Bends are now made up to 200 mm sizes. The bend sizes 160 mm and 200 mm are somewhat longer than the elbows. Fittings with copper lining or chrome plated fittings are also available.

Sealing rings for hot lines will in the future be made only of N5A with a Brinell hardness of 180 to 220. Somewhat softer rings are acceptable for special purposes. Bellows sealing rings are the same in material and dimensions for 325 and 700 atms. Blind sealing rings are alike in material and dimensions for both pressures. Cold blind sealing rings of the larger sizes differ in material and dimensions, hot rings only in dimensions. Material and pressure are indicated on the flap.

Manometer disks are furnished only with 1 mm bore.

It is desired that fittings, particularly bends, be provided in the welding as well as the screwed type. Welding bends for 325 and 700 atms. will in the future be kept in stock. It is also desired to provide limiting dimensions for the sealing rings in order to facilitate their fitting. These rings should also be made extra large in order to provide a greater fitting range.

Threads: Our threads are still cut to an accuracy corresponding to a fine fit. A medium fit should also be satisfactory just as in other industries. In order to avoid the freezing of threads they should be greased with hot-steam cylinder oil and colloidal graphite.

(Report from here on is very difficult to read but seems to deal largely with increase in stresses demanded by government authority, selection of materials, delivery details, drawings, stock lists, etc., all of which are not important for our purpose.)

KCBraun/HC/jw

7-26-1946

BUREAU OF MINES

T.O.M. Reel 162, pp. 752-756

T-61

January 27, 1943

APPROXIMATE CALCULATIONS OF THE HEAT TRANSFER COEFFICIENT OF A PREHEATER

The heat transfer coefficients have been calculated for a 10 li vapor phase converter using some simplifying assumptions. The equation

$$k = \frac{1}{\frac{1}{\alpha_i} + \frac{\delta}{\lambda} + \frac{1}{\alpha_a}} \qquad (1)$$

can be used in evaluating the factor k in $\frac{\text{kcal}}{m^2 \times h \times \text{degree}}$ which is a measure of the total amount of heat transferred. In this equation, α_i is the heat transfer coefficient from the inner tube surface to the liquid (or gas) flowing inside, α_a the heat transfer coefficient of heat absorbed by the outer surface, δ the wall thickness, and λ the heat conductivity of the tube material. The tube is heated from the outside with steam at a temperature around the saturation region, α_a is known to be very high, and may, as a first approximation, be neglected when computing the k value. The outside wall temperature may be set equal to that of the saturated steam (about 212°C at a pressure of 19 atm.). Computations are therefore essentially limited to the computation of α_i; i.e. of the heat transfer from the inner tube wall to the stream of the coal paste and hydrogen mixture.

In order to make computations we must first of all know the conditions of flow. The Reynolds coefficient must therefore be found from the expression

$$Re = \frac{d \times w \times \rho}{\eta}$$

where d - diameter of the tube, w - rate of flow, ρ - density, in g/cm^3 and η the absolute viscosity in g/cm x sec. Re is non-dimensional.

Re is determined separately for the coal paste and for the gas, on the assumption that the paste and the gas through the cross section at a given velocity, that is, that gas bubbles and droplets of the paste move with the same velocity through the tube without any mutual effect upon their individual movements. Under such conditions, the calculation of their motion is based on the following:

The velocity of the flow w is given from the total thruput by the equation

$$w = \frac{\text{throughput}}{\pi \times \frac{d^2}{4} \times \rho \times 3600} \text{ cm/sec for the throughput in g/h}$$

$$w = \frac{\text{throughput}}{\pi \times \frac{d^2}{4} \times 3600} \text{ cm/sec for the throughput in ccm/h}$$

The cross section of the tube is 1 cm, the total throughput consists of 10 kg paste and 17,000 liters gas/h (measured at 15°C and 1 atm. The volume of the 17,000 li. of H_2 under the conditions in the preheater (600 atm, abt. 200° C) is found from the equation

$$\frac{P_2}{P_1} = k \times \frac{v_1}{v_2} \times \frac{T_1}{T_2}$$

The integral compressibility factor k of hydrogen at 200°C is equal to 1.25. We get then:

$$v_2 = 1.25 \times 17,000 \times \frac{475}{288} \times \frac{1}{600} = 58.2 \text{ li.}$$

Should we assume the density of the paste to be 1.0, the total throughput of the system will be 58.2 li/h, assuming that the total volume of the system is equal to the sum of the individual volumes, i.e., that we may disregard the solubility of hydrogen. We shall get then an average rate of flow

$$v = \frac{68,200}{\frac{\pi}{4} \times 3600} = 24.1 \text{ cm/sec.}$$

No measurements are known of the viscosity of the paste under the preheater conditions. There is information that the viscosity of the converter contents (presumably that of the liquid constituents) at the converter temperature is that of water. The value of the latter at 20°C is equal to 0.01 $g \times cm^{-1} \times sec^{-1}$*). The viscosity of the paste under the conditions existing in the preheater are considerably higher, i.e.

$$\eta_{\text{paste}} \geq 0.01 \; g \times cm^{-1} \times sec^{-1}$$

which leads us to

$$Re_{\text{paste}} < \frac{1 \times 1 \times 24.1}{0.01} = 2410$$

The density of hydrogen at 15° C and 1 atm is 0.847×10^{-4}, from which we get its density under the preheater conditions as

$$\rho_{H_2} = 0.847 \times 10^{-4} \times \frac{17,000}{58.2} = 2.47 \times 10^{-2} \; g/cm^3$$

The absolute viscosity of H_2 at 200° is $2.211 \times 10^{-4} \; g \times cm^{-1} \times sec^{-1}$ and may be assumed as a first approximation to be independent of pressure. From this we may calculate the Reynolds number for hydrogen

$$Re_{H_2} = \frac{1 \times 2.47 \times 10^{-2} \times 24.1}{1.211 \times 10^{-4}} = 4930$$

---

*) corresponding to 1° E.

We must accordingly expect a laminar flow for the paste, and a turbulent flow for the gas, since in one case the Reynolds number falls below the critical value of 2300, in the other above it.

For a laminar flow in a long straight tube we get the Nusselt relationship

$$Nu = 3.63 \qquad (2)$$

where Nu is the non-dimensional Nusselt factor $Nu = \alpha \times \frac{d}{\lambda}$. we get from this

$$\alpha_{paste} = 3.63 \times \frac{\lambda}{d}$$ (λ = heat conductivity of the paste in $\frac{cal}{cm \times sec \times degree}$, or $\frac{kcal}{m \times h \times degree}$)

No experimental data are available for the heat conductivity of the paste. Values of 0.3 to 0.4 x 10^{-3} cal/cm x sec x degree may be found in literature for the different oils. For coal, also 0.4 x 10^{-3} ca/cm x sec x degree. We estimate therefore for the paste

$$\lambda_{paste} = \text{about } 0.4 \times 10^{-3} \frac{cal}{cm \times sec \times degree} = 0.144 \frac{kcal}{m \times h \times degree}$$

We get then

$$\alpha_{paste} = 3.63 \times \frac{0.144}{0.01} = 52.6 \frac{kcal}{m \times h \times degree}$$

For turbulent flow, such as must be assumed for the gas, according to Prendtl-Hoffmann, the following heat transfer equation must be assumed:

$$Nu = \frac{0.0395 \times Re^{0.75} \times Pr}{1 + 1.5 \times Re^{-0.125} \times Pr^{-1/6}(Pr-1)} \qquad (3)$$

$$Pr - \text{Prandtl factor} = \frac{\nu}{a} = \frac{\eta \times e_p}{1} \quad \text{(non dimensional)}$$

(ν - kinematic viscosity = $\frac{\eta}{\rho}$ $\frac{cm^2}{sec}$)
(a - thermal conductivity $\frac{1}{e_p \rho}$ $\frac{cm^2}{sec}$)
(e_p - spec. heat, $\frac{cal}{g \times degree}$)

with e_p of H_2 equal to 3.5 cal/g x degree and a heat conductivity $\lambda = 0.6 \times 10^{-3}$ cal/ m x sec x degree we get*):

---

*) we may neglect here the fact that the viscosity and heat conductivity of H_2 are not actually independent of pressure, because λ and η change in the same way and the deviations are mutually cancelled.

$$Pr = \frac{1.211 \times 10^{-4} \times 3.5}{0.6 \times 10^{-3}} = 0.71$$

The substitution in equation (3) gives

$$Nu_{H_2} = \frac{0.0395 \times 4930^{0.75} \times 0.7}{1 - 1.5 \times 4930^{-0.125} \times 0.7^{-1/6} \times 0.3} = 19.5, \text{ and from that}$$

$$\alpha_{H_2} = Nu \times \frac{\lambda}{d} = 19.5 \times \frac{0.216}{0.01} = 421 \frac{\text{kcal}}{m^2 \times h \times \text{degree}}$$

Equation (3) is only valid for smooth straight tubes, with a proportion of length to diameter $\geqslant 200$, otherwise the value α will be increased. Under the actual conditions, Nu might vary around the lower limiting value.

Should we assume the existence of some α value for the total heat transfer and that it is composed of those for paste and for hydrogen in proportion of their heat absorptions, we will get for 1° rise in temperature of 17,000 li H_2 an amount of heat of 17,000 x 0.31 = 5280 cal, while that for the 10,000 g of paste with an estimated specific heat of 0.475 cal/g will amount to 4750 cal.

$$\text{total} = \frac{5.28 \times \alpha_{H_2} + 4.75 \times \alpha_{\text{paste}}}{10.0} = 222 + 25.2 = 247 \frac{\text{kcal}}{m^2 \times h \times \text{degree}}$$

This value must therefore be introduced into equation (1) for α_i for the case of clean tubes, so that only the metallic conductivity (λ of the tube material estimated at 0.06 cal/m x sec x degree) is to be considered in the heat transfer through the tube walls, while the second summation factor in the equation (1) becomes small enough to be disregarded. In this case,

$$k = \alpha_i = 247 \frac{\text{kcal}}{m^2 \times h \times \text{degree}}$$

The actual k values observed were between 50 and 90, and they are therefore above the value for the paste, but much below the anticipated total values. This can only be explained by the poor conductancting layer which will at least interfere with the transfer of heat to hydrogen. Any other deviations which actually exist from the relationships assumed here, such as the greater velocity of rise of the hydrogen bubbles producing vortex motions in the paste, could only occasion a further increase in the k values. Should we assume, however, that hydrogen does not come into direct contact with the metal of the tube, say for reason of a continuously present layer of the paste, the k value will be immediately greatly reduced. Should one for instance assume that there remains a 1 mm thick layer of paste, we shall obtain, using the values for the heat conductivity of the paste assumed above,

$\lambda = 0.144 \frac{\text{kcal}}{\text{m x h x degree}}$ for hydrogen only

$$k_{H_2} = \frac{1}{\frac{1}{421} + \frac{0.001}{0.144}} = 107,$$

or only 1/4 of the value found previously. This would give

$$k_{total} = \frac{5.28 \times 107 + 4.75 \times 53}{10} = 80,$$

a value in the vicinity of those found.

Sternberg/MC/fkp

8-1-1946

T.O.M. Reel No. 181
Pages 625 to 631

Abstract of Technical Report on the Deformation of 120 mm Sealing Rings of N5 material due to inner pressure and with very tight drawn bolts, dated Ludwigshafen, 12 July, 1936

Up to this time the sealing rings were generally bored smaller in diameter than the nominal diameter. On 45 mm. N. D. the bore was 42 mm, which was decreased by as much as 2 mm by the bolts drawn tight in operation. The tube cross-section was thereby constricted up to 5 mm on the inner diameter of the 45 mm N. D. The 200 N. D. sealing rings were bored to 190 mm. On the basis of a previous report made in 1927 all sealing rings were then bored out to their N. D.

A test was now made to determine the deformation of an 120 mm N. D. sealing ring of N5 material according to drawing NB-2456-16. The inner pressure was produced by means of a hand pressure pump and a spindle press, using middle oil. A test at 325 ats. was also made with nitrogen. The tightening of the bolts was done, wherever possible, by a crane winch over a pull-dynamometer.

The N5 ring had just been taken from the lathe shop Lu 653, where it had just been completed. It had a Brinell hardness of 153, given us by the Lu Materials Testing Department. Flanges and bolts of N03 material were taken from stock. The bolts had an average strength of 74 kg/mm^2. The required pieces of tube of N8 material were fabricated by the Lathe Shop Lu 653. Tube-I showed a Brinell hardness of 197 in a strength test, Tube-II a Brinnell hardness of 236. The first figure is near the lower, the latter figure near the upper permissible limit. The assembly of the pieces was made according to Sketch N 2484 in which the required dimension-points are also shown. In order to eliminate the personal equation as far as possible all measurements were taken with precision instruments.

After the required dimensions of the individual parts were taken before assembly, the flange connection was made. The sealing ring was well aligned, the bolts were mounted without appreciable tension and the diameters and lengths were again measured. The bolts were then tightened by means of the pull-dynamometer with a 50 kg pull on an open wrench with a lever arm of 700 mm, and this condition was noted. 50 kg. corresponds to the strength of a man using one arm. After screwing on the closing caps, the first pressure test was made with middle oil. The pressure was slowly increased until at about 260 ats. a leak occurred and measurements were taken. The pressure was then released and measurements again taken. Since the first condition with 50 kg. pull on a 700 mm lever arm did not provide the seal necessary for an operating pressure of 325 ats. the bolts were drawn tight with 100 kg on a wrench with 700 mm lever arm. Based on our former experiments this corresponds approximately to the pull of a normal man using both arms. The measurements were also noted in this condition. In the then following pressure tests with middle oil the flange connection was absolutely tight at 325 ats. In this test with nitrogen and immersion in an oil bath no leaks of any kind appeared at 325 ats. After noting the measurements in this condition, middle oil was again applied and the pressure increased to the test pressure of 430 ats. and measurements were taken at this pressure. The pressure was then released and measurements were again taken. Since at 430 ats. no leaks occurred, the pressure was further increased to 500, 600, 700 ats., until at 780 ats. signs of leaks appeared, which was proved by the insertion of blotting paper in the slit between ring and tube, although the dynamometer showed no pressure drop. After noting the measurements, the parts were measured with the pressure released and with loose bolts. Since at 780 ats. the applied pressure was already 2.4 times the operating pressure and the closing caps were not constructed for high pressure, no pressure increase was attempted and the following tests were made with the inner [illegible] excess pressure. The screws were now pulled tight with a wrench 1400 mm long with 100, 150 and 195 kg. pull and then measured. A further increase in pulling power was not possible because the mouth of the wrench began to open and it was difficult to hold the flange connection against turning. A common method used in assembly was therefore resorted to and the bolts were tightened with a new wrench 700 mm long, which was given a tap with a hammer weighing 6 kg, 4 times in succession, and were then again measured. Since the stress on the ring and bolts was already above the elastic limit, the tapping could have been continued at will. After this operation the flange connection was again dismantled. The individual parts were measured and the permanent deformation determined.

The result of the deformation of the ring is summarised in Sketch W 2485. By means of a normal two-armed pull on a wrench 700 mm long, tightness up to 780 ats. is attained in a cold condition. A permanent deformation does not occur in the bore. The occurring changes of 0.02 mm are within the limits of the permissible inaccuracy in measurement. The original annulus pressure rings of both conical surfaces have been widened to pressure surfaces about 5 mm wide. However, these pressure surfaces have not yet assumed the spherical surfaces of the tube ends after loosening, but still form spherical surfaces with a radius somewhat larger than the original. The stress condition attained in this manner is therefore still elastic, which must be considered the only correct condition for this flange connection.

Due to the excessive pull and tapping of the bolts in this case, the ring bore was decreased by about 6.5 mm and its outer diameter about 4.5 mm. The ring has now assumed the form of the conical surfaces of the tube ends. The flowing of the rings toward the center causes a reciprocal compression of ring and tube on the outer periphery of the sealing surfaces. The strength of the sealing surfaces of the ring has now risen to a Brinell hardness of 161.

The deformation of the tubes is given in Sketch W 2486. No appreciable permanent deformations have occurred in the tube diameters and lengths even at the excessive loads. The decrease in the distance between both tube ends, which reached a maximum of 3 mm, is due to the decrease of the ring diameter.

The changes in length of the bolts is given in Sketch W 2487. These changes in length indicate the power applied to the flange connection. The test shows that the bolts are lengthened 0.05 mm by the application of a 2-armed pull (test No. 6). At a pressure of 780 ats. (test No. 10) this value rises to an average of 0.1 mm thru the increase of the sealing ring diameter (self sealing) and regains its former value when the pressure is removed (test No. 11). If the bolts are loosened (test No. 12) the original bolt lengths are regained. The stresses at these loads therefore were within the elastic limit. By the application of the operating pressure of 325 ats. the pressure circle of the sealing ring of 130 mm receives a load

$$P_1 = 13^2 \times \frac{\pi}{4} \times 325 = 43,000 \text{ kg.}$$

With the length of the 1-1/2" N03 bolts from center to center of nut of l = 200 mm and a shank diameter of 32 mm, the average change of length λ = 0.0065 mm in test No. 7. The power applied to one bolt

$$P = \frac{E \times F \times \lambda}{l} = \frac{2,200,000 \times 8 \times 0.0065}{20} = 5720 \text{ kg.}$$

and for the entire flange connection of 8 bolts

$$P_7 = 8 \times P = 8 \times 5720 = 45,660 \text{ kg.}$$

With a perfect sealing ring and clean ring surfaces of the tube ends, an initial stress of about 10% above the operating stress is satisfactory for a 120 mm cold line at 325 ats. Since a pressure of 730 ats. was reached with this slight initial stress, self-sealing of the sealing ring occurred, which is also apparent in test No. 10. For safety, however, the bolts would naturally be drawn tighter, about to the extent show in Test No. 13 (100 kg. on a lever arm of 1400 mm). The power applied to 8 bolts is calculated to be

$$P_{13} = \frac{8 \times 2,200,000 \times 8 \times 0.015}{20} = 106,000 \text{ kg.}$$

This loading condition provides an initial stress of about 2.5 times the operating load (P_1). According to former tests made in Oppau an initial stress of 1.8 times P_1 suffices for 120 mm flange connections. A stronger tightening than that indicated in Test No. 13 should be avoided. With about 200 kg. on a lever arm of 1400 mm the elastic limit of the bolts is reached and will be exceeded at the first tap.

