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JUL 18 1945

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TRANSLATION

AMMONIA PLANT MERSEBURG

ANNUAL REPORT 1940

Table of Contents:

	<u>Pages</u>
✓ Short General Survey	1 - 4
✓ Low-Pressure Activities	5 - 21
✓ High-Pressure Activities	22 - 31
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Salt Plants	32 - 37
✓ Organic Section	38 - 73
✓ Hydrogenation	74 - 103
Power Section	104 - 109
✓ Department of Economy Investigations	110 - 111
✓ Testing Laboratory (Pilot Plants)	112 - 124
✓ Analytical Laboratory	125
✓ Technical Department	126 - 135
Plant Protection Against Air Raids	136
Raw Material Procurement Department	137 - 138
Patent Office	139
Social Welfare Department	140 - 141
Law Department	142

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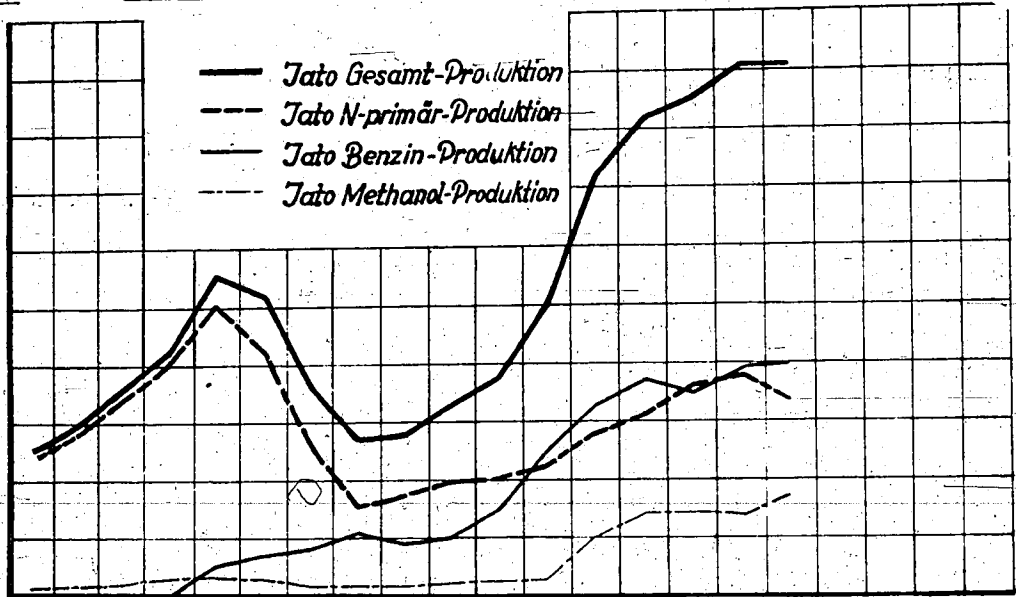
Jahres-Produktionen der Synthesen.

Jato Produkt

·000 000

900 000
800 000
700 000
600 000
500 000
400 000
300 000
200 000
100 000

— Jato Gesamt-Produktion
- - - Jato N-primär-Produktion
— Jato Benzin-Produktion
- - - Jato Methanol-Produktion



