

UNITED STATES BUREAU OF MINES

CLASSIFIED LIST OF TRANSLATIONS
BY THE COAL HYDROGENATION DEMONSTRATION
PLANT, LOUISIANA, MISSOURI
TO DECEMBER 31, 1946.

Arranged by W. M. Sternberg

THE FOLLOWING TRANSLATIONS ARE OMITTED
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BEEN REPRODUCED ON THIS REEL:

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192	Device for Low Temperature Carbonization of Carbonaceous Substances, 21 Dec. 1935. German Patent #699707, Class 10 a, Groupe 2601.
193	Pressure Hydrogenation of Coal or Similar Solid Carbonaceous Substances. German Patent #656364, 19 May 1933, and German Patent #675957, 4 Aug. 1935.
194	Process for Separating Oils from Mixtures with Solid Substances. German Patent #550157, 26 October 1927, and German Patent #630965, 30 April 1933.

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106.	Phenols. (A brief note by Hirschberger)	163	264-265
107.	Production of Useful Phenols During Hydrogenation of Various Materials 10/15/42.	163	266-267

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169.	Nitrided Plungers & Rods.	181	06703-4
170.	Hardened Plungers & Piston Rods for High Pressure Machines. Leuna April 30, 1942.	181	06705
171.	Hardened Plungers & Piston Rods for High Pressure Compressors. Gelsenkirchen, July 7, 1942.	181	06707-06720

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167.	Nitride Hardening of Piston Rods & Plungers.	181	06700
168.	Experience with Nitriding Plungers.	181	06701-06702
169.	Nitrided Plungers & Rods.	181	06703-4

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	Hardened Plungers & Piston Rods for High Pressure Machines. Leuna April 30, 1942.	181	06705
171	Hardened Plungers & Piston Rods for High Pressure Compressors. Gelsenkirchen, July 7, 1942.	181	06707-06720

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16	Temperature Equalization in the Lutzkendorf Preheater - Jan. 30, 1943.	1	Item 8
18	Properties of NiO Material for Harpin Tubes of a 700 Atm. Preheater.	1	Item 7
30	Proposed Modifications in the Scholven Preheater, June 18, 1937.	10	360-366
33	Heat Exchange & Preheater of the 700 Atm. Liquid Phase Hydrogenation at Blechhammer Ludwigshafen, Feb. 14, 1940.	11	Bag 2247 Item 9
37	Liquid Phase Stall Preheater, Scholven, 2-28-1942.	11	Bag 2247 Item 9
38	Preheater and Blower for 700 Atm. Liquid Phase, Scholven, III/266.	11	Bag 2247 Item 9
41	Preheater for Coal Stall - Gladbeck 11-3-1942.	11	Bag 2247 Item 9

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57	Report on the Strength of Tube Steel in 700 Atmosphere Preheaters - Ludwigshafen, 2-17-1943.	76	29-31
61	Approximate Calculations of the Heat Transfer Coefficient of a Preheater.	162	752-756
63	Instrumentation for the Gas Preheater of the Extract Hydrogenation Stall 17 of the Large Scale Pilot Plant at Welheim, March 4, 1917.	5	162-165
64	Report of the Technical Testing Laboratory at Oppau Number 543, Investigation of Finned Tubes.	76	48-68
68	Mounting Thermo-elements in Electric Preheaters Ludwigshafen on the Rhine January 25, 1943.	162	757-760
74	Preheater Problems in the Liquid Phase with Special Consideration of the Heat Pump. A Paper by Schappert presented at the Colloquium, March 24, 1944.	167	43
76	Construction of Preheaters with Horizontal Tubes. Lu, Sept. 27, 1944.	174	833-839
77	Maximum Preheating in the Liquid Phase, March 31, 1944.	145	489-490.
83	Reducing the Load on the Liquid Phase Preheaters.	104	974-806
111	Abstract of Report on Reducing Resistances in Gas-Fired Preheaters.	118	727-736
144	Dr. Kronig's Letter Regarding Incrustation of Preheaters, and the Action of the Catalysts in Causing Them.	75	909-912

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158	Velocity in Preheaters @ 600 Atm. By Oettinger, Lu, 22 Nov. 1938.	126	425-427	
191	Discussions on March 25, 1943 in Leuna of the N-10 Material.	129		Ref a-2

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166	Pump and Press Construction Materials.	181	06706	
122	Experiences with 700 Atm. Paste Presses at Bottrop by Chief Eng. Richter, Ruhrol-GMBH.	9	336-374	

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113	Different Processes for the Production of Water Gas. October 18, 1942.	163	27-29	
151	A Note on Koppers Gas Producer.	43	209	
152	Brabag-Zeitz Powdered Coal Gasification.	43	219-224	
153	Cost Comparison of the Winkler and the Keppers Powdered Coal Gasification Processes.	43	225-226	
154	Operation Costs in the Synthesis Gas Production in Rheinpreussen.	43	260-267	

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159	Direct Production of Synthesis Gas from Powdered Fuel.	43	267-268
188	Method of Production of Ammonia Synthesis Gas.	65	119-127
200	Operating Balance of Koppers Powdered Coal Operations.	188	20951-20960

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17	Vapor Phase Product Balance, Bottrop, July, 1944 and August, 1944.	9	193-194
24	Production Balance of the Vapor Phase Stall (54a for December 1941) (Bottrop February 4, 1942).	9	223-229

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166	Pump and Press Construction Materials.	181	06706
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T-No.		TOM Pp. or Reel Frames No.	
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40	Report on the Measurement of Heat Conductivity of Refractory Brick for the Inner Insulation of High Pressure Converters, Report #421, Ludwigshafen.	103	
44	Report on the Testing of Two Asbestos Cements for High Pressure Insulation.	103	Report No. H26-430
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70	Coal Preparation & Residue Operations at Gelsenberg Benzin A.G. Visit of April 28, 1944. Pöhlitz, May 1944.	78	69-72
71	Coal Preparation and Residue Operations at Hydrogenation Works Scholven, A.G. (Visit of 27 April 1944).	78	73-75
155	Working Up of Oil-Containing Residues (Kiln Operations).	126	843
160	Residue Processing.	163	125-127
104	Abstract of Report on the Processing of Hydrogenation Residue at Wesseling, based on a Visit to Scholven, 3 Jan. 1939 - Leuna Werke 20 Feb. 1939.	14	602-611

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62. 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, 1926. 181 625-631

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101. Abstract of Report on Sludge Plant Operation. Gelsenberg Benzin A.G. - Dr. Hu. Gelsenkirchen Horst, 17 July 1940. 5 182-188

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Oil Ludwigshafen/Rhine, 4-18-39.

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36 Installation for the Splitting Stall
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72 Report on Coal Preparation and Spray
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Oil Yield in Low Temperature Carbon-
ization by the Addition of Catalyst
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86 Steels Used in Hydrogenation.	163	221-226	
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94. Present Status of Investigations of Attacks by Hydrogen Upon Non-Loaded Test Pieces, as affected by Hydrogen Pressure, the Duration of the Experiment and the Hardening State of Material.	130	628-635	
96. Abstract of Report on Construction Materials for Hydrogenation.	130	591-596	
98-A Abstract of Lecture on High-Pressure Steels (See also T-93 and T-96).	130	597-611	
172. Results of Tests of N10 - Lenna, 1943.	130	612-622	
179. I.G. Construction Steels (Abstract)	130	559-587	
18. Properties of N10 Materials for Hairpin Tubes of a 700 Atm. Preheater.	1		Item 7
14. I.G. Specifications for K2A, N5 replacement Materials, N2A, N9 and N10 Steel. (I.G. Standards Book).			
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49. Sulfur Balance (on Hydrogenation Side) at the Scholven III Installation (S/t Gasoline and S/h at 250,000 Year/tonne) Scholven, January 10, 1939.	11		Bag 2247 Item 12
51. Summary of Results of Round Table Discussions on the <u>Sulfurization of Middle Oil</u> - November 1, 1943.	181	499-501	

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| 16 | Temperature Equalization in the Lutzkendorf Preheater - Jan. 30, 1943. | 1 | Item 8 |
| 68 | Mounting Thermo-elements in Electric Preheaters. Ludwigshafen on the Rhine January 25, 1943. | 162 | 577-580 |
| 39 | Heat Balance and Temperature Relationships of the Liquid Phase, Scholven, III/266. | 11 | Bag 2247
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| 95 | Pressure-tight Closure for Thermoelements. 192 | | |
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| 65 | Report on a Letdown Needle-Valve Stettin-Pöhlitz, 21 October, 1942. | 78 | 13-21 |

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17.	Vapor Phase Product Balance, Bottrop, July, 1944 and August, 1944.	9	193-194	
24	Production Balance of the Vapor Phase Stall (54a for December 1941) (Bottrop, February 4, 1942).	9	223-229	
27	Gasification with 700 Atm. in Vapor Phase.	126		Item 124
28	Inspection of 700 Atm. Vapor Phase Gasoline.	126	985	
29	Experimental Work on 700 Atm. Vapor Phase Operations.	126		Item 127
88	Abstract of Report on the Automatic Regulation of the Liquid and Vapor-Phase Hydrogenation Stalls at Leuna.	130	846-873	
89	Liquid and Vapor Phase Operations. Discussion at Pölitz - March 27 - 28, 1944.	170	534-547	
118	Experiments with Baffles in High-Pressure Vapor-Phase Hydrogenation Converters, Leuna Werke 21 December 1942.	180	836-846	
198	Arrangement of Gasoline Converters & Heat Exchangers for Various Catalysts.	180	833-835	
4	Data of Different Works on Vapor Phase Circuit.	24		Bag 1928

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- 82. Measuring the Viscosity of Pasting Oil, HOLD and Coal Paste from Scholven and Leuna Hydrogenation Works. 174 914-921

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- 43. Comparison of the Circulation washing and Circulation Purge at 700 Atm. Dr. Frese (Ruhrol) on April 24, 1939 in Scholven. 9 375-383

WASHING OF OIL

- 3. Wash Oil Inspection - Scholven. 24 Beg 1928

WATER GAS

- 113. Different Processes for the Production of Water Gas. October 18, 1942. 163 27-29

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WICKELMANTEL

99. Abstract of Discussion on Spiral-Wound
High Pressure Jackets. (Wickelmantel)
Lu, 25 May, 1939.

129 134-141

Reel 15

T-1

The Production of Aviation
and Heating Fuel from Upper Silesian Coal

The flow sheet is made up for working of coal only, small amounts of tar from the coal distillation are omitted. Quantitative data are calculated on an hourly basis; one year totals 8,000 hours. The analyses of products with number indices are tabulated in Appendix A. Letter indices refer to explanations in Appendix B.

APPENDIX A

Flow Sheet of the Hydrogenation Plant at Blechhammer

Analysis of the Products

Oil-Free Material of Sump Phase		% H ₂ O in Crude Product	% Ash in Dry Substance		% C in Pure Substance	
No.	Kind					
1	Crude Coal	10		5		81.0
2	Bayermass	15				
Oil-Containing Material of Sump Phase		% Solid	Distillation Analysis of Oils in %, °C			
No.			< 160	160-210	210-325	> 325
3	Grinding Oil	10.0	0	0	40.7	59.3
4	Thick Paste	51.0	-	-	-	-
5	Heavy Oil Let-Down	27.0	0	0	33.0	67.0
6	Thin Paste	43.0	-	-	-	-
7	Catch Pot Heavy Oil	0	0	0	0	100.0
8	Catch Pot	0	5.50	4.06	44.75	45.69
9	Dilution Oil	0	0	0	42.8	57.2
10	Centrifuge Mixture	16.0	0	0	37.8	62.2
11	Operational Loss during Centrifuging	16.0	0	0	37.8	62.2
12	Centrifuge Oil	11.2	0	0	37.8	62.2
13	Centrifuge Residue	38.0	0	0	37.8	62.2
14	Operational Losses during Distillation	38.0	0	0	37.8	62.2
15	Distillate from Flash Chamber	0	0	0	84.9	15.1

Flow Sheet of the Hydrogenation Plant at Bleckhammer
 Analysis of the products.

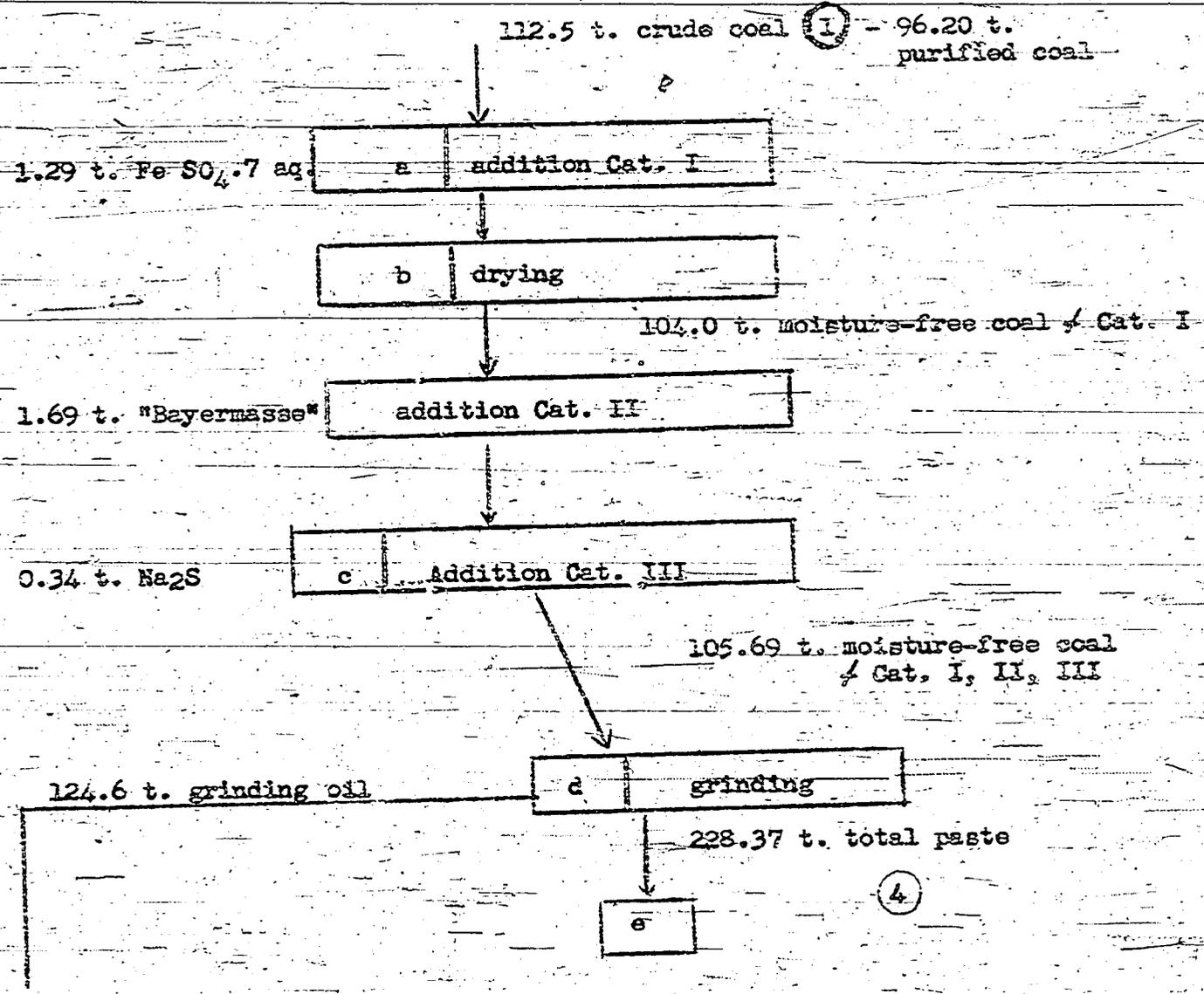
- 2 -

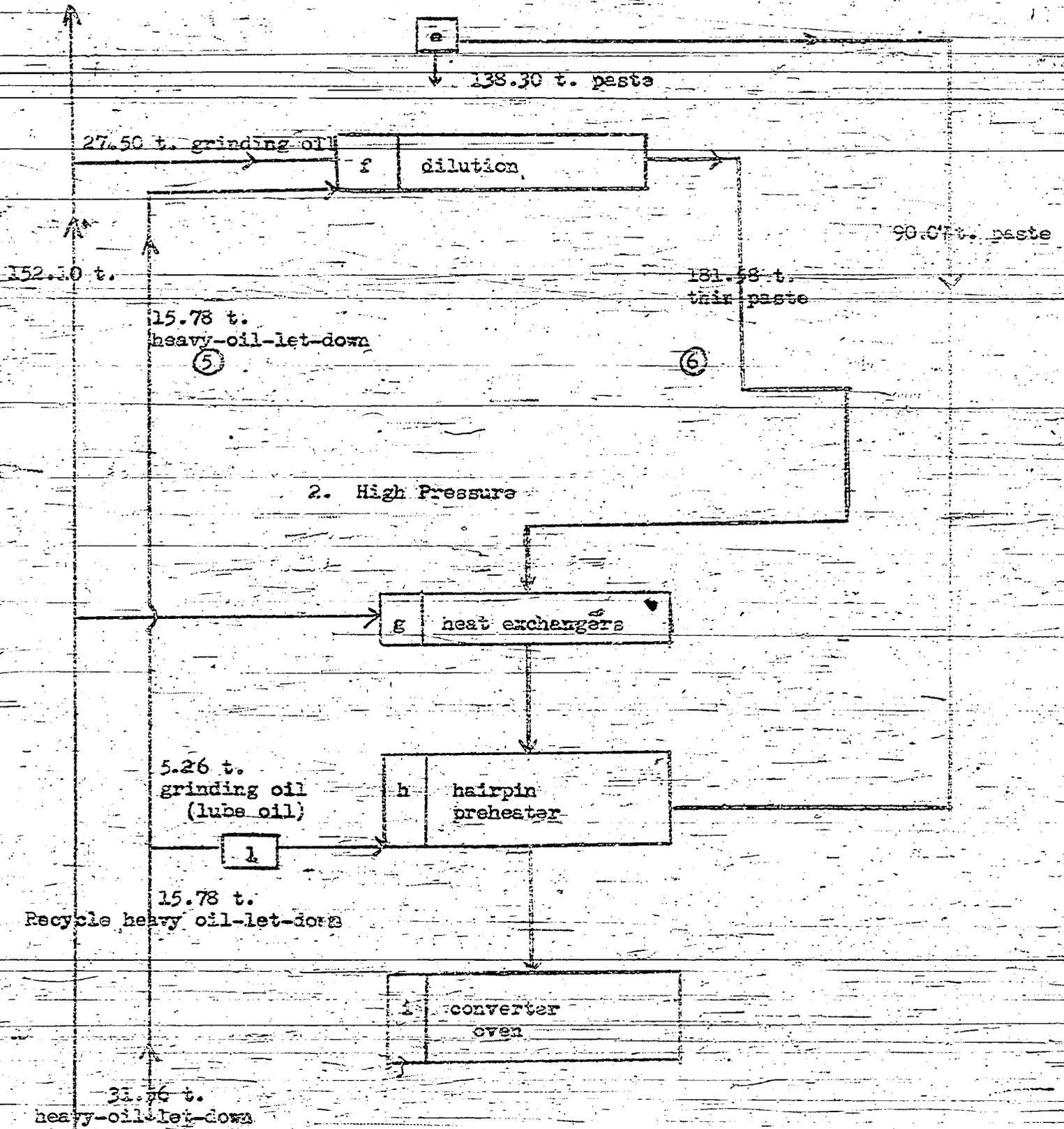
No.	Oil-Containing Material of Sump Phase	% Solid	Distillation Analysis of Oils in %, °C			
			- 160	160-210	210-325	>325
16	Concentrated Centrifuge Residue	53.0	0	0	5.0	95.0
17	Distillate	0	0	0	15.0	85.0
18	Distillation Residue	96.0	0	0	10.0	90.0
19	Operational Loss from A-Distillation	0	5.50	4.06	44.75	45.69
20	Fuel Oil for Sale	0	0	0	34.05	65.95
21	Gas Phase Products	0	23.1	17.1	59.8	0

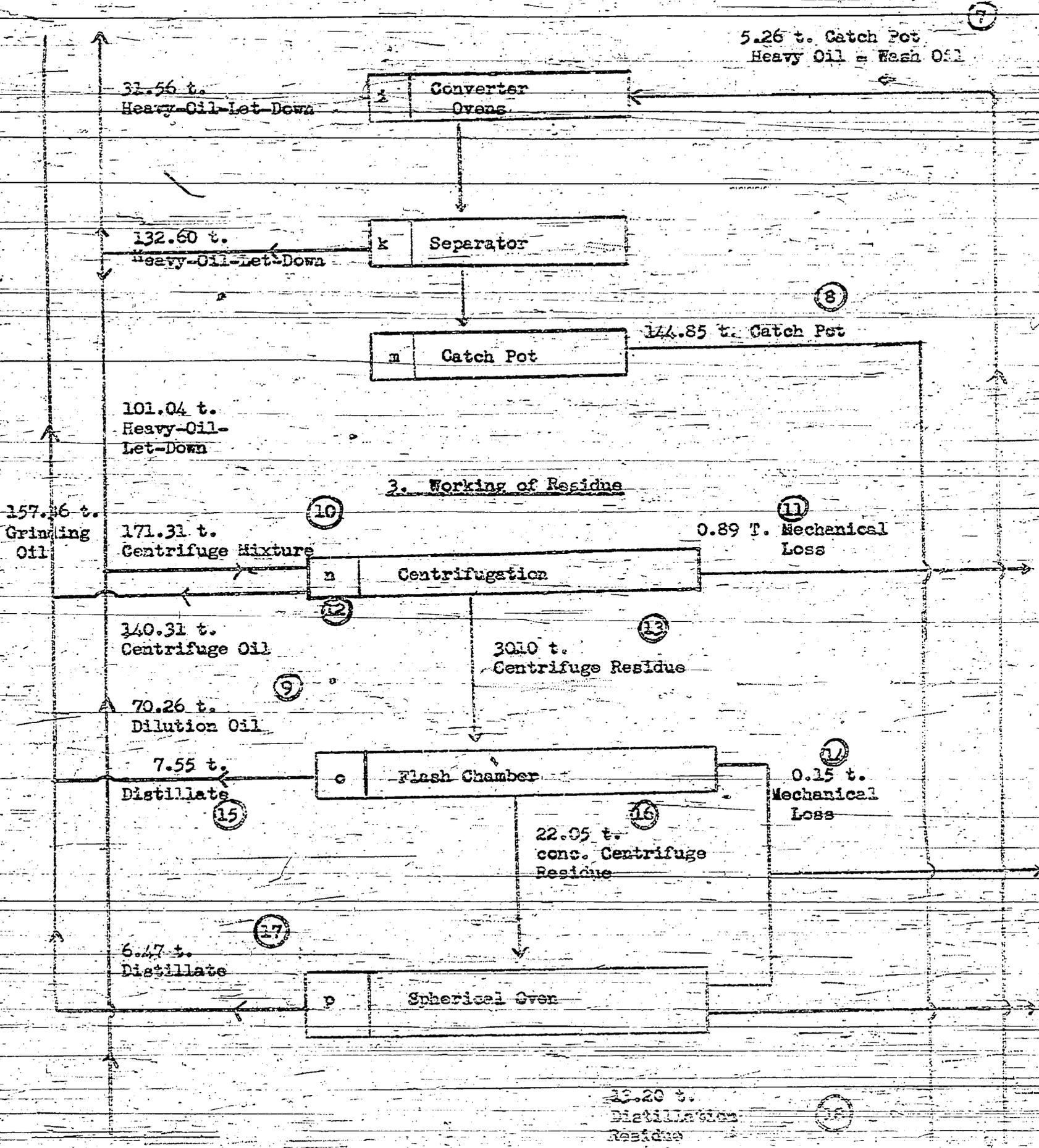
Material of Gas Phase	% Boiling			E.P.	V.P.	O.N.	
	-100°	-155°	>155°			No Pt	With 0.0% Pb
22 Catch Pot from Pre-Hydrogenation	-	35.0	65.0	-	-	-	-
23 Loss from B-Distillation	-	35.0	65.0	-	-	-	-
24 Gasoline Recovery, Catch Pot	-	49.6	50.4	-	-	-	-
25 Loss from C-Distillation	-	49.6	50.4	-	-	-	-
26 Aviation Fuel	55	98.0	-	157	0.5	74	88

I. SUMP PHASE

1. Preparation of the Coal







3. Working of Residue

5.26 t. Catch Pot
Heavy Oil - Wash Oil

31.56 t.
Heavy Oil-Let-Down

Converter
Ovens

132.60 t.
Heavy Oil-Let-Down

Separator

144.85 t. Catch Pot

Catch Pot

101.04 t.
Heavy Oil-
Let-Down

157.46 t.
Grinding
Oil

171.31 t.
Centrifuge Mixture

0.89 T. Mechanical
Loss

Centrifugation

140.31 t.
Centrifuge Oil

3010 t.
Centrifuge Residue

70.26 t.
Dilution Oil

7.55 t.
Distillate

Flash Chamber

0.15 t.
Mechanical
Loss

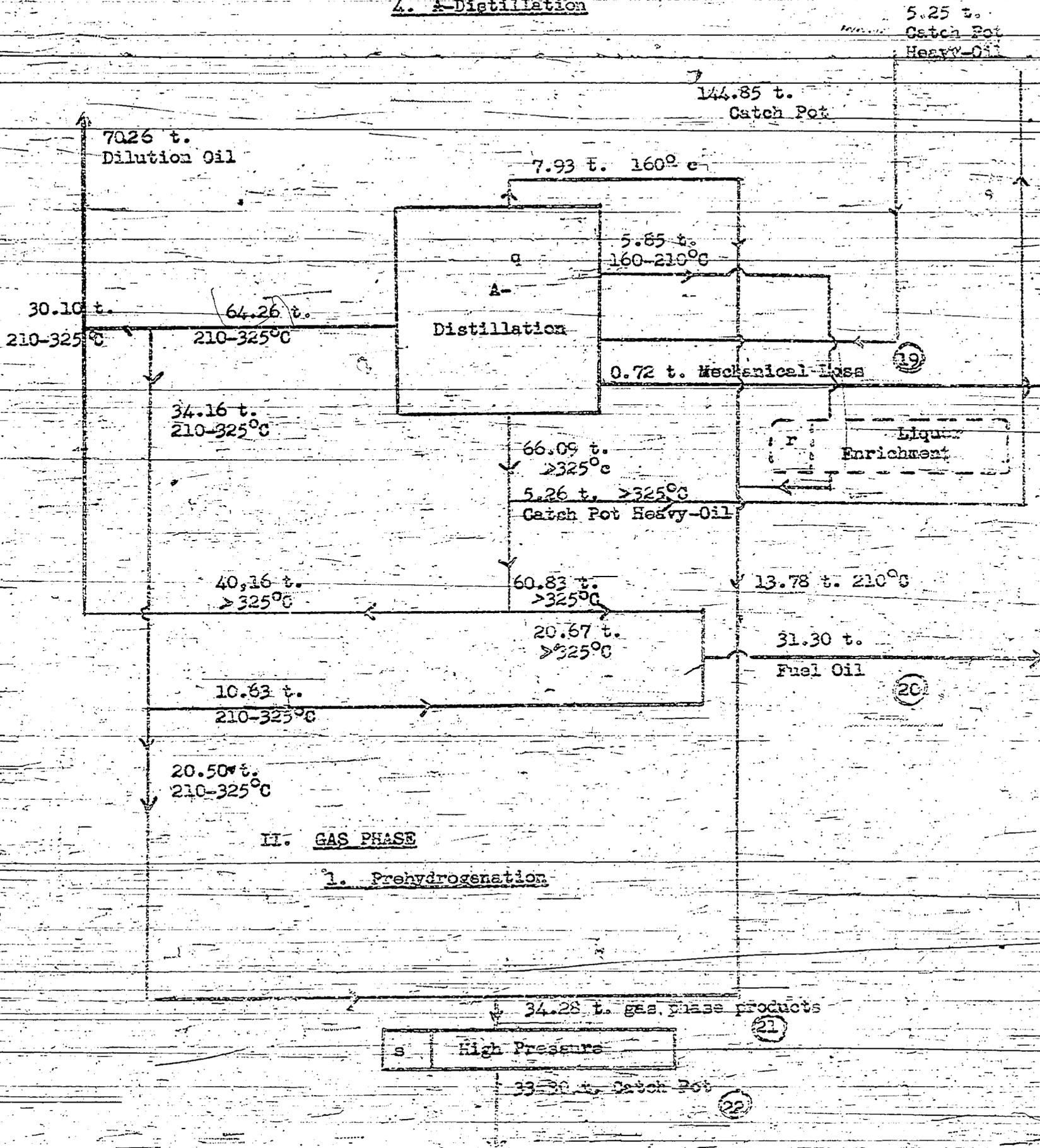
22.05 t.
conc. Centrifuge
Residue

6.47 t.
Distillate

Spherical Over

13.20 t.
Distillation
Residue

4. A-Distillation

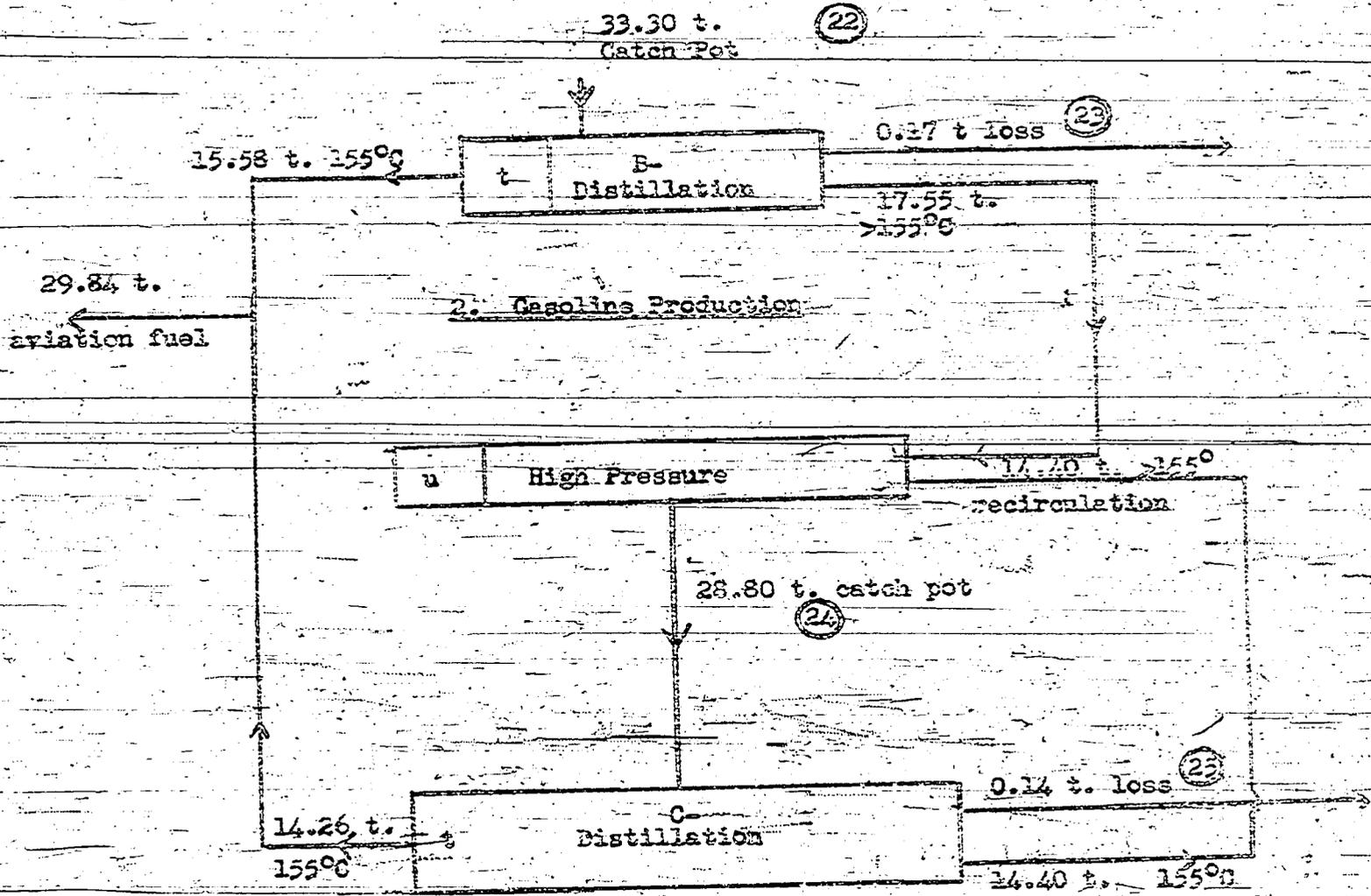


II. GAS PHASE

1. Prehydrogenation

s High Pressure

33.30 t. Catch Pot



Total Hydrogen Consumption
 128,000 H cu.m. (97.0%)

Total Yield of Gaseous Hydrocarbons:

- CH₄ = 5.50 t.
- C₂H₆ = 4.38 t.
- C₃H₈ = 6.50 t.
- C₄H₁₀ = 5.75 t.

- 3 -

APPENDIX B

Explanations

- a = The salt in water solution is sprayed on the moving coal (about 40°C crystallization temperature).
- b = Partial neutralization of the alkali in the coal takes place during heating by the SO_4 ions of the catalyst.
- c = Because of its sensitivity the salt is added just before the introduction of coal into the oil.
- d = The entire coal is ground to a thick paste; during this procedure the remaining water evaporates. The thick paste contains 103.77 tons moisture-free coal plus catalysts I, II, and III, also moisture-free.
- e = The thick paste is so proportioned that a large part (66.9%) of the total paste (271.65 tons) goes through the heat exchangers.
- f = The thick paste is diluted in order to be able to heat it in the heat exchangers. The solid content of the paste should not exceed 43%. Heavy-oil-let-down is also used for dilution to improve the carrying capacity of the not very viscous grinding oil by addition of oils rich in asphalt.
- g = There are four equally equipped chambers for the sump phase hydrogenation. The reaction conditions are 700 atm. total pressure, 585 atm. H_2 partial pressure in the entrance chamber, 3.5 M cu. ft. total gas/kg. coal. The yields are 96% of C in coal decomposed and 21% of the C gasified. Calculations were performed with a C percentage of 86.0 in the produced oil. There are three heat exchangers in each chamber. The temperature at the exit of the heat exchangers is 310°C.
- h = For each chamber one hairpin preheater with about 30 hairpins of a capacity of 12×10^6 Kcal. The temperature at the exit is 415, maximum 425.
- i = Each chamber has four ovens. The temperature is kept at 485. All four chambers have a combined reaction volume of 148.0 cu.m.
- k = For each chamber one separator.
- l = Since it suffices to have available only half of the recycle heavy-oil-let-down at higher temperatures, only that amount of the recycle heavy-oil-let-down is recycled hot to conserve energy.
- m = Each chamber has one cold catch pot.
- n = Contains de Laval centrifuges.

- o** = The centrifuge residue is heated to 450 under 50 atm. pressure, first, in the heat exchanger then in the hairpin preheater to keep it from drying out because of its high medium oil content. To increase the flow velocity, small amounts (maximum 0.1 N cu. m./kg. centrifuge residue) of compressed distillation gas are added. Expansion takes place in the flash chamber and a large part of the oil is evaporated and topped.
- p** = The concentrated centrifuge residue flows from the flash chamber under its own gravity into a spherical oven. The distillation residue is thrown away.
- q** = Gas heated distillation at 4 pressure gives the following fractions: To 160°, 160-210°, 210-325°, > 325°.
- r** = The fraction, 160-210, can be led through the liquor enrichment off the "Phenosolvan Plant," to enrich the liquor (8-10 g. phenol/l.) or the already dephenolized water (.2 g./l.) up to 18 g./l. Simultaneously, the more valuable phenols can be exchanged against the less valuable phenols. Material balance changes due to this procedure have not been considered in this flow diagram.
- s** = Two chambers with three ovens each of 8 cu. m. catalyst volume, two ovens, catalyst 8376, one oven, catalyst 5058; Conditions: 300 atm. total pressure, 410 temperature and 0.8 kg./l./hr maximum throughput. The reaction product has an IBP of 50°C.
- t** = Steam heat distillation at atm. pressure. Stabilization and washing of the gasoline are omitted in this scheme because everything was calculated with gas-free products.
- u** = Two chambers with two ovens each of 8 cu. m. catalyst volume, catalyst 6434. Conditions: 300 atm. total pressure, 390° temp., and 0.45 kg.-155°/l. catalyst volume per hour.