The tests show that no excessive tightening is required for sealing against 325 ats. A Brinell hardness of about 160 is enough for a sealing ring of H5 material. An increase of the sealing ring bore would not be required.

By ~~having~~ heavy tapping of the bolts the sealing ring bore may be decreased at will, in the present case by 6.5 mm, in Scholven by 9 mm. The bolts are permanently lengthened 1 mm by this. A flange connection in such an uncontrollable stress condition, to which mounting and heat stresses will be added in operation, must be considered unsafe for operating purposes.

KCBraun/pkl

I. G. N-2485

DEFORMATION OF A 120 mm SEALING-RING OF N5 MATERIAL
(Dimensions in m/m)

Test No.	Loads	Outer Dia.		Inner Dia.		Dimension Points Height				
		1	2	3	4	5	6	7	8	
1.	Without pressure, before assembly	160.06	159.70	120.10	120.06	25.06	25.05	25.04	25.05	
2.	Assembled, lightly drawn	160.06	159.70	120.10	120.06	Not	measurable	until	after	dismantlin
3.	With 50 kg. on a 700 mm lever arm	159.97	159.62	120.05	120.01	"	"	"	"	"
4.	At 260 ats., leaking slightly	160.20	159.82	-	-	"	"	"	"	"
5.	Pressure Released	160.00	159.65	120.04	120.01	"	"	"	"	"
6.	With 100 kg. on a 700 mm lever arm	159.90	159.55	119.96	119.94	"	"	"	"	"
7.	Pressure of 325 ats., middle oil and nitrogen	159.98	159.64	-	-	"	"	"	"	"
8.	Pressure of 430 ats., oil	160.05	159.70	-	-	"	"	"	"	"
9.	Pressure Released	160.01	159.67	119.97	119.955	"	"	"	"	"
10.	Pressure 780 ats.	160.32	159.98	-	-	"	"	"	"	"
11.	Pressure Released	160.00	159.63	119.98	119.97	"	"	"	"	"
12.	Loosened	160.11	159.75	120.08	120.04	24.99	24.99	24.98	25.00	
13.	With 100 kg. on a 1400 mm lever arm	159.60	159.32	119.34	119.36	Not	measurable	until	after	dismantlin
14.	With 150 kg. on a 1400 mm lever arm	158.62	158.30	117.93	117.97	"	"	"	"	"
15.	With 195 kg. on a 1400 mm lever arm	157.60	157.34	116.59	116.55	"	"	"	"	"
16.	One light tap	157.14	156.85	116.30	116.33	"	"	"	"	"
17.	Two heavy taps	156.64	156.35	115.08	115.06	"	"	"	"	"
18.	Three heavy taps	155.85	155.45	113.88	113.97	"	"	"	"	"
19.	Four heavy taps	155.40	155.05	113.30	113.33	"	"	"	"	"
20.	Loosened	155.52	155.23	113.50	113.50	25.22	25.21	25.16	25.17	
21.	Permanent Deformation m/m	4.48	4.47	6.60	6.46	0.16	0.16	0.12	0.12	

I. G.

W 2487

CHANGE IN LENGTH OF THE BOLTS IN THE DEFORMATION TEST ON 120 mm SEALING-RINGS OF N5 MATERIAL

(Dimensions and Deformation in m/m)

Test No.	Loads	Length & Changes in Length of the Bolts							
		29	30	31	32	33	34	35	36
1.	Without pressure, before assembly	262.26	262.24	262.27	262.22	262.24	262.25	262.20	262.24
2.	Assembled, lightly drawn	262.26	262.24	262.27	262.22	262.24	262.25	262.20	262.24
3.	With 50 kg. on a 700 mm lever arm	262.29	262.26	262.28	262.24	262.25	262.27	262.21	262.26
4.	At 260 ats., leaking slightly	262.30	262.26	262.29	262.24	262.26	262.28	262.22	262.26
5.	Pressure Released	262.27	262.24	262.27	262.23	262.24	262.25	262.20	262.25
6.	With 100 kg. on a 700 mm lever arm	262.30	262.29	262.32	262.28	262.28	262.30	262.24	262.30
7.	Pressure of 325 ats., middle oil and nitrogen	262.31	262.30	262.34	262.29	262.30	262.34	262.25	262.31
8.	Pressure of 430 ats., oil	262.32	262.32	262.35	262.315	262.31	262.36	262.26	262.33
9.	Pressure Released	262.28	262.26	262.29	262.26	262.25	262.27	262.22	262.27
10.	Pressure 780 ats.	262.36	262.34	262.37	262.33	262.33	262.38	262.29	262.38
11.	Pressure Released	262.30	262.29	262.32	262.28	262.29	262.32	262.26	262.32
12.	Loosened	262.26	262.25	262.27	262.23	262.24	262.26	262.205	262.25
13.	With 100 kg. on a 1400 mm lever arm	262.40	262.42	262.42	262.40	262.38	262.40	262.35	262.41
14.	With 150 kg. on a 1400 mm lever arm	262.47	262.54	262.48	262.56	262.46	262.47	262.42	262.54
15.	With 195 kg. on a 1400 mm lever arm	262.55	262.69	262.59	262.64	262.60	262.65	262.55	262.64
16.	One light tap	262.61	262.67	262.62	262.67	262.65	262.68	262.58	262.67
17.	Two heavy taps	262.77	262.82	262.80	262.83	262.79	262.87	262.75	262.80
18.	Three heavy taps	263.20	263.21	263.46	263.22	263.16	263.32	263.22	263.18
19.	Four heavy taps	263.69	263.51	263.75	263.51	263.55	263.55	263.66	263.65
20.	Loosened	263.31	263.30	263.46	263.23	263.19	263.31	263.30	263.33
21.	Permanent Deformation m/m	1.05	1.06	1.19	1.01	0.95	0.95	1.10	1.09
	Brinell Hardness	215	219	214	219	219	216	217	229

I. G.

W 2486

DEFORMATION OF THE TUBES IN THE DEFORMATION TEST ON 120 mm SEALING-RINGS OF N5 MATERIAL

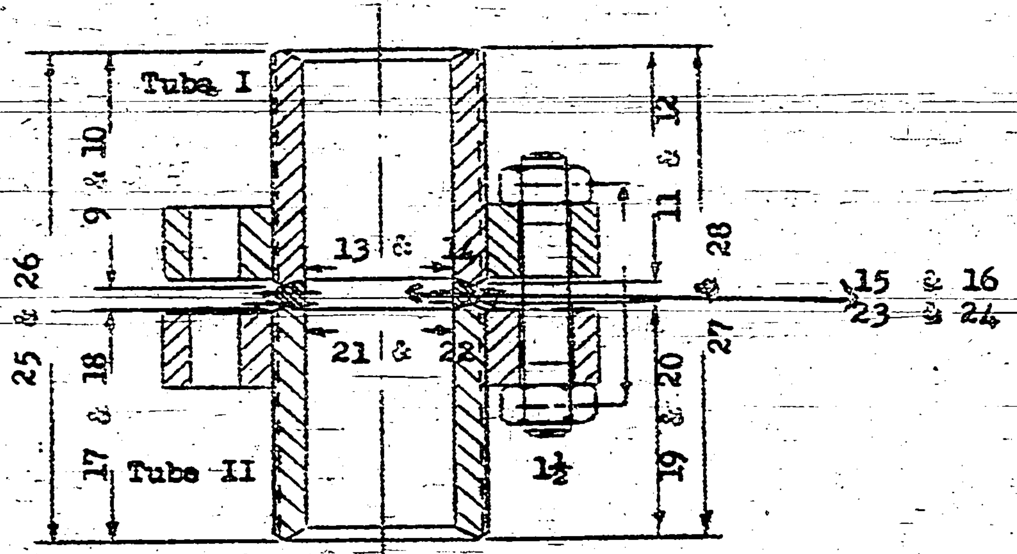
(Dimensions in m/m)

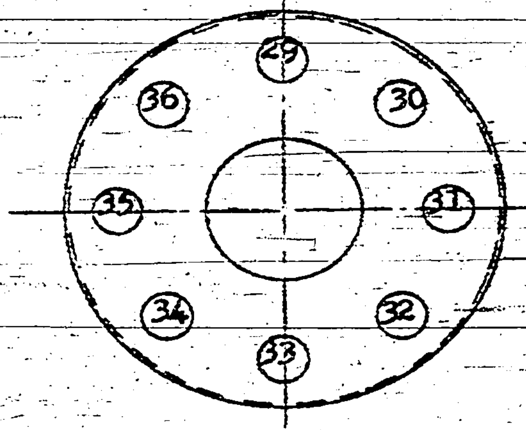
Test No.	Loads	Dimension Points Tube I 9	10	11	12	13	14	15	16
1.	Without pressure, before assembly	195.09	195.065	195.06	195.07	120.070	120.08	162.045	165.29
2.	Assembled, lightly drawn	(Not measurable until after dismantling)				120.07	120.08		
3.	With 50 kg. on a 700 mm lever arm		"			120.07	120.08		
4.	At 260 ats., leaking slightly		"						
5.	Pressure Released		"			120.07	120.08		
6.	With 100 kg. on a 700 mm lever arm					120.06	120.07		
7.	Pressure of 325 ats., middle oil and nitrogen		"						
8.	Pressure of 430 ats., oil		"						
9.	Pressure Released					120.06	120.07		
10.	Pressure 780 ats.		"						
11.	Pressure Released					120.06	120.08		
12.	Loosened	195.06	195.06	195.06	195.065	120.08	120.09	166.00	165.295
13.	With 100 kg. on a 1400 mm lever arm	(Not measurable until after dismantling)				120.06	120.07		
14.	With 150 kg. on a 1400 mm lever arm		"			120.03	120.04		
15.	With 195 kg. on a 1400 mm lever arm		"			120.02	120.02		
16.	One light tap		"			120.00	119.99		
17.	Two heavy taps		"			119.98	119.97		
18.	Three heavy taps		"			119.95	119.96		
19.	Four heavy taps		"			119.95	119.91		
20.	Loosened	195.065	195.06	195.05	195.06	120.04	120.05	166.00	165.260
21.	Permanent Deformation m/m	0.025	0.005	0.01	0.01	0.03	0.03	0.035	0.03

Dimension Points Tube II 17	18	19	20	21	22	23	24	25	26	Assembled 27	28
187.17	186.98	187.09	187.01	123.10	123.13	164.61	165.675				
(Not measurable until after dismant.)				123.10	123.13			396.7	397.0	396.7	397.0
	"			123.095	123.13			396.6	396.9	396.6	396.9
	"										
	"			123.10	123.14			396.6	396.9	396.6	396.9
	"			123.08	123.11			396.4	396.8	396.4	396.8
	"										
	"										
	"			123.08	123.11			396.4	396.8	396.4	396.8
	"										
	"			123.08	123.12			396.4	396.8	396.5	396.8
187.165	186.98	187.08	197.01	123.08	123.12	164.605	165.630				
(Not measurable until after dismant.)	"			123.06	123.08			396.5	396.5	396.3	396.6
	"			123.03	123.06			395.9	395.8	395.7	396.0
	"			123.01	123.03			395.5	395.3	395.2	395.5
	"			123.00	123.01			395.4	395.2	395.19	395.4
	"			122.99	122.99			394.9	394.8	394.7	394.9
	"			122.97	122.98			394.5	394.3	394.4	394.5
	"			122.97	122.98			394.3	394.0	394.2	394.3
187.140	186.915	187.08	186.93	123.09	123.11	164.59	165.65				
0.03	0.005	0.01	0.03	0.01	0.02	0.02	0.025				

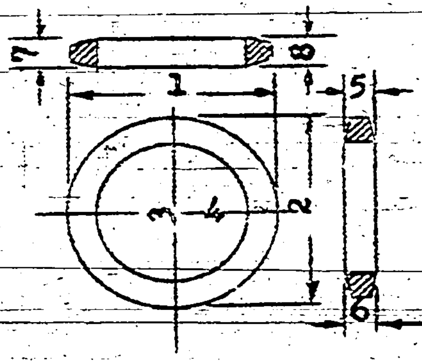
DIMENSION POINTS FOR DETERMINING THE DEFORMATION OF A 120 MM SEALING-RING OF N5 MATERIAL

W 2484





Flanges	N03
Tubes	N8
Sealing Rings	N5
Bolts	N03



For results of Test see
W-2485, W-2486, W-2487.

With Test Report of 12 July, 36.

T.O.M. Reel No. 5
pp. 162-165

T-63

Letter to the I.G. Farbinindustrie A.G.
March 4, 1937

INSTRUMENTATION FOR THE GAS PREHEATER OF THE EXTRACT HYDROGENATION STALL 17 OF THE LARGE SCALE PILOT PLANT AT WELHEIM

In the discussion between your Engineers Biegermann and Ledig and our Dr. Schulze on the first and third of March of this year in Ludwigshafen regarding the enlarging of the gas preheater in Leuna the following provisions were made for control measurements in the gas preheater of the extraction hydrogenation stall structures 5 and 17 of the large scale pilot plant, Welheim:

I. Amount of Fuel Gas.

The total amount of fuel gas is measured by means of a throttling damper and a pressure balance. The following has been set for the originally foreseen size of the gas preheater and the amounts of fuel gas:

"The total amount of heat introduced is 2,700,000 kcal/h; with heating values of the gas of

2,200 kcal/m^3, the volume of the gas is 1,230 m^3/h
3,500 kcal/m^3, the volume of the gas is 770 m^3/h
4,000 kcal/m^3, the volume of the gas is 675 m^3/h.

all referred to 0° and 760 mm mercury.

We may assume 1/6 of the normal amount as the minimum amount."

The amounts are increased for the enlarged preheater. We request information on amounts of fuel gas requirements for the enlarged preheater to enable us to make damper calculations:

Gas Pressure: P = 800-900 mm water column
Gas Temperature: About 15 - 20°C
Specific Gravity: About 0.79 kg/m^3 at 0° and 760 mm mercury
Diameter of Pipe Line: 150 mm. Should 150 mm diameter of the pipe line prove insufficient for the larger amounts of fuel gas than originally foreseen, we would request you informing us the size of the pipe line diameter which will be needed by us in the computation of the throttling slide.

II. Method of Operation.

The gas preheater contains a vertical arrangement of 4 circulation gas heaters, each one of which is equipped with 3 gas burners. The gas under a pressure reduced to 50 mm water is injected by means of air at 800 mm water pressure and is usually mixed before combustion. The circulation gas is introduced into the four heaters in a structure equipped with the 3 burners of the circulation gas heater.

When lighting the converter, the air damper is first opened and the converter purged with air. The pilot flame is then introduced or lit and only after that is the gas valve completely opened. The setting of the amount of gas is done by setting the air supply which is done by means of one slide for the 3 burners of a circulation gas heater.

1. The setting of the 4 air dampers may be done:

   a. Directly at the damper hand operation of the wheel.

   b. From a central location by using the Askania remote control, operating valve of the steering cylinder by hand.

There is in addition a hand operated coarse slide used for the general regulation of air supply.

2. The opening and closing of the gas valves can be made:

   a. Directly by hand at the valve.

   b. From a central location by using the Askania remote control, operating the steering cylinder by turning a valve by hand.

III. Safety Installation.

Should the blast be interrupted because of the shutdown of the electric power, or when the pressure in the combined air pipe line drops to below 200 - 300 mm water column, an automatic shut off valve will cut the gas supply from the fuel gas main before the gas is distributed to the burner.

2. In case of high excess pressure inside the gas preheater (explosion) that is when the pressure in the gas preheater exceeds 900 - 1,200 mm water column, there is again a quick shutoff of the gas supply at the main before its distribution to the burners by means of quick shutoff valves installed.

3. The quick shutoff valves mentioned under III, 1 and 2, may also be operated from a central location in case of general danger, for instance in case of fire.

4. A contact arrangement is to be built at each of the 2 circulation gas blowers of the circulation gas pump in Leuna, Böhlen and Magdeburg, which will start an alarm if the blower stops. The contact is closed by the dropping pressure in the suction pipe line of the pump which normally equals to 200 mm water column and becomes zero as soon as the blower stops. The alarm is sounded by a whistle with a simultaneous indication by a lamp. The location of the indicator lamp and of the whistle is to be determined at the plant (probably in the service department).

5. The pressure of the fuel gas (900 mm water column) is automatically reduced by a pressure reducing valve to 40 - 50 mm water column. In normal operations with a heating value of gases of 3,500 - 4,000 kcal/m3) of 150 - 200 mm water is sufficient, but with poor gas (about 2,200kcal/m3) a gas pressure of 500 mm water column is necessary when the burners are operated under full load.

IV. Temperature Measurements.

The following provisions must be made for the measurement of temperature:

1. For the gas path; of drawings L 2957-1, L 8497-2, L 8480-2, where the location of the thermocouples or their junctions are shown.

a. 12 thermocouples of iron-Konstantan for temperatures or mixtures of around 560°C, and which should under no conditions exceed 600°C. The junction of these thermocouples must be located in the center of the window through which the gas enters from the mixing chamber in the first path. Six thermocouples, namely the lower and the upper one inside of each circulation gas heater, are connected to the temperature indicators in the service stall. Four thermocouples, namely the middle one inside of each circulation gas heater, are connected through the contact galvanometer which produces an alarm when the temperature exceeds given limit.

b. Four thermocouples of iron-Konstantan for the gas temperature in the change from pass 1 to pass 2 of the gases. These thermocouples are so arranged that the junctions again are in the windows.

c. Four iron-Konstantan thermocouples to measure gas temperatures where the gas moves from the 2nd to the 3rd pass. These thermocouples are so arranged as to be again located in the centers of the windows in the 1st and 3rd passes leaving sufficient free way for the additional introduction of a hairpin tube as shown in the drawing L 8497-2.

d. Four iron-Konstantan thermocouples for the gas temperature at the outlet of the gas from the 3rd pass into the circulation gas pipe line, the junctions of these couples again located in the windows.

e. Two iron-Konstantan thermocouples for the circulation gas temperature at the intake of the circulation gas into the blower.

f. One iron-Konstantan thermocouple for the circulation gas temperature at the outlet of the circulation gas from the blowers which is a pressure pipe line from both blowers. All the thermocouples from b to f are also connected to the central instrument panel.