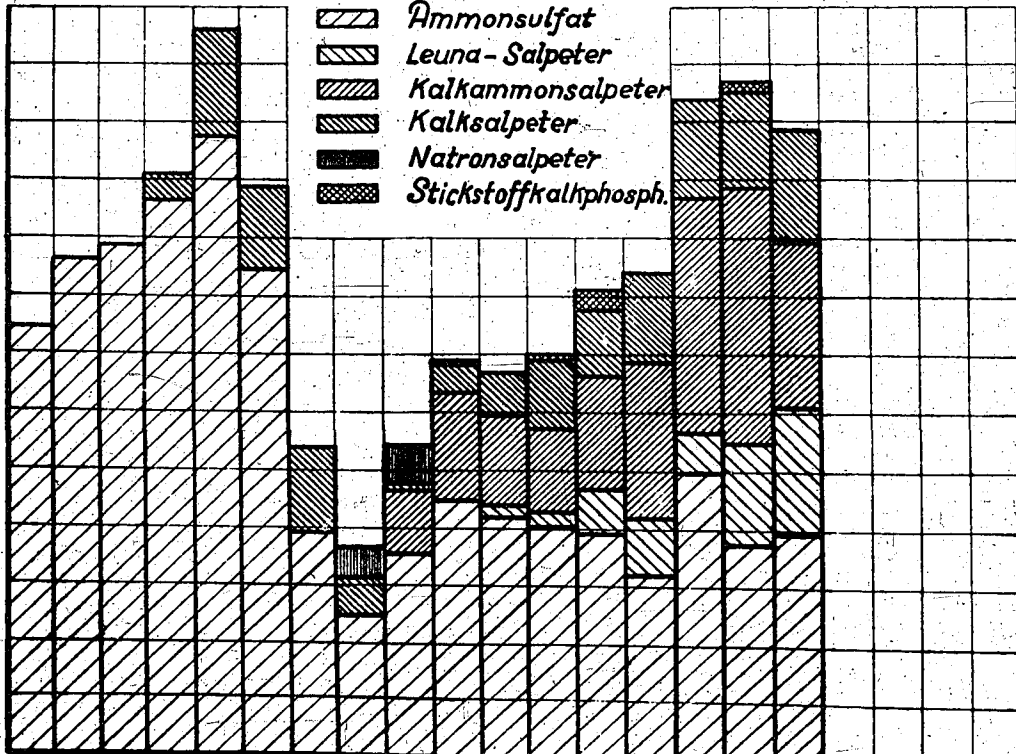
1924 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44

Salz-Produktion.

Jato N

260 000
240 000
220 000
200 000
180 000
160 000
140 000
120 000
100 000
80 000
60 000
40 000
20 000

Ammonsulfat
Leuna-Salpeter
Kalkammonsalpeter
Kalksalpeter
Natrumsalpeter
Stickstoffkalkphosph.



1924 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44

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Short General Survey of the Plant Operations during 1940
(Pages 1-4)

During the entire year 1940 the Leuna plant was entirely on a war basis.

The plant was attacked seven times by enemy airmen; during four of these attacks bombs were dropped upon the plant. Forty bombs fell on the plant itself, and about 80 bombs on the immediate vicinity. Material damage caused thereby amounted to about 2,000,000 reichmarks (RM). The drop in production caused by these enemy air attacks amounted all in all to 9600 tons (metric), 3300 tons of which were benzine, 1000 tons C₂-C₄ hydrocarbons, 2900 tons nitrogen, and 1100 tons alcohols. Air raid alarms were sounded 42 times in 36 nights, with a total duration of 80 hours.