T-2
Scholven, Jan 4, 1940

RECORD

Concerning gasification and coal hydrogenation balance during the production of 22.5 or 27 ton per hour of VT 705

The production of 22.5 te/hr. of VT 705 is done in 5 coal stalls. When a 6th coal stall is put in, the production will be increased to 27.0 tons per hour.

The following gasification values will be obtained:

Gasoline Production in Hydrocarbons in year-tonnes.	22.5 te/hr.	27 te/hr.
C ₁	22,700	26,950
C ₂	28,500	33,850
C ₃	36,600	43,400
C ₄₁	25,750	30,550
C _{4n}	25,750	30,550
Total Gasification	139,300	165,300

For a delivery of 68,000 year-ton of hydrocarbons to the Chemical Works Mills and an increase of the fuel gas delivery up to the limit given by the gas heating values, we will get the following requirements on hydrocarbons (Case 3 of the Record of December 14, 1939, with consideration of the fuel gas requirements T16 and T52):

Gas Production	22.5 te/hr.	27.0 te/hr.
Basic delivery in year-tons hydrocarbons	68,000	68,000
C ₄₁ delivery in year-tons hydrocarbons	16,000	16,000
Fuel gas delivery in year-tons hydrocarbons	26,000	44,000
Heating gas requirements in year-tons hydrocarbons	45,400	53,400
Total Consumption	155,400	181,400

The percentage gasification, from the above figures, is;

In the Coal Phase:

With pure coal throughput of 89.6 te/hr with 74.4 te/hr of C (83%) and a utilization of 96%, with 83% of carbon content in the gasified hydrocarbons $\frac{17.8}{71.4} = 25\%$

In the Gas Phase:

With a total gasoline production of 27.5 te/hr.

Subtracting 90% of the vapor phase stall production, $9.6 \times 0.9 = 8.6$ te/hr.

Therefore a gasoline production from the gas phase of 18.9

and with a 9.9 ton per hour C gasification computed as oil 11.5 te/hr.

Total 30.4 te/hr and $\frac{11.5}{30.4} = 38\%$

b): From the gasification data at Nordstern of 22%, the hydrocarbon share in the coal phase is calculated to be 18.9 te/hr.

We must consider the gasification in the vapor phase to be 20%, if we consider the conditions in Scholven. We have an additional 10% from the vapor phase stall. This brings us the following results:

Hydrocarbon separation from the gasoline production $\frac{18.9}{0.8} = 18.9 = 4.7$ te/hr.

$$\frac{4.7 \times 0.86}{0.83} = 4.8 \text{ te/hr.}$$

In addition

$$\frac{0.96 \times 0.86}{0.83} = 1.0 \text{ te/hr.}$$

Therefore a total hydrocarbon production of 5.8 te/hr instead of 11.39.

These figures lead us to a total gasification of 24.7 te/hr. or 178,000 year/te with 300 day throughput.

T.O.M. Reel 24, Bag 1928
T-3

(11)

The wash oil used was 5058 b product from P 1271 from stall 501. The wash oil was investigated in the fresh state, after a fairly long use ¹⁾ as well as after regeneration of the used oil by distillation. ²⁾

FRESH WASH OIL		USED WASH OIL		REDISTILLED USED WASH OIL		
SPECIFIC GR/15°	0.851	0.839		0.832		
ANILINE Pt.	+39.7	+40.6		41.9		
UNSATURATED	1%	2.5%				
IODINE NO.	5.3	17.8		7.53		
START BOILING	162°C	77°C		185°C		
% -- 180°	14.6	28.2				
% -- 200°	61.4	52.2		19.3		
% -- 250°	97.9	89.8		98.0		
RESIDUE %	99.9	91.9		99.9		
FRACTIONS:	SPECIFIC GRAVITY/°C	A.P.	IODINE NO.	SPECIFIC GRAVITY/°C	A.P.	IODINE NO.
-180°	0.827/15°	38.6°	16.9	0.783/15°	44.7°	24.9
180 - 240°	0.852/15°	39.9°	4.23	0.843/15°	41.1°	13.0
> 240°	0.890/15°	46.7°	3.24	0.912/15°	40°	24.9
ASPHALT BREAK DOWN						
NEUTRAL OIL		56.9		78.3		
WAX FORMERS		10.7		18.0		
OIL WAXES		1.9		3.1		
ASPHALT WAXES		3.0		0.5		
ASPHALTENE)						
CARENE)		0.2		0.1		
CARBOLE)						

- 1) MIDDLE OIL FRACTION FROM SOLVENT
- 2) THE WASH OIL HAS BEEN USED ABOUT 3 MONTHS, HOWEVER, HAD BEEN FREQUENTLY REPLENISHED WITH FRESH OIL DURING THAT TIME.

A.P. - ANILINE Pt.

P.D.T. # 24

T.O.M. Reel 24, Bag 1928
T-4

DATA ON DIFFERENT WORKS ON THE VAPOR PHASE CIRCUIT

PRESSURE RANGE (ATM)	300					
HYDROGENATION WORKS	POLITZ	GEISENBERG	SCHOLVEN	ME		
CATALYST (ATM)	6434	6434	5053/6434	5055	6434	5053/6434
PRESSURE ON SUCTION SIDE	283	290	285	285	265	315
PRESSURE ON PRESSURE SIDE	319	321	320	325	325	---
PRESSURE WHERE GASHYDRATE IS OBSERVED	---	300	---	---	---	---
TEMP. OF CIRCULATING GASES (°C)						
SUCTION SIDE	19	19	15-30	33	33) about
PRESSURE SIDE	34	27	35-45	47	47) 25-30
AT THE LOCATION OF THE GASHYDRATES	---	Occasionally 10°	---	---	---	---
TEMP. OF CATCHPOT	15-20°	35° (occ- 15-30° casual-ly 10°)	---	28°	27°	25-30°
OBSERVATION OF GASHYDRATE	NONE	Yes, in Products Cooler	NONE	NONE	NONE	NONE
KG E ₂ O/100m ³ CIRCULATING GAS IN CIRCUIT	---	---	2.5	1.8	3.6	---
KG E ₂ O/m ³ of HYDROCARBONS	?	?	0.23	0.10	0.78	---
COMPOSITION	SUCTION SIDE	SUCTION SIDE	SUCTION SIDE	OUTLET OF SUCTION FLASK	OUTLET OF SUCTION FLASK	CONVERTER OUTLET
CO ₂ /CO	0.0/0.6	0.0/0.0	About 0.014/0.0	0.02/0.19	0.15/0.0	0.00/0.28
H ₂ O	0.0	0.0	About 0.05	0.005	0.01	0.22
O ₂	0.0	0.0	0.0	0.0	0.0	---
H ₂	91.0	94.2	80.0	81.1	95.3	86.94
N ₂	5.0	3.0	7.0	11.0	3.4	5.46
CH ₄	---	---	7.0	5.9	0.11	5.57
C ₂ H ₆	---	---	2.0	1.0	0.10	0.21
C ₃ H ₈	2.9	2.8	0.7	0.39	0.53	0.55
10 ⁿ C ₄ H ₁₀ / n C ₄ H ₁₀	---	About	0.3/0.2	0.14/0.09	0.30/0.13	0.60
C ₅ and higher	2.21	H ₂ O: 3.0	0.7	0.05	0.08	0.15
% of Unsaturated	---	---	---	---	---	0.02
Gs NH ₃ /m ³	0.004	0.007	0-0.4	0.06	0.03	---
CONVERTERS	3, 4, 5	6	---	11.12	9.10	---
DATE	1/15/46	1/18/41	1940-42	---	---	5/24/40

PRESSURE RANGE atm

500-700

300

Hydrogenation Works

Politz

Gelsenberg

Welheim

Wesseling

Scholven

Me

Bohlen

Pressure on the suction side atm
Pressure on the pressure side
Pressure where gas hydrates have been observed

Temperature on circulating gases
On the suction side
On the pressure side
In front of the gas cooler
Behind the gas cooler
At the location of the gas hydrates

Observation of gas hydrates

Kg H₂O/100 m³ of circulating gas behind catchpot
Kg H₂O/100 m³ of circulating gas where gas hydrates have been observed
Kg H₂O/ m³ of hydrocarbons

Composition of the circulating gases

CO₂/CO
H₂S
O₂
H₂
N₂
CH₄
C₂H₆
C₃H₈
1 C₄H₁₀/n C₄H₁₀
C₅ and higher
% of unsaturated
Gm NH₃/ m³

	suction side	behind circulation side cooler	suction side	behind gas cooler	suction side	suction side	in front of cooler	behind cooler	Converter outlet	Converter Outlet	
CO ₂ /CO	1.0/0.5	0.3/0.9	0.03/0.5	0.00/0.51	0.0/0.7	4.18/4.29	4.18/4.29	0.12/0.4	1.13/1.3	4.74/5.94	0.41/2.9
H ₂ S	0.2	0.1	0.035	0.07	0.04	0.39	0.39	0.08	0.11	1.09	0.6
O ₂	0.0	0.0	-	0.00	0.0	0.0	0.0	0.0	0.0	0.0	0.0
H ₂	76.0	78.1	79	63.53	63.93	73.46	73.46	73.4	74.0	66.33	65.5
N ₂	6.9	6.0	0	5.54	8.30	3.25	3.25	7.1	7.3	5.87	4.8
CH ₄	11.1	11.9	8	17.31	21.1	11.7	11.7	14.5	15.0	12.85	21.5
C ₂ H ₆	1.5	1.3	2	6.36	4.0	1.6	1.6	2.3	1.4	1.46	2.7
C ₃ H ₈	0.8	0.7	0.7	4.00	2.0	0.7	0.7	1.8	0.72	0.96	1.1
1 C ₄ H ₁₀ /n C ₄ H ₁₀	0.05/0.25	0.05/0.25	0.07/0.43	0.26/0.43	0.08/0.42	0.04/0.2	0.04/0.21	0.07/0.35	0.03/0.21	0.04/0.24	0.06/0.34
C ₅ and higher	0.2	0.2	0.2	0.36	0.1	0.15	0.15	0.05	0.04	0.15	0.1
% of unsaturated	0.4	0.0	-	-	-	0.42	0.42	-	-	0.33	-
Gm NH ₃ / m ³	0.007	0.025	0.01	0.35	0.21	0.006	0.006	0.02	0.02	-	-

Note: 1) Estimated from per cent of hydrocarbons, average carbon and information from Leuna
2) Combined coal and vapor phase circuit

Converter Date

12 13 15 1-5
1/15/42 2/21/41 12/1/41 Dec 37 Jan 42 winter 41 March 42 March 42 12/17/40

T.O.M. Reel 24, Dec 1928
T-6

1)
SOLUBILITIES OF H₂, N₂ and CH₄
At 700 ATMOSPHERES (in liters/kg atm)
(100 Liters Converter used from July 24, 1938 to Sept. 25, 1938)

NUMBER	1	2	3	4	5	6	7	8	9	10	AVERAGE
DATE OF ANALYSIS	7/24/38	7/26/38	7/28/38	8/3/38	8/6/38	8/23/38	8/25/38	8/26/38	9/21/38	9/25/38	
PRODUCT TESTED	P 1301	P 1301	P 1301	P 1329	P 1329	P 1307	P 1307	P 1307	P 1307 P 1329 (1,1)	P 1307 P 1329 (1,1)	
CATCH POT											
SP. GR./0°C	0.904 /15 40°	0.910 /15 40°	0.916 /15 40°	0.968 /20 40°	0.966 /20 40°	0.956 /15 40°	0.946 /15 40°	0.946 /15 40°	0.968 /15 40°	0.962 /15 40°	
TEMP. OF SOLUTION	40°	40°	40°	40°	40°	40°	40°	40°	40°	40°	
H ₂ - SOLUB.	0.068	0.067	0.054	0.052	0.071	0.067	0.065	0.066	0.067	0.071	0.065
N ₂ - SOLUB.	0.153	(0.030)	(0.049)	0.116	0.104	0.153	(0.061)	(0.053)	0.110	0.147	0.130
CH ₄ - SOLUB.			0.170	1)				0.182	1)		
H.O.L.D.											
SP. GR./0°C	0.980 /50	0.970 /50	1.010 /20	1.048 /20	1.002 /20	1.040 /20	1.020 /50	1.032 /20	1.006 /20	1.046 /20	
TEMP. OF SOLUTION	48°	42°	43°	43°	43°	43°	43°	43°	44°	44°	
H ₂ - SOLUB.	0.017	0.017	0.020	0.023	0.015	0.107	0.179	0.205	0.075	0.095	
N ₂ - SOLUB.	0.013	0.026	0.039	0.029	0.013	0.196	0.160	0.194	0.109	0.103	
CH ₄ - SOLUB.			0.058	1)				0.264	1)		
WASH OIL											
SP. GR./0°C	0.851 /15° 20°										
TEMP. OF SOL.	20°										
H ₂ - SOLUB.	0.119	0.125	0.109	0.129	0.161	0.101	0.115	0.121	0.109	0.109	0.120
N ₂ - SOLUB.	0.134	0.207	0.187	0.248	0.252	0.250	0.116	0.129	(0.074)	0.237	0.196
CH ₄ - SOLUB.			0.460	1)				0.398	1)		

The values enclosed in parenthesis were wrongly determined and not used in the determination of averages.

1) Analysis of stock

P.D.I. 127

pkc

TABLE III (For computation of dew point curves)

Approx. values of equil. constant $K = \frac{\text{Mol } \% \text{ in gas}}{\text{Mol } \% \text{ in liquid}}$ In relation to total pressure

TEMPERATURE °C	0°						10°			
HYDROCARBONS	CH ₄	C ₂ H ₆	C ₃ H ₈	nC ₄ H ₁₀	iC ₄ H ₁₀	C ₅ H ₁₂	CH ₄	C ₂ H ₆	C ₃ H ₈	nC ₄
PRESSURE (ATM)										
3.5	30	5.2	1.4	0.34	0.48	0.12	35	6.0	1.8	0.48
7	15	2.6	0.7	0.17	0.24	0.06	17.5	3.0	0.90	0.24
14	7.5	1.42	0.41	0.11	0.16	0.037	8.5	1.7	0.51	0.16
21	5.3	1.08	0.32	0.085	0.12	0.032	6.2	1.24	0.41	0.126
28	4.3	0.90	0.28	0.071	0.10	0.025	5.1	1.05	0.37	0.105
35	3.9	0.80	0.25	0.065	0.092	0.029	4.5	0.90	0.32	0.095
42							3.75	0.75	0.30	0.090
49							3.2	0.64	0.28	0.085
56							2.8			

TEMPERATURE °C	-12.2°						
HYDROCARBONS	CH ₄	C ₂ H ₆	C ₃ H ₈	nC ₄ H ₁₀	iC ₄ H ₁₀	C ₅ H ₁₂	
PRESSURE (ATM)							
12	7	1.3	0.34	0.086	0.12	0.030	
21	5	1.0	0.26	0.07	0.10	0.024	

19°		15.6°						21.1°					
i C ₄	C ₅ H ₁₂	CH ₄	C ₂ H ₆	C ₃ H ₈	n C ₄	i C ₄	C ₅ H ₁₂	CH ₄	C ₂ H ₆	C ₃ H ₈	n C ₄	i C ₄	C ₅ H ₁₂
0.68	0.180	42						50					
0.34	0.090	20	3.1	0.0	0.30	0.45	0.011	24.5	4.0	1.17	0.35	0.54	0.136
0.228	0.055	9.5	1.83	0.60	0.185	0.28	0.067	12.2	2.0	0.67	0.2	0.32	0.084
0.178	0.045	7.0	1.36	0.47	0.149	0.22	0.052	8.1	1.45	0.51	0.17	0.26	0.069
0.148	0.039	5.5	1.14	0.40	0.129	0.19	0.046	6.1	1.2	0.44	0.15	0.23	0.058
0.134	0.035	4.8	0.97	0.36	0.117	0.17	0.041	5.0	1.0	0.40	0.13	0.20	0.050
0.120	0.033							4.0		0.383	0.12		
0.120	0.035							3.4		0.375	0.116		
								2.9		0.382	0.115		

P.D.I. # 28

PHI

COMPARISON OF GELSENBERG AND SCHOLVEN HYDROGENATION PRODUCTS.

Ruer-Scholven, June 12, 1940.

We give below the analysis of the products of the Gelsenberg-Benzin A.G. of May 1940 made in Scholven, and of our works in February 1940 and May 1940. At least two series of analyses were made to exclude accidental results as perfectly as possible. A simple comparison of analyses from the same works discloses differences which are particularly large in the Scholven vapor phase products and point to improvements which have since been introduced.

There are in addition occasional differences in results obtained in the different works, which result from differences in the process. It manifests itself in the liquid phase in the farther reaching coal utilization at Gelsenberg and the liquid phase becomes so highly enriched with iron in the ash (Table 2), that a recovery may be considered. This far-reaching utilization results however also in a progressive destruction of the aromatic structure, which is manifested in a lower C content of the Gelsenberg middle oil (Table 4), and recurs in the A gasoline (Table 5). The slight hydrogenation in the liquid phase may also be observed in a higher hydroaromatic content. It may perhaps be noticed that the pyridine base content of the Gelsenberg A gasoline is considerably above that of the Scholven A gasoline.

The great differences in the injection product of the 5058 stall (Table 6) are the result of A gasoline being also injected in Gelsenberg, in contradiction to Scholven. For similar reason, a comparison of the catch pot products must be made with certain reservations (Table 7). It is however of interest, that the amount of gasoline is nearly the same in the two cases, which leads to the conclusion that gasoline injection lowers correspondingly the production of additional gasoline. The hydrogenation effect is greater in Scholven, as is particularly

well demonstrated by the higher aniline point and the ultimate analysis of the catch pot product from 6434 (Table 9). These relationships are confirmed by analyses of the 6434 injection products (Table 8).

The differences become to a great extent negligible in the final state, as shown in the tests of the finished gasoline (Table 10). The light-test of the Scholven gasoline is not particularly good, because it depends on the poor light-fastness of the A gasoline. This is however merely a defect in looks, since the gasoline is not used in the light, and the test is accordingly not prescribed by the buyers. For purposes of experiment the A gasoline in Scholven has been refined over 5058 in spite of the attendant losses, and this resulted in a good light fastness.

It is naturally possible to find other differences. However, when they are small, they must be judged particularly critically, in order to avoid drawing conclusions concerning differences in processes which actually are simply the result in the differences in the taking of the different samples, or inaccuracies of analysis.

The vapor phase process in Scholven has been recently improved. The total fresh hydrogen addition of the vapor phase into the 6434 stall has improved the activity of the catalyst, which in turn has permitted to lower the temperature from 23 mV (833°F) to about 22 or 22.5 mV (804 or 819°F) (1) in spite of an increased oil injection. In addition, the undesired high hydrogenation of the preliminary refining 5058 could be limited by lowering the temperature from the former from 22.-22.5 mV to 21.5-22 mV (804°F - 819° to 788 - 804°F) (1). Since these measures have been introduced, the octane numbers of our gasolines are 73 and over.

6
(1). The conversion of mV into °F. was done with the aid of the table on p. 1878 in the Handbook of Chemistry and Physics, 26th edition. The mV data given in the text refer to a 30°C cold junction, and the composition of the thermocouple, not specifically mentioned here, is believed to be iron-constantan. Sternberg.

COAL PASTE

Gelsenberg

Scholven

	<u>Feb. 1940</u>	<u>May 1940</u>	<u>Mar. 1940</u>	<u>May 1940</u>
Solids in paste	58.2%	54.5%	54.2%	54.6%
Ash in solids	9.9%	10.4%	7.5%	8.1%
Asphalt in oil	6.9%	10.8%	10.5%	11.5%
Water	1.2%	1.0%	0.7%	0.6%

(Table No.1, T.O.M. Reel 10)

Blechhammer

	<u>Thick Paste</u>	<u>Thin Paste</u>
Solids in paste	51.0%	43.0%

(Appendix A, T.O.M. Reel 143)

- e The thick paste is so proportioned that 66.9% of the total paste goes through the heat exchangers.
- f The thick paste is diluted in order to be able to heat it in the heat exchangers. The solid content of the paste should not exceed 43%. H.O.L.D. is also used for dilution to improve the carrying capacity of the not very viscous grinding oil by additions of oils rich in asphalt.

(Appendix B, T.O.M. Reel 143)

TABLE 2

Investigation of H.O.L.D.

	Gelsenberg-H.O.L.D.		Scholven-H.O.L.D.	
	Feb. 1940	May 1940	March 1940	May 1940
d_{100}^a	1,200	1.206	1.250	1.250
Softening Pt.	10.8°C	10.5°C	38°C	38°C
of Solids	24.1 %	24.2 %	28.5 %	28.5%
Ash in Solids	55.2 %	58.5 %	27.4 %	27.4%
Asphalt in Oil	13.1 %	12.0 %	22.5 %	22.5%
% by wt. to 325°	8.6 %	7.6%	3.0%	3.0%
Soda in H.O.L.D.	0.0 %	0.08 %	0.0 %	0.08%

Gelsenberg-H.O.L.D. reacts neutral towards phenol phthalein. Its alkalinity towards m.-O. is equal to 12 mls. n/10 H₂SO₄/100 g. H.O.D.D.

Vacuum Distillation Curve
18 mm Hg

Beginning:	124°C	123°C	122°C	129°C
- 150°C	3 % by vol.	2 % by vol.	1 % by vol.	1 % by vol.
- 200°C	14 % " "	13 % " "	8 % " "	8 % " "
- 225°C	27 % " "	27 % " "	21 % " "	20 % " "
- 250°C	39 % " "	40 % " "	27 % " "	32 % " "
- 300°C	56 % " "	56 % " "	47 % " "	45 % " "
- 350°C	66 % " "	67 % " "	58 % " "	56 % " "
	362°/72	364°/72 Vol-%	365°/62 Vol-%	369°/61 Vol-%
Decomposition:	362°/	364°	365°	369°

Composition of Ash:

H ₂ O-Solubles		
Na ₂ SO ₄	7.6%	6.9% and Cl
CaSO ₄	2.6%	
MgSO ₄	1.2%	
H ₂ O-Insolubles		
SiO ₂	20.9%	32.2%
Fe ₂ O ₃	34.2%	12.8%
Al ₂ O ₃	23.5%	25.1%
CaO	0.9%	2.9%
MgO	2.2%	6.2%
Na ₂ O/K ₂ O	4.3%	4.9%
P ₂ O ₅	0.3%	0.9%
SO ₃	2.1%	7.7%

TABLE 3

Coal Catch Pot Investigation

	Gelsenberg-Catch Pot		Scholven-Catch Pot	
	February 1940	May 1940	March 1940	May 1940
d_{15}	1.004	1.003	1.022	1.019
Solids mg/l	38	40	46	60
Wt. Engler				
Begin boiling	154°F	73°C	156°F	153°F
to 311°F	3.8 Wt. %	4.2 Wt. %	4.3 Wt. %	4.2 Wt. %
337°F	5.4	5.3	5.4	5.6
410°F	10.7	11.6	12.4	11.5
483°F	21.7	20.6	24.0	22.4
527°F	29.4	28.0	29.6	27.8
572°F	36.9	37.4	39.2	36.5
617°F	50.7	48.7	49.6	48.6
653°F	63.0	61.5	61.8	59.6
Res. 653°F	36.6	38.2	37.6	40.1
Loss	0.4	0.3	0.6	0.3
d_{15} to 617°F	0.947	0.946	0.948	0.946

TABLE 4

Middle Oil

	Gelsenberg-M.O. May 1940	Scholven-M.O. May 1940
d_{15}	0.957	0.955
A.P. of Product Containing Phenol	- 21°C	- 19°C
A.P. of Product Containing no Phenol	- 19°C	- 19°C
Phenol	131 g/l	130 g/l
Sulfur	0.03 %	0.07 %
Unsaturated Hydrocarbons	26.0%	24.0%
B. P. Curve:		
Beginning:	136°C	284°F
to 311°F	3 Vol-%	4 Vol.-%
347°F	7	9
365°F	11	15
383°F	16	20
437°F	32	40
482°F	48	56
527°F	63	71
572°F	78	88
617°F	93	97
	340° 98	630°F / 98
	340° 1	63°F 1.0
Loss	1.0	1.0
Ultimate Analysis:		
% C	85.82%	86.73%
% H	9.57%	9.51%
% N	0.70%	0.55%
% S	0.03%	0.07%
% Cl	0.004%	0.004%

TABLE 1

A-GASOLINE INVESTIGATION

	Gelsenberg-Products(refined & distilled) February 1940			Scholven-Product (refined & distilled)	
	Delivered Products	From Catch 155° Cut.	May 1940 Delivered Sample	March '40	May '40
d ₁₅	0.7322	0.7526	0.7226	0.7480	0.7351
Cu/ Al-Strips	V.G.	V.G.	V.G.	V.G.	V.G.
A.P.	108°F	95°F	106.7°F	95°F	100°F
Doctor Test	neg.	neg.	neg.	neg.	neg.
Acid Content of Distill. Residue	0.0	0.0	0.0	0.0	0.0
V.P. at 99.9°F	0.67	0.26	0.65	0.40	0.36
Octane Number	68.6	64.7	70.2	65.1	70.4
Glass dish mg/100 mls.	0.0	0.0	0.0	0.8	0.0
ASTM-Boil. pt. curve:					
Beginning:	94°F	152°F	101°F	123°F	113°F
- 104°F	2 Vol.%	-	-	-	-
- 122°F	-	-	3 Vol.%	-	1.5 Vol.%
- 140°F	11 " "	-	11 " "	5 Vol.%	5 " "
- 158°F	22 " "	-	27 " "	11 " "	14 " "
- 176°F	34 " "	7.5 Vol.%	55 " "	20 " "	32 " "
- 194°F	64 " "	25.0 " "	84 " "	39 " "	64 " "
- 212°F	85 " "	47.0 " "	95 " "	60 " "	88 " "
- 230°F	94 " "	67.0 " "	221/97 " "	74 " "	94 " "
- 248°F	244/97 " "	80.0 " "	-	84 " "	233°/95" "
- 266°F	-	88.0 " "	-	91 " "	-
- 284°F	-	93.0 " "	-	96 " "	-
- 302°F	-	95.5 " "	-	98.5" "	-
		311°/96.5	-	-	-
Olefins	-	-	10%	6%	7%
Aromatics	-	-	5%	12%	12%
Naphtenes	-	-	57%	53.3%	58.1%
Paraffines	-	-	28%	28.7%	22.9%
Ultimate Analysis:					
% C	84.77	-	85.1	85.56	85.73
% H	14.15	-	14.0	13.67	13.76
% N	0.62	-	0.1	0.41	0.16
% S	0.004	-	0.0	0.003	0.02
% Cl	0.05	-	0.00	0.03	0.008
Pyridin base-N	0.056	-	0.02	0.007	0.009

TABLE 6

FEED FOR 505 CONVERTER

	Gelsenberg-Products		Scholven-Products	
	February 1940	May 1940	March 1940	May 1940
d_{15}	0.931	0.943	0.967	0.960
A.P. (of product containing Phenol)	-	-18°C		-20°C
A.P. (of product containing no Phenol)		-18°C		-20°C
Phenol g/l	121	107	157	141
<u>B.P. Curve:</u>				
Beginning	143.6°F	147.2°F	325.4°F	323.6°F
167°F	2 Vol.%	2 Vol.%		
212°F	5			
257°F	10			
311°F	15	11		
337°F	18	15	0.5 Vol.%	2.5 Vol.%
347°F	20	17	1.0	3.0
365°F	23	19	2.0	5.0
383°F	25	22	6.0	9.0
437°F	40	38	34.0	33.0
482°F	55	53	49.0	50.0
527°F	70	66	67.0	67.0
572°F	85	80	86.0	86.0
590°F	92	86	91.0	91.0
608°F	96	92	95.0	94.0
617°F	97	94	96.0	96.0
	635°/99	642°/98.5	642°/98	642°/98.5
<u>ULTIMATE ANALYSIS:</u>				
% C :	86.41	85.78	86.93	87.40
% H :	10.42	10.03	9.30	9.75
% N :	0.93	0.72	0.84	0.58
% S :	0.41	0.32	0.12	0.09
% Cl :	0.02	0.005	0.007	0.007

TABLE 7

CATCH POT OF THE 5058 STALL

	Gelsenberg-Catch Pot 5058		Scholven-Catch Pot	
	February '40	May '40	March '40	May '40
d_{15}	0.820	0.810	0.811	0.815
A.P.:	$\neq 106^{\circ}\text{F}$	$\neq 104^{\circ}\text{F}$	$\neq 115^{\circ}\text{F}$	$\neq 109^{\circ}\text{F}$
Phenol g/l	0.50	0.76	0.41	0.63
Column Distillation:				
Liquefied Gas	1.4 Wt. %	1.8 Wt. %	2.7 Wt. %	1.8 Wt. %
Gasoline - 155 °	36.5 " "	35.4 " "	37.0 Wt. %	34.8 " "
d_{15}	0.760	0.756	0.763	0.715
A.P.	102°	100°	103°	100°
Phenol g/l	0.63	1.12	0.50	0.95
Middle Oil:	61.6 Wt. %	62.4 Wt. %	60.0 Wt. %	63.0 Wt. %
d_{15}	0.863	0.863	0.846	0.852
A.P.	44°	43°	51°	48°
Phenol g/l	0.45	0.63	0.36	0.68
Volume Engler of Catch Pot				
Beginning: °	133°	111°	135°	108°
to 122° F				1.0 Vol. %
- 167° F	3 Vol. %	3.5 Vol. %	3 Vol. %	3. " "
- 212° F	10 " "	11 " "	10 " "	9. " "
- 257° F	22 " "	23 " "	22 " "	19. " "
- 311° F	36 " "	38 " "	37 " "	36. " "
- 347° F	45 " "	47 " "	46 " "	46. " "
- 383° F	54 " "	56 " "	62 " "	57. " "
- 437° F	67 " "	73 " "	77 " "	75. " "
- 482° F	80 " "	81 " "	87 " "	86. " "
- 527° F	90 " "	89 " "	95 " "	94. " "
- 572° F	96 " "	95 " "	545°/96"	532°/97.5"
	585°/97 " "	579°/96 " "		
Gasoline - 155°				
Olefines:	2.0 %	3.0 %	1.5 %	2.0 %
Aromatics:	11.0 %	9.0 %	10.5 %	12.0 %
Naphtenes:	59.4 %	54.2 %	57.6 %	57.2 %
Paraffines:	27.6 %	33.8 %	30.8 %	28.8 %
Ultimate Analysis of Product:				
% C:	85.17	86.24	86.08	86.54
% H:	12.89	13.36	13.74	13.25
% N:	0.51	0.25	0.0	0.03
% S:	0.02	0.0	0.02	0.0
% Cl:	0.003	0.007	0.003	0.004

TABLE 8

Injected product for 6434 Stall

	<u>Gelsenberg-Product</u>		<u>Scholven-Product</u>	
	<u>Feb. 1940</u>	<u>May 1940</u>	<u>March 1940</u>	<u>May 1940</u>
D ₁₅	0.858	0.852	0.838	0.849
A.P.	∠ 111°F	∠ 112°F	∠ 124°F	∠ 117°F
Phenol g/l :	0.32	0.45	0.50	0.50

B. P. CURVE:

<u>Beginning:</u>	<u>320°F</u>	<u>307°F</u>	<u>315°F</u>	<u>316°F</u>
- 338°F	1.5 Vol.%	5.5 Vol.%	5 Vol.%	3.5 Vol.%
- 347°F	3 " "	9 " "	15 " "	5 " "
- 365°F	10 " "	23 " "	23 " "	16 " "
- 383°F	23 " "	41 " "	40 " "	30 " "
- 437°F	62 " "	70 " "	73 " "	68 " "
- 482°F	78 " "	82 " "	95 " "	86 " "
- 527°F	90 " "	92 " "	97 " "	95 " "
	550°/95 " "	567°/98.5" "	529°/98" "	558°/98.5" "

Ultimate Analysis:

% C :	86.29	86.33	85.51	85.55
% H :	12.67	12.84	13.28	13.03
% N :	0.49	0.28	0.48	0.49
% S :	0.45	0.34	0.43	0.24
% Cl :	0.004	0.005	0.003	0.005

TABLE 9

CATCHPOT OF 6434 STALL

	Gelsenberg-Catchpot		Scholven-Catchpot	
	February 1940	May 1940	March 1940	May 1940
A.P. d_{15}	0.760	0.750	0.736	0.755
	+ 118°F	+ 117 F	+ 127 F	+ 120 F
Phenol g/l:	0.023	0.0	0.0	0.0
<u>Column Distillation:</u>				
Liq. Gas	12.5 Wt-%	9.6 Wt-%	10.9 Wt-%	7.5 Wt-%
Gasoline 155°	44.1 " "	56.6 " "	49.0 " "	51.0 " "
d_{15}	0.732	0.722	0.720	0.732
A.P.	+ 117°F	+ 118°F	+ 127°F	+ 120°F
M.O.	42.8 Vol.-%	33.4 Vol.-%	40.0 Vol.-%	41.4 Vol.-%
d_{15}	0.839	0.831	0.816	0.830
A.P.	+ 118°F	+ 115°F	+ 130°F	
<u>Engler Vols. of Product</u>				
Beginning:	88°	91°	90°	93°
104°F	2.5 Vol.-%	1.0 Vol.-%	1.5 Vol.-%	1.0 Vol.-%
122°F	5 " "	4 " "	4 " "	3 " "
167°F	17 " "	15 " "	12 " "	13 " "
212°F	26 " "	28 " "	28 " "	29 " "
257°F	37 " "	41 " "	40 " "	40 " "
311°F	48 " "	55 " "	54 " "	55 " "
347°F	57 " "	67 " "	64 " "	65 " "
383°F	67 " "	76 " "	74 " "	76 " "
437°F	79 " "	83 " "	82 " "	86 " "
482°F	84 " "	86 " "	85 " "	89 " "
532°F	87.5 " "	491°/86.58 " "	507°/86 " "	487°/90 " "
<u>Gasoline - 155°</u>				
Olefines	3.0%	2.0%	2.0%	2.0%
Aromatics	8.0%	6.0%	6.0%	7.0%
Naphtenes	38.5%	44.4%	45.3%	47.0%
Paraffines	50.5%	47.6%	46.7%	44.0%
<u>Ultimate analysis of Product:</u>				
% C:	85.44	85.49	84.88	85.08
% H:	14.20	14.08	14.91	14.52
% N:	0.00	0.00	0.0	0.00
% S:	0.03	0.01	0.05	0.03
% Cl:	0.002	0.005	0.004	0.003

TABLE 10
Investigation of finished gasoline

	Gelsenberg Gasoline		Scholven Gasoline	
	April 1940	May 1940	April 1940	May 1940
d_{15}	0.7370	0.7380	0.7342	0.7418
Octane Number with no Pb	71.6	72.6	71.2	72.3
Octane Number with 0.9 Pb	-	87.7	-	87.0
Cu-Al Strips at 50°	v.g.	v.g.	v.g.	v.g.
Oxydation Test				
With no Pb: mg/100 ccm		0.0		1.0
With 0.9 Pb mg/100		1.2		1.8
Distillation Start:	140°F	140°F	140°F	140°F
V.P. 99.9°F		0.40		0.45
A.P.	112°F	113°F	116°F	110°F
Iodine Number	-	2.6	-	4.5
Dr. Test	-	Positive	Positive	Positive
% Mercaptan	-	0.0011%	-	0.0044%
Coef. of refr.	-	1.4090	-	1.4106
<u>Light Test</u>				
After 7 Min	-	1	-	3
After 15 Min	-	1	-	5
After 56 Min	-	1	-	7
After 120 Min	-	1	-	7
<u>ASTM Boiling Curve:</u>				
Beginning:	127°F	118°F	113°F	113°F
10%	151°F	147°F	144°F	147°F
20%	162°F	163°F	160°F	165°F
30%	176°F	174°F	174°F	181°F
40%	189°F	187°F	187°F	176°F
50%	201°F	201°F	201°F	208°F
60%	216°F	215°F	214°F	219°F
70%	230°F	226°F	230°F	235°F
80%	248°F	246°F	250°F	252°F
90%	275°F	275°F	279°F	279°F
95%	298°F	293°F	300°F	300°F
End Point	306°F	304°F	311°F	313°F
Olefines:	2%	1.5%	3.0%	2.0%
Aromatics:	9%	7.5%	7.0%	9.0%
Naphtenes:	41.5%	46.9%	48.0%	51.9%
Paraffines:	47.5%	44.1%	42.0%	37.1%
<u>Ultimate Analysis:</u>				
% C:	-	84.86	-	84.59
% H:	-	14.75	-	15.02
% N:	-	0.13	-	0.09
% S:	-	0.002	-	0.002
% Cl:	-	0.006	-	0.005

Scholsen, Nov. 24, 1938

Material Balance, Liquid and Vapor Phases

Industrial Div. III.