Leuna does not approve the location of the thermocouples in the gas preheater suggested by Ludwigshafen in the drawing L 7591-2, because of the metallic connection between the iron packing nut of the thermocouples with the high-alloy Sicromal protection tube, which may result in errors because of the thermo electric forces produced. The safety case provided is also considered undesirable, because it greatly interferes with the control of the thermocouples during operations. Leuna suggests an arrangement of the thermocouples which has already proven satisfactory at Böhlen and at Magdeburg and has introduced no difficulties in temperature measurements over there. Scheppart agrees with the opinion of Leuna. The total iron shell of the gas preheater is made gas-tight, and Eng. Riegermann suggests improvement in the packing of the the thermocouple openings with asbestos cord by using stuffing boxes with grounding rings and inserts of porcelain in order to prevent metallic contact between the thermocouples and the protecting tube. This form of insertion of the thermocouples has been shown in the sketch made on March 1st this year.

2. The finned tubes:

The wall temperature of the finned tubes should be measured in 7 locations, numbers 1 - 5 at two of the five hottest hairpins in the 1st pass and numbers 6 and 7 on one tube each of the two hottest hairpins of the 2nd pass. cf drawing M 2801-d. The number of finned tubes shown in M 2801-4 has been taken from your drawing N 1445-1, and does not therefore correspond to the enlarged preheater.

When introducing thermocouples for measurement of wall temperatures your sketch N 3949-2 should not be used, because thermocouples built in accordance with this drawing are no longer interchangeable without shutting out the finned tubes. Temperature measurements would be missing should for instance the thermocouples become defective during operation or when for any reasons whatsoever a testing of the thermocouples would be found necessary during operation. For that reason, Leuna suggests inserting the thermocouples in accordance with drawing M 2801-4 with the 6 x 13 protecting tubes for the elements not placed as in the drawing, at a distance of 75 mm, but as close as possible to the high pressure pipe. The exact construction will be again discussed with the main shops of the ammonia works where the finned tubes are being manufactured.

V. Pressure Measurements.

The location of the pressure measurement may remain as originally suggested, but with the recommendation that pressure pipe lines be not fixed in position, because accurate determinations of pressure are only possible with inserted riser pipes. The vertical riser is a tube closed in front and with about 3 - 5 mm clear cross section and which has a hole in front for transmission of pressure, about ½ mm in diameter. Whenever measurements are made these riser pipes must be inserted through the places provided into the gas space of the preheater.

Drawings number L 2957-1, L 8497-2, L 3949-2, L 8430-3, K 1445-1, M 2901-1 and the sketch for the insertion of the thermocouples are appended. We request your sending us the latest drawing of the enlarged preheater in which the temperature and pressure measuring places are shown in accordance with this letter. (The numbers of the drawings are very difficult to read; no drawing go with this article - Translator)

/s/ Strombeck
Wolfrom

Sternberg/MC/jw

7-29-1946

T-64

T.O.M. Reel No. 76
pp. 48 to 68

REPORT OF THE TECHNICAL TESTING LABORATORY AT OPPAU NUMBER 543, INVESTIGATION OF FINNED TUBES

Part 5.

Measurement of heat transfer on a piece of a hairpin after 2 years operation. Heat conductivity of the crust. Effect of finning upon the measurements or wall temperature.

June 21, 1943

SUMMARY.

A piece of finned tubes from the hairpin G72 was cut out from a gas preheater at Gelsenberg after 15,040 hours of operation, and has been investigated thermally. The crust closely adhering to the inside was 8 mm thick and reduced the heat transfer by about one-half. The share of the heat resistance of the crust in the total heat transfer has become so large that the finning of the hairpins had but a small effect upon the heat transfer. The temperature in the wall of the high pressure tubes was measured with movable thermo-couples and the drop in temperature in the parts of the tube without fins was established. The error of measurement of temperature in relation to the number of missing fins is represented by a curve. After many experiments the heat conductivity of the crust was found to be λ = 0.9 kcal/mh/°C.

1. Introduction. One hairpin was removed from the gas preheater at the Gelsenberg Benzene A-G. The dimensions of the tube were 90/171 mm and it was strongly encrusted, so that the N10 material was probably harmed by the high heat strains. A sample of this hairpin (G72) was submitted by Engineer Schappert for investigation to determine on it the heat transfer coefficient of a finned tube after 15,040 hours of operation and in addition strongly encrusted. The fins were but very little attacked. The accurate knowledge of the heat transfer of the incrustation deposited on the inside of the tube to a thickness of 10 mm was here of interest; the incrustation consisted of a thin deposit of iron sulfide with overlaying coal layers. Measurements of heat conductivity can be performed only on the undamaged parts of the crust, because the knocked off and crushed crust was porous and therefore would have lower λ values.

The question of wall temperature measurements of hairpin was particularly important. The thermocouples were fastened on the gas side of the surface of the tube and protected by insulation against the direct effect of gas. The best temperature measurement could be made by locating the element between the fins proper for which purpose naturally part of the fin would have to be removed to save space. A much simpler mounting of the element consists in locating them in the free surface of the tube welds which are left without fins for a length of 100 to 200 mm and where 7 - 12 fins are missing. (This arrangement was used at Pölitz.)

In the latter case the question arises to what extent heat may flow by "cross conduction" through the tube wall from the neighboring finned parts, or by how much the temperature may sink at the free parts of the tubes in comparison with the finned parts.

Dr. Seibert at Gelsenberg is trying in a work at present in preparation to determine theoretically the processes of cross conduction. The dependability of the computations in such an extremely complicated heat transfer problem will have to depend on assumption of the distribution of the heat transfer coefficients and the shape of the field of flow. At a suggestion of Dr. Seibert, flow experiments were carried out, the results of which were then introduced by him in accordance with the impulse theory, but the heat transfer values gave too high technical results. It appeared basically simpler to measure the temperature distribution through cross conduction in the tube wall using movable thermocouples in our testing laboratory. This would offer the advantage of direct determination of the unknown temperature distribution.

2. Arrangement of the Test. Two experimental finned tubes were cut out from the hairpin received. The first arrangements (for tests of September 16, 1942) are shown with the numbers 37 and 38 in our experimental layout shown on sheet 1. Both tubes were set side by side at a distance apart of 400 mm, the width of the path 710 mm. The fins at the left tube 37, are 206 x 310 mm in size, and are somewhat too small in comparison with the normal size of 270 x 320 mm on the right tube 38. The latter was therefore used in measuring the temperature distribution.

Tube 38 is again shown on sheet 2 where the most important tube measurements are given. The edges of the tubes are turned in the usual way for the retention of 2 screens in which 2 steel balls were placed for the improvement of the heat transfer. The iron-konstantan thermocouples are soldered on to small copper plates 10 x 10 x 0.5 mm, and these places fastened to the walls with pointed steel rods attached to some cross section of the tube. There are 3 couples arranged at the front and the back surfaces in the direction of the gas stream. The measuring places are marked with a cross in the drawing. Drill holes 6.5 mm diameter were made in 4 spots along the length of the tube wall of tube 38, and they were 610 mm down from the upper end of the tube. A thermocouple can be inserted into the drill holes. The thermocouple consists of iron-konstantan wires fastened in a copper cylinder. This cylinder enters directly into the drill hole and is pressed against the wall of the tube by means of small steel springs, which help it to reach the temperature of the tube wall because of the good contact. The location of the measurement is assumed to be at one-half the cylinder height.

Tube 37 had no drill holes in the wall.

3. Measurements and Results.

a. Determination of the outer heat transfer coefficient.

These experiments were performed in exactly the same way as all the earlier fin tube investigations (of the report of Dr. Kling of the Technical Testing Laboratory #463). The numerical values have been summarized in table 1. There are 3 series of tests. September 16, 1942, both tubes were tested in the encrusted state. Tube 37 was next removed and the crust drilled off. The test made on October 14, 1942, gave the comparative values between an encrusted and non-encrusted tube.

Tubes 37 and 38 were next interchanged and a comparative test was repeated on November 18, 1942. The upper figure on sheet 3 represents the outer heat transfer coefficient α_a. According to the former definition, the amount of heat Q, referred to the outer surface F_a of the tube and the fin, as well as to the temperature interval between the gas temperature t_g and the inside wall temperature t_w measured with a thermocouple, is

$$\alpha_a = \frac{Q}{F_a \times (t_g - t_w)}$$

The relationship between α_a values from the fuel gas velocity in the empty pass in front of the tubes w_{Rg} has been plotted. The encrusted tubes show a heat transfer only one-half as great as in the non-incrusted tube.* The heavy crust destroys all effect of the heat transfer from the gas side, as shown in the almost horizontal straight line. In contradition to this, the non-incrusted tube shows a marked increase of α_a with the velocity of the gas. The difference in the α_a values of the encrusted tubes 37 and 38 is a purely mathematical conception, caused by different F_a values as a result of the different number of fins and surfaces of the two tubes.

The latter viewpoint as well as the fact that with increasing incrustation the inner diameter of the tube, on which a small piece of w is measured, becomes smaller, make it appear profitable to give a new definition of the outer heat transfer coefficient in which the inner tube surface effect is introduced by F_i-tube as reference surface:

$$\alpha_{a\,i\text{-tube}} = \frac{Q}{F_{i\text{-tube}} \times (t_g - t_w)}$$

The recalculated heat transfer coefficients are shown in the bottom figure of sheet 3. Both tubes, 37 and 38, transfer heat alike when in the encrusted state. The character of the curve is no different from the one in the upper figure. 8 mm incrustation will reduce the heat transfer of the hairpins by about one-half.

b. Wall temperature measurements. These measurements were made by installing tube 38 with the crust on the right. Tube 37 on the left merely served to fill the pass. The encrusted tube was intentionally used in these measurements because this was the state in which it was present in the gas preheater after a longer operation period, with the circulation gas temperature sufficiently high to affect the strength of the tube material. In the tests of April 22, 1943, tube 38 still had all the fins, in the next test of May 8, 1943, 3 fins were removed from the

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* The expression used here: "outer heat transfer" is of course actually a heat transfer which is composed of several consecutive and parallel heat resistances. (cf report of Dr. Kling technical testing laboratory No. 508.)

center, while in the test of May 11, 1943-9 fins were removed, while on May 13, 3 fins were again welded on and 6 fins were missing. The flue gas temperature in all cases was 410°, the velocity of the flue gas in front of the tubes was 8.4 m/sec., conditions which roughly correspond to the conditions existing in a gas preheater. The air stream through the tube was the same in all cases, G = 290 kg/h and the air entered the tubes at room temperature.

The temperature curves of the 4 tests, of 4-22, 5-8, 5-11 and 5-13-43 are plotted in sheets 4 to 7, with the distance of the upper tube end in mm as the abscissa, the mv read on the ordinate. The cold junction of the iron-konstantan thermocouple was at 0° and had to be corrected for the drop of potential through the wire (the resistance of the wires was 6.5 ohm, of the instrument 884.3 ohm). A section through the finned tube has been sketched in, and the wall of the pass is indicated by line with cross hatching. The fully finned tubes have a parabolic temperature field, caused by the flow of heat to the cold end of the tube inside of this tube wall. In test with some fins removed the original parabola forms a saddle, this is caused by the reduced heat transfer on the unfinned tubes. The difference between the temperature of the parabola leg of the fully finned tubes and the temperature at the lowest part of the saddle in a tube with missing ribs, in relation to the total drop in temperature $t_g - t_w$, represents an error of measurement caused by placing the thermocouples in the center of the unribbed tubular pieces of the hairpin. It has been plotted in the upper part of sheet 8 against the number of the missing fins. With 9 fins missing the wall temperature of the hairpin would be 11.5 per cent too low, an error which would amount to 17° in the operation of the gas preheater with a temperature drop of about 150°C. An accurate measurement of the wall temperature of hairpins is therefore only given with thermocouples directly at the foot of the fins in the finned part of the tube.

In the lower figure on sheet 8 are shown the amounts of heat transmitted and the outer heat transfer coefficient as affected by the finning. Heat conductivity Q sinks accordingly by only 7 per cent with 9 fins missing. With a strongly encrusted tube the heat resistance is primarily given by the crust, while the outer heat resistance is relatively small. This curve shows that all the known relationship are only valid in the operation of the gas preheater as long as it is not yet fouled.

c. Heat conductivity coefficient of the crust. Measurements of heat transfer made April 22, 1943, permit a simpler determination of the heat transfer coefficient λ of the crust. According to the formula for cylinders

$$\lambda = \frac{Q \; \ell n \; (d_i/d_w)}{2 \, x W x \, \ell \, x \; (t_i \; - \; t_w)}$$

where d_i is the diameter of the inner wall of the tube, d_w is the inner diameter of the pass; ℓ is the length of the crust and t_i the temperature of the inside of the tube. t_i was determined by plotting the temperature parabolas in sheet 4 inside the pass and finding planimetrically the average wall temperature at that place of the tube as being equal to 316°C. The drop in temperature from the diameter of the drill holes the tube to its inner wall can be calculated from the formula for cylinder ($\lambda_{iron} = 20$) from which t_i is found to be 287°C. $Q = 3,940$ kcal/h, $\ell = 0.47$ m, d_i is 91 mm, $d_w = 72.8$ mm, t_w is 59°C. Substituting these data we get the heat transfer coefficient of the crust

$$\lambda = 1.3 \text{ kcal/mh/°C}$$

from an average temperature of 173°C and in the air.

An attempt has also been made to determine the heat transfer coefficient λ of the crust by another method. For that purpose part of the hairpin was cut off and the fins were removed. This piece of tubing still encrusted on the inside was fitted with an axially arranged electric heating and was installed airtight in a high pressure furnace. All hollow spaces were filled with insulation or with sand to reduce the convection streams. The wires from the heating and from the thermal elements were passed through insulated stuffing boxes. The high pressure tube could be filled with H_2 or N_2 (the use of oil also was foreseen) at 200 atms. It was expected to find by means of these λ measurements the effect of other materials than air inside the pores as well as to show the effect of temperature upon λ.

Different difficulties of measurement (convection on frequent destruction of the heating wires and of the thermocouples through the action of gases, etc.) as well as a shortage of personnel forced us to break off these time-consuming tests. Even though the few available values are rather inaccurate as can be seen from their considerable scattering, they are recorded here:

Measured in:		Hydrogen		Nitrogen	
Pressure	atm	166	178	154	214
Temperature	°C	48	99	109	85
Heat Conductivity λ	kcal/mh/°C	0.80	0.98	0.88	1.21

The inaccuracies are too great to determine from these values the effect of pressure or temperature. The only important fact is the recognition that the heat transfer of the crust under pressure in hydrogen and nitrogen is of the same order of magnitude as in finned tubes measured in air under ordinary pressure.

We may judge the accuracy of the λ values of these different measurements of heat conductivities of the crust to be within the limits of 0.8 to 1.3 kcal/mh/°C, with but a slight effect of the different media in the pores, of pressure and of temperature. A careful calculation of the heat transfer in the encrusted hairpins would give us

$$\lambda = 0.9 \text{ kcal/mh/°C}$$

This approximate value should be sufficient for practical calculations, when one considers that even large differences may be caused by differences in composition or porosity of the crust in the gas preheater.

