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YEARBOOK 1939 - AMMONIAKWERK MERSEBURG - LEUNA

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TRANSLATION

AMMONIA PLANT MERSEBURG

ANNUAL REPORT
1939

Table of Contents: Pages

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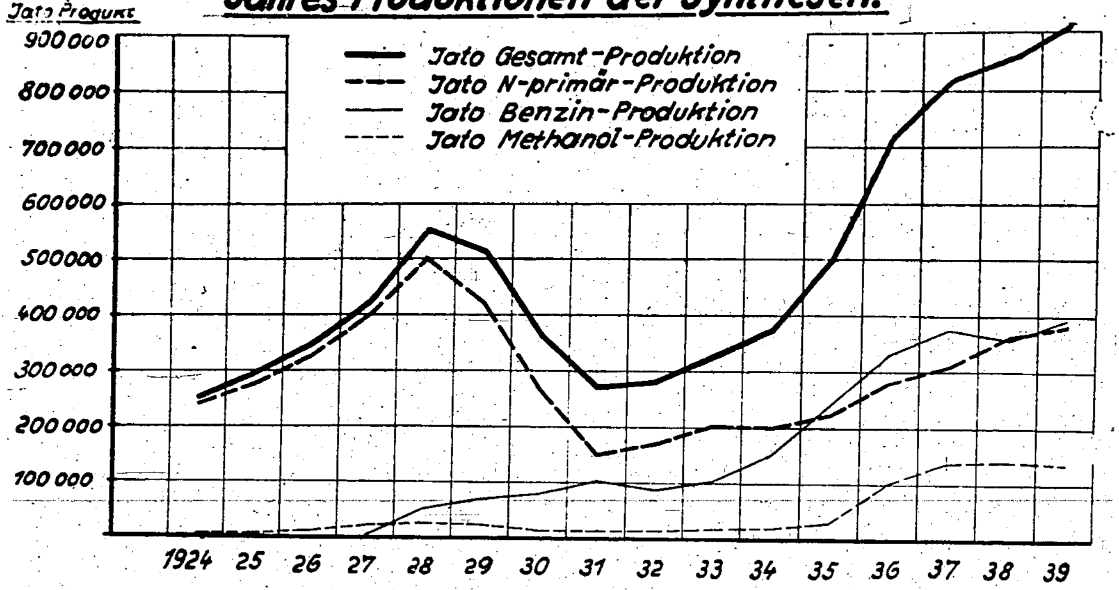
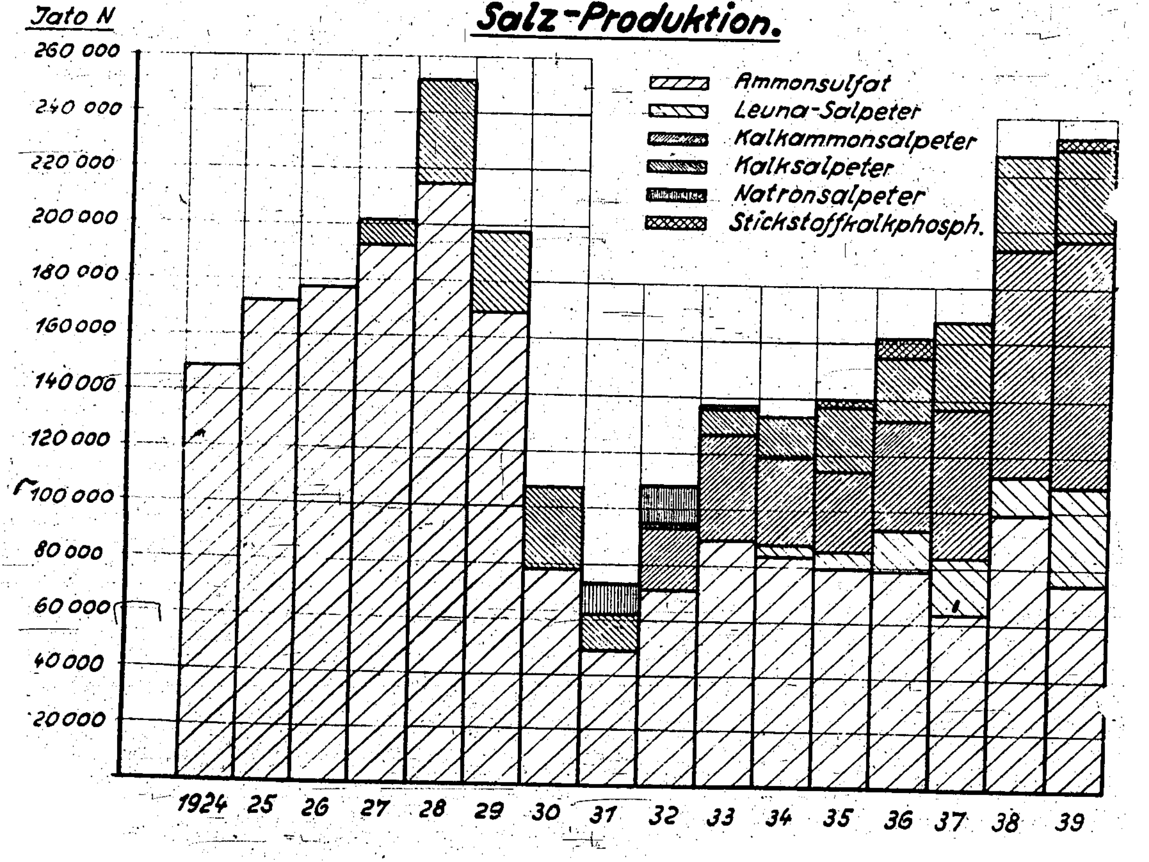
Jahres-Produktionen der Synthesen.



Salz-Produktion.



(Drawing Opposite Page 1)

Yearly Production of the Syntheses

Jato Produkt	Tons of product/year
Jato Gesamt-Produktion	Total production, tons per year
Jato N-primär-Produktion	Production of primary N products, tons per year
Jato Benzin-Produktion	Gasoline production, tons per year
Jato Methanol-Produktion	Methanol production, tons per year

Salt Production

Jato N	N tons/year
Ammonsulfat	Ammonium sulfate
Leuna-Salpeter	Leuna saltpeter
Kalkammonsalpeter	Calcium ammonium saltpeter
Kalksalpeter	Calcium saltpeter
Natronsalpeter	Sodium saltpeter
Stickstoffkalkphosph.	Nitrogen calcium phosphate

AMMONIAKWERK MERSEBURG - ANNUAL REPORT 1939

Short Summary of Plant Operations During 1939

The most important event of far-reaching consequences for our plant was the outbreak of war. Conversion to war production took place without difficulties, thanks to measures long previously planned. The most important conversion concerns the fuel production and the manufacture of aviation gasoline was raised from 80,000 tons per year at the beginning of the year to 150,000 tons per year in April and to 200,000 tons per year at the outbreak of the war. Production of Diesel oil was started at the end of September at the rate of 70,000 tons per year and was later increased to 190,000 tons per year at the expense of automotive gasoline. In spite of this change the total fuel production increased by 9.6% to 393,000 tons of which the portion produced from coal increased to 68.5%.

Regarding alcohol production the resumption of the manufacture of methanol for fuel purposes towards the end of the year as well as the considerable increase in the manufacture of pure methanol from 3200 tons to 17,000 tons should be emphasized. The manufacture of iso-octane which is important for the war economy could be increased from 3300 tons in 1938 to about 6000 tons in 1939 by the elimination of various bottlenecks, and the plant today has a capacity of 8000 tons per year.

It was further important to maintain the highest possible level of nitrogen production. The reasons for this are the necessity to furnish fertilizer for agriculture and to manufacture the war-important nitric acid. For the latter purpose, the manufacture of highly concentrated acid (Hoko acid) was increased to 600 tons per month and it is intended to further increase it to 2500 by the middle of 1940, and to 4000 tons per month by the middle of 1941.

A total of 2392 employees were called in the army including 9 graduate chemists, 272 employees, and 2111 workmen. Eighty-seven percent of the personnel required are deferred but 12% of this number (2700) only until April 1, 1940. In spite of the changeover to a 12-hour shift, it was necessary after the start of the war to hire 1900 men for the plant and 800 for outside companies engaged in important construction work. Housing of employees living at some distance, as well as of the new employees, caused at first considerable difficulty. Seventeen hundred men were placed in quarters in the vicinity of Dürrenberg, Merseburg, Halle and Weissenfels. A camp near Daspig for 1000 men is under construction. Improvement in the traffic system caused a certain amount of relief so that the number of resettled people decreased from 1150 to 200.

Additional food rations were given to 530 workers doing very heavy work, 5980 workers doing heavy work and 12,263 workers on night shift or shifts of extended length, making a total of 18,775 workers = 99.5%. 1033 = to 33% of the employees received additional rations for overtime and night work. At the beginning of the war the command posts and troops of the air raid protection group were brought to full strength. Today there exist only emergency troops besides the air raid protection command and the various plant groups. The volume of completed air raid protection space has been raised to 21,000 m^3 which means that 43% of the maximum number of workers present at the plant can be accommodated. The remainder of the workers who can be released from their work go into basements and slit trenches during an air raid. 95% of the workers have been equipped with gas masks. Blackout procedures have been so improved since the start of the war by the shielding of lamps and darkening of windows with paint or curtains that it was possible to intensify the illumination inside the buildings, (85 volt). The effectiveness of the blackout was tested by observation from the air and was found satisfactory.

The measures prepared and carried out during the last year to increase security in the plant have been put to a full test since the beginning of the war. By recruiting 139 guards from the plant personnel the number of the members of the plant security guard has been increased to 246.

Five 2-cm anti-aircraft cannons and 8 anti-aircraft machine guns have been placed on the high buildings in Works #5. One 2-cm cannon and two anti-aircraft machine guns are placed in the water works Daspig.

The sum of the primary products, ammonia, methanol, higher alcohols, and gasoline increased by 65,000 tons, as compared to the previous year, to a total of 920,000 tons. The increase was possible by the elimination of bottlenecks in the gas plants, including the high pressure plant, and by considerable balancing of the cycles of the three synthetic processes.

The following special events in the various departments must be reported:

1) Low Pressure Department

Mechanization of the operations in the gas plant was continued (installation of Brassert grates and automatic control devices). This resulted at the same time in an increase in the capacity of each generator from 5400 m^3/h to 6500 m^3/h equal to 20%. The change in the water gas production from coke to brown coal should again be taken into consideration because of the insufficient coke supply since the beginning of the war. In accordance with successful experiments it is intended to gasify brown coal hard coke in the Pintsch generator with superheated steam and a small amount of oxygen. Purification (Feinreinigung) of 35,000 m^3/h of Wi-Nullgas (hydrogen) by means of Alkazid served to relieve the burden on the desulphurization process by means of activated charcoal. The total amount of sulphur produced increased from 26,000 to 31,700 tons due to the higher hydrocarbon production. A new boiler for the Claus oven had to be installed. Among the special experiments in the Low Pressure Department should be mentioned the work on hydrocarbon syntheses and the checking of the results of Michael (Ludwigshafen) and Duftschmidt (Oppau), who worked in the same field.

2) High Pressure Department

The purification unit using water under pressure is being enlarged by new construction designed for 70-100,000 m^3/h of raw gas and it is intended to start operations in August 1940. An electric twin compressor for 24,000 m^3/h of gas and two electric standard compressors of 16,000 m^3/h each were installed. The third standard compressor will be ready for operation in March 1940. The rotary compressor first started operation in November after the many difficulties in the elimination of vibrations were overcome. The efficiency of a washer in the CO purification was increased by about 30% by the elimination of constrictions.

In the ammonia plant about half of the feed gas is purified in primary reactors; experiments with a catalyst which had been subjected to an after-treatment with oxygen in an electric furnace resulted in the high conversion of 8-9% accompanied by high efficency of the oven. Among new operations should be mentioned the manufacture of cyclohexanol for adipic acid by hydrogenation of phenols (production about 90 tons per month) and the manufacture of Luran (lactam of aminocaproic acid) from cyclohexanone and hydroxylamine, (production at present 10 tons per month).

3) Salt Manufacture:

By increasing the efficiency of the saltpeter plant to the utilization of the full capacity it was possible to increase the production of nitrate fertilizer from 127,000 tons to 158,000 tons, whereas sulfate production decreased from 99,000 to 74,000 tons. The production of nitrogencalciumphosphate which was started in May had to be discontinued in September because of the requisitioning of crude phosphate. Further expansion of the saltpeter factory by 50 tons per day has been started. By changing the operation of the ammonia nitrate evaporators it was possible to save 35% steam. The manufacture of adipic acid from cyclohexanol by oxidation with 62% nitric acid has been added to the salt plant. While adipic acid is used for the manufacture of a new synthetic fiber, methyladipic acid made by the same process is used in the linoleum manufacture to replace linseed oil.

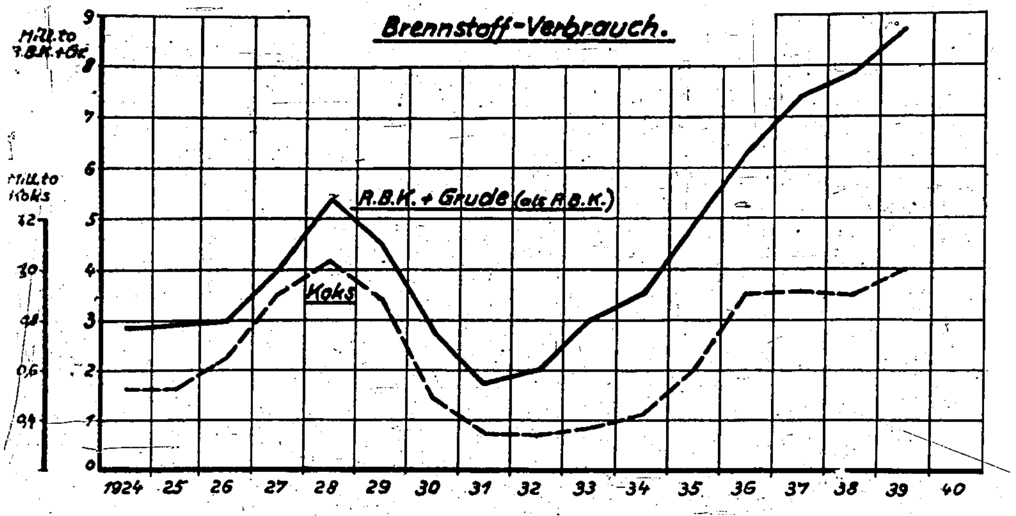
4) Organic Department:

In addition to the above mentioned increase in the production of methanol, isobutyl oil, and isooctane, the larger scale operation of the alcohol syntheses must be mentioned. The following processes were started on a commercial basis: Synthesis of amyl alcohol (lacquer solvent) trimethylolethane (glycerine substitute for the manufacture of alkyd resins), stearylamine (manufacture of waterproofing material), propionaldehyde (solvent). The output of Leuna carboxylic acids

(page 2 contd)

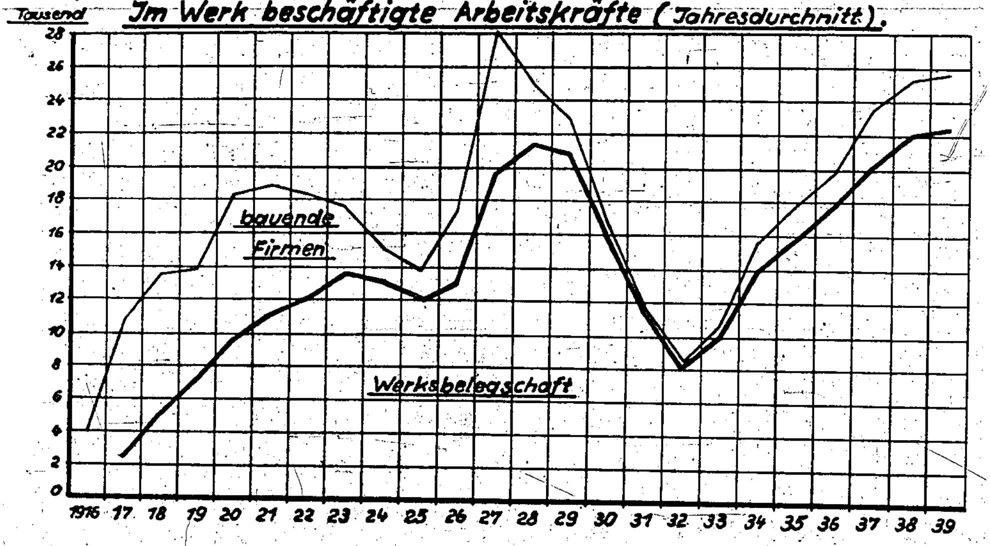
(substitute for naphthenic acids) has almost doubled, and this material is used for the manufacture of driers for the lacquer industry.

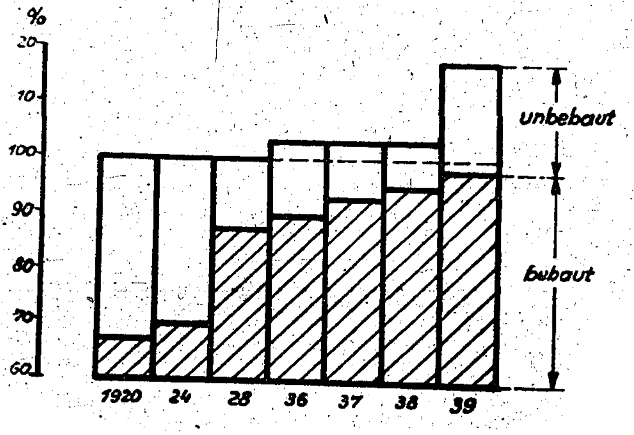
In the hydrocarbon field the considerable increase in the production of lubricating oil SS 906 (from 60 to 250 tons per month) caused by the war economy should be mentioned. In addition to the Klein oven (ethylene production from ethane and oxygen), the Häuber oven (cracking of ethane) was started and the expensive electric arc unit was shut down. The unit for the manufacture of ethylchloride (for tetraethyl lead) started operation and production amounts to 370 tons per year.



Brennstoff-Verbrauch.

Im Werk beschäftigte Arbeitskräfte (Jahresdurchnitt).





Gesamtfläche innerhalb der Werksumzäunung im Jahre 1920 = 2,32 Mill. m² = 100%.

(Drawing opposite page 3)

Fuel Consumption

Mill. to R.B.K+Gr.	- Million tons *RBK + Grude
R.B.K.+Grude (als R.B.K.)	- *RBK + Grude (as RBK)
Mill. to Koks	- Million tons of coke
Koks	- Coke

* RBK = pure brown coal

Number of Workers Employed in the Plant (Yearly Average)

Tausend	- Thousand
bauende Firmen	- Construction companies
Werksbelegschaft	- Plant personnel

unbebaut	- Vacant
bebaut	- Built up

Gesamtfläche innerhalb der Werksumzäunung im Jahre 1920 = 2.32 Mill. m^2 = 100%	Total area within the plant fences in 1920 = 2.32 mill. m^2 = 100%

The <u>manufacture of phenol</u> was increased from 5,000 to 11,700 tons. While up to now only the necessarily yielded phenols from waste water and gasoline refining were recovered, the so-called M-phenols have been extracted with 10% NaOH from middle oil since May of this year. The plant whose capacity at present is 400 tons per month is being expanded to 800 tons per month. Since the beginning of 1939 the mixture of phenols after refining with sulphuric acid and further purification by distillation is separated into its valuable individual components (capacity of the plant 500 tons per month). In the third stage of development which also was started this year, the individual components like phenol and cresols are converted into adipic acid or Luran. Both products are starting materials for new synthetic fibers. In 1939 about 800 tons of phenol was converted to adipic acid and Luran.

5) <u>Hydrogenation Department:</u>

Since the Gerlach unit was stared in March, by far the greatest part of the gaseous hydrocarbons obtained in the hydrogenation can be isolated and further processed. To increase the yield of ethane, the release pressure of the high pressure gasoline recovery plant was increased from 25 to 38 ats and it is now possible to recover about 80,000 tons per year of C_3 and C_4 and 16,000 tons per year of C_2. During 1939 42,800 tons of gas for automobiles and 2800 tons of propane for domestic purposes were delivered. The total amount of ethane produced and part of the propane was used for the manufacture of lubricating oil and ethyl chloride, or delivered in the form of ethylene to Buna for the manufacture of styrene and ethylene oxide. A distillation unit for raw butane (capacity 16,000 tons isobutane) was built for the manufacture of T52. Dewaxing the TTH residue by the propane process was tried out on pilot plant scale. Several difficulties with precipitation and filtration had to be overcome. The recovery of M-phenols from middle oil was successful.

Pilot Plant Work:

Experiments on catalytic cracking of oils at atmospheric and at higher pressure were continued. The hydroforming process for the manufacture of highly knock-resistant gasoline was investigated.

By hydrogenation of oils in the boiling range of spindle oil it was possible to manufacture white oil which up to now was made by sulphuric acid treating of imported spindle oil.

The sulfochlorination of oils was developed from the pilot plant to commercial scale. Mepasinsulfonate which was originally intended for textile purposes obtained increased importance as soap substitute because of the fat shortage caused by the war. The yields of Mepasin are more favorable as compared to the manufacture of fatty acids by oxidation of paraffin, and its cost price is only half of that of the synthetic fatty acids. The design of a plant for 20,000 tons per year of Mepasin was carried out in conjunction with the Organic Department.

For the design of a plant for the manufacture of T52 a number of small and large experiments were carried out. The construction of a unit for 19,000 tons per year of isooctane (T52) was started in the fall. The expansion of the production to 42,000 tons per year by changing to copolymerization or alkylation is intended.

Energies:

The increased steam and power requirements caused by expansion of the plant could be satisfied by increased production in the plant and outside supply of power (up to 20,000 kw.). Four new high pressure boilers were put into operation which increased the high pressure steam production to 560,000 tons per hour and the primary power production to 46,000 kw. At present

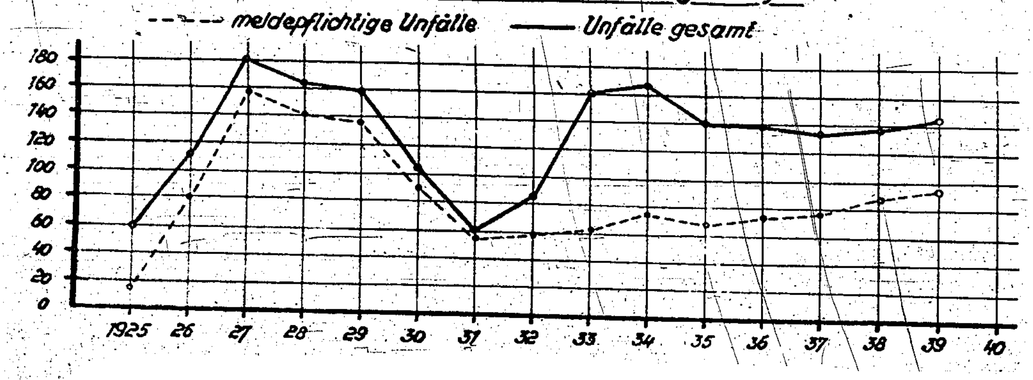
160,000 tons per month of salt coal can be utilized. At the end of next year the entire high pressure steam production shall be converted to the use of salt coal. The problem of utilizing salt coal which represents two-thirds of our coal supply has been furthered by promising experiments on low-temperature carbonization followed by leaching. The salt-free hard coke can be used either for power purposes or water gas production.

Wohnungswesen

	Stand Dez. 1924	Neubauten und andere Zugänge 25/27	28/30	31/33	34/36	1937	1938	1939 fertig	1939 i.Bau	Stand am 31.12.39	Ge-plan 194
1) **Werkseigene Wohnungen:**											
Leuna, Spergau usw.	1014	219	64	12	14	18	12	12	4	1369	6
Dürrenberg	–	–	–	522	–	–	–	–	–	522	
	1014	219	64	534	14	18	12	12	4	1891	
2) **Über Baugesellschaften bezuschußte Wohnungen:**											
Leuna, Dürrenbg., Mersebg.	20	209	1295	166	20	–	–	60	–	1770	350
Halle (S)	–	21	225	–	278	373	–	56	156	1109	204
Weißenfels, Naumburg	12	18	162	–	138	–	–	54	–	384	
	32	248	1682	166	436	373	–	170	156	3263	
3) **Darlehnswohnungen:**	3	38	157	1	143	14	5	7	–	368	
4) **Kleinsiedlungen:**											
Dürrenberg-Goddula	–	–	–	–	51	64	–	81	–	196	
Weißenfels	–	–	–	–	78	63	–	–	–	141	
Ammendorf	–	–	–	–	98	74	–	–	–	172	
Großkorbetha	–	–	–	–	–	–	–	–	104	104	148
	–	–	–	–	227	201	–	81	104	613	
5) **Niedersachswerfen:**											
Werkswohnungen	32	–	–	–2	–2	–	1	–	–	29	
Kleinsiedlungen	–	–	–	–	–	–	–	10	12	22	16
	32	–	–	–2	–2	–	1	10	12	51	
Gesamt:	1081	505	1903	699	818	606	18	280	276	6186	724
Stand 31.12.1927		1586									
" 31.12.1930			3489								
" 31.12.1933				4188							
" 31.12.1936					5006						
" 31.12.1937						5612					
" 31.12.1938							5630				
" 31.12.1939								5910			
Auf 1000 Mann eff. Gefolgschaft entf. Wohnungen:	87	72	252	373	256	266	252	251			

Die Abweichungen gegenüber dem Jahresbericht 1938 sind durch neu eingeführte Grundsätz für die Wohnungsstatistik begründet: Ledigenwohnungen gemäß Vereinbarung mit Ludwigsha abgesetzt, dadurch Zahl der Darlehnswohnungen erhöht; Wohnungen mit Penka-Darlehn mitg zählt.

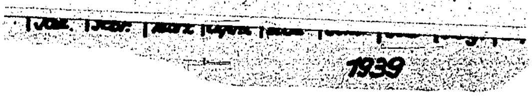
Unfälle je 1000 Mann Arbeiter-Gefolgschaft.



(Drawing opposite page 4)

Accidents per 1000 Workers

meldepflichtige Unfalle ——	Accidents subject to report
Unfalle gesamt	Total accidents



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Power Gas Supply:

Increasing quantities of recycle gas are being furnished from the hydrogenation unit and this gas contains a higher specific hydrogen content so that the addition of considerable quantities of coke gas (up to 45,000 m^3/h) becomes necessary. The problem to produce a Winkler fuel gas poor in hydrogen using Grude coke is still urgent.

Raw Material Supply:

	1938	1939	Increase
Consumption of crude brown coal and Grude coke (million tons)	7.7	8.7	13%
Consumption of coke (million tons)	0.9	1.0	11%

The supply of raw brown coal was very difficult during this winter because of blackout, 12-hour shifts, irregular delivery of other mass goods (red ore, coke) and, at the same time, increased requirements. Improvements in the transportation picture are urgently needed.

Because of the lack of freight cars of the Reichsbahn (State railroad) which became worse after the start of the war insufficient quantities of fresh coke were delivered. The difference in the quantities supplied had to be made up from the stockpile and the reserve sank from 122,000 tons in May to 58,000 tons.

Iron and Steel:

For new construction a total of 30,487 tons of iron and steel were allocated during this year.

In the current year the following plants were either built or put into operation with our participation: Brabag Works Zeitz-Tröglitz, the Italian hydrogenation plants Bari and Livorno, the Lützkendorf plant of Wintershall, the Wesseling unit of the Union Rhein, Braunkohlenkraftstoff, the hydrogenation works Stettin-Pölitz, and the hydrogenation plant at Brüx.

(Page 4 contd)

The Alkazid process will be used from now on not only in the hydrogenation plants but also for the recovery of sulphur from low-temperature carbonization gases.

Contracts:

The specifications covering the order for 4,000 tons per year of SS 906 were fixed with the Ministry for Aircraft. Contracts for T52 are under preparation. A supply contract for Kogasin II for the manufacture of Mepasin is being worked out with Brabag-Schwarzheide and Wintershall-Lützkendorf. The C.I.A. contracts have ceased to function with the beginning of the war and the same is true for the contracts with the I.C.I. and Norsk Hydro.

Twenty ha. of ground have been purchased at the south end of the plant. The expansion of the southern part of the plant has been started in August of this year and is intended for the production of Mepasin and T52.

Housing:

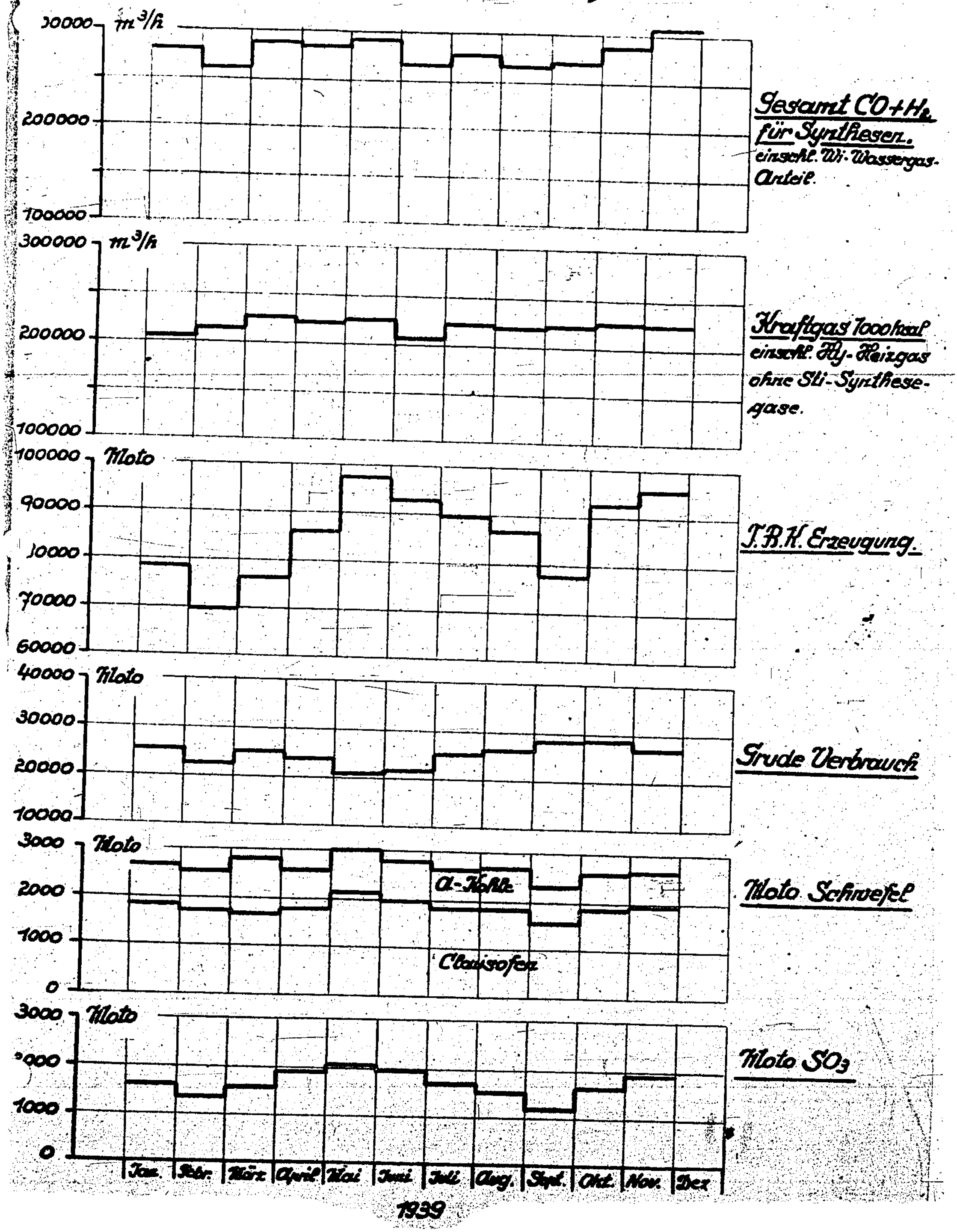
Ten one-family houses have been finished in the housing project. Four are under construction and six more are planned. Sixty apartments in Merseburg, 56 in Halle, 54 in Naumburg have been finished and 156 more in Halle are under construction; all are being subsidized. 150 subsidized apartments are planned for Dürrenberg.

Accidents:

3000 accidents occurred during 1939 of which 1910 were subject to report; these include 8 fatal accidents in the plant and two fatal accidents on the way to work. Whereas the number of accidents subject to report based on 1000 workers increased from 79.3 in 1938 to 97.4, a decrease is to be reported taking into consideration the longer working time (from 3.22 to 3.15 per 100,000 working hours).

Produktionen des Niederdrucks im Jahre 1939.

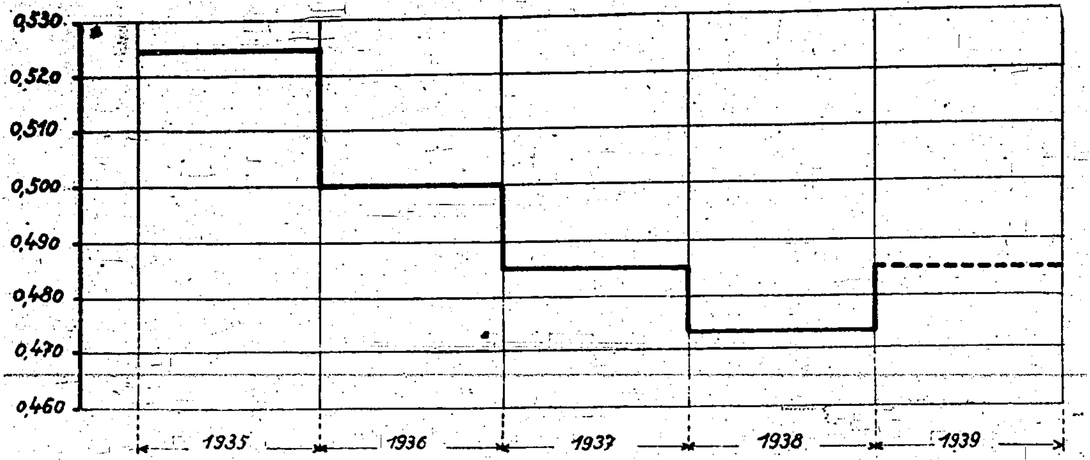
(und Grude Verbrauch)



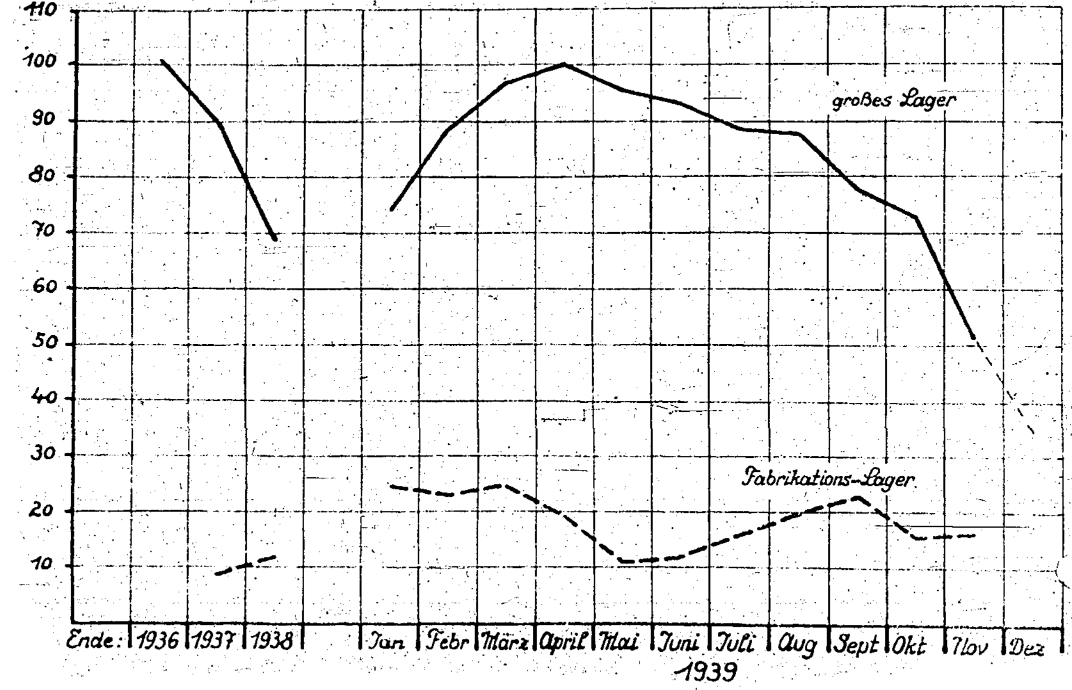
Production of the Low Pressure Department in 1939
(and Grude consumption)

Gesamt CO/H2 für Synthesen. einschl. Wi-Wassergas-Anteil	-	Total CO/H2 for syntheses including Winkler water gas
Kraftgas 1000 kcal einschl. Hy-Heizgas ohne Sti-Synthesegase	-	Power gas 1000 kcal including fuel gas from the hydrogenation but exclusive nitrogen synthesis gases
Moto	-	Tons per month
T.B.K. Erzeugung	-	TBK manufacture
Grude Verbrauch	-	Grude consumption
a-Kohle	-	Activated charcoal
Moto Schwefel	-	Tons of sulphur per month
Clausofen	-	Claus oven
Moto SO_3	-	Tons of SO_3 per month

Gasfabrik Me 1



kg Reinkoks/m³ CO+H₂



Kokslager

(Drawing opposite page 6)

Gas Plant Me 1

kg pure coke/m^3 CO/H_2

Coke Stockpile

coke, in thousand tons

large stockpile

plant stockpile

LOW PRESSURE OPERATIONS

Gas Plant Me 1

In the course of 1939 the last available free space was used for the erection of generators so that now a total of 50 generators are available.

Two generators have been equipped with new Pintsch-Brassert grates, and these have proven satisfactory during six and two months of operation respectively. Sixteen more of these grates have been ordered and shall be installed in the course of the next year. The generators are at the same time being equipped with less heavy linings so that their inner diameter is increased by 20 cm. This increase permits an increase of the hourly capacity by about 13%.

The boiler feed water used for cooling the Brassert generators is now returned to the boilerhouse after it has been heated by about 30°C. in the water jacket of the generators.

Work on the automatic control of the unit has continued. Six generators have been equipped with new simplified control devices (Leuna system) two of which are fully automatic. The installation of these control devices made it possible to operate the generators with a gas cycle of 3 minutes instead of 4 minutes as practiced up to now, and to increase the hourly efficiency without sacrificing the favorable coke consumption. A control apparatus supplied by Pintsch has been in operation since about a year without serious difficulty. Three more of these devices will be delivered during the next year.

Further experience has been collected on sludge bucket conveyors with the result that the installation of further conveyors can be accelerated. Some important parts must, however, be made from special materials (chromium steel, e.g., V17F, or V5M), since wrought iron is subject to corrosion by the acidic recycle cooling water from the gas plant.

(Page 6 continued)

The specific coke consumption in the year 1939 (485 g. pure coke/m^3 $CO \neq H_2$) has increased somethat as compared to 1938 (477 g. pure coke/m^3 $CO \neq H_2$). The reasons for this lie in the utilization of larger amounts of coke from the stock pile and in the processing of very many and different grades of coke. The following mines delivered coke: Auguste Victoria, Centrum, Morgensonne, Central-Kokerei, Gneisenau, Robert Müser, Sachsen, Recklinghausen, Friedrich Heinrich, Ewald Fortsetzung, Monopol, Radbod and Magdeburg.

Approximately since the beginning of the war the supply of coke has been insufficient (60 to 70% of requirement) because of the shortage of freight cars. The big coke stockpile therefore had to be used continuously.

If the present difficulties should continue the stockpile will be completely exhausted by the beginning of 1940. Efforts to improve the situation are being continued from all sides and supply from the Magdeburg coke supply was started on October 24, 1939.

Introduction of the 12-hour shift at the beginning of the war did not lead to the expected saving in manpower in the gas plant. The output of work decreased so that the gas plant today has the same number of workers as before the war at almost equal gas production.

Work Planned for 1940:

Conversion to automatic control shall be continued. Experiments are planned to manufacture water gas from brown coal hard coke by a discontinuous method.

Gas Plant Me 240

Of the ten rotary grate generators (Pintsch) which serve for the production of Sti-water gas (NH_3 synthesis gas), one or two had to be frequently run for the production of power gas (from air and CO_2) which was needed for the compressors. All ten rotary grate generators are now equipped for either the manufacture of water gas or power gas.

Until the middle of October, 3 tap generators (from a total of six) were used for the gasification of crude slag (from water gas manufacture) by means of oxygen and steam. By the middle of October one of these generators had to be converted to power gas for the compressors because of an insufficient supply of hydrogenation recycle gas due to increased gas deliveries for automotive purposes. The quantity of oxygen (5500 m^3/hour) available for producing water gas was used in two generators from this time on. For the same reason another generator had to be changed to the production of power gas by the middle of November.

The water gas generator with Brassert grate has operated without major difficulties from July 26, 1938 to January 13, 1939. During the overhauling period the generator has been equipped with a lining of Tertial bricks as substitution for the cooling ring. After a short operating period the reactor had to be cleaned because of excessive slag formation and stopping of the stirrer. Since the lining at the height of the stirrer was heavily attacked, it appears that satisfactory operation without cooling is not possible. The generator now has a grate with a spacing of 8-10 mm. for the gasification of Deuben hard Grude; when working on coke the spacing is 20 to 30 mm.

The gasification of Deuben hard Grude with oxygen and steam in a tap generator has been carried out successfully in an experiment in August. The water gas obtained had the following composition: 5.9% CO_2, 61.9% CO, 31.4% H_2, 0.7% N_2.

The sulphur content amounted to 12 g/m^3 of inorganic and 0.8 g/m^3 of organic sulphur. 8% sand was added to the wet fuel to obtain a slag of good flow qualities. When operating on raw slag from the water gas manufacture 15 to 18% lime is added.

Experiments on injecting dust into the generators were discontinued because of excessive back pressure and frequent difficulties. It is intended to secure a blower to inject the dust together with the water gas.

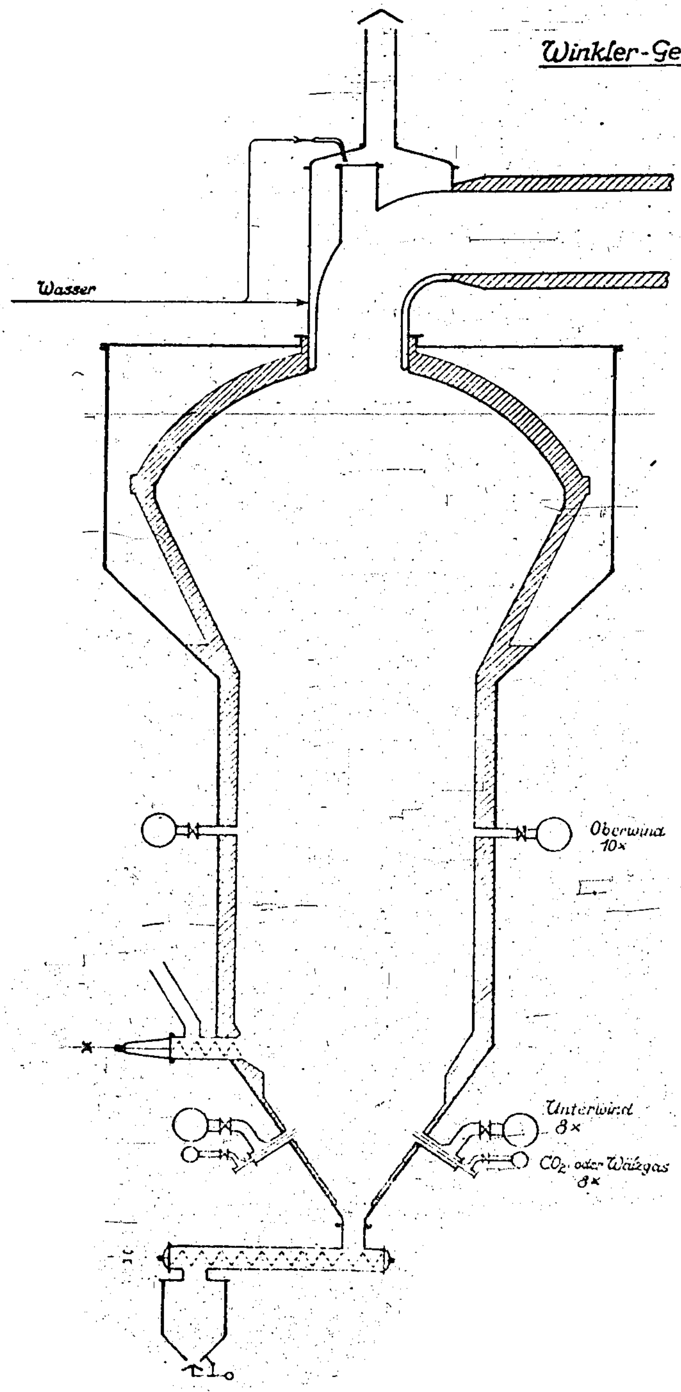
The electric bucket conveyor for slag at the south end of the building has been used since July.

Work Planned for 1940:

Experiments should be made on the manufacture of fuel gas from Deuben hard coke with air and CO_2 in the normal rotary grate generator and in the Brassert generator. The possibilities of the continuous manufacture of water gas from coke (from the low temperature carbonization of brown coal) with superheated steam, oxygen or air, should be investigated.

Braunkohlenvergasung

Winkler-Generator



(Drawing opposite page 8)

Brown Coal Gasification

Winkler-Generator

Oberwind	-	Upper air duct
Unterwind	-	Lower air duct
CO_2 oder Walzgas	-	CO_2 or recycle gas
Wasser	-	Water

Operation of Winkler Generators

During the reported period of operation a rather uniform supply of water gas and power gas was maintained. One generator was used for water gas and two mainly for power gas. No major interruptions occurred.

The waste heat boiler of generator #5 started operation and now all generators are equipped with boilers. This led to an improvement in the water situation because the temperature of the recycle water was considerably lowered.

Generator #1 was again put into operation. It has been converted to a "grateless" generator (see drawing) and does no longer have a dust collector. It was found that the temperature in the coal bed could be kept considerably higher than with other power gas generators. However, this advantage could not be utilized up to now because the temperature is still limited by slag deposits in the generator outlet up to the boiler inlet. Cooling of the gas outlet with water did not completely eliminate this difficulty.

Experiments on gasification of dust from the dust separators were successful; the entire separated dust is blown with CO_2 into the water-gas generators whereby fuel savings of about 5% can be effected.