Computation Basis for the Flow Sheet

Draw. No III 2001a-2, 250,000 year-te Auto Gasoline.

The basic values for the flow scheme No III 2001-2 of Nov. 10, 1938 were changed as follows on the grounds of discussions in Leuna on November 24, 1938:

The large amount of rinsing oil later required by the Technical Division at Leuna for the 700 at. paste presses were taken into consideration.

C-content of the coal	83.5%
Utilization	96.%
Gasification	24.%
Catalyst addition	3.6%
Dry coal in paste	45.5%
Solids in paste, including catalyst (without the rinsing and vehicle oil)	50.3%
Solids in vehicle oil	8.0%

Liquid Phase

Intake:	66.2	te/h	crude coal with 12% water and 4% ash
	59.6	"	dry coal with 2% water and 4% ash
	58.2	"	anhydrous coal with 4% ash
	55.9	"	water and ash-free coal
	46.6	"	pure carbon.

Amount of paste (without the rinsing and glide oils):
 Dry coal 59.6 te/h.

Catalyst

Cat. I	0.75	te/h
Cat. II	1.15	"
Cat. III	0.50	"

Vehicle oil	2.4	te/h
	68.7	"
	130.7	te/h.

Total Solids in paste (before paste pressing):

Anhydrous coal	58.2	te/h
" catalyst	2.25	te/h
Solids in vehicle oil (68.7 ± 8)	5.5	te/h
100	65.95	te/h

$\frac{65.95}{130.7} = 50.3\%$

Dry coal in paste:

Coal with 2% water	59.6	te/h
--------------------	------	------

$\frac{59.6}{130.7} = 45.5\%$

Amounts of catalyst calculated from Nordstern valves:

referred to crude coal	$\frac{2.25}{66.2} =$	3.6%
------------------------	-----------------------	------

<u>Vehicle Oil:</u>		5.9 te/h
Rinsing Oil: 300 liters/paste press +		
+ addition for other purposes =		
$12 \times 0.3 = 3.6$ te/h + 0.9 te/h =		4.5 te/h

Total intake into the stall:

Paste including catalyst	130.7	te/h
Film Oil	5.9	"
Rinsing Oil	4.5	"
Fresh gas	6	"
Water	6.4	"
	<u>153.5</u>	"

Total output of stall:

Coal catch pot + water	61.2%	94.0	te/h
Coal catch pot (anhydr.)	52.4% = 80.5	te/h	
Water	8.8% = 13.5	"	
	<u>61.2% = 94.0</u>	te/h	

HClD	12.7	te/h
Hydrogenating gas, including inert gas	16.1	"
Losses in circuit, 0.45%	0.7	"
Total	<u>153.5</u>	te/h

HOLD:

42.7 te/h

Solids in HOLD:

Coal Ash	2.3 te/h
Catalyst Ash	2.35 te/h
Non-utilized Coal (4% of pure coal)	2.30 te/h
Solids in vehicle oil	5.50 te/h
Solids in film oil (gleitöl)	0.45 te/h
	<u>12.30 te/h</u>

$$\frac{12.3 \times 100}{42.7} = 30\%$$

Centrifuges:

Intake: 42.7 to HOLD + 58% thinning oil
= 42.7 + 24.7 = 67.4 te/h to be centrifuged

Solids in centrifuge mixture:

$$\frac{12.8 \times 100}{67.4} = 19\%$$

Output:

Centrifuge oil = 11%	ref. to HOLD =	48.7 te/h
Centrifuge residue 44%	" " "	18.7 "

Solids in centrifuge oil and residue:

Of the 12.8% solids in the HOLD, retained in the residue:

Coal Ash	2.30 te/h
Catalyst Ash	2.25 te/h
Not utilized coal	2.30 te/h
Solids in film oil	0.30 te/h
	<u>7.15 te/h</u>

$$\frac{7.15 \times 100}{18.7} = 38\%$$

Should 3% catalyst be added instead of 3.6%, the solids content will be reduced to 36.5%.

Solids in centrifuge oil:

Total solids in HOLD	12.8 te/h
Total solids in centrifuge residue	<u>7.15 "</u>
	5.65 "

$$\frac{5.65 \times 100}{48.7} = 11.6\%$$

Kiln:

Intake: 18.7 te/h fugal residue with 38% solids

Output:

Kiln oil = 49.7% (let balance)	= 9.3 te/h with 0.3% solids
Kiln residue = 48.8%	9.1 "
Gas = 1.5%	0.3 "
	<u>18.7 te/h.</u>

Oil Loss in Kiln:

Oil Intake $\frac{18.7 \times 62}{100} = 11.6 \text{ te/h oil}$

Loss: $22\% = \frac{11.6 \times 22}{100} = 2.55 \text{ te/h oil in the form of } 2.25 \text{ te/h solids and } 0.3 \text{ te/h gas.}$

The total composition of the kiln residue is

Coal Ash	2.3 te/h
Not-utilized Coal	2.3 "
Catalyst Ash	2.25 "
Solids from oil loss in kiln	2.25 "
	<u>9.10 te/h</u>

New water formation in the liquid phase:

Coal catch pot oil + water	94 te/h
Water content = 8.8% of total	
intake into stall = $\frac{152.5 \times 8.8}{100}$	= 13.5 "
Of this amount, injected	6.4 "
Injection water	6.4 "
2% in dry coal	<u>1.0</u>
	7.3 te/h

Fresh water formations:

13.5 te/h
 - 7.8 "
 5.7 te/h
 $5.7 \text{ te/h} = \frac{5.7 \times 100}{55.9} = 10\% \text{ referred to pure coal.}$

Coal Catch Pot Distillation

Intake: 80.5 te/h of the water free catch pot.

Output:

Heavy oil = 57%	=	45.8 te/h
A-middle oil = 38%	=	30.7 "
Coal gasoline (3.9% to 135°)	=	3.1 "
Loss	1.1%	<u>0.9 "</u>
		80.5 "

Heavy Oil Balance (H. O. tarbage):

Intake:

Coal heavy oil	45.8	te/h
Kiln oil	9.5	"
Centrifuge oil	48.7	"
	<u>103.8</u>	"

Output:

Vehicle oil	68.7	te/h
Film oil	5.9	"
Rinsing oil	4.5	"
Thinning oil	24.7	"
	<u>103.8</u>	"

Solids in vehicle and film oil:

Introduced with

Centrifuge oil	5.65	te/h
Kiln oil	0.30	"
	<u>5.95</u>	"

Vehicle oil + film oil

68.70	te/h
+ 5.9	"
<u>74.6</u>	"

with $\frac{5.95}{74.6} = 8\%$ solids.

Circulating Gas in the Liquid Phase

From High Pressure tests:

Intake gas (including cold gas and fresh gas) = 4m³ gas/l kg
pure coal = 55,900 x 4 = 223,000 m³/h.

Circulating gas at the washer outlet:

223,000 - 60,000 = 163,000 m³/h (sic).

Output of circulating gas pumps + 10% =

163,000 + 17,000 = 180,000 m³/h.

Outlet gas from washer:

= 163,000 + 14,000 = 177,000 m³/h

Amount of wash oil: according to record of 11/18/38 =

0.75 m³ oil/1000 m³ gas = 0.75 x 177,000 = 133 m³/h.

Vapor Phase

The data for the vapor phase were calculated from the data for case 7
(Drawing # A 526-8)

The amount of fresh gas for the gasolins converters were set at 30,000
m³/h and the amount recalculated in the proportion of 5:3 for the 5055 and
6434 converters.

Fresh gas for the 5058 converter	18,750	m ³ /h
" " " " 6434	11,250	"
	<u>30,000</u>	m ³ /h

Circulating gas for converter 6434:

(Telephone communication of high pressure tests)

Gas intake — 1.6 to 2 m³/liter injection
Average 1.8 " "

$$\frac{42 \text{ t/a}}{0.87} = 48 \text{ m}^3/\text{h} \times 1.8 = 86,250 \text{ m}^3/\text{h}$$

Amount of circulating gas for converter 5058:

Outlet gas: 3,600 m³/1 t injection =
= 3,600 × 30.7 = 110,000 m³/h

The amounts of hydrogenating gas have been obtained from the operation control data of 10/8/36. Technical Section III. Signed Schivy.

Sternberg/pEI

PADBIENIAK ANALYSIS
T.O.M. REEL 11

DATE	TIME	KIND OF GAS	% CO ₂ / H ₂ S	% O ₂	% H ₂	% N ₂	% CH ₄	% C ₂ H ₂	% C ₂ H ₄	% H ₂ O	% H ₂ Higher	% C ₅ H ₁₂ Higher	% CO
3/1/40	11:00	Circulating Gas in Front of Oil Wash	0.2	0.4	69.1	11.0	14.8	2.6	1.1	0.5	0.5	0.5	-
3/1/40	11:00	Circulating Gas behind Oil Wash	0.2	0.4	75.2	10.7	12.2	0.8	0.5	-	-	-	-
2/28/40	10:20	Cold Converter II, Step I	1.8	0.6	52.4	7.6	23.6	6.1	4.6	12.4	0.9	0.9	-
2/28/40	10:10	Cold Converter Step II	2.0	0.5	10.2	5.6	32.6	17.5	15.7	7.0	5.4	3.5	-
2/26/40	8:50	Cold Converter Step III	2.3	0.4	2.0	3.0	23.8	14.2	14.7	3.0	24.4	7.2	-
2/26/40	8:50	Washer Step I	1.2	1.3	47.3	11.4	27.4	6.1	2.7	2.4	0.5	0.5	-
2/26/40	8:20	Washer Step II	0.9	0.7	27.6	2.6	34.4	11.8	4.6	1.1	0.3	0.3	-
2/26/40	8:20	Washer Off Gas I / II (calculated)	0.9%	0.3%	40.3%	9.3%	31.4%	10.7%	4.5	1.37%	0.35%	0.35%	-
2/26/40	8:10	Washer Off Gas III	1.0	0.0	0.4	0.7	15.2	37.2	29.3	9.4	2.1	3.5	-
2/26/40	8:30	Vacuum Installation Off Gas	0.2	0.0	0.4	0.4	-	19.9	40.0	4.0	25.3	9.8	-
2/26/40	11:30	Off Distillation Off Gas	0.5	0.1	0.5	0.9	-	5.1	23.5	9.9	37.5	22.4	-
3/2/40	13:20	H.O.L.D. Gas	1.3	0.2	56.2	6.2	23.7	4.5	2.1	1.9	1.9	1.9	-
		Fresh Gas	0.2	0.2	96.58	2.52	-	-	-	-	-	-	0.5

LEUNA HIGH PRESSURE EXPERIMENTS
OCTOBER 16, 1942

THE SUITABILITY OF COALS FOR HYDROGENATION

2. Relationship between the hydrogenating ability of coal and its other properties.

Catalytical hydrogenation under high pressure has been developed in the course of time to such an extent that practically any coal occurring in nature, from the oldest coals to the youngest brown Coals can be readily converted into oil. That should not, however, give reason for the claim that any coal is equally well adapted to large scale hydrogenation, because with as complicated chemical substances as coals, numerous factors assist which will affect hydrogenation into one or the other direction. On the strength of our experience with different kinds of coal it is possible and appears desirable to select the kinds which are best suited for large industrial use.

Among the coals proper the soft coals and the gas coals with C content between 78% and 86% must be chiefly considered. There is a certain crude relationship between the C content and the suitability for hydrogenation; however, one can not decide on the hydrogenation behavior of the coal on the grounds of the carbon content only. Coals with the same carbon content may possess different capacities for hydrogenation, depending on their origin. Thus, a coal with 83% carbon from the Slesia Coal Bed is throughout somewhat less favorable to hydrogenate than the Ruhr coal with the same carbon content. The reason for it is in the higher oxygen content of the Slesian Coal with the same carbon content and lower volatiles and sulfur than in the corresponding Ruhr coal. There is, moreover, important difference in the composition of the ash in that the ash of the Slesian Coals frequently reacts alkaline as a result of the C_2O .

It is in general true that the younger (lower-C) coals are more readily hydrogenated than the older coals; the hydrogenation results are in every way more favorable with them. The conversion of the carbon is higher, the formation into benzine and Middle Oil is better, the quality of the Heavy Oil is higher, because it contains less asphalt and the gas formation is lower than with the higher-C coals (cf curves). A certain relationship between the capacity for hydrogenation and the contents on volatiles, which is nearly universally proportional to the C-content, may be established. The oxygen content is of great importance with respect to the hydrogen consumption and effects hydrogenation in causing the formation of higher-asphalt products with higher oxygen content in the coal. The chlorine content and sulfur content, both of which favor hydrogenation, are important. However, not only organic constituents, but also the inorganic, affect the ease of hydrogenation by their amount and composition. Higher ash content reduces

the utilization of the coal, but on the other hand acts favorably on the asphalt yield. There is a way of regulating the ash content of coals either by mechanical beneficiation or by proper choice of coal, and we may set the ash content of coals used in large scale hydrogenation to 4-6%. The de-ashing of coal results in the reduction of the reaction space and reduces the work on the residues. Coals with stronger alkaline, higher-CaO ash make the acid reacting catalyst largely ineffective, because they neutralize in part the active catalyst and the alkali damages the frequently used catalyst from the metals of the sixth group. For this reason such high ash coals are advantageously neutralized with free acids to improve the hydrogenation.

The petrographic structure of coals also plays a certain role, in addition to its elementary analytical composition. The individual constituents such as the Bright Coal, Splint Coal, Fusain and Slate Coal behave differently during hydrogenation. The former two can be very readily hydrogenated while Fusain and Slate are difficult to hydrogenate. The carbon content of Fusain is usually very much higher, so is its ash content with very much more CaO in the ash than in the Bright Coal and the Splint Coal. Bright Coal and Splint contain large amounts of tar-formers (plant spores and algae) it produces good yields of oil even in low temperature carbonization.

We may summarize the characteristics of coals well adapted to hydrogenation in the following table:

	<u>Muhr Coal</u>	<u>Sear Coal</u>	<u>Slesia Coal</u>
Per cent carbon in coal proper	less than 84	less than 83	less than 82.3
Per cent volatiles in coal proper	over 38	over 37	over 37
Per cent ash in dry coal	low 6	not over 6.5	not over 6.5
Per cent CaO in the ash	low	low	low
Fusain and Splint content	low	low	low

Brown Coals are all readily hydrogenated because of their low C content, but in their case also the relationship between hydrogenation and carbon content may be observed, although not as pronounced as with coals. The hydrogenation is affected by the ash content, its composition, sulfur content and, most of all, by the oxygen content. Low sulfur and high oxygen Brown coals give upon hydrogenation a high asphalt as well as frequently unstable high phenol reaction products. The high sulfur content favors the reduction of asphalt and its breakdown. A high oxygen content is also unfavorable, because of the high oxygen consumption. The high ash content is objectionable for the same reasons as in the coals. The ash forms skeleton structures in the brown coals, and the Ca is in part combined chemically with the humic acid, and the mechanical de-ashing, such as frequently done with coals can not be readily produced. A neutralization of the more strongly alkaline ash with acid will therefore result in greater advantages than with the coals, the ash content of which is mostly lower. No improvement of hydrogenation by neutralization is produced in the now commonly used alkaline and catalyst and the neutralization will therefore be omitted.

Brown coal which can be readily hydrogenated will therefore have the following properties:

Ash Content	Below 13%
Oxygen Content	Below 25%
Sulfur Content	High

Pitch coals may be caused from their analytical composition as being older Brown Coals in so far as their hydrogenation is concerned, and virtually the same applies to them as has been said about Brown Coal.

2. Effect of Pressure on Hydrogenation of Coal.

Raising the hydrogenation pressure results in general in stronger hydrogenation and reduces the formation of the lower-hydrogenation products, which gives us the possibility to work at higher temperatures than can be done at lower pressures. High pressure will produce higher reaction velocities and will permit one to use up more coal in a given length of time. The use of higher hydrogenation pressures permits the utilization of older coals, because the effect of raising the pressure in this case upon the utilization of coal and the reduction of asphalt production is much greater than with younger coals. With the latter, raising the pressure permits increasing the through-out which results in an increased yield and lower gas formation.

Moreover, cheaper and non-corrosive catalyst (iron) can be used with higher pressures, because not raising the pressure is more effective with the less active catalyst than with the highly active ones. It is thus possible to obtain better results with iron catalyst at 700 atm. than with tin and chlorine at 250 atm. pressure. Similarly by increasing the reaction temperature at higher pressures the yield may be increased by about 50%.

CRASSL

Gasification Data for Gladbeck Works

T-13

Calculated from:

- a) Values obtained from Upper Silesia
- b) Nordstern gasification for liquid phase, Scholven for vapor phase

a) Coal throughput and gas formation:

	Lean Gases m ³ /hr. (15°, 735 mm)			
	Pure Coal te/hr	Coal Stall 50 atm.	Washer 50 atm.	Vapor Phase 26 atm.
Upper Silesia	48.1	3,620	13,800	2,160
Gladbeck	89.6	6,750	25,600	4,040

	Rich Gases m ³ /hr. (15°, 735 mm)			
	Coal Stall 1 atm.	Washer Off Gas 1 atm. plus Vacuum	Vapor Stall 1 atm.	Catch Pot Off Gas
Upper Silesia	1,350	5,320	3,880	2,450
Gladbeck	2,520	9,950	7,210	4,560

We get the following amounts of hydrocarbons (in te/hr.) from these gas amounts and the analyses of gases in Upper Silesia.

	C ₁	C ₂	C ₃	C ₄	Total	Carbon Content
Coal Phase	6.25	4.95	7.18	3.1	21.48	17.8 te/hr.
Vapor Phase	0.73	0.29	2.02	8.85	11.89	9.9 te/hr.
Total	6.98	5.24	9.20	11.95	33.37	27.7 te/hr.

atm. operation
 With 300 (tons) production per day, we get a total gasification of around
 240,000 year/ton of hydrocarbons.

The Available Gasified Hydrocarbons.	139,300	165,300
To be supplemented by Lean Gas Production, year-ton Hydrocarbons	16,100	16,100

If we assume that the lean gas (3 / 1 generator, with 38,000 year-ton coke consumption) is added to the total gas, the heating value of the fuel gas will amount to

3,400 or 3,540 W.E. Ho (Heat units, upperheating value?) m³/hr.

The heating value of the fuel gas coming to the coking plant for computation will amount to

2,800 to 2,850 W.E. Ho m³/hr.

if the lean gas battery be erected upon the coking plant grounds and the gas be returned to Hibarnia.

With reference to the gasification of the hydrocarbons from the amounts of gas given, we get the following:

a) During the production of 22.5 te/hr. VI 705:

	Lean Gas	C ₁	C ₂	C ₃	C ₄
Buna Deliveries, in yr/te Hydrocarbons	-	18,000	15,000	20,000	15,000
C ₄ Deliveries, in yr/te Hydrocarbons	-	-	-	-	16,000
Fuel Gas Deliveries, in yr/te Hydrocarbons	-	-	1,000	10,000	15,000
Used up in Heating, in yr/te Hydrocarbons	16,100	4,700	12,500	6,600	5,500
Total Consumption	16,100	22,700	28,500	36,600	51,500

b) In the production of 27.0 tons per hour of VI 705:

	Lean Gas	C ₁	C ₂	C ₃	C ₄
Buna Deliveries in yr/te Hydrocarbons	-	18,000	15,000	20,000	15,000
C ₄ Deliveries in yr/te Hydrocarbons	-	-	-	-	16,000

	Lean Gas	C ₁	C ₂	C ₃	C ₄
Fuel Gas Deliveries in yr/te Hydrocarbons			1,700	17,000	25,300
Consumption for Heating in yr/te Hydrocarbons	16,100	8,950	17,150	6,400	4,800
Total Consumption	16,100	26,950	33,850	43,400	61,100

SUMMARY:

About 80% of the C₁ and C₂ hydrocarbons were consumed in operations heating, i.e. the erection of a 6th coal stall will not permit replacing the C₂ and C₄ hydrocarbons in the Buna case by the C₁-C₂ hydrocarbons from the additional gasification. We had in this case, i.e. by substituting about 9,600 yr/te of C₁-C₂ hydrocarbons in the heating gas by the lean, reduced the heating value of the gas from

3,400 to 3,050 W.E. Ho m³/hr.

/s/ SCHIWI

I. G. Standards Book

F-14

SPECIFICATIONS

Steel K 2 A

Works Standard
12-80

Designations

I.G. Abbreviations
I.G. Key standard
Works designations

K 2 A
Croco 1, 1/4
Krupp: F 54

K 2 M
Manko 1, 1/4
C 56

Mauesmann:	Marwo 156 C	Marwo 582 Z
Baldenhitte:	D 2	-
D.R.W.:	-	K 2 M
Rheinmetall:	-	Mn 3 V/W
Rhurstahl:	D 3423	D 2418
Silesiastahl:	-	-

Method of Smelting:

Electric or high grade S.M. steel.

Uses:

Piping of Class B (v. Technical Specifications, #2).

Chemical Composition

Former values, K 2 A
C = (0.23) - 0.28%

Cr 1.0 - (1.5)%
Mo --

Mn (0.6 - 0.9%)
Si < 0.4%
V --

Present values for K 2 M
C = 0.22 - 0.28%
(at most 0.30%)

Cr none
Mo none

Mn 1.1 - 1.4%
Si 0.40 - 0.60
(V = 0.08 - 0.15%)

Max. permissible contents
according to specification
603, 8/17/42 #1, of
the list E 23 b, 7/1/42
of the Reichsstelle für
Eisen und metalle.

Mn = 1.4
V = (0.15%)

P and S: Maximum contents P = 0.03; S = 0.03, P+S = 0.05% for E steel
P = 0.04; S = 0.04, P+S = 0.07% for S.M. steel
in accordance w. DIN 1601 U, July, 1942.

For production of good weldability and maintenance of tempering values, the composition is limited to air tempering (not oil).

Tempering* Standards

Yield Point	Min. 35, preferably over 40 Kg/mm ² .
Tensile Strength	Min. 55, (preferably over 60) - 70 Kg/mm ² **.
Elongation	L = 5d lengthwise
Round rod	Standard 18%
Piping	-
Strips	-

Notched bar strength: for wall thickness <40 (<1.5748"), lengthwise 8, crosswise 5 mkg/cm²
for wall thickness >40 lengthwise 6, crosswise 4 mkg/cm²

Tempering Index, short rod lengthwise	L = 5d	1100	round rod strips
	(L = 10d)	900	
	(L = 10d)	800	

Brinell hardness about 170 - 200

The low yield point & tensile strength are for wall thickness
in excess of 40mm (>1.5748")
weldability*

Flush welding	good
Electric arc welding	good
Oxy-acetylene welding	only possible with great care, however better than with higher-Cr material.

Directions for Heat Treatment:**

1) Piping:

Tempered by cooling in air after heating above A₃ at the usual normalizing temperature of 800 to 920° C (1452 - 1688° F), followed by annealing at temperatures above 600° C (1112° F) (air tempering).

Directions for "Air tempering" does not exclude more accelerated cooling, e.g. in artificially produced air stream acting uniformly over the whole length of the tube, or in an oil bath. Particular attention must, however, be paid in such cases when testing by the receiver, that the chemical composition be within the

* Data are valid for K 2 A and also for K 2 M

** To facilitate reaching the required yield point of at least 40 Kg/mm², the upper limit of tensile strength may occasionally be exceeded up to 5 Kg/mm².

required limits in order to be assured that, in subsequent local after-tempering, welding will produce welds of required temper with normal air cooling, or in heat treatment of bands when hardening is not possible by accelerated cooling.

2) Local Treatment of Welds:

Automatic flush welding:

Oxyacetylene welding:

Arc welding:

Tempered as in 1)***

Tempered

In most cases sufficiently annealed.

It must however be judged for each individual case, with special consideration of the electrodes used.

***In most cases, a local heat treatment is sufficient. With simple annealing heating, the notched bar strength in the region of the weld is generally relatively low.

N5 Replacement Material
(N5A - N5B - N5C - N1K)

Works Standards
12 - 80

*)

4-Mn-free replacement materials with the following characteristics and principal applications are considered, namely:

- 1) N5A. Replacement material for N5 (A = Austausch, - replacement, interchangeable), strength the same as (soft) N5 and the resistance to deformation at 500°C (932°F) practically unchanged.
- 2) N5B. Special steel with an especial tensile applicability for tubular bundles (B = bundled tubing).
- 3) N5C. Special steel with higher C-content, especially for castings (C = with increased carbon content).
- 4) N1K. Special steel, particularly adapted to "cored" tubes for high pressure vessels with wickel wall, a modification of steel N1 (K = Kernrohr, "core" tubing).

Markings of Materials:

I. G. abbreviated notations:	N5A	N5B	N5C	N1K
I. G. Key designations:	Crovan 2½, 1/10	Cro 2½	Crovanko 2½, 1/10, 0.15	Croko 2, 0.15
Manufacturers notations:				
Krupp	FM 2121	F 212		F 210
Mannesmann:	Marwe 213 CV	Marwe 212 C	Marwe 522 CV	Marwe 521 C
Baillodhütte:	HD 5 A			
D.R.W.:	N5A	N5B	N5C	C 2215
D.E.W.:	HC 5			
B.V.:	N5A/J			N1K/J
Ruhrstahl:	D 3325 A	D 3325 B	D 3325 C	D 3507

Methods of Production:

N5A and N5B

N5C and N1K

Electric steel, in most cases from arc furnaces; S.M. duplex steel, insofar as the production is subject to process testing.

Electric steels, and very carefully smelted S.M. steels.

Applications:

N5A	N5B	N5C	N1K
Gaskets, for special cases of tubing, classes C and D; Construction pieces, as with N5C. Sheets clad with high-alloy steels	Piping, Class B, especially the so-called pipe bunches, in special cases also tubing class C; (gaskets).	Forged and cast pieces, especially built-in-parts (rod pipes, piping, etc.)	"Cored" tubes with nickel bodies (seamless rolled and welded) and corresponding forgings.

(Piping, classes C and D, see Technical Specifications, Section 2)

Chemical Composition:

N5A	N5B	N5C	N1K
C = 0.07 - 0.13%	0.07 - 0.13%	0.12 - 0.20% ***	0.12 - 0.20%
Cr = 2.5 - 2.8%	2.5 - 2.8%	2.5 - 2.8%	2.0 - 2.3
Mn = 0.3 - 0.5%	0.3 - 0.5%	0.3 - 0.5%	0.3 - 0.5
Si ≤ 0.4%	≤ 0.4	≤ 0.4	0.20 - 0.40
V = 0.1 - 0.2%	-	0.1 - 0.2%	(0.1 - 0.15%)
Mo -	-	-	-

P and S, maximum, each 0.03%, P&S max 0.05% for electric steels P & S ≤ 0.07%

The highest permissible contents, in accordance with regulations for exceptions 603, of 8/17/42, # 5 for N5 steels, and # 15 for N1K steel, from specifications E 23 b of 7/1/42 of the Reichsstelle für Eisen und Metalle:

Cr = 2.8	2.8	2.8	2.3
Mn = 0.8	0.8	0.8	0.8
V = 0.2	0.2	0.2	0.15

Tempering Standards:

Materials:	N5A	N5B	N5C	N1K
Brinnell Hardness: Tubing and castings air tempered) oil tempered)	140-170 for tubing	140-170 for tubing	abt $\frac{1}{0.35}$ * tensile str.	
Brinnell hardness for high pressure gaskets: 325/700 at.	180-220 Tolerance to 160	(180-220)	(180-220)	

Materials:	N5A	N5B	N5C		N1K	****	****
Uses:	325 & 700 at.	Construction tubes, 325/700	325/700 atm.	General standards 325 & 700 at.	"Cored" tubes 325 at.	"Cored" tubes 700 at.	
Resistance to creep strength, kg/mm ²		← about 30 →		Abt. 35	at 20° >30	at 350° >20	at 20° >35
Tensile str., kg/mm ²		about 50	abt 55	abt. 55	50 - 60 (65)		55 - 65

In agreement with the materials and construction requirements for stationary steam boilers, Section IV d, in peripheral direction.

Elongation	L = 5d lengthwise			With tensile str.	Elong L = 5d
				55 kg/mm ²	> 20%
				60 kg/mm ²	> 18%
				65 kg/mm ²	> 17%
Round rod	> 22%		> 20%		
Piping Strips					

Notched bar strength, kg/cm ²	N5A	N5B	N5C	N1K
	Lengthwise 12	Lengthwise 10	Lengthwise 10	About 8
	Crosswise 8	Crosswise 7	Crosswise 7	> 8
				> 6

These values apply to circular or cross sectional directions.

Tempering standard on short rod Lengthwise: 1200 cm a worked sample 1000 cm a tubular strip, ripped as a tube.

Weldability:

N5A, N5B	For butt welding: good) Much attention to be paid to proper preheating and immediate annealing.
N5A, N5B, N5C, N1K	For electric welding: good	
N5A, N5B	For oxyacetylene welding:	Can be done exceedingly carefully, however, somewhat more readily than with 3% Cr steels with higher C-content.

Directions for Heat Treatment:

N5A, N5C, N1K: Tempering and hardening from the normalizing temperature in air or in oil, followed by annealing to prescribed strength or hardness (annealing time at least 3/4 hour).
 With N5A, N5C (and also with N1K) the heating temperature may be raised

in special cases to 1000 - 1020°C (1832 - 1868°F), in order to take full advantage of the strength-increasing action of the V contents.

N5B

Unless otherwise directed, heat treated to tensile strength of about 50 kg/mm².

- a) Tempering by cooling in the air after heating above A₃, followed by annealing at about 730°C (1346°F)
- b) A somewhat simplified treatment may occasionally be used for cold drawn thin-walled tubing.

Treatment of Welds:

N5A and N5B:

Automatic butt welding: Annealing (about 3 hrs) further directions about time of heating given in Section 12.

N5A, N5B, N5C

Arc welding:

A simple annealing heat frequently suffices instead of tempering.

A usual after-treatment is in most cases sufficient.

N5A and N5B

Oxyacetylene welding:

Tempering as in a). In certain cases annealing is sufficient.

N1K

Arc welding:

Tempering or annealing of collars after welding of longitudinal seams. In peripheral welding a simple annealing may be used for large dimensions of tubing.

* The subdivision into 4 steels does not introduce difficulties in results from different manufacturers of the steels. The principal applications are the following:

N5A. Gaskets and clad sheets.

N5B. For pipe bundles, to replace N5A which offers difficulties in the production of longer cold-drawn thin tubes.

N5C. For steel castings and built-in tubes of larger dimensions.

N1K. For large sheets for core tubes or for very large rolled pipe shoulders for core tubes.

- ***) Insofar as there is an advantage in, e.g., the higher resistance to creep strength in comparison with N5B or the lower C-content against N5C, possibly for reasons of behavior on welding, or resistance to compressed hydrogen.
- ****) The C-content must be limited as much as possible because of welding properties. The higher values enter into consideration when higher elongation and tensile strength are required. The higher C-content of N5C and NK in comparison with N5A facilitates production, in particular the smelting of the steel in the S.M. furnace, which is especially emphasized by the manufacturers because of limitation of production capacity.
- *****) In special cases values are provisionally set from engineering calculations.

Dr. Sternberg

Markings of the Material:

I.G. Abbreviated Notation:	NSA
I.G. Key designation:	Crovanko 2, 1/4, 1/5
Manufacturers' notations:	Krupp: FMS
	DEW: ICS
	Mannesmann: Marwe 243CV
	B.V.: NSA/J
	D.R.W.: NSA
	Ruhrstahl: D 3513
	Rheinstahl: NSA
	Röchling: NSA

Method of Production:

Electric steel, in special cases S.M. Duplex steel.

Directions for Use:

Presently only up to 400°C (752°F) continuous temperature of material until additional information is available on the tendency of the steel to produce annealing brittleness from effects of long heating to higher temperatures (measured at 20°C, 68°F) and until the resistance to creep strength originally determined for the construction parts made of NS material is satisfactory for structural parts at above 400°C (752°F).

Chemical Composition:

Former values	Present specifications	Max. permissible amount according to approved exceptions 603 of 8/17/42, #2, of the order E 23 of 7/1/42 of the Reichsstelle für Eisen und Metalle.
C = 0.20 - 0.25%	0.19 - 0.24%**	
Cr = 2.5 - 3.0	2.0 - 2.3%	2.3%
Mn = about 0.25%	0.2 - 0.3%	0.3%
Si = 0.3 - 0.5	0.3 - 0.5	0.8
S = 0.25 - 0.4	< 0.4	

with a high degree of purity with respect to P and S:

Max %: P = 0.03; S = 0.03; P+S = 0.07.

. 2 .

Tempering Standards:***

- a) As delivered by the manufacturer, when a subsequent annealing of the whole unit or in the neighborhood of a weld is intended:

In this condition, the values given under b) are basically acceptable, provided the tensile strength and hardness values are mostly inside the upper half of the range given. To what extent the upper limit may be exceeded will depend upon proofs of a sufficient toughness.

- b) As used.

Brinell hardness:

Individual tests, about 220-260.

Experimental results are being collected for a conversion factor (tensile strength to hardness).

	<u>tempered***</u>	<u>annealed</u>
Yield Point	50 kg/mm ²	No performance values have been established for this state. The tubes must be easily machined in the relatively soft state and be weldable.
Tensile Strength, 20°C	75-90 kg/mm ²	
Elongation	L = 5d lengthwise	
Round rod	> 16%	
Tube	--	
Strip	--	

Notched bar strength: "Maximum" when using standard (DVMR) samples.
Standards: lengthwise 6-8 mkg/cm²
crosswise 4-5 mkg/cm²

The 70-90 kg/mm² limit for tensile strength and Brinell hardness of 220-260 are permitted for tubes of nominal width of 8, 10, 16 and 24. The hot yield point and the resistance to creep strength, ^{at 400°C} must approach as nearly as possible the values of the N-8 material. The resistance to creep strength at 400°C > 33, at 500°C > 10. The value of cold tensile strength must be considered from this view point. The requirement of resistance to creep strength at 400°C of at least 28 kg/mm² must be used as a basis for testing. Occasionally up to 25 kg/mm² may be allowed (DVM test with 5 x 10⁻⁴ %/h rate of elongation) with a 0.2% yield strength of at least 33 kg/mm² (standard 40 kg/mm²).

The tubes will be generally obtained from the manufacturers in the tempered state.

Weldability:

Butt-welding:

Good.

Arc welding:

Good. At present only with Mo-containing electrodes. Much attention to be paid to proper preheating and immediate annealing.

Oxyacetylene welding:

Only with extreme care; not recommended.

Directions for Heat Treatment:

A. Tempering of individual tubes:

Hardened by quenching from 950-980°C (1742-1796°F), e.g. in oil (with smaller cross sections also in a current of air) followed by careful annealing to the prescribed hardness over a time interval of at least 3/4 hr.***** Individual manufacturers have obtained good results by quenching in water at 87°C.

The hardening temperature indicated is considerably above the Ac3 point. It is a provisional temperature until additional experience is gained. Superheating must be avoided and a maximum strengthening effect of the V addition must be produced. The tendency to annealing brittleness during long continued operations should be less in the oil quenched than in the air quenched state.

B. Local treatment of welds and the treatment of weld-joined systems of tubes.

1) Working of tempered tubes:

After welding of fin-sheets, the tubes are generally subjected to a de-tensing heating somewhat below the annealing temperature. The hardness must not here become unpermissibly low.

Upon completion of a circular weld, it is locally heat treated. There exist the following possibilities:

- a) Local annealing; this must be carefully done at a suitable temperature (within the interval of 650-700°C (1202-1292°F), and without softening of the affected zone (see section 12). A lower notched bar strength immediately in the welded seam is produced, limiting the after-treatment of the automatic butt-welds to simple annealing.
- b) Local tempering of welds according to A, occasionally with an intermediate normalizing annealing for grain refinement in the superheated zone. The softened zone formed on both sides of the post-tempered zone will be kept by suitable means as narrow as possible.