Sternberg/mc/pkl
8-6-48

T-64

TABLE 1

Date	Hour	G	t_1	t_2	Q	t_w	α_a	α at tube	$t_g - t_w$	t_g	^{N}Rg	
		kg/h	°C	°C	kcal/h	°C	kcal/ $m^2h°C$	kcal/ $m^2h°C$	°C	°C	m/sec	
9/16/42	1140	284.5	19.0	75.5	3860	55	3.55	80.4	356	411	5.64	Tube 37, with crust, on the left
	1340	263.0	20.0	81.0	3850	59	3.61	81.7	350	409	4.42	
	1425	264.5	20.5	78.5	3710	56	3.43	77.6	355	411	2.72	
	1510	263.0	22.0	82.0		62	3.56	80.6	348	410	5.72	
	1555	268.0	21.5	81.5		63	3.65	82.6	347	410	6.74	
	1625	269.0	21.0					83.5	347	410	8.26	
	1720	275.0	22.0					79.3	353	412	3.12	
	1140								356.5	411	5.64	Tube 38, with crust, on the right
	1340								350	409	4.42	
	1425							75.5	352	411	2.72	
	1510							78.5	347	410	5.72	
	1555	264.5	21.5	81.5	3810	63	2.95	81.8	347	410	6.74	
	1625	266.0	21.0	81.5	3860	62	2.98	82.6	348	410	8.26	
	1720	267.3	22.0	79.0	3658	56	2.76	76.5	356	412	3.12	
10/14/42	1110	314.0	17.5		6570		6.6		317.2	410	4.78	Tube 37, no crust on right
	1200	313.0	16.0		6570	95.0	6.85		315.0	410	5.91	
	1410	308.7	21.5		6705	104.0	7.26	164.7	303.0	407	7.00	
	1445	308.7			6820	105.0	7.34		305.0	410	7.42	
	1500	310.2	20.0	115.0	7080	104.5	7.69	174.1	302.5	407	8.22	
	1640	308.0	20.0	116.5	7140	107.9	7.76	176.0	302.1	410	9.05	
	1720	311.0	23.5	108.0	6310	99.5	6.76	153.2	306.5	406	3.02	
	1110	306.3	17.5	68.5	3755	49.2		77.4	360.8	410	4.78	Tube 38 with crust, on the right
	1200								360.5	410	5.91	
	1410	301.3	21.5	74.0	3790	55.8	2.90	80.2	351.2	407	7.00	
	1445	298.2	21.0	74.5	3832	56.0	2.91	80.5	354.0	410	7.42	
	1500	299.0	20.0	74.5	3915	56.0	2.99	82.9	351.0	407	8.22	
	1640	297.2	20.0	75.0	3925	56.5	2.98	82.5	353.5	410	9.05	
	1720	296.8	23.5	74.0	3600	54.0	2.75	76.2	352.0	406	3.02	
11/18/42	1130	294.0	5.5	59.5	3810	40.0	2.72	75.4	375.0	415	4.70	Tube 38, no crust, on the right
	1300	311.5	6.5	58.0	3850	43.8	2.75	76.2	375.2	419	4.00	
	1410	317.0	7.0	54.5	3615	40.0	2.61	72.4	372.0	411	3.13	
	1520	316.0	7.0	58.5	3900	40.0	2.81	77.9	373.0	412	4.64	
	1620	315.5	7.0	59.0	3938	41.0	2.85	79.0	371.0	411	5.71	
	1700	317.5	7.0	60.0	4040	44.0	2.92	81.0	372.0	416	6.77	
	1720	318.5	7.0	60.5	4090	41.0	2.96	82.0	371.0	411	8.79	
	1130	288.0	5.5	100.0	6540	89.0	6.59	149.1	326.0	415	4.70	Tube 37, no crust, on the right
	1300	302.0	6.5	94.5	6380	88.6	6.34	143.6	330.4	419	4.00	
	1410	306.0	7.0	91.0	6180	84.8	6.20	140.5	327.2	411	3.13	
	1520	302.5	7.0	100.8	6815	90.0	6.93	157.0	323.0	412	4.64	
	1620	304.5	7.0	102.5	6965	88.8	7.08	160.3	323.2	411	5.71	
	1700	307.5	7.0	105.0	7240	95.0	7.41	168.0	321.0	416	6.77	
	1720	310.0	7.0	106.0	7370	95.0	7.64	173.0	317.0	411	8.79	
4/22/43		293.0	19.5	75.5	3940	65.3	2.97	82.2	344.7	410	8.35	Tube 38 on the right side w/the crust.
5/8/43		294.5	20.5	72.0	3647	61.0	3.14	77.8	349.0	410	8.50	
5/13/43		287.5	35.5	89.8	3752	83.0	3.89	85.3	327.0	410	8.39	
5/11/43		297.0	21.0	72.0	3642	58.0	4.06	77.0	352.0	410	8.43	

"Blanks" left where figures not legible.

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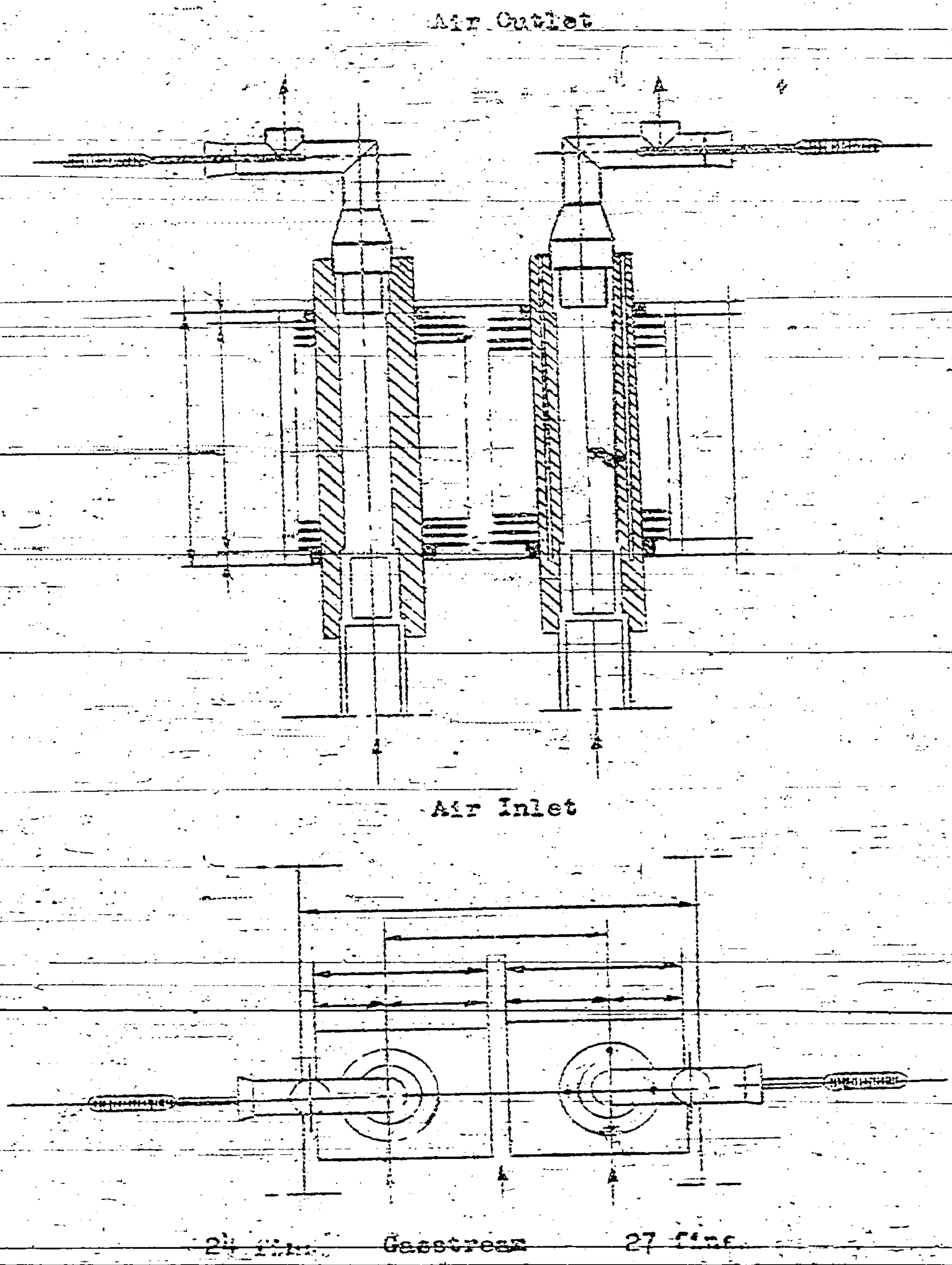
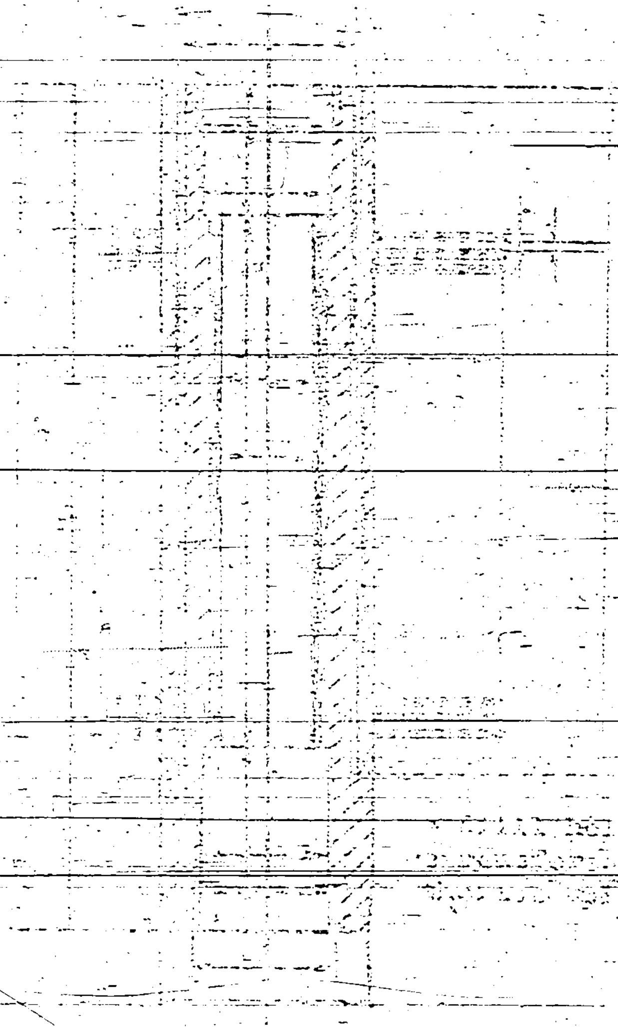
Sheet 1

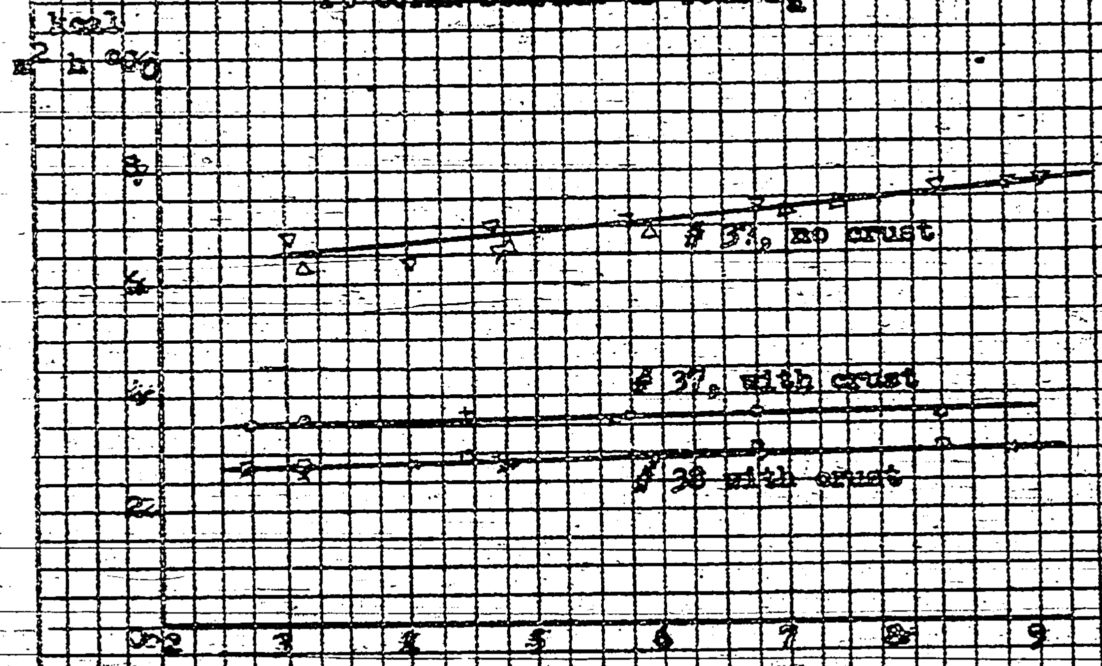
INSTALLATION OF THE FINNED TUBES IN THE PASS