The experiments for burning the dust under the boilers have been continued; they were interrupted because the draft blowers showed excessive wear. These difficulties would have been avoided if the blowers were placed behind the electric precipitators and this change is to be considered in the design of new boilers.

Experiments to gasify the salt containing Hermine-Henriette coal in the Winkler generators have been unsuccessful and the addition of clay to the coal did not prevent slag formation.

Work Planned for 1940:

Improvement of the power gas by increase of the gasification temperature and use of completely degassed Grude.

Improvements in the water economy by using pumps which pump the water from the receivers directly to the gasification towers. This relieves the ducts and avoids mixing of desulphurized and sulphur containing water.

Changing of the superheater of generator #4; the superheater is placed between the boiler tubes which should result according to previous experience in protection of the boiler tubes.

Sulphur Removal by F-Charcoal Me 284, 285 and 40

Since a high nitrogen production Me 284 is not sufficient, one apparatus in Me 40 was changed from hydrocarbon operation to nitrogen operation and a new mixed gas line to Me 285 was installed. Thereby it is possible to run 1500 nitrogen cycles in Me 40.

Improvements in the plant equipment and minor changes in operation are reported.

Because of the poor efficiency of the primary desulphurization of the T.B.K.-O-Gas by Alkazid (instead of 5 g. up to 12 g. S m/^{3}) the unit was heavily overloaded (over 400,000 m/^{3} of gas including recycle gas) at the beginning of April. Since the fall it has been possible to purify 35,000 m^3 of T.B.K.-O-Gas by means of Alkazid and feed it directly to the hydrogen plant. Relief for the sulphur purification: 1400 cycles.

For the prevention of corrosion the following steps were taken: one absorber was painted with Igelit; the spindles of the gas valves are continously provided with V2A protection.

Experimental Work:

1) The pilot plant experiments for the removal of organic sulphur from the methanol-O-gas by means of M-charcoal were finished. Result: The organic sulphur was removed to a value below 5 mg. The regeneration of M-charcoal is carried out by treatment with hot water and superheated steam. Because of the high cost it is not at present intended to use this process on a commerical scale.

2) In the course of experiments to crack the organic sulphur compounds it was possible to reduce the sulphur content from 250 mg.S to below 10 mg. by conversion into H_2S by means of Grude at 80 to 100°C. Experiments are being continued.

3) It was not possible to completely desulphurize T.B.K. gas (20 g S/m^3) by reaction which H_2S and SO_2 over F-Charcoal. Reaction between H_2S and SO_2 partly takes place before the gases reach the charcoal and this leads to a disturbance in the ratio of $H_2S:SO_2$.

4) Experiments for the desulphurization of air containing H_2S by means of activated charcoal which were carried out for synthetic wool factories were unsuccessful since the charcoal heats up and ignites.

5) Consulting work was done for the Schwefel G. m.b.h.

<u>Work Planned for 1940:</u>

Continuation of the study of possibilities to reduce the organic sulphur content of gases by means of Grude.

Hydrogen Manufacture

The hydrogen plant suffered from a lack of material, machinists and skilled labor so that the planned improvements could not be carried out and the plant was continuously overloaded.

Manufacture of Gas for the Nitrogen Plant:

Of the ten units which were intended for construction during this year only one unit was completed and put into operation.

The temporary catalyst shortage was relieved by sieving the waste and using a material of smaller particle size.

Manufacture of Gas for the Methanol Plant:

After completion of construction work 16 units were available. Since production for the methanol plant decreased during the summer, four units were switched to nitrogen production but returned to their original destination by the end of the year.

Gas Manufacture for Hydrogenation:

The plant was heavily overloaded during the greater part of the year. It also suffered very much from the salt content in the high pressure steam. Since the fall, up to 35,000 m^3/hour of water gas which had been desulphurized with Alkazid was worked up. The H_2S content of this gas is on the average higher than that of the gas treated by the F-charcoal unit. The dust content of this gas also is unsatisfactory (2 to 3 mg of dust/m^3) so that difficulties in the units occurred.

In order to provide for reserves for the hydrogenation, connections were provided which permit to obtain gas from the other cycles.

Experimental Work:

Experiments were carried out for Pölitz in order to find the lowest CO content at which gas conversion takes place over the catalyst without extraneous heating. Gas delivery to Schkopau had to be increased considerably at times necessitating also increase in pressure.

Purification of methanol synthesis gas

(A report on new equipment and repair work in the plant).

Purification of Synthesis Gas

By careful cleaning of a number of caustic coolers which resulted in lower operating temperatures it was possible to effect considerable savings in the consumption of sulphuric acid. Further decrease in the sulphuric acid consumption will be possible by treating with CO_2 the solution which is used for final washing of the gases since in this way the ammonium carbonate is converted into bicarbonate.

By using very impure ammonia water from gas works undesirable quantities of sulphur and phenol were introduced into the pure gas which resulted in damage to the activated charcoal and also affected the high pressure hydrogen purification. To eliminate this difficulty the ammonia containing vapors which were recovered from the gas water were no longer introduced into the atmospheric CO_2 absorbers but condensed to produce aqueous ammonia which was added to the solution supplied to the sulphate plant. In this way difficulties with foaming which previously sometimes occurred in the hydrogen purification unit were considerably reduced which makes it appear likely that this difficulty is connected with the impurities of the gas water.

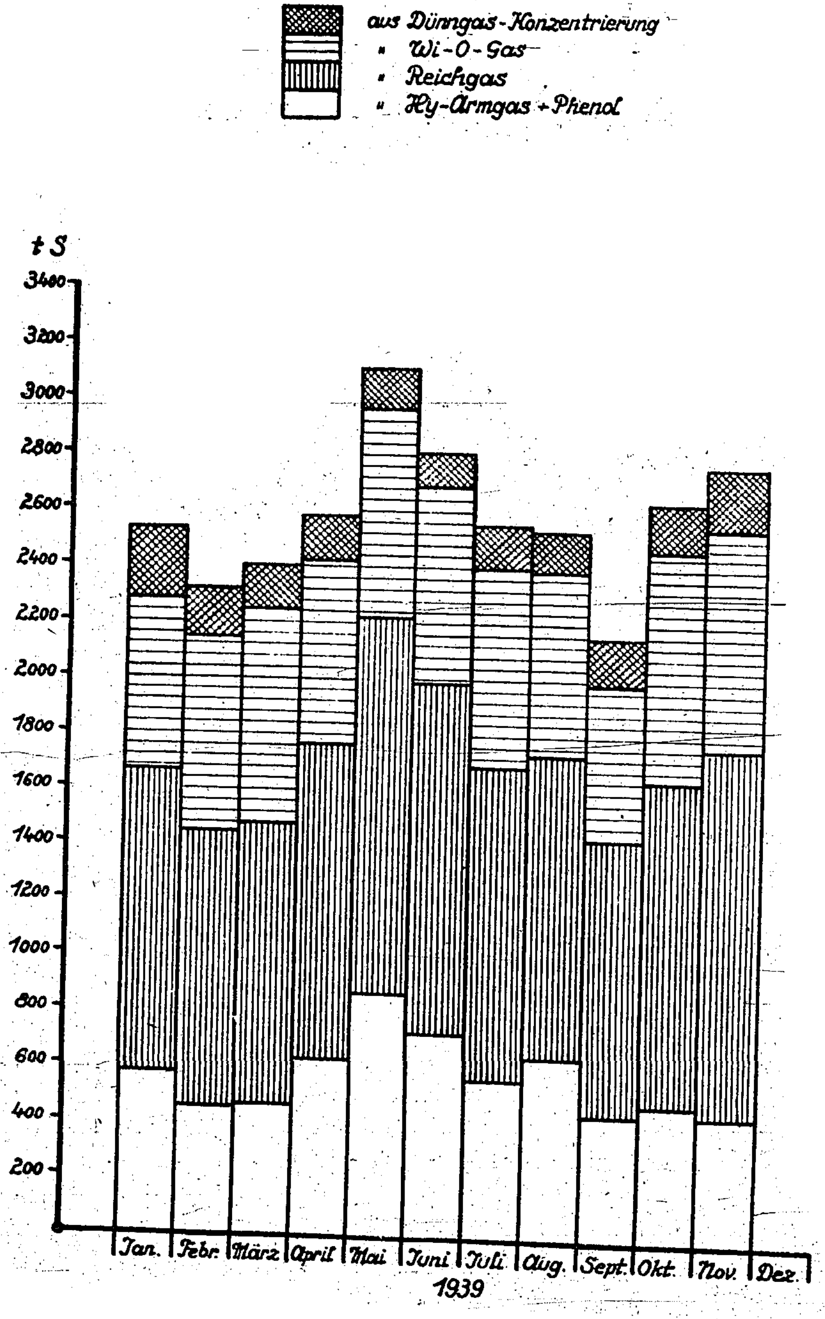
Further experiments on technical scale were made to investigate the possibility to operate the CO_2-absorption with ammonia in the absence of pressure in a cycle process. It was found that the procedure using salt suspensions or solid ammonium carbonate in the regeneration step appears promising even in comparison with the more recent processes now in operation.

Work Planned for 1940:

The cycle process based on the regeneration with salt suspensions or solid ammonium carbonate shall be tested in the suitably redesigned pilot plant to provide basic data for a commercial plant. Technical scale experiments are to be made to study further savings of sulphuric acid by cooling of the caustic.

Alkacid-Anlage

Ausgewaschene t Schwefel



(Drawing opposite page 13)

Alkazid Plant

Recoverd sulfur - tons

aus Dünngas-Konzentrierung	- From the concentration of low-concentration gas
" Wi-O-Gas	- From Winkler-O-gas
" Reichgas	- From high-concentration gas
" Hy-Armgas + Phenol	- From low-concentration gas from the hydrogenation + phenol

Alkazid Plant

Desulphurization of Low-Concentration Gas:

The sulphur content of the low-concentration hydrogenation gases decreased in the course of the year because of the increasing trend to incorporate the hydrocarbon components into the high-concentration hydrogenation gas. This resulted in a decrease of the H_2S content of the gases from the recovery unit. The installation of bubble plate wash towers for the production of recovery gas of higher H_2S concentration was delayed until the end of the year. The concentration of weak H_2S gases presented no difficulties during the entire year.

Purification of High-Concentration Hydrogenation Gas:

The quantity of high-concentration gas processed was further increased so that the unit which was designed for a maximum thru-put of 11,000 m^3/hour was overloaded already in November (thru-put, about 12,000m^3/hour) especially since the operating pressure which should lie at at least 2.3 ats. was mostly down to 1.7 ats. Because of the complete utilization of the high-concentration gas the release of excess gas to power gas after the first stage was discontinued.

A new pipe was laid to supply 80% H_2S for the treatment of middle oil so that the phenol-H_2S can again be removed from the low concentration gas.

Desulphurization of Winkler-Null gas:

The originally expected gas volume of 60,000 m^3/hour reached at times 80,000 m^3/hour with a sulphur content up to 23 g. S/m^3. The increase in the CO_2 content from 20% to 25% and even to 30% had very unfavorable effects because it decreased the concentration of the recovered gas and increased the load on the regenerator. The unsuitable box coolers for the recycle solution were replaced by steel heat exchangers. The steam jet refrigeration plant for 700,000 kcal/hour was put in operation in July. It was found to be very satisfactory mainly because the recycle water was clean and did not foul up the coolers.

(Page 13 contd)

The new Bamag bubble plate washer for final purification was put in operation in August and the specified sulphur content of the gases (max. 150 mg. S/m^3) was reached. Because of failure to obtain the new blower which had been ordered it was not always possible to maintain the design capacity of 35,000 m^3/hour. Furthermore, the removal of sulphur down to 2 g. in the preliminary purification step which is necessary for proper operation of the final purification unit will only be feasible for a thru-put of 80,000 m^3 of gas with high sulphur content in case that a second regeneration column will be available after the installment of the bubble plate washers for low concentration gas. In the final purification stage an iron reboiler was tentatively installed again and will be operated with the addition of arsenic to the absorption liquid.

Experimental Work:

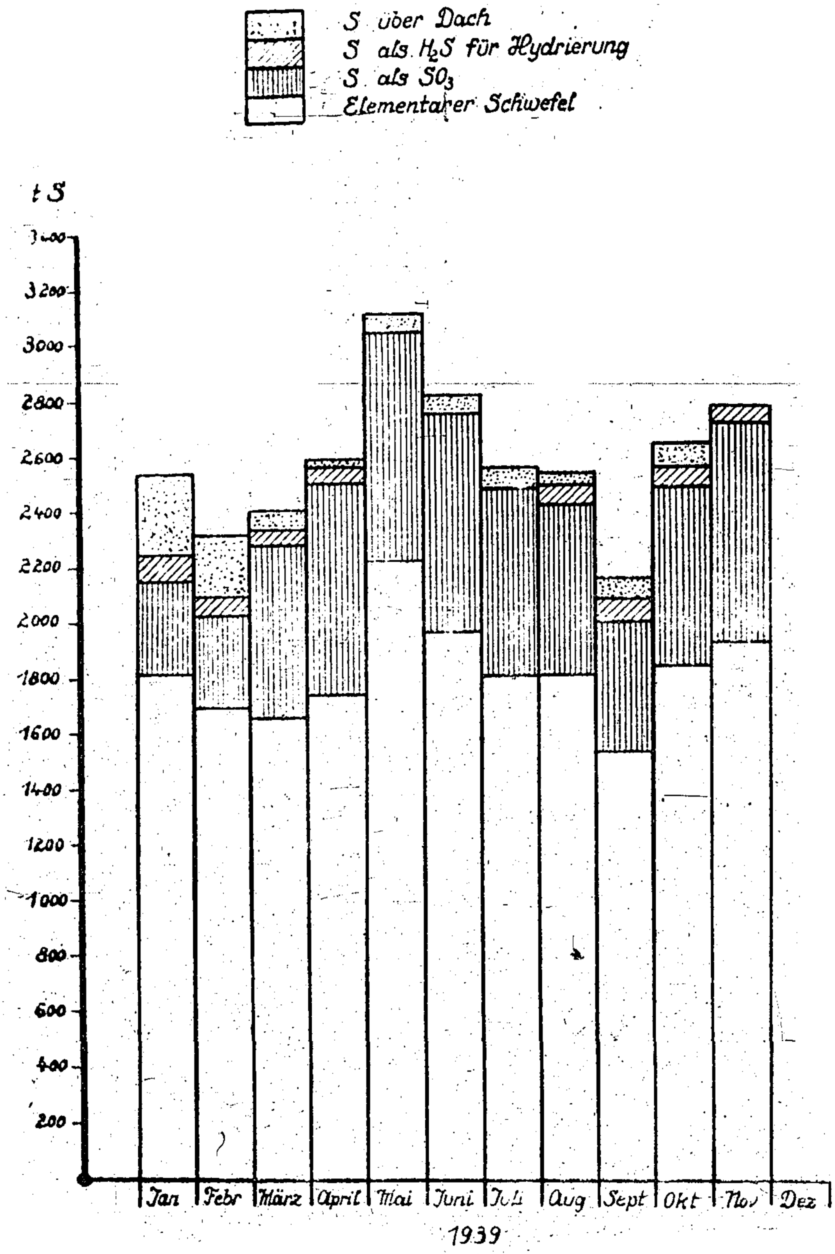
The results obtained in Alkazid and Claus oven experiments with low-temperature carbonization gas at Deuben were used in the design of plants at Espenhain and Deuben. Experiments to use a bubble plate column as regenerator did not show any advantages. The conditions for the concentration of dilute H_2S to highly concentrated H_2S were determined. Extensive experiments for the use of absorption liquids of low aminoacid concentrations were carried out on various gases for the removal of H_2S and CO_2. Corrosion tests were also carried out.

Work Planned for 1940:

Experiments with new absorption liquids. Replacement of filter presses for the removal of coal dust and other impurities by separators.

Clausofen-Betrieb

Ausgebrachte S-Produkte



(Drawing opposite page 14)

Claus Oven Operations

Recovered sulfur - tons

S über Dach	- S out of the stack
S als. H_2S für Hydrierung	- S as H_2S for hydrogenation
S als SO_3	- S as SO_3
Elementarer Schwefel	- Elementary S

Claus Oven Operations

Sulphur production increased considerably because of the increased desulphurization of water gas and the increase in the production of hydrogenation gas. In May the highest production so far obtained was reached with 2226 tons of sulphur. At the beginning of June the 50,000th ton of sulphur from the Claus oven operation was produced.

The Dürr boilers were inspected. The preheaters and superheaters which had been in operation for several years have been renewed. The steam tubes themselves were in good shape.

Frequent repairs were necessary on the electric lines of the electric precipitators because of the effect of moisture and vibrations.

The new air-cooled moulding machine was operated successfully. Normally, the production goes directly from the machine by means of a conveyor into railroad cars.

A German Bauxite from the Vogelsberg region has been found suitable as substitute for the French Bauxite which is no longer available.

Experimental Work:

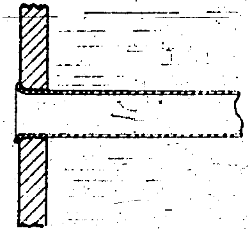
The washing experiments have been continued. Difficulties with sulphur separation in the centrifuge and clogging in the wash tower had still to be overcome. Extensive experiments are continuously carried out to find new catalysts. At Deuben an experimental Claus oven with air cooler for H_2S from low-temperature carbonization gas has been operated with good yields. Air-cooling as substitute for waste heat boilers can only be used for small units.

Work Planned for 1940:

Catalyst experiments on commercial scale. Experiments to increase the yield by installation of a hot zone behind the burner to destroy catalyst poisons. Installation of machinery to eliminate manual labor.

Schwefelsäure-Fabrik

Verbesserung der Wolff-Kessel

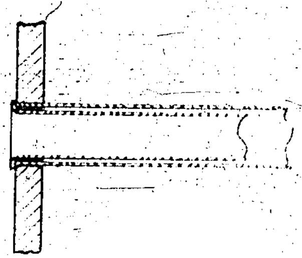


Gewöhnliches Siederohr

Wandtemperatur: 200°C

Schnelle Korrosion dann Kondensation von H_2SO_4 u. NH_4HSO_4

Laufzeit: ca 4 Monate



Siederohr mit Überrohr

Wandtemperatur > 235°

Keine Kondensation, keine Korrosion

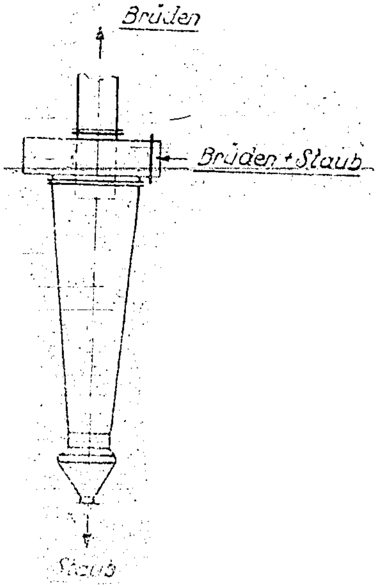
Laufzeit: bisher 14 Monate

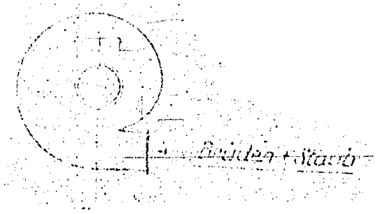
Sulfuric Acid Plant and Recovery of Alkazid Solution

(Report on changes and replacements of equipment of the sulfuric acid and Alkazid regeneration plant.)

Braunkohlentrocknungs-Betriebe

Hartmann-Zyklon



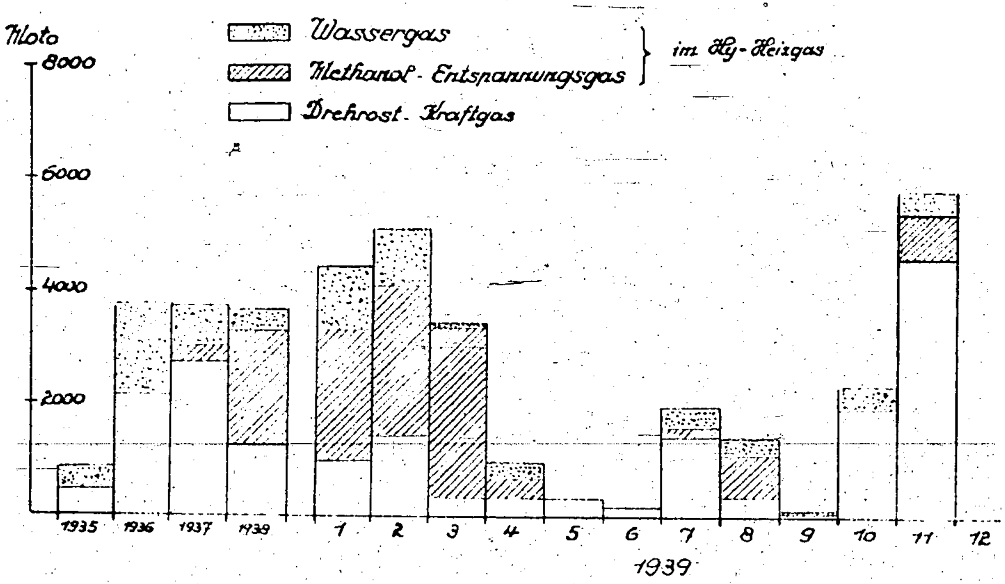


Plants For Drying Of Brown Coal

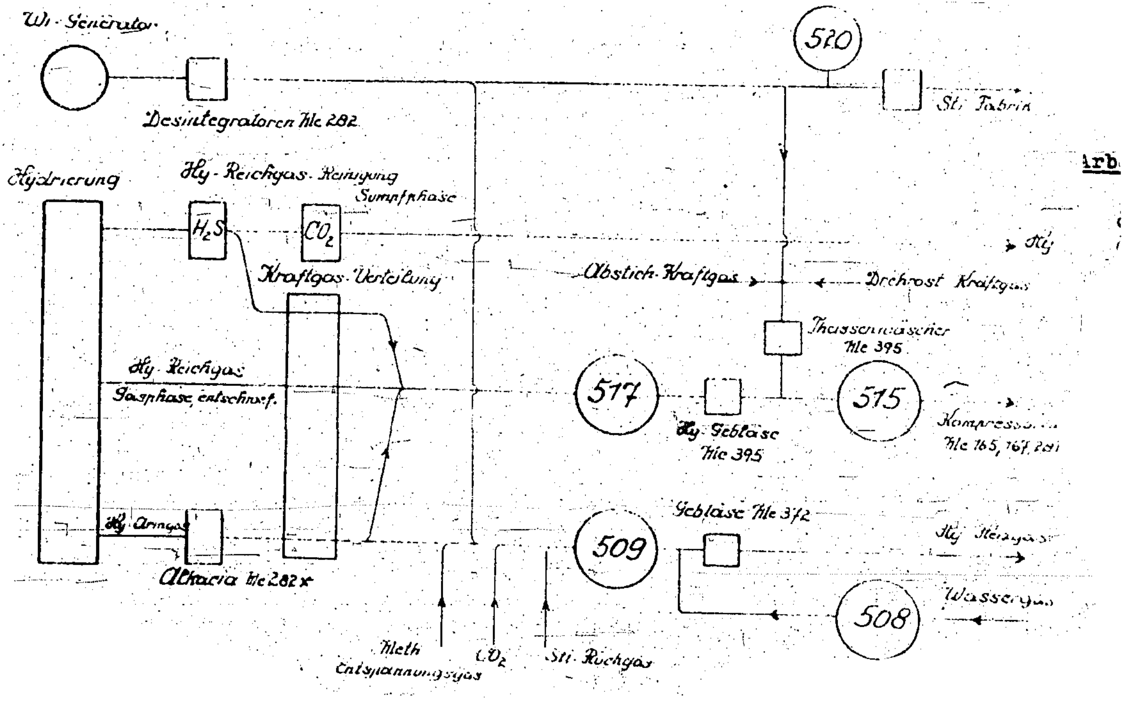
(Difficulties had been encountered when very wet, salt-containing coal and very wet red ore were worked up in the hammer mill; to protect the motors which in this case burned out frequently thermo-control switches were installed. A report on newly installed equipment is given).

Kraftgasverteilung.

Koksverbrauch für Kraftgas.



Schema der Kraftgasverteilung ab Dezember 1938



(Drawing opposite page 17)

DISTRIBUTION OF POWER GAS

Coke Consumption for Power Gas

Moto	Tons/month
Wassergas	Water gas
Methanol-Entspannungsgas	Methanol release gas
Drehrost-Kraftgas	Rotating grid power gas
im Hy-Heizgas	In hydrogenation fuel gas

Scheme of the Power Gas Distribution since December 1938

Wi-Generator	Winkler generator
Desintegratoren	Disintegrators
Sti-Fabrik	Nitrogen plant
Hydrierung	Hydrogenation
Hy-Reichgas-Reinigung Sumpfphase	Purification of high concentration gas from sump phase hydrogenation
Hy	Hydrogenat.
Kraftgas=Verteilung	Power gas distribution
Abstich. Kraftgas	By-pass power gas
Drehrost-Kraftgas	Rotating grid power gas
Theissenwäscher	Theissen washer
Hy-Reichgas Gasphase, entsch.	High concentration gas from gas phase hydrogenation sulfur free
Gebläse	Blower
Kompressoren	Compressors
Armgas	Low concentration gas
Heizgas	Fuel gas
Wassergas	Water gas
Meth	Methanol
Sti-Rüchgas	Nitrogen recycle gas
Entspannungsgas	Release gas

Distribution of Power Gas

The three recycle gases from the hydrogenation, viz. low-concentration gas, high concentration gas from the sump phase and high-concentration gas from the gas phase, are being distributed separately since December 1938. The first two gases are desulphurized with Alkazid whereas the third one is desulphurized in the course of the hydrogenation. By these measures it was possible to utilize the excess of high-concentration gases which are low in hydrogen as power gas for the compressors.

Since the end of October no high-concentration gas from the sump phase has been available for power gas. The entire desulphurized high-concentration gas from the sump phase is now returned to the hydrogenation after Alkazid treatment and is processed for the manufacture of hydrocarbons.

Because of the increased output of the hydrogenation plant since March the recycle gas delivery increased from 120,000 m^3/hour 1000 kcal. on the average during 1938 to 160,000 m^3/hour 1000 kcal. Since the hydrocarbons were removed increasingly from the recycle gases their hydrogen content increased to such an extent that it was not always possible to supply a power gas suitable for gas engines. Therefore, power gas from the rotary grate generators with additional CO_2 was mixed with the recycle gas and since October power gas from the tap generators was added.

The addition of power gas from coke was necessary at times because the quantities of hydrogenation recycle gas and Winkler power gas were insufficient For this reason the entire residual gas from the methanol plant was added to the hydrogenation fuel gas during the first quarter of ~~the~~ year (average January to March 17,000 m^3/hour 1000 kcal.). The addition of water gas to the hydrogenation fuel gas for the purpose of balancing periodic short-time variations in the hydrogenation recycle gas supply amounted on the average during the year to 2200 m^3/hour 1000 kcal.

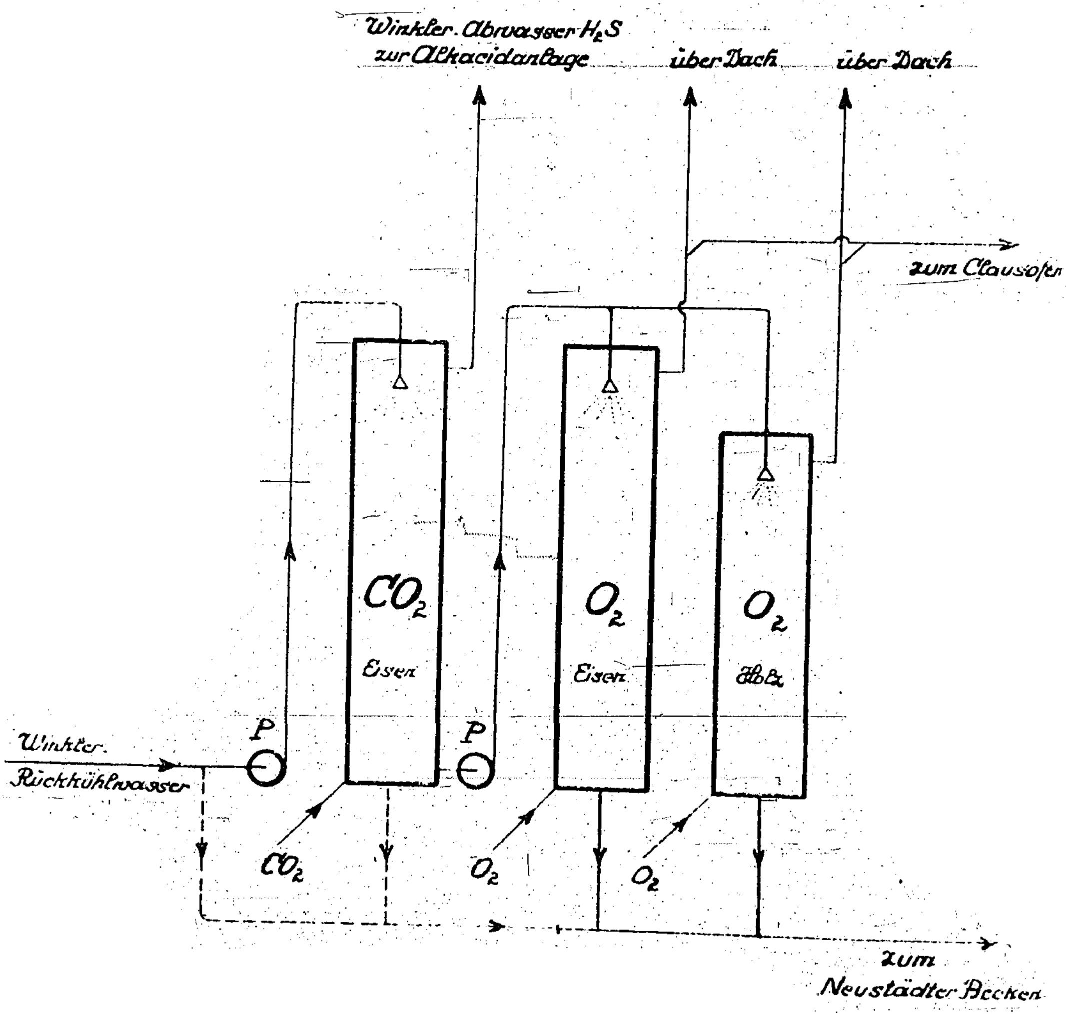
(Page 17 contd)

Work Planned for 1940:

In order to reduce the sulphur content of the hydrogenation fuel gas the Winkler power gas which accounts for about 80% of the sulphur content shall be taken out and replaced by hydrogenation recycle gas. The Alkazid unit shall then desulphurize the hydrogenation recycle gas for fuel purposes to 0.2 g S/m^3 1000 kcal., instead of the 0.5g. obtaining now. This measure results in a decrease in the heating value of the power gas for the compressors but the hydrogen content is not lowered proportionally; it is therefore necessary to add power gas from coke (which is poor in hydrogen) in suitable quantities.

Fabrikabwasser-Klärbetrieb.

Winkler-Rückkühlwasser-Entschwefelung mit Kohlensäure und Sauerstoff.



Treating of Waste Water

(A report on new equipment, mainly pumps. Since desulfurization of the recycle cooling water for the Winkler generators could not be achieved by CO_2 alone an after-treatment with oxygen was started and proved successful. A wooden tower is used for the oxygen treatment since iron towers are too easily corroded.)

Laboratory For The Gasification of Brown Coal Me420

In addition to current control analyses and special requests from the plant which form an essential part of the duties of the laboratory, the following experimental work was carried out:

Desulphurization of Waste Gases from the Claus Oven:

In connection with the negotiations on taking over the Szombathy process numerous wash experiments were carried out on the reaction of H_2S and SO_2 to form sulphur. The raw materials used included synthetic Claus oven waste gases, Winkler gas (Wi-O-gas) which had been subjected to preliminary purification, or raw synthesis gas (Roh-O-gas) with added SO_2. It was possible to obtain conversions up to 90% in Ströder washers as well as in special wash towers provided that special conditions were maintained (pH, etc.). This process is not suited for final purification. The conversion is comparatively low when calculated on the basis of reactor space.

Desulphurization of Winkler by-product water:

It was shown in laboratory experiments that Winkler by-product water can be desulphurized to a large extent by treatment with small amounts of CO_2 and simultaneous application of vacuum (below 200 millimeter Hg); this operation results at the same time in the production of a gas rich in H_2S. In actual plant operation this procedure is replaced by the more simple removal of H_2S with oxygen.

Sulphur Combustion by Means of an Immersion Burner:

A process for the manufacture of pure SO_2 from sulphur and oxygen by means of an immersion burner was worked out in the laboratory and on pilot plant scale. The special advantage of the process consists in the manufacture of SO_2 which is completely free from SO_3 and oxygen, and only contains traces of sublimed sulphur. Jets of a capacity up to 10 m^3 SO_2/hour were developed.

(Page 19 contd.)

Removal of H_2S from Winkler Gas Dust:

The various possibilities for the removal of sulfide sulphur from Winkler gas dust was worked out. It was found that scarcely 50% of the sulfide sulphur can be removed and even this result can only be obtained in a slow process. In order to obtain satisfactory results such a quantity of acid is required that the procedure is not economically feasible.

Experiments on the Reduction of Gypsum:

It was found that gypsum can be reduced at 900°C. in the presence of Grude. This reduction to CaS takes place by reacting the mixture of gypsum and Grude either as powder or in the form of briquettes; the reaction is complete but slow. Sweep gas improves the reaction, oxygen requires naturally more coal whereas the use of steam is detrimental. Gas dust or ash can be used instead of Grude. Coke from bituminous coal or Winkler power gas are too unreactive. The reduction also takes place with CO. H_2S (80%) can easily be prepared from a suspension of CaS in water by treatment with CO_2 and conversion of CaS to $CaCO_3$ is complete.

Work planned for 1940:

The following experimental work is planned: Complete removal of dust from gases; the manufacture of SO_2 by means of an immersion burner in a pilot plant.

Laboratory for Low Pressure Work

It was necessary to carry out extensive investigations in the Alkazid field to obtain design data for new projects. The solutions for almost all new plants had to be controlled. In practically all cases in which difficulties with the solution occurred this could be traced to the loss of free alkali by the formation of thiocyanate, thiosulphate, sulphate or complex cyanides. The solution Japan III, however, was an exception. In this case the decrease in the absorption efficiency was actually due to splitting off nitrogen from the aminoacids by oxidation. Experiments pertaining to this phenomenon which has first been observed in Japan are still under way.

New methods for the determination of small amounts of arsenic (corrosion preventive) in Alkazid solution as well as for the determination of HCN in gases containing H_2S were worked out.

A number of new solutions were tested for their use in the Alkazid process. Certain solutions which contained amines (Girdler) instead of alkali seem to be easier to regenerate at equal absorption efficiency. Solutions in which up to four-fifths of the Alkazid has been replaced by potash were found to be equally well suited for the removal of CO_2 from gases as regular Alkazid solutions. The same result was found in the laboratory for Dik solutions extended with potash. The observation concerning the M-solution of low aminoacid concentration was confirmed by experiments in the plant. With respect to the dilute Dik solution the plant experiments led to different results.

Wash tests with xylidine (Sulfidin process) for the removal of SO_2 from gases have been made and good results (2 g. SO_2/m^3) at higher solution throughput (about 100 g. SO_2/liter solution) were obtained by the use of bubble plate washers. Tests are under way to investigate the suitability of ammonium sulfite-bisulfite for the removal of SO_2.

(Page 20 Contd)

Much work was done on the utilization of salt coal. Experiments to leach the raw coal indicated that only the portion which is equivalent to the chlorine content of the raw coal can be extracted but that the remaining portion is insoluble. Experiments with solvents show that the alkali is predominantly bound to the humins. It was found that the sodium which is present as salt of humic acid can be converted into soda by carbonizing the raw coal and this procedure renders the alkali water soluble so that it can be washed out. Numerous small scale and pilot plant tests have been carried out on the removal of alkali from salt coal by carbonization and leaching. In order to convert the alkali into an extractable form it seems to be necessary not only to carbonize the salt coal but also to gasify it to some extent. Experiments on this subject have not yet been completed. Highly concentrated salt solutions were obtained in the leaching process using several extraction towers in series; the salt content of these solutions consisted to about 80% of soda. According to these experiments 100 tons of Grude yield about 3 tons of soda.

Special Experiments In The Low Pressure Plants During 1939

Hydrocarbon synthesis:

Investigations on the manufacture of hydrocarbons by synthesis at atmospheric pressure have been continued on a broad basis.

The laboratory tests furnished valuable information on the effect of pressure, composition of the synthesis gas and other variables on the course of the synthesis and the character of the products, especially with regard to the manufacture of olefinic materials over iron and cobalt catalysts.

Pilot plant experiments duplicating as much as possible actual plant conditions confirmed the improvements in the Fischer gasoline synthesis which had been previously found advantageous at Leuna. It was found that the yield of gasoline produced per cubic meter of catalyst space and the lifetime of the catalyst can be doubled, as compared to the operation of the regular Fisher units, if the number of stages is raised from 2 to 4 and the gas velocity and the width of the catalyst bed is also increased. It was further found that conversion of the gas for the same catalyst space can be increased by 40% by recycling the hydrocarbon rich exit gas at increased gas velocity. This kind of operation can be carried out with only slight changes in the Fischer plants which are working at present. The pilot plant work furnished definite data for economic calculations.

Towards the end of the year the work was directed towards the synthesis of paraffin wax and olefins.

Gasification Experiments:

To obtain data for the nitrogen project in Val d'arno, Italian brown coal was gasified with a steam-air mixture preheated to 650°C for the manufacture of synthesis gas.

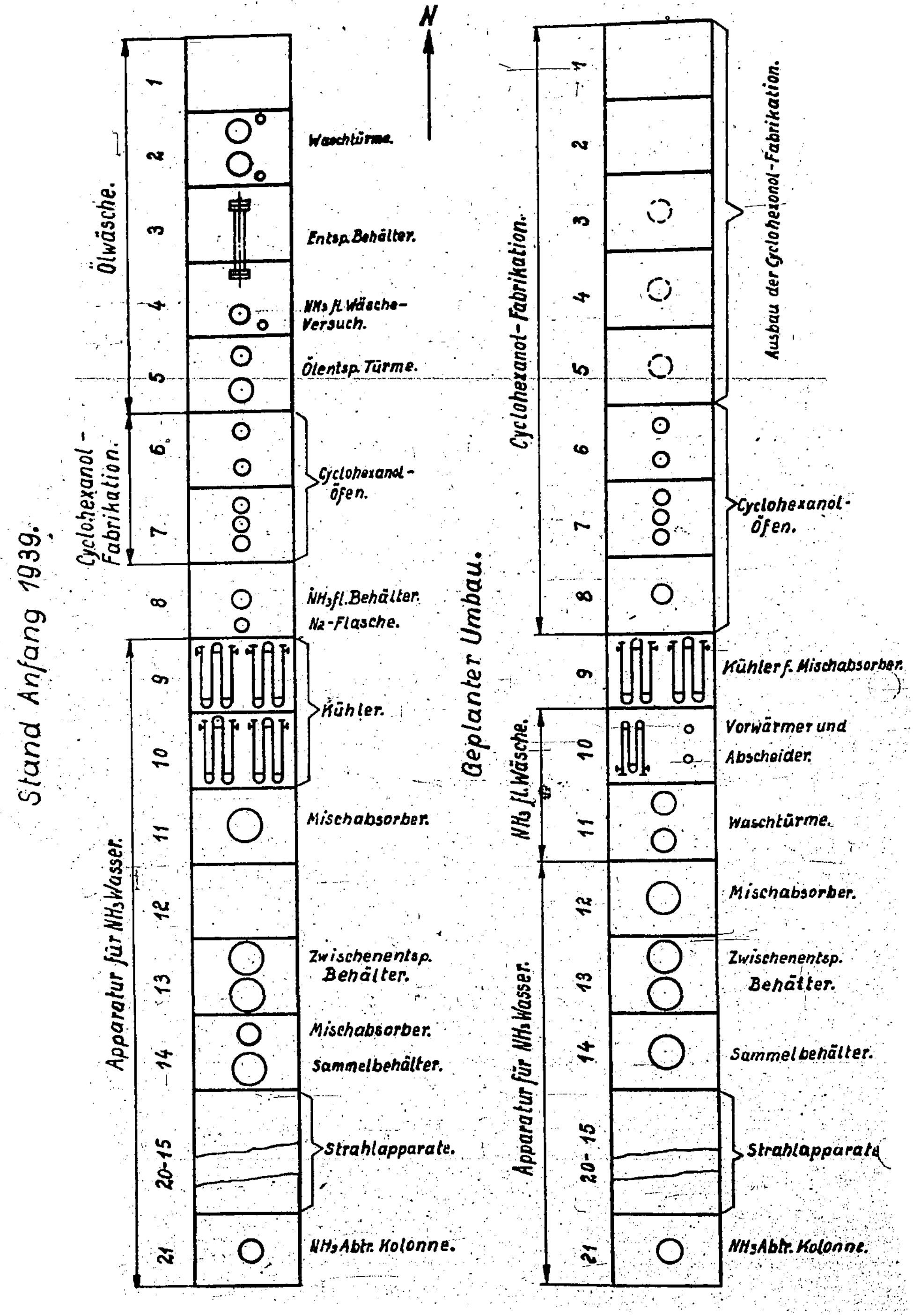
Previous experiments have shown that Deuben hard Grude can only be gasified slowly with steam of 900°C but further experiments show that this Grude can be gasified by a steam-air mixture preheated to 650°C and thereby furnish synthesis gas with good efficiency. To obtain data on the gas composition and material consumption for the steam-oxygen gasification of brown coal Grude the latter was gasified with steam-oxygen mixtures with varying oxygen content and increasing preheat. The tables and curves obtained show the trend from pure steam gasification to gasification with a steam-oxygen mixture containing a high percentage of steam.

Removal of Organic Sulphur:

For the hydrocarbon synthesis methanol gas containing 220 mg. organic S/m3 was purified to 0.0 mg S by means of Pattenhausengrude.

Ammoniakfabrik Bau 11.

Apparategerüst.



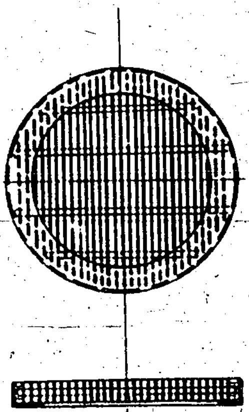
Ammonia Plant

(A report on changes in the operations and the installment of new quipment)

Wasserstoff-Reinigung

Kupferlaugewascher

Beseitigung von Querschnittsverengungen

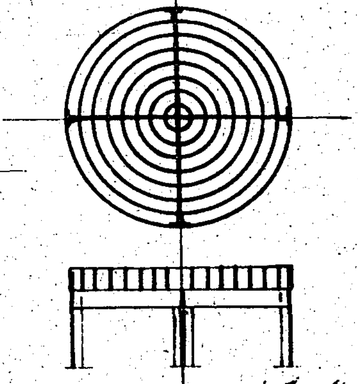


Bodenrost bottom support

alte Ausführung old design

bis 70% Querschnittsverengung

up to 70% construction

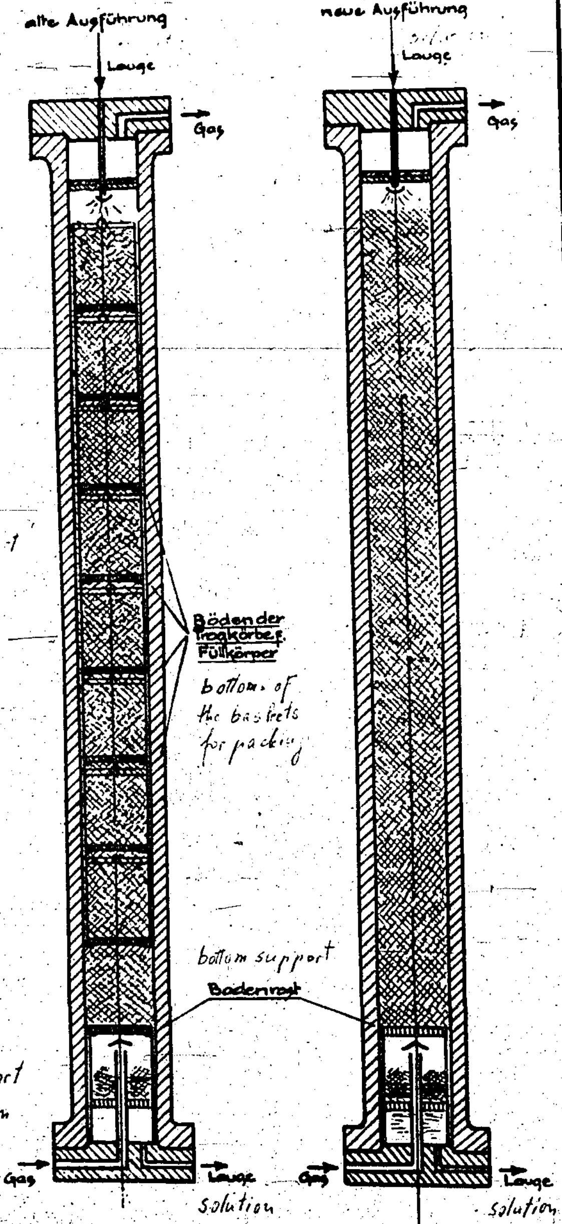


Bodenrost bottom support

neue Ausführung new design

~15% Querschnittsverengung

~15% constrictions



(Page 23)

Hydrogen Purification

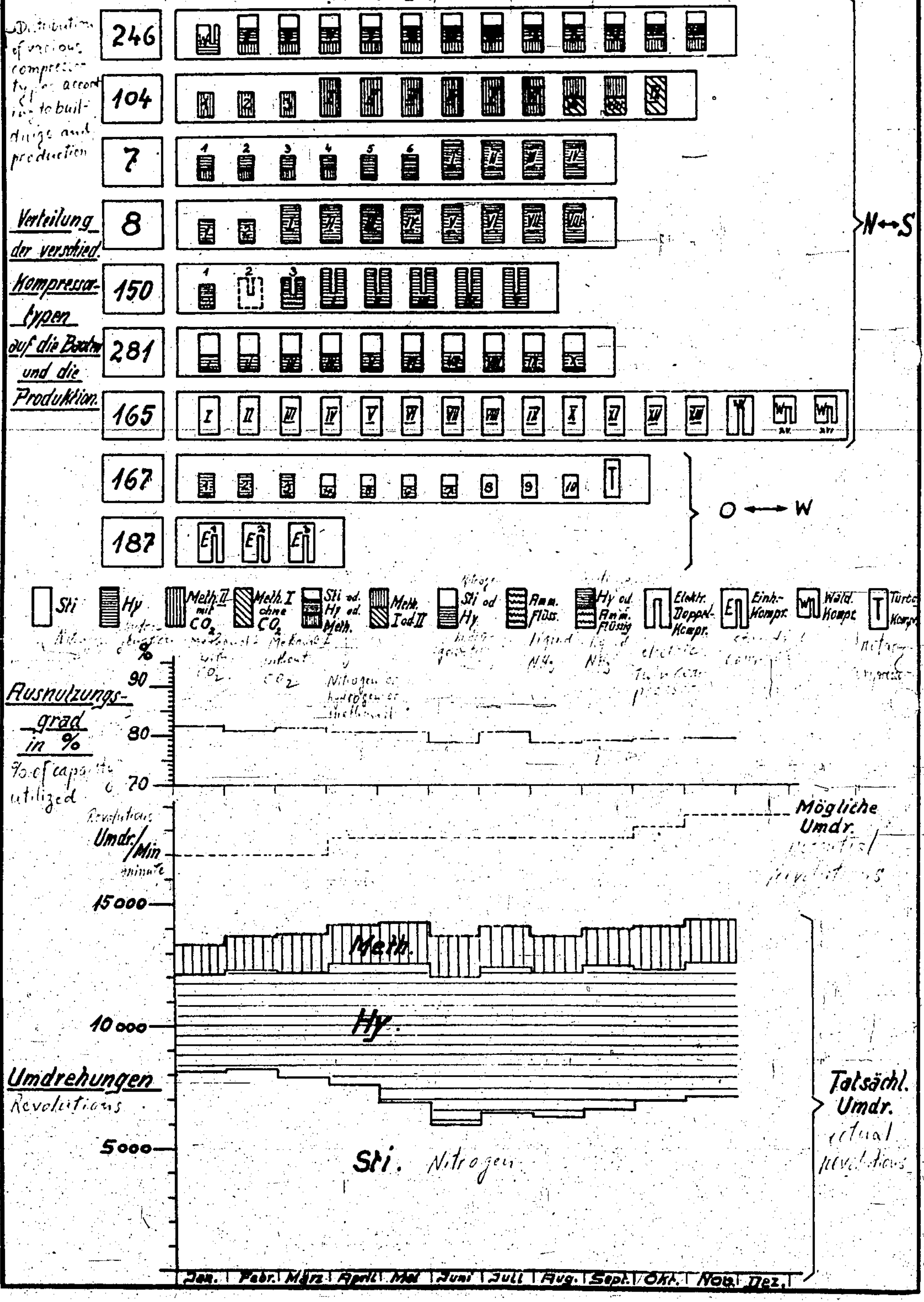
(Report on newly installed equipment and changes in the old equipment).