A purely de-tensing annealing appreciably below the annealing temperature immediately upon the conclusion of circular welding does not replace the careful after treatment given under a) and b), which produce a high toughness with sufficient strength, in particular resistance to creep strength, in the region of the weld.

2) Working of annealed tubes:

When a post-tempering of the welded tubes is foreseen, it is done as given under A. After the circular welding is finished, the immediately following annealing may exceed the usual annealing temperature, not

however above the lower transformation point, in order to produce high toughness and to make the as yet not-tempered length of tube in the neighborhood of the joint insensitive to transportation strains.

Lengths of tube hardened by tempering and not yet annealed are to be handled with particular care during transportation

* The composition and the resistance to hydrogen under pressure are altered during welding, especially in the fusion butt welding machine. The hot strength properties at 400°C must therefore be set under conditions of not excessively high cold-strength, which will permit also local heat treatment of welded parts at relatively high annealing temperature. It is desirable that the manufacturers should take into consideration any new information on the possibility to suppress the tendency of chrome-vanadium steels to annealing brittleness over longer heating periods. The phosphorous content, e.g., must be kept as low as possible. When setting the analytical limits for Mn, V, C and Cr, the fact was taken into consideration that raising their contents increases the tendency to annealing embrittlement.

** With the upper limits of 0.18 - 0.25%.

*** These values are used provisionally, until further information becomes available.

**** The receiving requirements for tempered tubes and for section of tubes for testing.

***** Information is to be collected on the tendency of the steel to annealing embrittlement during slow cooling from the annealing temperature of the individual melts and in different condition of manufacture. When there is danger of annealing embrittlement the cooling from the annealing temperature to at least around 400°C must be done rapidly; should annealing embrittlement have taken place, a subsequent treatment must be undertaken, consisting of re-tempering or of heating a little below the annealing temperature and rapidly cooling over the critical temperature interval.

I. G. Specifications for Material N9

September 1943

Markings of the Material:

I. G. abbreviated notation:	N9
I. G. key designation:	Cromovan 2½, 1/4, 6/10
Manufacturers' Notations:	
Krupp:	FKM9
Mannesmann:	Marwe 245 EV
D.R.W.:	N9
Röchling:	N9
Ruhrstahl:	D 5517
D.E.W.:	HC9
Baildonhütte:	HD9
Rheinmetall:	N9
B.V.:	N9/J

Method of Manufacture:

E-Steel (Arc furnace product is usually understood under E-Steel).

Use:

Tube of Class C (v. technical specifications, Section 2.)

Chemical Composition

Former Values	Present Specifications	Highest permissible contents according to permitted exceptions 603, of 8/17/42, # 3 of the order E 23 b, of 7/1/42 of the Reichsstelle für Eisen und Metalle.
C = 0.19 - 0.24% (outside limits 0.18 - 0.25%)	0.18 - 0.25%	
Cr = at least 3 - 3.5%	2.5 - 2.8%	2.8%
Mo = 0.2 - 0.3%	0.2 - 0.3%	0.3%
V = 0.45 - 0.55%	0.55 - 0.65%	0.65%
Mn = 0.25 - 0.50%	0.3 - 0.5%	0.8%
Si = < 0.4%	< 0.4%	
with a high purity to P and S: Max. %: P = 0.03; S = 0.03, P & S = 0.07		

The analysis of material handed to the receiving official must be particularly reliable with respect to the carbon content, because the chemical composition forms the basis for connecting the tubes to bundles of tubes and furnishes information for the heat treatment in later working of the tubes.

Tempering Standards:*

Brinnell hardness:

Tempered

Annealed

a) As delivered by the manufacturer, or, if a subsequent re-annealing is intended; v. sheet 2, Section 3.

Standards have not been established for this state. The tubing must be relatively easily worked in the soft state and be suitable to welding

Air Tempered:
formerly:

at least 230 (possible min. 240) - 280

oil tempered:

240 - 280 **

b) As used:

air tempered:

formerly: (at least 220 to about 260

oil tempered:

about 240 - 280 **

At present

240, - 280 **

Standards for Tensile Strength:

Tempered

Air tempered:

(formerly 75-90 kg/mm²)

at present 80-95 kg/mm²

oil tempered 80-95 kg/mm²

The following limits are permissible in tempered tubes of Nom. width 6, 10, 16 & 24:

Tensile strength 80-100 kg/mm²

Hardness 240 - 300.

The values for hardness have a predominating character over tensile strength.

In this case the tensile strength values are of greater importance than hardness.

Yield Point:

Air tempered: at least 50 kg/mm²

Oil tempered: " " 55 "

Elongation L = 5d lengthwise

Round rod (at least 15% with a strength of 75-90 kg/mm²)

at least 14% " " " " 80-95 "

Tubing

Strips

Notched bar strength "maximum" when normalized (DVHR) samples are used.

Standards: air tempered, lengthwise 6 - (8) mkg/cm^2 , crosswise 4 - (5) mkg/cm^2
oil tempered, " 8 " " " " "

Tempering standards of short rods

Lengthwise 1100 - 1200

Min. resistance to creep strength at 500°C N/kg/cm^2 ***

The creep strength resistance (resistance to deformation) in the oil tempered state (assuming equal annealing hardness and equal cross section of the pieces) is in most cases lower than in the air tempered state. It increases with increasing annealing hardness. The resistance to creep strength is not the only deciding factor for the behavior of the material in practice, but the high resistance to creep strength alone is insufficient for approval in practice, and the behavior of steel under high mechanical stresses when under action of highly compressed hydrogen has a deciding influence.

Whenever the purchaser calls attention to the necessity of having tubing delivered in the hardened state, undergo additional annealing heating either as a whole or locally, as in the neighborhood of welds, it is recommended that the manufacturers of tubing maintain the values of hardness at the upper limit.

Weldability:

Fusion butt welding:

good at cross section suitable for machine operation.

Arc welding:

is now being successfully developed. Much attention must be paid to proper preheating and immediately following annealing heating.

Directions for Heat Treatment:

A. Tempering of single tubes.

Tempering by:

1) Hardening after heating to 1000 - 1020°C (at a suitable composition, and when conditions are very favorable with respect to furnace operations and temperature control) to at least 980°C with sufficient time during quenching.

a) in artificially produced air current, uniformly acting over the whole length of the tube.

b) in an oil bath. Oil quenching comes particularly under consideration for castings. Hardening in water should not be done.

In case of double hardening, as in the case of a required later treatment, special combinations may be used with suitable tests (structure, toughness, resistance to creep strength):

2. Annealing to the required hardness; kept for several hours (3 to 6 hours, depending on the annealing temperature).

For quenching hardness when controlled in the air tempered state, the standards are 360 to about 400. The values are higher in the oil tempered condition.

The pause during heating for hardening is determined with a viewpoint of producing a complete uniform solution, especially that of vanadium carbide, for high resistance to creep strength, while avoiding overheating or too prolonged heating which will lower the ductility.

A special attention is called to the necessity of having the heat treating effects during hardening as well as annealing spread over the whole tube, as well as for using suitable tests. The rate of cooling during hardening should be at least 25°C per minute in the temperature interval of 800-600°C, which corresponds to a cooling rate of 100° in 4 minutes. It is advisable to record during operations also the total cooling time from the hardening temperature to 600°C.

The pieces must have proper markings to indicate whether hardening was done in an air current or in an oil bath.

The material must possess in the tempered state a uniform, sufficiently fine grained well developed temper structure, without pronounced grain boundaries or free ferrite. The surface, in particular the inner surface must be as nearly free as possible from decarbonizing phenomena or from other unfavorable effects upon the structure of the outer zone caused by heat working or heat treatment. Understanding must be reached on this point in individual cases. (where drilling of the tubes is intended, this should preferably be done after the conclusion of heat treatment).

In general, tubing must be delivered by the tubing manufacturers in the tempered state, unlike the N10 tubing which must in most cases be delivered by the manufacturer in the annealed state and only be tempered after subsequent working in order to produce the greatest uniformity in even the welded parts of the material.

B. Local treatment of welds and the treatment of systems of tubing joined by welding.

1). Working of tempered tubes:

After the fin-sheets have been welded on, tubes are in general subjected to a de-tensing or annealing heating. Hardness must not become unduly lowered.

After the completion of circular welding it must be subjected to a local heat treatment. There exists the following possibilities:

- a). Local annealing heatings; this must be carried out carefully at the proper temperature (within the temperature interval of 650 and about 700°C) and without appreciable softening of the affected zone (v. section 12).
- b) In special cases a local tempering of the weld as in A, occasionally with intermediate normalizing heating for grain refining in the superheated zone. The softening zone on the two sides of the re-tempered zone must be kept at a minimum by the use of suitable means.

A true de-tensing heating, appreciably below the annealing temperature, immediately upon the completion of the circular welding, is not a substitute for the careful after-treatment described under a) and b), and is intended to produce in the weld a high ductility with sufficient strength, particularly creep resistance strength.

2) Working of annealed tubing:

When an after-tempering of the whole welded tubes is planned, this is done as directed in A. The usual annealing temperature may therefore be exceeded during the annealing heating which immediately follows the circular welding, but not above the lower transformation point, and produce in this way a high ductility and a freedom from stresses, making the welds of the tempered system of tubing insensitive to strains during transportation.

Systems of tubing hardened during temper treatment and not yet annealed must be particularly carefully handled during the necessary transportation.

- * Receiving directions for tempered tubing or sections of tempered tubing for testing.
- ** Preliminary values before the availability of further experiments on resistance to creep strength and time resistance to creep strength under the influence of hydrogen under pressure; with sufficient ductility, in special cases up to 300 Brinnell is permissible.
- *** (DVM) process, but referred to a rate of elongation of $5 \times 10^{-4}\%$ /h instead of the usual $10 \times 10^{-4}\%$ /h measured between the 25 and 35 hours of test, or 500 (1000) hour test with 3/4% (1/2%) permanent elongation/year measured during the 500 (1000-1100) hour.

I. G. SPECIFICATIONS FOR N-10 STEEL - SEPTEMBER 1943

MATERIAL N-10

Markings of the Material:

The I. G. abbreviated notation: N-10

The I. G. key designation: Grossowran 2 $\frac{1}{2}$, 1/3, 2/3, 3/4.

Notations of different manufacturers:

Krupp	FKDM 10
D.E.W.:	MC III V
Mannesmann:	Marwe 246 EVW
Baldenbutte:	HD 10
Ruhrstahl:	D 5610
Rheinmetall:	N 10
Roehling:	N 10
D.R.W.:	N 10
Mitteld. Stahlwerke:	N 10

Method of Production

E-Steel (E-steels are mostly materials made in the arc-furnaces.)

Uses

Tubes of the Class C (v. technical requirements, Section 2.)

Chemical Composition

Highest permissible content according to specifications GOK of 8-17-1942, # 4 from the arrangement 5 13 G of July 1st, 1942 of the State Institute for Iron & Metals.

Former Values

Present Specifications

C = 0.19 - 0.24%	0.18 - 0.25%	
(extreme limits 0.18 - 0.25%)		
* Cr greater or equal 2.5%	2.7 - 3.0%	3.0%
V = 0.70 - 0.85%	0.70 - 0.85%	0.85%
Mn = 0.25 - 0.50%	0.30 - 0.50%	0.8%
Si less or equal 0.4%	0.4%	
Mo = 0.35 - 0.50%	0.35 - 0.45%	0.45%
N = 0.3 - 0.5%	0.30 - 0.45%	0.45%

* Remarks: The original chromium content 2.8%; this has been changed by direction of the State Institute ES II 6276 Rtz/Bch of April 3, 1943 to 3%.

With a high degree of purity with respect to P and S: Maxima in percent: P = 0.03, S = 0.03 and P + S = 0.07

The manufacturer's analysis furnished to the fabricators must be particularly dependable with respect to the carbon content, because the chemical composition furnishes the basis for connecting the tubes to return bends and for the heat treatment in the subsequent handling of the tubes.

Tempering Standards

Brinell Hardness:

Tempered

In the tempered state the hardness was:

Formerly:

Tempered in the air 210 (min. 200) - 250
(connecting tubes)

220 - 260 for heated hairpin tubes
and flanged tubes)

Oil tempered: 240 - 280 *
(connections, forged pieces)

At present
240 - 280 *

Annealed

No definite norms have been established for this state. The tubes must be relatively soft, readily workable and suitable for welding.

When the tubes are to be subsequently welded, they are usually delivered by the manufacturer in the annealed state and only tempered after the later operations so as to produce the greatest uniformity in the region of the welds.

Directions for Tensile Strengths:

Air Tempered - formerly 70-85 kg/mm²
At present - 80 to 95 kg/mm²

Oil tempered:
80 - 95 kg/mm²

The hardness values are of greater importance than the values for tensile strength.

In the tempered tubes with the nominal width (I.D.) of 6, 10, 16 and 24 mm the following limits are permissible:
Tensile strength 80 - 100 kg/mm².
Hardness 240 - 300.

The values for tensile strength have in these cases greater importance than the values for hardness.

Yield Point:

Air tempered: at least 50 kg/mm²
Oil " : at least 55 kg/mm²

(with reference to the water pressure test the yield point may not be below 45 kg/mm².)

Elongation, L = 5d, lengthwise

Round Rod, Air Tempered: Over 14, but preferably over 16, with tensile strength of 70 - 85 kg/mm².

Oil Tempered: Over 14

Tube --

Strips --

Notched bar strength "maximum" when using normalized samples (DVMR)

the rate of cooling in the temperature range of 600 - 600° C must be 25°/min. or 150° C in 4 minutes. For comparison it is advisable to record in actual operation the total time of the hardening temperature to 600° C.

The material must be stamped with characteristic markings to show whether it was air quenched or oil quenched.

In the tempered condition, the material must possess no visible grain boundaries or free ferrite, and have a uniform, sufficiently fine grained, well developed temper structure. The surface, in particular the inside surface, must be as nearly as possible free from indications of decarburization or from other unfavorable effects upon the structure of the outside zone by the action of heat or the heat treatment. In certain cases one may have to come to a specific understanding (When a drilling of the tubes is anticipated this must preferably be done upon the conclusion of the heat treatment).

B. Local Treatment of Welds and the Treatment of Welded Systems of Tubes.

1) Treatment of Tempered Tubes.

If, in certain cases, welding is done on tempered tubes in accordance with directions given under #9 (supplement 5, sheets 3 and 4 will apply.)

The treatment is done as in B; (2) insofar as the arrangement permits.

2) Working of Heated Tubes.

Whenever this tempering of the welded tubes is foreseen, as is mostly the case with # 10, it must be done as directed in A. After performing a circular welding job, the usual annealing temperatures may be slightly exceeded during the annealing which follows immediately after welding, however not above the transformation point, so as to produce a high tensile strength and a freedom from strains, and make the tube assembly insensitive to transformation stresses in the neighborhood of the welded joints.

Tempered but not yet annealed tubes must be particularly carefully handled during the necessary transport.

With a sufficiently high tensile strength, a Brinnell hardness of 300 is permissible. With a Brinnell hardness of 250 - 300, the notched bar strength must be at least 3 kg/cm² when the cross sections are very great in the cross sectional directions of the D V M samples; with smaller cross sections, it must be correspondingly greater.

Referred to the time of reaching the lower limit of the hardening temperature given.

Sternberg

T.O.A. Reel No. 1
Bag No. 2168
Item No. 9

SUBJECT: High pressure Experiments
February 11, 1942 O Leuna Works

HEAT BALANCE IN THE SUMP PHASE PREHEATER - LUTZKENDORF

Enclosed please find the computations of the Lutzkendorf preheater as promised at an earlier date.

In addition to the operating conditions recorded at that time in column 1 of the table, we have made computations for three other cases, namely:

	<u>CHANGES</u>	<u>INCREASE IN THRUPT BY:</u>
Case 1.	Increasing the inlet temperature in the regenerator from 50° C to 60° C.	1.0 te/h
Case 2.	Raising the inlet temperature of the heating gases from 565 to 573°	0.9 te/h
Case 3.	Additional increase by using new hairpins at 585° C	1.1 te/h

Computations were made on the following assumptions:

- a. The recirculated amount (Introduced in front of the preheater) was in all cases: 9.5 te/h
- b. The FOD residue was in all cases: 3 te/h
- c. The bottom temperature was: 450° C
- d. The inlet gas amounted to: 14,000 m³/h
- e. The heat effect was assumed to be sufficiently large to cover the radiation losses and no cold gas was used.
- f. The K value of the regenerator amounted to: 130 K cal/m²
In the gas blower the K value was assumed to be 135; the calculated larger thrupt should lead us to expect still higher K values. °C.h.

g. The radiation losses amounted to:

In the regeneration	200,000 K cal/h
For the hot circuit	400,000 K cal/h
For the preheater	400,000 K cal/h

h. Amount of heating gas was: 53,000 m³/h

We may mention that a further increase in throughput of about 0.6 t/h could be achieved by adding the HOLD after sending the gases first through the preheater, instead of before, because temperatures lie closer together at this point. This effect is greater when the regeneration becomes poorer.

T.O.M. Reel No. 1
 Bag No. 2168

Present Operating Conditions

Throughput
 Including 3 to/h of
 cold HOLD (Abschlamm)

Inlet Gas

Regeneration
 Temperature range

K Values

Heat Efficiency

Exhaust
 Temperature range

Heat Efficiency
 Scrubbing (?) " 100%

Case 1

Case 2

Case 3

to/h 10.3 11.3 12.2 13.3

olm/h 14,000

°C 137 ← 450 → 50 → 294 163 ← 450 → 80 → 306 169 ← 450 → 80 → 303 176 ← 450 → 80 → 300

Cal/°C.h.m² 130 135 135 135
 10³ Cal/h 2900 2850 2960 3090

°C 408 ← 565 → 329 → 458 412 ← 565 → 336 → 458 412 ← 573 → 334 → 458 414 ← 585 → 329 → 458

10³ Cal/h 2420 2370 2490 2690

Sternberg/pt.1

T. O. M. Reel No. 1
 Bag No. 2168
 Item No. 8

SUBJECT: Temperature Equilisation in the Lutskendorf Preheater.
 Report of visit on January 30, 1943.

Suggestions for improvement to attain more nearly uniform temperature distribution in the preheater inlet and outlet as well as of a loss of pressure in heating gas for raising the efficiency of the heating gas blower.

The evaluation of the operating conditions from the standpoint of the heating gas were made in the hydrogenation works of Politz in 1939 with the preheater constructed on the Ludwigshafen plan, and in Lutskendorf on the preheater of the Laura model.

Both preheaters have two Schiele heating gas blowers of the type 1160. The following operating conditions have been found:

		Politz	Lutskendorf
Number of hairpin tubes:		20-1/2	24
Volume produced by the blowers (15°, 735 mm):	m ³ /h	About 85,000	About 55,000
Inlet Temperature:	°C	593 - 598	555 - 515
Outlet Temperature:	°C	435 - 425	455 - 410
Total Resistance:	mm/water	260	325
Composed of: -			
Pressure piping	"	60	80
Suction "	"	110	145
Gases "	"	90	100
Maximum velocity of heating gas in the gases:	m/sec	About 13.5	About 15
Loss in pressure per hairpin tube with heating gas velocity of 20 m per sec	mm/water	6.2	10.7
Effective heat conduction with 300,000 and 200,000 K cal. per hour	k cal/h	1,450,000	1,820,000

We have determined the resistance of hairpin tubes in a number of operating measurements and experiments and found them to be 6.5 mm water per tube with a velocity of 20 m/sec. and the specific gravity of the heating gas of 0.42 kilograms/m³. (This value includes the resistance of the changes in direction of gas).

The value measured by you, 10.7 mm water per hairpin tube may in part be explained by the non-uniform distribution of heating gas over the cross sections of the gases.

We are enclosing as an Appendix our sketch # L-15200-2 of February 17, 1943, for the heating gas movement in the preheater at Lutsendorf which has permitted achieving a uniform distribution of the heating gas on the pressure side; to what extent equal temperatures become established will depend on a uniform distribution of the temperature and the pressure of the heating gases from the inlet on. Should the latter be non-uniform the heating gases must be regulated. (Corresponding experiments have been performed by yourself.)

Constructional changes were at present desired in the preheater proper, and we have not changed the gas outlet supports in the preheater. They have an adverse effect upon the temperature distribution at the outlet from the preheater. A uniform temperature at the outlet could be produced by changing the piping distribution over the height, as is done in the inlet. This would also result in additional eliminations of pressure losses.

To permit but a short shut-down of the chamber, the heating gas blower located the furthest from the chamber was installed during operations. During the shut-down, most of the inlets could be closed and the chamber then operated with only one blower. The efficiency with one blower amounted to about 70% of the efficiency with two blowers.

According to our computations the following resistances were found with an amount of heating gas of 80,000 cubic meters per hour (15° 735 mm) using the installation represented in the appended sketch.

- | | |
|-----------|--------------------|
| 1. Piping | About 140 mm water |
| 2. Gases | About 310 mm water |
| | About 450 mm water |

The pressure with your newly ordered blower amounts to 440 mm of water with 100,000 cubic meters per hour, and more than 450 mm water will surely be produced with a lower yield.

We might recommend, in order to produce a somewhat higher pressure in the new blowers, that the yield be reduced to about 90,000 c.m. per hour and use the already ordered motors at full capacity.

The distribution of the amounts and of the temperature permits a complete utilisation of the temperature drop of the gas and of the material of the hairpins.

(NO SKETCH)

Stenberg/pH

(8) - T-17

T.O.M. Reel No. 9
 Bag No. 2733
 Pages 193 & 194.

Bottrop - August 1944.

Vapor Phase Product Balance - Stalls 17 & 54a
 for June 1944

Operations, hours : 720
 1) Injection (12.7 P101/31.3 P102/13.2 P103/0.5 P 18/41.8 P302) : 19.86 te/h; Load te/m^3 : 0.717
 1) New Input (21.9 P101/54.5 P102/22.7 P301/0.9 P18) : 11.57 te/h; Load te/m^3 : 0.418
 Catch Pot : 17.70 te/h
 Gasoline (VT 330) of analysis B. 83 : 9.27 te/h; Load te/m^3 : 0.335
 Production Factor (Gasoline + gasification + salts : Gasoline) : 1.232

Per 1/te gasoline, consumed: 0.0707 te H_2 = 879 m^3 fresh gas (assured value)

Per 1/te injection, consumed: (0.033) H_2 .

From 1033.0 kg injection + H_2 produced:

	kg	% referred to injection + H_2
Products		
Catch Pot (gas free)	891.00	86.400
Containing gasoline B. 83	165.20	45.220
Middle Oil B. 83	124.30	41.180
Reaction Water (pure)	9.16	0.387
Salts in injection water & reaction water	4.96	0.481
Consisting of NH_3	3.77	0.365
H_2O	0.99	0.096
CO_2	0.10	0.010
Phenols	0.10	0.010
Gases		
3) Free H_2 in gases after pressure reduction	4.53	0.443
Containing free H_2 in Press. Red. II	3.88	0.376
Free H_2 in Press. Red. 12 & 13	0.70	0.067
2) Hydrocarbons (C = 2.499)	125.43	12.156
Consists of C_1	14.59	1.412
C_2	25.60	2.480
C_3	34.15	3.308
C_4 (normal)	26.25	2.544
C_4 (iso)	10.20	0.990
C_5	14.69	1.422
CO_2	0.01	0.001
H_2O	0.83	0.080
NH_3	1.35	0.131
Total	1037.38	100.579

Continued:

T.O.M. Reel No. 9
Bag No. 2733
Pages 193 & 194.

- 1) Boiling point 101 to 195°
- 2) The large amount of hydrocarbons, and correspondingly high total result of 101 percent is the result of the washing of the liquid phase still.
- 3) Computations based on analyses of May 1944, because the analytical results of June 1944 were destroyed by aerial attack.

Sternberg/pkl

Vapor Phase Product Balance - Stalls 17 & 54
for May 1944

- Operations, hours : 744
- 1) Injection (10.9 P101/23.1 P102/16.0 P54) : 16.61 te/h; Load te/h/m² : 0.618
- 1) New Input (21.8 P101/46.2 P102/35.0 P54) : 3.31 te/h; Load te/h/m² : 0.409
- Catch Pot : 14.70 te/h
- Gasoline (VI 330) of analysis B. 83 : 6.82 te/h; Load te/h/m² : 0.336
- Production Factor (Gasoline + gasification + salts : Gasoline) : 1.280

Per 1/te gasoline, consumed: 0.0727 m³ H₂ = 902 m³ fresh gas (assumed value)

Per 1/te injection, consumed: 0.0095 te H₂

From 1029.9 kg injection + H₂ produced:

	kg		% referred to injection + H ₂
<u>P R O D U C T S :</u>			
Catch Pot (gas free)	834.80		85.910
Containing gasoline B. 83		410.00	39.840
Middle Oil B. 83		474.80	46.070
Reaction Water (pure)	23.28		2.262
Salts in injection water & reaction water	5.62		0.547
Consisting of NH ₃		3.86	0.375
H ₂ S		1.37	0.134
CO ₂		0.27	0.026
Phenols		0.12	0.012
<u>G A S E S :</u>			
Free H ₂ in gases after pressure reduction	5.48		0.532
Containing Free H ₂ in Press. Red. 11		4.72	0.458
Free H ₂ in Press. Red. 12		0.76	0.074
2) Hydrocarbons (C = 2.481)	143.65		13.947
Consists of C		17.22	1.674
		29.25	2.842
		29.00	2.785
(normal)		30.10	2.920
(iso)		11.49	1.116
		15.58	1.610
H ₂ O	0.04		0.003
CO	1.00		0.098
NH ₃	1.61		0.156
	1055.43		105.454

1) B.P. 101 - 1950

2) The large amount of hydrocarbons in the resulting high total of 103 percent was caused by the washing of the liquid phase.

Sternberg, etc.

T.O.M. Reel No. 1
Item No. 7

2-17-1943

PROPERTIES OF NIO MATERIAL FOR HAIRPIN
TUBES OF A 700 ATM. GAS PREHEATER

The relationships between the temperature of the heating gases, tube wall temperatures and resistance to creep strength, as well as the connection between the temperature of the tube walls and the incrustation of the tubes under operating conditions have been at the center of considerations in the design of the 700 atm. gas preheater.

One could in general strive to limit the temperature of the heating gases in the 325 atm. preheater to that temperature which corresponded to the resistance to creep strength of the tube walls at complete incrustation; however, conditions existing in the 700 atm. preheater, in particular the totally absent or only partially feasible regeneration of the paste, as well as the large thru-put, forced the selection of higher wall temperatures than corresponded to the creep resistance of the incrustated walls, or, in other words, to utilize as far as possible the creep strength values of the incompletely incrustated preheater. An earlier discussion resulted in laying out a preheater on the basis of a maximum crust of 10 mm. thickness which still provided a certain safety margin with the values of the creep strength then available. The maximum wall temperature was computed from such a thickness of the crust to be 550°, the presence of the latter being assumed for a limited time.

Intermediate observations of the investigation of materials tested showed, however, that evaluation of material was not dependent on the strength of creep test strength alone. The material showed a time relationship of the creep strength under the existing mechanical and chemical influences at the high temperature of the material.

We may observe here that the closer connections are not as yet fully cleared. It remains in particular yet to investigate the effect of the

crust or the absence of a protective oil coating caused by the existence of a crust. The corresponding laboratory experiments are being carried out. We have also inaugurated experiments to find the factors which are responsible for the formation of the incrustation with the purpose of holding the incrustation back; at any rate the purpose of our newest investigation is to pay closer attention to the thickness of the crust.

Preliminary investigations of the material have shown that the temperature at which NiO can be used for a longer time without difficulties must be limited to 520° of the inside wall temperature, and only subsequent experiments will have to clear whether this temperature may be displaced upward with corresponding improvement of material and corresponding change in the analysis of the charge. We may, however, say even now that this temperature limit is strongly affected by the mechanical strains on the tubular walls, that is by the operating pressure. If the operations are carried out at 650 ats., the relationship becomes considerably more favorable. According to our newest experiments the harder NiO tubes have longer time creep resistance to hydrogen than the softer tubes. Formerly, soft NiO tubes had been recommended, which was in general entirely correct from mechanical considerations. Soft NiO tubes have a greater initial toughness while their creep resistance is still satisfactory. As mentioned above, the newest experiments showed that the creep resistance itself does not determine the life of the tubes but that the time creep resistance in hydrogen is important in that connection, and that this time creep resistance is no longer satisfactory with soft NiO material. Experience has further shown that the greater toughness of the soft NiO tubes was of no benefit. This means that the tubes could be harder. Moreover all the hairpin tubes which had been used in hot gases at wall temperatures exceeding 520° at least at some plants, and which had been used at least 10,000 hours in the hot gases could be

profitably rehardened. When such tubes have been operated for considerably longer time, structure strengthening may be used which can no longer permit regeneration by subsequent retempering.

Care is frequently taken not to exceed 520° on the inner surface of the wall temperatures and then there will be no need to retemper the new hairpins after 10,000 hours of operation. We expect to achieve similar conditions in the old hairpins after the first retreatment.

As has already been mentioned, lowering of the heating gas temperature without reducing the thru-put is in most cases impossible, since it would require an enlargement of the preheater.

When the blower output is increased, the colder hairpins can be more strongly loaded and they may outlast the hotter ones. However, one cannot readily obtain newer and larger blowers and motors.

Even the limited amount of information present permits us to conclude that the tube wall temperature of the cold gas preheater should be increased after one year to 520°. According to our computations this would correspond to the thickness of the crust of 5 mm. At the expiration of one year the preheater will have to be shut down and the crust removed. The operation time will naturally depend greatly on the product, in particular, from its tendency to form crust, and from the methods of operation.

In the future, particular attention must be paid to the supervision of the wall temperatures, and an agreement has been reached with the men in control of operations at Gelsenberg-Bensin as well as at Pölitz to introduce a hairpin in the usual arrangement of the elements in the chambers to be built at Pölitz and Gelsenberg. We will notify you of the result of measurements and simultaneously on the best arrangement to be recommended based on results obtained.

We may mention also that B9 and B3V have also a creep strength depending on time, although less so than B10.

To summarize: to increase the life of hairpins according to our present directions:

1. The hottest spots should be made of hairpins with the greatest permissible Brinell hardness.
2. All hairpins which have been operated for around 10,000 hours in hot gases at 700 ats. should be retempered.
3. If the wall temperature does not exceed 520° at the inside of the wall, the new hairpins as well as the retempered, and which therefore have a greater hardness, will no longer require any additional retempering.
4. It is most urgently required to remove the crusts at the latest after one year of operation.
5. The lower pressure, for instance 650 or even 550 ats., at which some plants are at present operating, retempering after 10,000 hours of operation of pins of normal hardness is not necessary to the same extent.

It is our intention to acquaint the hydrogen plants of any new experience on the question.

I. G. FARBENINDUSTRIE A.G.

Sternberg/m3

T.O.M. Reel No. 126
 Bag No. 2075
 Item No. 121-1

April 16 & 17, 1941

COMPARISON OF 300 AND 700 ATM.
 OPERATIONS IN LEUNA - 558

A comparison of experimental results with the Welheim catalyst using Scholven middle oil from coal for feed stock gives the following results:

1. The fraction up to 100°C is higher in octane number under Welheim tests than in Leuna. No satisfactory explanation for this has, as yet, been found. Dr. Iding brings out in the reported investigations that in the laboratory distillations the cuts were not made very sharply. However, samples sent from Welheim were distilled in a large size semi-commercial unit.

Dr. Donath says that the gas evolving in the catch pot in the Leuna tests and hence the gasification were lower than in the Welheim tests, which may be the cause of the lower proportion of the parts distilling below 100°C.

2. The iodine number is lower in the Welheim tests (3.5) than in Dr. Donath experiments (11.5), while tests made in a 200 mm. converter also gave 3.5.

These differences are not the results of analytical manipulations although more recent methods with more detailed requirements used at Leuna for the determination of the iodine number give higher results.

3. The large differences in the concentration of the aromatics is to be attributed largely to the method of determination. Welheim used only the Kattwinkel method, as described by Riesenfeld-Bente and entered in the operating directions of 1940.

Dr. Hirschberger says that the corrected aniline point method gives very reliable results which have been carefully tested for pure aromatic mixtures as well as with results of the SO₂ extraction.

Dr. H. tells us that only aniline points below -5 have been determined in mixtures of Kahlbaum gasoline. The differences from direct aniline point determinations were estimated graphically in making corrections.

Dr. Pier emphasizes that uniformity is being gradually attained in the method of determination of aromatics, and that with the existence of sufficient testing laboratories this uniformity will be gradually achieved. It is particularly important to prescribe definite concentrations of aromatics in the different laboratories. Dr. H. says that lower aromatic values are found in Darben by the Kattwinkel method than in Welheim. Dr. Iding is also of the opinion that the Kattwinkel method should not have been selected. Discussion has resulted in an agreement that the percentage of aromatics in Welheim is 5 to 12% higher by volume than in Leuna, and in the range tested

- 2 -

the difference is about 9%. This is in agreement with comparative investigations which have been run in Scholven in the tests of the different methods of determination.

4. The determination of phenol is made in Welheim in the form of triphenylmethane; in Leuna, by direct weighing of the extracted phenols. According to Dr. Iding the method used in Leuna gives too high results.

5. The gasoline made in Leuna with the Welheim contract has not been refined but washed with soda. It is not light-proof. Samples made at Welheim have been refined with diluted sulphuric acid, merely for safety's sake as told by Dr. Iding, in order to deliver gasoline which would meet specifications. Dr. Winkler says that if the VT706 be used and the product refined with H_2SO_4 , the product will be equally light-proof. The H_2SO_4 refining installation is available. The refining is cheap and is used even though probably not essential. Dr. Winkler estimates the refining losses to be around 0.5%.

The iodine numbers obtained in Leuna in the two liter converter would, however, necessitate refining losses of 1.5 to 2% when the refluxing of the redistillation residue (about 3%) is taken into consideration.

Dr. Pier says that in the liquefaction of coal the danger of obtaining high iodine numbers is greater than in the liquefaction of tar. A catalytic second refining may have to be taken into consideration in that case instead of a simple sulphuric acid refining.

The alkali scrubber at Welheim is operating at present with sodium hydroxide. The washing is done with the lye containing phenol and traces of phenol remain in the gasoline. No other inhibitors were used in addition to this "natural inhibitor."

Dr. Donath asks whether the redistillation losses, which are of importance in the calculation and designing, have been properly recorded.

6. There are additional differences in the gasification which have not, as yet, been explained. Dr. Winkler is emphasizing again that in the large-scale units at Welheim the gasification is steadily 2% more than in the five liter converter. He may repeat in that connection that there is in general good agreement in the results on gasification obtained in small-scale tests and in industrial units, but that as a rule the gasification is higher in industrial operations, instead of lower.

7. The hydrogen consumption values given in Welheim and at Leuna differ very greatly. Welheim claims 640 m^3 /te of gasoline with the gasoline free of the original product, and 530 m^3 /te of gasoline with the gasoline containing the original product, while the computations for Leuna give 910 m^3 /te of gasoline corresponding to 750 m^3 of chemically utilized H_2 .

Contrary to Dr. Winkler's conception that 100 kg of middle oil obtained from tar produces 82 kg of gasoline and about 31 kg of gasoline

is obtained from coal, Dr. Donath mentions the following figures:

1000 kg of gasoline free middle oil produces 790 kg crude gasoline with 90% gasification, or 782 kg. of refined gasoline.

The hydrogen consumption calculated from an average of Leuna and Welheim analyses is as follows:

1000 kg of middle oil = 86.5 C
With 10% H₂ requires 86.5 kg H₂

165 kg of carbon gasification with 23.0% H₂ requires 37.9 kg H₂

14 kg loss with 13.8% H₂) requires 96.5 kg H₂
782 kg gasoline with 13.8% H₂)

Total	134.4
Less	86.5
Net	47.9 kg H ₂

corresponds to 585 m³/te of middle oil or 750 m³/te of gasoline.

In these computations the factor for leakage used was 40 m³/te of middle oil, and for the dissolved hydrogen 110 m³ = total amount 900 m³/te of gasoline.

6. The overload curves for the two gasolines (with coal stall gasoline and without it in the two-liter converter in Leuna and in Oppau) were below C₃ specification which doesn't state, as emphasized by Dr. Pier, that it may not be changed. Dr. Winkler was informed by Dr. Frese over the telephone that two samples of fuel obtained from Welheim were of the C₃ specification fuel. This difference may be the result of the differences in the Oppau and RIN testing methods. One should expect from the lower concentration of aromatics in the Welheim gasoline samples that their overload should be lower than the Ludwigshafen samples.