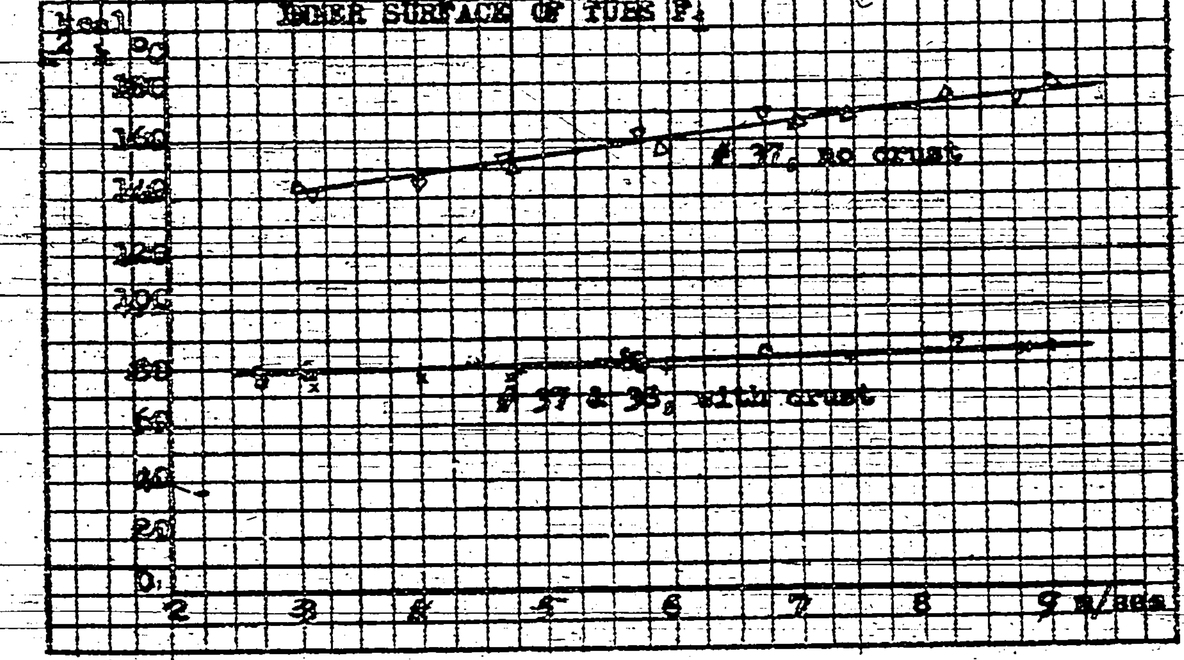


[illegible] No. 75

OUTER HEAT TRANSFER COEFFICIENTS REFERRED TO OUTER SURFACE OF TUBES F_a



OUTER HEAT TRANSFER COEFFICIENT, REFERRED TO INNER SURFACE OF TUBES F_i

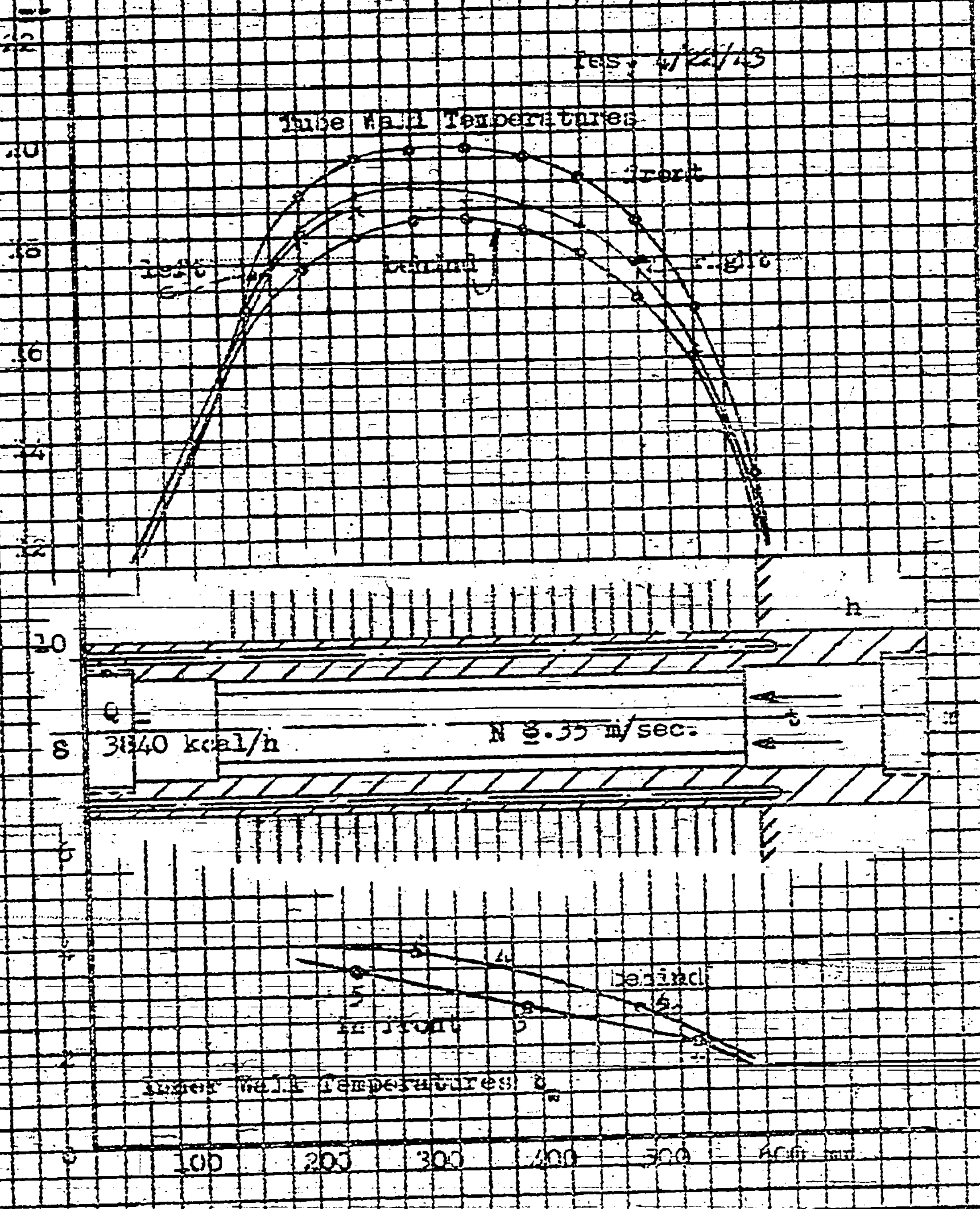


Technical Testing Laboratories
Oppau

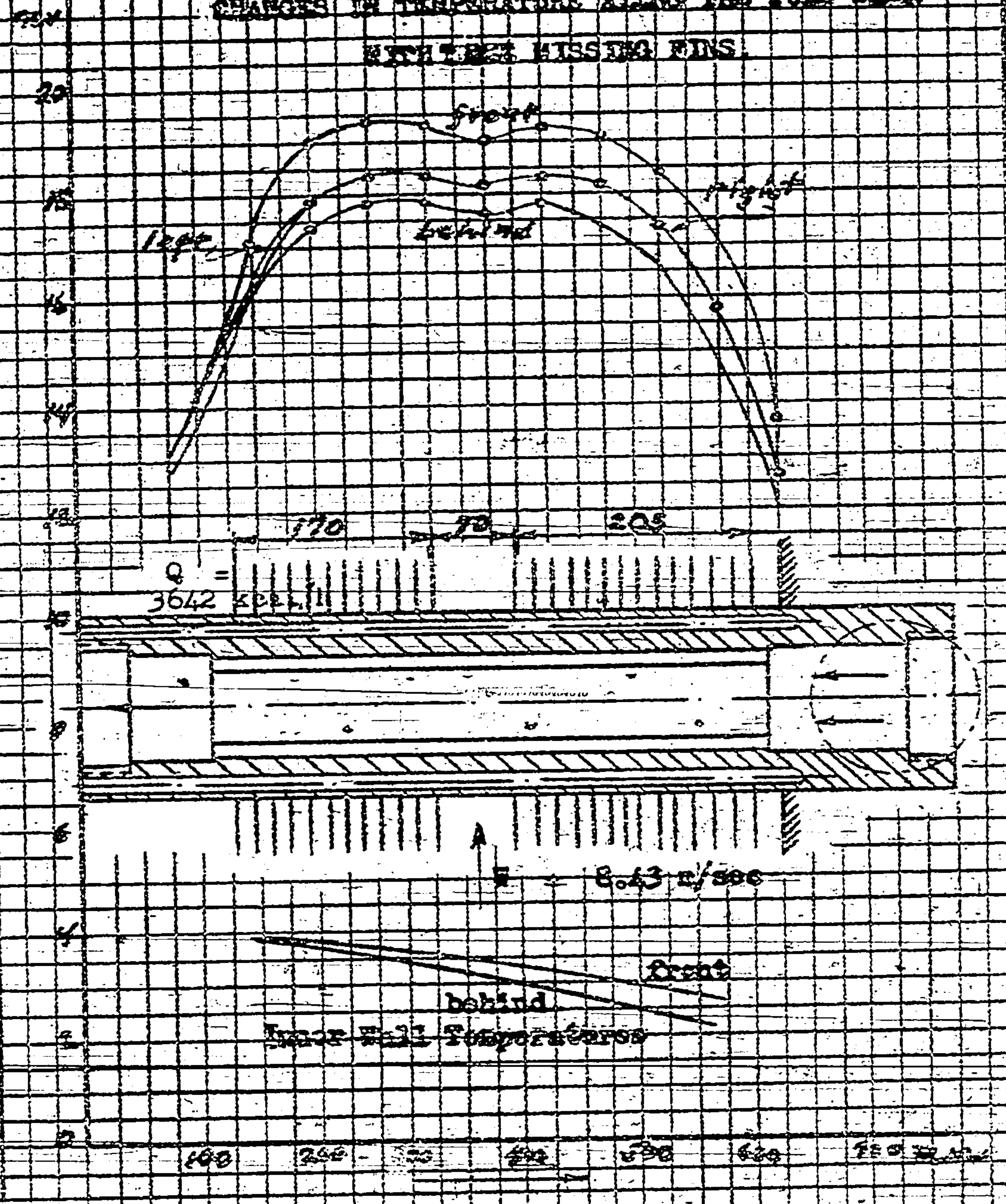
Sheet 4.

CHANGES OF TEMPERATURE ALONG THE TUBE WALL

Tes. 4/22/43



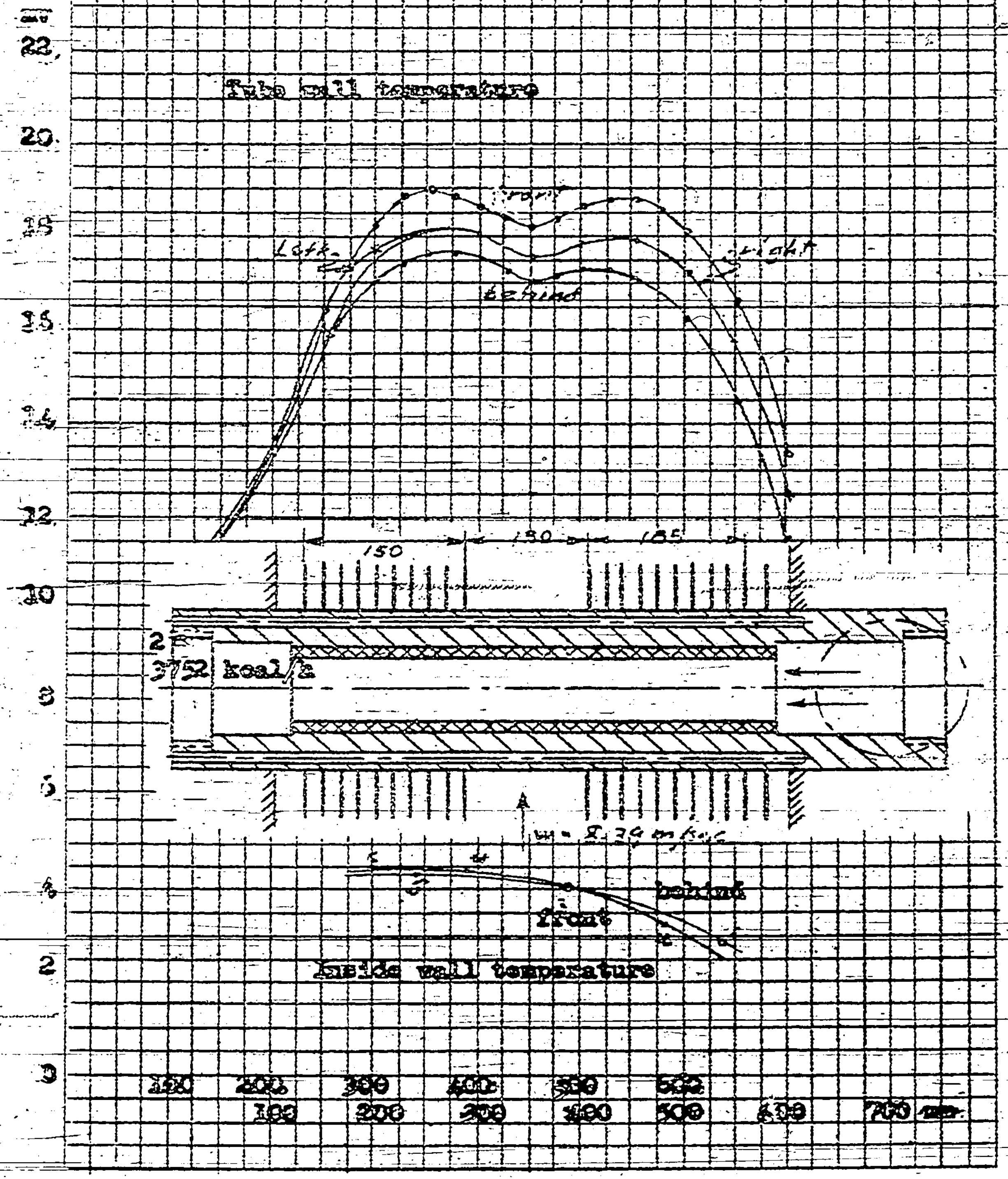
CHANGES IN TEMPERATURE ALONG THE TUBE WALL WITH THREE MISSING FINS.



Sheet 6

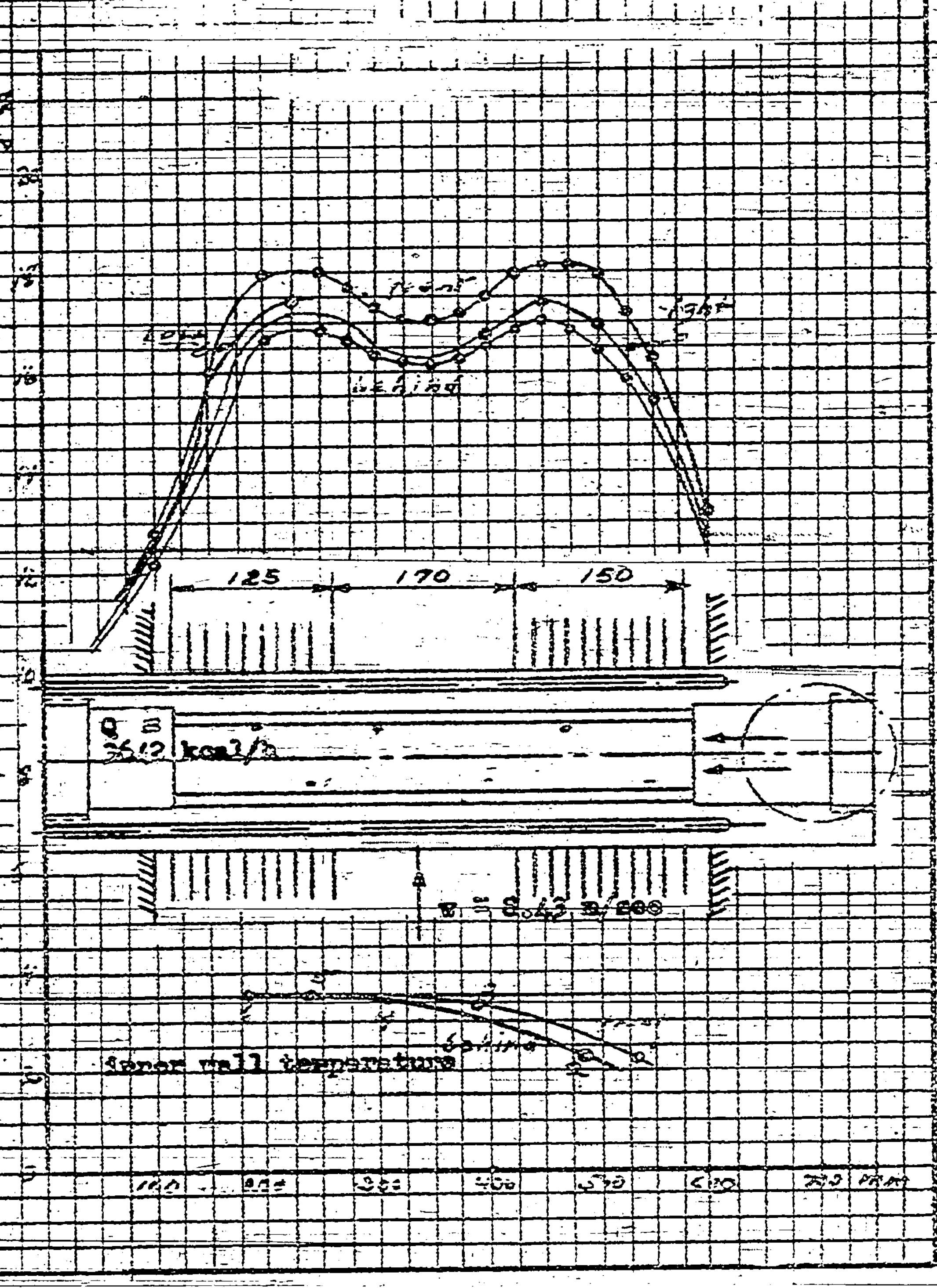
T-6i

CHANGES IN TEMPERATURE ALONG TUBE WALLS
WITH SIX MISSING FINS



TEMPERATURE CHANGES ALONG TUBE WALL WITH 9 MISSING FINS

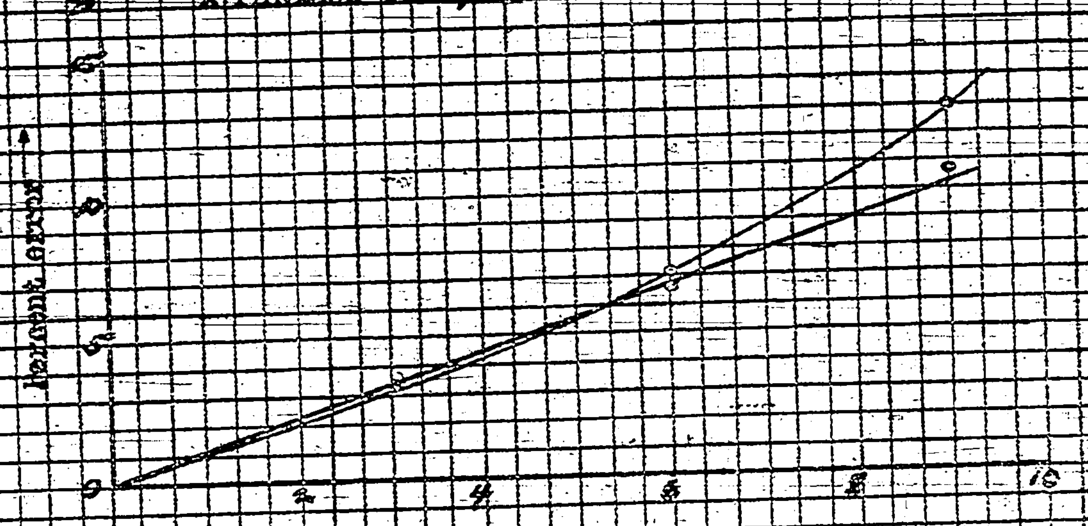
Sheet 7



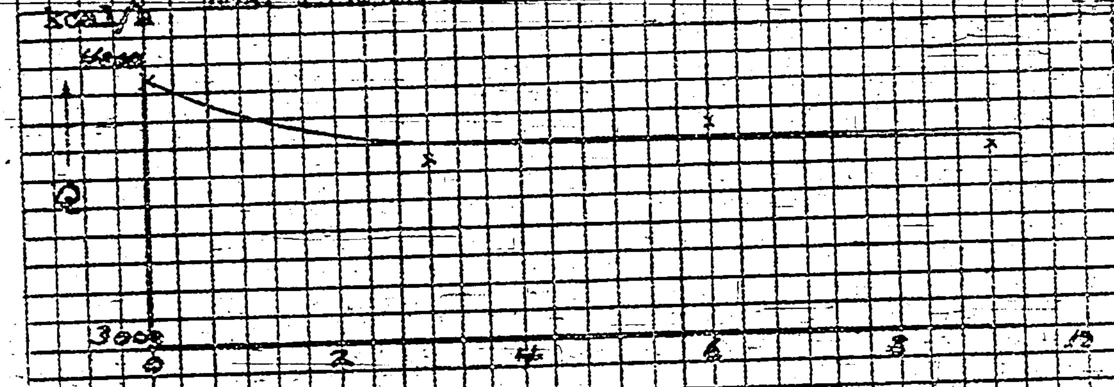
Sheet 8

T-64

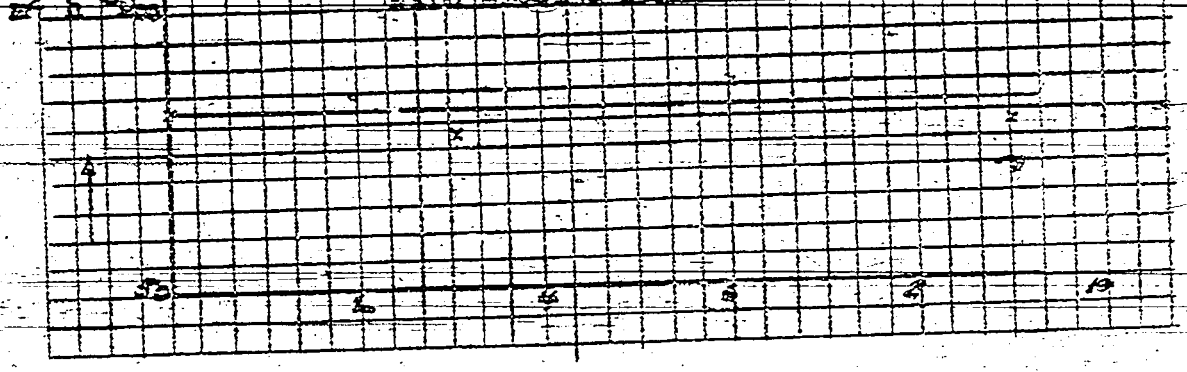
PERCENT TEMPERATURE DROPS IN CENTER OF A FINLESS TUBE, AGAINST FINNED PIECE



HEAT THROUGH A TEST PIECE WITH MISSING FINS



HEAT TRANSFER COEFFICIENT OF TEST PIECE WITH MISSING FINS



T.O.M. Reel No. 78
Bag 4
Target 30/4.13
Pages 13 to 21

REPORT ON A LETDOWN NEEDLE-VALVE

Stettin-Pölitz, 21 October 1942

The hot oil letdown of the liquid phase chambers is now generally accomplished by the use of needle-valves NW 45 (45 mm), drawings N 5910a-2 and 9645a-2. (These drawings not available). The letdown is made through a cylindrical opening, 10 mm diameter by 40 mm long, which is lined with hard metal. Not over 30-40 hours operating time was achieved at Pölitz with these valves. After this time, either the supporting shell for the hard metal inserts, the holding nut for the nozzles, or the supporting ring for the nozzle body, was so damaged that the letdown could no longer be performed in the regular manner.

Because the hard metal parts (Wallramit from Meutsch, Voightlander & Company, Essen) showed practically no wear during these operating periods, we now tried to protect the other valve parts by means of liners. For the time being, the component parts were left in their existing form, but were protected on their stream side by welding on a layer of Tizit or Guronit. The improvement in their life duration was inconsequential because operating times of only 60-70 hours were attained; the damage to the material remained the same.

Because it was difficult to procure supporting rings of NC-material, we tried to retard the wear by inserting liners into the N8-body of the sealing ring (lens). The material for these liners was hardened tool steel, Guronit or Granitherm castings. The life duration of all of these three materials was about 170 hours, which was still unsatisfactory.

In a further experiment the pressure drop was sub-divided by the arrangement in series of orifice plates with hard metal inserts. For this purpose orifices of 10 and 15 mm diameter respectively were built in behind the nozzle proper at intervals of about 400 mm and the needle valve at the letdown vessel was replaced by three orifice plates at intervals of 400 mm, and 8, 10 and 15 mm diameter respectively, the largest orifice being located at the valve inlet. While the needle valve at the letdown vessel could be successfully replaced in this manner, the use of the orifices in the stall-letdown brought no immediate improvement, because it was shown that the nozzle cross-section required for the successive pressure reduction could not be made to agree with that required for the thru-put volume.