Gaskompressoren.

Gas compressors

Stand am 31. 12. 1939

status 12-31-1939



Operation of Gas Compressors

The rotary compressor which in November 1938 has been used for the first time ran only for 14 weeks in 1939. In the vicinity of 4300 revolutions per minute which is the regular number of revolutions at which the compressor operates, vibration set in so that in spite of many changes and experimental runs no satisfactory operation of the compressor could be achieved. The changes which were made to eliminate vibration are as follows:

Removal and reinstallation of the rotor wheels, balancing of the rotors, use of Novotext instead of bronze bushings for coupling bolts, increase of the play in the bearings, introduction of oil grooves into the bearings, heat and vibration measurements of the foundations, punching of the low-pressure rotor shaft. Finally, the bearings of the turbine and the low-pressure rotor were changed in such a way that a firmer seating of the shaft in the bearings resulted and the oil pressure is only throttled after the oil leaves the bearings. These measures finally resulted in quieter running of the compressor so that it could be used for regular production by the beginning of November.

Utilization of the capacity of the gas compressors at the end of 1938 was 30% and was kept at this value on the average during the year. In spite of the fact that no major breakdown of any machine took place, a number of unusual repairs had to be made. All fissures were found on inspection of endangered engine parts so that no breakdown occurred.

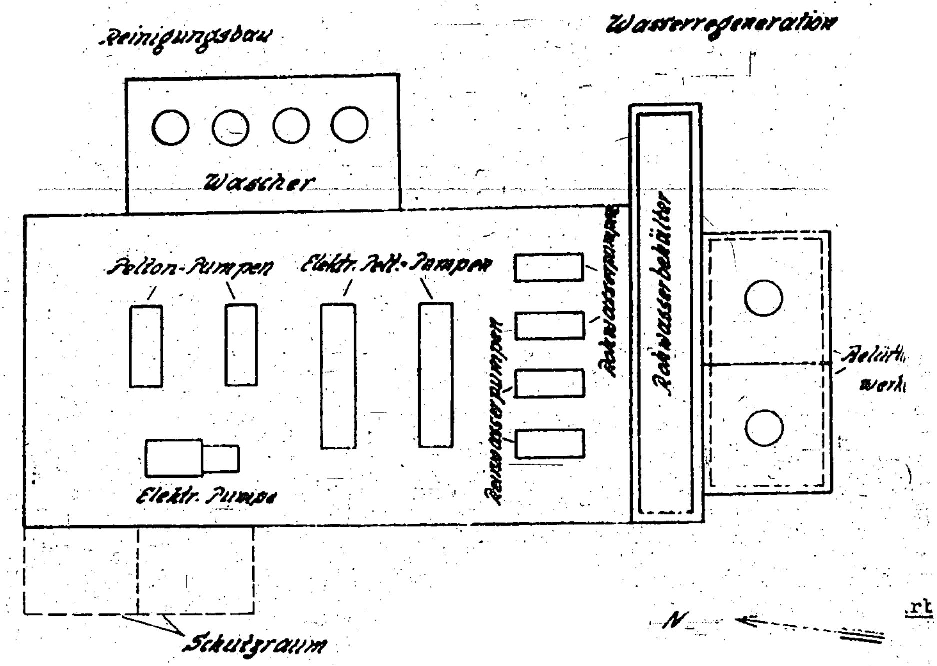
In addition to fatigue damage at the normal places (threads of piston rods, frames of gas engines, pistons of the third compression stage, etc.) fatigue damage on the cylinders of the fourth and fifth compression stage was found for the first time. Six of the twenty-four cylinders had to be replaced. This difficulty will be eliminated after discussion with the steel works by setting up new specifications.

(Page 24 contd.)

In order to permit operation of gas engines with a power gas of a higher hydrogen content and lower heating value, 18 of the 33 gas engines have been equipped with valves for additional air input. No improvement resulted from tentatively increasing the compression.

Druckwasserreinigung.

Neuer Bau 78.



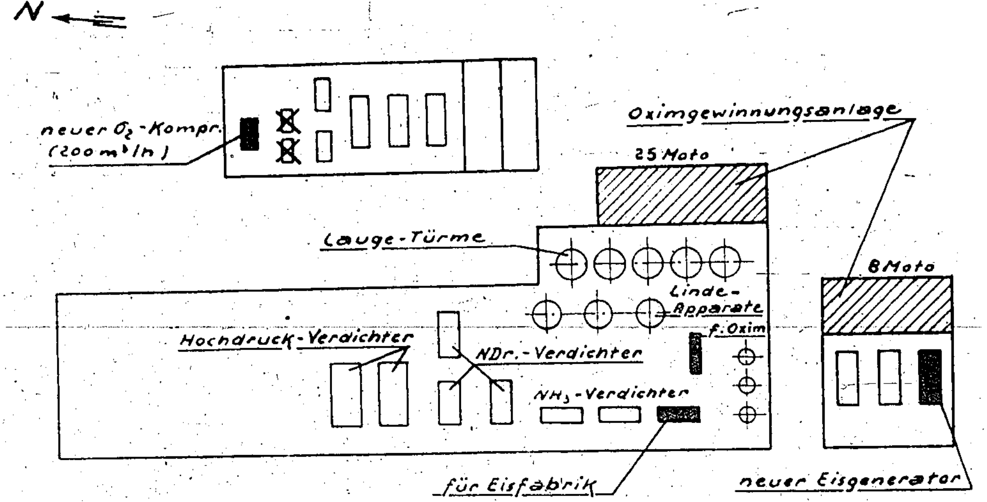
Reinigungsbau 169

Purification with pressure water and regeneration of the water

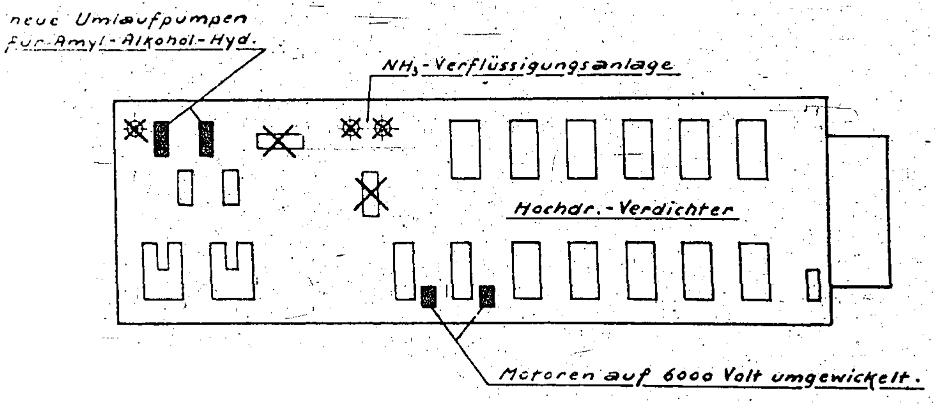
(Report on new equipment and changes in the operation.)

Stickstoff- und Sauerstoff-Fabrik

Bau 337 u. 337a



Bau 12



(Page 26)

Nitrogen, Oxygen and Krypton Plant

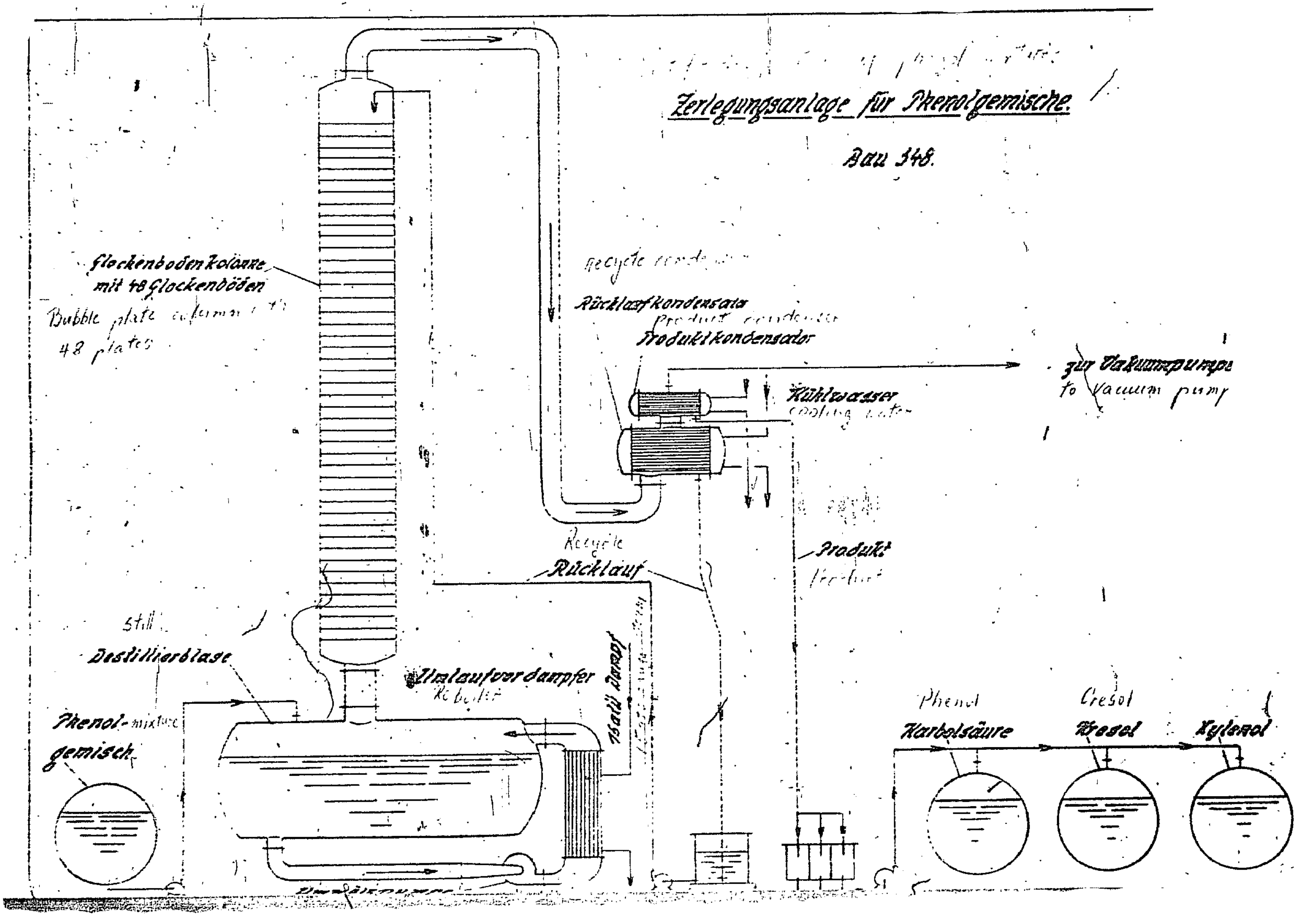
(Report on newly installed equipment.)

Auxiliary plants for the Nitrogen and Oxygen Unit

(Report on newly installed equipment)

Zerlegungsanlage für Phenolgemische.

Bau 348.



Phenol Plant

In order to test the biological purification process for the removal of residual phenol from phenol waste water after extraction with trisodium phosphate, an Emscher filter with an effective coke filling of 300 m^3 has been put into operation near the Neustadt basin; in this installation the phenols are decomposed by bacteria after the addition of phosphates. The efficiency of the filter was found to be about 100 g./m^3/24-hours.

Phenol Processing:

A fourth distillation unit with two new storage tanks made of aluminum was installed and put into operation. It is now possible to produce about 1000 tons of refined phenol oils per month.

The RD Phenol oil from R Phenol oil had up to now a water content of 1% which for certain purposes is too high. By taking a larger quantity of distillate overhead and rerunning part of this distillate with the next batch it was possible to reduce the water content to 0.1 to 0.3%.

Phenol Separation:

The phenol separation unit was completed and put into operation. The capacity of the discontinuous plant was found to be 600 tons per month of SR phenol oil or 500 tons per month of SD phenol oil.

Since different phenol oils are processed at various times, and since their separation products must be stored separately, new storage facilities had to be installed.

Work Planned for 1940:

In previous years the phenol content of the hydrogenation water increased from 3 g. of phenol per liter to about 4.5 g. per liter. In order to be able, in spite of this change, to remove the phenols to such an extent that the concentration in the waste water is about 0.5 g. phenol per liter, it is planned to add a third settling tank and another extraction stage.

(Page 28 Contd.)

A fourth triphosphate regeneration unit will be installed to work up the increased phenol water production from the hydrogenation.

The most important problem of phenol removal by means of tricresylphosphate consists in the purification of the contaminated phosphate solution. In laboratory experiments it was possible to carry out the purification by extraction with naphtha in aqueous methanol solution. An experimental unit will be installed in the plant.

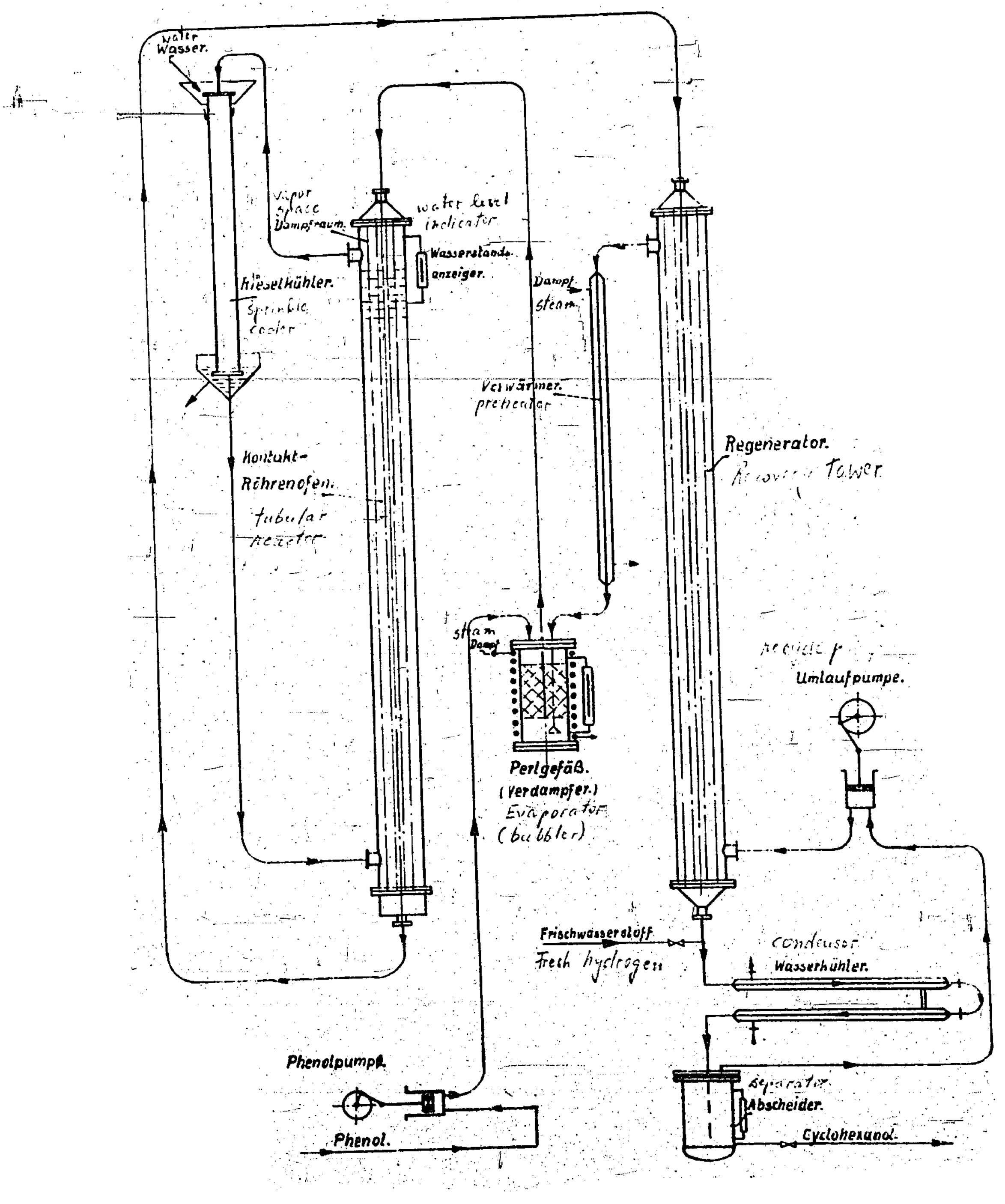
The Leverkusen ester process will be tested together with Leverkusen.

Ammonia plant

Ammoniakfabrik.

cyclohexanol manufacture

Cyclohexanolfabrikation.



Cyclohexanol Manufacture

A unit was erected for the manufacture of cyclohexanol by catalytic hydrogenation of phenol. The hydrogenation takes place at a pressure of 20 ats at 140 - 150°C over a nickel-alumina catalyst. The plant consists of four reactor units with one common circulating pump. The individual unit consists of a reactor, recovery tower, condenser and separator.

Water circulating around the catalyst tubes by the thermosyphon system serves to dissipate the heat in the reactors; in circular furnaces the water circulates around the circular catalyst bed. The reaction temperature is controlled by regulating the pressure of the circulating water.

Phenol is introduced into a vaporizer in which it is partly vaporized by hot hydrogen so that the higher boiling components which are detrimental to the catalyst remain as residue in the vaporizer and are removed from there with a small amount of residual phenol. At present, three tubular furnaces of 300 mm I.D. and one so-called circular furnace of 300 mm I.D. are on hand. The length of all furnaces is 8 m. The total capacity of the unit is 100 tons per month. Production which was started in February 1939 amounted to 60-70 tons per month on the average but increased towards the end of the year to 85-90 tons of pure cyclohexanol per month.

Work Planned for 1940:

The capacity of the cyclohexanol plant will be preliminarily raised to 500 tons a month. Two of the reactors required in this program are under construction; they have a diameter of 800 mm. The first of these reactors shall be in operation by the beginning of 1940, the second one in March 1940, and the remaining units shall be put into operation towards the middle of the year.

(Page 29 contd.)

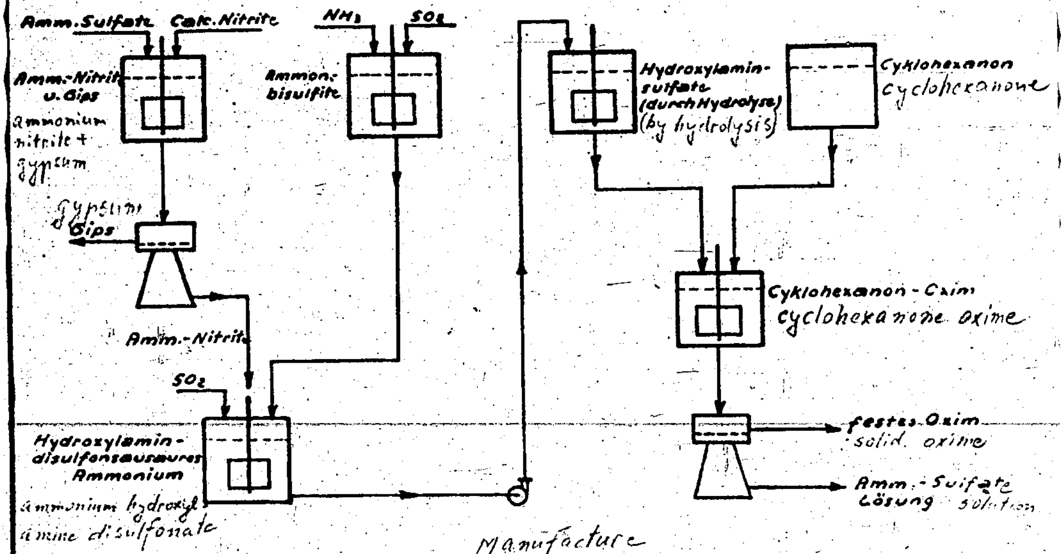
In order to obtain pure cyclohexanol with less than 0.05% phenol which is required for the manufacture of adipic acid, it is necessary to distill the reactor product. A distillation unit with a capacity of 300 tons per month of cyclohexanol is being constructed and shall start operations at the beginning of 1940. For the preliminary purification of crude S-oil a hydrogenation step is planned to replace the sulphuric acid treatment with its accompanying losses. Hydrogenation in this case must be carried out at 450°C. and 100 ats. pressure. The necessary plant with a capacity of 100 tons per month of S-oil will be constructed and should be in operation by the middle of March 1940.

Manufacture

Luran-Fabrikation.

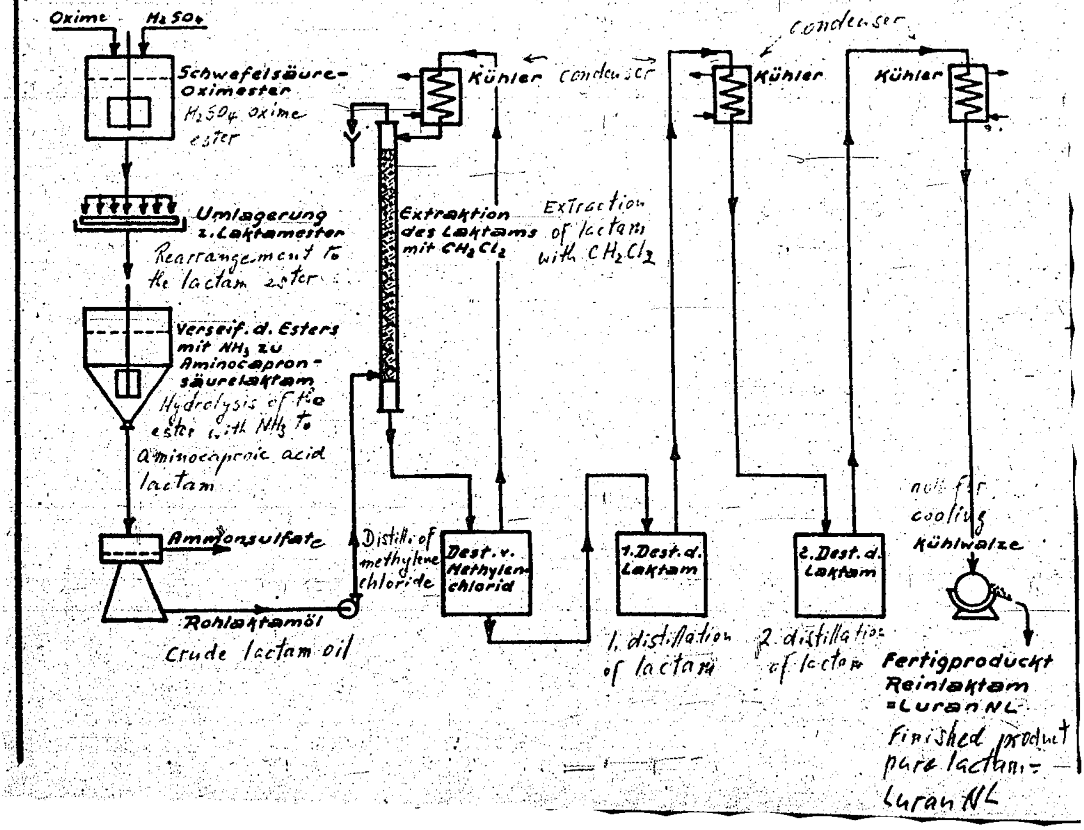
Hydroxylaminsulfat-u. Oxim-Herstellung.

Manufacture of hydroxylamine sulfate and oxime



Manufacture

Luran-Herstellung.



Luran Manufacture

For the annufacture of the new synthetic fiber Perluran at Wolfen, the intermediate product cyclohexanone-oxime and/or aminocaproic acid lactam (Luran-N) is required and these products shall be manufactured at Leuna. For the individual manufacturing steps new processes have been worked out in the laboratory and in the pilot plant.

1) Cyclohexanone-oxime

The oxime is made from hydroxylamine sulphate and cyclohexanone. The manufacture of hydroxylamine sulphate at first was carried out with sodium nitrite, SO_2 and NaOH. For economic reasons it has been tried to replace the sodium salts by calcium salts for which a cheap raw material source exists in the calcium nitrite solution from the nitric acid plant. A small pilot plant for 30-50 kg/day was in operation for a short time.

The manufacture of hydroxylamine sulphate could be considerably improved and cheapened by the use of ammonium salts instead of the corresponding calcium salts. Ammonium sulphate is reacted with calcium nitrite to form ammonium nitrite and gypsum which latter is filtered off. Reaction of SO_2 and NH_3 with ammonium nitrite results in the formation of hydroxylamine sulphate and ammonium sulphate. The presence of ammonium sulphate is advantageous because it salts out the cyclohexanone-oxime formed on addition of cyclohexanone to the solution.

A pilot plant for the manufacture of 8 to 10 tons of oxime per month is in operation at present but the unit is to be expanded to a capacity of 25 to 30 tons per month by the beginning of 1940.

2) Aminocaproic acid lactam (Luran-N)

The oxime is esterified with sulphuric acid and rearranged (according to Beckmann) to the lactam ester of aminocaproic acid. In order to dissipate the large heat of reaction which can easily lead to carbonization of the product, the liquid ester is passed through a distributor to a heated ferrosilicon tube, on which it spreads as a thin film and rearranges without carbonization.

The ester is hydrolyzed with ammonia. The lactam formed contains impurities which are removed by extraction with methylene chloride. A continuous extraction apparatus has been developed which proved satisfactory in the laboratory as well as in the plant.

The purification of the product is effected by double distillation under vacuum. It is first distilled in the presence of about 2% NaOH followed by a second distillation with about 2% phosphoric acid. The product so obtained is pure white and remains stable to light during polymerization. In a pilot plant the production of about 10 tons of Luran per month is carried out.

Laboratory For High Pressure Work

Experiments with the apparatus for 2000 ats were continued only with great difficulties because of lack of material and skilled personnel.

The experiments on the effect and elimination of catalyst poisons in the ammonia synthesis gases were continued in the laboratory. The investigations in the plant have been concluded for the time being until the changes planned for the improvement of the gas purity have been carried out.

Most of the work of the laboratory is concerned with the investigation of the manufacture of cyclohexanol and its homologs by hydrogenation of phenols. These experiments were carried out at 20 ats. analogous to plant conditions. Eighty catalysts were tested in 10 small reactors with 100 cc. catalyst space and investigations were carried out as to the degree of the conversion and the lifetime of the catalysts. In order to exclude catalyst poisons, pure phenol and cracking gas from liquid ammonia (as hydrogen source) were used. The nickel-alumina catalyst which is being used on commercial scale was found the most satisfactory of all the catalysts tested. This catalyst is prepared by reduction of the hydroxides of Ni and Al, which have been precipitated together from a nitrate solution.

In 8 other reactors of the same construction phenol oils from different sources were tested over the same catalyst. It was found that a considerable quantity of contact poisons can be held back by charging the hydrogen with phenol vapors in an evaporator and avoiding the complete evaporation of the liquid in the evaporator. In this way crude phenol oil can be hydrogenated without additional processing and the catalyst will not be prematurely poisoned. It is then only necessary to refine the residue in the evaporator in the usual way with acid or caustic or to hydrogenate it under mild conditions.

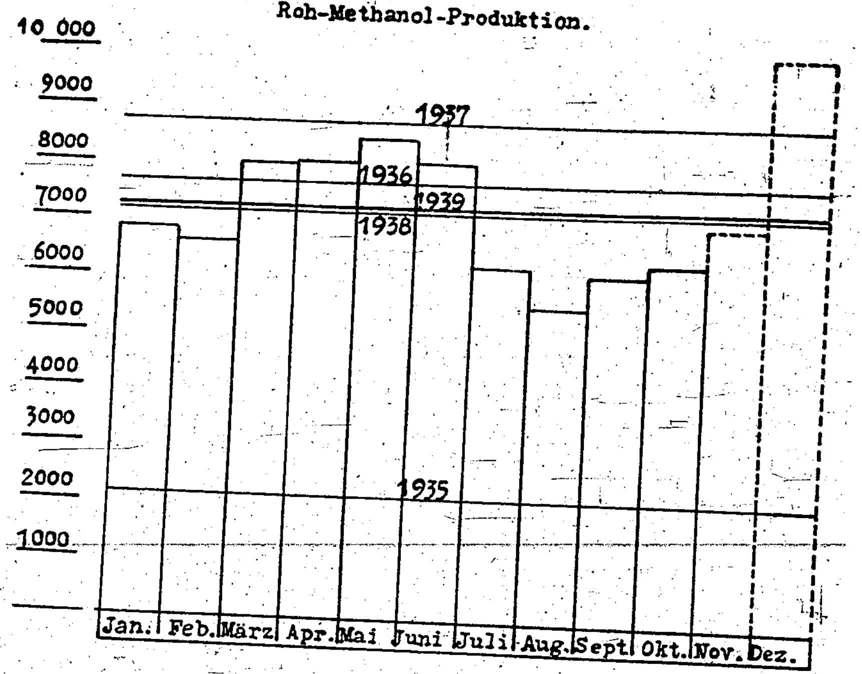
This process has been worked out with good success for M-cresols. The experiments were made in a reactor with 3 liter catalyst space, using recycle gas from the cyclohexanol plant. Further experiments are concerned with the hydrogenation at atmospheric pressure and the use of recycle gas from the ammonia plant.

Work Planned for 1940:

The experimental work on the manufacture of cyclohexanol and methylated cyclohexanols from phenol oils are to be continued and the suitability of the process of partial evaporation will be tried in reactors with 3 liter catalyst space.

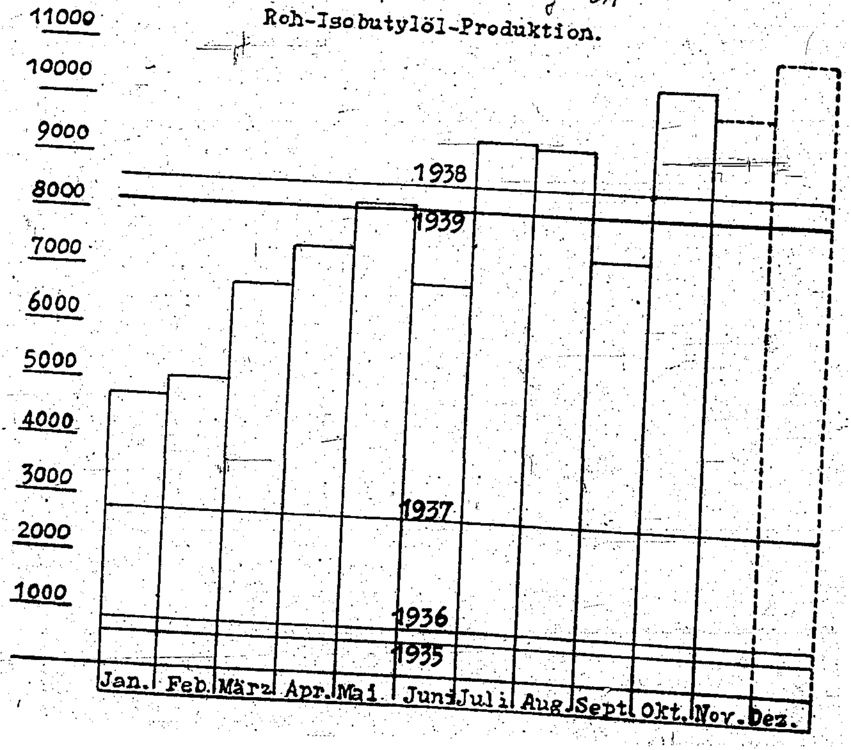
Production of crude methanol

Roh-Methanol-Produktion.



Production of crude isobutyl oil

Roh-Isobutylöl-Produktion.



ORGANIC DEPARTMENT

Production of Methanol and Isobutyl Alcohol

a) **Methanol Plant:**

Because of discontinuation of the addition of methanol to motor fuel the methanol production decreased slightly. The increased consumption for other purposes, especially during the last months, had a sufficiently great effect on the production so that the yearly average was about the same as during the last year.

For the quick separation of the methanol cycle from the isobutyl alcohol cycle, a valve has been installed which shall later be replaced by an electrically operated quick-acting gate valve.

For 1940 it is planned to carry out large scale experiments to obtain equal capacity per reactor with decreased catalyst space.

b) **Isobutyl alcohol:**

Delivery of higher alcohols to the alcohol monopoly for the purpose of stretching the alcohol used in motor fuel was discontinued during the last year so that the report year was entered with a small production. In the course of the year the consumption of isobutyl alcohol and other products increased so much due to the war economy that the loss of this outlet was almost completely balanced. At times up to 12 reactors were operated. The production at the end of the year lies only slightly below the best month of the previous year and the total production has almost reached that of the previous year and will exceed it in 1940. By further increasing the efficiency of the individual reactors (by changes in the operation) it has become possible to produce the entire output in building #333. The maximum output of one furnace has reached now 56 tons per day.

(Page 38 contd.)

In the course of the year dimethyl ether which is obtained in the methanol and isobutyl oil plant was either sold or recycled; the latter practice led to a corresponding saving in gas.

By the end of the year it was possible for the first time to succeed in the large scale manufacture of ethyl alcohol. It was, however, necessary to establish a special cycle since the cobalt carbonyl formed leads to severe disturbances in the methanol cycle.

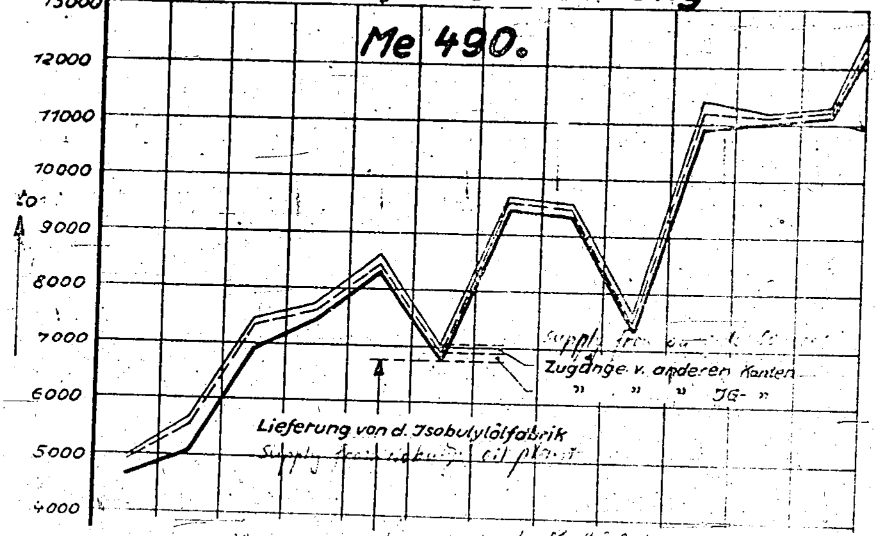
For 1940 it is planned to increase the efficiency of the reactors by the installation of electric preheaters. It is planned to wash the pressure release gases with water or methanol in order to supply a purer gas to the ammonia plant. The ethyl alcohol experiments will be continued on a larger scale after completing the new cycle.

c) Laboratory Work:

In addition to less important work, the manufacture of adipic and methyladipic acid by oxidation of cyclohexanone and methylcyclohexanone with oxygen in the presence of manganese salt was investigated. Two units for pilot plant work were built. These experiments will be continued in 1940 with the aim of putting the reaction on a commercial basis.

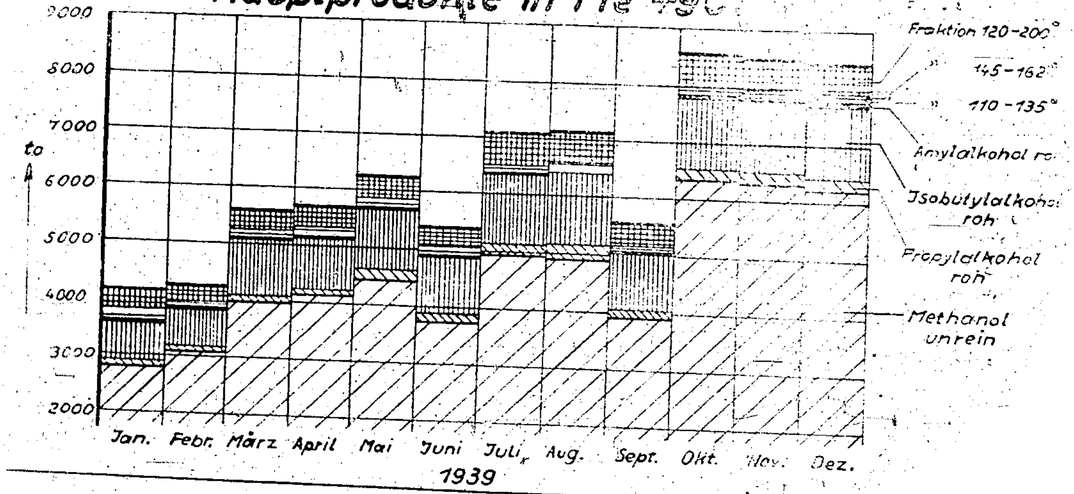
Processing of crude isobutyl oil

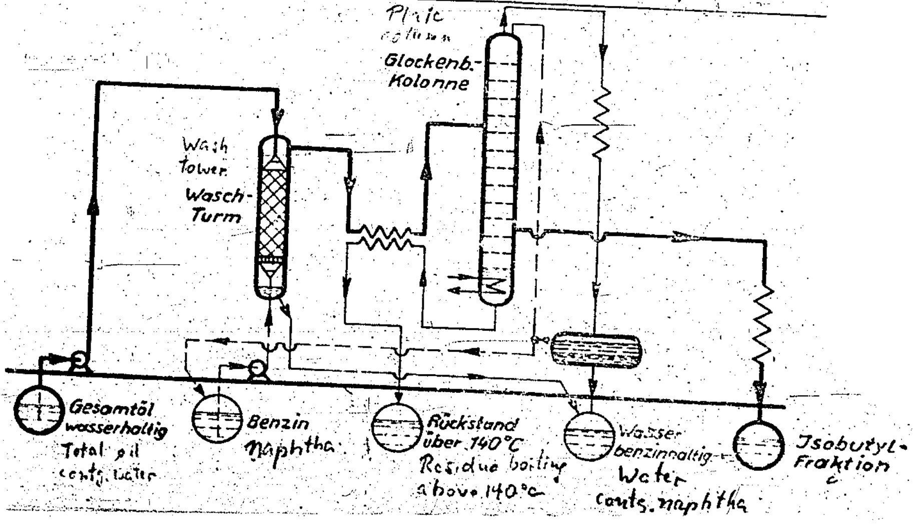
Roh-Isobutylölverarbeitung



Main products in plant Me 490

Hauptprodukte in Me 490





Distillation of Isobutyl Oil, Removal of Ether from Methanol and Isobutyl Oil, Manufacture of Pure Methanol from Dimethyl Ether

Isobutyl Oil Distillation

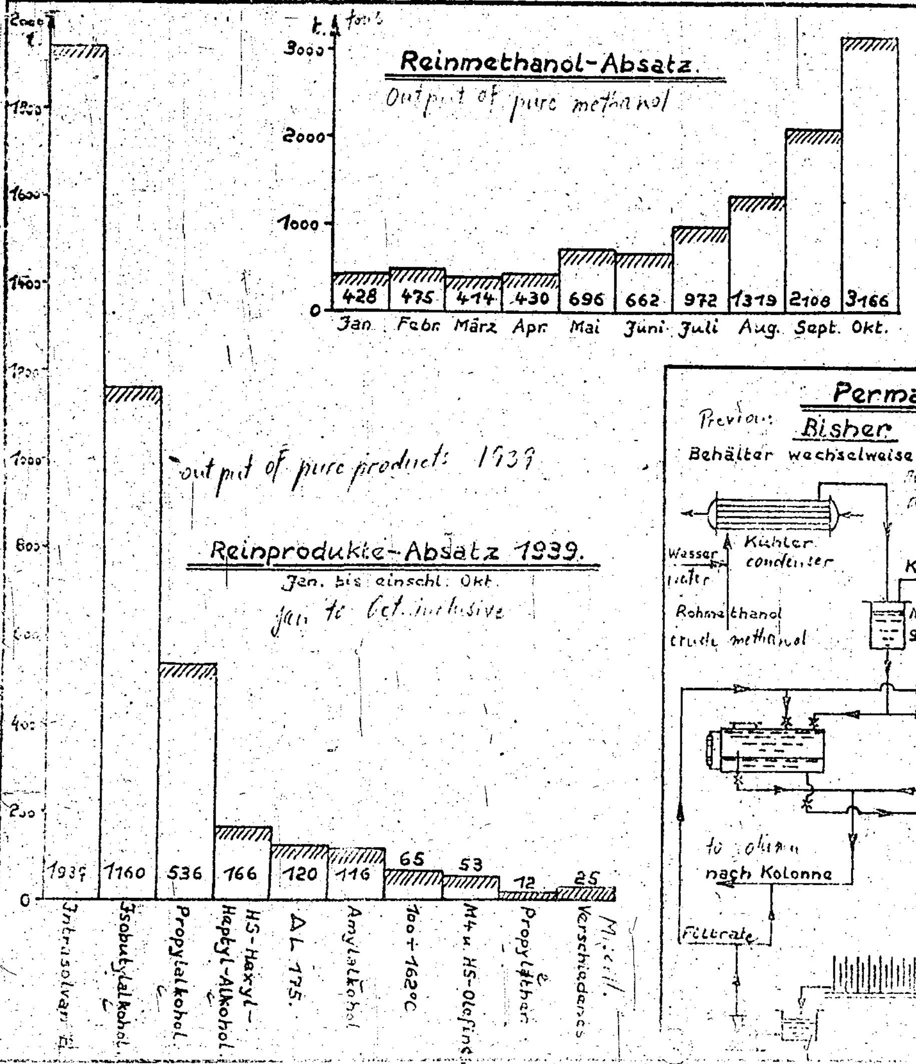
The isobutyl oil distillation successfully fulfilled its task to prepare anhydrous alcohols or alcohol fractions from isobutyl oil without major interruptions. The removal of water from the higher alcohols was improved by using an extraction tower with naphtha as extraction medium. In this way operations were improved as compared to the previously used azeotropic distillation and the cost could be lowered by about 25%. The change in operation also saved one column. For 1940 it is intended to increase the isobutyl oil production to about 14,000 tons per month. Successful experiments are under way to separate the isobutyl oil residue in a packed tower into two layers and thereby save the centrifuges required at present for the separation of the emulsion which forms frequently.

Removal of ether from methanol and isobutyl oil and manufacture of pure methanol from dimethyl ether

The manufacture of methanol from dimethyl ether has been discontinued and the dimethyl ether is recycled to the synthesis except for the quantity which is sold outside.

The dimethyl ether from isobutyl oil has been recycled completely to the isobutyl reactors. On the pilot-plant scale it was possible to purify this ether by removal of low boiling hydrocarbons by absorption under pressure. A former distillation column is being prepared for the removal of dimethyl ether from the entire output of isobutyl oil.

It is planned for 1940 to install a larger column for the removal of dimethyl ether from methanol since the ether from the methanol process is almost free from impurities (in contrast to the product from the isobutyl oil process) and Ludwigshafen intends to take about 300 tons per month.



Reinmethanol-Absatz.

Output of pure methanol

Reinprodukte-Absatz 1939.

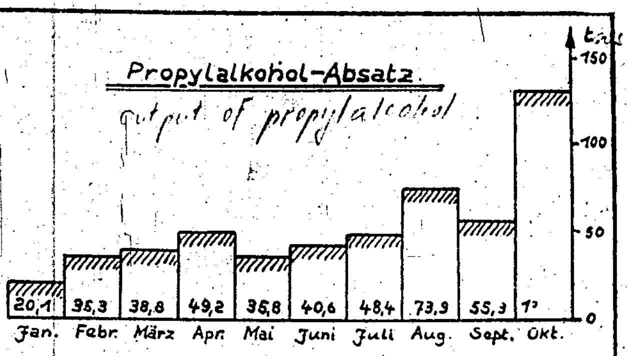
Jan. bis einschl. Okt.