Dr. Pier points out that great care must be used in judging the overload curve. There are at present two C₃ groupings at RIN, of which the lower contains reserve for the automobile firms. We have always considered the higher C₃ grouping as being important. According to a recent telephone communication, the lower C₃ grouping is also important in evaluating gasoline. A different picture is obtained dependent on which of the two different C₃ groupings is used in comparison, and from which standpoint the tests are made.

Dr. Pier shows in an investigation of the first aromatization of gasoline from Politz which he had in the form of a mixture from the 7019 and the DAP steps were both within the lower limit in comparison with the tests of the Oppau testing laboratories, while tests taken at three of the spots put it into the C₃ grouping. On our part, we will especially emphasize the following circumstances:

Samples sent from Welheim to the RIN consisted of middle oil without any coal stall gasoline or of middle oil with as little coal stall gasoline

as can be obtained in the coal phase from gasoline plus middle oil. The proportion of coal phase gasoline is essentially larger in the process for fuel oil production (40% up to 200°C) and according to experiments in Leuna this will produce further lowering of the aromatics and a considerable deterioration of the overload behavior. One has to deal with a still higher proportion of sump phase gasoline in Upper Silesia II.

Discussions have shown a difference in the conception or judgment of the overload curve. We have placed the principal value upon the height of the minima of the curve, while the gentlemen in Welheim judge the overload curve primarily by its thickness in the region of aliphatics. Dr. Winkler is of the opinion that he is in agreement with this idea of the aviation administration. On the other hand, we can point to the fact that the aromatic fuels are anyway little utilized in the aliphatic region, and therefore greater steepness may even be undesirable. (This topic was re-opened in a discussion about VT706b and CV2b during which Dr. Winkler re-emphasized the viewpoint that the CV2b is inferior to the VT706b, judging by all the overload curves and also according to the opinion of the RIN.) Our material has been presented to Dr. Deha and Dr. Hirschberger who did not concur in this statement.

9. In summarizing, we may say that our comparison tests in Leuna and Welheim are in the main in agreement as shown by the following figures:

	Welheim	Leuna 2 Liter Converter	Leuna 0.2 Liter Converter
Specific Gravity	0.777	0.780	
% to 100°C	45.5	35	28
End Point	167	167	
Aniline Point	+ 12	+ 8	
Iodine Number	3.5	11.5	3.6
% Aromatics by volume Kattwinkel method	47	(51)	
% Aromatics from Aniline Point		42	
Gasification	21	19	18

Dr. Winkler mentions the fact that the pressure has dropped at Welheim from 600 to 530 atm. and the hydrogen partial pressure from 68 - 71 to 63% H₂. The H₂ pressure in Welheim is accordingly in excess of 334 atm.

10. Dr. Pier states that the test results indicate the importance of careful consideration of the limiting values to be obtained during the operation with sump phase gasoline in comparison with tar. The table below gives a few pertinent values according to Dr. Winkler and Dr. Donath:

	Aromatics Rattwinkel	Aromatics An. Pt.	% below 100°C Helheim	% below 100°C Leuna	Gasifi- cation Helheim	Gasifi- cation Leuna	Aniline Point Helheim	Aniline Point Leuna
Tar	50-60	50	18-21					
Coal Lique- faction with no gasoline	45-50	42	45	35	(18)-21	19	12	8
Coal lique- faction with gasoline.	41-45	36	48	47	(18)-21	16	18	15

Dr. Pier added the following remarks on the question of preference of 300 or 700 atm. in the vapor phase, by stating that it was as wide as it was long and must be answered in reference to the production of (a) automobile gasoline with high anti-knock value and (b) high-test gasoline.

Historically the question has been developed as follows:

The 700 atm., sump phase, has been found in certain cases to be necessary for the working of asphalts. Tests performed at 700 atm. in the vapor phase had already indicated certain advantages at that time:

1. There is greater freedom from molybdenum and tungsten. This means the saving on rare materials. Dr. Simon's experiments with petroleum have shown that work can be done entirely without Mo. and W., using terrane alone.

2. Higher yields are produced.

3. The gasification losses were lower than with 300 atm.

A certain setback has been experienced when the 700 atm., sump phase has been introduced into the industry and the installation has been found to be more expensive than first supposed.

This retarded at first the introduction of the 700 atm. vapor phase into the industrial installations. In the meantime, suggestions to use intermediate pressures, for instance 500 atm., were also neglected in order to avoid having to deal with too many different pressures. Intakendorf built one installation for 700 atm. in order not to work with two different pressures.

The question of materials already played an important role in the decision. Leuna originally was inclined to use not too costly materials, even though they are limited in their hot strength, while the Americans decided in favor of high priced materials (D2A). One has to make a similar decision today between the N3 and N10 materials. As is always the case with new materials, reversal of decision is not excluded.

When analyzing the question one may harbor some such doubts, but when it comes to giving advice to others one is inclined to recommend or urge things without much consideration of the difficulties.

The gasoline production in the 300 atm. vapor phase is good. It produces automobile gasoline with a 90% yield and 83% yield of 87 anti-knock gasoline. The whole aromatization with 3,510 was only intended originally for the improvement of anti-knock gasoline. Later the question of toluol became real, and the question was renewed of producing aromatic gasoline with the minimum gasification, for which purpose new catalysts have been developed. The Air Force was at that time opposed to all aromatics, which may probably have been caused by the failure with benzol and which was connected with the poorer refining of otherwise poorer quality of the automobile benzol.

Ludwigshafen-Oppau at that time recommended aromatics for the air service (in this connection Dr. Pier reminded us of the good Borneo gasoline which contained about 30% of aromatics).

The A coal catalyst 7019 for 300 atm. was then developed at Leuna; it could be used in the available installations to produce toluol. It produces very good aromatic gasoline and high-hydrogen B middle oil, the boiling point curve of which is below that of the A middle oil.

Dr. Winkler reminds me that high-hydrogen B middle oil is not to be recommended. Dr. Urban had to withdraw a certain amount of B middle oil in order not to impair the overload curve. Dr. Pier claims this to be in no ways necessary. Peters mentions that according to remark recently made by Dr. Schmidt in Scholven the reflux proportion of the B middle oil must be kept constant at 1:1 to retain an absolutely constant quality of the CV₂b. Only a slight excess of B middle oil is removed and added to the 5058 step. Dr. Pier then added the following: It had been immediately recognized that the 7019 catalyst was not an ideal catalyst but had important advantages over 3510 or similar catalysts by causing less gasification and no condensation. The gasoline produced also had certain peculiarities. For instance, the iodine number offered certain difficulties which could, however, be readily overcome by refining catalyst in the cooling branch. Nevertheless, 7019 is an interesting catalyst especially inasmuch as it occupies a peculiar position in comparison with those previously known.

Dr. Winkler explains that the 7019 gasoline distills too little below 100°, so that the Air Force obtains no C₃, while the Welheim process produces C₃ directly. Dr. Donath remarks that catalysts are already available for 300 atm. which produce 50% of aromatics and distill 45% below 100°. The middle oil obtained in the liquefaction of coal at Scholven contained only 42 to 45% of aromatics. Such a production does not necessarily have the C₃ quality, but has the advantage to require only 10% of 1-octane instead of 20 in order to give C₃.

Dr. Pier mentioned in that connection that we as well have for a long time conducted tests along these lines and not only with the hopes of producing directly C_2 or C_2 qualities in one step, but also in combination with DHD, where the normal gasoline is slightly aromatized or dehydrogenated. At the same time, the production of light gasoline is intensely studied from higher alcohols or from normal gasoline production.

In general, for instance, the DHD gasoline from coal is in the lower C_2 group. The DHD gasoline from coal mixed with light gasoline containing 40% aromatics has the C_2 quality according to the determination of the DVL.

Dr. Pier told them the second principal method for the production of aromatic gasoline:

- a. Dehydrogenation without catalyst breakdown at 50 atm. has been tried repeatedly, but there invariably was a certain deterioration in the catalyst activity.
- b. The DHD process which has been developed from a similar American process for improvement of the anti-knock properties is known, however, to work with a deteriorating catalyst. It has been improved to make it possible to work with in too short regeneration periods.

The DHD process permits one to obtain good gasoline from the most different raw materials. One disadvantage is the formation of small amounts of condensation and displacement of the boiling point curve. There is, however, an immense advantage of the DHD gasolines compared to the aromatization gasolines in that, with the same proportion of aromatics, they consist of only aromatics in the upper part of the boiling curve. This means that the gasoline represents for definite aromatic concentrations practically the ideal fuel with regard to overloading, provided the residual gasoline is of good quality. However, there still remain a number of aromatic residual gasoline constituents in the upper part of the boiling point of the aromatization gasoline, and their anti-knock properties are poor.

The DHD process is now carried out industrially, after Politz pilot plant experiments, in Politz, Moosbierbaum (BF process), Leuna, Scholven and Upper Silesia I.

Dr. Pier next discussed the question of the best available means to use for the production of high-test gasolines:

1. 300 or 700 atm. aromatization (with addition of i-octane or i-octane substitutes).
2. Benzination at 300 or 700 atm. + DHD.
3. Mild aromatization + DHD.

4. Mild aromatization at 300 or 700 atm. for the direct production of C_3 with no addition of i-octane.

In that connection the production of light gasoline, alcohol gasoline and i-octane or alkyl-octane is to be considered.

Catalysts investigations have been performed at Ludwigshafen over all of these fields. We must emphasize here the importance of terrana or similar carriers which are generally applicable for aromatization of the 300 and the 700 atm. Initial failures at 300 atm. caused at first to disregard terrana and to use the coal catalyst.

In addition to the general wide considerations of the problem, Silasia II. is our primary interest because of the large amount of sump gasoline produced together with middle oil. (In the first construction step there are a 300 atm. vapor phase and a DHD installation.) Our computations have shown the following:

	300 ATM.	700 ATM.
Cost of installation without auxiliary units	Somewhat lower than 700 atm.	Somewhat higher than 300 atm.
Production cost	About the same as with 700 atm.	About the same as with 300 atm.
Thruput	0.35	0.50
Gasification/gasoline + loss	17	15
Quality	C_3	Barely C_3

This should make one inclined to say that the proper thing to do is to continue developing the 300 atm., in particular because this will introduce no new points for consideration.

Dr. Pier added that he nevertheless is personally inclined to recommend the 700 atm., leaving for the present the question open regarding subsequent operations. He bases his recommendation on the following:

The installation at present considered is not large, a 700 atm. in sump phase is available, the sump phase gasoline could be worked together in

the 300 atm. vapor phase (Silesia I.), where DHD is available. The principal advantage is the greater flexibility in the 700 atm. operations.

Naturally tests to solve the question of 300 or 700 atm. must yet be continued. The development work done so far has already resulted in the production of catalysts which produced more aromatics at 700 atm. There exists the added possibility, which is not yet ripe for industrial development, which consists in the aromatization with short-lived catalysts at, for instance, 300 atm. or lower.

However, the question of whether the Air Force will begin to use shortly the C_2 fuel is important for the choice of 300 or 700 atm.

A lively discussion resulted with respect to this question.

Winkler: The Air Force requires at present primarily such C_3 .
 Dahn: The present automobile program will soon permit the use of C_2 .
 Pier: C_2 is available and can be easily produced with DHD without the addition of 1-octane.

Winkler: And how shall one make C_3 from CV_2b_3

Donath: With light gasoline:
 $CV_2b + \text{light gasoline} = C_3$
 DHD with 40 aromatics = C_2
 DHD with 32 aromatics = C_3
 300 or 700 atm. aromatization with 40 aromatics = C_3 , but no means is at present available for the production of C_2 .

Winkler: Why not?

Donath: When aromatics are increased, 1-octane becomes necessary.

Pier: This is possible when tar or pitch are used because the quality in this case is high; this is, however, not the case with coal or with sump gasoline with middle Rhine or with middle German brown coal. The best residual gasoline is obtained by benzination at 300 or 700 atm. using 6434. A one-step process gives poorer residual gasoline than a two-step process. The quality of the residual gasoline increases in the following series:

- | | | |
|----|-------------------|----------------------|
| 1. | 500° 40 aromatics | 60 residual gasoline |
| 2. | 460° 20 aromatics | 80 " " |
| 3. | 420° 5 aromatics | 95 " " |

where in the case of 3) the best residual gasoline is obtained with DHD. Insofar as we can foresee mild aromatization plus DHD is at present the cheapest. This permits obtaining any concentration of aromatics required, for instance C_3 , C_2 or 50 volume per cent aromatics or more. Should one wish to remain with 40% of aromatics, the aromatization may be performed with 300 or 700 atm.

However, when designing a 700 atm. installation for aromatization (preheater) work may also be done at lower temperatures, and there are plenty of possibilities to use it in conjunction with DHD.

Winkler : There is no difference in the aromatization of the liquefied coal and of pitch, and there are in addition some possibilities of development of catalysts. Should the demand be made, a larger amount of aromatics can also be produced (underscored in pencil in the original).

Donath: Our experiments have already shown that 60% aromatics may be produced in liquefaction at 600 atm.

Pier: At 600 atm. the same amount of aromatics is produced as at 300 atm. when the 7019 is used.

Winkler: The super gasoline is of no significance in the 600 atm. aromatization (underscored in pencil in the original).

Donath: However, it lowers the aromatics.

Peters: According to our experiments the super gasoline contains 10% of aromatics when prepared by the fuel oil method.

Pier: The Air Force counts on the i-octane and alkyl-octane. Whatever butane is produced must be worked up. As much i-octane must be prepared as at all possible.

If production is directed toward the 87 gasoline a maximum of 10% i-octane or 20% alkyl-octane is obtained which may be used for blending of high-test gasoline or for other purposes.

The question is: What is primarily attained by installation? Quality is the first consideration, price the second.

Miss Ewing and Donath: Calculations give us the following yields:

METHODS	AROMATICS	GASOLINE	TOTAL GASOLINE INCLUDING ALKYL OCTANE
Normal densination (middle oil and heavy oil process)		83-84	100-102
Mild aromatization / DHD	40	78	85
Densination / DHD	40	77-81	92-95
	50	75-80	92-95
Aromatization at 700 atm.	33	83	87-88

Pier: I have a feeling that no mistake will be made if the installation is to be made for 700 atm.

Donath: 700 atm. would cost somewhat more.

Pier: Having 300 and 700 atm., aromatization may be readily done at 700 atm. to say 20 aromatics, and followed then with DHD.

Donath: The installations are inter-connected. One must take into consideration the possibilities that one installation may be shut down.

Pier: We are dealing at present with ordering the material.

Winkler: It is not impossible to produce 40% sump gasoline with the 700 atm. aromatization.

Oettinger: The installation must offer development possibilities.

Winkler: Should the whole sump and vapor phase be built for 700 atm., we shall have a saving in circulation washing and reserve containers.

Donath: With a common circulation, constituents will be present in the gas which will affect the vapor phase. Dr. Pier points to the possibilities to produce fuel oil at 700 atm. over a fixed bed catalyst.

Winkler: Such experiments have been made at Helheim; 60 to 65% gasoline plus middle oil has been obtained with a thru-put of 1.0.

In the 600 atm. aromatization tests - Leuna used brown coal liquefaction middle oil with sump gasoline, with a thru-put of 1.1, losses of 15.18%, 50% gasoline with 50% aromatics was made in the five liter converter.

Sternberg/mc/pki

T.O.M. Reel No. 1
 Bag No. 2168
 Item No. 11

3-21
 Indwighshafen, March 7, 1939

GAS EQUILIBRIA IN THE COAL STALL
AT 700 ATM. AT SCHOLVEN III. *

The equilibria calculated and investigated were:

- (1) Relationship between the H_2 partial pressure at the converter inlet to the amount of gas at the converter inlet.
- (2) Relationship between H_2 partial pressure at the converter inlet to the amount of gas sent to the Linde installation.
- (3) The effect of increasing CO content in the fresh gas:
 - a. With constant oil scrubbing.
 - b. With 26,500 m³ of gas sent to the Linde installation.
- (4) The amount of wash oil in relationship to the CO content at about 590 atm. H_2 partial pressure.

BASIC FIGURES AND ASSUMPTIONS:

The amounts have been taken off the drawing of the IG Lemna and N6557a-2. This presupposes a pure coal thru-put of 55.5 te/hr. with the following amount of hydrocarbons produced at 250,000 year to gasoline production.

CH_4	C_2H_6	C_3H_8	C_4H_{10}
21,300	24,900	34,800	16,400 te/yr.
73.2	45.6	43.4	15.5 m ³ /te of pure coal
36.0	44.08	64.0	30.6 kg C/te of pure coal

The assumption is made that in 100% gasification of carbon, 0.5% CO_2 and 2.2% CO are formed.

CH_4	36.0 kg C/te of pure coal
C_2H_6	44.8 " " " " "
C_3H_8	64.0 " " " " "
C_4H_{10}	30.6 " " " " "
	175.4 = 97.3%

100% C gasification = 180.275 kg C/te of pure coal

CO_2 : 0.905 kg C/te of pure coal, equal about 1.80 m³ CO_2 /te of pure coal

CO: 3.97 kg C/te of pure coal, equal about 8.07 m³ CO/te of pure coal

* Drawing, sheet #2, not found in the original.

With the C content of the coal equal to 83.5% and an assumed utilization of 96%, the amount of C utilized:

$$1000 \times 0.835 \times 0.96 = 802 \text{ kg C/te of pure coal}$$

The gasification accordingly amounted to 22.5%.

Of the 1.05% sulphur in the dry coal, about 80% is converted into H_2S , producing about $6.1 \text{ m}^3 H_2S/te$ of pure coal.

With 10% of oxygen in the pure coal there is $1000 \times 0.1 \times 0.96 = 96 \text{ kg}$ of O_2 present, which will form $108 \text{ kg } H_2O$. The dry coal contains 2% of water, so that 22 kg of water/te of pure coal are introduced. The total amount of water at the converter outlet is $130 \text{ kg } H_2O/te$ of pure coal. The water injected into the regenerators has not been taken into consideration.

SOLUBILITIES AT 700 ATM.

Gas	HOLD about 1050 Dynamic	Hot Catch Pot about 1000 Dynamic	Static	Wash Oil Dynamic	Static
H_2	0.0866	0.037	0.05	0.078	0.1
$CO + N_2$	0.0762	0.04	0.08	0.083	0.16
$CO + H_2S$	0.524	0.80	2.70	1.3	4.70
CH_4	0.152	0.13	0.43	0.31 - 0.25	0.50
C_2H_6	0.286	0.35	2.23	0.80	3.2
C_3H_8	0.305	0.63	4.56	1.20	12.9
C_4H_{10}	0.267	2.0	35.0	3.50	50.0

Explanation of values:

m^3 of gas (15°C , 735 mm. of Hg)/te of oil or product (at 20°C) and 1 atm. of gas partial pressure for the HOLD and the hot catch pot product, m^3 of gas (15°C , 735 mm. Hg.)/ m^3 of wash oil (20°C) and 1 atm. partial pressure of gas.

See (1), page 1, and Table 2.

4,032 4,400 4,750 * m^3/te of pure coal, converter intake
589.89 583.0 590 * atm. of H_2 partial pressure

The amount of wash oil was assumed to be $3 \text{ m}^3/te$ of pure coal ($166.5 \text{ m}^3/hr.$), since with $2.5 \text{ m}^3/te$ of pure coal ($133 \text{ m}^3/hr.$) the partial pressure of H_2 at the converter intake would be considerably lower because of the lower solubilities of CO and N_2 in the wash oil. Raising the amount of gas at the converter inlet results in no appreciable change of the partial pressure of H_2 .

See (2), page 1, and Table 4.

The partial pressure of H₂ going to the Linde installation is on page 1 as 38,000 m³/hr., and was found from a rough balance sheet.

In comparison to the process with oil washing, with approximately the same H₂ partial pressure at the converter inlet, the following change in the analysis will take place:

COMPOSITION AND PARTIAL PRESSURES
AT CONVERTER INLET

Gas	Oil Scrubbing 166.5 m ³ /hr. 0.15% CO in the Fresh Gas		Linde Fraction ** 26,500 m ³ /hr. 0.15% CO in the Fresh Gas	
	Atm.	%	Atm.	%
H ₂	589.89	84.27	585.396	83.628
CO	18.20	2.6	7.280	1.040
N ₂	44.45	6.35	20.440	2.92
CH ₄	40.60	5.8	51.100	7.3
C ₂ H ₆	5.285	0.755	19.670	2.81
C ₃ H ₈	1.414	0.202	13.300	1.90
C ₄ H ₁₀	-	-	0.966	0.138
CO ₂	0.035	0.005	0.462	0.066
H ₂ S	0.126	0.018	1.386	0.198
	700.000	100.000	700.000	100.000

** The analyses and the pressures are given on the assumption that the Linde installation returns into the circuit only pure H₂ and no other gases.

(3) a. (See sheet 2 and Tables 1 and 3
Amount of wash oil m³/ts of pure coal (166.5 ts/hr.)

b. (See Tables 4 and 5)
Assumption is made here that the Linde installation delivers only pure H₂.

(4) (See Sheet 3)

The picture is set up under the assumption that the partial pressure at the converter inlet remains as shown in Table 1. The converter outlet is assumed to contain a constant partial pressure of CO equal to 24 atm. (the actual pressure in the scrubber).

8.07 m³ CO/te of pure coal are produced in the converter and are removed with the CO introduced in the fresh gas.

<u>Fresh Gas</u>	<u>% CO</u>	<u>m³ CO</u>
1162.0	0.15	1.74
1163.7	0.30	3.5
1166.1	0.50	5.83
1168.4	0.70	8.15
1178.8	1.00	11.60

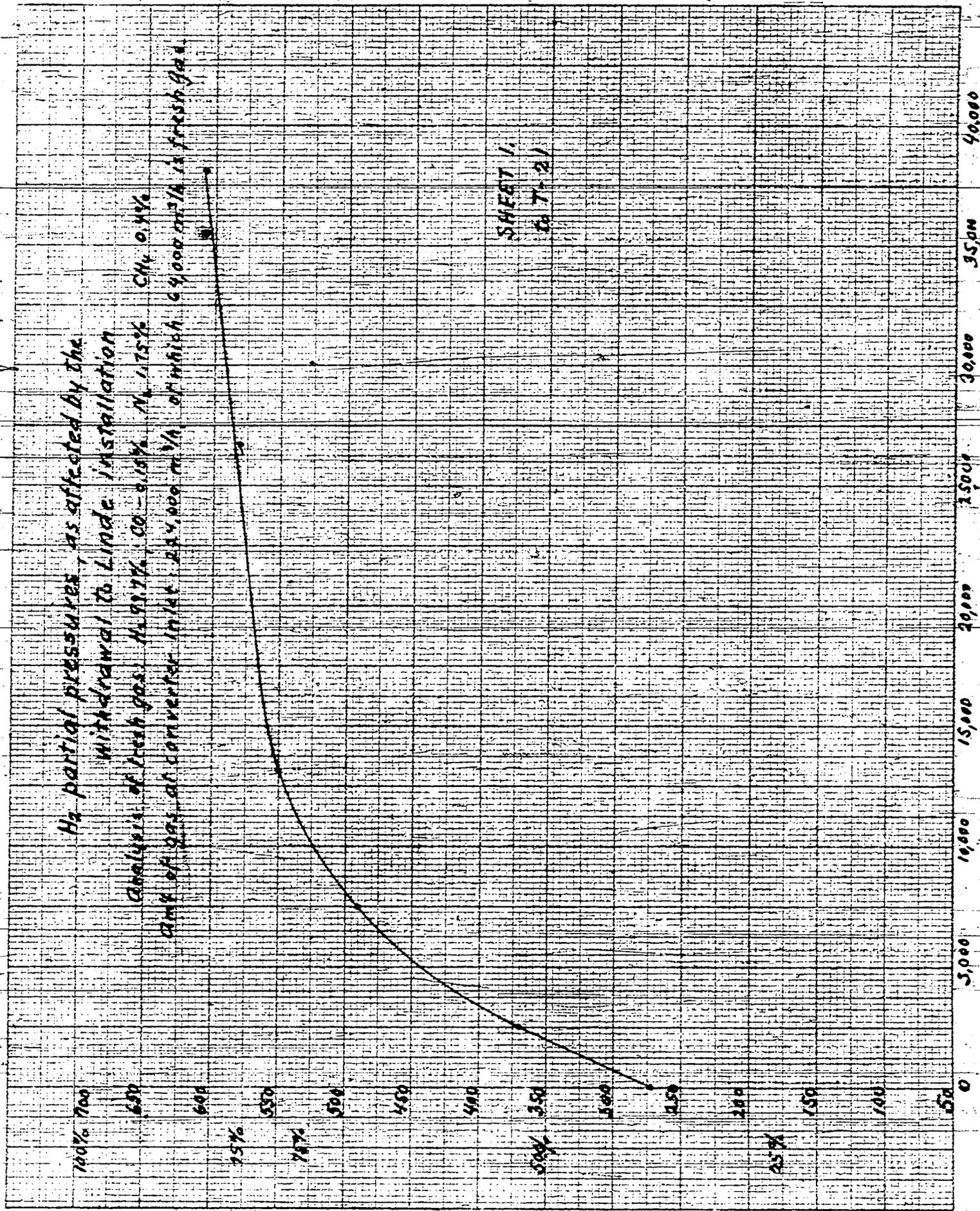
The total amount of gas is constant at the converter inlet.

Remarks: The decimals do not represent a measure of accuracy.

Sternberg/mc/pkl

KEUFFEL & ESSER CO. N. Y. NO. 38871-K
 2 1/2 X 3 1/2 to the 4 inch 2 1/2 inch accepted
 MADE IN U.S.A.

H₂ partial pressures, as affected by the withdrawal to Linde installation
 Analysis of fresh gas: H₂ 97.7%, CO - 0.16%, N₂ 1.75%, CH₄ 0.34%
 Amt of gas at converter inlet: 25,000 m³/hr of which 6,000 m³/hr is fresh gas.



SHEET 1
 (67-21)

Ludwigshafen/Main, March 3, 1939.

Table 3

GAS EQUILIBRIA, LIQUID PHASE, 700 ATM.

0.35% CO in the Fresh Gas

No	Fresh Gas		Converter		Inlet		New		Converter Inlet		Dissolved in		Less		Converter Outlet 3 m ³		Dissolved		Removed		CO ₂ Gas from Converter
	m ³	%	m ³	%	m ³	%	m ³	%	m ³	%	m ³	%	m ³	%	m ³	%	m ³	%	m ³	%	
12	1125.23	84.27	3997.74	389.89	2457.74	522.830	24.5	28.5	522.830	24.5	28.5	1.15	1.15	12.13	232.12	522.830	329.85	1855.83	2282.51		
13	8.74	2.6	104.89	16.2	122.9	24.10	0.644	1.032	24.10	0.644	1.032	1.15	1.15	0.524	109.09	24.0	6.6	2.81	150.09		
22	20.39	6.35	256.0	44.45	256.0	54.9	2.19	3.26	54.9	2.19	3.26			1.28	249.27	54.9	19.65	20.39	235.62		
334	4.53	1.3	244.0	40.6	207.2	65.6	5.24	12.7	65.6	5.24	12.7			1.53	247.73	63.9	30.10	77.83	212.55		
1215	0.735	0.22	70.6	5.245/45.6	76.00	18.25	2.43	6.44	18.25	2.43	6.44			0.33	64.71	14.27	34.35	45.6	30.69		
334	0.272	0.08	2.24	1.434/49.0	53.94	11.19	1.760	10.42	11.19	1.760	10.42			0.28	32.43	8.70	21.12	45.6	24.14		
2470	0.675	0.20	0.795	1.84	15.99	3.820	0.464	9.650	3.820	0.464	9.650			0.078	5.108	1.125	5.033	15.5	0.0		
118	0.018	0.01	0.018	0.01	0.018	0.01	0.01	0.01	0.018	0.01	0.01			0.01	1.124	0.304	1.194	1.64	0.19		
118	0.018	0.01	0.018	0.01	0.018	0.01	0.01	0.01	0.018	0.01	0.01			0.01	1.124	0.304	1.194	1.64	0.19		
TOTAL	120	1222.04	150	4082.0	3276.11	730.080	97.845	76.465	730.080	97.845	76.465	1.35	1.35	16.914	924.243	669.000	275.449	1324.81	2970.69		

Values % 1 to. of pure total

% Calculated to 25° C, 757 mm pressure

53.5 g/m pure coal through at Section III.

Table #2

GAS EQUILIBRIA, LIQUID PHASE

4400 m³/to Pure Coal at Converter Inlet, 0.15% CO in Fresh Gas

Gas	Fresh Gas		Converter		Inlet Part.Pr.	Newly Added	Conv. Inlet & New Addition Part.Pr.	523 kg. H ₂ O	Dissolved in 1485 kg. Catch Pot	130 kg. H ₂ O
	%	m ³	%	m ³						
H ₂	97.7	1135.23	83.300	3662.16	583.00	-948.20	2723.96	23.6	28.6	
CO	0.15	1.74	2.640	116.80	18.80	+ 8.07	124.87	0.950	1.42	1.15
H ₂	1.75	20.38	6.540	288.00	45.50		288.00	2.190	3.28	
CH ₄	0.40	4.65	6.330	279.80	44.20	+ 73.20	353.00	5.380	13.05	
C ₂ H ₆			0.850	38.40	6.10	+ 45.60	84.00	2.460	3.50	
C ₃ H ₈			0.305	13.20	2.00	+ 43.80	57.00	1.750	0.25	
C ₄ H ₁₀						+ 15.50	13.50	0.425	8.85	
CO ₂			0.007	0.34	0.05	+ 1.84	2.18	0.112	0.48	
H ₂ S			0.028	1.30	0.25	+ 6.10	7.40	0.390	1.680	
TOTAL	100	1162.00	100.000	4400.00	700.00(?)	-754.09	3645.91(?)			1.15
					699.90		3653.91			

Values to 1 te. of pure coal

% Calculated to 15° C, 735 mm. pressure

55.5 te/h pure coal thru-put at Scholven III.

Less 0.5%	Conv. Outlet Part.Pr.	Dissolved in 3 m ³ Wash Oil	Removed	Fresh Gas to Converter
m ³	at.	m ³	m ³	m ³
13.63	2648.13	520.37	187.03	2526.93
0.63	120.72	23.70	9.81	115.06
1.44	281.09	55.20	20.38	267.62
1.77	392.80	65.20	77.85	275.15
0.42	72.76	14.22	45.60	38.40
0.29	44.71	8.76	43.80	13.20
0.08	6.175	1.21	15.50	
0.01	1.571	0.31	1.84	0.34
0.04	5.290	1.03	6.10	1.30
	3513.246	690.00	291.091	3238.00

Sternberg/mo/rkp

Table #3

GAS EQUILIBRIA, LIQUID PHASE

0.3% CO in Fresh Gas

Gas	Fresh Gas		Converter		Inlet Part.Pr.	Newly Added	Conv. Inlet & New Addition	Part.Pr.	Dissolved in 523 kg. HOLD	Dissolved in 130 kg. H ₂ O	Loss 0.5%
	%	m ³	%	m ³							
H ₂	97.57	1135.23	63.340	3360.070	583.340	-934.000	2406.070	514.590	24.193	28.11	12.030
CO	0.3	3.50	3.530	142.500	24.750	+ 8.070	150.570	32.220	0.887	1.908	0.752
N ₂	1.74	20.38	6.350	256.000	44.450		256.000	54.800	2.190	3.260	1.280
CH ₄	0.39	4.65	5.800	234.000	40.600	-173.200	307.200	65.800	5.240	12.700	1.530
C ₂ H ₆			0.755	30.400	5.285	-165.600	76.000	16.250	2.430	8.440	0.380
C ₃ H ₈			0.202	8.140	1.414	-143.800	51.940	11.130	1.780	10.420	0.260
C ₄ H ₁₀						-15.500	15.500	3.320	0.464	9.850	0.078
CO ₂			0.0057	0.190	0.035	+ 1.840	2.030	0.425	0.119	0.517	0.010
H ₂ S			0.018	0.700	0.126	+ 6.100	6.800	1.455	0.398	1.247	0.034
TOTAL	100	1163.76	100	4032.000	700	-759.89	3272.11	700	37.698	76.452	16.354

Values to 1 te. of pure coal

% Calculated to 15° C, 735 mm. pressure

55.5 te/h pure coal thru-put at Scholven III.

Conv. Outlet Part. Pr.	Disassembled in 3 m ³ Wash Oil	Removed	Fresh Gas to Converter
m ³	m ³	m ³	m ³
2341.740	514.031	1135.230	2224.840
147.023	32.240	11.570	139.000
249.270	54.900	20.380	235.620
287.730	63.300	77.820	229.250
64.750	14.270	45.600	30.400
39.480	8.700	43.800	8.440
5.108	1.125	25.500	
1.284	0.304	1.840	0.190
5.121	1.130	6.100	0.700
3141.606	690.00	1357.870	22868.240
		1367.870	

Stenberg/mc/ckp

Ludwigshafen/Rhein, March 9, 1939

Table # 4

GAS EQUILIBRIA, LIQUID PHASE, 700 AT. WITH 26,500 m³/h to
 Lande Installation
 with No Oil Scrubber, 0.15% CO in Fresh Gas

Gas	Fresh Gas		Converter Inlet		Newly Added	Conv. Inlet & New Addition		523 kg. HOLD	Dissolved in	
	%	m ³	%	m ³		Part. Pr.	at.		Part. Pr.	1485 kg. Catch Pot
H ₂	97.70	1135.23	83.628	3370.16	-1000	2370.16	515.998	23.40	28.40	1.15
CO	0.15	1.74	1.04	42	+ 8.07	50.07	11.0	0.440	0.655	-
N ₂	1.75	20.38	2.92	118.88	-	118.88	26.40	1.05	1.52	-
CH ₄	0.40	4.65	7.30	294.65	+ 73.20	367.85	80.00	6.36	15.45	-
C ₂ H ₆			2.81	113.4	+ 45.60	159.00	34.60	5.18	17.98	-
C ₃ H ₈			1.90	76.5	+ 43.80	120.30	26.10	4.07	24.4	-
C ₄ H ₁₀			0.138	5.75	+ 15.50	21.25	4.620	0.645	13.710	-
CO ₂			0.066	2.66	+ 1.84	4.50	0.976	0.267	1.158	-
H ₂ S			0.198	8.00	+ 6.10	14.10	0.306	0.842	3.640	-
TOTAL	100	1162.00	100	4032.00	-805.89	3226.11	700	42.254	106.913	1.15

Values to 1 te. of pure coal

% Calculated to 15°C, 735 mm. pressure

55.5 te/h pure coal thruput at Scholven III

Loss 0.5%	Conv. Outlet	Circulating Gas to Converter
m ³	m ³	m ³
	%	%
11.81	2306.55	75.398
0.25	47.575	1.56
0.63	115.68	3.81
1.84	344.20	11.25
0.80	135.04	4.40
0.6	91.23	2.97
0.106	6.789	0.221
0.02	3.055	0.10
0.07	9.548	0.31
16.126	3099.667	100
	2580.00	100

amt of circulating gas $\times 0.24611 = 1162 \times 0.023 = 402 \times 0.16372$
 $= 2580 \text{ m}^3/\text{te}$ of pure coal $= 143,000 \text{ m}^3/\text{hr}$. of circulating gas to the circulating pumps.
 $59.667 - 2580 = 479.667 \text{ m}^3/\text{te}$ of pure coal $= 26,600 \text{ m}^3/\text{hr}$ to the lands installation.