Since the aforementioned experiments, of which only the most important are indicated, led to no satisfactory results, we endeavored to arrive at a satisfactory solution of this problem by influencing the flow conditions, and by a corresponding hydro-dynamic shape of the nozzles. The manner in which the wear on the nozzle supports occurred (Figure I) led to the idea that a process similar to that caused by sound waves was taking place. This conclusion was particularly supported by the fact that the worn surfaces had assumed the form of a parallel-stream nozzle. If we consider that in a pressure drop during the letdown the gases dissolved in the letdown are freed (particularly hydrogen) and the lighter boiling constituents vaporized, it may be visualised that a considerable volume-enlargement of the substance discharged from the nozzle takes place. If it is required that in the same unit of time the same quantity of the mixture pass through the nozzle, the pressure will decrease with increasing velocity, according to Bernouilli's equation, and a further volume-enlargement will result. This proves that the cross-sectional area of the stream must increase with rising velocity. It seems obvious, therefore, that stationary sound waves (over-expansion and its equalization by means of succeeding thickening shocks, so-called "Mach"-waves) are present. These have their start particularly on the edges or uneven spots of the walls enclosing the stream. It may now be readily visualized that the outlet of a cylindrical nozzle is the starting point of such waves. The conditions in the plane of the outlet of the 10 mm cylindrical nozzle are similar to the discharge of powder gases from the mouth of a gun. From photographs taken of a gun discharge, the form of the discharged stream, first calculated in 1907 by Prof. L. Prandtl, may be recognised. (Figure II). These observations now led to the decision to build the letdown-nozzle so that the course of the pressure is a constant function of the nozzle length, that is, opposed to the erstwhile cylindrical construction, in which the course of the pressure was unsteady, the nozzle received a conical form. It is of deciding importance that the angle of the opening not exceed 10°, so as to assure a positive contact of the stream on the enclosing nozzle walls under all operating conditions.

Experience with the letdown nozzle shown on drawing P 1021-16 clearly indicated that the inlet edges on the side of the closing cone must be well rounded to assure positive contact of the stream since otherwise cavitation would appear because of the formation of a hollow space at the stream change. We should also aim to apply a fine finish to the surfaces coming in contact with the stream, because, as mentioned above, every uneven spot is the start of destructive "Mach"-waves. The shape of the valve cone (disc) should be as streamlined as possible. Because of construction difficulties a compromise solution shown on drawing P 1036-16 had to be chosen.

In the present construction with lantern nuts the chosen valve cone form is not of deciding importance, since considerable inlet disturbances occur, due to the lantern. The wear in the lantern caused by these disturbances, was mitigated by a hard metal liner inserted in the lantern opening. It is therefore desirable to reduce these inlet disturbances to a minimum by the omittance of the lantern. We have made an agreement with Meutsch, Voigtländer & Co. on the corresponding form.

With the aforementioned firm, 3 additional nozzles coming successively closer to the ideal form were agreed upon:

The first construction is shown in drawing P 1036-16.

The second form provides for the one-piece construction of the hard metal inserts with the cylindrical part shortened to a length of 10 mm; the valve stem would have to be made to fit.

The third construction merely provides a practical way out of the shop difficulties, if a one-piece casting should not be possible. It provides that the nozzle be assembled of individual rings. It should be noted that in all of these forms the hard metal insert is shrunk into the supporting jacket.

Results:

The nozzles made according to drawing P 1021-16 have given very satisfactory operating times. These have reached a thousand hours to this date at 15 to 20 m3/h letdown volume. Findings based on the dismantling of the nozzles tend to confirm our expectation of considerably larger operating times, since only very slight wear was noticeable. Neither did the valve body nor the letdown tube show any damages of any kind.

The changes noticeable in the hard metal are as follows:

1.) Inlet side of the nozzle with scarfed surface. (The stream forms its own hydrodynamically correct form).

2.) The formation of small flat grooves immediately behind the inlet in the cylindrical part of the nozzle. (This must be considered a form of cavitation. The hollow space required for this is caused by the change of the stream flowing around the comparatively sharp edges.)

3.) At the points of thrust, or impact, of the 2 nozzle parts the fluid enters the hollow space behind it, causes, however, very little damage here.

4.) Nozzle arrangements, with and without throttling disks (orifice plates) of 10 and 15 mm diameter behind the nozzle, were compared with each other. In the arrangement with the throttle disks no changes were observed while in the other case slight point shaped cavities were found in the nozzle outlet.

It is to be expected that the form of nozzle shown on drawing P 1036-16 will show further diminution of the 4 changes just described, particularly if the surfaces enclosing the stream are finely finished.

Summary:

The form of needle letdown valve in use up to this time could not meet the operating demands, because operating times of only about 30 hours were achieved. Therefor, a number of experiments were conducted with the object of reducing the wear caused by the letdown. The nozzle form shown on drawing P 1021-16 was developed after the most far-reaching adaptation of hydrodynamic requirements. With this nozzle, whose form may still be improved, a thousand operating hours have been achieved with practically insignificant wear. The findings indicated in its dismantling led us to expect a further lengthening of the operating time.

It should be noted, that as a result of this considerably longer operating time a recheck of the condition of these needle valves at longer intervals is made possible. A further result is that the shutoff valves now need to be changed only at longer intervals.

Technical Department High Pressure Stalls.

/S/ Bernau

KCBraun/MC/jw

7-30-1946

Appendix 1 & 2 T-65

Anlage 1

Bild 1

FIG. 1.

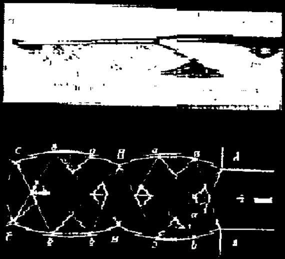


Bild 2

FIG. 2.

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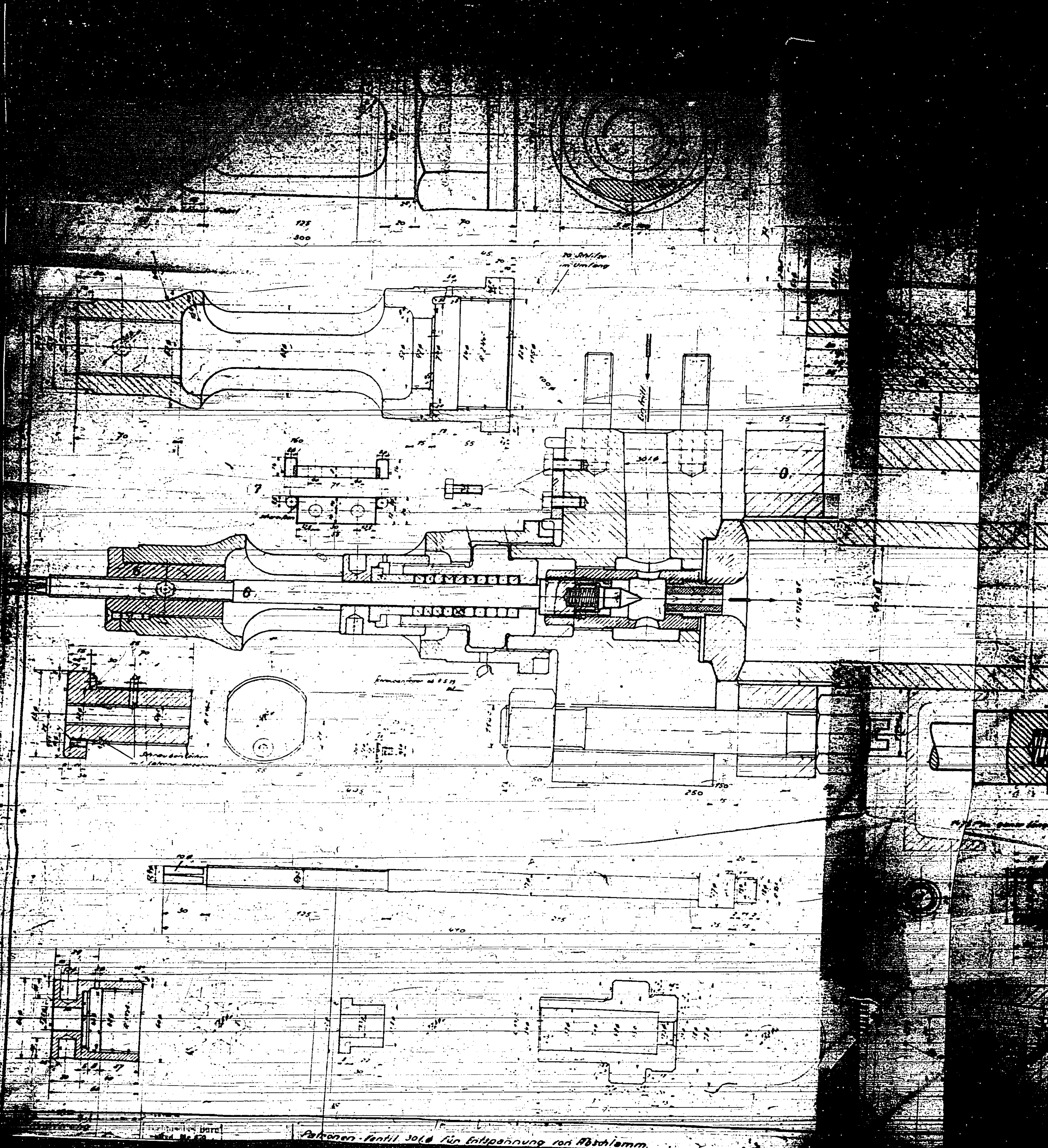
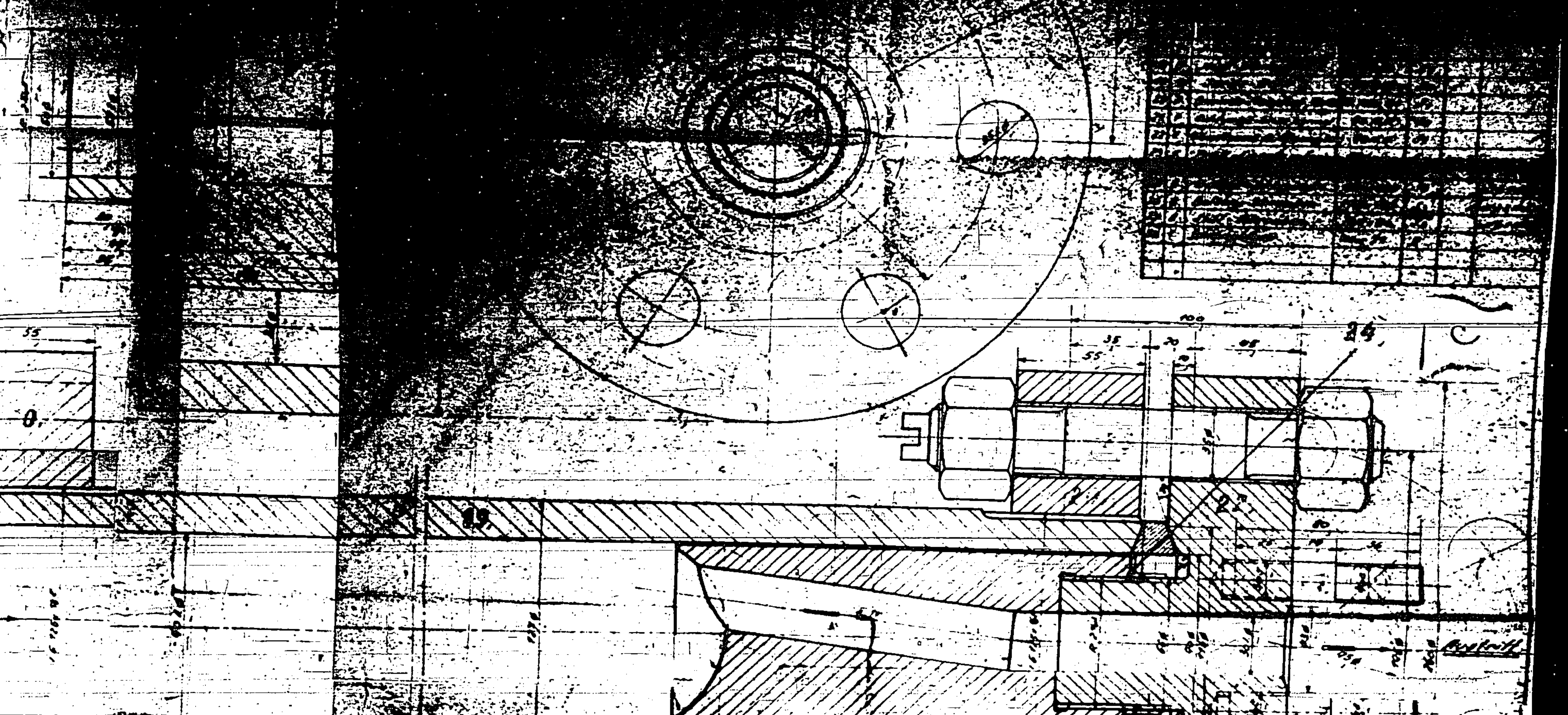


Patronen-Ventil 30 l.ø für Entspannung von Abschlemm.



Ammoniakwerk Merseburg G. m. b. H.

M 1871 d — 1

stoffverbrauch geprüft:

591

T.O.M. Reel 145 T-66

pp. 487-488 February 23, 1945

High Pressure Experiments
Lu i.

MEANS FOR IMPROVING HEAT EXCHANGERS IN THE SUMP PHASE

Reply to Dr. Peukett, Rheinbraun.

The hydrogenation works Wesseling have experienced difficulties in the coal stall heat exchangers, resulting from low heat transfer values caused by depositions of the catalyst (Bayer mass) and coal paste solids in the forward pass of the cold heat exchanger.

The following means have been used lately to counteract it:

1). Charging the cold heat exchanger with gas alone;

2). Increasing the flow velocity by reducing the size of the bundle (increasing the velocity of flow from 0.4 to 0.8 m/sec);

3). A downward instead of an upward flow in the forward passage.

Experience has shown that increasing the rate of flow had no desirable results, but reversing the direction of flow proved beneficial.

On the strength of this experience Wesseling has developed a new heat exchanger in which the paste and the gas enter the forward pass of the heat exchanger separately, with the paste moving downward from above and the gas upward from below. The new heat exchanger operated for 12 days with no reduction of the K value prior to the closing of the works. Moreover, the K value obtained was better than any previously measured and the demands on the preheater were low.

The following must be noted:

The rate of flow was increased by using only 108 instead of 199 tubes, i.e., with the same thruput, not only the outward flowing paste-gas mixture, but also the distillate flowing on the inside required less time and surface for heat transmission than with the usual heat exchanger construction. This counteracted the effect of the reduced deposition.

According to our past experience, the downward flow in the forward pass of the heat exchanger with a downward flow in the return pass as well should have lowered the production capacity of the heat exchanger with regard to the final temperature in the forward pass. Dr. Peukert's report does not state whether the downflow was retained in the return pass. Changing the outlet of the paste-gas mixture over to the bottom end of the heat exchanger favors the removal of any incipient deposition. Dr. Peukert gives on us no information on the emptying of the forward pass with this direction of flow.

The degree of filling and the fluidity are definitely improved by the method recently introduced in Wesseling when compared with the former combined downward flow of the paste and gas, and a good removal of the initial deposition is assured. The coalescence of the gas bubbles which swim in the paste possibly proceeds differently when gas is added to the exchanger than when the gas had been previously mixed with the paste, possibly forming a cushion in the narrow tubes which remain in the exchanger. This may affect readily the formation of a deposit (settling) (drying) as well as the heat transfer by contact with the hot wall.

(The last sentence is illegible).

Sternberg/jw
8-7-46

T-68

T.O.M. Reel No. 162

Frame No. 577-580

pp. No. 757-760

MOUNTING THERMO-ELEMENTS IN ELECTRIC PREHEATERS

Ludwigshafen on the Rhine, Jan. 25, 1943

Exchange of experiences on electric preheaters:

We refer you to damages to electric preheaters of which you have previously been informed. Because the control of the preheater tube wall temperature is important, we send you herewith in the appendix the arrangement of the thermo-elements as it is done at Werk Böhlen.

I. G. FARBENINDUSTRIE A.G.

/s/ Zimmermann

On the occasion of the conference for the exchange of experiences in Ludwigshafen on the 10th and 11th of December, 1942, the temperature control of the electric preheaters was also discussed in detail. On this occasion Dipl. Ing. Schappert asked that the improved method of mounting the thermo-elements on the individual shanks of the preheater hairpin tubes developed by us be submitted to him.

The method of mounting of these new elements may be seen on the attached sketch.

Before we adapted this new method of mounting, the mounting was done in the usual way, which caused many interruptions. In particular, ~~a part~~ some of the thermo-elements were torn off by the settling of the cinder wool after operations had been under way for some time, causing interruptions in the temperature measurements. This is being avoided by the new mounting method.

A strip of asbestos is applied over the element mounted on a sheet of mica, the asbestos strip fastened by an asbestos-covered wire and protected by a shell. To this shell is attached a steel tube of 25 mm inside diameter, through which the thermowires lead to the connecting clamps in the preheater jacket. To assure good insulation in all cases, these steel tubes, which are brought to within about 30 mm of the preheater jacket, are further protected by a refractory protecting tube at the preheater jacket. The steel protecting tube welded to the shell is filled with asbestos insulation, which is soaked in water glass at the ends, so that the thermo-measuring-wires are protected against damage and contact with steel parts on their way to the clamps.

At present the mounting is not done in place, as formerly, but the elements are assembled in the workshop and the solder points pasted between sheets of mica on an asbestos insulation, which is enclosed within one-half of the shell of the element holder. The details may be seen on the sketch. The construction of the elements in the workshop is done so that after the preparation of the shells and tubes the required lengths of tubes in the preheater may be measured, followed by a pre-assembly on a mounting tube. After the moist insulation has been thoroughly dried out the individual shells are removed from the mounting tube and can now be attached to the preheater tubes without the necessary cumbersome and time-consuming fitting at present required.