Jan to Oct. inclusive

output of pure products 1939

Monat	Reinmethanol-Absatz (t)
Jan.	428
Febr.	475
März	414
Apr.	430
Mai	696
Juni	662
Juli	972
Aug.	1319
Sept.	2108
Okt.	3166

Produkt	t
Intrasolvan E.	1939
Isobutylalkohol	1160
Propylalkohol	536
HS-Hexyl-Heptyl-Alkohol	166
AL 175.	120
Amylalkohol	116
100 ÷ 162°C	65
M4 u. HS-Olefine	53
Propyläther	12
Verschiedenes (Miscell.)	25



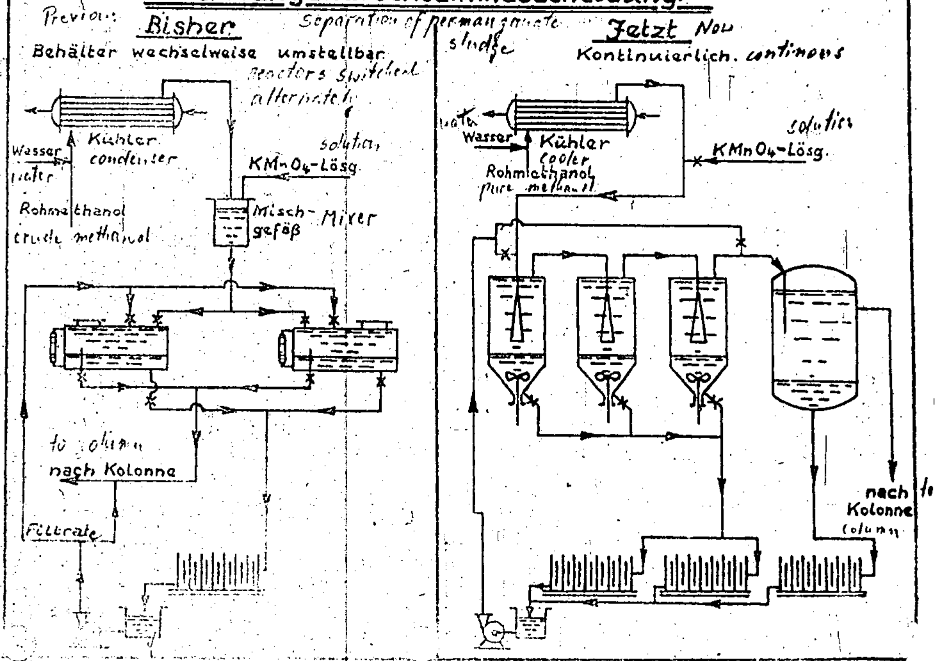
Propylalkohol-Absatz.

output of propylalcohol

Monat	Propylalkohol-Absatz (t)
Jan.	20,1
Febr.	35,3
März	38,8
Apr.	49,2
Mai	35,8
Juni	40,6
Juli	48,4
Aug.	73,9
Sept.	55,3
Okt.	1?

Permanganatschlammabscheidung.

Separation of permanganate sludge



Distillation of Pure Methanol and Isobutyl Alcohol Manufacture of Propyl Ether and M_4

After the contract for the supply of methanol for automotive fuel purposes had terminated at the end of September, the distillation equipment could be used entirely for the manufacture of pure methanol. By conversion of the batch separation of manganese sludge to a semi-continous process and by the installation of two new filter presses and the installation of some new bubble plates in the column, it was possible to reach a capacity of about 5000 tons of pure methanol per month. The previous deliveries lay at about 3200 tons per month and will probably rise further.

The demand for pure products from isobutyl oil increased further as compared to the last year and a further rise, especially for foreign delivery, took place since the beginning of the war. The apparatus for the manufacture of propyl ether was in operation when required. The uses for propyl alcohol which originally was almost completely recycled have multiplied so much during the year that towards the end of the year about 85% of the production was further processed or shipped out.

About 80 tons of cyclohexanol were distilled in a batch vacuum still. A new unit for this purpose is to be installed at the beginning of the next year.

Work Planned for 1940:

Expansion of the unit for the manufacture of mixed polymerizate to produce 30 to 40 tons of M_4 per month to be used in the manufacture of Igepal. Installation of a fractionating column for the separation of methanol from the methanol-oil-water mixture originating from crude methanol.

Refining of Alcohols

Three 500 type reactors are available for the after-hydrogenation of higher alcohols. These reactors will be sufficient for the near future since it was possible to increase their capacity by expanding the supply of electric power. Their capacity is now limited by the electrical equipment.

Three 500 type reactors with a capacity of about 600 to 800 1/h hour each are available for the manufacture of gasoline from those higher alcohols which can at the present time not be sold. The reactors are equipped with a line for cooling gas. The gasoline production amounts to about 600 to 800 tons per month.

Several times during the year Kogasin was hydrogenated for the manufacture of Mepasin; Spindle oil was subjected to after-hydrogenation to manufacture white oil. For these hydrogenations a gasoline reactor with special heavy electrical equipment is being used.

It is intended in 1940 to locate the Kogasin hydrogenation somewhere else so that more spindle oil can be processed.

Laboratory for Work on Isobutyl Oil Me 19/19a

Analytical Work

The production of the methanol and isobutyl oil distillation as well as the manufacture of propyl ether and the production of the pilot plants were continuously controlled. A laboratory has been built for testing of lacquers and lacquer raw materials.

Experimental Work

The manufacture of isoamyl and isooctyl ether has been investigated since the manufacture of propyl ether has reached the production stage.

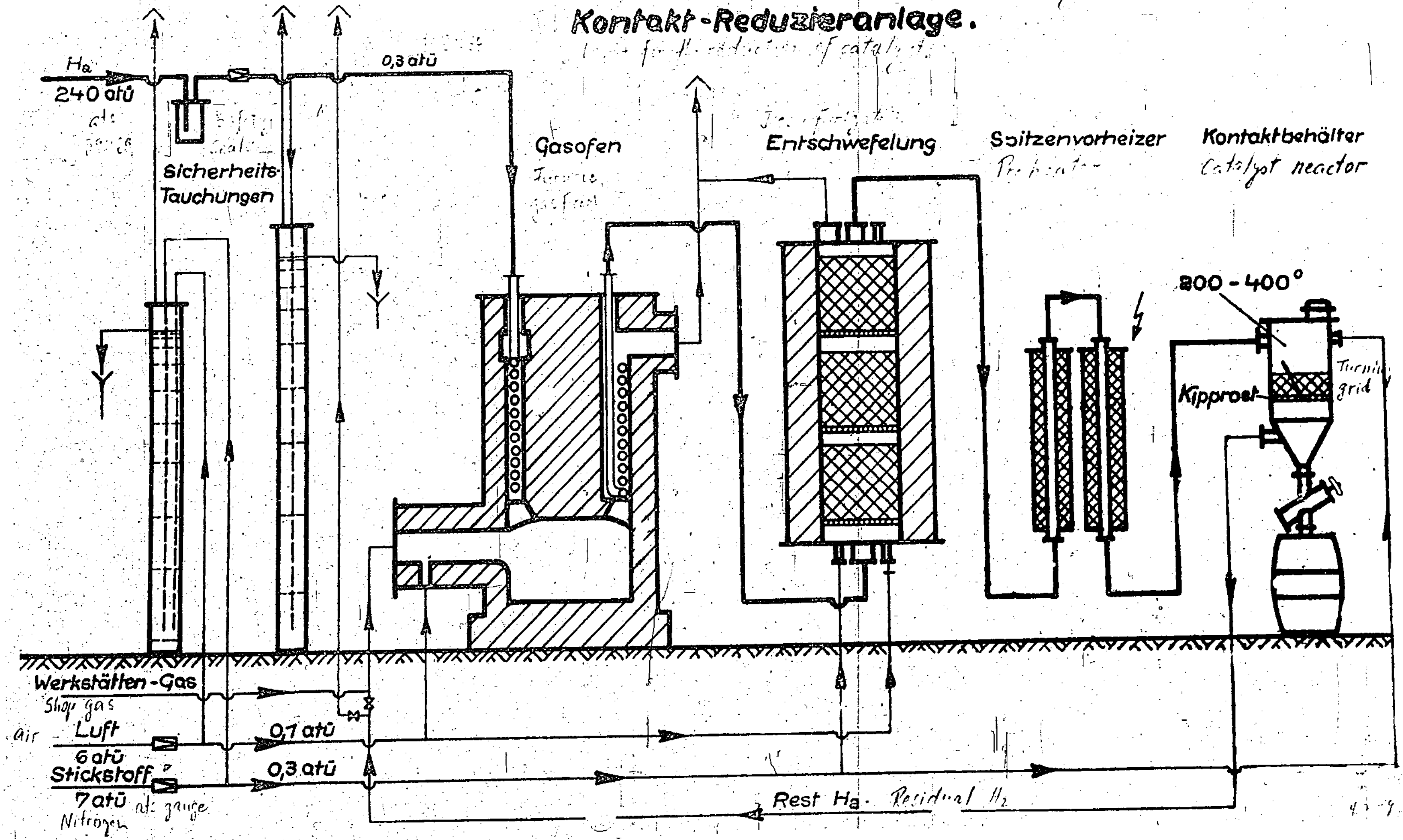
The work on the preparation of lower and higher ketones from carboxylic acids by decarboxylation and from alcohols by dehydrogenation has been continued. Interest was centered mainly on the dehydrogenation of isopropyl alcohol and of sec.-butyl alcohol (from Treibstoffwerke Rheinpreussen) for the manufacture of acetone and methylethyl ketone.

Systematic investigations were carried out to increase the yield obtained in the preparation of multivalent esters and these experiments have not yet been concluded. The purification of Palatinol HS was worked out in larger scale experiments in cooperation with Ludwigshafen. Experiments for the manufacture of plasticizers similar to Palatinol were extended to products from Leuna.

Methanol-Kontaktfabrik.

Kontakt-Reduzieranlage.

Plant for the reduction of catalyst.



Methanol Catalyst Plant, Gelberde Catalyst Plant (Catalyst 5058)

Catalysts for the following processes have been incorporated into the manufacturing program:

Hydrogenation of phenol to cyclohexanol, hydrogenation of tiglic aldehyde to amyl alcohol, hydrogenation of acetylene to ethylene by the Häuber-Hirschbeck Process, manufacture of stearylamine. The manufacturing program also included five-component catalysts for syntheses developed in the laboratory. A considerable lowering in the manufacturing cost for precipitation catalysts could be obtained by complete removal of oil and iron from the Condensate water by means of activated charcoal instead of by the more costly distillation process. The condensate treated in this way corresponds in its purity to the distillate and can be used as wash water without any objections. The cost for activated charcoal amounts to less than RM 0.10/m^3 of water, as compared to RM 1.00 for the distillation process. The savings amount to about RM 5000.00 per month. For those catalysts which for operational reasons must be reduced outside the reactor a plant was built in which the catalysts can be reduced with hydrogen at atmospheric pressure. Production figures are as follows:

Catalyst for the Manufacture of	1938	1939
Methanol	45 tons	54 tons
Isobutyl oil, Leuna and Ludwigshafen	204 "	272 "
Methanol recovery	4 "	0.2 "
Methanol after hydrogenation	15 "	-
Amines	7 "	5.9 "
T 52 and Di 1000 and gasoline from higher alcohols	15.5 "	16.3 "
Fischer Synthesis	2.2 "	0.4 "
Anol (cyclohexanol)	-	12 "
Ethylene after-hydrogenation	-	2.9 "
Ethyl Alcohol	-	5.7 "

The following equipment for the pretreatment of catalyst masses and for shaping was newly installed: a ball mill for 100 kg. product, a mixer for very tough masses, two extruding machines, one rolling machine for the shaping of spheres from plastic masses which set on drying, one large tabletting machine for preliminary pressing of masses which are difficult to shape.

Work Planned for 1940:

Since several large scale experiments had been successful in which isobutyl catalyst was used which was made of equal parts of fresh material and used materia it is intended to work up the used isobutyl catalyst to a large extent for the manufacture of fresh catalyst. It is estimated that in 1940 about 100 tons of zinc oxide and 50 tons of chromic acid of a total value of RM 80,000 can be saved in this way. For this purpose the installation of a continuous drying kiln is planned.

The catalysts required for the T 52 plant shall be developed to the commerical scale.

No catalyst 5058 is at present being made since there is no demand for this material because of the long lifetime of the catalyst. The tungsten containing residues which were collected during the last year have been worked up to usable catalyst and the used catalysts which were received were processed in the same way. The plant was overhauled and started again in December.

Work Planned for 1940:

The roasting plant for used catalyst must be brought to a capacity of 40 tons per month by the installation of a new oven since in the near future (after further gasoline plants will have been started) only used catalyst will be processed to make fresh catalyst. Drying of ammonium sulfotungstate which up to now has been carried out by means of hot nitrogen at considerable expense shall be carried out in a heated pressure filter or a drying pan which will result in a saving of about 150,000 m^3 of nitrogen per month.

(Page 43)

Catalyst Experiments

About 1000 new catalysts were again prepared. The following problems were of greatest interest:

(1) Alcohol synthesis from CO/H_2, (2) hydrogenation of unsaturated aldehydes and alcohols, (3) manufacture of T 52.

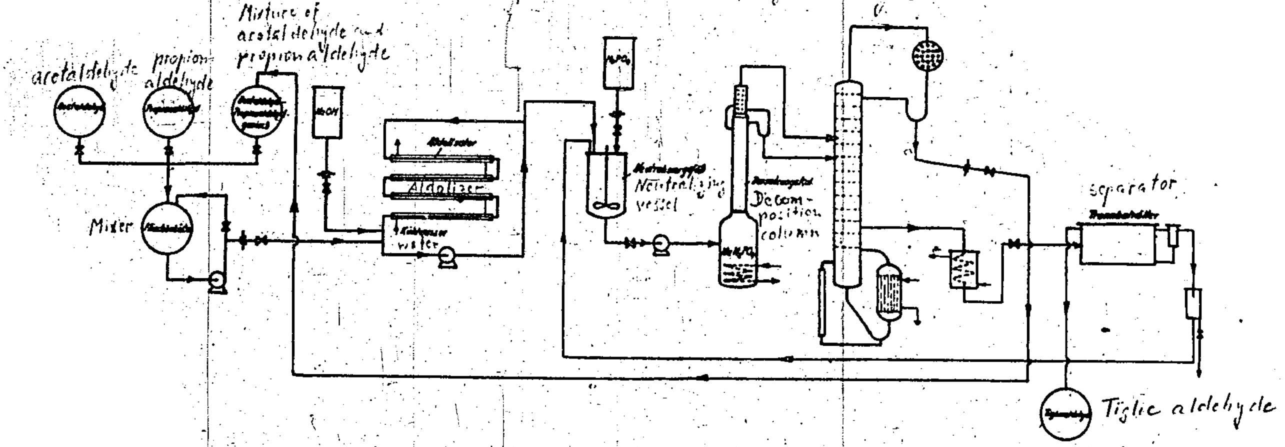
For the first of these problems a five-component catalyst has been developed which permits to stop the alcohol formation at the ethyl alcohol stage and which has also given satisfactory results in the pilot plant. With regard to the second problem, it was possible to develop a Cu catalyst for the hydrogenation of dimethylolethanealdol (P_2) and a Ni catalyst for the manufacture of Anol (cyclohexanol) and amyl alcohol; the manufacture of Raney nickel was successfully taken up. The work on the improvement of the catalyst for the manufacture of T 52 has not yet been concluded.

Work Planned for 1940:

The above problems are to be followed up especially the alcohol synthesis from CO/H_2 with the aim to develop specific catalysts which stop the alcohol formation at the propyl or isobutyl alcohol stage.

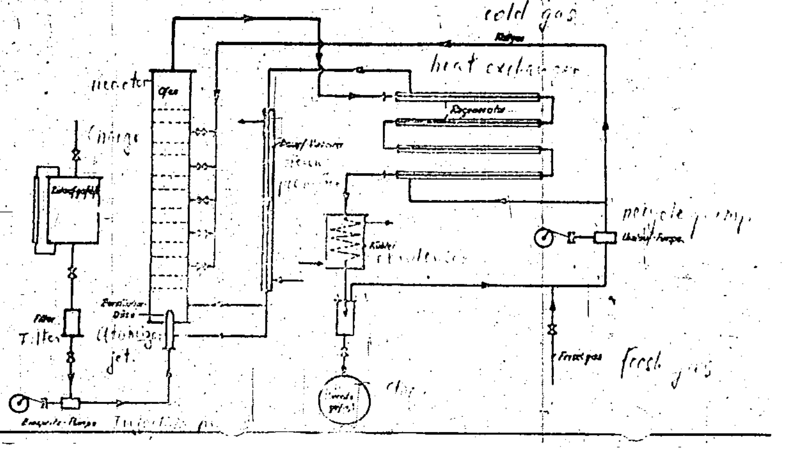
Schema der Tiglinaldehydherstellung.

Flow sheet of the manufacture of tiglic aldehyde



Tiglinaldehyd-Hydrierung 180°C, 10-20 atü

Hydrogenation of tiglic aldehyde, 180°C, 10-20 ats gauge



Manufacture of Amyl Alcohol

For two years the supply of the German amyl alcohol market has been insufficient. It was possible to manufacture amyl alcohol from acetaldehyde and propionaldehyde at Leuna on a technical scale.

Acetaldehyde and propionaldehyde are mixed in a mol ratio of 1.4:1 and condensed with dilute NaOH at p_H 8-9 to acetopropionaldole; conversion is about 50%. After neutralization with dilute phosphoric acid (to a p_H 5-7) water is split off from the aldole in the decomposition column and the tiglicaldehyde formed is distilled with the unreacted acetaldehyde and propionaldehyde. The unreacted aldehydes are taken overhead whereas the tiglicaldehyde-water azeotrope is taken off as a side stream in the lower third of the bubble plate column. After cooling, the azeotrope separates into an upper layer of tiglicaldehyde and a lower aqueous layer which contains 10% tiglicaldehyde in solution and is recycled. The tiglicaldehyde which contains about 5% water can be hydrogenated to amyl alcohol without further purification. The hydrogenation is carried out 160° to 180°C. and 10 ats. over a nickel-alumina catalyst. The space velocity is 0.5. The temperature of the strongly exothermic reaction is controlled by cold gas and a sufficiently large amount of recycle hydrogen.

The capacity of the aldole apparatus and the hydrogenation unit is about 60-100 tons per month. As by-products n-butyl alcohol, isohexyl alcohol and resins are formed besides a small amount (1 to 2%) of n-amyl alcohol. The main and side reactions are given in the following equations:

$$CH_3CHO + CH_3CH_2CHO \longrightarrow CH_3CHOHCH(CH_3)CHO \xrightarrow{-H_2O} CH_3CH{=}C(CH_3)CHO \xrightarrow{+2H_2}$$

Acetaldehyde + Propionaldehyde   Acetopropionaldole   Tiglicaldehyde

(Page 44 Contd.)

$CH_3CH_2CH(CH_3)CH_2OH$

Isoamylalcohol

$2CH_3CHO \longrightarrow CH_3CHOHCH_2CHO \longrightarrow CH_3CH{=}CHCHO \longrightarrow CH_3CH_2CH_2CH_2OH$

Acetaldehyde Acetaldole Crotonaldehyde n-Butanol

$2CH_3CH_2CHO \longrightarrow CH_3CH_2CHOHCH(CH_3)CHO \longrightarrow CH_3CH_2CH{=}C(CH_3)CHO \longrightarrow CH_3CH_2CH_2CH(CH_3)CH_2OH$

Propionaldehyde Propionaldole Hexenylaldehyde Isohexyl alcohol

$CH_3CH_2CHO + CH_3CHO \longrightarrow CH_3CH_2CHOHCH_2CHO \longrightarrow CH_3CH_2CH{=}CHCHO \longrightarrow CH_3(CH_2)_3CH_2OH$

Propionaldehyde Acetaldehyde Propionacetaldole Pentenealdehyde Amyl alcohol

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Amyl alcohol is used for the manufacture of solvents (amylacetate), plasticizers (amylphthalate), as flotation agent (amylxanthate) and for other purposes. The by-products are disposed of in the solvent field.

Work Planned for 1940:

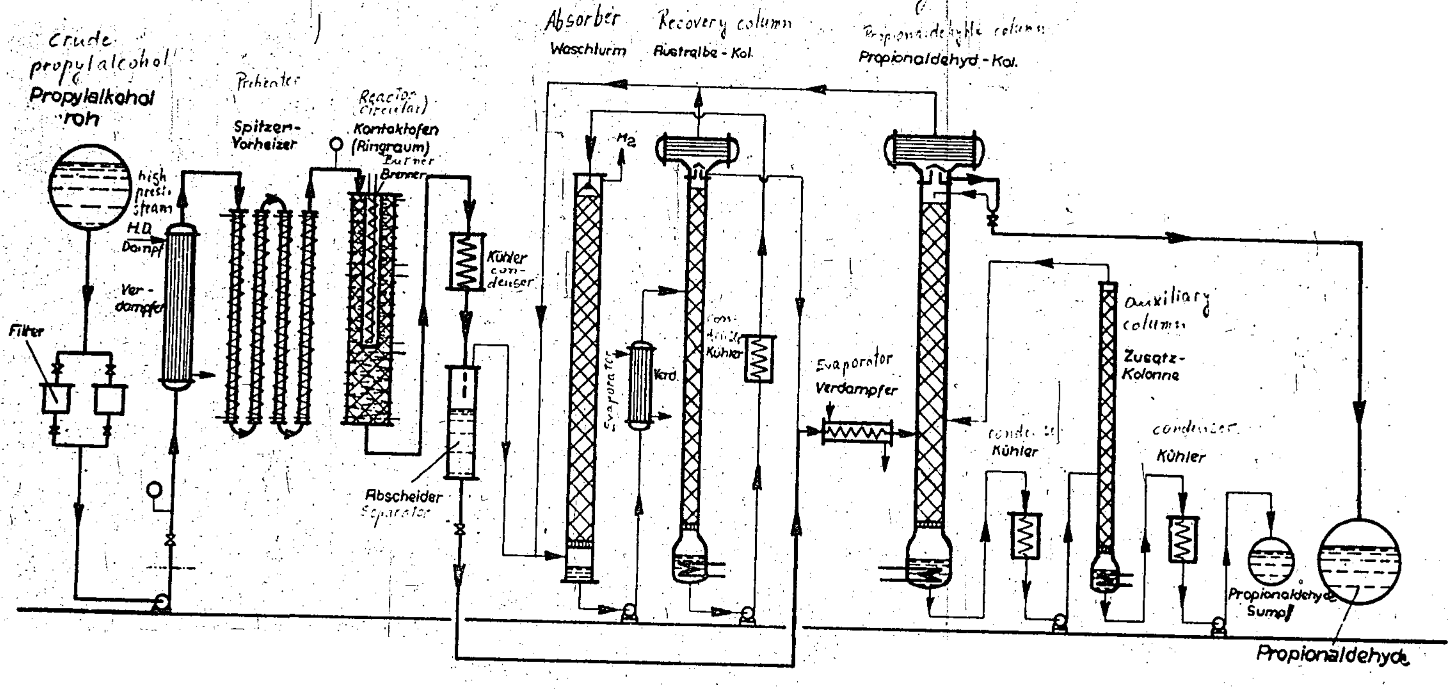
It will be attempted to suppress the formation of by-products by finding the optimum residence time in the aldole reactor as well as the best acetaldehyde: propionaldehyde ratio. Furthermore, it is necessary to improve the separation of the unreacted aldehyde from tiglicaldehyde.

Manufacture of aldehyde

Aldehyd - Fabrikation.

Schema der Propionaldehyd - Herstellung.

Flowsheet of the manufacture of propionaldehyde



Manufacture of Aldehyde and Cyclohexanone

Aldehyde:

For the manufacture of aldehydes, especially of propionaldehyde, from the corresponding alcohols from isobutyl oil a new circular reactor (Leuna system) with 200 l. catalyst space has been installed. The heat input for the strongly endothermic dehydrogenation takes place through electric heating on the outside and through a burner inside the reactor. Ten copper lined electrically heated N8 tubes of 8m. length are used as preheaters. The preheat required as well as the energy absorption in the reactor are of the magnitude expected. After a prolonged period of operation, however, alcohol or aldehyde vapors diffuse into the space between the iron wall and the copper lining. Subsequently, cracking accompanied by carbon formation takes place on the outer iron wall and the carbon ruptures the copper lining. This disadvantage will be eliminated in a new reactor by fabricating both the preheater and reactor from V2A extra material.

The losses in the manufacture of propionaldehyde were very high because the hydrogen liberated during the dehydrogenation step carried away about 10% of the aldehyde in spite of low-temperature refrigeration with ammonia. By a newly installed oil absorption step it has been possible to lower the loss of 2%; the isobutyl oil fraction 200 to 250°C was ~~found~~ suitable as absorption oil and could be continuously regenerated by heating.

Further quantities of aldehyde were lost with the bottom product of the aldehyde column which contains about 5% aldehyde. By the installation of an additional column this source of loss was also eliminated.

In laboratory experiments it was found that propionaldehyde can be prepared in good yield by passing a mixture of propyl alcohol and air over a silver catalyst at 400 to 600°C. The yield in a small scale experiment was as high as 87% but in a pilot plant operation which could not be as yet completely controlled it amounted to 80 to 83%. Isobutylaldehyde was prepared by this process with a yield of 90% of the theory and higher aldehydes can also be made in this way. It is also possible to oxidize ethyl alcohol to acetaldehyde and isopropyl alcohol to acetone with good yields. A plant for the manufacture of 2 tons of propionaldehyde per day will be ready for operation at the beginning of 1940.

Cyclohexanone:

For the Luran manufacture and for the manufacture of adipic acid by the oxygen process cyclohexanone made by dehydrogenation of cyclohexanol is required. On the basis of the work by Dr. Herold's group brass turnings with 58% copper are used as catalyst at 520 to 530°C. Only at these high temperatures a conversion exceeding 90% is obtained. Two to three percent cyclohexene and cyclohexane 0.5 to 1% of water, 0.3 to 1% phenol and a small amount of higher condensation products are formed as by-products. The separation of cyclohexanone from unreacted cyclohexanol cannot be completely accomplished by distillation because of the close boiling range of the two products and it is therefore necessary to keep the conversion very high.

At the above mentioned temperatures the carbon formation on the catalyst is so heavy that the apparatus has to be shut-down after eight to ten days since it is clogged up by that time. According to information obtained from Dr. Herold's group, a brass catalyst with 20% copper and 80% zinc gives a 90% conversion at 400 to 450°C. The catalyst which is used on a commercial scale at Ludwigshafen is now being tested.

(Page 45 Contd.)

Work Planned for 1940:

Aldehyde:

Installation of the V2A-dehydrogenation reactor. Starting of the 2 ton/day reactor for propionaldehyde from propyl alcohol and air; experiments with this reactor for the manufacture of formaldehyde, acetaldehyde, etc.

Cyclohexanone:

Testing of the Ludwigshafen catalyst on a technical scale. Testing of the copper - zinc catalyst at 400 to 450°C. Installation and starting of a plant for the manufacture of cyclohexanone and methylcyclohexanone with a capacity of 150 tons per month.

Pilot Plant Laboratory Me 13a

Preliminary investigation of catalysts for the manufacture of methanol, isobutyl and ethyl alcohol. Several multi-component catalysts for synthesis of alcohol from $CO + H_2$ were tested. The testing of the five-component catalyst in a pilot plant reactor with 10 liters of catalyst confirmed the results of previous smaller scale experiments. At 400 - 420 °C a specific yield of 0.8 l. (calculated on 1 l. of catalyst) was obtained and the product consisted of 45% methanol, 13% ethyl alcohol, 1% propyl alcohol, 6% ester (mainly methylacetate) and 25% water. Experiments for recycling of methanol for the manufacture of ethyl alcohol were negative. It is planned for 1940 to continue development of the ethyl alcohol catalyst.

Laboratory for Experiments at Very High Pressures. Me 238

The problems investigated concerned the effect of very high pressures on the synthesis from CO/H_2 and the preparation of acids from alcohol and CO.

The expansion of the pilot plant laboratory was continued and twelve reactors for 700 ats, 4 reactors for 200 ats, and 16 low pressure reactors have now been installed; 2 reactors for 2000 ats. will be completed in the near future.

In the experiment on the formation of acetic acid it was not possible to suppress the formation of water. Autoclave experiments with dimethyl ether at 3000 ats. gave about the same results as with methanol (20 - 30 per cent acetic acid).

The hydrogenation of dimethylolpropionaldehyde (P_2) to trimethylolethane over copper - magnesium oxide and copperchromite catalysts at 700 ats. could be sucessfully carried out with a yield of 75%; however, these catalysts are not yet sufficiently stable to loss by attrition and their efficiency decreases too rapidly.

(Page 46 Contd.)

The manufacture of propionic acid from propyl alcohol and aqueous NaOH could be carried out continuously at 200 ats and about 350°C. Yields up to 85% could be reached. A semi-technical experiment is under preparation.

Experiments on the manufacture of dimethylamine did not as yet show satisfactory results.

<u>Work planned for 1940:</u>

Continuation of the experiments on acetic acid. Two new silver lined reactors for experiments with phosphoric acid are being installed.

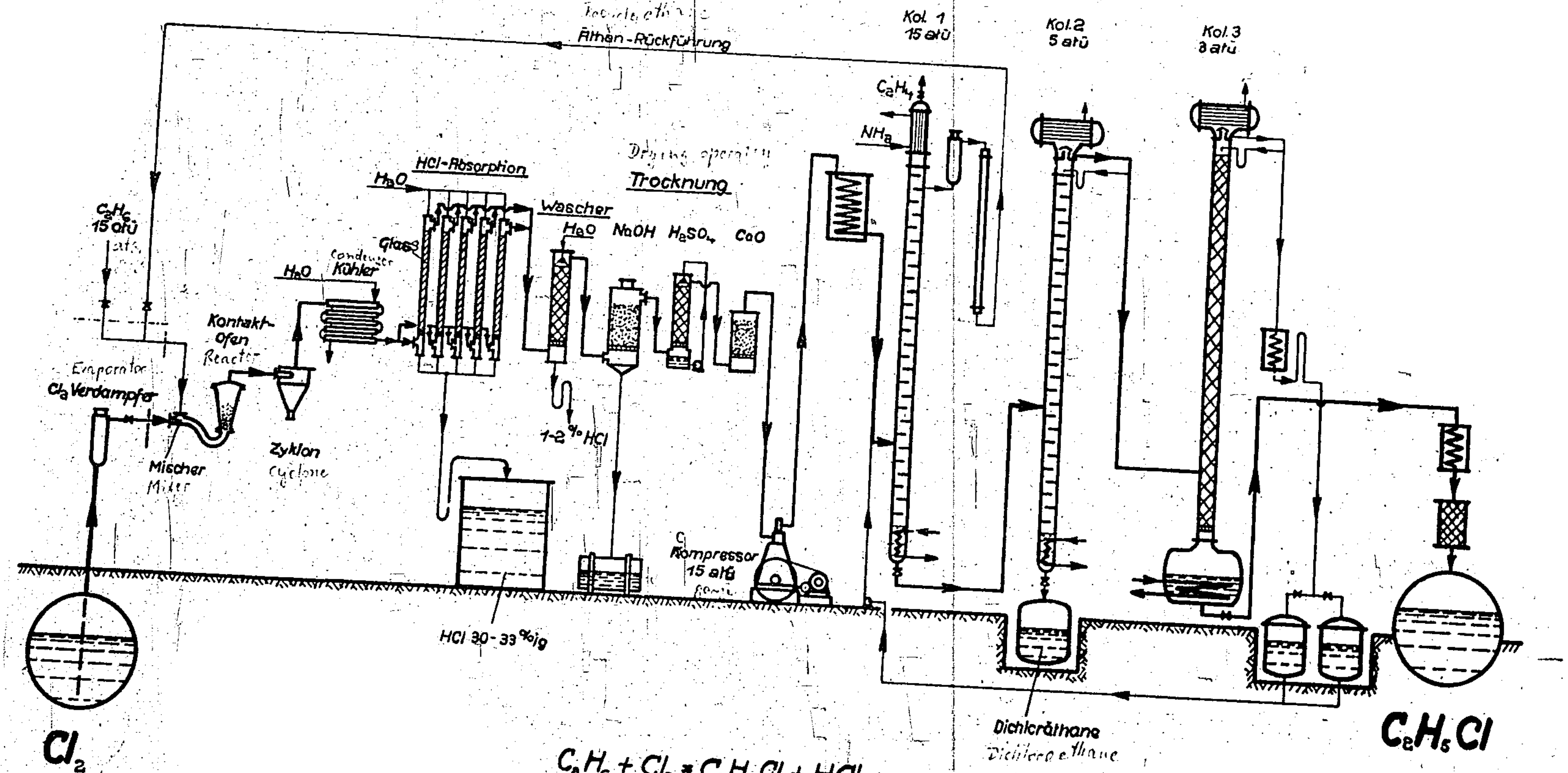
Investigation of the formation of ethyl alcohol in relation to pressure and CO concentration.

Experiments on construction materials for syntheses at very high pressure.

Manufacture of ethylchloride

Chloräthyl - Fabrikation.

Herstellung von Chloräthyl.



$$C_2H_6 + Cl_2 = C_2H_5Cl + HCl + 37\,\text{kcal.}$$

47-9

Ethyl Chloride Plant

The HCl absorption unit made at Oppau was damaged by corrosion to such an extent that it had to be replaced. The new unit consists of 5 parallel glass tubes of a diameter of 70 mm. and a length of 12 m. which are packed with Raschig rings. The same efficiency can also be reached by using only two glass tubes with a diameter of 150 mm. but these tubes are not sufficiently safe in operation because of manufacturing difficulties at Schott, Jena, resulting in internal stresses in the glass tubes. The HCl obtained by absorbtion has a concentration of 30 to 32% and can be sold as technical HCl. The on-stream time of the Oppau circular reactor with chromium-nickel wire netting as catalyst was increased to 4 to 5 weeks by changing the heat exchangers, the catalyst space and the method of heating.

After several experiments a new reactor was put in commercial operation at Leuna in which the reaction of ethane and chlorine takes place on "dancing" carbon in accordance with the process developed on the pilot plant scale by Dr. Herold. The important feature of this funnel shaped reactor is the carbon separation by means of a cyclone which is arranged behind the reactor. The advantages of the new reactor as compared to the Oppau circular furnace include better yields, higher thru-put and savings in fuel gas because the reactor can be operated without supply of outside heat. The capacity of the plant was increased mainly through the new reactor from 25 to 60 - 70 tons per month.

The quality of the ethylchloride was improved by removing 5 to 10 percent of a preliminary distillate which is recycled. This distillation was made possible by the installation of an additional column. In spite of this improvement I. G. Gapel had difficulties in processing this ethylchloride since an undue pressure rise occurred during the reaction with lead-sodium alloy. The degree of purity of the ethylchloride lies between 99.5 and 99.7% and it must be investigated

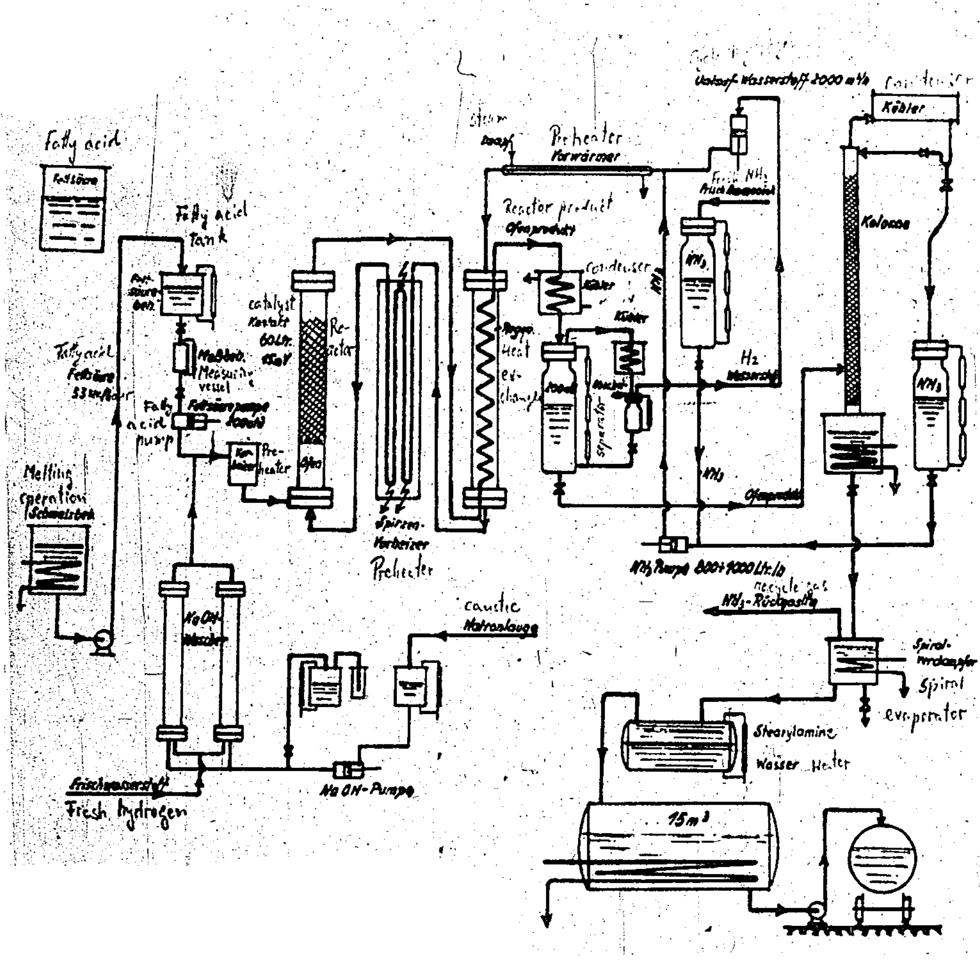
(<u>Page</u> <u>47</u> <u>contd.</u>)

what substances are present in the remaining 0.5 to 0.3%.

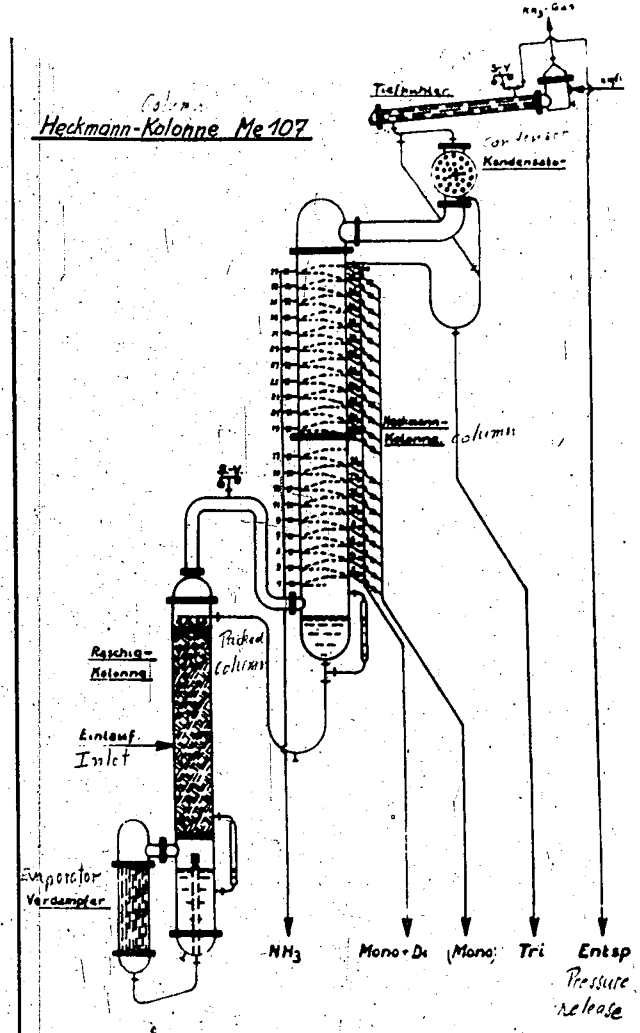
<u>For</u> <u>1940</u> it is planned to raise the capacity of the plant to 120 tons per month which will require enlarging the reactor and the HCl absorption plant as well as the installation of a new compressor.

Unit for the manufacture of stear[illegible]

Anlage zur Herstellung von Stearylamin in Me 13



Column
Heckmann-Kolonne Me 107



Plant for the Manufacture of Amines

A new column with 40 plates has been installed. A column packed with Raschig rings is arranged ahead of the new column; in the lower part of this packed column the reactor product is dehydrated whereas the separation of the mixture of mono and dimethyl amines from ammonia and the ammonia-trimethylamine azeotrope takes place in the upper part.

Since packing with iron bodies lead to frequent clogging of the dehydration columns due to sludge formation this packing was replaced after some experimentation by ceramic Raschig rings which are not attacked.

Since the demand for dimethylamine, especially for Alkazid solution, was very great during the year and since the necessarily produced monomethylamine could not be entirely disposed of, the latter was recycled instead of methanol.

In addition to the manufacture of monoisobutylamine the manufacture of diisobutylamine was taken over from the pilot plant. Experiments on the recovery of trimethylamine by washing the azeotropic mixture of trimethylamine and ammonia with NaOH solution were continued.

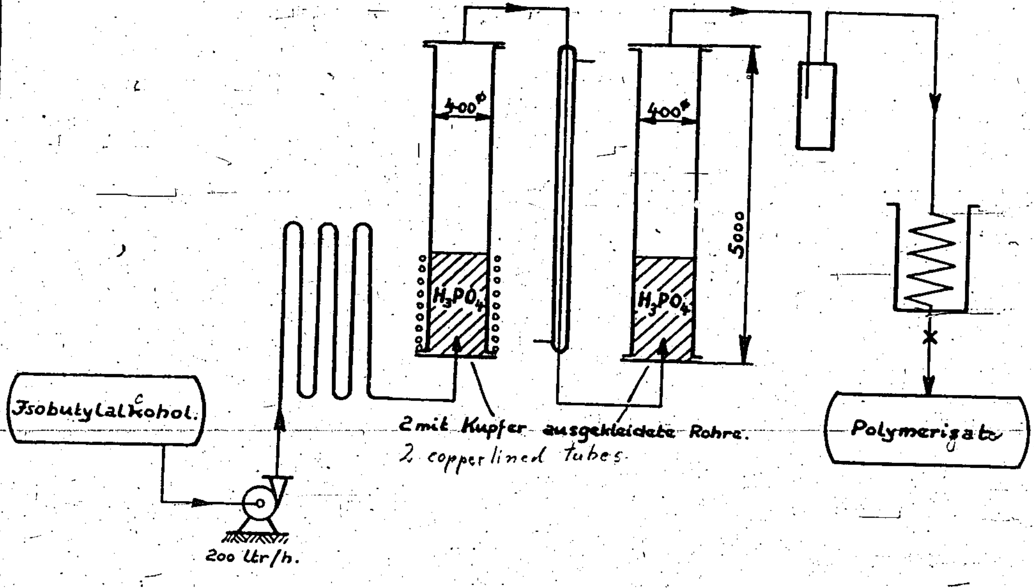
A larger unit for the manufacture of stearylamine was installed and started up. The product obtained does not quite come up to specifications and must be improved by changes in the catalyst.

Dehydration and Polymerization

Wasserabspaltung und Polymerisation.

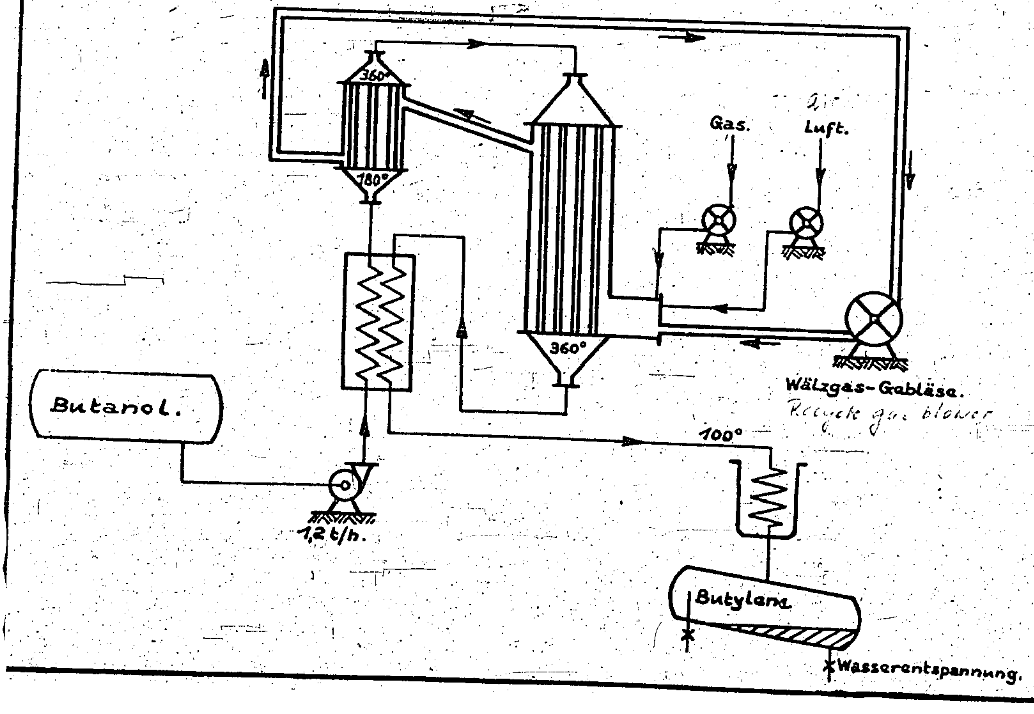
(in einem Arbeitsgang.)

in a single step



Gas fired Dehydration reactor

Gasbeheizter Wasserabspaltofen.



Manufacture of Isooctane

Dehydration of isobutyl alcohol:

As in the previous year the Oppau catalyst 695A is still in use. The lifetime could be raised to 2 to 3 months. A wash tower using warm water under pressure was installed for the recovery of unreacted isobutyl alcohol (1 to 2% of the amount injected) and isobutylaldehyde (0.5% of the amount injected) which are yielded together with higher hydrocarbons (1 to 2% of the amount injected) in the isobutylene column. In this way 80 - 90% of the unreacted alcohols are recovered.