Sternberg/mo/ptl

Table #9

GAS EQUILIBRIA, LIQUID PHASE, 700 at., 26,500 m³/h to
 Like Installation

No Oil Scrubber, 15 CO in Fresh Gas

	Inlet Gas		Converter Inlet		Part. Pr. Added		New Addition		Part. Pr. Catch		Total	
	%	m ³	%	m ³	at.	m ³	at.	m ³	at.	m ³	at.	m ³
H ₂	96.66	1135.25	82.16	3310.86	575.12	-1000	2310.86	508.298	22.84	28.64	23.80	1.15
CO	1.0	11.72	2.508	101.30	17.556	+ 8.07	109.37	23.70	0.947	1.415	1.415	1.15
N ₂	1.744	20.38	2.92	118.88	20.440		118.88	26.40	1.05	1.52	1.52	1.15
CO ₂	0.596	7.45	7.30	296.65	51.200	+ 74.20	367.85	90.00	6.36	13.95	13.95	1.15
C ₂ H ₆			2.81	113.40	19.670	+ 45.60	159.00	34.60	5.18	17.98	17.98	1.15
C ₃ H ₈			1.90	76.50	13.300	+ 43.80	140.30	26.10	4.07	24.40	24.40	1.15
C ₄ H ₁₀			0.138	5.75	0.966	+ 15.50	21.25	4.62	0.645	13.71	13.71	1.15
CO ₂			0.066	2.66	0.462	+ 1.84	4.50	0.976	0.267	1.158	1.158	1.15
H ₂ S			0.198	8.00	1.386	+ 6.10	14.10	0.306	0.842	3.64	3.64	1.15
TOTAL	100	1172.00	100.00	4032.00	700.00	-805.89	3226.11	700.000	42.201	107.913	107.913	1.15

Values to 1 te. of pure coal

% Calculated to 15° C., 735 mm. pressure

55.5 te/h pure coal thru-put at Scholven III.

Unit	Urea to be Circulated	Urea to be Circulated to Converter	%	%
11.52	2247.86	1895.60	73.79	73.479
0.546	105.342	84.515	10.66	3.46
0.63	115.63	98.50	3.81	3.81
1.81	361.20	290.00	11.25	11.25
0.80	135.04	113.40	4.40	4.40
0.60	91.23	76.50	2.91	2.91
0.106	6.788	5.75	0.22	0.221
0.02	3.053	2.64	0.10	0.100
0.07	9.548	8.00	0.31	0.31
16.132	3058.714(?)	2580.00	100	100
	3055.711		100.31	

Amount of circulating gas X x 0.26521
 = 1172 x 0.023 = 4032 x 0.1784
 X = 2580 m³/te of pure coal in the
 circulating gas = 143,000 m³/hr.
 3058.724 - 2580 = 478.74 m³/te of pure
 coal = 26,500 m³/hr. to the Linde installation.

Sternberg/mc/ckp

HYDROGEN CONSUMPTION
DURING THE HYDROGENATION OF GASOLINE-CONTAINING SCHOLVEN MIDDLE OIL
WITH THE RUHRÖL CATALYST K 413.

Temperature: 26.5 MP (501° C, 934° F)
Pressure: 540 atm.
Load: 1.45 kg/liter/hour.

The "Comprehensive Report on the Hydrogenation of Coal Liquefaction Oil
Product at Pressures over 300 atm, Using the Ruhröl Catalyst K 413" gives the
following data:

109.0 kg. A middle oil, with 87.5% C and 10.2% H	95.4 kg. C	11.12 kg. H
85.6 kg. gasoline	87.9% C	12.1% H
23.4 kg. loss = 21.5% of product loss.	75.25 " C	10.35 " H

Recalculated to C:

95.4 kg. C in A middle oil
75.25 kg. C in gasoline
+ 20.15 kg. gasified C with a carbon number of 2.94 (21.1%)
24.65 kg. hydrocarbons
+
20.15 kg. C + 4.50 kg. H

Recalculated to H:

11.12 kg. H in A middle oil + x kg. H consumption = 10.35 kg. H in gasoline
+ 4.50 kg. H in hydrocarbons.
11.12 kg. H in A middle oil + 3.75 kg. H consumed = 10.35 kg. H + 4.50 kg. H
For 95.40 kg. C in A middle oil → 3.75 kg. H consumed
for 1000 kg. C in A middle oil → 39.1 kg. H consumed
789 kg. C in gasoline
897 kg. gasoline → 39.1 kg. H
1000 kg. gasoline → 43.47 kg. H = 527 m³ H₂ (15° C, 735 mm Hg)

1 to gasoline from gasoline containing Scholven middle oil obtained with the
consumption of 527 m³ H₂.

CONSUMPTION OF HYDROGEN
DURING THE HYDROGENATION OF THE WELHEIM MIDDLE OIL
WITH THE RUFEROL CATALYST K 413.

Bottrup-Boy, 4/12/1941

Temperature: 26.5 mV (501° C, 934° F)
Pressure: 630 atm.
Load: 1.06 te/m³/h.

The data used are taken from a report of 12/24/1940.

9955 kg. C in A middle oil with 89.7% C and 8.9% H

8477 kg. C in gasoline with 88.25% C and 11.75% H
+ 1478 kg. gasified C with a carbon number of 2.54 (14.9%)
1820 kg. hydrocarbons
1478 kg. C + 342 kg. H

Recalculated to H:

938 kg. H in A middle oil + 2 kg. H consumed = 1128 kg. H in gasoline
+ 342 kg. hydrogen in hydrocarbons.

938 kg. H in A middle oil + 482 kg. H consumed = 1470 kg. H

For 9955 kg. C in A middle oil → 482 kg. H consumed
1000 " " " A middle oil → 48.34 kg. H consumed.

946 kg. gasoline → 48.43 kg. H
1000 " " → 50.20 kg. H = 610 m³ H₂ (15°, 755 mm Hg).

1 te gasoline from Welheim coal tar gasoline obtained with the consumption
of 610 m³ H₂.

Sternberg/1cp

ABSTRACT OF A TEA REPORT ON THE DEVELOPMENT
OF HYDROGENATION IN LEUNA, 1941

A few points in the history of development of hydrogenation in Leuna are discussed (very briefly). It consisted in increasing the fuel production to a maximum, bringing the costs down to a minimum, improving the quality of the product and in overcoming the deficiency in critical materials.

The production of the liquid products has increased from 1927 (beginning of operations) to 1940 from 0 to 400,000 t/annum. The adequate original planning is proven by the fact that the structural facilities were adequate for such an expansion and permitted increasing the size of the units. This is illustrated by the development of the reaction space of the stalls, with the converter changes from a single converter with a stirrer and a capacity of 2.74 m³ to the four converter stalls of 27 m³ reaction volume each.

The original boiler type stills with a thruput of 2.5 m³/hr and heat requirements of 250,000 h.u./t were replaced with tubular stills of 130 m³ thruput and 100,000 h.u./t heat requirements, with a corresponding great reduction in the costs of heat, service and repairs.

The original ball mills used for the grinding and pasting of coal had a capacity of 8 ton of dry coal/h. Replacing these with Krupp's Concentra mills doubled the output for the same fineness and for the same volume of the mills, and by doubling their volume the capacity has been increased fourfold.

The capacity has been further increased by increasing the specific thruputs of the units. One of the factors was the increased operating time, at the cost of the repair time, and the table of the development of the life of the units, reaction volumes and capacity is enlightening:

	1927	1933	1940
Average life, days	11	54	155
Average time of repairs	12	21	20
Reaction space per stall, m ³	2.7	16	27
Injection of coal paste, m ³ /h	3	8	30
Middle oil yield per stall, t/annum	1100	11,500	55,000

This was made possible by a careful study of dismantling results and small scale experimentation. It has thus been found,

that paste and H_2 must be kept thoroughly mixed to avoid coking, which at first was done by mechanical stirrers, until found that introducing coal paste with the gas resulted in a turbulence which brought about a perfect mixing. Dismantling showed the necessity of avoiding any kind of obstructions in the equipment. Other observations led to a very rapid increase in the vapor volume of the hot catchpots to avoid entrainment of the hot oil which would coke. The vertical pipe lines had either no crust, or else very little of it. A slight cooling of the wall completely overcame the crust formation.

The cavier formation was one of the interesting but unpleasant experiences of hydrogenation of brown coal. Iron catalysts which replaced the acidified Mo catalysts, introduced the formation of small spherical bodies up to pea size, especially in the first converter. Up to 12 tons of them were collected from a single stall. The spheres consisted of concentric layers of $CaCO_3$ mostly around a quartz nucleus. The $CaCO_3$ crystallized as beta arragonite. It was overcome by continuously withdrawing small amounts of the paste from the first converter.

The life of the vapor phase stalls was increased by the selection of catalysts varying little in activity, and resistant to abrasion over a long period of time.

The heat of the reaction had to be mastered. Increases in temperature must be stopped as quickly as possible to prevent any overheating of the high pressure lines, which results in explosions and major fires. Rising temperatures are lowered by the introduction of cold gas, which can be done at different levels of the catalyst bed. The distribution of the cold gas over the whole cross section was found to be of the utmost important, and this has led to the introduction of blends. The catalyst temperature is kept from rising by careful proportioning of the feed when changes are made to feeds with different heats of reaction. The temperature distribution in the vapor phase converter is made easier by an upward flow of the feed. However, with light catalysts or greater velocities of flow, this advantage must be disregarded because of the increased abrasion and the entrainment of the catalyst dust, which increases resistance. Exhaustive experiments have shown that catalyst pills are set in motion at $1/5$ to $1/6$ of the velocities required to suspend the particles in space.

Because of the high operating temperatures and pressures, the pipelines must possess a high resistance to H_2 and great strength. Fires can be avoided only when using proper construction materials. Their composition is supervised by testing samples of each alloy casting. Over 36,000 such samples have been analyzed for this purpose since 1936, and of these 14,000 were for other works. A card index is kept for alloyed parts, with 30,000 cards

for the Leuna works. Finally, all alloy castings and tubes are tested micrographically before being installed. Some 800 pieces must be thus examined after the repairs on a stall, and a great amount of experience for such testing is presupposed. All tubes and bends which may be subject to corrosion or abrasion must have their wall thickness tested either mechanically or by means of X-rays.

When making repairs, some 50 tests must be made on equipment to make sure of tightness, internal resistance and perfect operation of the parts. Upon the erection of a stall, drawings are made, with each pipe, casting and valve in the cold as well as the hot lines listed and numbered. As a result of these elaborate tests the number of fires have been greatly reduced.

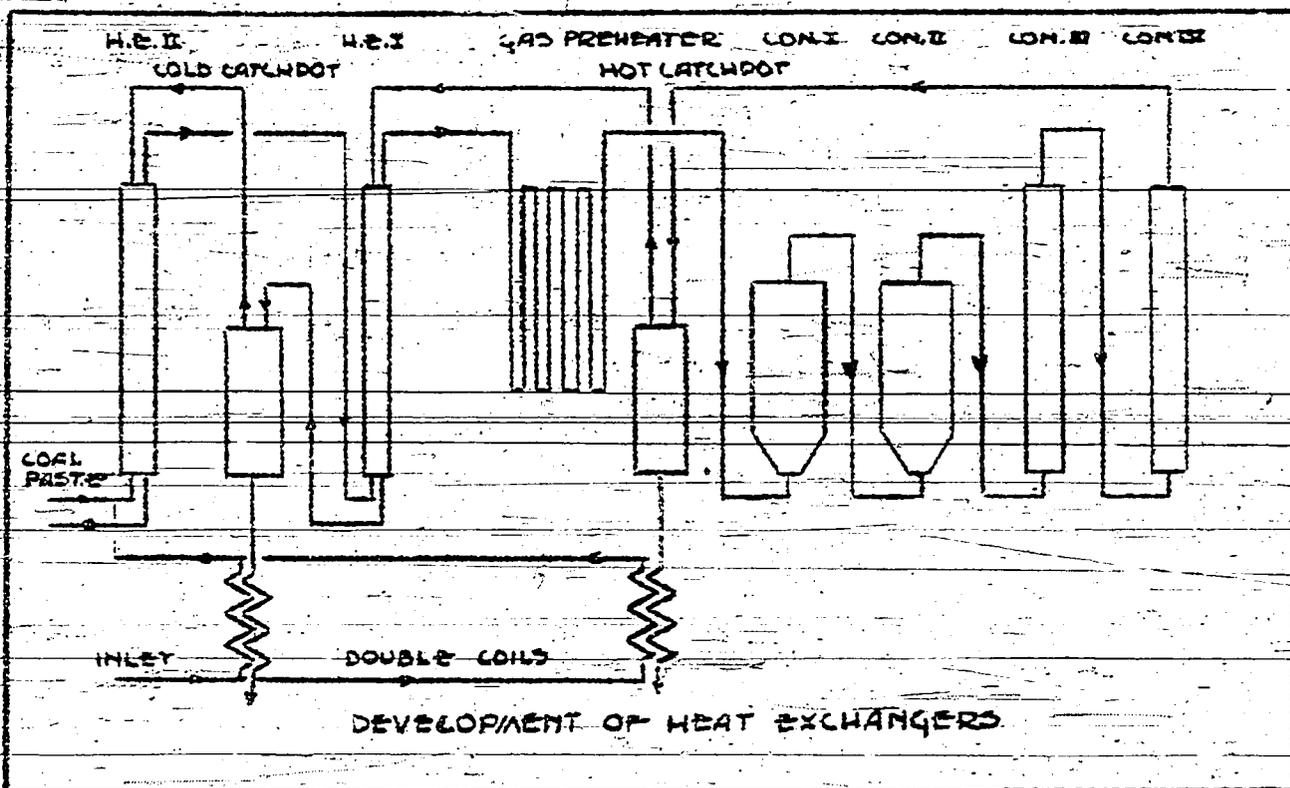
The reduction in accidents was in part due to improvements in operations and the training of experienced operators. The work of the latter is aided by refinement in instrumentation and the automatization of operations.

Thruputs are further raised by proper operation of the units. Mastery of the heat of the reaction prevents the passing of the unreacted feed and increases the thruput. The breaking down of the asphalts is rapidly reduced by increasing the throughput. The remedy was hard to find, because different stalls, operating under apparently identical conditions, varied much in their asphalt utilization. However, this has been accomplished, and within the last three years the thruput has been increased by about 80%, from 18 to 32 m³ coal paste per hour, without placing any additional burden upon the working up of the residues.

While operating at all costs was the prime consideration during the first years of operation, growing experience, and in particular the use of heat resistant steels, has permitted a more economical use of power, and the use of the cheaper forms of power.

In the original stalls, the peak heating was obtained by the use of electrical preheaters, and only a small amount of heat regeneration was done in the converter itself in order to avoid hot pipe lines. Only gas was preheated in the tube-bundle heat exchangers. The coal paste was preheated to about 300° in a molten bath and with hot water. The temperature was limited by the possibility of coking. The very complicated and costly heating with hot water was then necessary to avoid over-heating and the frequent plugging up of the equipment.

Today coal paste and gas are preheated together in heat exchangers. The peak heat is reached in the preheater by fuel gas. This preheater was developed here and served as a model for other preheaters and in the distillation. Finally, the HOLD heat was transferred to the intake gas.



The heat requirements were reduced to one half the 1927 requirements by the reduction of the proportion of gas per m² of paste, from 620,000 h.u. to 315,000 h.u. In 1927, 250,000 units of the amount had to be supplied from outside sources, while in 1941 only 18,000 h.un. had to be supplied from the outside, and 94% were obtained from heat exchangers.

The improved yield was a further factor in making the process more economical. Increasing the utilization of coal from 65% without the use of catalysts to 97% with Mo-catalysts was a deciding factor. The amount of oil lost with HOLD and in the residue working-up is lessened with better coal utilization. The losses of oil in the liquid phase may be compared in the following table:

	1927	1940
Utilization of pure coal, %	65	97
High pressure oil production, kg/t pure coal	351	524
Solids in HOLD " " "	394	227
Oil in HOLD " " "	800	461
Loss of oil in working up residues, in % of HOLD oil	23	17
in % of high pressure oil	52	15
Actual oil production, kg/t pure coal	165	445

This success of the Ludwigshafen development work has permitted the resumption of coal liquefaction in Leuna.

Gasification was reduced in the vapor phase, and as far as possible the gases produced were utilized. The reduction was primarily due to the discovery of the catalysts 5058 and 6434.

The more perfect gasoline removal of the off-gases, and the separation of butane, propane and ethane were other developments. Gasoline and hydrocarbon-containing gases, obtained when the pressure on the liquid products was released, were at first simply burned in waste gas burners, later added to the fuel gas. Two compressors were available for the compression and cooling of the volatile fractions, which were added to the motor gasoline. The removal of gasoline from the distillation off-gases was for years done by washing them with middle oil, with a thruptut of 2000 m³/h. The composition of fuel obtained in hydrogenation was:

Motor gasoline, with no gasol	80%
Gasol	6%
Butane	6%
Propane	8%

Gasol, as well as butane and propane have been recovered for some time not only from the distillation off-gases, but also from the hydrocarbon-rich off-gases of the vapor and the liquid phases. Connected with this was the separation of ethane for the conversion into ethylene, and later into lubes. The gases had first to be freed from hydrogen sulfide by alkazid washing, and from the organic sulfur by a special catalytic treatment.

The different properties of the fuels necessitated the introduction of separating units for obtaining gasolines of required vapor pressures. This stabilization permits having a maximum amount of butane in the finished gasoline.

The manufacture of isooctane necessitated the separation of the two isomeric butanes. The following are the different stabilization units needed to-day:

	Operating pressure	Capacity m ³ /hour
4 stabilization units	6	136 (liquid, total)
2 alkazid units	1	12,600 each
2 units for the separation of organ.S	1	8,000 "
2 gasol units	15	12,000 "
1 Linde unit for recovery of hydro-		
	carb. 15	5,500 "
1 Gerlach unit " " " " "	15-20	10,000 "
1 unit for the separation of butane	7	8 (liquid)

Except for the Linde unit, all the rest have been designed and installed by ourselves.

In addition to the separation of the hydrocarbons, provisions had also to be made for their disposal. New ways of using them had to be found: propane for household uses, for cooking, heating, illuminating the homes and industrially, and a propane/butane mixture as a fuel gas. As an illustration of the utilization of byproducts, we may mention that during 1940, 26,000 t. elementary sulfur and 16,000 t. SO_2 were obtained from the hydrogenation gas, including the hydrogen production, and the waste water.

A detailed cost accounting was made necessary by the high cost of the gasoline produced. The causes of losses had to be established, corrections rapidly and surely developed, and the costs correctly distributed between the raw material and the finished products costs. In doing this, operational supervision had to solve a number of problems of measurement.

In addition, specific standards (yields, hydrogen consumption, etc) had to be found for the various raw materials and finished products. This was frequently impossible to get by direct measurement, because many different feeds were simultaneously treated in the same equipment, and the many closed circuits made decisions difficult. Statistical methods, and conclusions from large number of values solves the problem. The data obtained could also be used for the design of new units.

The future development will primarily consist in refining the details. The operating time of the high pressure units will have to be further increased. It already appears probable, that the operating life of a liquid phase stall can be increased to one year. The operating pressure will have to be increased to 300 atm as soon as iron and labor again become available, because most of the equipment is already adapted to it.

A 700 atm unit must be introduced to contribute to the general development through our own experience.

8,000 t. of phenols are already produced from the middle oil, and the amount could be increased to 13,000 t/annum. About 1,800 t additional phenols is obtained from the off-water.

Work is being done at present on the recovery of acetone and methyl ethyl ketone from the liquid phase.

The present working up of the residues by a low temperature coking is no entirely satisfactory solution, because of the large losses and the low thrupt per unit, especially for our high-ash coals.

So far attempts to find a better solution have been unsuccessful; but experiments in that direction will be resumed as soon as conditions improve.

A dehydrogenation unit for the prehydrogenation gasoline for the production of aviation gasoline high in aromatics is now being erected. This unit will also be used for the production of higher antiknock gasoline, when the demands for aviation gasoline have been reduced.

This might pave the way for the separation of middle oil into paraffin-rich and high aromatics portions. The latter would give in the vapor phase a gasoline equal in quality to the bituminous coal gasoline; the former could be used as a diesel oil, provided higher antiknock brings a corresponding difference in price, and the price of diesel oil comes closer to that of gasoline.

When the contemplated change of the compressor drives to electric drives is made, the higher-hydrocarbon portion of the hydrogenation off gases will no longer be needed for the antiknock gas machines, which would create a substitute for hydrogen-rich off-gases as a fuel gas, while the hydrogenation gas could be produced by a complete breaking down of ethane (2400 m^3), methane (6000 m^3) and H_2 ($11,500 \text{ m}^3$), or a splitting unit would produce all the H_2 required, some $20,000 \text{ m}^3$.

One ton of gasoline from brown coal requires $2 \frac{1}{2}$ times as much H_2 as from low temperature coking tar, and the capacity of the plant can be increased about 150,000 t/annum by substituting tar for brown coal, with very little additional investment in the hydrogenation proper. It will however require putting in low temperature coking furnaces, preferably by ourselves, because sad experience has shown, that when obtained from the outside, it may easily lead to a gasoline production by the tar producer, instead of ourselves.

W.M. Sternberg
12/17/46

Reel 9

Report on the Results of the Vapor Phase Study - October - 1942

Material Balance of the Vapor Phase Still
for October 1942

Report
February 4, 1943

Total weight of gas feed : 744
 Total weight of A-Product : 10.96 to/h; loss to/h, m³ : 0.369
 Total weight of B-Product : 4.65 to/h; loss to/h, m³ : 0.275
 Total weight of C-Product : 10.04 to/h
 Total weight of D-Product : 4.15 to/h; Yield to/h, m³ : 0.245
 Total weight of gasoline used : 0.024 to E₂ : 900 m³ fresh gas (assumed value)
 Total weight of Injection used : 0.0275 to E₂
 Total weight of Injection + E₂ obtained :

	kg	% referred to Injection + E ₂
Gasoline (gas feed)	916.00	89.140
Gasoline B. 83	378.40	36.730
Oil B. 83	537.60	52.410
Water (used)	13.85	1.347
Injection - React. gas	4.89	0.473
Hydrocarbons		
Benzene	3.05	0.295
Toluene	1.23	0.120
Xylene	0.39	0.038
Paraffins	0.21	0.020
Inert gas	3.18	0.310
Inert gas in beddown 12	2.99	0.285
Inert gas in beddown 12 & 16	0.25	0.025
Inert gas (0.12 & 80)	18.90	1.840
Inert gas	0.00	0.000
Inert gas	0.77	0.075
Inert gas	2.40	0.233
Total	1092.93	106.34

The high amount of hydrocarbons resulting in a total of 106.3% has been caused by the washings of the liquid phase circuit.

Stearberg/pk

Product Balance of the Vapor Phase Stall
5/4-a in January 1942

Hours of Operation: : 26 1/2
 Injection (52.9 A-/47.1 B-Product) : 7.79 te/h; Load te/h/m³ : 0.477
 New Input (A-Product) : 4.11 te/h; Load te/h/m³ : 0.252
 Catch Pot : 6.98 te/h
 Gasoline (VT 706) per analysis B. 83 : 2.88 te/h; Production te/h/m³ : 0.177
 Production Factor (Gasoline + gasification + salts, % Gasoline) : ≈ 1.27 (Including shutting down and starting the stall)

Per 1/te gasoline used: 0.0750 te H₂ ≈ 900 m³ fresh gas (Assumed amount)

For 1 ton of Injection: 0.0277 H₂ has been used.

From 1027.7 kg. injection plus hydrogen obtained:

	kg	% referred to injection + H ₂	
<u>P R O D U C T :</u>			
Catch Pot (gas free)	896.00		87.170
Including gasoline B. 83		369.20	35.940
Middle Oil B. 83		526.80	51.230
Reaction Water (pure)	30.68		2.983
Salts in Injection & Reaction Water	7.44		0.725
Including: NH ₃		4.92	0.479
E ₂ S		1.76	0.172
CO ₂		0.41	0.040
Phenols		0.35	0.034
<u>G A S E S :</u>			
Free H ₂ in letdown gases	2.85		0.278
Including free H ₂ in letdown 11		2.41	0.235
and free H ₂ in letdowns 12 & 16		0.44	0.043
Hydrocarbons (C = 2.62)	127.40		12.390
CO ₂	0.00		0.000
H ₂ S	1.41		0.137
NH ₃	3.06		0.298
	1068.84		103.981

1) The high amount of hydrocarbons resulting in a total of 104% has been caused by the washings of the liquid phase circuit.

T.O.M. Reel No. 9
Bag No. 2733
Page No. 225

Bottrop - January 26, 1942

N O T I C E

H₂ balance - Stall 54 a.

The present H₂ balance of stall 54-a refers to November 24, 1941, while the stall was operating in its own gas circuit. Computations are based on analysis made by the Main Laboratory. The amount of free H₂ in the waste circuit gas, and the amount of the hydrocarbons (C₁ - C₄) with the H₂ were calculated from the gas analysis of the stall; C₅ is entered as an oil product.

Sternberg/pkl

H₂ - Balance of Stall 5622 - November 24, 1941

Hours of Operations: 24 Injection: 12.36 te/h (A to B product) 48.5 52) Catch Pot: 11.62 te/h
 Catalyst: K 429 Density of inlet gas: 0.191 (= 88.86% H₂) Gasoline: 4.79 te/h
 Converter: 268 mV (= 48200) 3) Stall Pressure: 595 atm.

	m ³	to	% H ₂ by volume	Pure Hydrogen m ³	te	% of Pure Hydrogen	Remarks
Fresh gas from unit 7	155,000		97.20	150,600	12,620	100.00	C ₁ = 13.40%
Oil products 1)				33,350	2,750	22.15	C ₂ = 19.02%
Reaction water		5.50		7,410	0.611	4.92	C ₃ = 29.48%
OH ₂	4,430	2.91		8,830	0.728	5.86	n-C ₄ = 26.58%
C ₂ H ₆	4,150	5.17		12,540	1.034	8.32	1-C ₄ = 11.52%
C ₃ H ₈	4,750	8.81		19,410	1.601	12.90	100.00%
n-C ₄ H ₁₀	3,420	8.38		17,520	1.445	11.63	Hydrocarbon = 43.75%
1-C ₄ H ₁₀	1,480	3.63		7,590	0.626	5.04	1-C ₄ in total = 30.2%
NH ₃ } in water		0.51		1,090	0.090	0.72	
N ₂ S } in water		0.26		181	0.015	0.12	
Free N ₂ in letdowns 11, 12 and 16.							
Waste Circulating Gas			86.80	14,340	1.182	9.52	
Undeclared losses (0.5% from the catch pot computed as gasoline.)	26,000			22,550	1.860	14.96	
				1,965	0.162	1.31	
				146,776	12,104	97.45	

H₂ Consumption

Including waste gases = 1350 m³ of fresh gas per te gasoline. Not including waste gas = 1150 m³.
 Fresh gas: = 9.20 m³ of fresh gas per te of gasoline plus gasification.

- 1) Computations given in the appendix. 2) Analysis in the Appendix. 3) Temperature in centigrade assuming constanten couple (Sternberg).

T. O. M. Reel No. 9
Bag No. 2733
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H₂ - Balance of Oil Products

Inlet:

Injection = 296.4 to with 8.92 % H₂

Outlet:

Gasoline = 114.9 to with 11.76% H₂

Middle Oil = 164.0 to with 9.55% H₂

= 26.43 to H₂

= 13.52 to H₂

= 15.66 to H₂

29.18 to H₂

26.43 to H₂

H₂ - Addition

2.75 to H₂

Stamberg/pkl

Gasification of C during the Experiment

Starch: Gasoline = 4.79 to/h with 88.24% C
 Hydrocarbons = 760 m³ C₁ - C₄ with 2.56 average carbon C number = 4.230 to C
 = 0.955 to C

Gasification of C: 18.4% referred to S in gasoline + C
 in C₁ - C₄

Hydrocarbons	m ³ /h	kg C/h	% of gasified C by weight
CH ₄	184.6	90.8	9.50
C ₂ H ₆	173.0	170.2	17.90
C ₃ H ₈	198.1	292.0	30.55
C ₄ H ₁₀	162.6	210.3	29.35
C ₁ - C ₄ H ₂ O	61.7	121.3	12.70
	760.0	954.6	100.00

Investigation of Injection Products and of the Catchpot

Injector Production:

Density, 15°C.	= 0.950	Phenol	= 2.8% by weight
Fluctuation Point	= - 12.5°C.	Pyridine	= 2.7% by weight
Solution Point	= + 0.0	C	= 89.86 - 89.90%
Start of breathing point	= 65°C.	H	= 8.89 - 8.94%

Engler Distillation

Vol. %	°C.	Vol. %	°C.	°C.	Vol. %
	169	65	236		
15	195	75	248		
25	204	85	270	170	6.5
35	210	95	297	250	75.5
45	216	98.5	314		
55	224	Residue	1.0%		

Catchpot:

Density, 15°C.	= 0.877	Analine point of the fraction up to 170°C.
Beginning Breathing Point	= 40°C. Engler	= with gasoline gas - 4.0°C.
Column Distillation	= 25°C. after column	Without gasoline gas - 5.9°C.
	43.0% by volume	
	below 162°C.	

Engler Distillation

Vol. %	°C.	Vol. %	°C.	°C.	Vol. %
5	69	75	236		
15	102	85	232	100	15.8
25	128	95	277	170	42.8
35	156	97.5	300	200	62.8
45	179	Residue	0.6%	300	98.8
55	192	Low Cooling	1.3%		
65	207				

Elementary Analysis:

Fraction below 162°
in column

Fraction above 162°
in column

C	88.09 - 87.99	90.36 - 90.36
H	11.97 - 12.03	9.51 - 9.58

T.O.M. Reel No. 126
Bag No. 2075
Item No. 122

(Leuna) April 15, 1941

THE PROPORTIONS OF n-BUTANE AND I-BUTANE
AT 300 AND 700 ATM.

The two tables below contain the different possible proportions of normal, i- and total butane produced in the vapor phase under different operating pressures. The values given are but rough approximations, but give nevertheless information on the amount of butane production under different operating conditions.

1. In the method for the production of gasoline containing up to 10% aromatics (87 anti-knock I gasoline) (6434), with or without pre-hydrogenation, the proportion of i-butane to gasoline is over 6%. The proportion of i-butane at 300 atm. (7846/6434) with over 12% gasoline production with pre-hydrogenation (300 atm.) and benzination (700 atm) is but 7% because of the lower gasification at 300 atm.

If dehydrogenation follows, the proportions are changed but slightly. There is in general a slight shifting of the i-butane toward the normal butane proportion.

2. During aromatization, the proportion of i-butane referred to gasoline is about 4%, except when 7019 is used, when it amounts to about 2%. With milder aromatization followed by dehydrogenation the proportion of the C₄ hydrocarbons is hardly at all affected by the DHD.

3. In the method of production for gasoline and fuel oil the proportions are similar to those for the manufacture of gasoline and middle oil. The i-butane proportion is on the average somewhat lower and the normal butane proportion somewhat higher.

Stenberg/mc/ckl

TABLE 1.

PROPORTION OF BUTANE (ISO AND NORMAL) IN THE METHOD OF PRODUCTION FOR GASOLINE AND MIDDLE OIL

VAPOR PHASE	300/7846	300/7846	300/7846	300/7846	700/6434	700 Bleaching Earth	700 Bl. Earth	300 Bl. Earth	300 Bl. Earth	300 Bl. Earth
Step I. Pressure/Catalyst	300/6434	300/6434	300/7846	300/7846	700/6434	-	50/DHD	-	-	200 Bl. Earth
Step II. "	"	"	"	"	"	"	"	"	"	"
Step III. "	"	50 DHD	"	50 DHD	"	"	"	"	"	"
% Gasoline/Liquid phase gasoline + middle oil	82.7	75.1	87	77.2	84	83.7	79.2	75.6	77.8	79.2
m ³ /t of gasoline	935	945	895	940	1000	645	650	815 ?	750	680
% by volume of aromatics in gasoline	7	50	5	50	10	-30	50	50	50	40
% Normal butane in the vapor phase/gasoline	4.5	5.6	3.1	4.4	3.8	4.1	4.9	5.1	6.8	5.7
% i-butane in vapor phase/gasoline	11.6	10.1	6.1	5.6	5.6	2.8	3	1.1	3.3	3.8
% Total butane in vapor phase/gasoline	16.1	15.7	9.2	10.0	9.4	6.9	7.9	6.2	10.1	9.5
% Normal butane in liquid + vapor phase/gasoline	11.1	12.8	9.4	11.5	10.3	10.7	11.8	12.3	13.8	12.6
% i-butane in liquid + vapor phase/gasoline	12.6	11.2	7	6.7	6.6	3.8	4	2.2	4.4	4.8
% Total butane in liquid + vapor phase/gasoline	23.7	24.0	16.4	18.2	16.9	14.5	15.8	14.5	18.2	17.4

TABLE 2.

PROPORTION OF BUTANE (ISO AND NORMAL) FOR THE METHOD OF PRODUCTION FOR GASOLINE AND FUEL OIL

VAPOR PHASE	300/7846	300/7846	300/7846	300/Al.S1	700/Al.S1	700/Al.S1
Step I. Pressure/Catalyst			300/7846			700/Al.S1
Step II. "	300/6434		300/6434			50/DHD
Step III. "			50/DHD			
% Gasoline/liquid phase gasoline + middle oil	84.3		76.5	80.7	82.8	78.2
n ² B ₂ /te of gasoline	750		760	600	575	600
% by volume of aromatics in gasoline	10		50	-40	-33	50
% Normal butane in the vapor phase/gasoline	4		5.9	5	4.7	5.7
% I-Butane in vapor phase/gasoline	9.7		9.7	2.6	1.6	1.8
% Total butane in vapor phase/gasoline	13.7		15.6	7.6	6.3	7.5
% Normal butane in liquid + vapor phase/gasoline	14.3		17.1	15.7	15	16.7
% I-butane in liquid + vapor phase/gasoline	11.2		11.3	4.2	3.1	3.7
% Total butane in liquid + vapor phase/gasoline	25.5		28.4	19.9	18.1	20.4

Sternberg/mo/prl

T.O.M. Reel No. 4, p 124-130
 Target 30/4.08
 Bag No. 2745

CALCULATION OF RELATIONSHIP BETWEEN THE
 TEMPERATURE OF THE MATERIAL AND THE
 THICKNESS OF INCRUSTATION ON PASTE HAIRPINS.

Gelsenkirchen-Horst, Feb. 4, 1943.

It has been observed that as the operating time is lengthened the drop in pressure at the outlet side of the paste preheaters increases rather steadily while other conditions remain unaltered, and with it the necessary inlet temperature of the heating gases for the same amount of heat transferred, and also naturally the temperature of the hottest part of the hairpins which is used as a characteristic of strains related to creep strength. These phenomena are explained on the strength of actual experiment on poorly conducting incrustations on the inside of the hairpins, which increase in thickness with time. The question was studied by Dr. Wilde at I.G. Oppau. I wish to record here, with his permission, the computations recommended by him.

1. Temperature of materials.

The value of heat transfer to the outside, referred to a smooth surface, is designated as a constant α_2 for preheaters of some definite construction and operating conditions. The temperature characteristics of the preheater must naturally be known. The most dependable temperatures are taken from the records of the stalls, the rest are estimated as well as possible. The heat losses of the preheater, amounting to 3×10^5 to 4×10^5 kcal/hour must appear reasonably distributed among the paths, and the mixing temperature for the gas and paste must be introduced into the computations. The individual average specific heats used are:

For heating gas	0.32 kcal/mm ³ o
hydrogen	0.34 " "
paste	0.55 " /kg ^o

The numerical value 0.55, which also includes the rinsing oil, is, according to the data of the I.G., valid for the total temperature interval of about 250 - 450°, and with an average solid content of about 48%. When the concentration varies more or less strongly, the specific heat varies within narrow limits according to fig. 1. Obviously the different amounts of material moving with the paste and mixture must

be taken into consideration. It remains to measure the degree of efficiency of the preheater, which must amount to around 70%, and which gives information on the correct choice of the amount of heating gas.

Dr. Wilde gets the numerical value of α_1 from the simple case of the hairpin. The heat transfer number for the ribbed surface is calculated for it as $k =$ about 10 to 12 kcal/m²h°. The inside surface of the hairpin is throughout $F_1 = 8 \text{ m}^2$, the total outer surface in the narrow 14 mm ribbed part $F_e = 200 \text{ m}^2$, and with the wider 20 mm spacing $F_w = 150 \text{ m}^2$. A heat transfer number calculated to the inside becomes in general:

$$K_1 = k \frac{F_e + F_w}{F_1}, \text{ where } \frac{F_w}{F_1} = 18.7, \text{ and } \frac{F_e}{F_1} = 25.$$

We get therefrom for the more important narrow rib-spacing:

$$K_1 = (10 \dots 12) \times 25 = 250 - 300 \text{ kcal/m}^2\text{h}^\circ$$

For the unribbed tube, considered as cylindrical, having the heat conductivity λ_R of the material, we have

$$\frac{1}{K_1 D_1} = \frac{1}{\alpha_1 D_1} + \frac{\ln \frac{D_2}{D_1}}{2\lambda_R} + \frac{1}{\alpha_a D_2}$$

$$\frac{1}{K_1} = \frac{1}{\alpha_1} + \frac{D_1 \ln \frac{D_2}{D_1}}{2\lambda_R} + \frac{1}{\alpha_a} \frac{D_1}{D_2}$$

We may take $\lambda_R = 30 \text{ kcal/m h}^\circ$, and $D_2 = 0.171$, for $D_1 = 0.090$, and we get:

$$\frac{1}{K_1} = \frac{1}{\alpha_1} + 0.00096 + \frac{1}{\alpha_a} \cdot 0.526; \quad \frac{1}{\alpha_a} = \frac{\frac{1}{K_1} - \frac{1}{\alpha_1}}{0.526}$$

α_1 acquires a high value for pure hydrogen under high pressure. The lower limit is estimated at about 2000, the upper limit as ∞ .