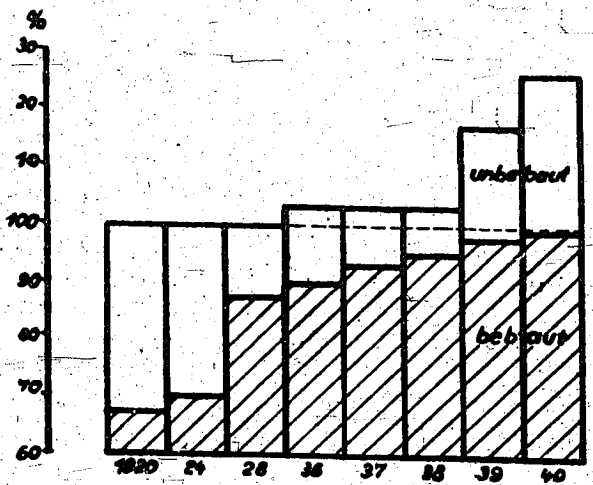
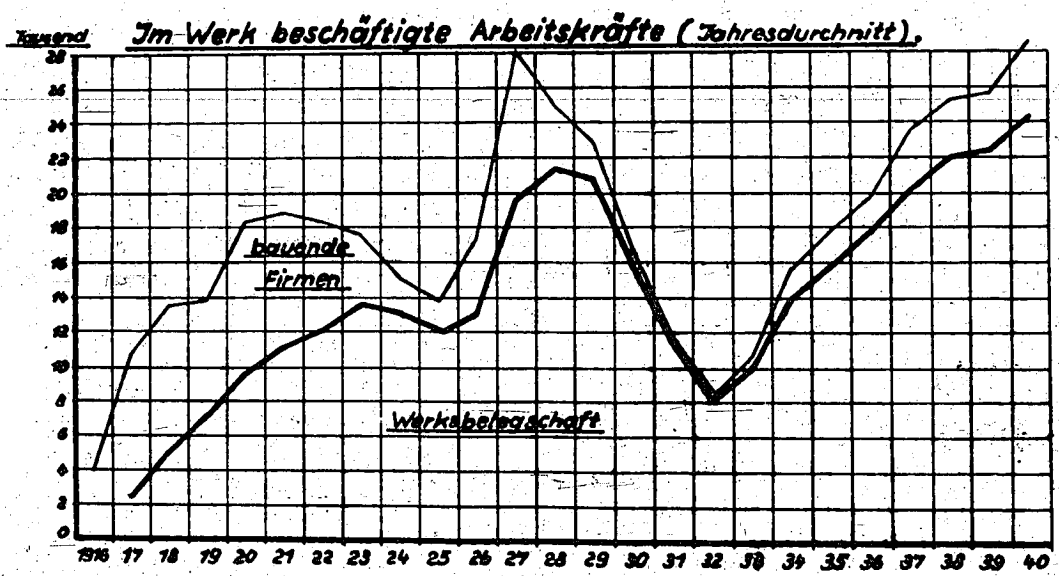
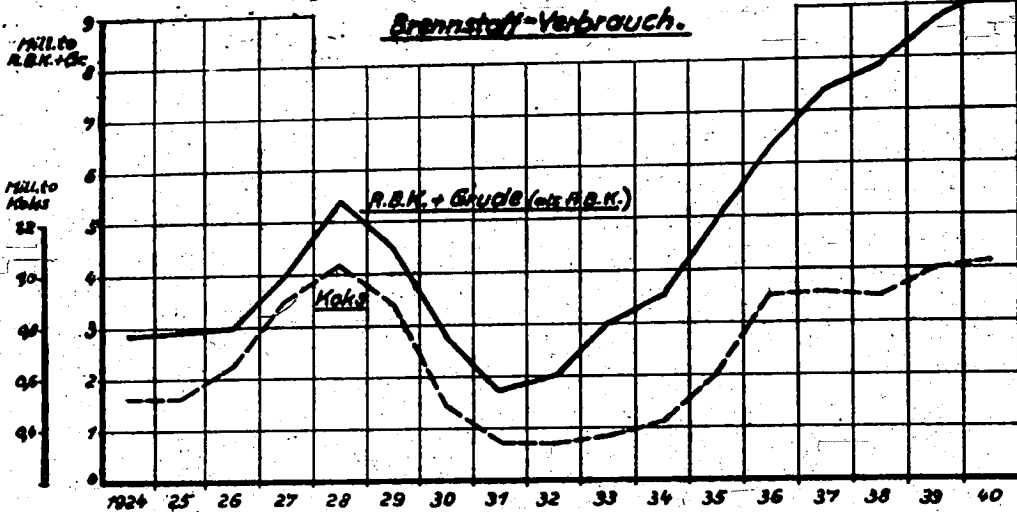
The protection of the plant was further extended. Particularly important structures, equipment, and machinery were protected against bomb fragments by the erection of dams, brick walls, or bags filled with cement. The tank installations were lined with preformed concrete slabs.