Polymerization of isobutylene:

A fourth reactor was installed to increase the capacity so that the production of the polymerization plant now amounts to 8000 tons of isooctane per year. The lifetime of the activated charcoal catalyst has reached 3 to 4 months. The asbestos catalyst intended for the T52 plant was able, at the same lifetime, to give better polymerization of n-butylene and isobutane. Since April triisobutylane is hydrogenated together with diisobutylene and delivered as ET 110 so that further experiments to improve the ratio of diisobutylene to triisobutylene are at present unnecessary.

Cracking of triisobutylene:

Triisobutylene cracking was discontinued in March. The stock of triisobutylene will be slowly blended up.

The reactors which were used for the cracking process have been changed to permit the manufacture of amylene from amyl alcohol and the manufacture of n-butylene from the sec.butyl alcohol. During the rest of the year the reactors were used for the dehydration of isobutyl alcohol whereby the capacity of the dehydration unit was raised to 8000 tons per year of isooctane.

Hydrogenation of diisobutylene:

By separating the two hydrogenation reactors into two completely independent units the capacity of 10,000 - 11,000 tons of isooctane (ET110) per year was reached. It was found that up to 30% of triisobutylene in the diisobutylene can be hydrogenated completely and without decomposition at the previously used hydrogenation temperatures.

Losses:

Losses by leakage amounted to about 4% during 1939, whereas the losses due to the formation of by-products (isobutyl alcohol, isobutylaldehyde and higher hydrocarbons) amount to 2 to 3% and the losses by the formation of tetraisobutylene come to 0.5% of the alcohol charged. By the installation of the warm water washer 1.5% (isobutyl alcohol and isobutylaldehyde) are recovered, hydrogenated in an after-hydrogenation apparatus at atmospheric pressure and returned to the operation.

Production and delivery:

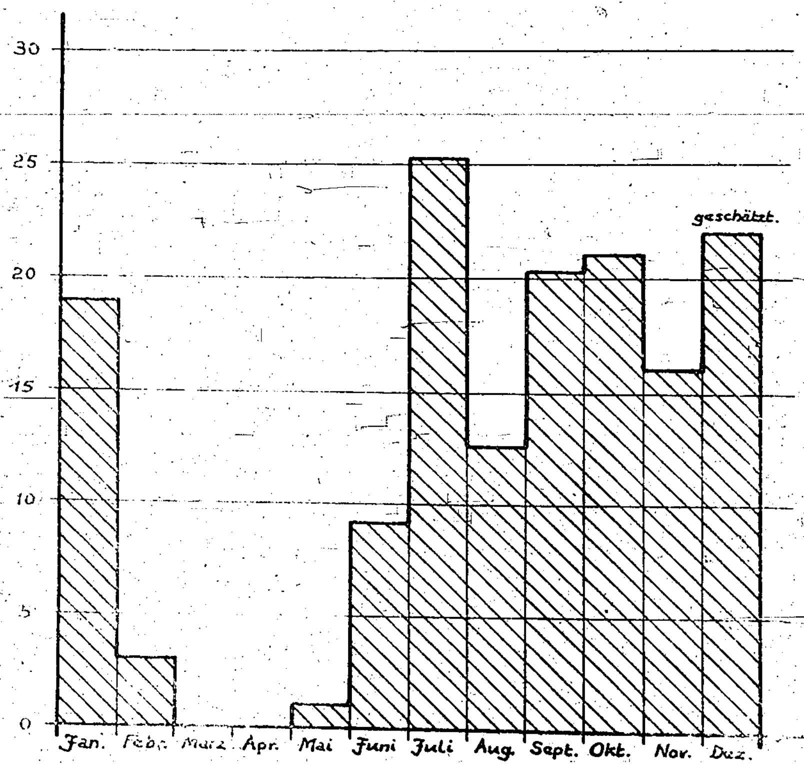
The production of isooctane (ET 110) amounted to about 6000 tons in 1939 of which 200 tons came from the T52 plant. 5900 tons were delivered. The total production since operations were started in the fall of 1937 has now reached 10,000 tons.

Work planned for 1940:

On the basis of preliminary experiments which were carried out at Ürdingen a new process is being investigated which permits to combine dehydration and polymerization into a single process. Experiments today indicate that this process is promising.

Production of Leuna carboxylic acids

Produktion an Leuna-Carbonsäure 1939.



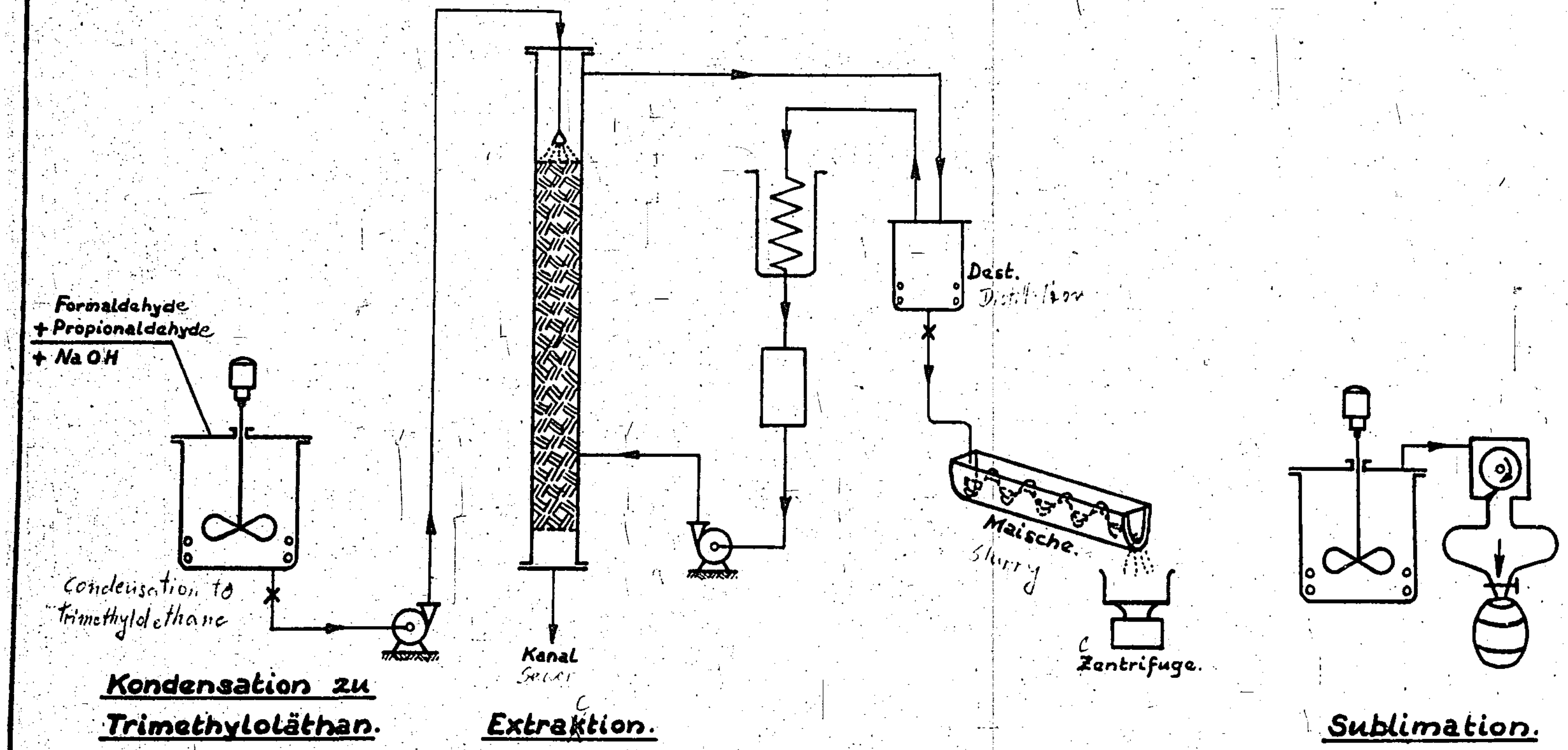
Manufacture of Leuna Carboxylic Acids

The plant was only periodically operated. During 1939 193 tons of acids were delivered to Griesheim.

Since a request for the supply of isovaleric acid was received, the conditions for the manufacture of this acid from the amylalcohol fraction was worked out in the organic laboratory and 8 tons of this acid were made in the plant. Acids of lower molecular weight were made by the same process partly in the laboratory and partly in the plant.

Herstellung von Trimethyloläthan nach dem Extraktions=verfahren mit anschließender Sublimation.

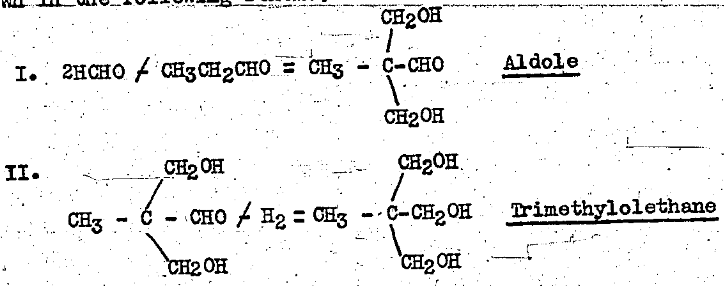
Manufacture of trimethylolethane by the extraction process with subsequent sublimation



Manufacture of Trimethylolethane and its Ester

With the equipment on hand 5-6 tons of trimethylolethane were made which were sent mainly to Ürdingen to be tried out for the manufacture of alkyd resins. Since the interest for the product increased the apparatus was detached from the carboxylic acid plant and enlarged. A condensation vessel and a centrifuge were installed and this equipment is sufficient for the production of 20-30 tons per month. The remaining equipment has either already been installed or is under construction.

A new process by which trimethylolethane is made from two mols of formaldehyde and hydrogenation of the aldole formed has been considered in the laboratory. The new process is shown in the following scheme:



$$\text{I.}\quad 2HCHO + CH_3CH_2CHO = CH_3 - C(CH_2OH)_2-CHO \quad \underline{\text{Aldole}}$$

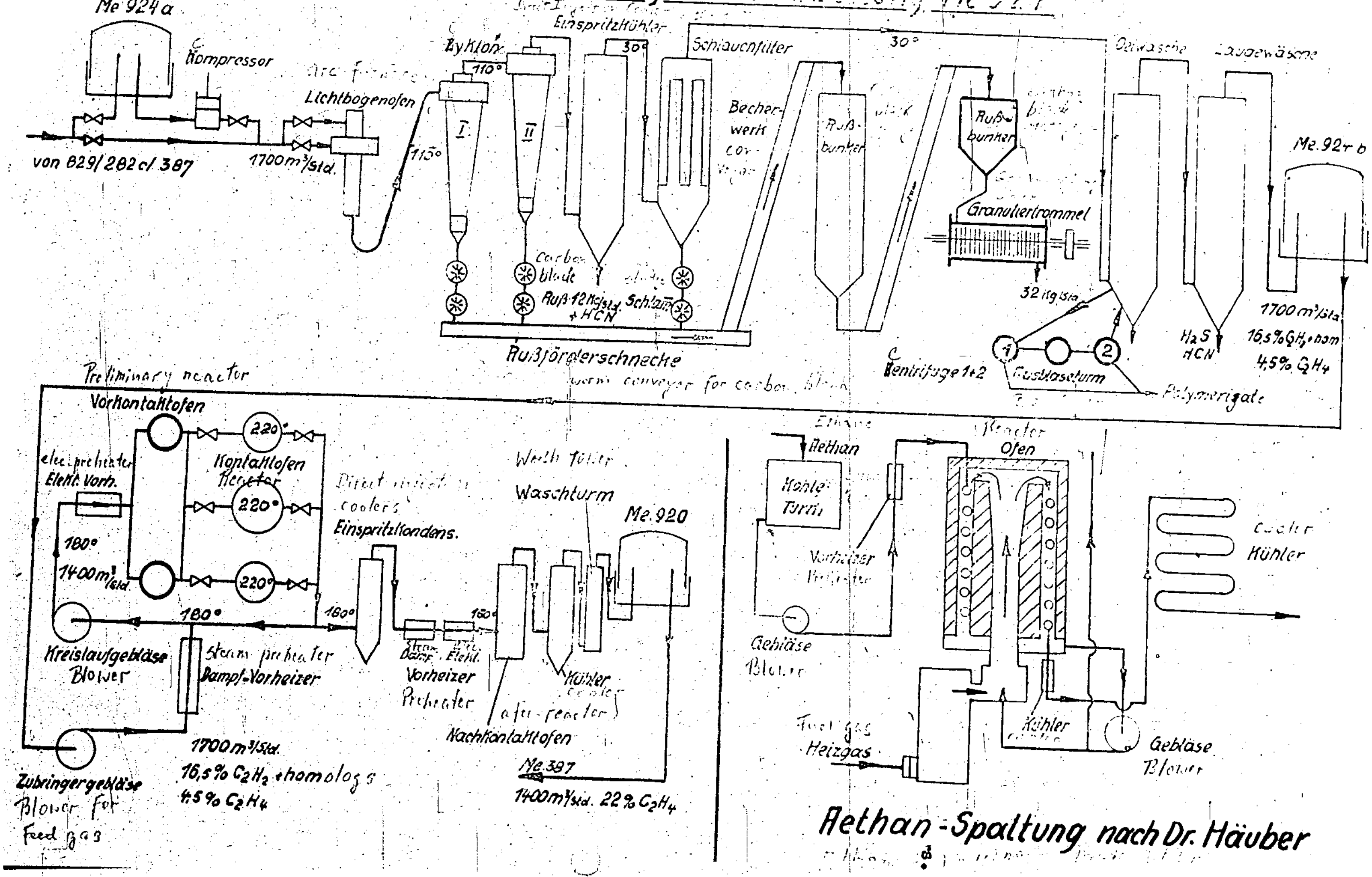
$$\text{II.}\quad CH_3 - C(CH_2OH)_2 - CHO + H_2 = CH_3 - C(CH_2OH)_2-CH_2OH \quad \underline{\text{Trimethylolethane}}$$

Further work has been done on the preparation of esters of trimethylolethane with Leuna acids in close contact with the testing stations for special lubricating oils. A plant for the manufacture of these acids would be necessary if the tests are satisfactory.

Work planned for 1940:

In the enlarged plant which should be started in February with a capacity of 30 tons per month the crude product shall be extracted from the aqueous solution by means of a solvent and be further purified by sublimation. (See diagram)

Schema der Acetylen-Verarbeitung Me 924



Aethan-Spaltung nach Dr. Häuber

Processing of Acetylene

On the average 200 tons of ethylene per month were made by hydrogenation of acetylene. The raw material for cracking in the electric arc furnace consisted of high-concentration gas from the hydrogenation, propane, butane and residual C_3 gas from the Linde plant in mixture with low-concentration gas from the hydrogenation. Major operating difficulties were due, as before, to the formation of carbon black. It was possible to convert the carbon black which is separated by a cyclone ahead of the Beth filter into a commerical product suitable for printing purposes; this is achieved by treating the carbon black in a granulating drum with rotating beaters. The apparent density was lowered from 0.03 kg to 0.25 kg per liter. After outbreak of the war the carbon-black business does not seem very promising.

An improved regeneration process for the wash oil was introduced (the wash oil is used for the removal of polymerization products formed in the electric arc). The innovation consists in heating the oil containing the polymerizate to 180 to 200°C until solid polymerizate separates. After cooling the polymerizate is centrifuged off and the wash oil is again ready for use. This improvement in the oil regeneration results also in a better purification of the arc gases.

The installation of small preliminary reactors filled with catalyst has proved very valuable in the acetylene hydrogenation. These small reactors can be easily opened and reassembled if the catalyst loses activity or becames contaminated. These reactors together with the above mentioned improved purification of the gas by washing with oil increased the on-stream time of the main reactors considerably, viz., from an average of 45 days to 65 days.

At the beginning of November a Häuber-Hirschbeck reactor was put into operation. In this process ethane is passed with a speed of 35 m/sec. at 0.6 ats pressure through a coil heated to 800 degrees C; the coil is made out of the special material FF30.

The exit velocity is 130 m/sec. because of the increase in volume by cracking and heating. The cracked gas is passed into the Linde unit after cooling. The advantage of this process as compared to the Klein process consists in the fact that no oxygen is used for cracking and therefore no CO and CO_2 is formed. This eliminates the Alkazid and caustic wash ahead of the Linde unit. The ethylene yield is more favorable than with the Klein process. The Häuber reactor has been in operation for several weeks without interruption.

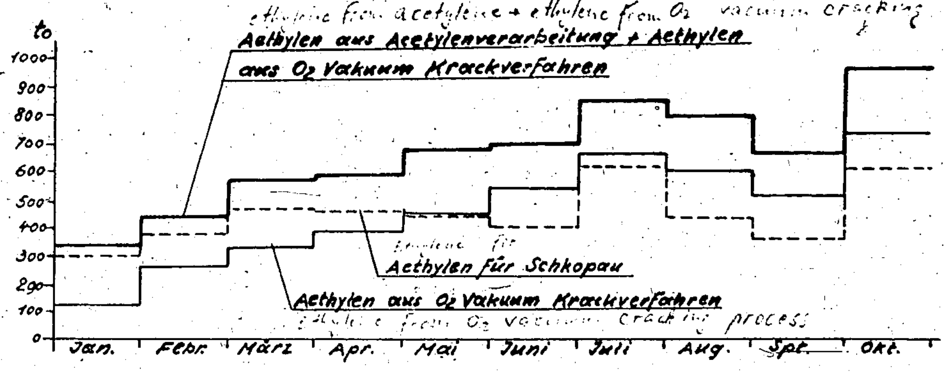
After the installation of this reactor the manufacture of ethylene by the electric arc process will no longer be necessary since this process is more expensive than either the Klein or the Häuber process.

Work planned for 1940:

Expansion of the cracking unit (Häuber-Hirschbeck) to 3 units with 2 coils. Installation of a copper and oil wash unit for this cracking plant for the concentration of ethylene and the recovery of unreacted ethane.

Monthly ethylene production

Monatliche Aethylen-Produktion.



Flow sheet of C_2H_2 after-hydrogenation

Schema der C_2H_2 Nachhydrierung

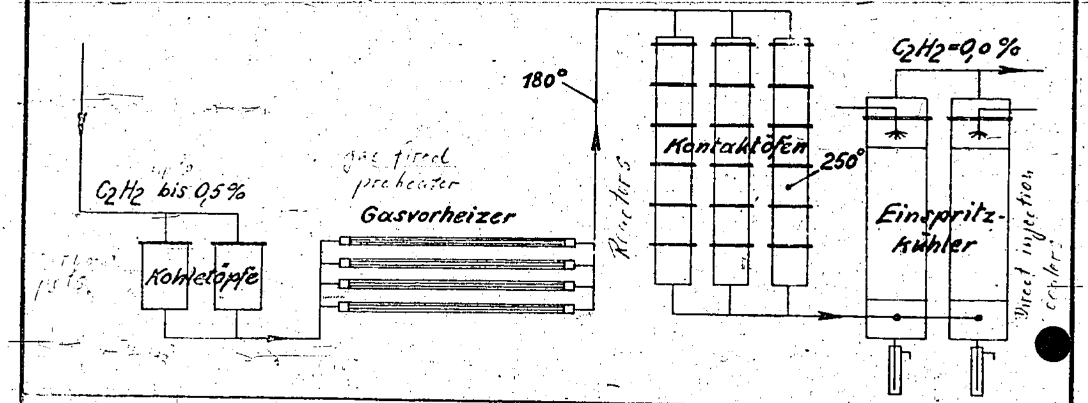
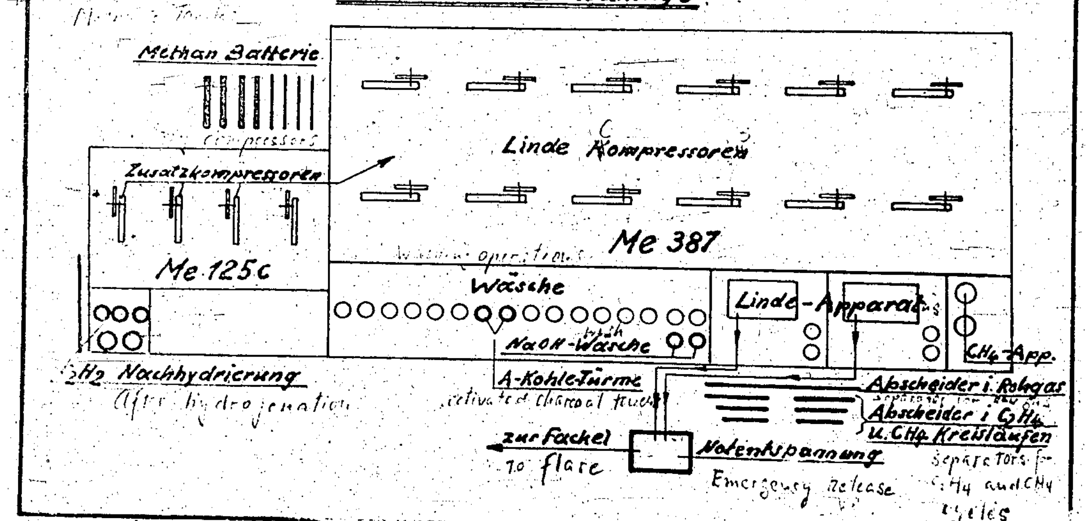


Diagram of the Linde unit

Schema der Lindeanlage



Processing of Ethane

Cracking Plant:

Production was increased proportionally to the amount of ethane or propane supplied by the hydrogenation plant. By the middle of the year all four units were in operation. The capacity of the reactor could be raised to 1100 m^3/hour of cracked gas. This increase in capacity required the enlargement of coolers and pipes as well as an increase in the gas holder pressure to overcome the back pressure in the lines. The cracking furnaces are on stream on the average for 9 months before they must be cleaned from coke. This indicates the success of the careful design of the gas distribution in the furnaces. By improving the control of the furnaces the undesirable formation of CO could be reduced from 12 to 10% and the oxygen consumption could be lowered from 26 to 22%.

Linde Plant:

Various parts of the washer unit for the preliminary purification of the cracking gases had to be expanded. Two new activated charcoal towers and two new caustic towers were installed. The main difficulty in the washing step still consists in foam formation in the Alkazid towers. A change in the plate design of the wash columns seems to improve this condition. To protect the Linde unit from carried-over foam a large raw gas separator has been incorporated into the unit. For the methane and ethylene cycles of the two Linde units large separators for entrained lubricating oil have been installed. By these measures the operating period of the Linde unit has been extended to 3 to 4 months.

An acetylene after-hydrogenation unit has been tentatively installed to remove the acetylene from part of the raw gas and this process has been fully successful. This unit works according to the proposal by Häuber-Hirschbeck with a selective catalyst which only reduces acetylene to ethylene but does not hydrogenate ethylene to ethane. In this way the acetylene content of the raw gas is reduced from 0.3-0.4%

to quantities which can no longer be detected.

The entire ethylene production at Leuna is at present 10,500 tons per year.

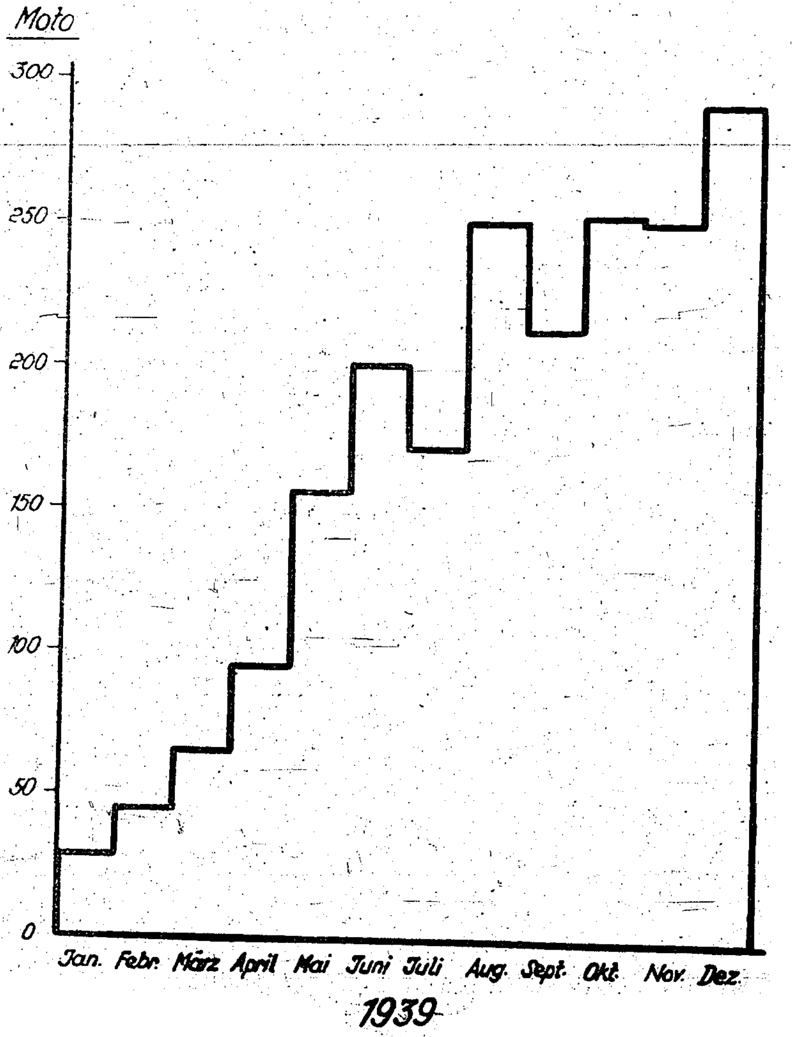
Ethylene of 97% purity is supplied to Schkopau. The yield in the Linde unit is about 90%.

The Linde apparatus for the manufacture of methane for the methane cooling cycle filled all requirements. The excess of pure methane was sold for chemical purposes in pressure tank cars and cylinders.

Provisions are being made for emergency cases to remove the liquid hydrocarbons as fast as possible from the unit and introduce them into a flare after gasification. Furthermore, a water sprinkling system for the Linde unit proper was installed. These measures were taken after a fire in July.

[illegible] SS 104

Monatliche SS-Produktion 1939



Manufacture of Lubricating Oil From Ethane

The expansion of the lubricating oil plant from a production of 700 tons per year to 3600 tons a year took place in several stages beginning in April and a production of 250 tons per month was reached in August.

The two 500 type stirrer autoclaves were replaced by four N6 autoclaves 800 x 8000. The Hösch stirrers which are inserted from the bottom were kept and proved to be satisfactory in the big autoclaves. The reaction time of 8 hours remained unchanged.

The experiments to continuously remove the catalyst sludge after the polymerization by means of a centrifuge were concluded and lead to the following operating method: The autoclave contents are released hot over a period of two hours into a scraper centrifuge and the oil which has been purified in this way is finally transfered into the decomposition tank via a Laval separator.

The $10\ m^3$ vessels with cone bottoms which were used in the small plant as settlers are now utilized as decomposition vessels. The decomposition process which up to now was carried out with caustic methanol has been improved by changing to a treatment with methanol followed by neutralization with a small amount of ammonia. A considerable improvement could be achieved by carrying out the treatment with methanol and ammonia immediately after polymerization prior to centrifuging of the sludge. In this way the amount of sludge removed in the centrifuges is increased and the filters behind the decomposition vessels proper are very much relieved. Difficulties with filtration which used to be very frequent and lead to time consuming interruptions have since decreased to a point where they are unimportant.

The distillation unit had a sufficent capacity since the unused vacuum column will be operated at atmospheric pressure. In case that the Air Ministry should require delivery of SS 903 which would make it necessary to start up the depolymerization plant an expansion of the distillation unit would be necessary.

The final treating process has not been changed. SS 906 has been shipped without inhibitor in tank cars of the Wifo.

It has been decided to expand the lubricating oil plant in 1940 to a production of 10,000 tons SS 906 per year.

Laboratory and Pilot Plant for the Organic Department

Work on lubricating oil

Experiments were made on the preparation of oil blends. SS 906 with a viscosity of about 6°E at 100°C was blended with four different natural lubricating oils in the ratio 1:1. The four blends were tested in aviation engines after the addition of an inhibitor. It was found that all these blends in all engines were better than the aviation oil from natural petroleum which is used at present.

By polymerization of propylene with $AlCl_3$ a product was obtained which proved to be extremely well suited for the coating of cable insulating masses as indicated by tests at the Siemens-Schuckert Cable Works in Berlin. The electrical properties, the good resistance to oxidation as well as the viscosity-temperature characteristics of this material are far superior to the products used up to now (colophonium dissolved in mineral oil). Work is being done on the improvement of this product.

It was found that the inhibitor used up to now, diisobutylphenolsulfide, can be considerably improved in its activity by replacing the hydrogen of the hydroxy group completely or partly by tin. The tin containing product is about 10 times as effective as the unsubstituted product. Further development work is being done on the inhibitor to increase its efficiency.

Work on motor fuels

Work was being done in conjunction with the isooctane plant to improve the ET 100 fuel. Development work is under way on a substance to lower the ignition temperature of Diesel oils and on an ignition oil for the combination Otto-Diesel engine.

Work on amines

It was attempted to prepare morpholin which is used as catalyst for condensation reactions from $\beta\beta'$- dichloroethylether and ammonia. These experiments were

(Page 55 contd. and
Page 56)

stopped for economic reasons. Condensation of morpholin or piperidine with crotonaldehyde led to products which interested Leverkusen as accelerator for the vulcanization of rubber.

Work on the condensation of aldehydes

Work has been continued on the preparation of trimethylolethane (P_3) by condensation of 1 mol propionaldehyde with 3 mols of formaldehyde with the simultaneous formation of sodium formate. For the realization of a new process based on the reaction of 1 mol propionaldehyde and 2 mols of formaldehyde the corresponding aldole has been prepared and also the reaction products which are obtained on hydrogenation. The condensation of isohexyl and isoheptylaldehyde with formaldehyde to dimethylolhexane and dimethylolpentane has been further developed. About 2 tons of C_{12} and C_{14} glycols which are useful as anti-foam agents have been made from isohexyl and isoheptyl aldehyde. A small plant with a capacity of about 8 tons per month is under construction. Work is under way to improve the C_6 and C_7 acids which are formed in this reaction.

The process developed by Dr. Zorn at Oppau for the manufacture of higher molecular unsaturated aldehydes (Polyenealdehydes) from crotonaldehyde by means of organic catalysts has been further developed, and also the processing of the hydrogenated polyenealdehydes to saturated or partly saturated alcohols and the oxidation to fatty acids.

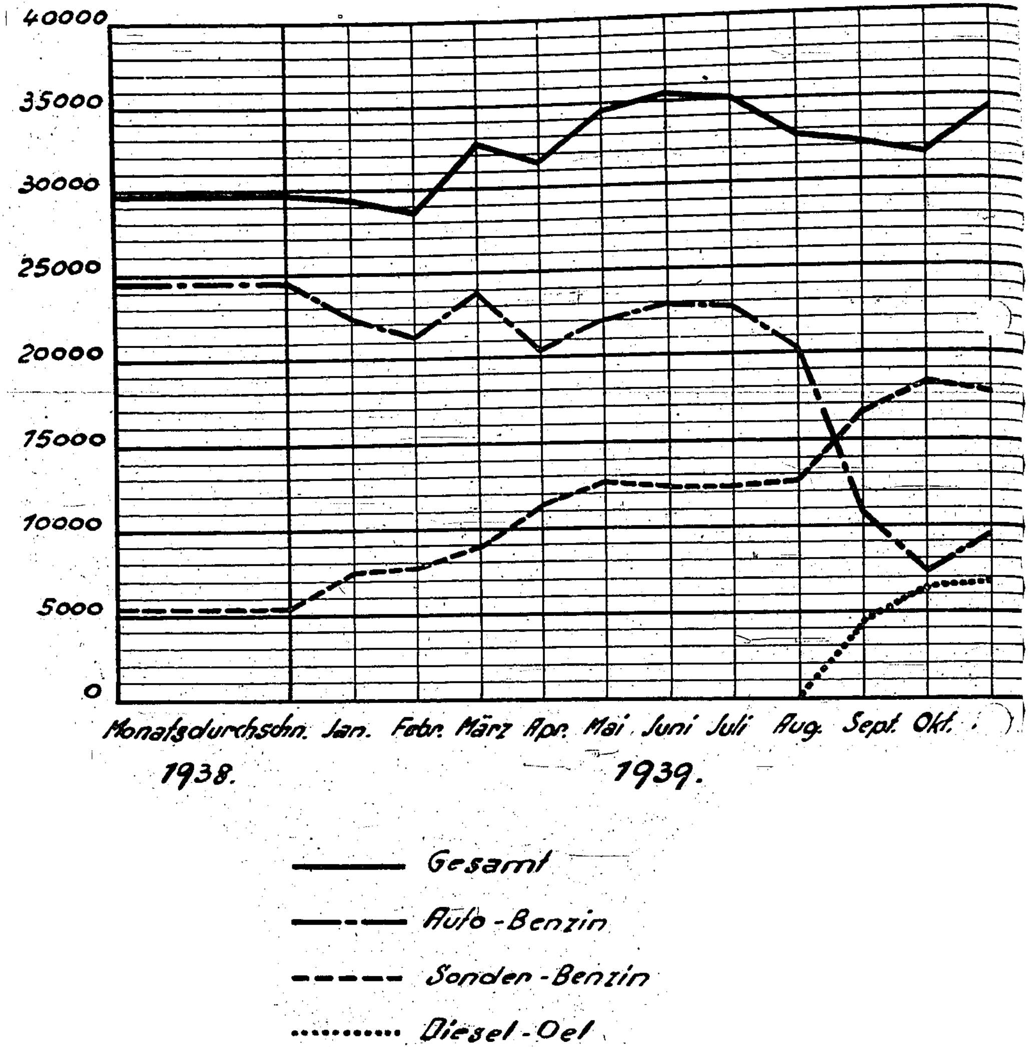
Acids

The best conditions for the manufacture of isovaleric acid from isoamyl alcohol and for the acid from the isobutyl oil fraction 140-190°C. have been worked out. The laboratory procedure to oxidize an aqueous propyl alcohol solution with NaOH to propionic acid has been utilized to work out a continuous process.

(Page 56 Contd.)

Esters

Esters of polyfunctional alcohols with Leuna acids have been prepared and tested as to their lubricating oil properties. This field has been considerably expanded by including blends with hydrocarbon oils, the use of acids from the oxidation of paraffin wax and glycols furnished by other plants. Many samples have been prepared and tested and important data on the relation between lubricating characteristics and structure have been obtained.



Benzin- und Dieselöl-Erzeugung.

(Page 57)

Hydrogenation

Production of hydrogenation plants in 1939 showed appreciable progress over 1938 and significant changes in the type of product.

Production rose by 9.6% from 358,000 tons to 393,000 tons. While in the previous year aviation gasoline represented only 17.9% of total production, in 1939 it rose to 39% of the increased production. Also products made from coal rose from 61.8% to 68.5%. Since outbreak of war manufacture of motor gasoline became secondary. Diesel fuel, whose production was resumed with the outbreak of the war, and aviation gasoline were made experimentally during December. Thanks to precautionary measures, which were prepared long in advance, these conversions could take place without significant difficulties.

With the stoppage of butylene experiments and the removal of Dr. Baehr, the "Research Group Dr. Baehr" was dissolved in May. Work carried on in Leuna was reported under "Labo 907" and "technical experiments," by plant group dealing with low pressure.

Among the licensed firms Magdeburg and Boehlen operated without incidents; at Zeitz-Troeglitz gasoline and Diesel fuel are prepared by low temperature hydrogenation. The paraffin wax and lubricating oil production will start up later. The Luetzkendorf pressure hydrogenation plant is now being started up. Latter will process petroleum middle oil for the time being.

Regarding coal hydrogenation plants, which are located mainly near Ludwigshafen, Scholden operated satisfactorily; at Welheim coal tar pitch was hydrogenated instead of coal extract as originally planned; newly started up was the coal hydrogenation plant of the Gelsenberg-Benzin A.G.; during the first half of 1940 the plant at Stettin-Poelitz will start up which will process upper Silesian coal in addition to petroleum residue.

The hydrogenation plant at Brux which will produce 600,000 tons of motor fuel from low temperature carbonization tar, and the plant of the Union Rheinische

(Page 57 contd.)

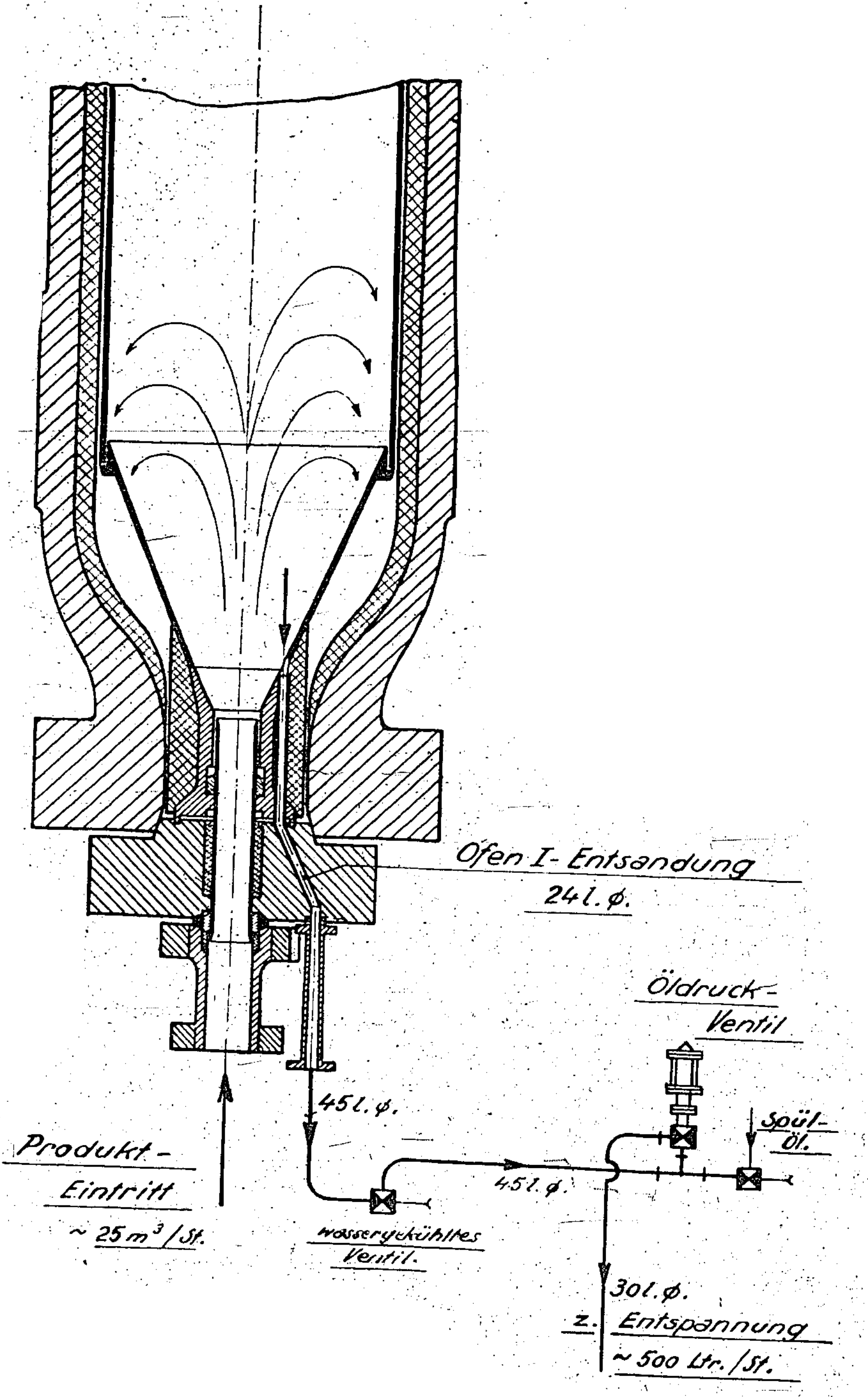
Braunkohlen-Kraftstoff at Wesslig are under construction. Further plants in the east are planned.

Both Italian plants at Bari and Livorno are now back in operation after initial difficulties.

Members of "hydrogenation" took significant part in the planning, erecting, and starting up of plants at (report goes on mentioning names of places and persons).

Personnel of "hydrogenation" have spent 26,000 man-days in "other" plants. For most of the plants mentioned above over 200 men were trained during 15,500 man training days.

Schema einer Ofen I - Entsandung.



HYDROGENATION

Operation of Reactors

a) Coal reaction chambers

Work in 1939:

Single runs of coal reaction chambers were raised from 280 days maximum to 339 days. By adjustment of temperature for optimum conditions high charge rates of 23-25 cubic meters per hour per reaction chamber could be maintained. It was possible to maintain experimental raising of charging rates at 27 cubic meters per hour for some chambers. In spite of this increase in charge rates and length of runs no increased damage to catalyst pipes or insulation were observed.

By means of introduction of "reactor-I-sand removal" coarse sand and solid coal particles were removed from the reaction system and "Caviar" formation, which was previously quite pronounced, was prevented thereby. It is expected that the retarding influence of sand and Caviar will be avoided. In addition to increasing length of runs of reaction chambers this method also permits stopping of coal reactors within short time and without consequent repair of reactor-I. This type of "sand removal" was built into six reactors and has been in operation for 175 days in one unit. Material removed by the "reactor-I-sand removal" scheme contains twice as much mineral matter as the normally removed slurry and is distilled along with the latter.

Automatic level control has proved satisfactory without exception in the units equipped with slurry separators.

The immersion coolers built into the slurry removal system after the high temperature separator is not yet satisfactory. An improved design will shortly be installed.

Local overheating damaged several gas preheaters. Brickwork was replaced and repaired and the use of blanketing gas was introduced.

On a semi-plant scale tests were made at 650 atmospheres regarding deposit formation in preheaters and its prevention by use of Rhineland brown coal.

Work for 1940:

1. Further experiments to increase charge rates and length of runs along with study of asphalt formation in reactors.
2. Installation of automatic level controls.
3. Improvement of the "reactor-I-sand removal" system and installation into all reactors.
4. Continuation of experiments with immersion coolers because they are easier to galvanize and to repair than the customary double coils and are cheaper besides.

b) Tar reactors

Work in 1939:

Instead of the customary use of two tar reactors per four furnaces, the use of one tar reactor per six furnaces was started up. Charge rates up to 30 cubic meters per hour were obtained using tar and coal "heavy" oil as a charge. Pure tar "heavy" oil would permit higher charge rates still. . . .

Work for 1940:

Replacement of furnaces or reactors of "three parts" design as they are required elsewhere for high pressure plants.

c) Gasoline reactors

Work in 1939:

Reactors with catalyst No. 5058

Attempts to reduce ammonium chloride deposition ana consequent high resistance to flow by means of changing hook-up of regenerators were without success. After thorough tests and calculations it became possible to inject more water more effectively by installing water sprays above the upper tube bottoms of regenerator II and beneath tube bottoms of regenerator I. The length of runs on regenerators II was extended from 3 months to 6 months thereby, after which time an increase in resistance to flow became noticeable.

Reactors with catalyst No. 6434

In all units sulfurizing with hydrogen sulfide was introduced which prevented the dirtying of outsides of reactor bundles. Sulfurizing with elementary flowers of sulfur was discontinued. . .

Based on successful experiments the new gasoline reactor is equipped with a water washing tower for the removal of last traces of ammonia from the charge gas. A substantial increase of yield and life of catalyst is probably attainable thereby. . .

For the warm up and starting up of the 6434-reactors a special gas flow cycle was set up. . . Decomposition of catalyst through unavoidable water condensation at time of start can be avoided thereby.

As of September 7 production was converted to war needs, motor gasoline, aviation gasoline and Diesel fuel being manufactured. The conversion to war production proceeded without a hitch.

(Page 59 contd.)

Work for 1940:

1. Continuation of tests to increase throughput of 5058 reactors by reduction of resistance to flow. . .

2. Reduction of pressure drop in the clean gas cycle through installation of a separator. Two-unit or three-unit parallel hookups can be tied in with clean gas cycle thereby. At the same time (consideration is given to) installation of a by-pass of charge gas to clean gas cycle whereby the bottleneck at the entrance to the washer is avoided. A 25% increased yield of 5058 reactors could be anticipated thereby.

d) T.T.H. Reactors (low temperature hydrogenation)

Work in 1939:

Tests continued. The reactor was used for the time being as prehydrogenation chamber and could thereafter again be operated as a low temperature hydrogenation reactor (L.T. reactor) without difficulty. Latest tests showed reduction of length of runs due to build-up in resistance to flow. Reason therefor was dirtying of regenerators and first layers of catalyst with tar polymers.

The products of L.T. reactors were sent to the dewaxing plant for further processing.

Tests with circulating catalyst 3510 were terminated. Space velocities of 0.66 were obtained thereby as against 0.45 when using fixed bed catalyst 5058. The end products are the same for both methods of operation. Evaluation of these results on large scale were no longer possible, as (plant) Zeitz was equipped with fixed bed catalyst since end 1938. Furthermore, the relatively high loss of catalyst in small scale equipment could not be investigated.

Work for 1940:

Tests to raise operating time of L.T. reactor are to be continued. This reactor furthermore must supply charging stock for the dewaxing plant.

e) Miscellaneous

Work in 1939:

The pressure seals of the de-slurrying equipment were changed. A drain to the pit was installed, so that loss of seal would not cause soiling of surroundings.

Tests with various corrosion preventive paints of cooler coils for de-slurrying were promising.

Furthermore, two more electrically controlled Ferranti gate valves were placed into the gas cycle to permit operation from a distance in case of accident. . .

Tests with highly errosian resistant material "Wigran" were satisfactory. However, this material could not be used owing to its cost.

As a result of increase in raw gas up to 110,000 cubic meters, and other reasons, copper solution was entrained. A trap was installed along with an automatic level indicator.

(Page 60 contd.)

Work for 1940:

1. Further investigation of anti-corrosive coatings for cooling coils and gas coolers.

2. Further testing of erosion-resistant materials and stuffing box packings.

3. Further use of electrically controlled gate valves.

4. Further change in de-slurrying.

5. Introduction of a second horizontal trap for separation of entrained copper solution from charge gas.

6. Change and enlargement of central pressure release in Me 809.

7. Investigation of substitutes for asbestos containing heat insulating materials. . .

(Page 61)

Slurry Pumps
Scrubbers
High Pressure Absorption Gasoline
Gas Circulating Pumps

a) Slurry Pumps

Work in 1939:

The new charging pumps for H_2S-containing middle oil went into operation; also a second pipeline for middle oil from the tank farm. In line with foreseen measures taken to assure increased charging rates, foundations were laid for two more electrically operated middle oil pumps. . . To avoid fouling of reagent, filter tests in middle oil B were carried out. Tests are not yet terminated. . . .