$\alpha_1 = 2000$		$\alpha_1 = \infty$		
$K_1 = 250$	$K_1 = 300$	$K_1 = 250$	$K_1 = 300$	
$\frac{1}{\alpha_a}$	$\frac{0.00400 - 0.00146}{0.526} = 0.00480$	$\frac{0.00333 - 0.00146}{0.526} = 0.00355$	$\frac{0.00400 - 0.00096}{0.526} = 0.00580$	$\frac{0.00333 - 0.00096}{0.526} = 0.00450$
α_a	208	282	172	222
AVERAGE 245		AVERAGE 197		

α_a is used to calculate the inside and the outside wall temperatures from the temperatures of the heating gases and of the products, and too high values are to be preferred to too low values; we take therefore

$$\alpha_a = \text{abt. } 250 \text{ kcal/m}^2\text{h}^\circ$$

The average heat transfer number K , which is to be recalculated to K_1 is obtained from the above mentioned temperature changes of the preheater and from the occupancy of the hairpins with all the gases as well as from the initial and final temperature differences \bar{t}' and \bar{t}'' . The accurate average logarithmic values of the temperature difference $\frac{\bar{t}' - \bar{t}''}{\ln \frac{\bar{t}'}{\bar{t}''}}$ are however only necessary when the ratio $\frac{\bar{t}'}{\bar{t}''}$ differs very much from 1; in all other cases the somewhat larger arithmetical average $\frac{\bar{t}' + \bar{t}''}{2}$ is sufficient. v. Table 1.

Table 1:

$\frac{\bar{t}'}{\bar{t}''}$	1.0	1.5	2	2.5	3
$\frac{\bar{t}' + \bar{t}''}{2} \text{ log.av.}$	1.00	1.01	1.04	1.07	1.10

The required tube wall temperatures form at any time a definite part of the temperature differences of the heating gas temperature t_g at the proper place and the value α_1 found purely by computation. If the corresponding temperature of the product is t_p , we will have in general $\bar{t} = t_g - t_p$, and, with the help of the auxiliary value K_1 the wall temperatures at the

outside: $t_a = t_c - (t_c - t_p) \frac{1/\alpha_a \frac{D_1}{D_a}}{1/K_1} = t_c - (t_c - t_p) \frac{K_1}{\alpha_a \frac{D_a}{D_1}}$ inside: $t_1 = t_c = (t_c - t_p) \frac{1/\alpha_a \frac{D_1}{D_a} + \frac{D_1}{2\lambda} \ln \frac{D_a}{D_1}}{1/K_1}$
 $t_2 = t_c - (t_c - t_p) \frac{K_1}{475}$

Some of the temperatures found in this way, when compared to actually measured temperatures, give an idea on the unavoidable variations.

The heat transfer value α_1 which is used in computations can be calculated from K_1 and the coefficient α_o . It should be valid for the inside of the paste hairpins,

when the latter are free from all incrustations. Should some other value be used instead for α_B for the heat transfer number from paste to wall determined in some other way, and should it be larger than α_1 , we would be in position to calculate the thickness of the crust, δ , using the heat conductivity of the crust $\lambda = \text{abt. } 1.5 \text{ kcal/m h}^\circ$. We may consider for the sake of simplicity the layer as a plane wall, and get:

$$\frac{1}{\alpha_1} = \frac{1}{\alpha_B} + \frac{\delta}{\lambda} ; \delta = \lambda \left(\frac{1}{\alpha_1} - \frac{1}{\alpha_B} \right)$$

However, computations of this kind have so far but little actual value, because α_B can not be reliably estimated. The measured K , determined with new hairpins, and used with the selected α_2 calculated in the same way as α_1 , may furnish some ideas on the corresponding values of α_B ; they vary between 150 and 350 kcal/m²h^o, amounts confirmed by the K values of coal-paste regenerators.

2. Increase in the Pressure Reduction.

One should also be able to draw conclusions from the drop in pressure on the average thickness of incrustation. We shall introduce the friction coefficient ζ .

The indicator θ could be used for a clean tube.

$$\Delta P_o = \zeta \frac{L}{D_1} \%$$

L in this formula represents the straightened out hairpin and including the revaluated bend, and the percentage of standing pressure $\frac{W_o^2}{2g}$ in the unstricted

cross section depends directly on the thru-put of hydrogen, paste and oil, as well as on the temperature and pressure. The indicator "b" represents here the operating conditions. The diameter may be decreased from D_1 to D_b , so that $D_b = D_1 - 2\delta$, where the thickness of incrustation δ need not be numerically equal to that used in Section 1. The velocity of the products increases in proportion to the cross section, and the true pressure decrease, ΔP_b kg/m², in proportion to the theoretical value, will become

$$\frac{\Delta P_b}{\Delta P_o} = \left(\frac{D_1}{D_b} \right)^5$$

An exact solution is obtained after introducing δ .

$$\frac{\delta}{D_1} = \frac{1}{2} \left(1 - \sqrt[5]{\frac{\Delta P_o}{\Delta P_b}} \right)$$

We may use the simplified expression when the thickness is small in comparison with the diameter:

$$\frac{\Delta P_o}{\Delta P_b} = \left(1 - \frac{2\delta}{D_1} \right)^5 \approx 1 - 10 \frac{\delta}{D_1}; \quad \frac{\delta}{D_1} \approx \frac{1}{10} \left(1 - \frac{\Delta P_o}{\Delta P_b} \right).$$

ΔP is again obtained from measurements of the pressure reduction during operation, corrected for conduction outside the preheater, the check valve (equivalent to about 30 m of straight high pressure tube NW 90) and the regenerator, and recalculated to a single hairpin, so that P_o may be calculated under certain assumptions. The proof may be limited to the mixed portion. Dr. Wilde suggests the following convenient way, with the necessary coefficients:

Thru-puts:

Hydrogen:

N m³/h

$\rho_n \approx 0.25$ kg/m³

Paste (including vehicle and rinsing oil):

B kg/h

$\rho_p \approx 900$ kg/m³

Let the average pressure and temperature of the preheater be 700 atm. and 375° at which the compressibility may be set $\lambda = 1.2$, and therefore:

$$\frac{\delta W}{\delta B} = \frac{700}{1} \times \frac{288}{273 + 375} \times \frac{1}{1.2} = 258$$

	Operating Volume	Weight	
Hydrogen	$\frac{W}{258}$	$W \times 0.25$	Velocity of product: $w_b = \frac{(W + B)}{258 + 900} \cdot 3600 \times \frac{\pi D_1^2}{4}$
Paste and oil	$\frac{B}{900}$	B	

The pressure will now become:

$$F_o = \frac{\left(\frac{W}{258} + \frac{B}{900}\right)^2}{2g \left(\frac{3600 \pi D_1^2}{4}\right)^2} \times \frac{W \times 0.25 + B}{\frac{W}{258} + \frac{B}{900}} = \frac{(258 + 900) \left(\frac{W}{4} + \frac{B}{900}\right)}{19.62 \left(\frac{3600 \pi D_1^2}{4}\right)^2}$$

or, rearranged for the paste thru-put B,

$$\text{percent} = \frac{1}{19.62} \frac{B^2}{\left(\frac{3600 \pi D_1^2}{4}\right)^2} \left(1 + \frac{1}{4} \frac{W}{B}\right) \left(\frac{1}{258} \frac{W}{B} + \frac{1}{900}\right) =$$

$$= \frac{1}{19.62 \times 900 \left(\frac{3600 \pi D_1^2}{4}\right)^2} B^2 \left(1 + \frac{1}{4} \frac{W}{B}\right) \left(1 + \frac{900 W}{258 B}\right)$$

The law of squares can now be readily recognized, and a correction factor must be added depending on $\frac{W}{B}$, the value of which one would prefer to have close to 1. To accomplish it, we select as a "reference" mixture about $W = 30000 \text{ mm}^3/\text{h}$, or

$\frac{W}{B} = 1$, which will give us:

$$\text{percent} = \frac{1}{19.62 \times 900 \left(\frac{3600 \pi D_1^2}{4}\right)^2} B^2 \left(1 + \frac{1}{4}\right) \left(1 + \frac{900}{258}\right) F =$$

$$= \frac{5.6}{19.62 \times 900 \left(\frac{3600 \pi D_1^2}{4}\right)^2} B^2 F$$

using the correction factor

$$F = \frac{\left(1 + \frac{1}{4} \frac{W}{B}\right) \left(1 + \frac{900 W}{258 B}\right)}{5.6}$$

For $D_1 = 0.09$ m. = 9 cm, we get:

$$\text{percent} = \frac{5.6}{19.62 \times 900 \times 22.9^2} \quad B^2 F = \frac{F^2}{1.65 \times 10^6}$$

Should $\frac{B}{W}$ be considered an independent variable, the following values of F may be obtained from table 2 and fig. 2:

Table 2:

$\frac{B}{W}$	0.5	0.6	0.7	0.8	0.9	1	1.1	1.2
$\frac{1}{4} \frac{W}{B}$	0.5	0.417	0.357	0.313	0.278	0.250	0.227	0.208
$3.48 \frac{W}{B}$	6.96	5.80	4.98	4.35	3.87	3.48	3.16	2.90
F	2.13	1.72	1.45	1.26	1.11	1.0	0.91	0.84

This shows immediately that, at least in the range considered, the approximation entered as broken lines in fig. 2 is valid:

$$F \sim \frac{W}{B}$$

and also:

$$\text{percent} \sim \frac{B \times W}{1.65 \times 10^6}$$

For values of $\frac{B}{W}$ smaller than shown in fig. 2, F increases more rapidly than required by the approximation.

The fall of pressure per hairpin with clean walls becomes, when L is about 30 m long, and the friction value $\zeta = \frac{1}{30} \pm 5\%$ (the range of operation measurements of I.G. was between 0.032 and 0.035):

$$\Delta P_0 = \frac{1}{30} \frac{30}{0.39} \zeta = 11.1\% = \frac{6.7}{10^{10}} B^2 F \text{ kg/m}^2$$

One may use to advantage the unit

$$\Delta P_0 = \frac{6.7}{10^{10}} B^2 F$$

for a comparison of the observed with the theoretical pressure drops.

To eliminate the powers of 10, we may set B in cm^2 , and obtain the simpler expression

$$\Delta P_o \sim \frac{6.7 B^2 (te/h)}{10^4} \quad \text{or} \quad \frac{B^2 (te/h)^2}{1500}$$

For computation purposes we may use the approximation:

$$\Delta P_o (\text{atm}) \sim \frac{6.7 B^2 (\text{kg/h})}{10^{10}} \frac{N}{B} = \frac{6.7 B (\text{kg/h}) N (\text{mm}^3/\text{h})}{10^{10}}$$

$$\sim \frac{B (te/h) N (100 \text{ mm}^3/\text{h})}{1500}$$

We may now plot ΔP_o against $B (te/h)$ using N as the parameter, as has already been done by Dr. Wilde, and the curves will then naturally be expressed in terms of B . When plotted on a logarithmic scale with the drop in pressure ΔP_o against the thru-put B , parallel straight lines are obtained, as in fig. 3. The range of $B = 15$ to 50 tons has been chosen. By entering δ against $\frac{\Delta P_o}{\Delta P_b}$ we obtain a simple functional relationship, fig. 4, which gives us directly the thickness of the crust. For the sake of uniformity, we have selected the same scale as used in the discussion of the drop in pressure, fig. 2. Solutions for δ in section 1 could also be conveniently tabulated by drawing three uniformly spaced lines thru a point, and this creates even more forcibly the impression than fig. 4, that this computation was built on a solid foundation.

The growth of the crust during operation may be observed not through the many uncertain assumptions of recalculated temperatures of supposedly identical thru-put, but more dependably and simply from the drop in pressure calculated to a single hairpin, ΔP_o , from which the apparent friction coefficient ζ_b may be recalculated for the original diameter D_1 .

$$\Delta P_b = \zeta_b \frac{L}{D_1} \frac{B^2}{F}; \quad \zeta_b = \frac{D_1 \Delta P_b}{L \frac{B^2}{F}} = \frac{0.09 \Delta P_b 10^4}{30 \frac{B^2}{F}} = 1.65 \times 10^6$$

$$= \frac{\Delta P_b (\text{at})}{B^2 F} \times 50 \times 10^6$$

$$\zeta_b = \text{abt. } 50 \times \frac{\Delta P_b (\text{at})}{B^2 (te/h)^2 F}$$

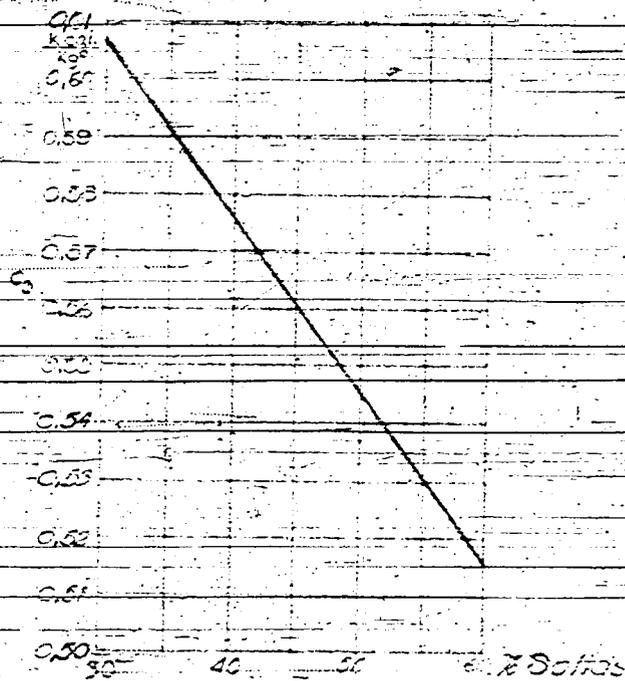


Fig. 1. Average sp. heat C_p of the coal paste in the temperature range of $250-400^\circ$ in relation to the solid contents (30-60%). Values based on:
 Coal 0.39 kcal/kg
 Oil 0.70 " " "

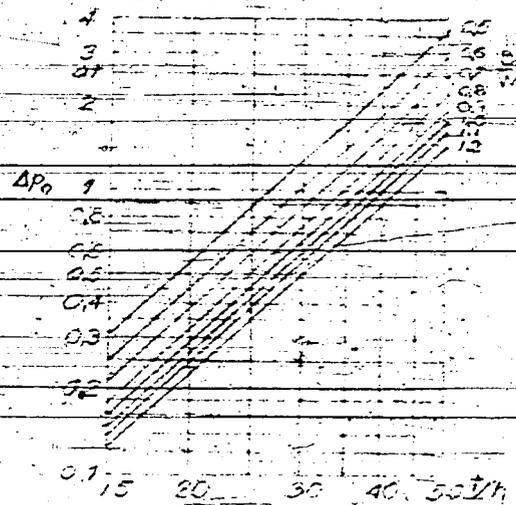


Fig. 3 Reduction in pressure in the non-intruded hairpin, in relation to the paste throughout the parameter B/W .

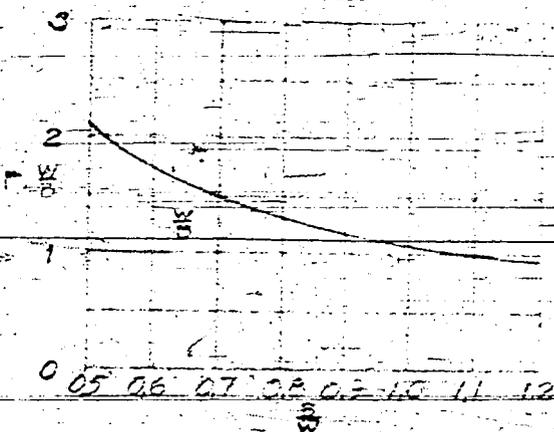


Fig. 2 Factor F from the relationship between amounts of paste & gas (W) .

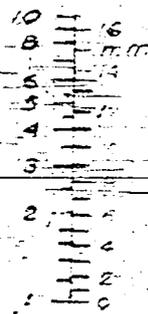


Fig. 4 Thickness of crust δ in relation to the proportion of paste.

T.O.M. Reel No. 126
 Bag No. 2075
 Item No. 124

Gasification with 700 atm. in Vapor Phase

DETAILS OF DISCUSSIONS IN WELHEIM,
 MARCH 26 AND 27, 1941

Industrial size equipment in Welheim was formerly operated at 640 atm., now at 510 atm. As reason for it, the possibility was given of difficulties one might possibly meet with the NiO materials at too high pressure.

Dr. Ibing's experiments were carried out at a minimum of 500-550 atm. Deterioration of the catalyst could be observed at lower pressures. The gas used contained 98% E₂.

The converter used had 5.3 liter catalyst volume and was operated at all thru-puts with 15 m³ of gas (gas measured at atmospheric pressure at the outlet).

Tables below were collected during discussion in Welheim.

COMPUTATION OF GASIFICATION BY
 THE DREESCHMIDT ANALYSES
 (Page 6 of Report)

<u>Welheim 24 hr. Balance</u>		<u>Calculated by the Leung Method of Computation</u>
<u>5.11. Converter</u>		
Feed Stock	184.6 kg.	
Gasoline production (Gasoline contains 2% C ₄)	74.5 kg.	87.5% C = 65.20 kg. C (C ₄ -free correction = 73.0 kg. for 87.5 C = 63.8 kg.
<u>Converter outlet gas</u> Converter gas 325 N m ³		
Hydrocarbons	1.47%	6.98 kg.
Carbon number	2.16	3.16% C = 10.3 m ³ of C = 5.55 kg. C

The catch pot expansion gas = product gas = 9.943 m ³		
Hydrocarbons	31.5% = 6.9 kg.	
C-number	3.29	10.35% C = 10.30 m ³ C = 5.49 kg. C
	Gas 13.9 kg.	11.04 kg.
Gas/the injector	7.5%	(Correction for 2% C ₄ from gasoline 12.28 kg. C
Gas/Gasoline + Gas (Recalculated to C ₄ -free gasoline	<u>15.8%</u> <u>17.6%</u>	Gasification/gasoline + gasification 14.5% (Calculated/C ₄ -free gasoline 16.2%)

COMPUTATIONS OF GASIFICATION FROM STOCK ANALYSES (Page 7 of Report)

<u>Welheim 24 hr. Balance</u>		<u>According to Leuna Computation Method</u>		
<u>5 li. converter</u>				
Feed	184.6 kg.			
Gasoline production	74.5 kg	87.5% C		65.20 kg C.
Hydrocarbons - 325 m ³ of converter gas		% C-gas	m ³ of C-gas	
	0.54%	0.54		
	0.33%	0.66		
	0.61%	1.83		
	0.00%	-		
(CO 0.16)	6.0 kg.	3.03	9.85	5.29 kg. C

Production Gas 9.943 N m³

C ₁	2.82		2.82		
C ₂	5.42		10.84		
C ₃	10.10		30.30		
n-C ₄	5.8		20.72		
i-C ₄	5.72		22.88		
<hr/>					
C ₁ -C ₄		5.70 kg.	87.56	8.72	4.67 kg. C
C ₅	5.13	1.65 kg.	25.65	2.58	(1.37 kg.)
<hr/>					
Catch Pot		167.3			
C ₂	0.02% by weight				
C ₃	0.35% by weight				
i-C ₄	0.61% by weight				
n-C ₄	1.10% by weight =	3.48 kg.	2.08		2.88 kg. C
<hr/>					
C ₅ + 3.36 =	Σ				12.84 kg. C

Gasoline + vapor phase gasoline 181.25 kg.

66.57 kg.
79.41 kg.

Yield of crude 98.2%

C gasification/gasoline +
gasification = 16.2%

When the rough balance of 24 hrs. agrees as in the case above, the gasification at Welheim is computed as follows:

100 kg. Feed stock:

- 0.79 kg. C₁
- 1.18 kg. C₂
- 3.49 kg. C₃
- 1.27 kg. i-C₄
- 1.79 kg. n-C₄

of 10.8 kg. in the gasoline = 7.7 kg. C₁-C₄ gas.

184.6 kg. feed
100 kg. feed
Added 2% C₄H₁₀ to the gasoline

75.5 kg. gasoline
40 kg. gasoline
0.8 kg. C₄H₁₀

40 kg. gasoline
7.7 kg. gas

=
16.0% gasification

Starnberg/mc/pk1

Inspection of Gasoline derived from Scholven
Soft Coal Liquid Phase Gasoline + Middle Oil
Treated in 700 Atm Vapor Phase Process
(Ruhroel Method)

Operating Conditions

Pressure	550 Atm Fresh Gas
Temperature	26.5 m.v.
Throughput	1.42 Kg/l/h
Gravity at 15°	0.779
Vapor Pressure	0.42 atm
<u>Distillation (A.S.T.M)</u>	
1 B ²	41.0°C
5 Vol. %	63.0 "
15 "	77.5 "
25 "	85.5 "
35 "	92.0 "
45 "	98.5 "
55 "	105.0 "
65 "	113.0 "
75 "	123.5 "
85 "	136.0 "
95 "	158.0 "
F.B.P.	98.0 % / 167.5°C
Residue	1.0 %
Loss	1.0 %
10 Vol. %	71.5°C
90 "	145.0 "
100° Point	47.5 Vol. %
Factor	105.2
Aniline Point	+ 16.0°C
Corrosion Test	negative
Doctor Test	"
Gum Test-Glass Dish	0.0 mg
Bomb Test	
+ 0.12 Vol. % Tel	
Fitch, taken directly	10.2; 9.1 mg /100 ccm
Lead Residue	8.8; 9.0 mg /200 ccm
Iodine Number	1.09; 1.22
<u>Hydrocarbon Classification</u>	
Aromatics - Olefins	42 Vol. %
Naphthenes	37 "
Paraffins	21 "
<u>Element Analysis</u>	
C	87.88%
H ₂	12.06%
<u>Octane No. (Mot. Meth.)</u>	
Unleaded	77.0
+ 0.05 Vol. % Tel	86.0
+ 0.12 " "	89.5

DEW. Dir. Dr. Winkler
4 x Reg.

T.O.M. Reel No. 126

T-29

Bag No. 2075

Item No. 127

EXPERIMENTAL WORK ON 700 ATM.
VAPOR PHASE OPERATIONS

Discussion with Dr. Winkler, January 4, 1941

Dr. Winkler reports his results on aromatization at 700 atm. He worked at a total of 630 atm. pressure which corresponds to about 450 hydrogen pressure with a thru-put of 0.4 H₂ gasoline/li. of catalysts/hr. Increasing the thru-put would have been possible on the small scale experiments but does not appear to be possible on the industrial scale even by using blends. The concentration of the aromatics in the 165° end point gasoline obtained from tar middle oil (including about 8% of sump gasolins) amounts to about 55-58% by weight (50-53% by volume). Dr. Winkler has, however, recently used catalysts which in small scale tests (5 li. converter) produced 70% of aromatics with tar middle oils. The thru-put amounted to 0.4. No information is yet available on the gasification. Dr. Winkler tells the following on the yield:

The loss calculated to the middle oil used amounted to about 12.2-12.4%. There is in addition a 3.5-5% gas loss during this stabilization in Scholven. Moreover, the gasoline must be refined with sulphuric acid and redistilled. Dr. Winkler assumes here a very small loss of about 1%. The total carbon gasification calculated from these figures amounts to about 17.6-19% calculated on gasoline + gasification.*

* The carbon gasification is computed as follows:

100 part middle oil with 89.7% C	= 89.7 parts C,
87.7% of gasoline with 87.5% C	= 76.7 parts C,
Carbon loss	= 13.0 parts C

There must be added to it 3.5-5 parts of stabilization loss with 80% C = 2.8-4 parts of C, making the total loss = 15.8-17 parts C.

Referred to the 89.7 parts of C used in the middle oil, we get a gasification of 17.6-19%.

These values are in agreement with the 1-liter experiment performed at Ludwigshafen.

Dr. Winkler makes an additional statement about the yield by saying that he obtained 87 parts of high-test gasoline from 100 parts of middle oil; and out of that 82 parts of refined and stabilized gasoline, which corresponds to a production factor of 1.22.*

There was an agreement on about a 3% reduction in yield when substituting tar middle oil by coal liquefaction middle oil because of the lower C content of the middle oil. Moreover, the yield of the coal liquefaction middle oil would be lower because of the higher gasification of that oil if the same content in aromatics is to be obtained as with the tar middle oil. Dr. Winkler found that a reduction of 2-3 in the octane number is obtained under the same operating conditions when coal liquefaction oil is used instead of the tar oil.

Dr. Winkler has not yet tested the effect of sump gasoline when operating on coal liquefaction product, because during tar hydrogenation only a little sump gasoline is obtained and of a high boiling point. This question is, however, of great importance during the operation with coal middle oil, in particular when operating in Upper Silesia for the production of fuel oil, where about 35% sump gasoline is present in the gasoline + middle oil of the sump phase.

Some other points have been discussed which must be considered during the production of aromatic gasoline. The amount of i-butane is first to be mentioned because it forms but a fraction of that obtained during normal benzinization for aviation gasoline or during benzinization + DHD. Even when conducting the 700 atm. aromatization at a lower temperature, in order to produce gasoline suitable for DHD with a low gasification, less i-butane will be produced because the temperature is much higher than at the present two-stage benzinization.

* This is to be compared with the yield of 5058/6434 gasoline of the benzinization + DHD process which consisted of 86 parts and from which only 73 parts of DHD gasoline were produced. We countered this statement by saying that the coal liquefaction middle oil as raw material would produce at 700 atm. aromatization not 82 parts gasoline, as does the tar middle oil, but only 78 parts. Moreover, the 86 parts yield during benzinization at 300 atm. is too low a value. With our best DHD catalysts, we figure for a yield on a small scale test of 78 parts, or the same as with the 700 atm. aromatization, and with the catalysts industrially used we assume a yield of 75 parts.

It is to be noticed that the quality of DHD gasoline from coal liquefaction middle oil is equal to the aromatic gasoline from tar middle oil, producing therefore a somewhat better overload yield than the aromatized gasoline from the liquefaction middle oil.

Dr. Winkler amended the data in the record that the hydrogen consumption/te gasoline of 165° C H. was only 700 m³ instead of the 900 m³ previously given.

Further study of the problem will be made by Dr. Winkler by using middle oil from Scholven and Gelsenberg and running high pressure experiments at Ludwigshafen with 2 te from each batch, and also using 2 te of his tar middle oil and 30 li. of his new catalysts in 10 mm. pills. The yield and hydrogen consumption during 700 atm. aromatization at Welheim and Ludwigshafen will again be tested in a different way using the same raw materials and the same catalysts.

Dr. Winkler will run his tests with the circulating gas while the experiments at Ludwigshafen were performed with fresh gas of a different pressure and with the addition of 0.1-0.15% H₂S. Experiments with coal middle oil will be carried out for the production of gasoline with 50% by volume of aromatics. The gasolines will be refined with 96% sulphuric acid, redistilled to an end point of 165°C and stabilized to vapor pressure of 0.4.

In addition, Dr. Winkler will work out the cost of the 700 atm. aromatization for the production of 235,000 or 120,000 te of gasoline in the high pressure experiments at Ludwigshafen.

Dr. Winkler gave this additional information based on his tests: The average C on gasification is 2.5; there is a total of 30-36% of iso C₄ in the total C₄; 280 mls. of hydrocarbons are produced in a converter with 10.5 mls. of catalysts and they are distributed as follows:

(The Data Referred to "High Pressure Gasoline"; Stabilization Off-Gas Not Included)

		Recalculated: 20.6% by vol. 8.8% by weight			
C ₁	55.3 mls.	30.3%	"	24.4%	"
C ₂	81.8 "	25.4%	"	30.0%	"
C ₃	68.7 "	23.7%	"	36.8%	"
C ₄	64.0 "				
C ₅	10.6 "				

The middle oil used contained 89.7% C, 9.4% H and 4% phenols. The converter with 10.5 mls. catalysts had 3000 li. of middle oil injected into it on the first day at a temperature of 24 mV. and with refluxing, on the second day 6000 li., and on the third day 9400 li. The temperature was so maintained that the concentration remained about 50-55%, which corresponded to a specific weight of the catch pot of 0.830-0.840. This corresponded mostly to a minimum temperature of 24.8 mV. and a maximum of 6.2 mV. (40° cold junction). The 5 li. converter was operated at 25.5 mV. temperature, which corresponded to the average temperatures of the full size converters. The b middle oil (A.P. - 25°) produced a better yield and a gasoline with higher aromatics than the a middle oil. The amount of gas is about 25,000 m³ (at times only 13,000 m³) to which must be added some 6000 m³ of cold gas. The catalyst gives at once the proper concentration of aromatics (A.P. of the gasoline -4° to -5°), with new catalyst the aromatic concentration tends to be even somewhat higher than is obtained later. It is artificially neutralized right from the start. The new catalyst may also be used at 300 atm. but it deteriorates if the product is not de-phenolized.

The cost of the catalyst is around 1200-1500 RM. Regeneration of the catalyst would not pay, its life is between six months and one year.

Dr. Winkler makes the additional remark that the coal stall yield with low hydrogen pressure is higher than with higher pressure. He draws this conclusion from the difference in the hydrogen sulphide content in the circulating gas and furnishes the following data:

90% H₂ corresponds to 0.05% H₂S and 71-75% H₂ represents 0.1-0.15% H₂S (450-470 atm. of H₂).

Sternberg/mc/fkp

T.O.M. Reel No. 10
 Target 30/4.09.
 Bag No. 2743
 Item No. 2, pp 360-366

Scholven, June 16, 1937

PROPOSED MODIFICATIONS IN THE
 PREHEATER CONSTRUCTION AT
 SCHOLVEN

The Gas Heated Preheater for Proposed Expansion, Discussed
 from the Standpoint of our Most Recent Knowledge.

I. The previous investigations of heat transmissions in the gas heated preheaters of the liquid phase at Scholven gave throughout a very low K value for the second part of the preheater. A direct relationship between the K value and the total concentration of solids of the paste can be established. The highest value of K is obtained when working with vehicle oil (10% solids).

The preheater at present is divided into three parts, which is done for purely constructional reasons, since otherwise the poor heat transfer will go over into the Part III. (see report "K values of the gas heated preheaters for the cold paste", June 11, 1937). Additional determinations of temperatures in Parts I and III have been undertaken to determine in what part of the Part I does one find the beginning of the low values of K, and where they end in Part III. The change cannot be sudden and the measurement of heat conductance of the last few hairpins of the first part and of the first hairpins in the third part will determine a valuable intermediate point. These results may be of value in a general way, and also specifically in the expansion program at Scholven; they will find confirmation in subsequent viscosity measurements in Leuna.

II. Should we base our computations on the low values of K, we will obtain very large surfaces, or very many hairpins, because little heat is transferred in the second part of the preheater. We must therefore try to have everywhere a maximum K value, since otherwise the preheater would become too large and the resistance will then become too great. There are several possibilities to meet these requirements:

(1) A lower concentration of solids, for instance 40%. However such a concentration is in general uneconomical; should it be produced with vehicle oil which is repeatedly used under pressure, heated and distilled, there is always a certain element of uncertainty and the reaction space moreover is unnecessarily overloaded. However, the maximum amount of the hot circulation material to be pumped over must be established and its effect be determined on the yield, gasification, etc., because hot circulation pumping may yet prove to be the most economical way for lowering concentration.

(2) Operations of Converter II which had an operating time of 37 days with 46% concentration, without the use of chlorine confirmed, or rather

strengthened, the opinion held from the start that the plugging-up was caused by dry cold paste plugs, which also reduced the heat transfer, proving that additional means to relieve such plugging must be found.

a. The swelling is one of the characteristic properties of coal; some coals may swell more than others. The swelling must be taken into consideration at least in an effort to reduce the swelling zone (see Section III.)

b. The swelling of the coal particles absorbs the oil present during mixing because of increased surfaces; extremely great fineness of grinding acts in a similar way. Should both causes be present, that is if an extremely finely ground coal is permitted to swell, a completely dry state of the paste evidently may be produced. A sieve analysis of the Scholven paste has shown that 70% of the coal passes through 10,000th mesh. This fineness is normally high when compared with the 30-40% in Leuna and in I.C.I., even aside from the fact that brown coal does not swell. It is not yet certain whether this 70% through the mesh actually contains the fine dust of Lurgi which is present to the amount of 10 to 15% (a question mark in pencil). The following facts which may furnish the solution of the whole problem:

1. Breaking down of a cube into cubes of half the length of the edge, doubles the surface of the newly formed cubes, in analogy to spheres;

2. With 46% total solids the high plugging up disappears almost completely when the percentage is only reduced a little, say from 46% to 40%, that is by 6% (in pencil: a question mark and correction to 10%);

3. The Scholven paste has 30% more of the fine particles than the paste of I.C.I. (70% against 40%).

The proportion of the finest particles must be reduced; it may be sufficient to reduce it from 70 to 30-40%.

It is suggested that immediate contact be made with the firm Krupp requesting them to hurry with experiments along that line with the Nordstern coal.

The requirement one usually tries to meet of producing the largest amount of grinding in the first operation may be modified so as not to reach the other extreme, namely of permitting the coarser particles to be above a certain size, as is also done at present.

The recently observed plugging up of the gas heat exchangers is explained by the carrying over of the fine dust, which is very difficult to centrifuge in the HOLD, causes one to expect that should elimination of excessively fine grinding prove of value in the preheater it will also simultaneously relieve many difficulties elsewhere.

III. The above mentioned ways of improving the K value, in particular by changing the grinding, can only be tried out after some time. Should our expectations be fulfilled, the new preheater could be reduced 20% compared to the size based on our present K values. In the meantime other means must be used

to shorten the region of the plugging zone. The former temperature jump between I and III which amounted to around 50°C through the addition of hot hydrogen, should be increased to 75°C. This can be done by raising the temperature of the hydrogen from the heat exchanger and introduced into two or three hairpins, from 400 to 465°C. This hot hydrogen is first added to the hot circuit, the temperature of which will thereby be raised from 330° to 410°. This mixture will be added to the paste after Part I., causing the temperature to rise by around 75°C. Dr. Pier sets the maximum temperature of the reaction of the medium added as + 20°, because hydrogen may readily produce harm at higher temperatures. Temperature balances show that the full benefit of the temperature is not being utilized.

a. Increasing the size of the present preheater (fig 1). It was first intended to use three gas hairpins. When considering the excess temperature of the material, and the fact that the effects of such connection can be obtained with two hairpins, only two hairpins are at present intended to be gas heated, because the use of the V₂A ED eliminates the difficulties with the material. It is being determined whether the temperature of the hot circuit can be raised.

At any rate tests will show whether the temperature can be increased without harm to the production, whether the mixing process requires certain time, and whether the plugging zone can be reduced.

b. A similar heat balance is obtained when designing the converter with 25 te of paste/hr.

IV. Pumps. The poor heat transfer is closely connected with the question of "one or two pumps".

Should one succeed in raising the concentration by changing the grind and by producing a better heat transfer, one pump would be sufficient for the two new converters. For the old converters, in which construction permitted not over nineteen hairpins (formerly 16) two pumps would still be necessary; one cannot foresee at present whether the above changes will permit to increase the K value sufficiently and maintain it at that higher value.

T.O.M. Reel No. 10
 Target 30/4.09
 Bag No. 2743
 Pages 414 to 422

THE DESIGN AND PROPERTIES OF MATERIALS OF
 HYDROGENATION OF THE T52 INSTALLATION AT
 SCHOLVEN AND PÖLITZ (Condensed translation)

Ludwigshafen, April 6, 1940

I. Distribution of Work:

It is intended that the laying out of the stalls and the preparation of the installation be done at I.G. Ludwigshafen. The former are to be sent to Leuna, the latter are to be delivered by the I.G. work shops directly to Scholven for transmission to ? and Uhde, while orders outside of the I.G. are to be shipped to Scholven with a transmission to Uhde. Next follows a list of drawings to be sent to Uhde (omitted).