Every member of the staff was given a gas mask. At certain exposed places small shelters were built for those operators who had to remain at their posts during the attacks. All in all a sum of 6.5 million reichmarks (RM) has up to the present been spent for air-raid protection purposes inclusive of that for plant protection.

The output of the main syntheses could be maintained at the same level as in the preceding year, viz., 910,000 tons per year. In consideration of the war requirements, power fuel production was pushed to a maximum. There were produced 400,000 tons of benzine, 237,000 tons

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

85
 86
 87
 88
 89
 90
 91
 92
 93
 94
 95
 96
 97
 98
 99
 100

C O N F I D E N T I A L

of which were aviation gasoline, 76,000 tons Diesel oil, and 87,000 tons automobile gasoline. Power gas output amounted to 76,000 tons, as compared with 42,000 tons during the preceding year. Power fuel obtained from coal amounted to 69% of the total production (as against 68.5% in 1939).

In the alcohol field the production rose sharply from the 140,000 tons (metric) in 1939 to 174,000 tons in 1940. The production of pure methanol rose particularly sharply from the 17,000 tons in 1939 to 52,000 tons in 1940. As in the preceding year the production of isobutyl alcohol for iso-octane was pushed to a maximum.

The nitrogen production had to suffer a recession from the 1939 output of 385,000 tons to 334,000 tons in 1940 due to the important increase in production in the two other synthesis plants. The production of "Hoko" acid ("Hokosaure") for derivatives of strategic importance rose from 6000 tons to 9800 tons.

The projects relating to strategically important products, the production of which was taken over by Leuna, were pushed intensely. In furtherance thereof 528,000 square meters of new land situated southwardly of the works were acquired by purchase, and the total area of the works was thus enlarged to cover 2957 million square meters.

For organizational purposes the new projects were assigned to the Organic Section.

Organic Section

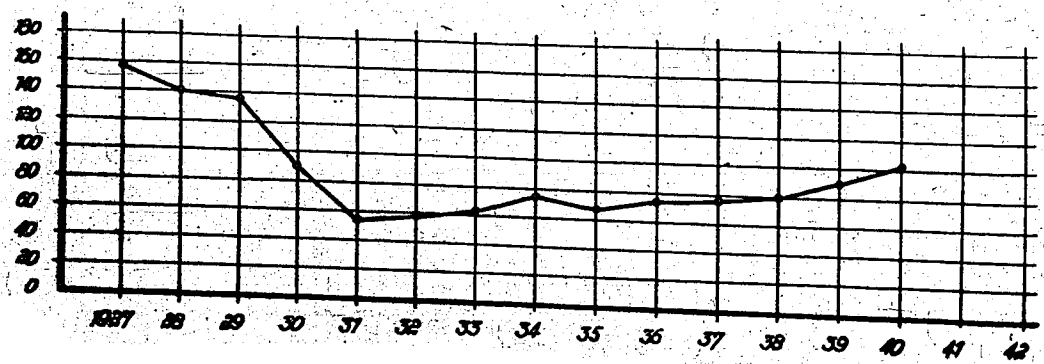
In the course of the year the production of "Mersol" was started. At the end of the year it had reached a production capacity of 20,000 tons per year, and is being enlarged to reach 50,000 t.p.y. (tons per year). The T-52 plant is in course of construction for an intended output of 19,000 t.p.y. Upon completion of the plant and by changes