Work for 1940:

(Deals with mechanical features of plant operation)

b) Scrubbers

Work in 1939:

A nitrogen compressor for 1000 atmospheres was installed and is operated at present for tests with Rhineland brown coal using hydrogen at 200-700 atmospheres.

. . . Intermediate expansion pressure was raised from 25 to 38 atmospheres to increase ethane yields by furnishing more raw gas from scrubbing oil. Line pressures of scrubber oil charge pumps had to be raised by insulation of additional stage in high pressure centrifugals.

Work for 1940:

1. Reinforcing of both of last two expansion engines.
2. Removal of two scrubbers from gasoline cycle for use as separator in clean gas.

c) High pressure absorption gasoline.

Work in 1939:

Gases of expansion from de-slurrying (coal or tar), which are relatively rich in hydrogen, were taken out of sump-phase-rich gas and fed into lean gas.

. . . To raise throughput of the high pressure absorption gasoline plant the two scrubbers were hooked parallel. Furthermore, a new and more direct suction line to the gasometers was installed. . . A second gas cooler with higher capacity was also installed to permit occasional cleaning of one without disturbing the other.

(Page 62)

Work for 1940:

(Deals with mechanical plant detail.)

d) Gas Circulating Pumps:

Installation of new gas circulating pumps was delayed due to supply conditions and this seriously affected anticipated increases in production. . . (Balance deals with minor plant details.)

An all enclosed exhaust system for the expansion gases from the separator (of pumps and gas cycle) was started up to remove odor nuisances.

Work for 1940:

(Items 1, 2, 3, 5, deal with problems peculiar to local plant . . .)

4. (Study of) Added protection against entrained liquids.

e) General

Work in 1939:

(Here blackout and air raid protective measures were discussed.)

Work for 1940:

(Some more of air raid precaution.)

Small Scale Experiments Me 869

Work in 1939:

I. Small reactors:

Current examination of products and catalysts used in plant.

Examination of new catalysts and their suitability for plant. Experiments show that catalyst 7019, recommended by Ludwigshafen, rapidly loses its effectiveness when used for aromatizing of A-middle oil and that it will become necessary to add more active catalyst on the inert carrier material.

Tests to establish influence of water washing of circulating gases on life of catalyst 6434 were continued and it was established that water washing the gas (free of ammonia) prolongs catalyst life. Sensitivity of the fixed bed catalyst 6434 is especially high to ammonia. A reactor equipped with water washing will shortly start up in the plant.

Examination of catalysts of others. (Drabag is only one mentioned.)

II. One kilo reactor experiments at 200 atmospheres:

Control testing of products and catalysts of plant coal tar reactors.

Investigation of a "Quickie" hydrogenation ("Kurzhydrierung") especially to improve yields of the coal sump phase at expense of reduced asphalt decomposition, was conducted. Both anthracite and brown coal were investigated under conditions of increased charge rates. Slurry samples were sent to Dr. Baehr for briquetting and distillation experiments.

Examination of products from others for suitability in our plants. (Bohemian tar, petroleum and its residues)

Examination of tars from Zeitz. Hirschfelder tar was easier to hydrogenate than tar from Kulkwitz in spite of its higher asphalt and phenol content.

(Page 63 contd.)

Investigation of "dehydrogenating refining" of sump phase gasoline at 50 atmospheres and various catalysts in order to get highest possible octane numbers. Results to date not satisfactory and work is continuing.

III. One kilo reactors at high pressures.

Tests with Rhineland coal were more or less terminated inasmuch as we succeeded to establish causes for salt deposition and found ways to eliminate them. It was found that deposition was due to decomposition of humates and that preheater temperatures therefore should be kept preferably below 400°C. Furthermore salt deposition can be strongly repressed by additions; acidulating coal affects almost exclusively alkali- and alkaline-earth-humates-- while addition of sulfur to coal slurry only decomposes iron humates. A large scale test is in progress.

Tests with zinc-sulfur catalysts on our A-product did not arrive at the desired goal due to the failure to reduce phenol content and the tests are now extended to other catalysts.

Oil centrifuged from coal-unit cannot be hydrogenated at 200 atmospheres in the sump phase but could easily be converted to middle oil and gasoline at 700 atmospheres.

Work for 1940:

. . . . Further processing to gasoline and middle oil of the vacuum residuum from the oil centrifuged from the slurry. This vacuum residue appears similar to coal extract.

Investigation of salt deposition lately found to be objectionable in the coal reactors. Reproduction of formation, explanation of conditions leading to its formation. . .

Oil Processing Laboratory Me 870

Objectives:

The study and survey of plant operations and small scale experiments., and examination of products developed in the plant.

Work in 1939:

Tests were made in the plant in the following:

1) The high charging rates of 23-25 cubic meters could be maintained by adjustment of temperature changes in the coal reactors. Some reactors could be operated experimentally as high as 27 cubic meters charge rates.

Increase of average charge rate to 27 cubic meters is as yet not possible because the preheater coils have reached the limit of their capacity and are very sensitive to variations in asphalt content. An additional burden on the preheater coils was the rise of the ash content of coal and high solid content in the starting oil (Anreibeoel). The latter condition was caused by 100% centrifuging of slurry. Only 85-90% of slurry will henceforth be centrifuged.

An attempt to distill uncentrifuged slurry directly in preheaters led to increased down time for equipment and was discontinued for the time being. Further tests shall be made later to investigate distillation of slurry in a shell still.

2) Prehydrogenation over catalyst 5058 had to be conducted at low temperatures as the resistance in the reactors and the manufacture of Diesel fuel did not permit higher temperatures.

Catalyst 6434 attained a space velocity of not more than one ton per cubic meter per hour during 350 days. In order to meet the requirements for Diesel fuel attempts are made, contrary to war assignments to date, to make more Diesel at the expense of motor fuel without affecting aviation gasoline.

(Page 64 contd.)

3) Semi-plant scale tests with circulating catalyst from low temperature hydrogenation experiments gave space velocities of 0.66 and the same end products as fixed bed catalyst.

Small scale experiments with new gas phase catalysts from "Lu" (Ludwigshagen ?) showed that they were not suited for Leuna.

Attempts are under way to refine dephenolized sump over gasoline without loss in antiknock value.

4) . . . Because of its high sand and ash content coal from the mines at Tannenberg could not be used for hydrogenation.

5) Investigation of scale formation in coal reactors revealed that the thickness of the scale, especially in the lines between the first and second preheater, has increased during the last two years with increasing length of runs. Analyses showed that in addition to the (usual) calcium carbonate last year's (scale) also yielded increasing content of alkali- and magnesium salts.

6) An automatic device for the determination of ammonia in gases, which was suggested by us, was being tested for its suitability and may soon be ready for plant use.

7) Work in identifying phenols boiling above 220°C. was carried on and water from the coal separators was tested. Fractional distillation and crystallization yielded 40-50% of Resorcinol and methyl-resorcinol.

8) Development of a laboratory method for titration of H_2S . . . as substitute for iodometric titration method. . .

9) Plant observations show that initial fresh gas consumption per ton of coal varies by some 10-15%. Various attempts to tie these variations in with chemical composition of the coal have failed to date.

Laboratory Hydrogenation Me 907

Work in 1939:

1) Conversion of Fischer-gas oil (above 230°C.) and Fischer heavy gasoline (140-230°C.) into high antiknock gasoline by means of "gas reversion," gas oil cracking and naphtha reforming.

"Gas reversion" experiments with saturated gas oil components and oil from cracking were conducted during the last year and supplemented with gas oil cracking and naphtha reforming experiments.

The purpose of these experiments was to verify data by Kellogg by means of our own data and then to compare it with other processes to establish possible advantages of the "gas reversion" process. Working in accordance with the methods recommended for Leuna, i.e., use of two coils (pre-cracking of C_3-C_4 mixture) did not give indicated gasoline yields for given octane numbers. When working with single coil the indicated Kellogg yields could be confirmed only at low octanes of 60 motor method or less. The maximum knock rating reported by Kellogg of 69.5 motor method was never obtained. Comparisons with Kellogg data are however difficult to make because we could not use the 15-20% by weight of propane and butane (basis charge) which was indicated by Kellogg.

Yields and octane by gas oil cracking and naphtha reforming equalled and sometimes exceeded Kellogg data. It was possible by means of processing of C_3 and C_4 from latter two methods, with available saturated Fischer gas oil, to substantially exceed yields of poly-gasoline obtainable by "gas reversion" at somewhat above 60 octane.

2) Manufacture of alkylated benzols and their utilization as antiknock agents and safety fuels.

(Page 66 contd.)

It was possible to produce 40 well-defined alkylated benzols by alkylation of benzol and its homologues with propylene or isobutylene in the presence of strong sulfuric acid--or with methyl and ethyl chloride by Friedel and Craft; and to test their anti-knock qualities and as safety fuels. Without exception octane numbers of above 100 (motor and research) were found. The anti-knock improvement in mixtures with gasoline was greater than could be obtained with similar percentages of benzol-toluol-xylol and gave less fading (heat fading) and same or better lead sensitivity. Owing to their high boiling points (150-250°C.) they are very well suited for use as safety fuel; their low temperature characteristics are good having a pour point below -50°C. Their use would substantially widen stocks available for manufacture of aromatic 100 octane fuels without further demands on isobutane. (Use of isobutylene as alkylation partner; see reports Baehr/Kolb of 3/24/39.)

3) Poly- gasoline made from normal butane at 700 atmospheres in once-through operation.

Experimental work done since end of December to polymerize normal butane in once-through operation shows following: raising of pressure from 200 to 700 atmospheres raises gasoline yield by 100/120%; catalysts can favorably influence both yield and octane number. It is not clear at present which catalysts (dehydrogenating, polymerizing, or aromatizing) are most satisfactory. Best runs at 700 atmospheres gave 28.5% by weight poly-gasoline in once-through operation. The olefin content of these poly-gasolines is low as expected from the high pressure (iodine number about 45-70). The stability in mixtures with Leuna gasoline is good (20% Leuna gasoline, 80% poly-gasoline). Depending upon operating conditions, octane numbers were found to be 76-81 (research) and 72-77 (motor). The once-through experiments to date are now being changed to include recycling of butane.

4) Analyses of mixtures of phenol by means of superfractionation in a laboratory bubble tray column.

Mixtures of phenols ("Phenoloele") were fractionated in a laboratory distillation apparatus which was two meters long equipped with 80 plates and manufactured by I. H. Braun, etc. . . Among other results it was possible to prepare crystallized carbolic acid of 95-99% purity. Experiments with known mixtures showed it possible to determine presence of carbolic acid, ortho-cresol and meta or para-cresol quantitatively within a few per cent, based on total.

Work for 1940:

1) Continuation of work to repair poly-gasoline from normal butane.

2) Further development of superfractionation of mixtures of phenol, etc. . .

3) Resume work to isomerize normal butane to isobutane.

Planning and Layout of Distillation and Gas Recovery Plant

I. Distillation Plants

1) Work for Leuna.

Assistance was rendered in distillation experiments and erection of plant unit for processing raw phenols which are obtained by extraction from sump phase middle oil.

. . . (Mentions various plant operating problems) . . .

A suggestion was made by "Lu" (Ludwigshafen ?) to subject brown coal tar to a so-called pressure distillation in the presence of coke dust (Grude-staud) in order to reduce its asphalt content. . . .

2) Work for "others"

. . . (Lists plants for whom designs were prepared) . . .

II. Gas Recovery Plants (by fractionation)

1) Work for Leuna

. . . (Lists work done)

2) Work for "others"

. . . (Refers to assistance rendered to others) . . .

(Page 69)

. . . (Lists number of plants for whom services were rendered) . . .

Coal De-slurrying, Centrifuging and Distillation

a) **Centrifuging:**

Work in 1939:

Continuation of work of installing "cyclo" gearing in Swedish centrifuges; in all, 38 machines so equipped. Total of 34 supplied with jackets and sprayer rings, and 22 with bearings of synthetic resin. Odor nuisance obviated by drawing off exhaust gases from centrifuges and tanks and burning them under furnaces. To overcome sudden large fluctuations in solid content of slurry from centrifuging operation, an attempt is now being made to effect a fixed control by means of a continuous density measurement. Further experimentation with this method of operation was carried out to gather additional experience.

Work for 1940:

1) Further installation of "cyclo" gearing; also of jackets and enlarged spray rings in remaining centrifuges.

2) Further experimentation with automatic density indicators.

b) **Distillation:**

Work in 1939:

The rise in the volume of coal gasoline in the current year has meant an increase in the number of furnaces operating, from 12 to 17 on the average. The installations were also kept running a longer time. The average operating time for the upper coils was 265 days; that of the lower coils 58 days--the mean being 90 days. Furnaces equipped with flue-gas circulation (1 and 2) showed more even temperatures and made possible a substantially greater throughput. No economies are thereby effected, however, for the reason that the saving in fuel gas is offset by greater consumption of energy.

The badly eroded caps and the top sections of the stacks on furnaces 11 to 16 were replaced. The fire chambers of all furnaces have now been lined with sillimanite. The revolving valves that have replaced gate valves in furnaces 14 and 16 last longer and are easier to take care of.

(Page 70 contd.)

Because of lack of space to work in, experiments for dry removal of residues from the furnaces (worm conveyor) were continued on a special outfit.

The direct distillation of the slurry without prior centrifuging was again given up, for the time being, partly because the concentration fluctuated too much, partly because the ratio of asphalt to solid matter resulted in a too rapid coking of the coils.

To get rid of the odor produced by the flue gases from the furnaces the distillation gases are sent into the hydrogenation recycle gas by two blowers instead of being burned in the furnaces. The automatic pressure regulation device is found to work satisfactorily.

Montan wax pitch procured from Riebeck since early in 1939 is being dissolved in heat-exchange oil in steam-heated vessels and sent to storage as a grinding oil.

The experimental distillation furnace, which is just now equipped with rods instead of balls, has for some time been operating satisfactorily on a mixture of residue from the sand-removal operation and of the solids from the centrifuges.

Work for 1940:

1) Distillation of original slurry in experimental distillation unit after reduction in inclination.

2) Progressive development of the dry removal idea.

Preparation of tar, low-temperature hydrogenation unit, tar filtration

a) Preparation of tar:

Work in 1939:

An average of 1530 tons a month of Koppers tar from the Brabagwerke (Schwarzheide), containing an average of 1.8% of water and 4.8% of solid matter, was processed. For experimental purposes, it was centrifuged, without any addition of acid, in a 1:2-mixture with Deuben and Voss tar. The solid content of the tar from the centrifuge was 0.3%.

To reduce sedimentation and the nuisance of cleaning the tanks by hand, it has been the practice since November to lead the raw tar into the former sand-removal tanks of the coal-deslurrying operation, where the residues are removed mechanically.

The quantity of waste oil from all hydrogenation operations, centrifuged in 1939, was on the average 890 tons a month as compared with 680 in 1938.

• • •

To overcome the odor nuisance, the exhaust gases from the two Haubold waste-oil centrifuges standing in the open air were drawn off and burned under the furnaces.

Centrifuging and filtering tests were carried out with the Lurgi tar obtained from Brüx brown coal.

Work for 1940:

1) Re-installation of a fourth Haubold centrifuge

2) Making 3 Heine outfits gas-tight, in order to work with CO_2. Installing mechanical devices for removal of residue. • •

3) To save iron: coating the tanks and parts of the waste-oil processing outfit with acid-proof material

4) Processing tests with new tars.

(Page 71 contd.)

b) Low-temperature hydrogenation operation, tar filtration

Work in 1939:

It was found possible to remedy occasional reductions in filter-press efficiency, resulting from the unfavorable character of the tar, by adding small amounts of kieselguhr as a filter aid.

During the final periods of operation the low-temperature hydrogenation reactor showed an increased difference in pressure, attributable to a deposit of polymerization products from the filtered tars. Tests are to be made, noting what effect a greater aging of the tar, prior to filtering, and a re-filtering through immersion-type filters, would have, in order to determine whether this condition is due to a new formation of polymerized resin in the tar already purified.

Work for 1940:

1) Continuation of tests looking to increase in filtration efficiency in cases where nature of tar is unfavorable.

2) Determination of substances unfavorably affecting filtration.

3) Filtration experiments on sediments forming in course of aging.

Catalyst Plant

a) Catalyst for sump oven:

Work in 1939:

There has been a substantial reduction in the excessive consumption of H_2SO_4 required for neutralizing the coke, though no change has been made in the mode of operation of the Winkler generators, Deuben coke having as before been gasified in a mixture with Tannenberg coal.

Since March the vapors resulting from the indirect drying of the catalyst have been freed of dust to a substantially higher degree, before they pass into the fire chamber of the furnace, by the installation of spray nozzles in the original cyclone. Of late, carbide sludge has been sent into the pressure seal of the cyclone, the effect being to cause part of the sulfurous acid from the vapors to combine, and, particularly, to eliminate the unpleasant odor.

The balls in the Polysius mill, which is used for the fine grinding of the catalyst for the sump oven (mixture with grinding oil), were replaced by rod-like members 20 x 20 mm. The grinding time for fine grinding could thereby be reduced by over 25%. Moreover, the mill operates almost noiselessly and without the hard pounding which used to cause shutdowns due to damaged bearings.

Work for 1940:

Completion and starting up of plant for manufacture of catalyst for sump oven operation, for the Pölitz A.-G.

b) Presses for catalyst pellets:

Work in 1939:

The presses have not been in operation since 1939.

For the purification of sump-oven rich gas, the intention had been to use "Lautamasse" (15% moisture content) in the form of pellets. The "Lautamasse" was ground to 62-82% below 0.06 mm. in size and could then be pressed into pellet form. The large-scale processing of "Lautamasse" in pellet presses proves impracticable, however, because of the damage to the presses caused by the harshly abrasive character of the material.

The poisonous catalyst powder must be entirely exhausted from the presses and working rooms. . . (details) . . .

Tar and Coal Separator Distillation

a) Tar and sump-oven separator distillation:

Work in 1939:

The larger of the two towers was entirely overhauled.

The installation of throttle diaphragms with pressure scales for the measurement of the individual throughputs of the 3 (or 2) coils hooked up in parallel has been completed on all 5 oil preheaters, and these are giving satisfactory service.

The corrosion in the oil condensers at the distillate entrance was eliminated by means of aluminum shields. Operating time now runs about 1100 days.

The piping of the fuel gas was simplified.

Work for 1940:

1) Installation of a modern gas preheater with turbulent blow, and of U tubes connected in series.

2) Improvement of towers by installation of modern bottoms to improve efficiency and secure sharper separation of distillates.

3) Further experimentation to prolong life of water condensers.

b) Coal separator distillation:

Work in 1939:

Both installations were given a complete overhauling.

West (680 days of operation), total throughput 1,243,000 tons

East (1036 days of operation), total throughput 2,155,000 tons

No signs of corrosion or erosion were found on any of the parts examined, and the heat exchangers, condensers and coolers were all right. An illumination by mesothorium of the first and hottest U pipes of the gas preheater, made of S_2 material, revealed that in some places the wall thickness of the bottom bend had been reduced from the original 8 mm. to 5 mm. N8 bends were attached at these places.

(Page 73 Contd.)

The coefficient of heat transfer at the gas preheater and heat exchanger remained uniformly satisfactory throughout the operating period, and so was the quality of the product.

In one of the installations there was a fractional condensation of the A middle oil. In the lighter fraction are the more valuable phenols, that are obtained in the caustic-soda de-phenolizing operation.

• • •

Work for 1940:

1) Replacing S_2 tubing in the first section of the gas preheater with N8.

2) Experiments with enameled elbows in the lower bends of the U pipe of the first section.

Processing of Coal

Work in 1939:

The load limit of the concentration mills, with adequately fine grinding, is about 36 tons per hour for the large-sized outfits, and about 15 tons per hour for the small ones (as delivered, 75% TBK (solid?), 25% oil). As the throughput increases the degree of fineness drops rapidly.

It is possible, by a constant making up of the losses suffered by the grinder bodies at certain intervals, to keep the grinding efficiency, and so also the fineness of the coal paste, at a high degree of uniformity.

The iron losses due to the abrasion suffered by the grinder bodies amount to about 0.1 kg. per ton of dry stock.

Owing to the surprisingly large amount of wear observed in a large mill equipped with baffles it was decided to try out a new design, with a square opening in the concentrating plates as a device to equalize the charge. . . . (Suggested improvements to overcome stresses suffered in manganese-steel plates due to pounding action. . . etc.) . . .

The effectiveness of the magnetic separator device installed before the oscillating screens was the object of several months' experimentation. It removes at least 50% of the iron present in the paste; what remains consists almost exclusively of particles of minute size - short, fine threads and flakes, also granules of magnetite, which are not likely to do so much damage.

The oscillating screens are loaded with a full charge of 50 cu.m. of paste per hour. With the November paste throughput, which reached 176 cu.m., the plant operated without reserve capacity. The average length of time the screen tension held was 10 months.

. . . (Reference to a plant fire) . . .

(Page 74 contd.)

Work for 1940:

1) Installation of a central connection for conveyance of coal.

2) Installation of a new type of concentration outfit.

3) Experimentation on electromagnetic removal of iron from dry coal, behind the proportionating scale.

1940 projects for completion and further development of low-pressure operation.

1) Project for an 11th battery of furnaces in the residue-processing operation.

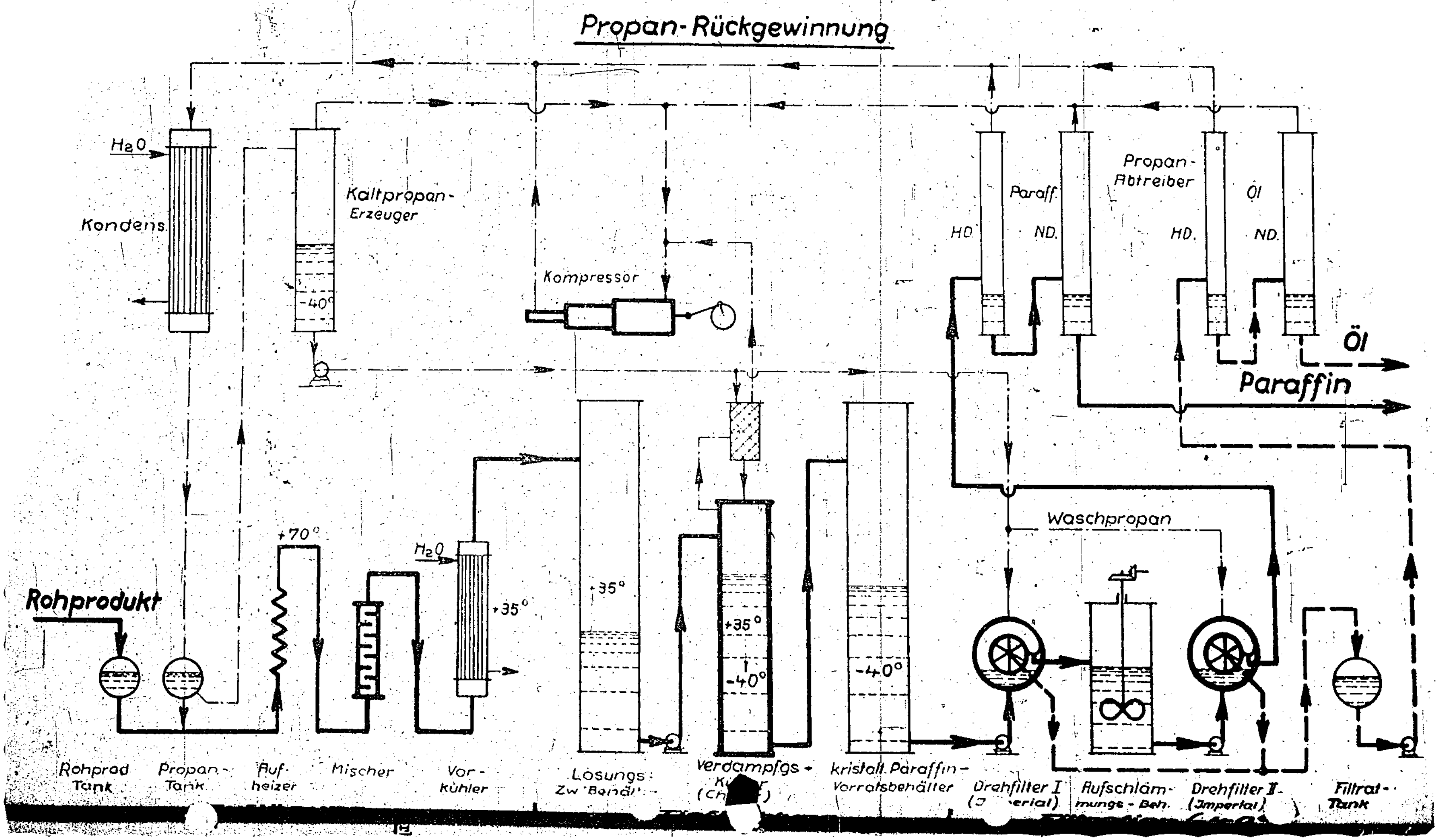
2) Transfer to the hydrogenation area of the equipment for causticization of the material used for purifying boiler-feed water.

3) Plant to obtain phenols from tar middle oil after the caustic-soda operation.

4) Plant to obtain phenols from phenol-rich water with heavy gasoline (replacing the expensive "Triverfahren" (tri-process)).

Hydrierung - Versuchsbetriebe.

T.T.H.-Rückstands-Entparaffinierung mit Propan.



(Drawing opposite page 75)

Test Lay-Out for Hydrogenation

De-waxing of low-temperature hydrogenation residue with propane

Propane recovery

Kondens	-	Condenser
Kaltpropan-Erzeuger	-	Cold propane product
Kompressor	-	Compressor
Propan-Abtreiber	-	Propane separator
ND	-	Low pressure
HD	-	High pressure
Waschpropan	-	Scrubber propane
Rohprodukt	-	Raw product
Rohprod. Tank	-	Tank for raw prod.
Propan-Tank	-	Propane tank
Aufheizer	-	Heater
Mischer	-	Mixer
Vorkühler	-	Pre-cooler
Losungs. Zw Behälter	-	Interm. solution storage tank
Verdampfgs-Kühler	-	Chiller
Kristall Paraffin-Vorratsbehälter	-	Storage tank for cryst. paraff.
Drehfilter I (Imperial)	-	Imperial rotary filter I
Aufschlämmungs-Beh.	-	Filter-cake tank
Drehfilter II (Imperial)	-	Imperial rotary filter II
Filtrat-Tank	-	Tank for filtrate

Technical Experimentation

I. De-waxing of low-temperature hydrogenation separator residue

1) With dichloroethane

Work in 1939:

The experimental equipment for de-waxing the residue from the low-temperature separator was in operation up to the first part of April 1939. It was designed to provide a simple method of preparing larger quantities of paraffin, Diesel oil and lubricants and to determine the utilizability of these products by placing them in the hands of consumers.

The Heckmann lubricating-oil distillation outfit, which had not yet given complete satisfaction during the previous year, yielded good cuts between Diesel oil II, spindle oil and lubricating oil after several additional plates had been installed.

2) De-waxing with propane

a) Small-scale tests

Work in 1939:

It was shown, by the small-scale tests with the de-waxing of propane, to obtain an aged paraffin from the residue from the low-temperature hydrogenation separator, that the following conditions have to be observed if a paraffin utilizable for obtaining fatty-acid is to result:

1) The amount of filter aid added to improve the quality of the paraffin and the filtration efficiency has to be 0.5% of the said residue.

2) Thorough removal of the oil constituents adhering to the paraffin requires a two-stage operation.

3) After the paraffin has been treated twice with propane it has to be separated as follows by distillation: 0-10%, preliminary paraffin run to separate the low-boiling constituents; 40-95%, main fraction, representing the

finished paraffin; and 95-100%, a residue which contains the filter aid and which is again added to the starting material

4) The paraffin distillation causes the residue to become enriched in substances which replace the filter aid and make superfluous any repairing of paraflow losses. For Zeitz de-waxing, the saving thereby effected amounts to 80 tons a year of paraflow, or RM. 240,000.00.-

5) Re-refining with Tonsil and aluminum chloride.

Work for 1940:

Tests for the preparation of an aged wax without purification with aluminum chloride and Tonsil.

Tests for processing Austrian petroleum.

Determination of causes of fouling of regenerator and catalyst in low-temperature hydrogenation.

Experiments in replacing propane with propylene-containing propane from Fischer synthesis.

b) Large-scale experiments

The following advantages offered by the use of propane were the decisive factors in determining the choice of de-waxing agent:

Suitability as a hydrogenating product;
Reduction in losses under chilling, due to cooling by expansion;
Greater speed of filtration.

The propane de-waxing process resolves itself into three stages:

1) Solution (continuous)

The residue (plus filter aid) is thoroughly mixed with liquid propane after being heated to 65°C. The resulting solution is first cooled down to 38°C. with water and placed in so-called hot-solution tanks.

2) Wax precipitation (non-continuous)

The solution is cooled in the chiller, batch by batch, down to -40°C., by direct propane evaporation, and stored in the filter charge tank. To make up the propane loss there is added during the cooling process liquid propane pre-cooled in a special tank equipped with its own evaporator. For equalizing the temperature of the gas passing off and the liquid coming in, a heat exchanger is installed above the chiller.

3) Filtration (continuous)

The paraffin is separated from the cold mixture in a rotary filter, and washed with cold liquid propane. It is next re-worked into a paste with propane and again filtered to remove the remaining oil. The paraffin and the filtrate are freed of propane in flashers and then pass to the distilling and refining outfits for further processing.

The propane gas passing off from all parts of the outfit is compressed and comes back into the cycle via the propane tank.

The outfit built for de-waxing of 1.5 tons of residue an hour was put into operation from about the beginning of June, and in several trial periods has come close to what was expected in throughput and in quality of product.

The experience thereby gained, which was utilized for the Zeitz plant, may be summarized as follows:

It is a fundamental requirement, for a satisfactory performance of the process, that the prescribed temperatures and pressures be most rigorously adhered to, especially during the filtration.

The formation of anti-crystal-forming nuclei during the preparation and processing of the solution must be prevented; these nuclei may be due to the moisture-content of the propane, to incompleteness of solution, or to local cooling.

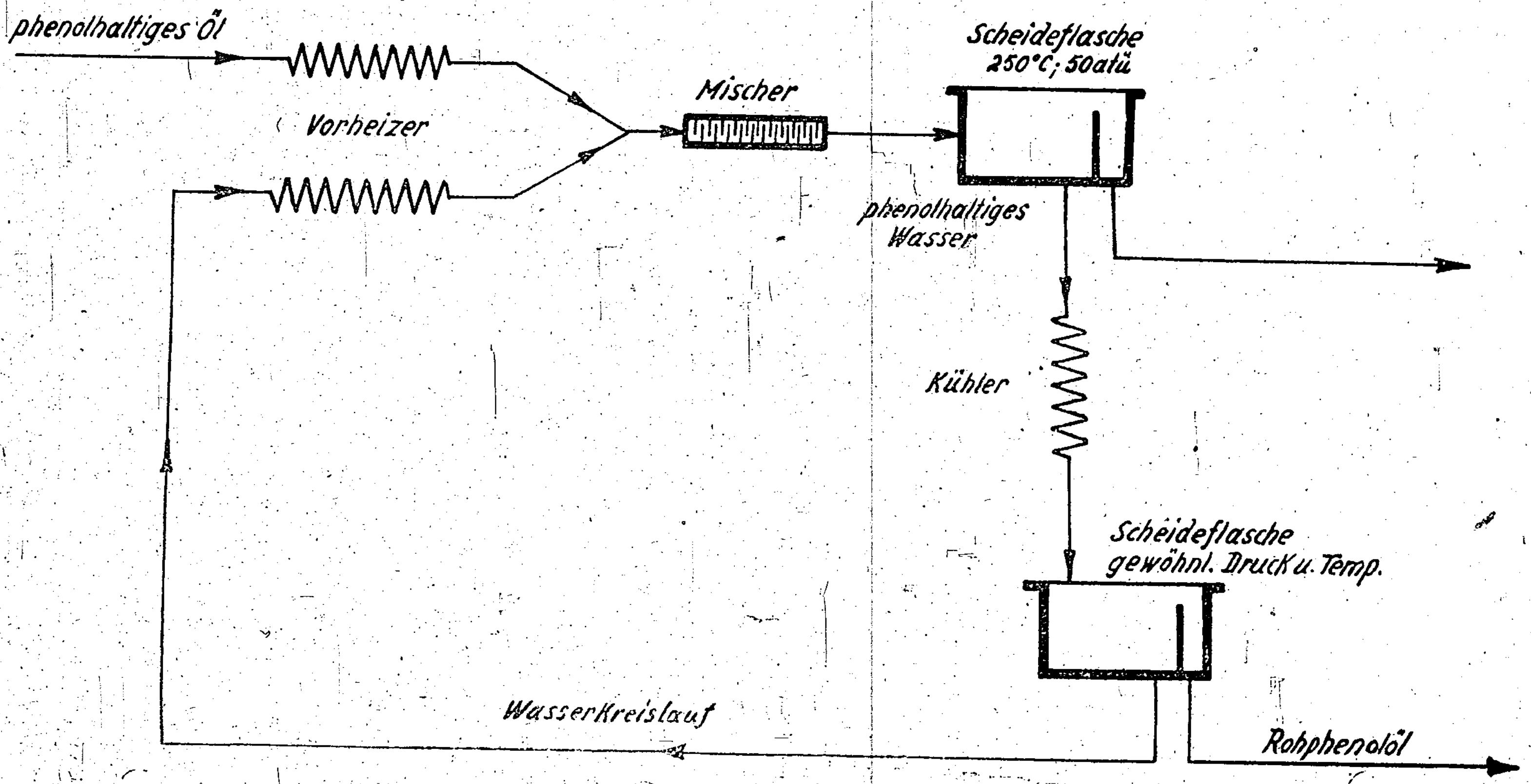
Wax crystals in suspension must be protected against mechanical destruction, which may for instance be caused when the mass is pumped by a multi-stage centrifugal pump.

Since the precipitated wax tends to settle, some way should be devised to keep up a continuous agitation in the storage tower and in the filter.

The mode of operation of the level indicators and regulators should be suited to the special requirements of the process.

Since on account of the serious difficulties encountered it has so far been possible to carry out the propane process in the large-scale test outfit only in short periods of operation, the experiments should be continued for the purpose of improving the installation to the point where operations can be carried on smoothly and without interruption.

Phenolgewinnung nach dem Heißwasserverfahren.



(Drawing opposite page 77)

Obtaining Phenol by Hot-Water Process

phenolhaltiges Öl	-	Oil containing phenol
Vorheizer	-	Preheater
Mischer	-	Mixer
Scheideflasche	-	Separator
phenolhaltiges Wasser	-	Phenolated water
Kühler	-	Chiller
Rohphenolöl	-	Raw phenol
Wasserkreislauf	-	Water circuit

II. Production of ammonia from phenolic water

The construction plans and calculation data for a large-scale plant to obtain ammonia and hydrogen sulfide from the phenol-rich waters in the hydrogenation process, by the direct-steam method, were based substantially on the experiments carried out as early as the first part of the year in Leuna and Hüls by Dr. Bähr's test group. The only unsettled question was of what material to build the heat-exchange unit for fresh and de-aerated phenolic water. Observations at the Hüls plant showed that corrosion would be a serious problem. To ascertain whether corrosion trouble was to be expected also with the Leuna water, the test column in Me 820 was put in operation again in July with separator water from the 5058 gas-phase reactors, and 3 heat-exchange tubes, of wrought iron, cast iron, and chromium steel, were installed in parallel.

After about 1200 hours' operation the test was stopped and the tubes were cut away, and the chromium-steel and cast-iron ones were found to have no corrosion, while the wrought-iron tube was badly corroded in spots, on the de-aerated water side. In contrast with this, it is noteworthy that the chromium steel at the Hüls plant had shown an extraordinary amount of corrosion after only a brief period of operation, which would seem to indicate that the Leuna water is far less corrosive.

• • • •

III. Obtaining phenol from hydrogenation oils.

1) Sulfide-solution process.

There was a continuation of the large-scale experimentation begun in 1938 for the extraction of middle oil with sodium sulfide solution. The agitator method was replaced by an extraction column equipped with Raschig rings and mixing coils, the yield from which, when middle oil from the coal

(Page 77 contd.)

separator was used, was 40-50% of the usable phenols present in the middle oil; this was in line with the results of the small-scale tests previously effected. An attempt was also made to increase the yield still more by directing a jet of steam into the vapor and gas contents of the column to churn them up--but without much success. The phenol yield so far achieved may be said to represent the maximum obtainable in a single stage of extraction. It is likely that a further increase can be brought about through the introduction of a multi-stage counterflow method and by the use of a middle oil fraction richer in phenol.

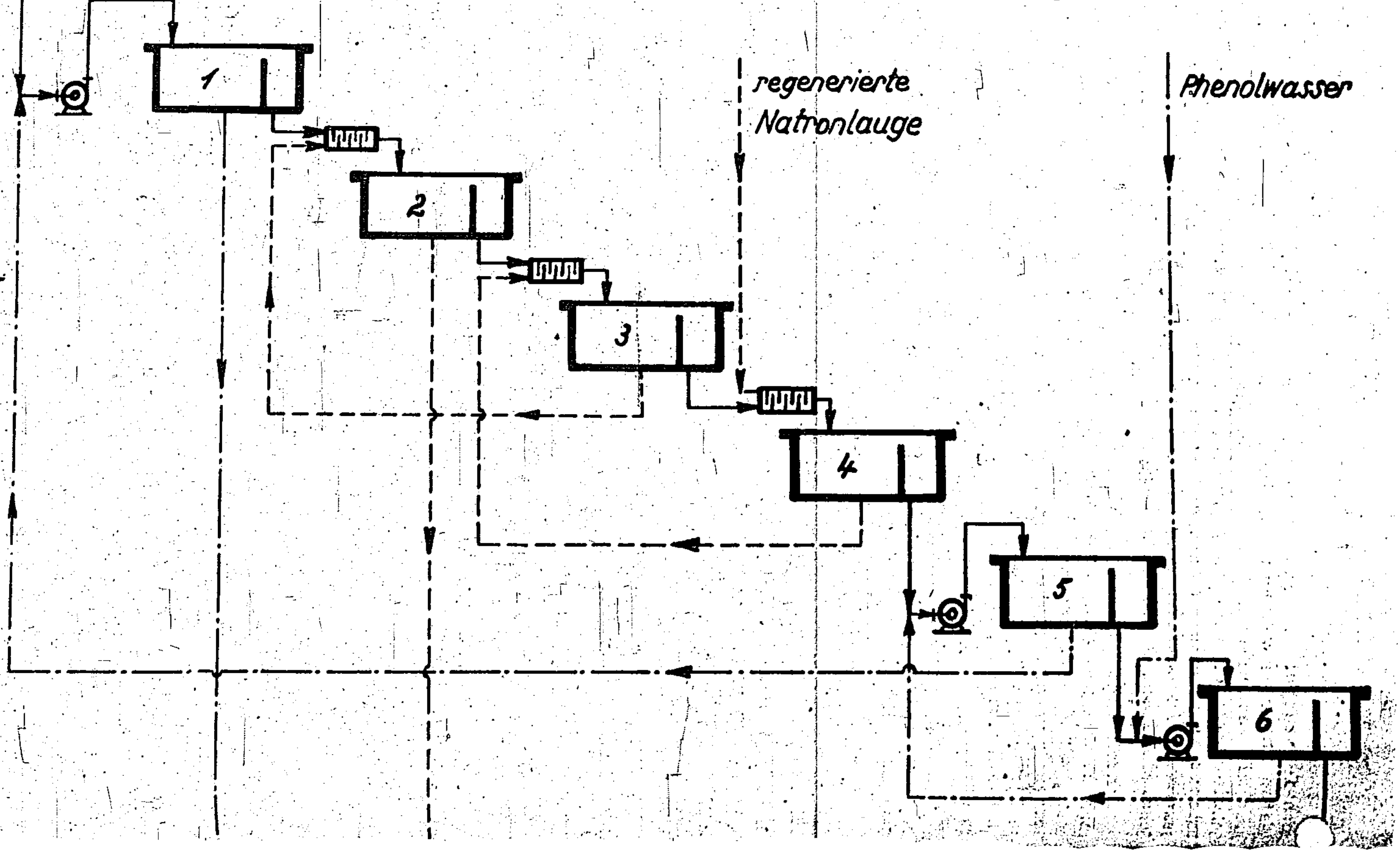
A study of the test data showed that the sulfide-solution process is able, under the best conditions, to achieve the economy which the caustic-soda counterflow method does, even when the latter has the advantages of multi-stage counterflow and of the use of a richer middle oil fraction. Against it, on the other hand, are its high installation cost, the difficulty of obtaining the materials, and the risk which hydrogen sulfide involves.

The experimentation was discontinued in May 1939 and use was made of the outfit whereby phenol is obtained by the caustic-soda counterflow method.

2) Hot water process.

According to this method, oil containing phenol is subjected to extraction with water at about 250° under (?) atm. pressure. At 250° water dissolves phenols out of the oil and, as a result of chilling, re-precipitates them as raw phenol oil, after the separation of the de-phenolized oil. The water, still weakly phenolic, then passes back into the cycle. The fraction between 180 and 210° is distilled out and is then dissolved in caustic soda solution to remove the neutral oil. By carbonization of the phenolate solution the phenols are separated in a form practically free of neutral oil and by re-distillation are obtained free of water.

Phenolgewinnung nach dem Natronlaugeverfahren.



(Drawing opposite page 78)

Obtaining Phenol by Caustic-Soda Process

A-Mittelöl — A middle oil

regenerierte Natronlauge — Regenerated caustic soda

Phenolwasser — Phenol water

In September an extraction plant erected in Building 803X for an hourly throughput of 9 cu. m. of middle oil from the coal separator was put into operation. The phenol oil produced was processed elsewhere.

The extraction unit was in operation without any particularly long interruptions until the end of June and its throughput was in all about 36,000 tons of middle oil, the raw phenol oil production being about 2,466 tons Analytical calculations showed the content of pure phenol fraction 180-210° to be about 500 tons, and it was found possible to increase the production of this fraction from the originally planned 75 tons a month to a maximum of 150.

The installation was comparatively simple to operate and it worked from the outset with no interruptions worth mentioning. The anticipation had been that putting in a multistage counterflow extraction arrangement to replace the existing single-stage process would presumably have meant a doubling of the yield of phenol without any substantial increase in steam consumption: an economy test, however, based on this favorable assumption, showed the hot-water process to be considerably more expensive to operate than either the sulfide or the caustic-soda process. The tests were accordingly discontinued at the end of June.

3) Caustic-soda process.

By this process the phenols are extracted from the oils containing them, by means of a 10% caustic-soda solution, at ordinary temperature, and the phenolate liquor formed is saturated with CO_2, with the result that the dissolved phenols are separated out. The resulting carbonate liquor is causticized with lime and then returned to the extraction outfit.

Large-scale experiments conducted in 1937 had shown that substantial quantities of valuable pehnols can be obtained by this method from phenolic hydrogenation products. Those tests were then discontinued, for the reason that the outfit was required for Dr. Bähr's sulfide process, at that time deemed more advantageous. The caustic-soda process was meanwhile developed further on a laboratory scale, and it was found that the amount of solution per ton of phenol required by economy considerations can be substantially reduced by performing the extraction operation with caustic soda in counterflow. The process thereafter seemed so promising that further investigation seemed called for, with a new large-scale test, and the outfit was rebuilt with that in view.

As indicated on the accompanying drawing, the Me 906X extraction outfit consists essentially of 6 mixers and their corresponding separation vessels. In mixers 2, 3, and 4, the counterflow extraction of solution and oil takes place in three steps; in mixers 5 and 6 the de-phenolized oil is subjected to a 2-stage washing with water (e.g., phenolic waste water) in counterflow, to remove the remaining NaOH. In mixer 1 the oil to be de-phenolized is subjected to treatment to cause the gases dissolved therein (CO_2, H_2S) to combine with the weakly alkaline wash water. The mixers used for solution and oil are what are known as orifice-mixers fitted with baffle plates, while in the case of water and oil the mixture is effected by means of a centrifugal pump.

The installation was put into operation at the end of May, with an hourly throughput of 4 to 6 cu. m. of middle oil from the coal-furnace separator, and the results achieved in a series of tests carried on up to August 1, went far to confirm the favorable ones the laboratory experiments had given. The quantity of 10% caustic-soda solution employed was on the average 15% by weight of the oil throughput.

The yield of raw phenol oil obtained was 5.4%.

The more valuable phenols of b.p.180-210° were selectively dissolved out of the oil by the caustic-soda counterflow process.

The yield of low-boiling phenols was 3% by weight of the middle-oil charge. The fraction consisted of carbolic acid, 30-40%; cresols, 50-60%; xylenols, about 10%; and neutral oil less than 1%. The caustic-soda requirement was on the average 6 tons of the 10% solution per ton of 180-210° phenol oil fraction.

After the washing with 5-10% of phenol water the de-phenolized oil was found entirely free of caustic soda. An exhaustive verification showed it to be entirely satisfactory for use as a gas-phase injection product.

Upon conclusion of the large-scale experimentation the test outfit was developed to the point where it could be adapted to the increasing requirement of phenol, for an output of 400 tons a month of phenol oil (fraction 180-210°) and it was put in operation at the end of August.

Since the beginning of November, normal middle oil has been replaced, for the time being, with a lighter product obtained by fractional condensation, constituting about 40% of all the middle oil, the advantage of this replacement being that a raw phenol oil particularly rich in valuable phenols can be so obtained with a lesser expenditure of solution. The separate processing of the light condensate is however practicable only as long as not all the utilizable phenols contained in the middle oil have to be subjected to extraction.

The raw phenol oil separated from the phenolate liquor by carbonization in Me 288 has to be given another carbonization to thoroughly free it from the last traces of solution, after which it can without difficulty be fractionated in non-continuous vacuum stills.

(Page 79 contd.)

IV. Phenol-oil distillation.

To provide computation data it was found necessary, before the new distilling unit was put into operation, to prepare a phenol-oil residue corresponding to the product yielded in the new distilling outfit Me 950. For this purpose an already existing experimental pipe-still installation was rebuilt and was employed, from September on, for the separation of raw phenol oil from extraction unit Me 906X.

The distillation tests are still in progress, bad corrosion having developed in the outfit built of ordinary iron. Among other places, corrosion showed in spots where, judging from the results of the earlier tests on the material, such damage was not to be expected. On the basis of the new findings, it was found that there was still time to make some changes as regards the materials to be used in the large-scale installation now in course of construction.