II. The ordering of the materials is discussed (not translated). The originally intended steam preheater for the T52 installation will cause different difficulties in operation as well as relatively high operating costs (for instance where the steam must be maintained at an elevated temperature even during the autothermal operations). The question has been discussed of equipping the stall units with electrical preheaters, because the partial conversion of Nordstern and Politz works to 7019 catalyst would provide the transformers and the other electrical equipment for the heating of the preheater. One transformer with the four tubes would be sufficient for the heating of each T52 stall. The question whether Nordstern or Politz can give up the necessary transformers is being discussed from Leuna for Scholven and from Uhde for Politz.

I. Heat Exchanger.

The maximum thru-put through the converter amounts to

$$\begin{aligned} 52,000 \text{ m}^3/\text{h Gas } \gamma \text{ (150)} &= 0.2 \text{ kg/m}^3 \\ 6,500 \text{ kg/h Product } \gamma \text{ (150)} &= 1.0 \text{ kg/ltr} \end{aligned}$$

The heat value amounts to 20,000 k cal/°C

When the operations are carried out isothermally there is the additional temperature picture to be included

$$K = \frac{20,000 \times 160}{71 \times 190} = 230 \text{ k cal/m}^2 \text{ } ^\circ\text{C, h.}$$

This presupposes therefore 600 diameter heat exchanger 12 m long with 301 tubes or with around 190 m².

The accompanying table H2741-16 shows the relationship of cross sections of the different heat exchanger constructions. A 500 heat exchanger is sufficient for Stettin.

The inlet temperature in the converter must be reduced when the values of K are higher, as may happen at the start of operations or with smaller converter temperature differences between inlet and outlet, and it is advisable to have a cold gas inlet near the converter intake.

II. The electric preheater contemplated permits bringing the system to the temperature with about 450 kW. 600 kW, or around 500,000 k cal/h. may be brought in with the aid of a transformer, and in current operation with poor K values of the heat exchangers or with narrower temperature drops across the catalyst, a heating of the feed mixture of $500,000 + 20,000 = 25^{\circ}\text{C}$ may be done.

III. The cooler must remove 1,300,000 k cal/h. by a cooling of 45°C .

This will require a surface of $F = \frac{1,300,000}{35 \times 500} = 125 \text{ m}^2$
The water requirements are 37 m^3 .

Scholven has adopted a 70 cooler of their own construction with four bunches of tubes and six locations, corresponding to a surface of 150 m^2 . The usual distribution in Scholven is with five units connected in one spot. One system each with two bundles in six positions is sufficient for Stettin; its total surface is 75 m^2 each. The structure is arranged in eight tiers.

Resistance of the Converter

1. Piping and preheater:	
Length including 20% addition for turns (estimated)	120 m
Average temperature	150°
Gas volume	285 m ³
Feed volume	10 m ³
<u>Total volume</u>	<u>295 m³</u>

The velocity reached for a 90 tube exchanger is 13 m/sec.

Weight of the gas	10,400 kg.
Weight of the feed	6,500 "
<u>Total weight</u>	<u>16,900 kg.</u>

ρ of the mixture 57 kg/m³

$\Delta p = 0.025 \times \frac{120}{0.09} \times \frac{13^2}{20} - 57 = 1.6 \text{ atm.}$

2. Converter

Velocity in the 500 converter (empty)
Mixed weight ρ

0.18 m/sec.
50 kg/m³

From experiments with pressure drop across pills
(Dr. Kling) we get

$$J = 25$$

$$J = \frac{1}{D} = 25 \times \frac{7.5}{0.01} = 19,000$$

$$\Delta p = 19,000 \times \frac{0.18}{20} \times 30 = 0.15 \text{ atm.}$$

Considerations of the hose through the cover increases it to 0.5 atm.

3. The resistance of the heat exchanger is primarily a hydrostatic resistance and a variation of 1.0 in either direction is allowed under considerations of the cover hose and the bends.

4. The circulation pipe is made with 9 mm. diameter and according to computation its resistance is 0.5 atm.

The total resistance of the stalls:

a. Pipe and preheater	1.6 atm.
b. Converter	0.5 "
c. Heat exchanger	2.0 "
d. Circulation piping	0.5 "
e. Pre-pressure (cold gas?)	10.0 "

Total about 15 atm.

The circulating pumps are therefore normally laid out with 20 atm. difference in pressure.

Sternberg/ac/pkl

Table 1

	Scholven	Polits
Yr. te of T ₅₂	22,000	12,000
Normal Feed te/h	4.0	1.7
Feed Maximum	6.5	3.4
Number & capacity of pumps	2 & 40	2 & 1.7
Inlet gas, normal	32,000	14,000
Inlet gas, minimum	52,000	28,000
Capacity of circulating pumps with p = 20 atm.	35,000	-
Number	2	-
Number of stalls	1	1 + 1
Dimension of converter	500 x 8	300 x 12
Heat exchanger	600 x 12	500 x 12
Temperature of converter inlet		199° (180°)
Temperature of converter outlet		276° (270°)
Heat exchanger kg.cal/ltr. kg. feed		282
Fresh hydrogen, normal m ³ /h	1000 - 1200	
Fresh hydrogen, 4 to maximum	2000	
Off gas (at 5 atm.), normal m ³ /h	400 ÷ 600	220 ÷ 300
Off gas (at 5 atm.), minimum	1000	440 ÷ 600
Temperature of products in the catch pot		45°
Probable amount of cold gas m ³ /h	10,000 ÷ 18,000	5000 ÷ 10,000

Table 2

thickness of insulation in the upper section of a 600 sqt exchanger of different subdivisions, and suggested increase in the insulation for the 500 heat exchanger.

	Brabeg 600 i.d.	Scholven 600 i.d.	Nordstern 600 i.d.	Italian 600 i.d.	Summa 500 i.d.	Suggestions for 500 i.d.
Number of Pipes	241	241	199	301	151	158
Sections (7)	30	29.5	29.5	27 *	30	28
Inside Diameter	0.037 m ²	0.037 m ²	0.030 m ²	0.046 m ²	0.023 m ²	0.024 m ²
Outside Diameter	0.088 "	0.083 "	0.068 "	0.070 "	0.051 "	0.046 "
Average Surface	288	288	190	286	145	146
Thickness of insulation in the upper section	7.5 ⁱⁿ /m	11 ⁱⁿ /m	32 ⁱⁿ /m	7.5 ⁱⁿ /m	15 ⁱⁿ /m	10 ⁱⁿ /m
						151 22, 28.5 0.023 0.044 140 20 ⁱⁿ /m

* The ends of the pipes are drawn out.

T.O.M. Reel No. 130
Reference No. q, pages 321 to 329

Ludwigshafen, October 8, 1941

RESULTS OF LARGE SCALE INVESTIGATION
OF HEAT EXCHANGE OF PASTE WITH KSO₄,
LUDWIGSHAFEN, 1941

It has been required for some time to have the thru-put through the 4-unit stall of the Upper Silesian installation increased to 50.2 te/h. of cold paste, which would represent a pure coal thru-put of 0.59 kg./ltr./h. This represents a 70% increase over the thru-put at Nordstern or around 30 te/h. It is now obvious that the heating of this large amount of paste would require special attention, especially after having arrived at the conclusion that no substantially larger preheater could be constructed than the Nordstern preheater.

There were at first two possibilities to be considered to heat up the required amount of paste.

1. By the Nordstern method.

Figure I. represents the Nordstern stall scheme. The incoming gas is heated practically to the reaction temperature in the cold path of the gas heat exchanger and added to the cold paste when at about 280°. The mixture is then heated to the reaction temperature in the succeeding hairpins. Were one to heat by this method, a preheater would be required with 45 hairpins and the resistance of the preheater alone would amount to 48 atm. with 90 hairpins.

The heat requirements with a 70% efficiency would amount to 14×10^6 k.cal/h.

The thru-put has in the meantime been increased in Upper Silesia to 60 te/h. and the number of hairpins had to be increased correspondingly to 50. Even if the inside diameter of the hairpins be increased to 100 or even to 110 mm., such a large preheater becomes impractical of construction.

2. The second possibility, shown in figure II., consisted in heating the paste in the same manner as has been successfully done for over ten years with the brown coal phase at Leuna. Heating of all of the paste to 332°C has been done with a K value 130. This showed it to require a relatively low heat consumption of around 6×10^6 k.cal/h. using a preheater with 26 hairpins. It has, however, been previously known, and confirmed by the large scale experiments here that paste cannot be heated in this way, because of the swelling of the coal, which begins at about 250°. The heating could only be performed up to about 250°, by using for instance only two heat exchangers, which would however make it difficult to keep it sufficiently far below the dangerous temperature, while the heat exchanger is still very efficient. This method of interrupted heating would however greatly increase the required number of hairpins and this will greatly raise the resistance of the preheater and increase the heat requirements by about 30%. This method therefore could not be considered for the high thru-put in the Upper Silesian installation, primarily because of the high resistance.

We have accordingly laid out a method of operation for Pölitz, shown in figure III., in which the following advantages of methods 1 and 2 are incorporated:

1. The paste is heated in heat exchangers but with a lower concentration of fresh-coal than would prevail in Method 2. It was assumed that with lower fresh-coal content of the paste the well known heat exchange difficulties will be overcome.
2. The deficiency in the fresh-coal was compensated by means of the paste of a higher concentration in the cold path of the preheater, that is with the high temperature difference of Method 1. Both streams are mixed and brought to the reaction temperature in the hot path of the preheater.
3. The division of the paste into two parallel streams appreciably reduced the pressure drop of the whole system. High thru-puts will become possible when the high resistance has been overcome.

The preheater will require 23 hairpins, the pressure difference will amount to 28 atm. and the effective heat requirement will be around 8×10^6 k.cal/h.

The method numbered 4 is the same in principle as the method numbered 3 but the paste in the two streams is kept at the same concentration. With this method of operation the preheater could have a favorable design with only 18 hairpins, $\Delta p = 8$ atm. with an effective heat requirement of 6.5×10^6 k.cal/h. The large scale tests performed here have shown it to be wrong that a much higher concentration of solids can be used in producing gasoline and fuel oil than when working for gasoline and middle oil, and primarily for the reason that the paste would carry less swelling material by carrying less fresh coal with recycling HOLD. This method had also certain advantages on the low pressure side of the paste with respect to the setting-up the pumps when compared with the method numbered 3.

The local large scale tests had the primary technical objective to clarify the question of heat exchange. It was therefore arranged in such a way as to permit operation by the method 3 or 4.

Figure 5 shows the relations of the heat transfer in the large scale experiment as compared with the heat transfer obtained in Pölitz for the Upper Silesian installation.

Figure 6 shows the changes of the K values during the experiment: K I. II. III. combined. The curve for the total heat transfer is at its lowest and produces no average curve because of the relatively high radiation losses of the piping between the individual heat exchangers which strongly affects the total K value, while naturally the K values of the individual heat exchangers do not have these radiation losses.

We may in general consider the following as being settled:

1. The K values are rather high at around 350° for a paste with 40-43% of solids.

2. No lowering of the K values has been found at the end of the test. Toward the close of the experiments the operating conditions were the same as initially.

3. The K value depends very strongly upon the concentration of solids. The relationship from the concentration of the fresh coal is especially apparent because, after all, it represents the swelling substance. The film oil pumps were stopped for one hour on August 23; these pumps deliver the film oil necessary to produce the lower concentration. The concentration increased suddenly from 41 to 51.6% solids or from 34 to 45% of fresh coal.

Figure 7. The K value dropped inside that hour from 300 to 100, but has not become permanent and one can anticipate a further drop.

The pressure difference, Figure 8, also rose suddenly from around 2 atm. to 4 atm. for the exchanger II. - I.

This unintended test has furnished the proof for the strong dependence of the K values on concentration. After the film oil pumps were started again after one hour of rest the normal values were again obtained.

At the end of the test the concentration was intentionally increased to 47% solids or 39% fresh coal. The K values dropped from around 300 to 120, but assumed again the former value when the initial concentration was reduced to 41% solids.

Figure 9 shows the changes in the pressure difference in the heat exchanger II. and I. during the high concentration run when the concentration was increased from 41 to 47%. Not only did the absolute value increase from 1.8 to 2.5, but a local plugging-up in the heat exchanger could be suspected from the records of the differential pressure indicator. The records are the same as obtained in the level measurements of the catch pot when it is very greatly loaded. Fortunately the plugging was overcome the moment the concentration has again been reduced. The normal values for K as well as the normal recording of the pressure difference lead one to expect that the regenerator was not fouled, at least not very much so. This however will be determined by examination of the unit.

This result is of the utmost importance. It is not always possible to maintain the desired paste concentration in large scale operations and variations of at least 1% are permissible. On the grounds of these experiments we may conclude that one can work close to the limiting values such as have not yet been found to be lastingly harmful in the past.

K Value of the Preheater

Figure 10 reproduces again the figures for the preheater.

The thin paste is heated to 300° in Part I. The thick paste is heated to 430° in Part II. The total paste is heated to 450° in Part III.

The following K values have been obtained referred to the outer surface, at first for the partial stream passing through the exchanger at the concentration of 41%:

Part I. (41%)	about 8.5 k.cal/m ² , °C, h.
Part II. (51.6%)	" 3.8 " " " " "
Part III. (45%)	" 7.1 " " " " "

After the concentration of the thin paste was increased to 47.5%, that is the concentration in all three parts has been made the same or 47.5%, the following values were obtained:

Part I. (47.5%)	8.2 k.cal/m ² , °C, h.
Part II. (47.5%)	4.3 " " " " "
Part III. (47.5%)	6.2 " " " " "

This shows that:

1. In Part I., where heating is done to about 300°, that is where it does not extend appreciably into the swelling range, the K values are not greatly affected by the concentration, and are therefore high.
2. The K value in Part II. is low; the swelling range is entirely inside that part.
3. Part III. is outside the swelling range and the K values are again higher.

The conclusion we may draw about the preheater is that the thick paste component should be preheated in the cold part to about 300 to 330° and that the actual swelling range should be bridged over by the addition of the stream coming from the regenerators. This range is between 300 and 360°. This method has been suggested for Pblitz and has been found most acceptable there. It may be that the work in Stettin will clarify this question.

To summarise, large scale tests had the important result of showing that the regeneration may only be done with thin paste in the method for gasoline and fuel oil. For an average concentration of 47% total solids in the converter feed, the heat exchange of a concentration of 41% is certainly permissible, and it is to be expected that 43% concentration will offer no difficulties. The concentration of the thick paste must correspondingly be raised to 54%. Even paste with 56% can be used if care be taken that the temperature in the pumps be not below 115° and the velocity of the paste is kept below 1.5 m/sec. by suitable choice of piping.

Figure 11 shows the economy of this process.

Earlier tests have shown that the large expenditure in capital for the production of two additional heat exchangers and additional paste pump is holding its own against the lower expenses for smaller preheaters with correspondingly fewer hairpins and a small cooler. It remains to consider the current operating costs:

-5-

We will consider as normal a stall with a gas heat exchanger, in which case the savings in heat will be shown in Figure 11.

With a cost of heat of 0.5 pfg./1000 k.cal., the saving represents a lowering of the operating cost of around 35 RM/h. stall, with a thru-put of only 50 te/h.

The cost for water will also be lower because if the heat for heating is less the requirements in cooling water will be correspondingly reduced. This saving is small however in comparison with the saving in heat and is therefore not taken into consideration.

With an installation of the size of the Upper Silesian which will have a thru-put of 60 te/h./stall, the total saving on heating will amount to 4 (stalls) x 42 x 8000 = 1,350,000 RM/yr.

It is equally important however, that in the method for gasoline and fuel oil without paste regeneration, the design would not be made for a 3 unit stall, not to mention a 4 unit stall, without adopting still larger and completely untested pipe sizes. A much greater number of stalls must be built to reach a certain yearly production.

It remains only to briefly consider the pressure differences in the stall:

Figure 12 represents schematically the stall arrangement and the measured pressure differences in the large scale experiment in comparison with the calculated values for Upper Silesia. The two streams are arranged in parallel and we had to take into consideration only the higher value of the preheater stall when calculating the total and the lower value is therefore enclosed in parenthesis.

We may add the following about the individual pressure differences:

1. (a) value of 0.07 is calculated for the preheater in Leuna, a value of 0.04 is used for Upper Silesia. The higher value of 0.07 indicates a crust formation of about 1.5 mm.
(b) An average crust formation of several mm. is computed for the preheater of the thick part. It is however difficult here to subtract the part contributed by the piping and by the first hairpins, where the viscosity of the paste is still very high. However, pressure measurements as well as the K values of this part tell us that the thick paste probably should not be heated to so high a temperature.
2. The measured pressure difference in the converters is primarily a measurement of hydrostatic pressure. The current weight of the converter contents is measured and the records frequently lead to valuable conclusions of the operating conditions at any time.

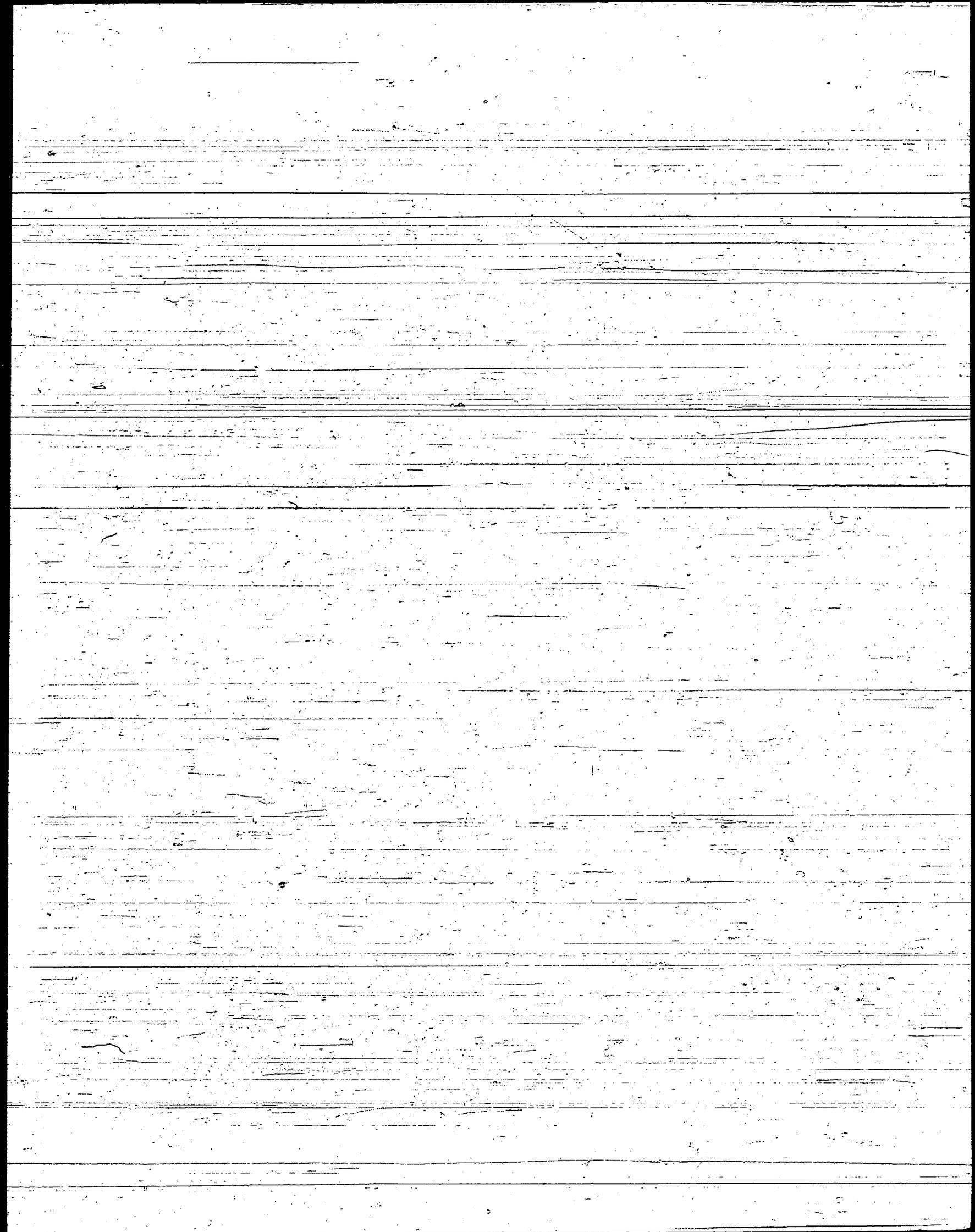
The regeneration of thin paste gives thus the possibility to conduct economically operations in a 4-unit stall with a higher thru-put and relatively small preheater.

Sternberg/mc/ikp

	Case 1	Case 2a	Case 2b	Case 2c
4 fold coal still Total throughput 50.2 te/h. Concentration 47.5% solids.				
Paste through heat exchanger	t/h	20	25.1	32.5
Total concentration of solids	%	38	41	44
Paste direct to preheater	t/h	30.2	25.1	17.7
Total concentration of solids	%	54	54	54
Number of exchangers		3	3	3
K value	Cal/°C, h, m ²	250	200	170
Heating in the preheaters	°C	410	418	380
Preheater				
Capacity of pumps	m ³ /h	168000	168000	168000
Inlet temperature of circulating gas	°C	590	590	590
Outlet temperature of circulating gas	°C	409	505	500
Heat requirements ($\eta = 70\%$)	10 ³ Cal/h	11000	6350	6800
Cost of heat (0.5 m ³ /1000 Cal.)	RM	70	31.75	34
Saving against case 1	RM		38.25	36
Number of hairpins		45	18	20
Δp of hairpins (100 mm)	at	48	22	25

Regeneration	Coal	
	Stall 824, Leuba	Upper Silesia
Pressure	700	700
Heat exchangers	Reg III + II + I	Reg III + II + I
Diameter of shell	600*	600
Length of heat exchanger	11000	18000
No. of small tubes	30	199
Size	18/10	23/14
Grouping	2 1/2	29.5
Outer cross section of flow	00065	0.068
Inner cross section of flow	000235	0.030
Throughput of paste	1.5	20.6
" " filmoil	—	—
Total concentration of solids	47.5	43
Concentr. of fresh coal	40	36
Amount of gas	1400	40000
Catchpot	1.0	16.2
Total gas	4000	48000
Temperature change	318 ← 408 296 → 391	188 ← 434 117 → 405
Number of heat exchangers	1+2	3
Average heat. surface	15.3 + 2x7.3	3x188
Velocity through exch.	0.34	0.55
" return	2.26	3.1
K value	100	226

*With heavy inside insulation.



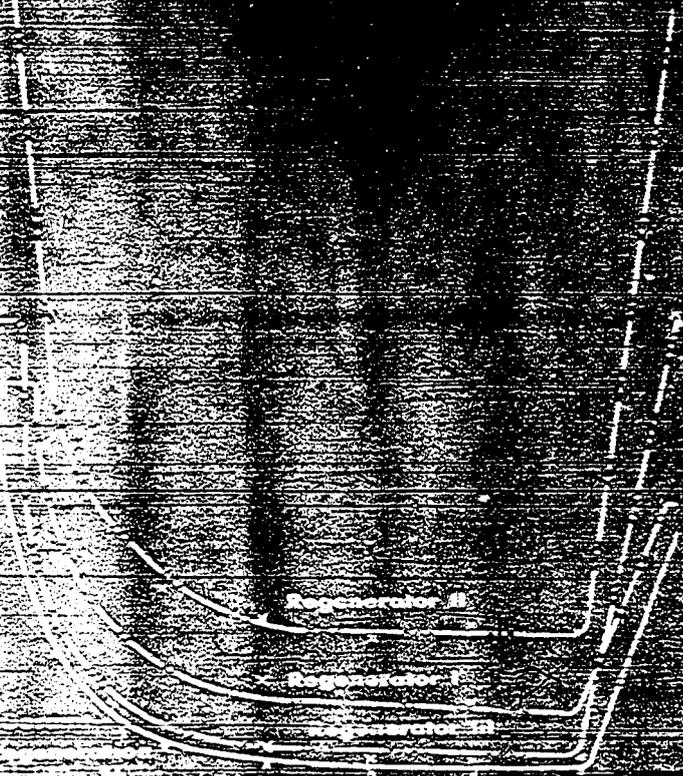
THE HEAT EXCHANGERS

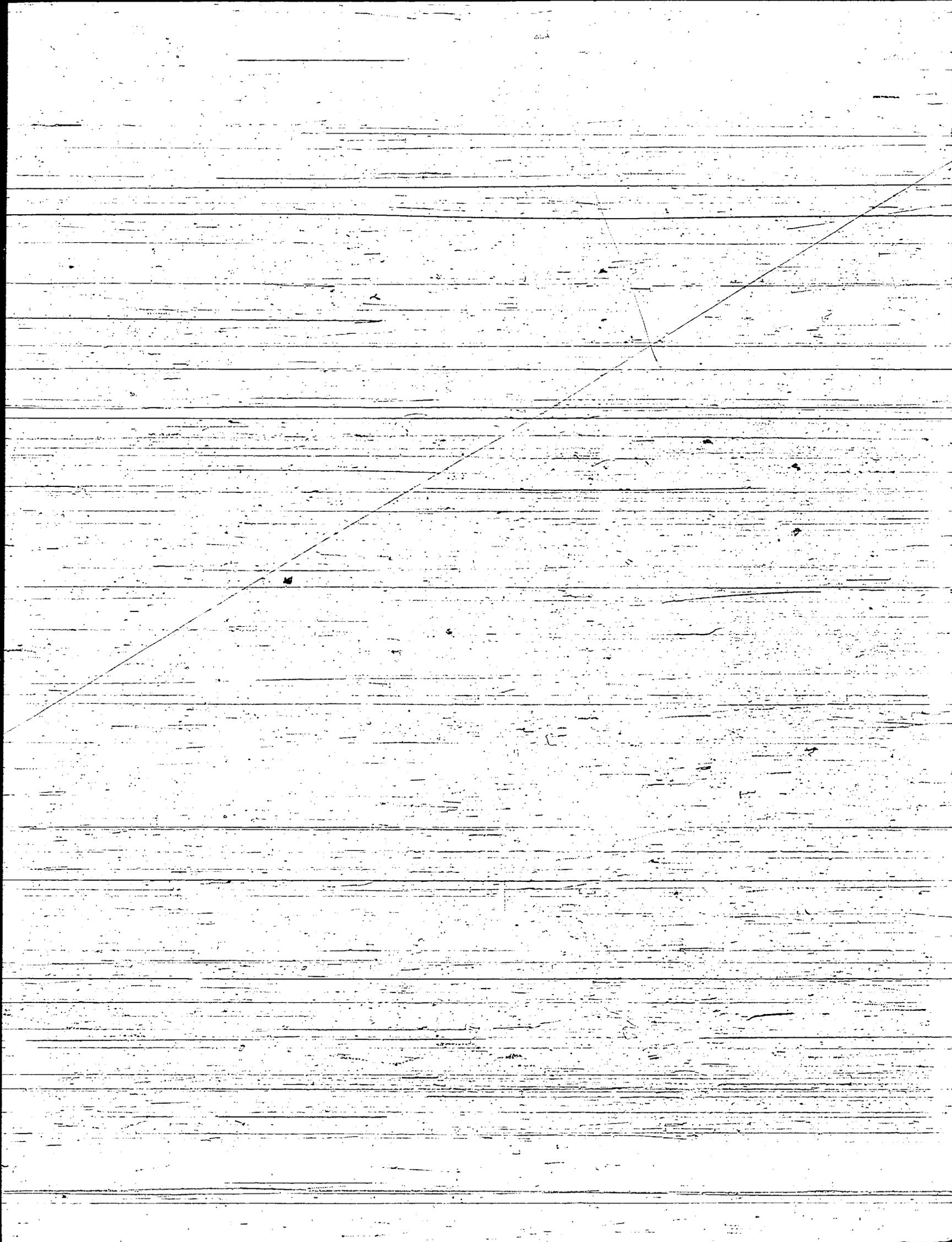
der Regeneration

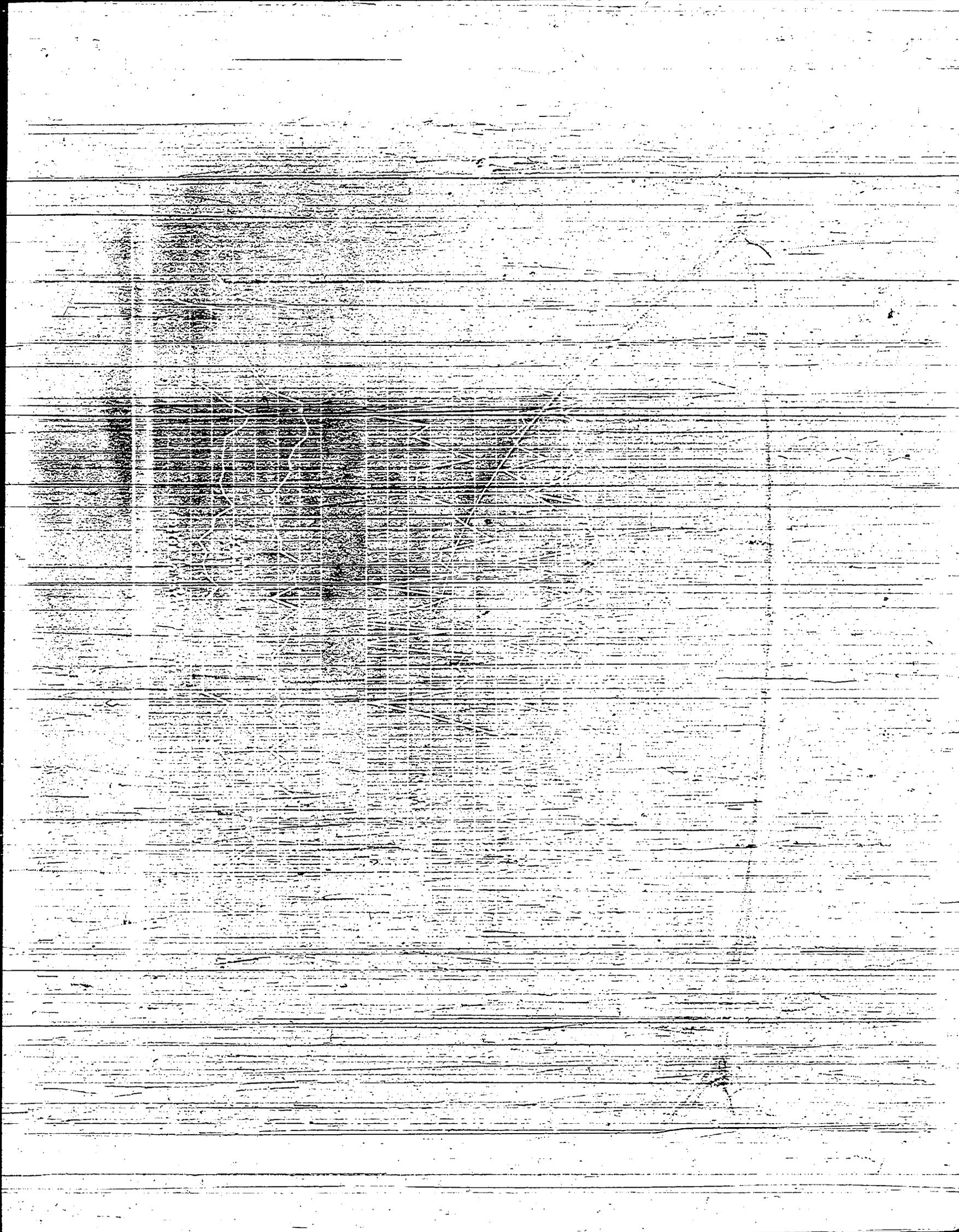
STALL
Kammer 804

23.24.8.41.

Concentration of solids	41%	57%
Konzentration	41%	57%
fresh coal	34%	45%
frischkohle	34%	45%



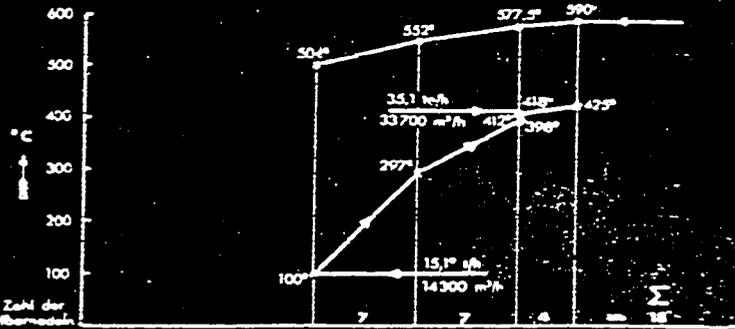
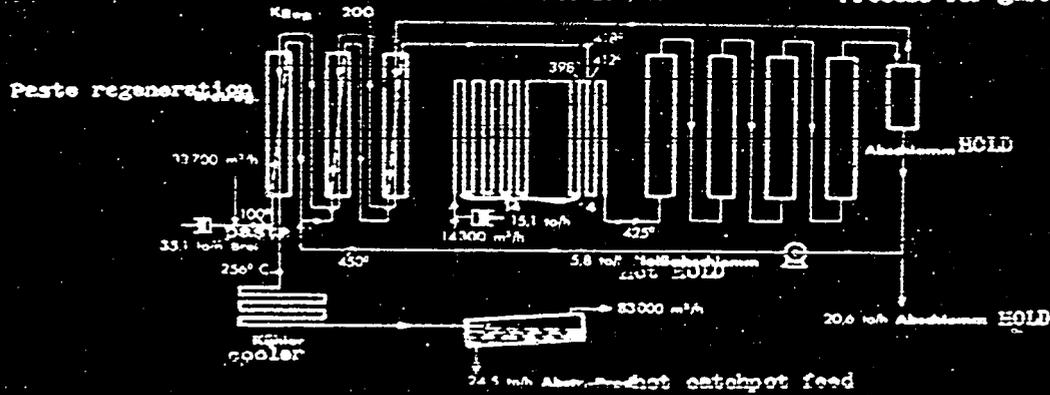




Breaking down of paste into two streams of the same concentration (lower viscosity)
 Zerlegung des Breis in 2 Ströme mit gleicher Konzentration (bei niedrigerer Viskosität)

Fahrweise Bi + H5

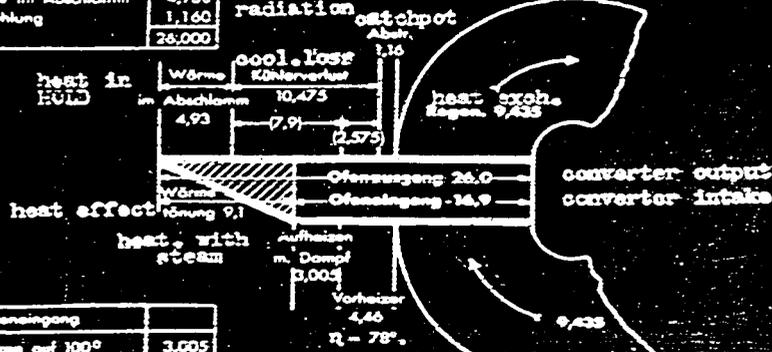
Process for gasoline & fuel oil



No. of hairpins	Zahl der Haarstangen	K	Col/F C m²h	°C	heat transfer	Übers. Wärme Col/h	Δp
7	7	57	3215	2550000	—	—	
7	7	4.4	215	1310000	—	—	
4	4	6.6	165	600000	8	8	
Σ	Σ	—	—	—	4460000	8	

Ofenaustrag	
1. Reg.	9,435
2. Kühnverlust	10,475
3. Wärme im Abstrahlm	4,930
4. Abstrahlung	1,160
	26,000

Converter output
 heat exch.
 cooling loss
 heat in HOLD
 radiation



converter intake
 heat. to 100°
 preheating
 heat exchangers
 heat effect

Ofenaustrag	
1. Aufheizen auf 100°	3,005
2. Vorheizen	4,450
3. Reg.	9,435
4. Wärmeerzeugung	9,100
	26,000

Scale Maßstab: 5 mm 1000000 Col.
 Die Zahlen bedeuten Millionen Col.
 The numbers represent millions of heat.