The steel protecting shell serves a second important purpose; it serves to repel the induction current acting in the preheater. Experiments have shown that measuring points with protecting tube show a temperature ½ mv lower than measuring points without a protecting tube. The temperature indication itself is also much steadier.

This method of measurement has been successfully used by us within the past year.

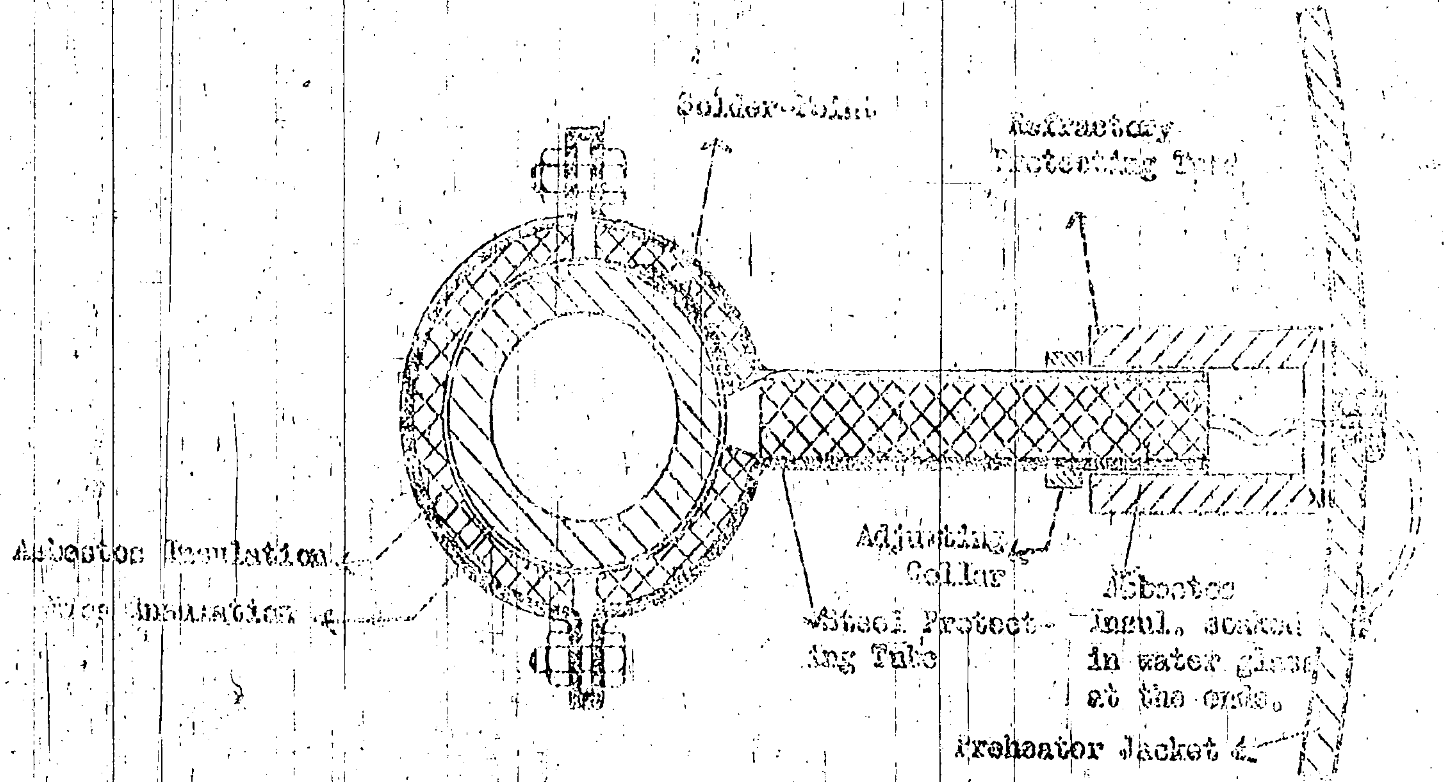
Braunkohle-Benzin A.G.

/S/ von Felbert and Hartmann

KCBraun/MC/jw

7-30-1946

[illegible]


Solder-Joint
Refractory Protecting Tube
Asbestos Insulation
Pipe Insulation
Adjusting Collar
Steel Protecting Tube
Asbestos Insul. soaked in water glass at the ends.
Preheater Jacket

T.O.M. Reel No. 75 T-69

pp. 400-416

Hydro. Works, Pölitz

Pölitz, October 14, 1943

ECONOMIES OF PASTE HEAT EXCHANGE OF A STALL

The concentration of the 2 partial streams of thick and thin paste are of deciding influence on the economies of paste heat exchanging. For this reason the very low concentration process with 35% in the thin paste was considered as the lower limit and compared with a process involving 40% thin paste, which however should not be construed as the upper limit. We compare below this method of paste heat exchanging (a) with the Nordstern method where only the intake gas is subjected to Heat exchanging (b).

Thru-put.

Total paste,	te/h	44.5
Total solids in	%	45.5
Fresh coal solids TK_0 in %		27.0
Gas through Heat exchanger,	m3/h	26.000
Gas to thick paste,	m3/h	4.000
Gas to hot catchpot,	m3/h	8.000
Gas to the stall outlet,	m3/h	55.000
Catchpot feed (exclusive of H_2O),	te/h	22

		a) Paste Heat Exchange		b) Gas Heat Exchange method of Nordstern
Thin paste concentration	%	38	40	
" " "	te/h	23	27	
Thick paste concentration	%	52	54	
" " "	te/h	21.5	17.5	
Heating during heat exchange	°C	122→415	122→415	30 → 420
Heat gain during heat exchanging,	10^6kcal/h	7.0	8.0	3.4
Heat supplied in pre-heater,	10^6kcal/h			
a) Useful		5.0	4.1	8.5
b) Effective = 75%		6.67	5.5	11.5
Resulting saving in paste heat exchanging against case b,	10^6kcal/h	4.83	6.0	
or fuel gas saving (H = 1450),	m3/h	3.300	4.150	
Required hairpins,		21	17	27
Saving in comparison with b,		6	10	--
Electric power for blowers,	kW	400	400	500
Saving in comparison with b,		100	100	
Cooling in cooler,	°C	198 →55	166 →55	320 →55
Cooler yield,	10^6kcal/h	4.5	3.5	8.3
Saving in comparison with b,	10^6kcal/h	3.8	4.8	
Saving in water	m3/h	85	105	

Comparison of Costs

a) Installation cost increase

2 heat exchangers	400,000	
1 paste pump	100,000	
Total	500,000	

Reduction in installation cost

Hairpins	180,000	300,000
Pipelines	80,000	
"	40,000	
"	10,000	
"	30,000	
Total	340,000	460,000

The preheater may be designed right from the start for a higher concentration of the thin paste by equipping it with 17 hairpins, in which case the increase and reduction in costs become practically equal, and the difference amounts to only 15% of the estimate.

b) Operating costs
Saving in case a)

	a) Paste Heat Exchanging 38%	40%
1.) Cooling water RM/h	85 x 0.01 = 0.85	108 x 0.01 = 1.08
2.) Power (blowers) kW/h	150 x 0.02 = 3.0	150 x 0.02 = 3.0
3.) Fuel gas, at the cost of fuel gas of:		
1,000 heat units = 1.2 Pfg.	57.5	72.0
1.0 "	48.0	60.0
0.8 "	38.0	48.0
0.6 "	29.0	36.0

This shows that the saving in cooling water and electrical energy is entirely subordinate to the saving in the fuel gas.

4.) The saving in relationship to the fuel gas prices per hour and per year with 8,000 operation hours is shown in the following table. The possible additional repairs with the paste regeneration are added in comparison to the saving in the repair-time which are balanced against the value of the saving. It is based on the hourly catchpot shutdown for the finished gasoline per 6.5 to stall with the price per ton of about RM300. There is in addition, savings in power and raw products, amounting to about 50% are shown, so that the shutdown per stall amounts to RM975/h or RM23,400/day.

Pfg./1000 heat units	1.2	1.0	0.8	0.6
Saving RM/h with				
38% paste	61.35	51.85	41.85	32.85
40% paste	76.08	64.08	52.08	40.08
Saving in 1000 RM/yr. with				
38% paste	490	415	350	263
40% paste	610	514	418	320
Days of repairing with				
38% paste	21	17.5	15	11.5
40% paste	26	22	18	13.5

The above table is represented graphically in the appendix.

It shows the economy increases vary strongly when the thin paste concentration is raised from 38 to 40 per cent solids.

We may counter the possible objection that any saving will be nullified by the shutdown of the heat exchange:

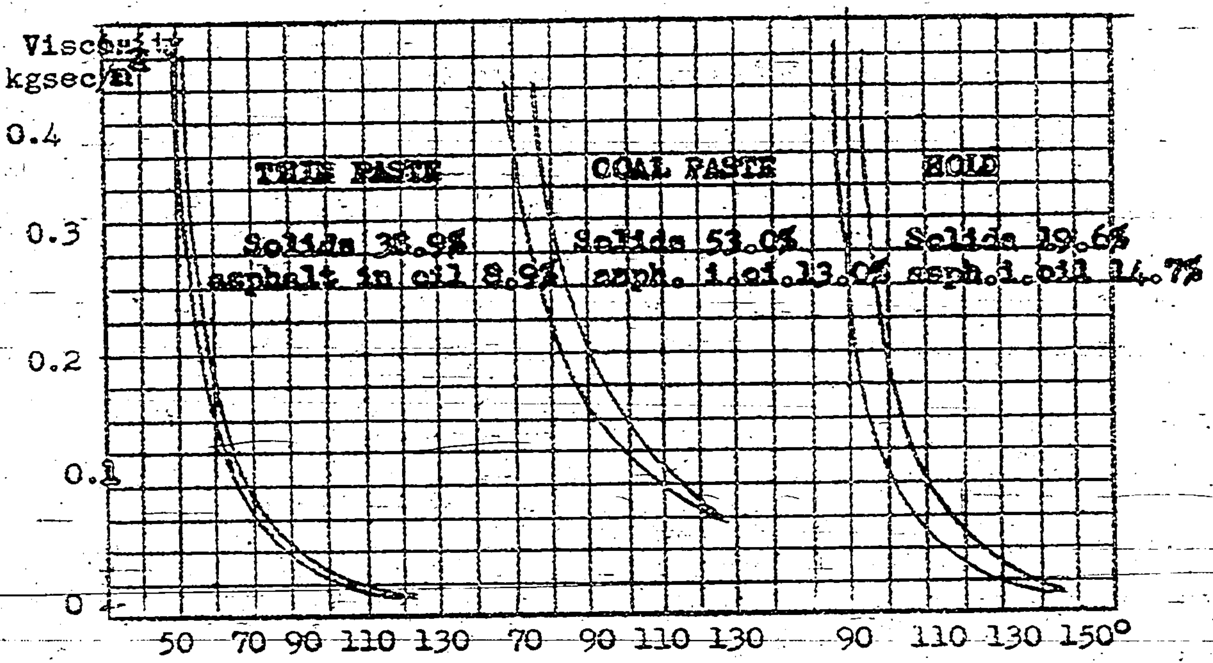
1.) The former operating experience is too slight to make any definite statements. According to our present short experience, it seems that the shutdown caused by poor heat exchange can be foreseen and that the heat exchangers may be regularly interchanged in the shortest time, for instance in 3 - 4 days, if the stall is not shut down for some additional reasons, and not on account of the heat exchanging alone.

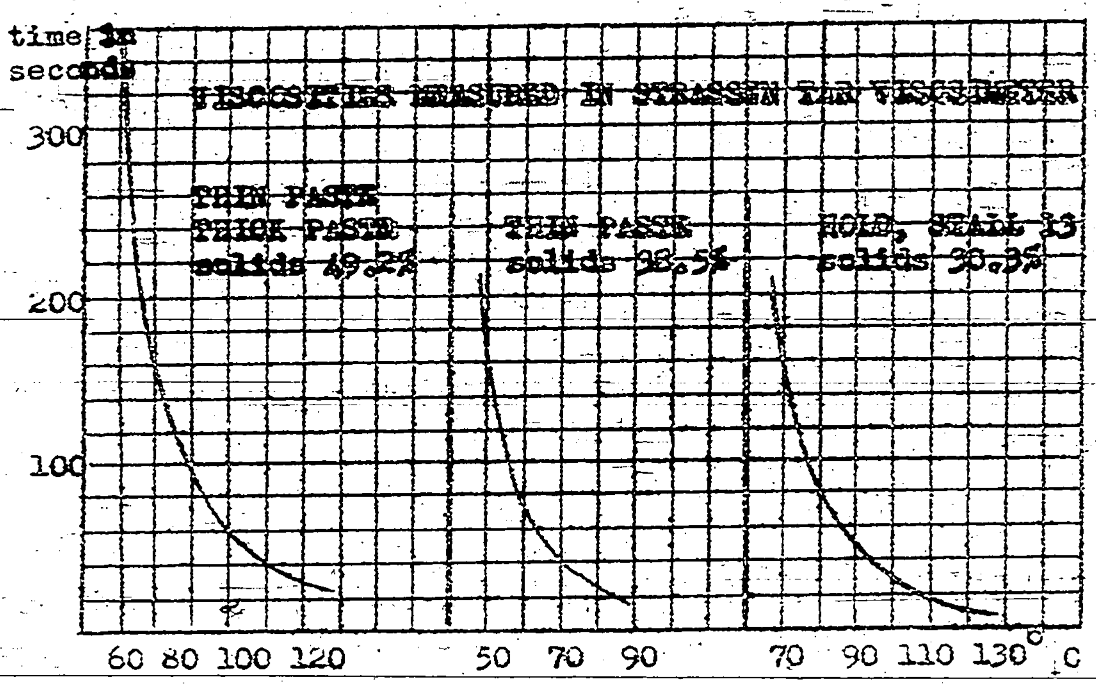
2.) The saving in the paste heat exchanging is so large that considered only from the economic view an additional stall might be introduced which would take care of the unforeseen shutdowns - not necessarily only those caused by heat exchange troubles, - so that no reduction in production over the year would result (spare stall, as proposed by Schappert).

3.) It must be mentioned here that the question of paste heat exchanging is subordinate to the fact that for each ton of production of the finished gasoline with 10,000,000 heat units, the amount of heat value set free is at least 1,000,000 heat units of energy because of the more economical heat utilization during the heat exchanging, a saving which is of great importance in the power economy.

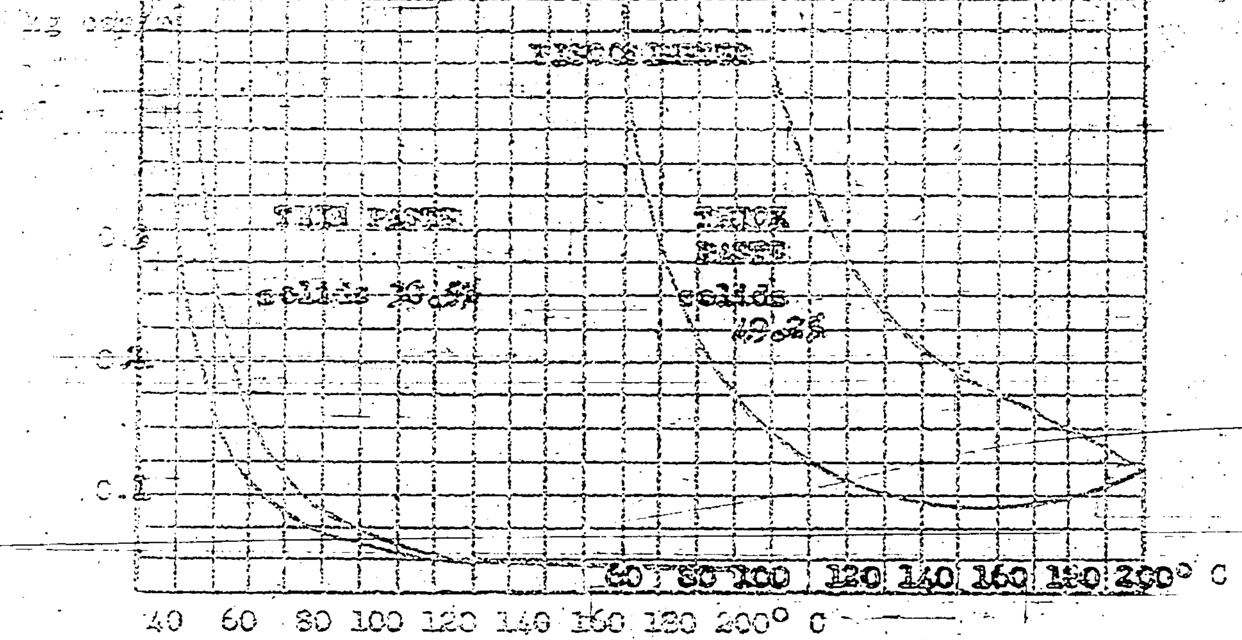
Sternberg/MC/ jw

7-31-1946

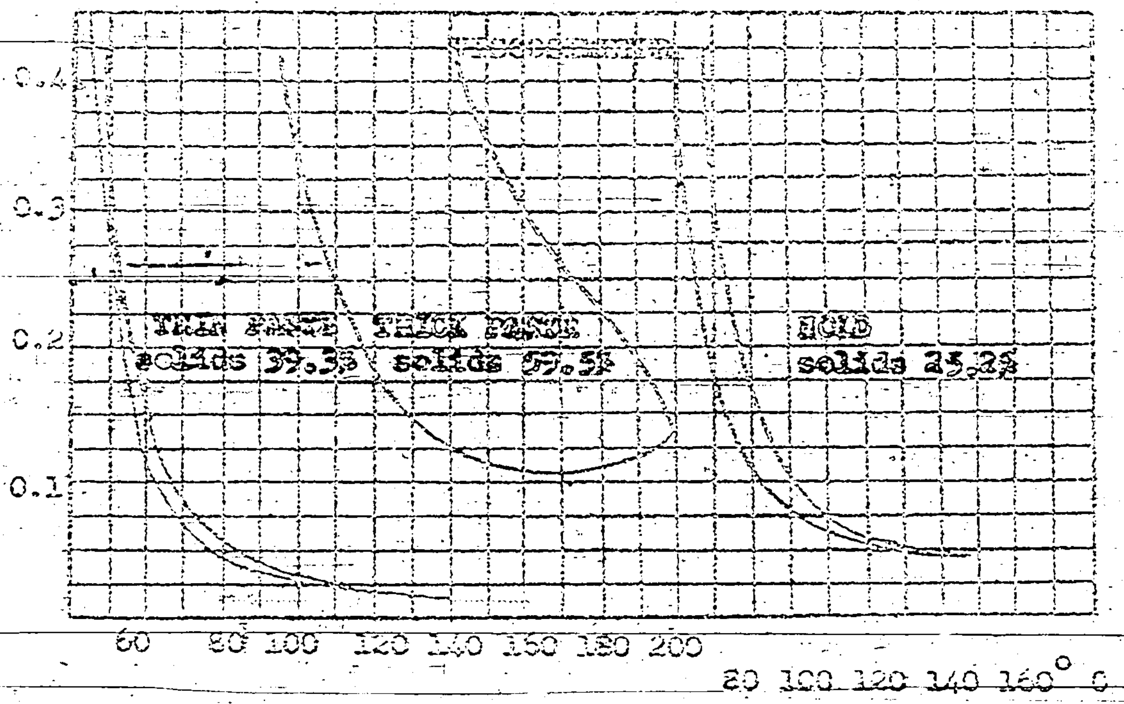

Viscosity kgsec/m²
0.4
0.3
0.2
0.1
0
THIN PASTE
Solids 38.9%
asphalt in oil 8.9%
COAL PASTE
Solids 53.0%
asph. i.oil 13.0%
HOLD
Solids 19.6%
asph.i.oil 14.7%
50 70 90 110 130
70 90 110 130
90 110 130 150°


time in seconds
300
200
100
VISCOSITIES MEASURED IN STRASSEN TAR VISCOMETER
THIN PASTE
THICK PASTE
solids 49.2%
THIN PASTE
solids 38.5%
HOLD, STALL 13
solids 30.3%
60 80 100 120
50 70 90
70 90 110 130° C