It is also to be determined, in the experimental distillation outfit, whether it is possible, in a lateral column, to make a technically pure carbolic acid cut from a mixture of phenol, cresol and steam. If this test proves successful, a lateral column for the production of carbolic acid will be installed also on the main outfit.

Laboratory Me 885

(Managing personnel listed)

Work in 1939:

In addition to plant analyses the following was done:

1) Most favorable recovery of slurry from low-temperature-hydrogenation-lubricating-oil-refining was found to be distillation of residues after their neutralization with lime with recoveries as high as 90%.

2) By means of addition of minute traces of Oppanol of various molecular sizes the viscosity index of the low-temperature-hydrogenation-lubricating-oil could be materially improved. Stability under heat was not adequate, however. Continuous operation of a lubricating oil at higher temperatures, whose viscosity index had been improved with Oppanol, showed it not to be sufficiently stable.

3) Fractionation of paraffin wax, from the low-temperature-hydrogenation, in vacuum by means of a Wittworth-Column was carried out for purposes of gaining data for a large scale paraffin distillation plant.

4) Sensitivity of pour point and low temperature filtration characteristics of low-temperature-hydrogenation-Diesel-fuels to paraflow and POV (Polyvinyloleate). The lowering of the pour point was more appreciable with POV than with Paraflow although both products could be used. The improvement of the low temperature filtration characteristics through a fuel filter of a Diesel engine was however considerably less affected thereby than the pour points.

5) Tests to establish PVO as standard fluid for calibration of Viscosimeters at high viscosities.

PVO in pure condition, or when mixed with paraffin-oil to desired viscosity, showed constant viscosity so that it is suitable for calibration purposes because of its high viscosity and its complete transparency.

(Page 80 contd.)

6) Low-temperature-hydrogenation-Diesel-fuels were found to be completely compatible with most other Diesel oils on the market.

7) Comparison of stability of low-temperature-hydrogenation-Diesel-fuels with that of commercially available Diesel oils by means of addition of "Kogasin." Low-temperature-hydrogenation-Diesel-fuels are equivalent to other Diesel oils on the market and showed no asphalt-like separations.

8) Impregnation of a hydrogenation catalyst with iron sulphate and determination of the degree combination with catalyst and investigation of corrosiveness due to liberation of acid.

The alkali content of the dust from the electro-filter of the Winkler coke ("Grude") was definitely sufficient to prevent formation of free acids under estimated catalyst concentrations. The mineral salt content of the coke dust is sufficient to react completely with the iron content of the impregnated catalyst so that extraction with water after impregnation can no longer remove iron from the catalyst.

Work for 1940:

1) Because of shortage of iodine new analytical methods for determination of phenol and hydrogen sulfide will be investigated.

2) Extension of tests with paraflow and PVO to affect pour point and low temperature filtering characteristics will include commercially available Diesel fuels.

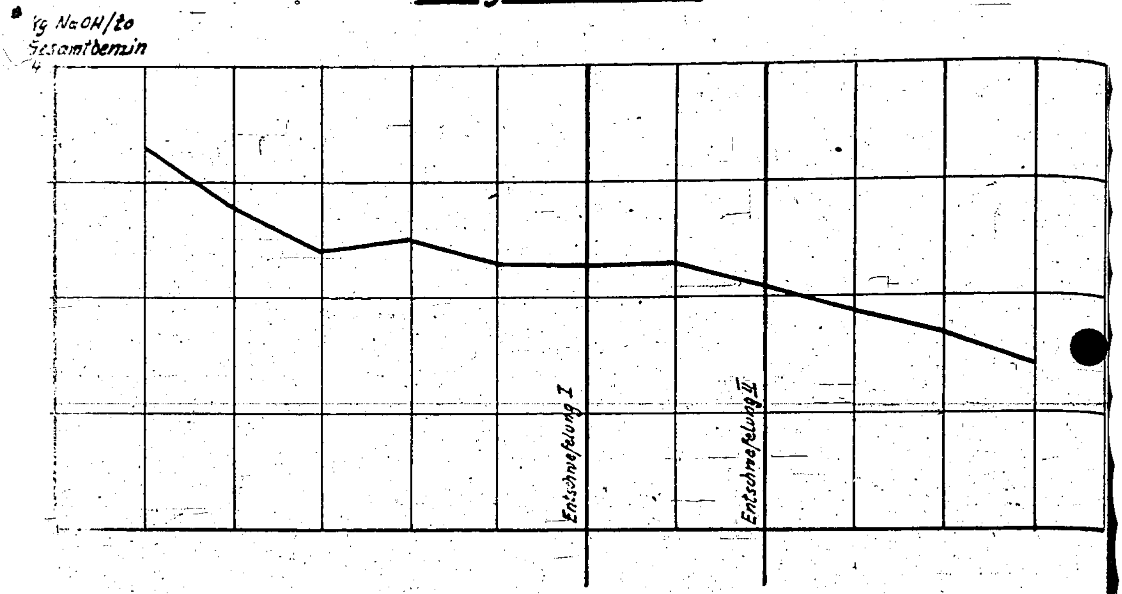
(Following Page 80)

Hydrogenation

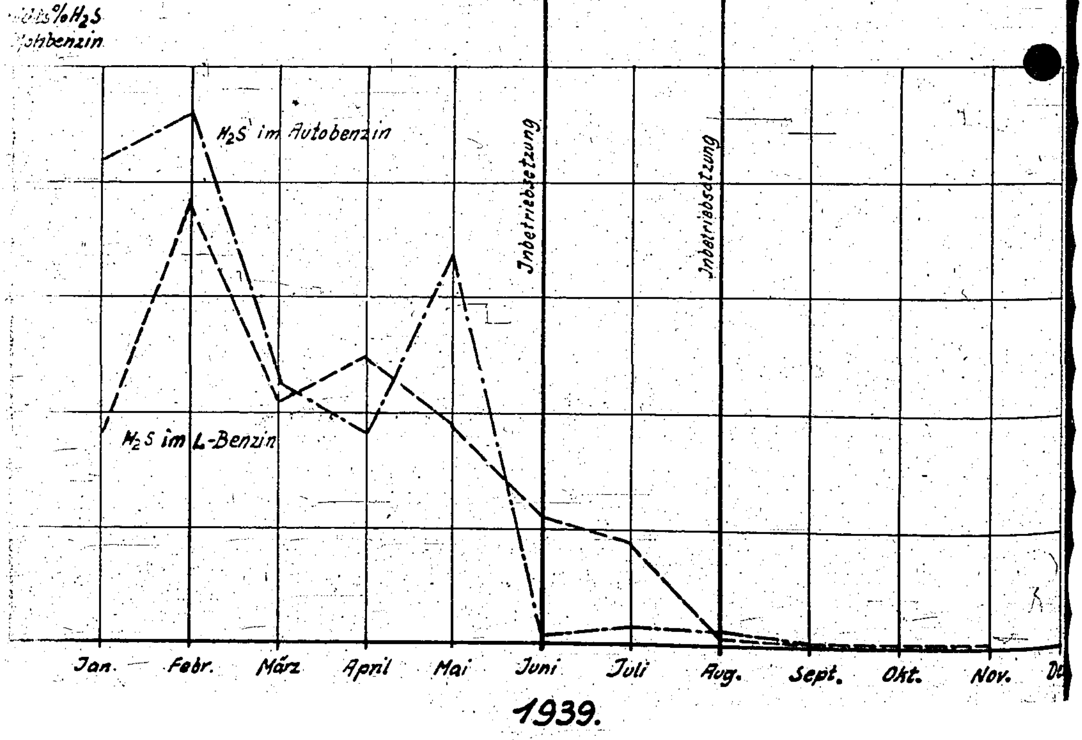
c) Distillation Group

Benzin Abstreifer-Destillation, Laugewäsche.

Laugeverbrauch.



Konzentration von H_2S im Rohbenzin.



(Drawing opposite page 81)

Benzin Abstreifer-Destillation, Laugewäsche - Gasoline-Separator-Distillation, Caustic Wash

Laugeverbrauch - Consumption of Caustic

Konzentration von H_2S im Rohbenzin - Concentration of H_2S in raw gasoline

(Page 81)

Gasoline-Separator-Distillation, Caustic Wash

Gasoline-separator-distillation

Work in 1939:

Only overhauling and careful checking of plant III was carried out although more work had been planned. In the course of this work the preheater tubes which were corroded by sulfur containing flue gases were rebuilt and heating surface reduced. Tubes located in the so-called radiation zone were removed. The combustion chamber was lined with sillimanite (highest temperature 1450°C.) and combustion gas and the flue gas lines were equipped with new throttles. This plant was equipped with a new control board from which both tower and preheater could be handled.

. . . (Details regarding a fire in Unit II) . . .

The preheater of Unit IV was checked in line with experience with Unit II The heating surface was reduced by removal of one path in order to keep flue gas outlet temperatures sufficiently high and to thereby avoid corrosion.

Similarly the preheater of Unit V was checked, revealing strong corrosion. Heating surface was also reduced here.

. . . (Details regarding what to do in case of accidents) . . .

Aviation gasoline production was increased at the expense of motor gasoline. The increase was from 7500 tons in July to 17,000 tons in September. From September on 6000 tons of Diesel fuel were manufactured in addition thereto.

Work for 1940:

. . . (Points 1), 2), 3), and 5) deal with plant details) . . .

4) Preparation of means for prevention of preheater corrosion.

(Page 81 contd.)

b) Alkali Wash

Work in 1939:

Three desulfurizing towers were installed in order to reduce consumption of caustic. In these towers the hydrogen sulfide in raw gasoline is substantially removed by counter-current passage of hydrogen sulfide-free hydrogenation-gas. Before these towers were placed into operation the hydrogen sulfide content was 20-50 mg. per 100 g. gasoline, whereas thereafter less than 1 mg. of hydrogen-sulfide per 100 g. of gasoline was found. The consumption of NaOH (100%) per ton of raw gasoline decreased from 3.28 kg. in January to 2.3 kg. in June and 1.4 kg. in November. The chief advantage of this counter-current blowing operation was that after separation of phenols the caustic solution could be brought up to strength and recycled for further caustic wash.

A gasoline washer was set up for the oil containing condensate used for washing the waste gas and liquid in the absorption gasoline plant. Oil traces were thereby removed from condensate water which (traces) otherwise would be entrained in the fuel gas.

Depropanizing and Absorption Gas Separation

(Managing personnel listed)

Work in 1939:

. . . (Details of plant operation) . . .

During the inspection of a cooler in units 887 strong corrosion of the bi-metallic tubes was observed after a short period of operation. The corrosion may be due to high chlorine content of the water of the river Saale. Fewer operating personnel were required in unit 890 owing to installation of various automatic controls . . .

In the course of increased production of aviation gasoline resultant yields of absorption gasoline were separated into bottoms, and overhead. The bottoms were added to aviation gasoline while the overhead is separated into pentane and a pentane-containing butane. It was in this manner that it was possible to increase the production of aviation gasoline within desired steam pressure. The present increased production could be further augmented. Besides, this permitted a clearer picture as to the quantities of pentane resulting from (increased) aviation gasoline production. An incidental benefit of this method of operation was an easing of the load on the absorption gasoline plant in which nowadays only small quantities of pentane and butane are recycled.

Work for 1940:

An additional depropanizing unit capable of handling 40 cubic meters shall be built, as at present only one unit is available for each of the aviation gasoline and motor gasoline (plants).

Gasbenzin - Niederdruck I.
Aethan - Erzeugung.

Tail Gas (Low-Pressure) I

(Managing personnel listed)

Work in 1939:

To make it possible to process the whole of the sump-phase rich gas and come up to the required ethane production of 16,000 tons a year, suitable conversions were made so that the compressors and the related gas-recovery units could operate in parallel. Both the alkazid plants also had to work parallel from November 1 on. A load test has shown that a throughput of 13,000 cu. m. of gas in one of the outfits is possible.

The northern alkazid unit was overhauled in the early part of the year. Corrosion having been observed also on the iron tube clusters of the generator of the south unit, an attempt was made in August to remedy this condition by adding arsenious acid to the alkazid liquor (0.6 kg. per cu. m.). It remains to be seen whether this will be successful.

In July, after four years' operation, the column of gas-recovery unit Me 887 had to be overhauled, and the new unit Me 936 was accordingly put into operation with the GHH compressors. To increase the gas throughput, improvements were effected in both tail gas units by the installation of precoolers behind the compressors. The resulting condensate was then conveyed in liquid form into the column.

In August it was finally possible to put into operation the BBC compressor which had been ordered long previously. It has performed satisfactorily so far.

The scrubbing of the liquid gas mixture from the tail-gas plant with condensate containing a small amount of oil, for the removal of the ammonia, brought small quantities of oil into the liquefied petroleum gas (Treibgas); in June 1 accordingly, on occasion of putting the Gerlach unit into operation, means had to be contrived for removing the oil from the condensate by washing with gasoline.

(Page 83 contd.)

Advantage was also taken of the overhauling of tail-gas unit Me 887 to install a series of automatic regulators.

Work for 1940:

1) Both alkazid installations are to be fitted with aluminum tube clusters in the regeneration unit.

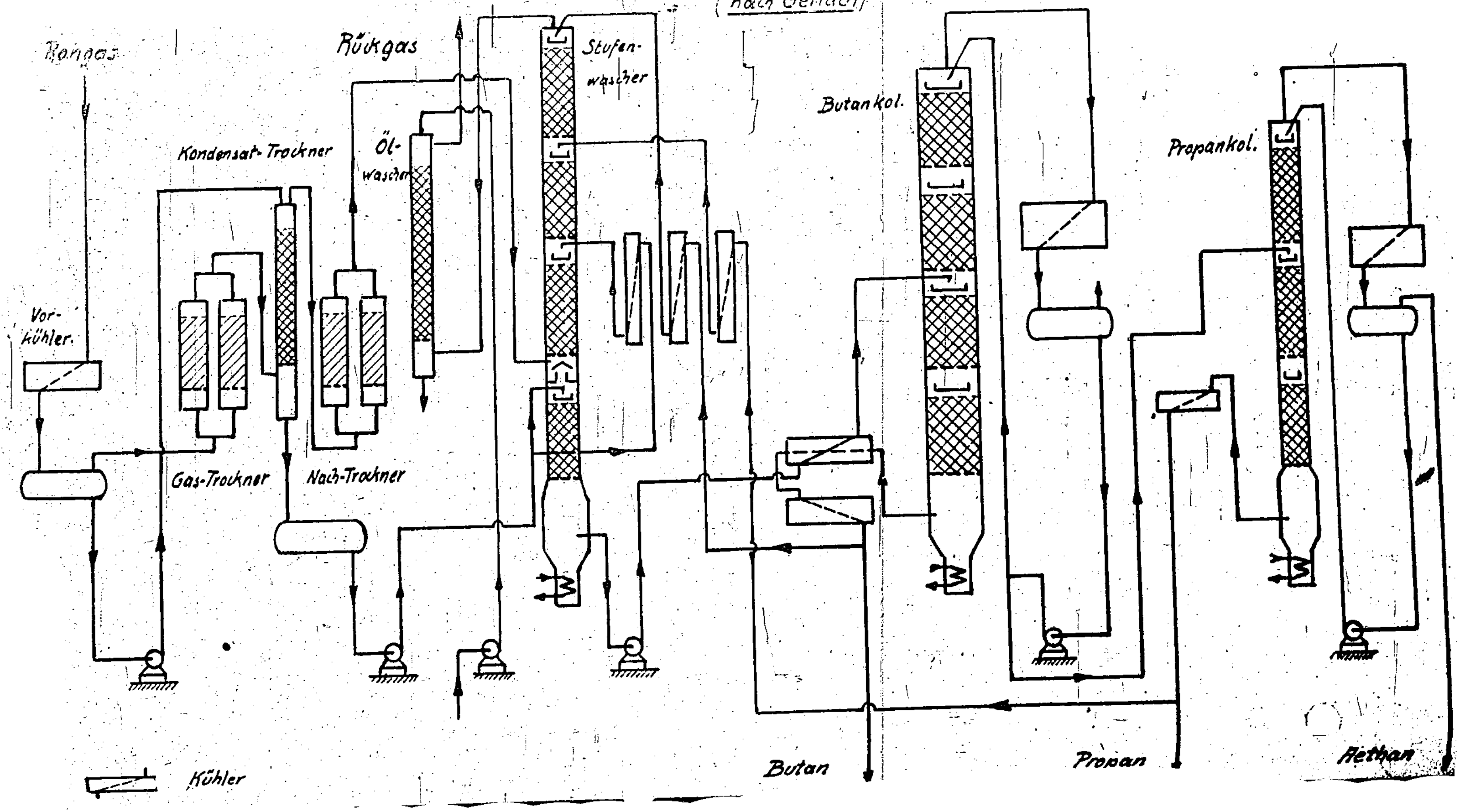
2) The delivery of a larger-sized compressor, scheduled for 1941, must be hastened.

[illegible] Niederauden I

Me 914

[illegible]gaszerlegungs-Anlage.

(nach Gerlach)



(Drawing opposite page 84)

Tail Gas -- Low-Pressure II

Me 914

Gerlach plant for separation of rich gas

Rohgas	Raw gas
Rückgas	Residual gas
Stufenwascher	Absorber and stripper
Kondensat-Trockner	Condensate drier
Ölwascher	Overhead vapor washer
Butankol.	De-propanizer
Propankol.	De-ethanizer
Vorkühler	Partial condenser
Gas-Trockner	Gas drier
Nach-Trockner	Second-stage vapor drier
Kühler	Condenser
Butan	Butane
Propan	Propane
Aethan	Ethane

Tail Gas (Low-Pressure) II

(Managing personnel listed)

Work in 1939:

The new Gerlach-model gas separation plant Me 914 was put into operation June 3rd. As a departure from the Linde method, in this unit the separation of the gases lighter than CO_2 is effected at the beginning by means of a multi-stage scrubber the distinguishing feature of which is that the gas mixture is scrubbed at the top with butane and below with propane (see drawing). Butane is then obtained in another column, at the bottom, while the mixture of propane and ethane issuing at the top passes to the neighboring ethane column, to be separated. The pressure maintained in the multi-stage scrubber is about 15 atm. and in the other two columns 21 atm. From the raw gas coming in, a pre-cooler separates a mixture of gases which, after being dried with silica gel, is conveyed to the scrubber. The remaining gas passing out of the latter is scrubbed with middle oil to obtain the entrained butane. Refrigeration is effected by 3 single-stage ammonia compressors (including one in reserve).

There has been a change in the composition of the raw gas (increase in butane content) since the installation was planned; operations have however gone comparatively smoothly, after a few initial difficulties. It is now possible with this single plant to process the whole of the rich gas yielded by the gas and sump phase, for which the two Linde units proved insufficient.

The Linde-type gas separator installations remained in operation until the Gerlach unit was started; they were then shut off for an overhauling. A stone wall has been put up to protect the pipe-bridge passing Me 914 on the east from a fire in the Gerlach unit.

Work for 1940:

Installation of a dephlegmator for the scrubber will make possible an increase in gas throughput. To get the butane column to operate better with the given gas composition, the installation of a preheater is planned.

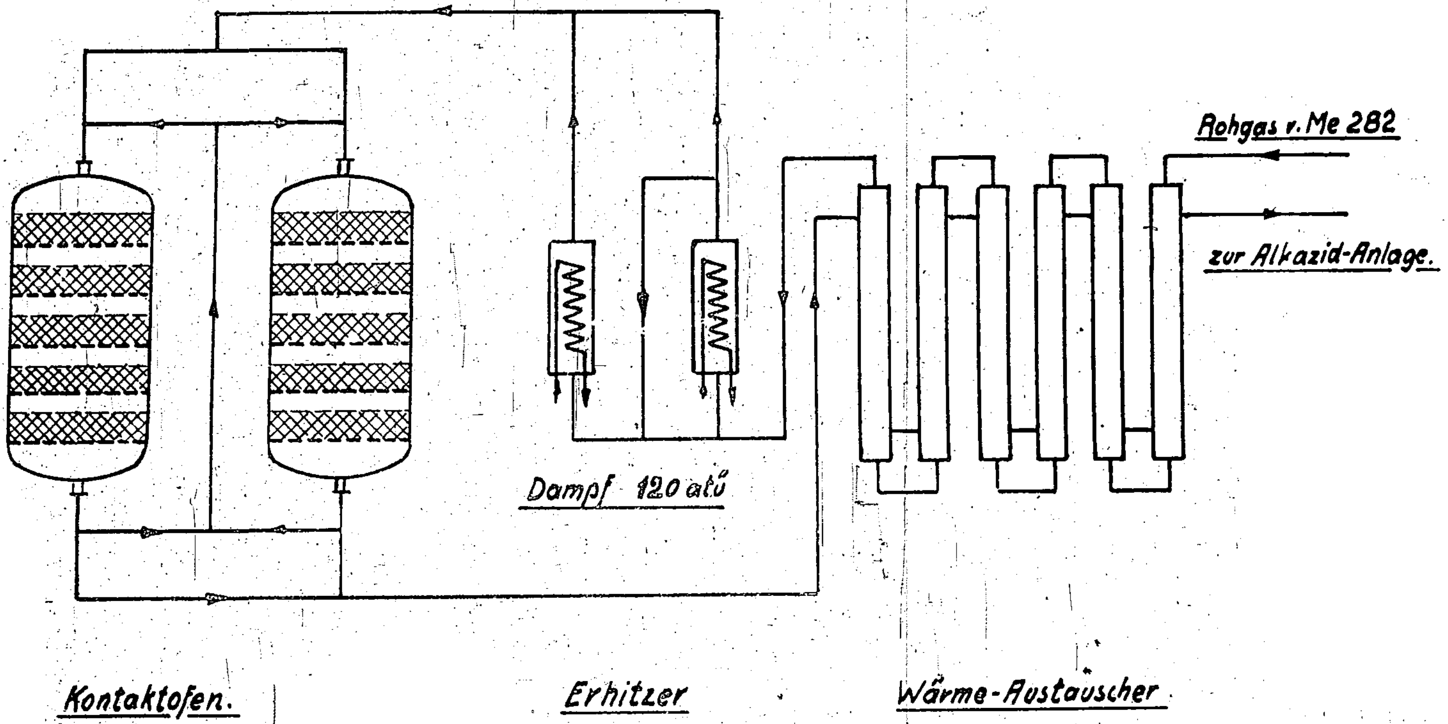
To produce isobutane a larger separating unit for n- and isobutane will be erected (to produce 16,000 tons a year).

Plans are being made for a rapid evacuation of the large quantities of liquid gas in the Gerlach unit in the event of a fire risk, by passing them into an underground tank of 50 cu. m. capacity. . .

Sumpfphase Reichgas-Reinigung.

Me 940.

Sumpfphase - Reichgas - Reinigung.



(Drawing opposite page 85)

Refining of Sump-Phase Rich Gas

Rohgas v. Me 282 - Raw gas from Me 282

zur Alkazid-Anlage - To alkazid plant

Dampf 120 atü - Steam 120 atm.

Kontaktofen - Catalytic furnace

Erhitzer - Heater

Wärme-Austauscher - Heat exchanger

Purification of Rich Gas from Liquid-Phase Process

(Managing personnel listed)

Work in 1939:

Plant Me 940, planned in 1938 for the processing of larger quantities of liquid-phase rich gas, was put into operation in January, and the smaller test outfit shut down completely in May. The unit was heated with steam under 120 atm. pressure from Me 204. Only one of the two furnaces charged with catalyst containing iron oxide is in operation at any one time. The operating temperature is 300°. The increase in the sale of "Treibgas" (a liquefied petroleum gas) and in the ethane requirements made it necessary to take in larger quantities of liquid-phase rich gas, and this was made possible by resorting to special arrangements in the high-pressure unit (increasing the expansion pressure).

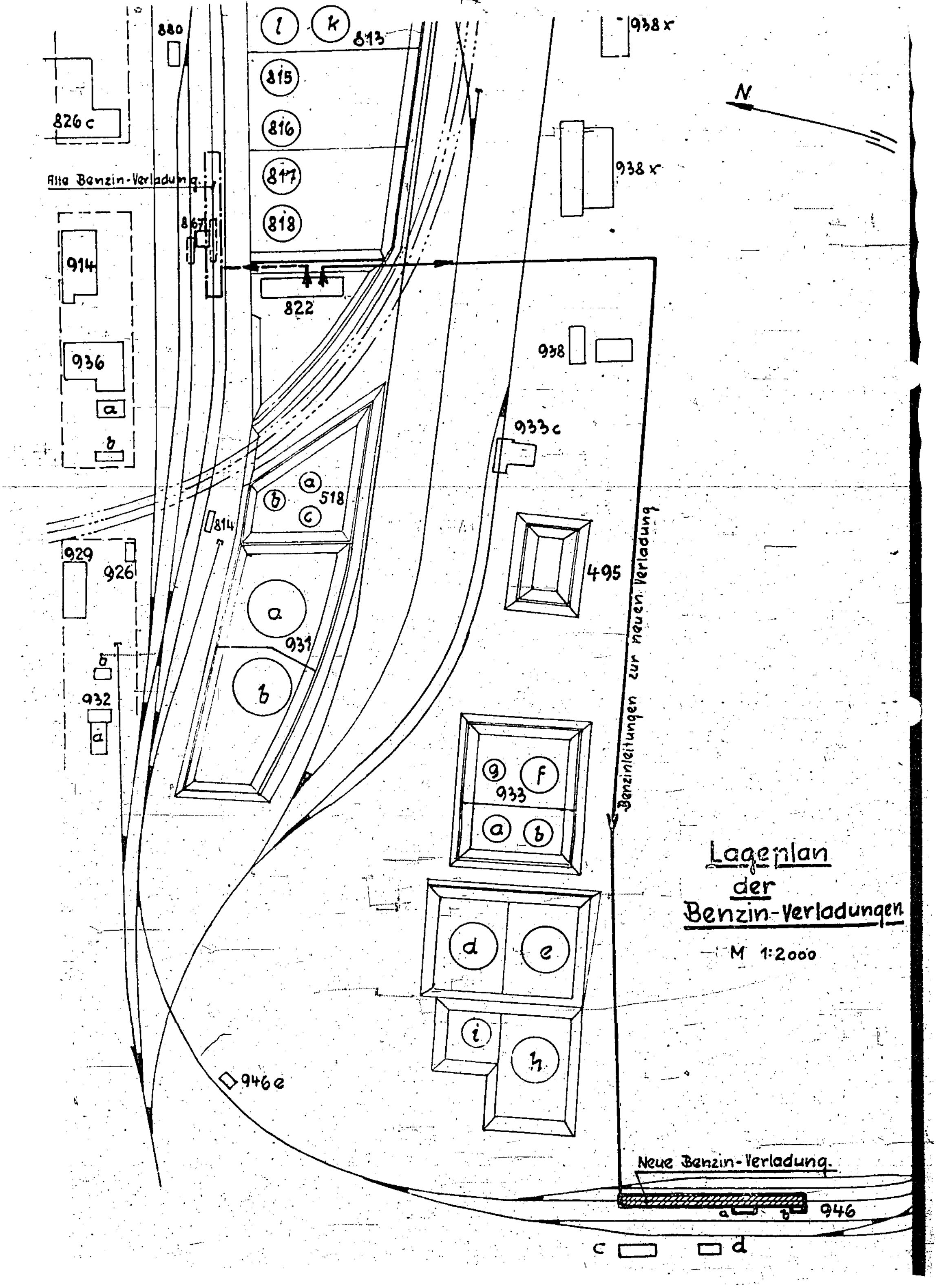
To augment the throughput quantities, the gas pre-refined in the alkazid pressure washing process was delivered under a pressure of 1.3-1.8 atm. By November an increase to about 6,000 cu.m. an hour had been achieved. It was with this that the whole production of liquid-phase rich gas was processed.

Working up a raw gas that contains about 600-700 mg. of organic sulfur per cu. m., the installation passes on to the alkazid plant a gas with only 5-10 mg. Not until 10 months had elapsed did the catalyst show any weakening.

Work for 1940:

A suction device is to be provided, for the safe changing of the catalyst, an operation otherwise risky owing to possibility of fire.

Benzin-Versand.



Lageplan
der
Benzin-Verladungen

M 1:2000

(Drawing opposite)
(Page 86)

Gasoline Shipment

Alte Benzin-Verladung	-	Former gasoline loading installation
Benzinleitungen zur neuen Verladung	-	Gasoline lines to new loading area
Lageplan der Benzin-Verladungen M 1:2000	-	Site plan of gasoline loading installation Scale 1:200 m.
Neue Benzin-Verladung	-	New gasoline loading plant

Shipment of Gasoline

(Managing personnel listed)

Work in 1939:

As far as the shipment of gasoline is concerned, quite a number of conversions and changes were effected during the year in the storage tanks, blending outfits and tank-car filling equipment, in consequence of the multiplicity of new regulations governing power fuels.

As of May 1 the shortage of alcohol in Germany made it necessary to divide Germany into a northern and a southern zone; in the former only a blend of 88% gasoline and 12% "monopoly spirit" was allowed, and in the latter only leaded-gasoline. The octane number of both had to be at least 74. Throughout the country there could be used, along with a straight gasoline-benzol fuel, a so-called "super fuel," of about 78 to 80 octane, consisting of the gasoline-benzol blend plus tetraethyl lead.

Operations had to be changed to suit these conditions. The transformations were made possible without too great expense by the removal of the Leuna alcohol-blending outfit to the German Gasoline Company's storage plant located in the northern zone.

The installation for the tetraethyl lead operations was enlarged by the erection of a blend-injector for auto gasoline and of a pump; the storage possibilities for tetraethyl lead were correspondingly augmented.

The large tanks of 5,000 cu.m. were fitted up to receive south-zone gasoline. With the increase in our aviation-gasoline production from 80,000 to 150,000 tons per annum, the 500 cu.m. tanks used for adding alcohol to gasoline were likewise adapted, on the 1st of April, for the reception of leaded aviation gasoline.

(Page 86 contd.)

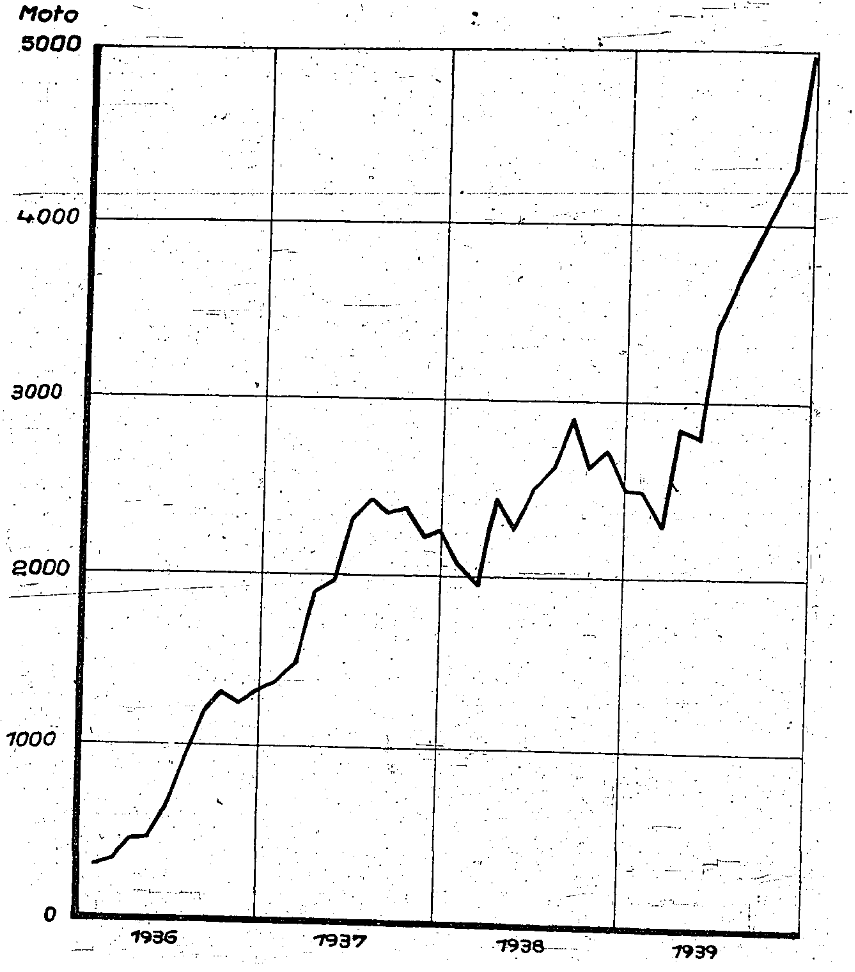
The terms of the order for mobilization made it necessary to produce Diesel oil as a new source of power. Two tanks of 2,500 cu. m. each were accordingly provided, and the loading equipment designed so that the packaging could be taken care of separately from the gasoline loading. The increase in production of aviation gasoline to 200,000 tons a year, coming at the same time, made it necessary to include altogether 4 tanks of 2,500 cu.m. as storage space, in order for production not to be interrupted when any trouble was experienced with either the packaging or shipping activities.

Work for 1940:

The new loading installation, started in July, is to be in readiness by the beginning of 1940.

The tank-truck loading installation, scheduled to be ready this year, has been postponed, and is to be completed in 1940.

Kohlenwasserstoffe - flüssig - Versand.
Treibgas - Versand.



(Drawing opposite page 87)

Shipments (tons per month) of
liquefied fuel gas (Treibgas)

Storage and Shipment of Liquid Gas

(Managing personnel listed)

a) Storage of liquid gas:

Work in 1939:

The erection of the 7th and 8th tanks (150 cu.m.) completed the new underground storage plant. Still, the storage facilities lag behind production, and the old-type surface storage must, especially since the outbreak of war, be kept at a decidedly low level.

Present circumstances make it as yet impossible to complete the ethane storage facilities.

Work for 1940:

Completion of the ethane storage.

b) Shipment of liquid gas:

Work in 1939:

"Treibgas": The sale of this liquefied petroleum gas is on the upgrade again (see graph). During the first half of the year it was affected by the lack of fresh supplies of bottles. The unfavorable transport conditions (haulage by truck) at the outbreak of the war caused a decided decline in the shipments of liquid gas. As early as October, however, it was possible to check this downward trend and to make up for it by a substantially increased efficiency in the packaging operations.

From September on, the sale of Treibgas was taken over by the Central Mineral Oil Bureau in Berlin. Four packaging stands were re-erected in the packaging area.

Propane for industrial and household use: The sale of propane in 1939, 260 tons a month, was again exceeded by about 60%. Difficulties experienced in the procurement of iron caused a shortage of bottles. We were badly

(Page 87 contd.)

behind in deliveries, so that all marketing activity had to be suspended from autumn on. The business done in 300-kg. drums went up again. Two more weighing outfits were installed in the packaging area; two of these were also provided for the bottling of butane in Me 928.

There was an increase in the shipment of liquid gases in tank trucks and tank cars.

In Me 925, owing to the increased demand, Sunday work had to be instituted; more personnel was also employed.

The bottling plant in Munich increased its output to about 420 tons a month. Operations were carried on continuously in shifts of 8 to 9 hours; there will be a change to a 2-shift arrangement in 1940. The Stuttgart plant is still under construction. Difficulties have arisen due to delays in the deliveries of the transformer equipment. A third installation is to be erected. The site, earlier figured on as Königsberg, has not as yet been decided.

Work for 1940:

The makeshift loading equipment for tank cars in Me 928 is being improved. Tank-car deliveries of butane to manufacturers of engines (being planned in 1939) will be undertaken at the beginning of 1940.

(Page 88)

Laboratory, Technical Institute and Engine-Testing Room

(Managing personnel listed)

Work in 1939:

In the laboratory, in addition to the researches carried out on raw, intermediate and finished products as a necessity for plant supervision, further investigations with respect to the final distillation for the separation of hydrocarbon mixtures were started.

. . . (Members of research carried on in collaboration with government agencies, etc.) . . .

The internal features of the technical institute, organized in 1938, were further developed.

For engine tests on motor fuels the existing two CFR units have been augmented by another I.G. test motor; an I.G. test Diesel, with the necessary measuring devices, is to be set up some time this year.

In addition to the tests made on various hydrogenation products, wide-ranging experimentation was also effected on motor fuels of the organic division, derived from the non-pressure hydrocarbon synthesis tests, as also on the anti-knock value, shrinkage in anti-knock value and lead sensitivity of the gasolines produced in the main laboratory's experiments. Analytical fuel-product researches were also carried out.

Work for 1940:

Contemplated new plans have been put aside, since production space has to be the prime consideration in a war economy.

Deaprtment of Economics

The development of processes for the manufacture of hydrogen from bituminous coal, brown coal, exit gas from the hydrogenation, coke oven gas and natural gas have been investigated thoroughly from the technical and economic standpoint. These new developments have been utilized for Leuna and for new hydrogenation and nitrogen projects. The following processes were especially investigated: the Koppers, Viag and Lurgi processes, the gasification of coke from the low-temperature carbonization of bituminous coal, the partial combustion of coke oven gas and especially the processing of the exit gases from the hydrogenation and Fischer plants. Furthermore, the questions concerned with the expansion of the synthesis gas production at Leuna and the change from coke to brown coal (power house in connection with low-temperature carbonization, desalting of Grude, manufacture of soda) have been subjected to preliminary investigations.

With regard to gas purification further licensing of the Alkazid process in Germany and abroad has been undertaken in connection with the plant and the Director's Office of Sparte I. The sulfoammonium process for coke oven gas and hydrogenation water has been investigated as to its economics. The German sulfur market has been continuously followed and additional production possibilities for sulfur at Leuna have been examined. A series of cost calculations have been made for the manufacture of krypton.

In the phenol field economic questions concerned with the manufacture, additional production (outside purchases, extraction of middle oil and tar oil), refining, utilization and the market situation have been considered.

In the field of organic products the work consisted in increasing production and deliveries, disposal of by-products, etc., and this was achieved by cooperation with the processing plants and the sales department of the I.G. as well as with government departments and trade organizations. Procurement of raw materials and the negotiation of contracts was included in the duties of the department. Special interest was taken in the use of the Mepasin products as soap substitute, textile agents and plasticizers. The development of cyclohexanols, adipic acid, Luran, product 512, white oil and toluene from benzol and methanol was followed up.

The development of cracking and dehydrogenation processes for the manufacture of ethylene, propylene, iso-butylene and butylene, the manufacture of SS 906, T 52 from isobutane, butadiene from n-butane as well as the manufacture of polygasoline from saturated and unsaturated gases was followed from a technical and economic standpoint. The necessary preliminary work on contracts was carried out throughly and contracts were negotiated.

The manufacture of hydrocarbons in its broadest aspects, the hydrogenation of bituminous coal, brown coal, tars and natural petroleum, low-temperature carbonization, furthermore, the Fischer process in its various modifications (at atmospheric pressure, at elevated pressure, Michael process) and catalytic cracking were considered as to their economics. The experience with these subjects of some of our men has been utilized by the "Reichsstelle für Wirtschaftsausbau für die Minerolölplanung im Rahmen des Vierjahresplanes" (Department for economic development for planning in the petroleum field Section of the four-year plan) and, since the beginning of the war by the Committee for Hydrogenation, Synthesis and Low-temperature carbonization.

The purchase of ores for the manufacture of hydrogenation catalysts at Leuna and Ludwigshafen and the fixing of the sales price has been undertaken by the department.

On request of the Southeast Europe committee of the I.G. the technical cooperation with the countries in that area was centralized in the Berlin office. An intensive study of the possibilities of development and cooperation with the Rumanian chemical industry was undertaken in May 1939 together with men from Sparte II by request of the Ministry of Economics. At the beginning of the war the practical cooperation with Rumanian companies was increased by assisting in the sales of nitrogen plants, licensing of chemical processes, acquisition of interests, etc. By request of the German Ministry of Economics the members of the Russian economic mission who are responsible for petroleum problems have been acquainted with the technical status of the industry in Germany. Assistance was provided for negotiations on the construction of larger hydrogenation plants in Russia.

The work on the practical utilization of the technical experience of the I.G. in Brazil which was started in 1938 has been continued.

Pilot Plant Work

Experimental laboratory including pilot plants

The following subjects have either been brought to a conclusion or have reached an advanced stage of development:

(1) Hydrogenation in the sump phase and the liquid phase.

(2) Catalytic cracking and hydroforming.

(3) Manufacture of extremely knock-resistant fuels from gas fractions from the hydrogenation.

(4) Chlorination of gases (from the hydrogenation) and oils.

(5) Work on phenol oil

(6) Work on isobutyl oil and amines.

(7) Work on catalysts.

The following products have been made in the pilot plants during the course of the year:

255 tons of polymerizate from isobutylene, delivered to the ET 100 plant.
18 tons of pure isobutane for use at Leuna and other I.G. plants.
35 tons of n-butane " " " " " " " " "
1.2 tons of pentane from hydrocarbon gas for use at Leuna.
410 tons of a mixture of phenol and cresols from MW oil
643 tons " " " " " " " " ML "
9.4 tons diisobutylamine supplied to I.G. Mainkur

(Page 97 contd.)

40 tons of crude octadecylamine supplied to I. G. Wolfen, dyestuff plant.

50 tons of Mersol D for the manufacture of plasticizers and for the addition to soaps.

50 tons of Mesapon N of which 20 tons were used internally.

15 tons of Mesamoll (plasticizers) mainly for I. G. Bitterfeld.

(Page 98)

Hydrogenation in the Sump Phase and in the Gas Phase

In addition to previous experiments on raw material for the hydrogenation plant at Lützkendorf the hydrogenation of Bohemian generator tar (36% boiling up to 380°C., 28% asphalt) in the sump phase (700 ats, 3 liter reactor) was investigated at the beginning of the year for a short time. The product could be processed in 2 stages in the sump phase at 440-460°C. using molybdenum Grude as catalyst with a yield of oil (boiling up to 350°C) of about 0.45 with high gasification.

Tar from the low-temperature carbonization of brown coal (d_{20} 0.960) has been processed for a maximum yield of Diesel oil by dewaxing, solvent extraction with butane and SO_2 and pressure distillation of the brown coal paraffin wax. In this way 52% Diesel oil with a cetene no. of 49 has been obtained. The experiments have been concluded.

Work planned for 1940:

Experiments for Lützkendorf on the manufacture of gasoline from middle oil (boiling up to 325°C) from cracked oil residue.

(Page 99)

Catalytic Cracking and Hydroforming

1) The work on catalytic cracking at atmospheric pressure with fixed bed catalysts which has been started in 1938 in conjunction with Standard Oil has been continued and systematic studies were made with respect to products, catalysts and process details. Further progress was made in the manufacture of highly active alumina-silica catalysts. The best catalyst for automotive gasoline was found to be a magnesium oxide-silica catalyst, which for the same yield of gasoline gives a better distribution in the products yielded, as compared to synthetic aluminum silicate.

2) The favorable results obtained with fluid catalytic cracking in America could not be reproduced at Leuna and further work had to be postponed because of a shortage of suitable workers.

3) Catalytic cracking of middle oil under pressure is being carried out as follows:

The middle oil together with 2 to 10% of catalyst powder (cheap natural clay) is passed at 40-100 ats. through a high pressure coil heated to 450-510°C; the quantity passed through the coil amounts to up to 20 times the volume of the coil. The distribution in the products obtained by this method is better than by other methods. However, the octane number of the gasoline is lower by about 5 octane numbers and it contains more olefins. This process is at present only suited for the manufacture of automotive gasoline, especially from Kogasin products.

4) The cracking of high molecular petroleum residues (boiling at about 400°C.) into a middle oil boiling from 200-400°C has been investigated in two ways.

(a) Visbreaking at atmospheric pressure with steam at 450-490°C with pumice as catalyst.

(b) Pressure distillation at 5-25 ats. with 5% Grude as catalyst at 440-480°C.

With both processes 20 to 30% of newly formed middle oil was obtained in a single pass. The second process proved to be simpler in operation and permitted thruputs up to 15 kg/l/h.

5) A special method for the manufacture of highly knock resistant gasoline was used, namely, the catalytic reforming of higher boiling gasoline fractions in the presence of hydrogen (hydroforming). The experiments were at first carried out in accordance with the work of Standard Oil using Cr_2O_3-Al_2O_3 and MoO_3-Al_2O_3 catalysts at 10-30 ats. pressure and 500-540°C in the presence of hydrogen. Several 2- and 4-liter reactors and one 25-liter reactor were used. Straight-run gasoline and gasoline from the preliminary hydrogenation from Leuna were used as charge stock. The best results up to now were obtained with a molybdenum catalyst. Gasoline with an octane number of 79 was obtained from natural petroleum naphtha and gasoline with an octane number of 82 resulted from hydrogenation gasoline.

Work planned for 1940:

Experiments will be conducted on the processes which are suited for the manufacture of aviation gasoline. A 100 liter pilot plant for catalytic cracking with a fixed catalyst bed at atmospheric pressure shall be put into operation to obtain data for the design of larger units. The hydroforming of Rumanian, Russian and hydrogenation gasoline from different hydrogenation plants shall be investigated with the aim to produce aviation gasoline.

Dr. Kaufmann spent several weeks in the USA with our contract partners in the oil industry and obtained information on their experience and the technical status of the development of catalytic cracking and hydroforming.

All experimental work in the laboratory has been concerned with the manufacture of aviation gasoline since the beginning of the war.

Manufacture of Extremely Knock-Resistant Special Fuels from Fractions of Hydrogenation Gas

The original project to erect a unit for the manufacture of 4000 tons of isooctane (T 52) per year has been expanded to 3 large units for 12-20,000 tons per year each. In the course of the design work it was found necessary to obtain additional data by several small- and large-scale experiments.

Dehydrogenation of isobutane

Small-scale experiments

The precipitation catalysts on chromium oxide basis have been abandoned and a cheap technical aluminum oxidehydrate coated with chromium salts has been used as basis for catalyst development work. It was possible to prepare a spherical catalyst of very great mechanical strength which shall be used in the large-scale unit instead of the carbon catalyst.

The catalyst experiments are being continued with special consideration of the effect of the moisture content of butane.

Large-scale experiments

A sluice-type process was chosen as best suited for plant scale operations. In a 100-liter tubular reactor experiments were made with a fixed bed catalyst and a six hour cycle of dehydrogenation and regeneration; regeneration of the catalyst was carried out in the reactor itself and was effected by burning with dilute oxygen. The results were less successful than the small-scale experiments and the detrimental effect of moisture in the feed gas was ascertained.

The experiments in the tubular 25-liter reactor showed that countercurrent flow is not suitable since the catalyst begins to dance and disintegrates. The use of tubes with larger diameter (80 mm) did not show any advantages.

Dehydrogenation of n-butane

Small-scale experiments have been continued to prepare n-butylene for the manufacture of highly knock-resistant fuels (alkylation, copolymerization). The reaction takes place in tubular furnaces with similar catalysts as with isobutane but at somewhat lower temperatures.

Polymerization of isobutylene

Experimental work was conducted to decide on the selection of a fixed bed polymerization process for the T 52 projects. The small-scale experiments can be considered as finished since it was possible to regenerate the catalyst by means of steam. The following conditions for the reaction have been worked out: Temperature 80-120°C, pressure 50 ats, catalyst: preheated technical o-phosphoric acid on asbestos or rock wool. The results of small-scale experiments were confirmed in tests with the 250-liter reactors. Polymerization experiments using liquid hot mineral acids as catalyst in a one-stage process did not show any advantages above the other procedures. The two-stage sulfuric acid process was also investigated on semi-technical scale but this process, also, did not have any advantage over the fixed bed procedure.

Experiments on the cracking of triisobutylene have been discontinued because triisobutane is now considered as a useful special fuel.

(Page 101)

Copolymerization

The experiments on the mixed polymerization of iso- and n-butylene with a fixed bed catalyst (phosphoric acid-asbestos) and also with liquid hot phosphoric acid confirmed the results received from America. It was possible to prepare a copolymerizate which after hydrogenation had an octane number of 93-95 at a conversion of 50-60 percent per pass. Further development work will not be carried out because of the promising alkylation experiments.

Alkylation

Because of the lower consumption of sulfuric acid and the good yield obtained only n-butylene was used as the olefin component. Alkylation takes place at atmospheric pressure at 0°C. and an olefin-isobutane ratio 1:20 with a yield of total alkylate of up to 230% based on the olefin charged. The olefin-isobutane mixture is added as gas. The alkylate formed is practically saturated and contains up to 86% of C8 hydrocarbons besides a very small amount of light ends and heavy alkylate. The main fraction (98-120°C) has an octane number of 93-95. A semi-technical unit of a capacity of about 0.5-0.8 tons of alkylate per day is being started in order to test this procedure on a somewhat larger scale.

Further experiments on the alkylation of normal butylene at 2-5 atm. pressure and at higher isobutane concentrations gave favorable results.

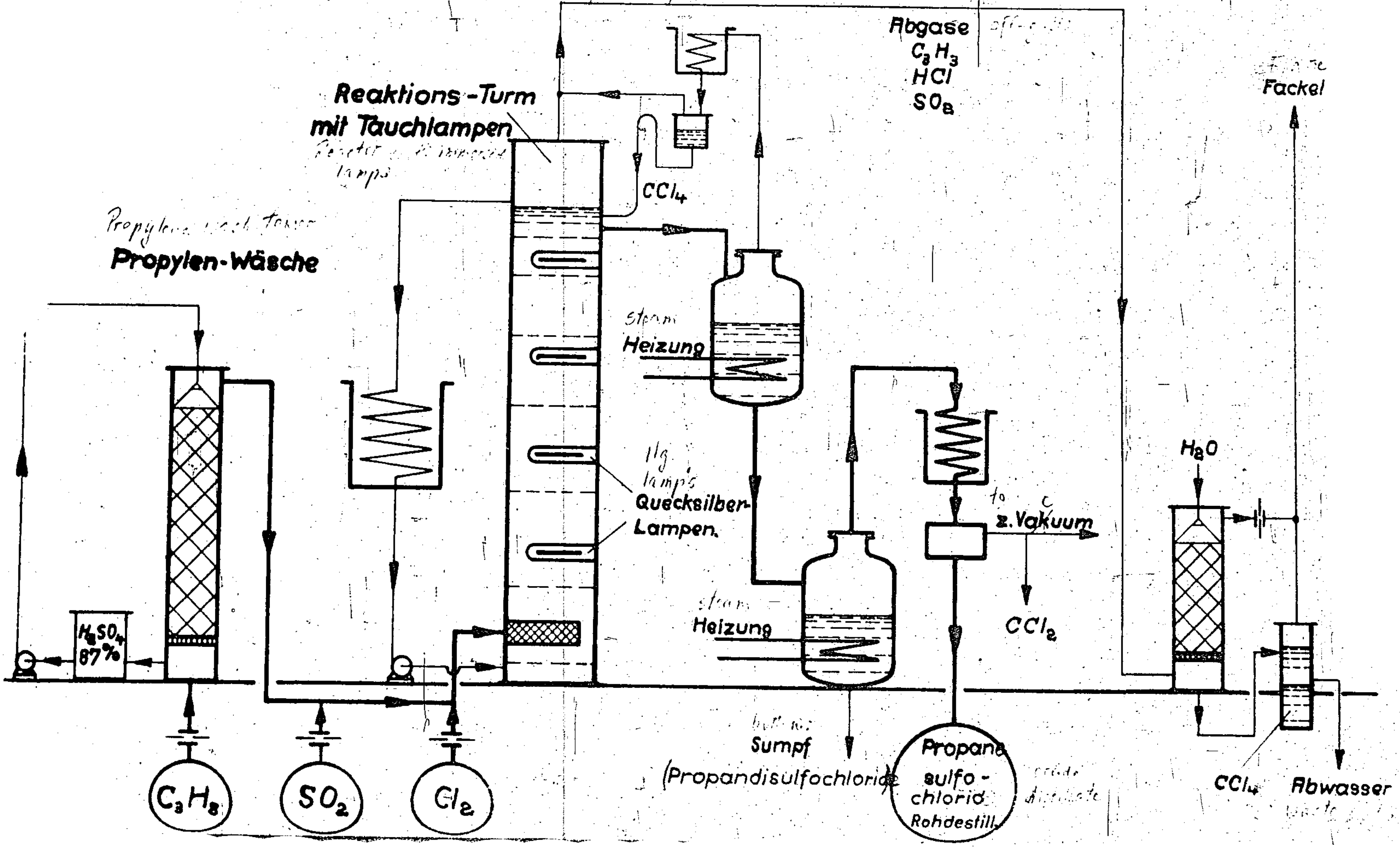
Isomerization of n-butane

It was possible to convert n-butane up to 50% into the iso-form without great losses using either a WS_2 catalyst in the gas phase or $AlCl_3$ in the liquid phase.

During his trip to the USA Dr. Kaufmann familiarized himself with the status of the development of isomerization.

Schema der Versuchsanlage zur Herstellung von Propansulfochlorid.

Flowsheet of the manufacture of propane sulfochloride



Chlorination Experiments on Gases and Oils from the Hydrogenation

Chlorination in the gas phase

The semi-technical unit for the sulfochlorination of gaseous paraffin hydrocarbons in the presence of shortwave light was started during the second quarter of the year and it could be shown that the reaction proceeds easily on a technical scale. The difficulties caused by construction materials were overcome by using ceramic materials. The unit was built for the production of 7 tons of propane sulfochloride but actually a total of 10 tons of propanesulfochloride and 1 ton ethanesulfochloride were made. Irradiation was carried out by immersing double walled quartz tubes with Heraeus burners near the sides of the reactors. Osram-low pressure quartz lamps also seem to be suitable. A commercial unit for 40 tons per month is being planned.

Chlorination of oil

The sulfochlorination, especially of Mepasin, was carried out on a semi-technical scale and in an experimental plant unit. In the laboratory sulfochlorination was carried out with Kogasin and various saturated oils, e.g., products from the preliminary hydrogenation and hydrogenated petroleum middle oil, and it was found that oils with a high content of paraffin hydrocarbons are best suited. Several selective solvents were found for the separation of the sulfochloride from unreacted hydrocarbons. SO_2 seems to be the best for technical purposes. By extraction with this solvent a suitable raw material for the soap industry (Mersol E) has been made.

An experimental plant unit for sulfochlorination has started operations at the end of April and gives satisfactory results which are in line with those obtained in small-scale tests. Irradiation was carried out by means of windows and Hg lamps arranged outside the reactor. The design in which light is applied through tubes inserted into the sides of the reactor has also been developed. The processing of Mepasin sulfochloride to plasticizer (Mesamoll) and Mepasin sulfonate (Mesapon N) has also been carried out on a larger scale. The quality of Mesamoll did not quite reach that of the product prepared on semi-technical scale. Mesapon N could be made in larger quantities without difficulties. Saponification of the half-sulfochloride and drying of the sulfonate solution on a roller type drier was carried out without special difficulties. The unit produces at present about 50 tons of Mesapon N and about 20 tons of Mesamoll per month.

Larger units for the manufacture of these products are under construction. Extensive experiments are being conducted on the introduction of Mepasin solfochloride into the soap industry.

Development work is being carried out in the laboratory on the utilization of definite Mepasin fractions as starting material for the manufacture of textile agents with especially favorably surface-active properties. The calcium and magnesium Mepasin sulfonates have become important in the manufacture of fat-free hard soaps.

Sulfamides were made by reacting ammonia and amines with sulfochlorination products. The manufacture of propane sulfamide was extensively investigated.

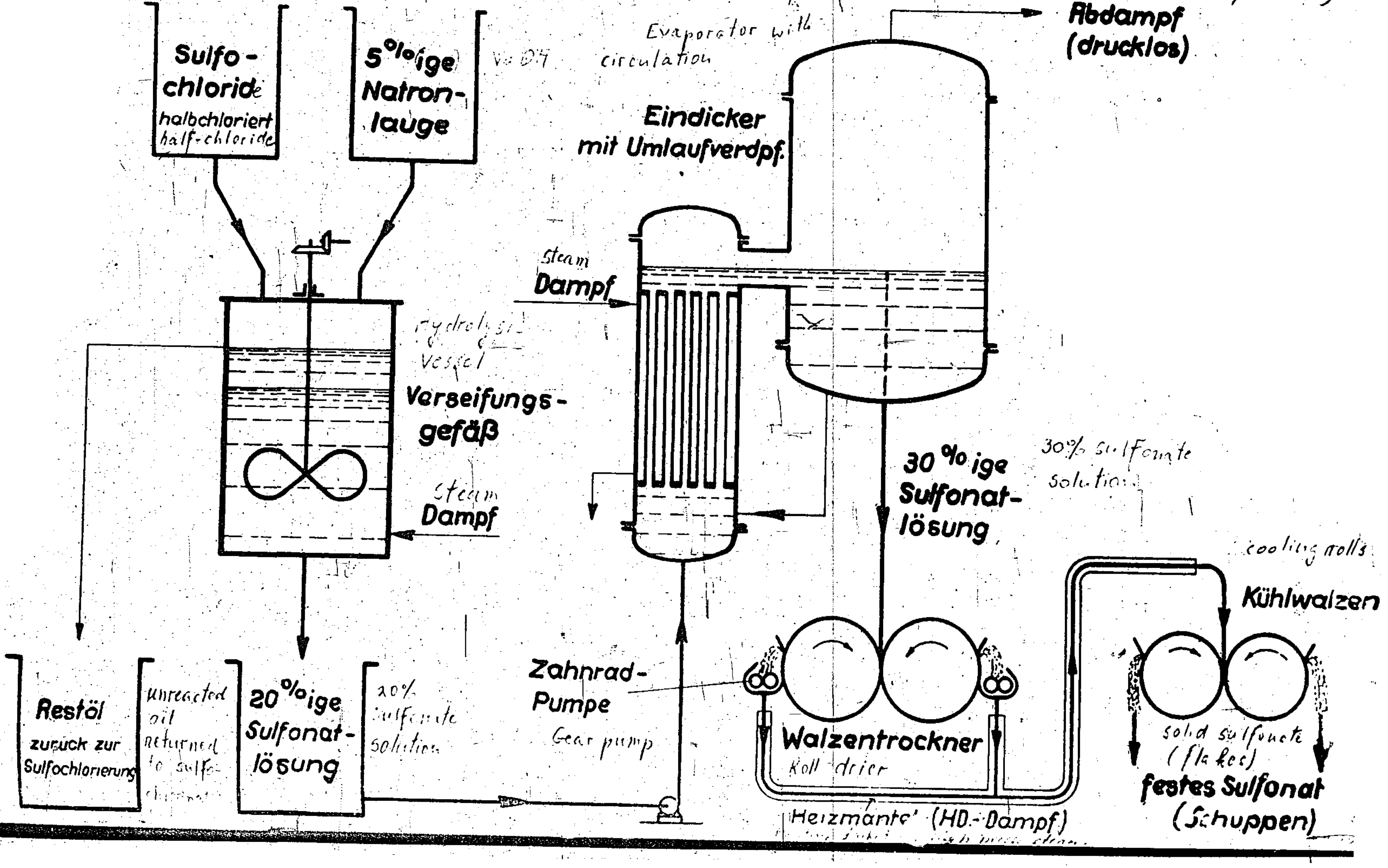
Work planned for 1940:

Cooperation in planning and starting the plant for the sulfochlorination of saturated gaseous hydrocarbons. Sulfochlorination of chlorinated hydrocarbons. Further development work in the plasticizer and sulfonate field. Cooperation in the planning and starting of plant units for the sulfochlorination of oils. Development work on the extraction of sulfochlorides with liquid SO_2.

Versuchslaboratorium. Experimental laboratory

Schema der Mepasinsulfonat - Herstellung.

Flowsheet of the manufacture of Mepasin sulfonate



(Page 103)

Work on Phenol Oil

The recovery of phenols from oil fractions by extraction with phenol water can be carried out successfully at certain definite concentrations. At Leuna the conditions are unfavorable because of the lack of an oil fraction rich in phenol. In the carbonization plant Deuben it was possible to increase the concentration of phenol in the carbonization water from 8-9 g/l. to 11-12 g/l. by mixing the water with a suitable tar fraction. In this process the less valuable high molecular phenols in the water are replaced by the valuable ones and the phenol removal by means of tricresylphosphate is thereby made more economical. The semi-technical concentration process with subsequent phenol removal has operated for two months.

The experiments to remove the neutral oil from the mixture of phenols obtained by the hot water pressure process have been continued. Extraction by means of aliphatic, low boiling hydrocarbons in conjunction with SO_2 and NH_3 was not completely successful.

The same is true for the steam distillation of the phenol oil which contains neutral oil; this treatment results in the concentration of the neutral oil in the distillate but does not completely remove the neutral oil from the residue. The addition of benzidine to the crude phenol oil resulted in the formation of a crystalline benzidine-phenol (or cresol) molecular compound which is easily separated and decomposed but the loss of benzidine due to solubility is too large to make the process practical.

Some quantities of the "hot water oil" obtained in the hydrogenation plant were distilled for the removal of neutral oil after redissolving in NaOH; 1518 tons yielded 410 tons of a mixture of phenol and cresols. After changing the method of recovery of phenols from the middle oil to countercurrent extraction with NaOH solution the resulting phenol oil-M was also worked up; 1439 tons yielded 643 tons of a mixture of phenol and cresols (phenol oil MD). In both cases the distillates were sent to Leverkusen for the manufacture of tanning agents.

The high yield of xylenols in the separation of the phenol oil initiated experiments for their reduction and demethylation. Reduction could be carried out over the methanol catalyst with a conversion of almost 100% and a yield of 98% aromatics. The low price differential between xylenol and xylene makes this process uneconomical. The demethylation with a Co-Cr catalyst on a carrier yielded 36% phenol and cresols but also 23% aromatics. The experiments are being continued.

Whereas no preliminary purification was necessary prior to the hydrogenation with a Ni catalyst of RD phenol oil and SR phenol a separate purification step was required for crude S oil. Purification took place under weakly hydrogenating conditions at high space velocity with subsequent removal of hydrocarbons and H_2S by blowing with steam. The resulting product must be protected from air during the transfer to the hydrogenation unit but can be hydrogenated to Anol (cyclohexanol) continuously without difficulty.

In the manufacture of adipic acid from these "Anols" it was possible to obtain the adipic acid as white crystals free from homologs by evaporation of the mother liquor obtained after oxidation.

The dehydrogenation of pure cyclohexanol—and also of its homologs to the corresponding ketone (Anon) was at first carried out without success using oxide and sulfide catalysts. Dehydrogenation was successful with brass catalysts of varying composition. Temperatures above 500°C. are required and iron and copper must not be used as construction material. The reaction product contained up to 95% of the ketone. Lowering of the zinc content in the brass to 5-15% permitted a lowering of the temperature by about 100° to about 420-460°C. A patent for this process has been applied for. A new process has been worked out for working up the reaction product (steam distillation of the oxime formed from the ketone). The dehydrogenation process has also been successfully applied to isopropyl alcohol.

In cooperation with the Benzol-Verband experiments have been made to prepare toluene from benzene and methanol using phosphoric acid catalysts. The problem consisted in finding an effective catalyst of long life with which it would be possible to suppress the formation of higher aromatics. The promising experiments are being continued.

On a small scale mesitol could be converted to misidine with almost 100% conversion over an oxide type catalyst of Al-Zn-Cr.

Work planned for 1940

Recovery and refining of phenols. Decomposition of higher phenols including xylenol. Oxidation of Mepasinsulfonic acid with HNO_3. Catalytic oxidation of phenols to maleic acid. Work on the dehydrogenation of cyclohexanol. Manufacture of toluene.

Work on Isobutyl Oil and Amines

The experiments on the preparation of isobutyl oil at 700 ats. did not lead to worthwhile results and have been abandoned in favor of work in the Mepasin field.

Plant manufacture of isobutyl amine from isobutyl alcohol and ammonia gave the same yields as the semi-technical work.

The experimental plant for the manufacture of octadecylamine from stearic acid, ammonia and hydrogen produced a total of about 40 tons octadecylamine. This product was supplied to the dyestuff plant Wolfen and processed for the textile plasticizer Soromin BS. Attempts to start production on plant scale caused some difficulties. The reason for this lies in the difference in the catalysts used, since the catalyst for the pilot plant was made by wet precipitation of NiS and MoS_3 whereas the catalyst used in the plant was prepared by a dry method. These catalyst questions must be clarified in small scale tests.

Adipic nitrile was made with a yield of about 60% by the reaction of ammonia with adipic acid in the presence of catalysts. Considerable equipment difficulties were encountered in the continuous operation of the process. At present a small unit has been developed for the study of catalysts. A new method for the preparation of diamines is being considered which consists in the dimerization of isobutylene chloride and subsequent reaction of the dichloride with ammonia or amines; the first step of the reaction has been successfully carried out.

Work planned for 1940

No further work is planned on the synthesis of amyl alcohol and isobutyl oil. No extensive experimental work is planned for the isobutyl amine synthesis except occasional testing of catalysts. Catalyst studies are to be made on the preparation of amines from carboxylic acids and also on the preparation and hydrogenation of dinitriles.

Work on Catalysts

Synthetic catalysts of the composition Al_2O_3-SiO_2 have been found superior to natural or activated clays with respect to their efficiency for cracking of middle oil. It was possible to prepare mechanically stable catalysts which gave good yields of olefinic gasoline. Future work is directed towards the manufacture of wear-resistant alumina-silica catalysts of definite shape which shall be used for the manufacture of knock-resistant cracked gasolines with low olefin content.

Catalysts of the type Al_2O_3-Cr_2O_3 have been further developed for the dehydrogenation of butanes and combinations of $SiO2$ and Cr_2O_3 were investigated systematically. An increase in the activity shall be obtained by subsequently applied additives.

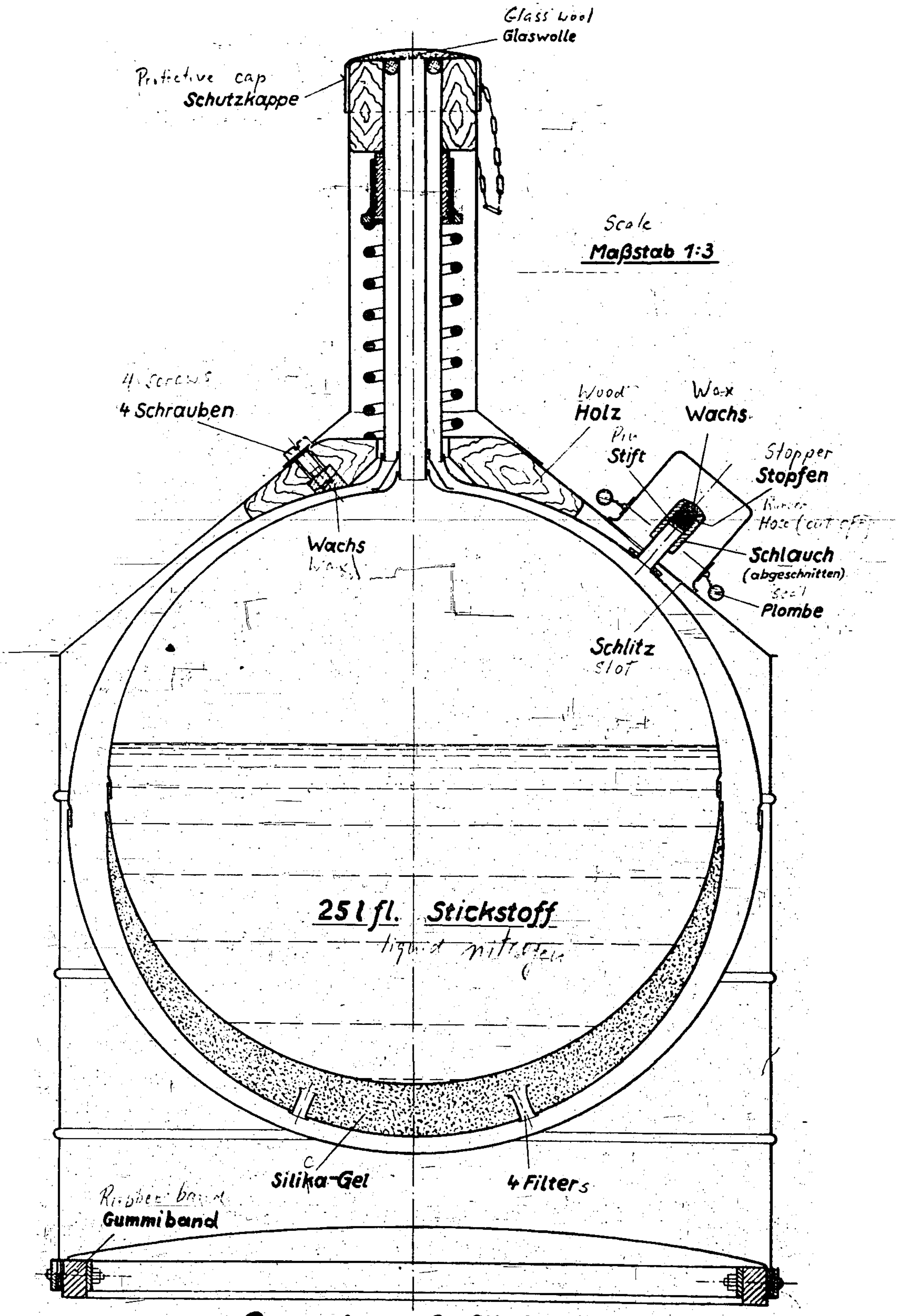
The preparation of the asbestos-phosphoric acid catalyst for the polymerization of olefins caused difficulties since asbestos is not easily obtained. Experiments are therefore under way to replace the asbestos by rock wool.

The experiments on the demethylation of xylenols are still in the first stages with respect to the catalyst. Catalysts on the basis Al_2O_3-SiO_2-Cr_2O_3 as well as oxides and sulfides of the metals of the 8th group have been developed.

The experiments on the reduction of xylenols have been concluded and the regular methanol catalyst and a zinc oxide-chromium oxide-iron sulfide catalyst have been found suitable. For the experiments on the synthesis of toluene catalysts of the asbestos-phosphoric acid type have been used.

The system Al_2O_3-Cr_2O_3 was thoroughly investigated for use in the hydroforming process. In spite of the short time of this development work it was already possible to prepare catalysts of satisfactory activity.

The sulfochlorination of hydrocarbons as a photochemical reaction has been investigated and it was found that wave length above 500 Å are not effective. The most active light lies in the region 3600-4400 Å. However, no experiments have been as yet conducted with narrow wave length bands below 3600Å.



Gaſanalyſe Dr. Wetzel

Analytical Laboratory

The inorganic group carried out the current analyses of metals, catalysts, fertilizers, etc., and also investigated a large number of coal samples from drillings in the Hermine-Henriette II Field to obtain data on their alkali content. A summary was prepared on the distribution of alkali in the Ammendorf and Wallendorf coal region. New methods have been worked out for the determination of arsenic in plant liquors and of small amounts of fluorine in phosphate sludges containing nitrose.

A quantitative method of analysis for non-conductors (ashes, catalysts) was worked out by means of spectral analysis and this method is largely independent from the composition of the sample. Experiments for determination of Pb in urine have been continued with satisfactory results. Numerous glasses, filters, organic substances and solutions have been investigated with respect to their absorption in the visible and ultra-violet light. The determination of aromatics in hydrocarbon mixtures has been started.

The organic group continued to work on analytical methods for phenol oils. A method to be used as substitute for iodometry had to be worked out due to present circumstances.

The gas analysis group was charged with so much control work that no development work could be carried out during the last half of the year. The analysis of butadiene has been further developed. The apparatus for the determination of small concentrations of CO by the iodine pentoxide method has been improved and a new safe container for the transport of liquid nitrogen was designed.

The hygienic group carried out the determination of lead in blood as a routine task. During the second half of the year the investigation of biological material from the tetraethyl lead plant at Frose has been started. A semi-automatic distillation apparatus has been developed to satisfy the increased demand for double-distilled water.

(Page 107 contd.)

The work on the analysis of air (outside atmosphere) has been consolidated with that on waste water control but the field work remains with our laboratory. Observations of the vegetation were carried out during the year in cooperation with the Wabolu Berlin and the surroundings of the Buna plant were included. Several complaints on damages caused by waste gases and fly dust have been investigated and settled.

(Page 108)

(Page 109)

Technical Department

Main shops

(A summary of new installations of machinery and extension of the facilities).

Testing of Materials

Work on intercrystalline corrosion has been continued. A heat treatment has been developed by which iron of regular commercial quality can be protected against intercrystalline corrosion. By the use of this method it is possible to make profile iron, chains, welding seams, etc., of the same properties as those made by Krupp with IZ material. The process developed at Leuna has been called "Izett-ing" and has proved itself satisfactory in the plant.

Experiments have been started to develop a material which is resistant to the attack by carbon and the work seems to be promising. The experiments are intended to replace FF 30 which is difficult to handle and to work.

Ruptures of the plugs of the preheater return bends at Gelsenberg made it necessary to measure strains on the weld. The measurements showed that in this case the weld is subject to an especially high strain in spite of the fact that it only serves as a "sealing" weld. It was necessary to change the design of the plug and of the weld.

Several fires during the transport of gas for automotive purposes made it advisable to test the behavior of gas cylinders with and without rupture discs in a fire. Motion pictures of these experiments showed that it is necessary to increase the diameter of the release openings on the valve. The experiments had to be stopped before all questions could be answered.

Pitting by water of V2A tubes of the sprinkle coolers of the nitric acid plant is avoided by using V6A-Extra material. The use of V4A instead of V2A was detrimental.

The separation of HCl and SO_2 has been investigated and it was found that it can be carried out by water at about 60°C. It was attempted to replace the rust preventive oil from Rhenania-Ossag by products made at the plant and it appears that middle oil with ammonium-magnesium sulfonate is suitable for the purpose.

Plants for protection against rust have been highly developed during the last years. The present raw material situation makes it practically impossible to use the old formulas. Since tested substitutes are not yet available and since paint tests take a long time unsatisfactory results must be expected.

[redacted]

Plant control

Betriebskontrolle Me 201.

Anlage zum Jahresbericht 1939.

Hg-loser Mengenmesser ND 25.

free flowmeter

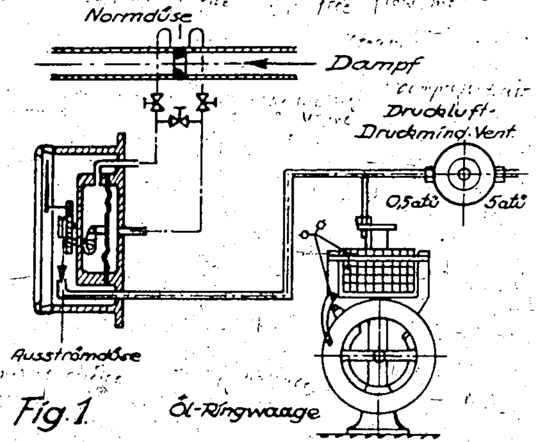


Fig. 1.

Elektr.-Hochdr.-Mengenmesser ND 700.

Electr. high pressure flowmeter

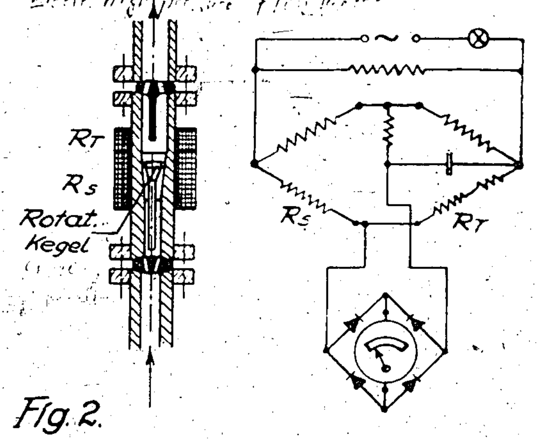
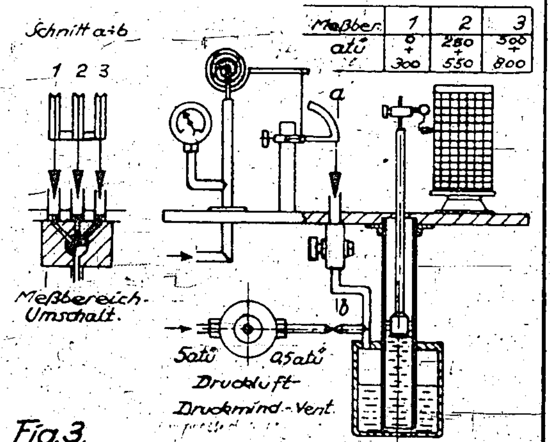


Fig. 2.

Hochdr.-Schreiber mit unterteilt. Meßbereich.

recorder



Meßber.	1	2	3
atü	0 ÷ 300	280 ÷ 550	500 ÷ 800

Fig. 3.

Membran gesteuertes Überdruck-Ventil.

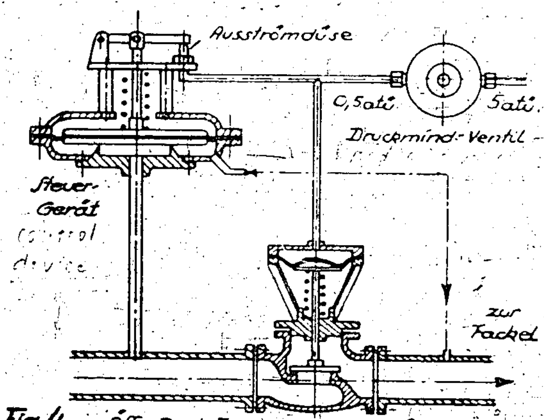


Fig. 4. Öffn.-Druck: 700 m/m W.S. Schließdr. 280 m/m W.S.

Flüssigkeits-Dichteschreiber ND 35.

recorder

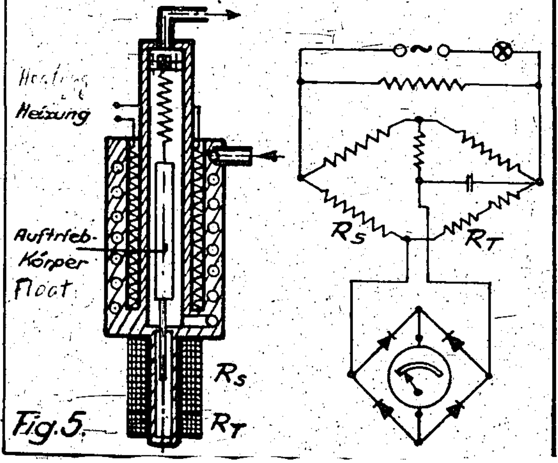


Fig. 5.

P_H-Messung für ND 10.

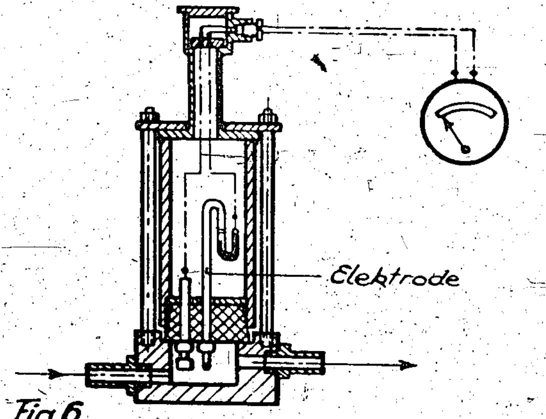


Fig. 6.

Plant Control

Measuring instruments, panel boards and automatic control devices have been provided for several plant units.

The consumption of energy and raw materials at Leuna was continuously determined by the department and these quantities have been estimated for new plants under construction elsewhere. Much work was done in the field of vibration measurements at Leuna as well as in other plants.

Among new designs for measuring and control purposes the following instruments have been newly developed. A membrane differential manometer measuring up to 25 ats. and using a dial has been designed to replace Hg differential manometers as panel board instruments and as air controlled recorders especially for Alkazid units; for the latter purpose an aluminum apparatus is used. A rapid indicating instrument for flow meter to be used at high pressures and which also serves as control instrument for automatic valves has been designed and was tested at pressures up to 700 ats. at various locations. A new apparatus for pressure measurements up to 800 ats. had to be developed which could be used in three different pressure regions, viz., 0-300 ats., 250-550 ats. and 500-800 ats.; according to the requirements, the measurements are carried out by switching to one of the three orifices corresponding to the pressure to be measured. A new membrane-controlled instrument has been built which permits to control the gas flow to a flare; it lets, e.g., pass the gas into the flare at a pressure of 700 mm water column whereas the gas flow below a pressure of 280 mm water column is shut off. New instruments have been developed for work at pressures up to 10 ats. for the determination of the density of liquids and for pH measurements. Furthermore, instruments were designed for the automatic determination of NO, the determination of Pb and Hg in urine and for the detection of traces of H_2S, the latter with an automatic alarm. For the pressure release engines a quick-closing device was built in case of ruptures. A safety valve operating at 2000 mm. water column was built for the Pattenhausen generator for hot gases (900°C.). Automatic level control for the hot separators of the coal hydrogenation plant have been successfully developed after prolonged experiments.

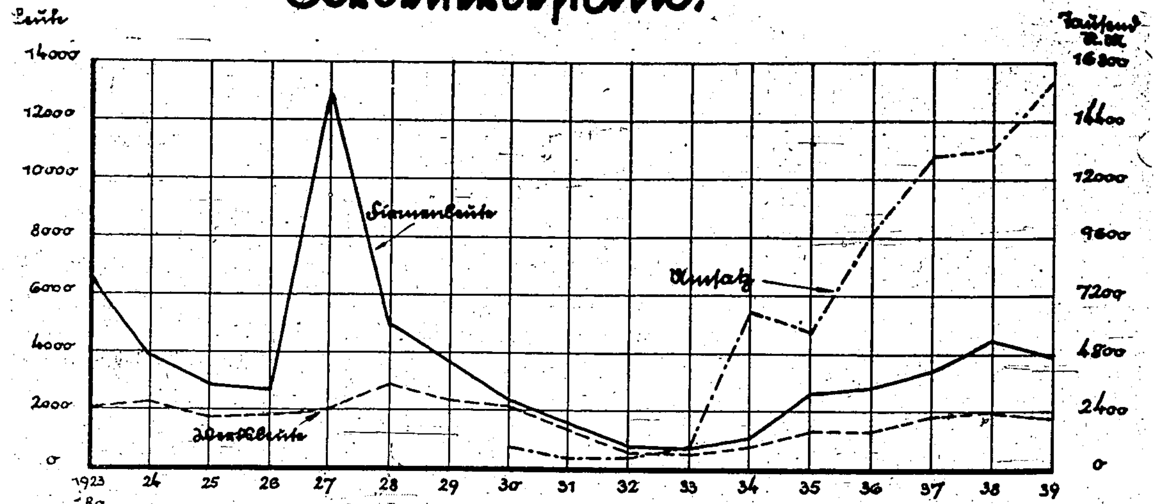
Investigations of the fine structure have been taken up again in the laboratory and structural changes of catalysts and special minerals have been investigated by means of Debye-Scherrer diagrams. The investigations of high-pressure construction materials by means of spectrum analysis have been extended and a new instrument for the determination of traces of metals has been developed using an A.C. discontinuous arc. A new automatic apparatus for the determination of the sulfur content of gases (H_2S and SO_2 besides other combustible gases) has been developed which determines the total S by means of an excess of iodine solution and registers the SO_2 portion by titration with NaOH. The instrument measures the ratio of SO_2:total sulfur. An explosion proof and portable apparatus for the determination of ammonia in plant gases has been built which determines the quantity of gas required to neutralize a known quantity of absorption liquid. Some physical constants like solubility, p_H value, specific heat, ignition temperature have been determined on adipic acid, Mepasin oil, phenol oil.

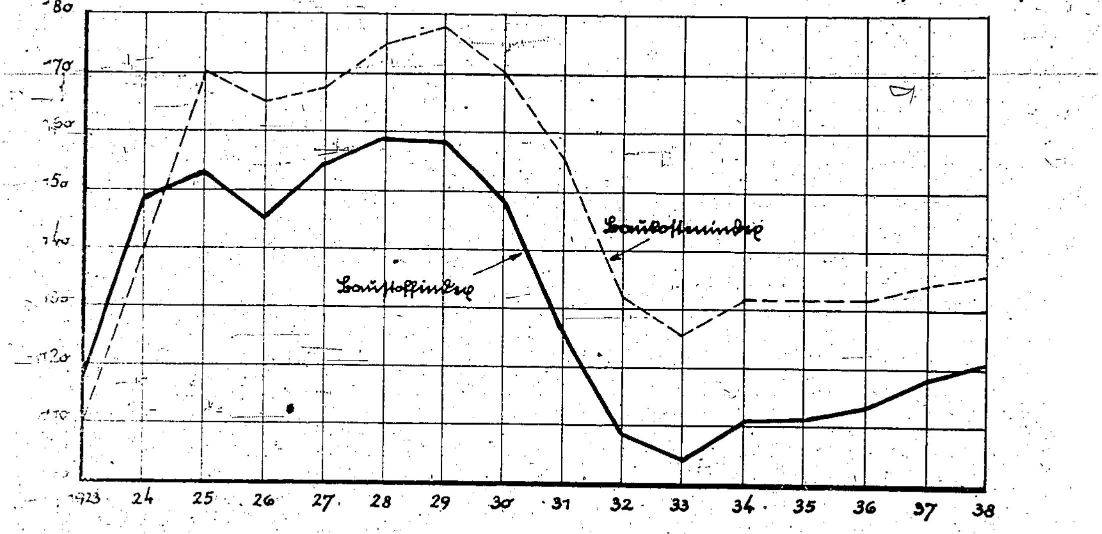
A patent has been secured covering the manufacture of the high pressure manometer.

Electrical department

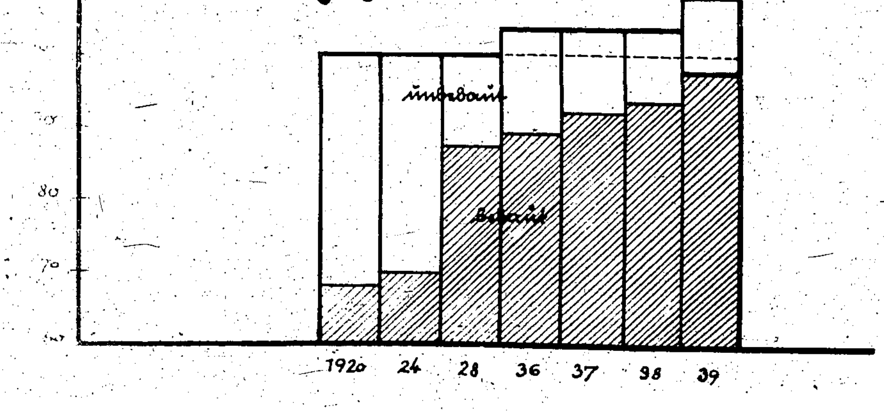
(A summary of improvments and new installations in the electrical equipment of the plant including the telephone system.)

Arbeiterbestand.





Gesamtfläche innerhalb der Dorfumzäunung im Jahre 1920 = 2,32 Mill. m² = 100%.

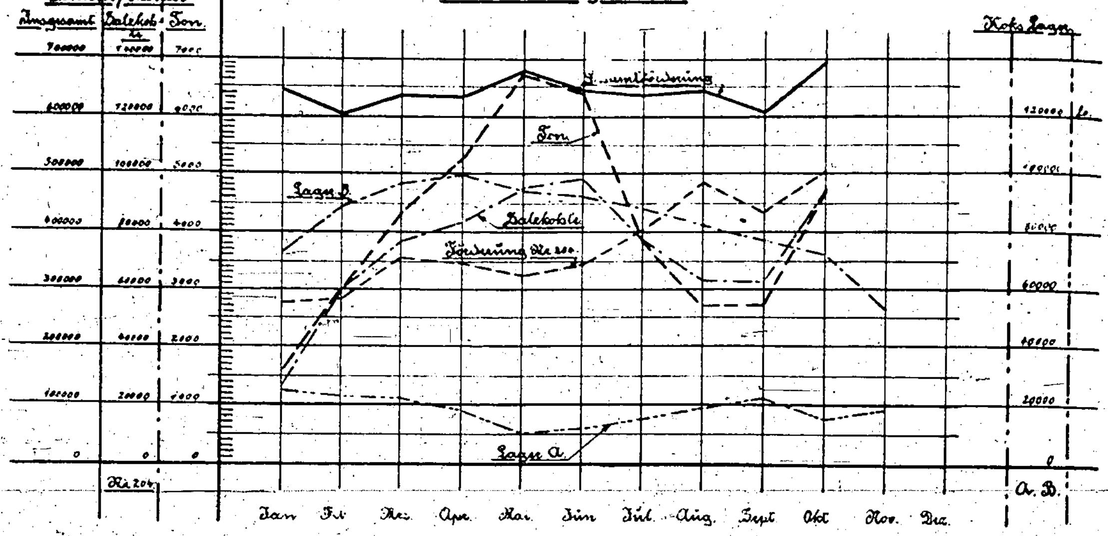


Construction department

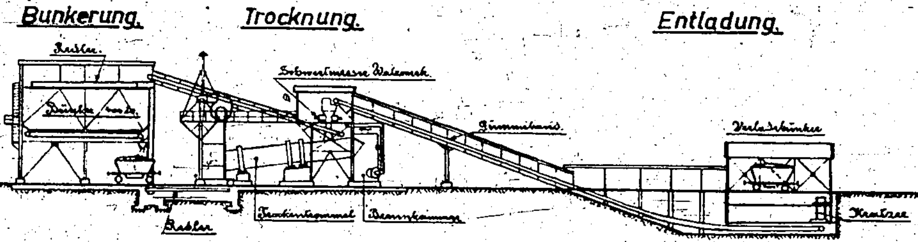
(Report on the construction work carried out during 1939).

Kohlen-u. Koks-Förderanlagen Me.

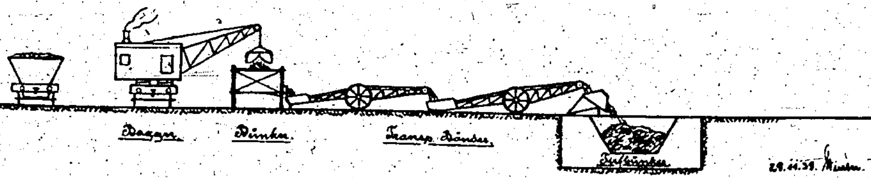
Förderleistungen 1939.



Schema der Tonaufbereitung Me 142.



Automatische Frostwagen Entladung.



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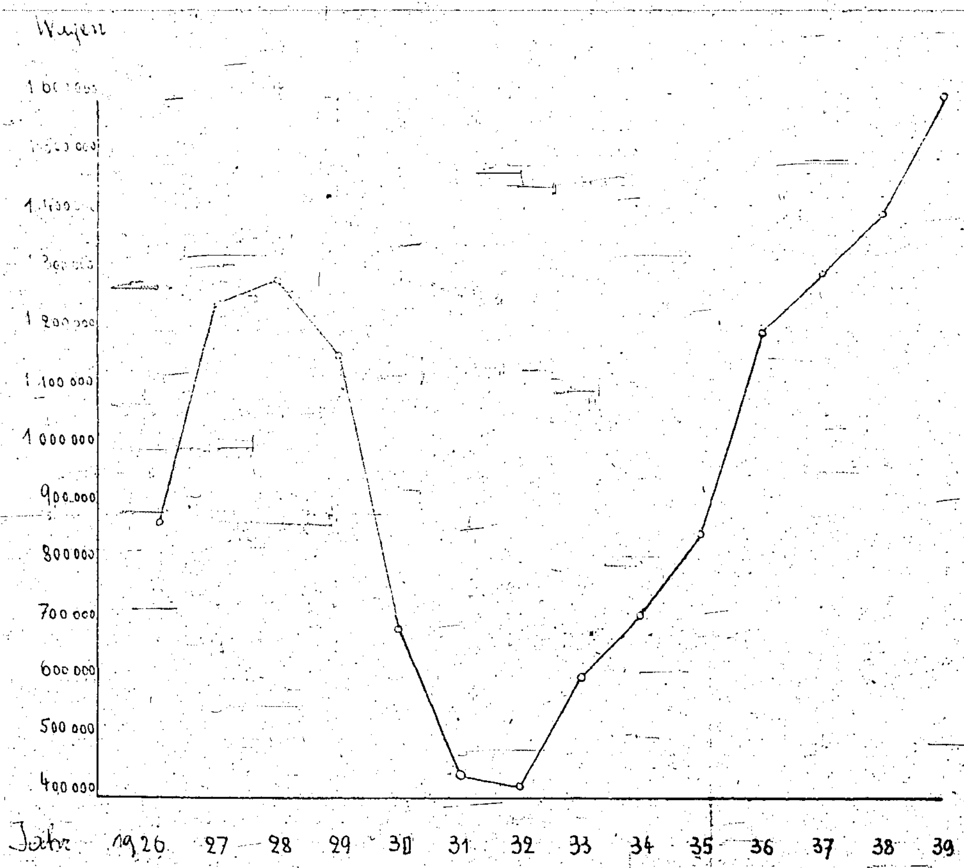
Coal and coke transportation

(Report of the quantities of coal and coke transported during the year together with a summary of changes in the transportation equipment.)

Gesamt - Wagen - Umlauf



Rail transportation

(Report on freight car loadings and the status of freight transportation by rail).