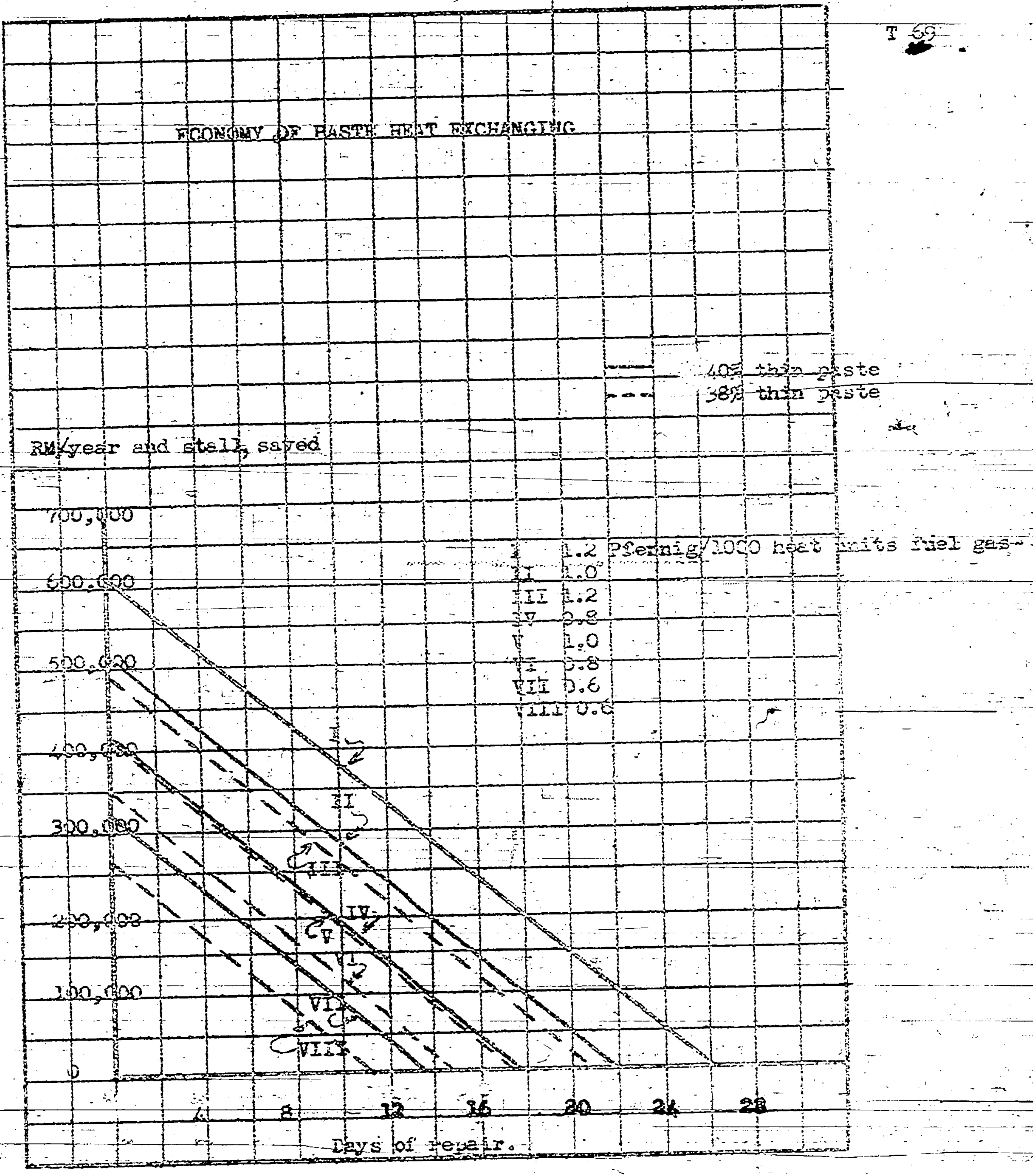
VISCOSITY CURVES DETERMINED IN COAL PASTE

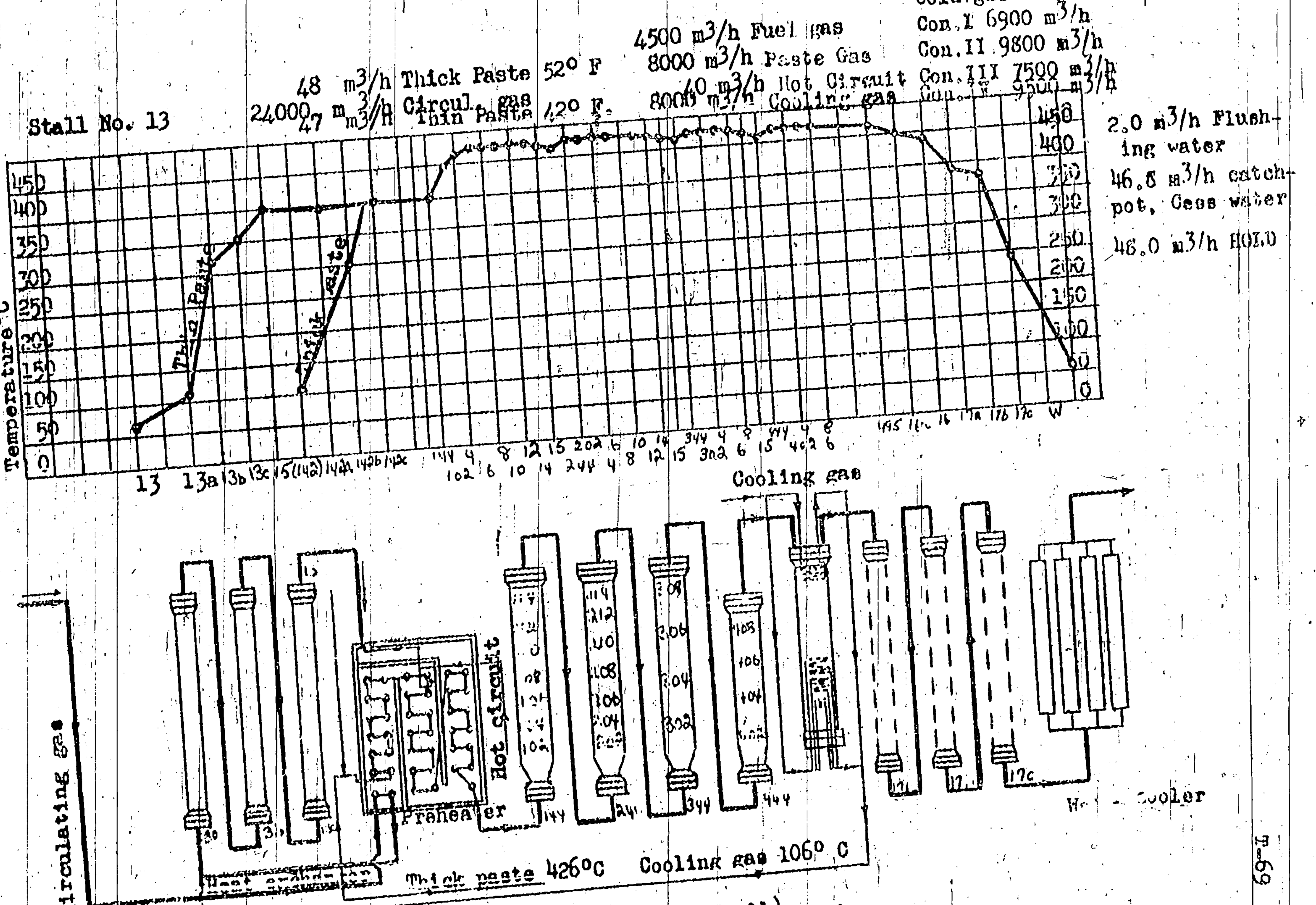


VISCOSITY CURVES MEASURED WITH COAL PASTE



ECONOMY OF PASTE HEAT EXCHANGING





Temperature Changes (Coal Stall)

Heat Exchange		STALL 13 9/30/41	10/9/41
Paste thruput	te/h	18.6	20.0
Solids	%	39.1	37.7
Coal	%	30.3	35.7
Am't. gas	m^3/h	26,000	26,000
Catch pot	te/h	16.9	19.3
Total gas	m^3/h	45,700	49,500
Temperature	°C	189 ← 324 ← 362 ← 435 105 → 272 → 319 → 408	195 ← 319 ← 365 ← 437 105 → 263 → 319 → 410
Number of Exchang.		3	3
Over.heat. Surface	m^2	3 x 188	3 x 188
Velocity, forw.pass	m/sec	0.50	0.53
Velocity, return pass	"	1.07	2.14
K value	cal/°C/h/m^2	302 182 127	289 143 349
Total K value		266	265

Heat Exchange		STALL 14 9/30/41	10/9/41
Paste thruput	te/h	18.5	20.8
Solids	%	39.1	38.5
Coal	%	39.1	27.9
Am't. gas	m^3/h	26,000	26,000
Catch pot	te/h	16.9	19.3
Total gas	m^3/h	54,300	59,200
Temperature	°C	205 ← 324 ← 421 ← 439 122 → 270 → 399 → 420	201 ← 327 ← 401 ← 425 126 → 284 → 382 → 411
Number of Exchang.		3	3
Over.heat. Surface	m^2	3 x 188	3 x 188
Velocity, forw.pass	m/sec	0.49	0.55
Velocity, return pass	"	1.97	2.22
K value	cal/°C/h/m^2	265 446 182	374 442 192
Total K value		296	287

Preheating after Paste Heat Exchange.

STALL 13

Preheater		9/30/41								10/9/41							
Paste thruput	te/h	39.3 (incl. 20.7 thick paste)								37.8 (incl. 17.8 thick paste)							
Total conc. sol.	%	46.6								43.5							
Am't. of gas	m^3/h	Paste gas 6,000 + 26,000 intake gas								Paste gas 4,000 + 26,000 intake gas							
Flue gas temp.	°C	384	409	409	500	500	560	560	594	428	456	456	506	506	543	543	594
Temp. of feed	°C	51	119	101	277	347	394	420	394	55	137	103	306	361	403	430	403
		Gas		Paste		Mixture				Gas		Paste		Mixture			
Heat absorbed	cal/h			6,135,000								6,180,000					
Av. Diff. Temp.	°C	312		266		156		169		356		277		142		149	
K value	cal/°C/h/m^2	11.0		7.1		9.6		6.1		11.6		7.2		9.1		6.9	

STALL 14

Preheater		9/30/41								10/9/41							
Paste thruput	te/h	39.0 (incl. 20.5 thick paste)								40.2 (incl. 19.4 thick paste)							
Total conc. sol.	%	46.6								43.6							
Am't. of gas	m^3/h	Paste gas 6,000 + 26,000 intake gas								Paste gas 4,000 + 26,000 intake gas							
Flue gas temp.	°C	407	444	444	510	510	550	550	594	407	448	448	500	500	543	543	594
Temp. of feed	°C	39	124	79	282	362	399	430	399	47	129	106	296	360	394	430	394
		Gas		Paste		Mixture				Gas		Paste		Mixture			
Heat absorbed	cal/h			6,595								6,075					
Av. Diff. Temp.	°C	353		297		138		155		345		273		144		153	
K value	cal/°C/h/m^2	13.1		7.4		8.6		7.8		13.0		6.5		7.5		9.2	

Operations with Coal.

Pölits, Oct. 14, 1941

	Pölits Process								
	Calculated	Stalls							
		13 7/10	13 7/18	13 8/15	13 8/23	14 9/18	13 10/11	14 10/11	Value reached with 40% Thin Paste 10% Solids in Pasting Oil
	1	2	3	4	5	6	7	8	9
Number: Heat Exch.	3	3							
Hairpins	20	20							
Converters ($35.5\ m^3$)	4	4							
Temp.Rise in Heat Exch. (°C - °C)	(100-385)	(103 - 400°C)							
Gas: Intake $m^3/h \times 1,000$	24	21	24	24	26	26	26	26	26
Paste gas $m^3/h \times 1,000$	8	6	8	8	6	6	4	4	4
Cooling gas $m^3/h \times 1,000$	8	8	8	8	8	8	8	8	8
Paste: Thin, te/h	17.3	17.3	17.1	16.4	15.8	18.1	19.8	21.4	21.4
%Solids in thin	37	38.4	39.9	44.5	42.1	39.3	42.1	42.1	40.0
Thick, te/h	19.7	20.4	19.2	22	22.2	20.0	17.0	19.8	19.8
%Solids in thick	52	52.1	51.9	56.4	52.7	54.5	54.1	54.1	54.0
Total, te/h	33	37.7	36.3	38.4	38.0	38.1	36.8	41.2	41.2
Total, %solids	46	45.8	46.7	50.5	49.0	46.7	47.7	47.8	46.2
Pasting Oil: Film Oil	1.1	1.0	-	0.5	1.0	2.1	1.6	-	-
% Solids	6.0	6.9	6.6	8.1	8.9	12.2	16.1	16.1	10.0
% Ash in Solids	-	27.8	24.0	26.2	33.0	37.0	44.7	44.7	35.0
Coal: te Coal, te/h	13.8	15.64	15.25	17.8	16.8	15.0	13.72	15.44	16.8
% Ash	about 4.0	9.6	6.8	7.2	7.6	8.5	6.9	6.9	6.9
Pure coal, te/h	-	14.1	14.2	16.5	15.5	14.2	12.8	14.4	15.6
% Total catalyst	about 3	about 3.4							
Utilization		93 - 95%							
Oil Prod., k/li		0.28 - 0.31							
Gas + M.O. in Oil		80 - 92%							
Gasific./gas + M.O. + gasific.		23 - 25%							
Oil prod./te Pure Coal		670 kg							
H_2 Consump./te Oil prod. (m^3/te)		2,100 (?)							

T.O.M. Reel No. 78
pp. 69-72
Bag No. 4
Target No. 30/4.13

Pölitz, May 3, 1944.

COAL PREPARATION AND RESIDUE OPERATIONS AT THE GELSENBERG BENZIN A.G. VISIT OF April 28, 1944

A) Coal preparation. Gelsenberg uses coal from the Nordstern mine. This is washed at the mine with a barium sulfate solution (specific gravity 1.35) and separated into 53% hydrogenation coal, 20% gang, and 27% middle product with 30% ash. The hydrogenation coal contains 4½% ash, 6-7% water and 40% volatile constituents.

The coal, which is almost all lump coal, is crushed in two hammer mills. These have a capacity of 130 T/h each. After adding the catalysts, 1.8% by weight Bayer mass and 1.2% iron sulfate (the 0.3% sulfigran are added later), the coal is weighed wet and enters the Uni-mill.

There are 5 systems available, which are run with a thru-put of 26 to 34 T/h. At the present time 2,000 T/day, or 83 T/h, are prepared. Three systems were in operation with a load of 26 T/h each. With an increase in the thru-put per system the spray-grain (spritzkorn) quantity increases as follows:

Spray-Grain Quantity	Load on the Uni-mill
300 kg/day	26 te/h
600 " "	28 " "
900 " "	30 " "
1,200 " "	32 " "
1,500 " "	34 " "

The spray-grain is run into the yard and consists of 40% wood, 20% oil and the remainder coal.

Each system contains a ball mill, a screen in which the coarse grain is caught, a Lurgi-filter and a tube paste mill. The coarse grain is taken to the ball mill, where the coal is also dried. Wistra-burners are used for heating. In order to keep the content of the circulating air below 7%, 8,000 m3/h carbon dioxide are introduced into the system. The volume proportioning of pasting-oil and coal is done automatically.

The following analyses show what excellent milling is done in the plant, which makes an excellent impression in the arrangement of its equipment, safety of operations and cleanliness:

Screen Analyses

	Raw Coal	Coal Dust	Coal Paste
Over 1.0 mm	57%	0.6%	0.0%