C O N F I D E N T I A L

Wohnungswesen

	Stand Dez. 1924	Neubauten und andere Zugänge									Stand am 31. 12. 40	Geplant 1941
		25/27	28/30	31/33	34/36	1937	1938	1939	1 9 4 0 fertig i. Bau			
1) <u>Werkseigene Wohnungen:</u> Leuna, Spergau usw. Dürrenberg	1014	219	64	12	14	18	12	12	-	50	1415	18
	-	-	-	522	-	-	-	-	-	-	522	-
	1014	219	64	534	14	18	12	12	-	50	1937	-
2) <u>Über Baugesellschaften bezu- schützte Wohnungen:</u> Leuna, Dürrenberg, Karseburg Halle (S) Weißfels, Naumburg Großkorbetha	20	209	1295	166	20	-	-	60	-	154	1924	350
	-	21	225	-	278	373	-	56	155	66	1174	244
	12	18	162	-	138	-	-	54	-	-	384	-
	-	-	-	-	-	-	-	-	-	-	-	16
	32	248	1682	166	436	373	-	170	155	220	3482	-
3) <u>Darlehenswohnungen:</u>	3	38	157	1	143	14	5	7	-	-	368	-
4) <u>Kleinstedlungen:</u> Dürrenberg-Goddula Weißfels Ammendorf Großkorbetha	-	-	-	-	51	64	-	81	-	-	196	100
	-	-	-	-	78	63	-	-	-	-	141	-
	-	-	-	-	98	74	-	-	-	-	172	-
	-	-	-	-	-	-	-	-	-	105	99	204
	-	-	-	-	227	201	-	81	105	99	713	-
5) <u>Niedersachswerfen:</u> Werkwohnungen Kleinstedlungen	32	-	-	-2	-2	-	1	-	-	-	29	-
	-	-	-	-	-	-	-	10	12	-	22	-
	32	-	-	-2	-2	-	1	10	12	-	51	-
Gesamt:	1081	505	1903	699	818	606	18	280	272	369	6551	728
Stand 31. 12. 1927		1586										
• 31. 12. 1930			3489									
• 31. 12. 1933				4188								
• 31. 12. 1936					5005							
• 31. 12. 1937						5612						
• 31. 12. 1938							5630					
• 31. 12. 1939								5910				
• 31. 12. 1940									6182			
Auf 1000 Mann eff. Gefolgschaft entfallene Wohnungen:	87	72	252	373	256	259	248	239	220		233	

Unfälle je 1000 Mann Arbeiter-Gefolgschaft.
(meldepflichtige Unfälle)



C O N F I D E N T I A L

in the process and enlargement of the plant it is intended to produce up to 44,000 t.p.y. of alkylate gasoline ("Alkylatbenzin").

Also in course of construction are plants for the production of the dehydrogenation and polymerization catalyst for the Leuna, Scholven and Poelitz T-52 plants. In other buildings equipment for the production of the catalysts for the Moosbierbaum and Leuna HF plants and for the Waldenburg toluene plant was installed. The substitute catalyst for 5058 is being produced in a new plant situated to the north of the works.

In an intermediate-product building products from the isobutyl alcohol and methanol synthesis are further processed. In this building are to be made formaldehyde, propionaldehyde, trimethylolethane, and various high-molecular esters for special lubricating purposes. Furthermore, a pure methanol plant now being erected is to be put into operation in the spring of 1941.

In another part of the southside part of the works, plants for the further processing of the phenols are in course of construction. According to the planned program about one half of the phenols and cresols recovered are processed to yield "Luran" (about 4000 t.p.y.) and adipic acid (about 7000 t.p.y.). In the available plant in the old part of the works 10,300 tons of phenols / cresols were isolated in the course of the year 1940, of which 4700 tons were M-phenols extracted from middle oil A. The capacity of this plant will be expanded to reach 8000 t.p.y. The production of "Luran" amounted ^{to} 67.6 tons, that of adipic acid 650 tons.

The production of lubricating oils rose from 1900 tons in 1939 to 3900 tons in 1940. Expansion to reach an output of 10,000 tons is nearing completion.

C O N F I D E N T I A L

C O N F I D E N T I A L

The construction of a plant for the production of 3600 t.p.y. of acetic acid by the maximum-pressure process from methanol and carbon monoxide will be started; it is intended to enlarge it later on to reach 12,000 t.p.y.

Low-pressure processes: In the gas works the reconstruction of the Brassert grates and the mechanization of the operation is being continued. Up to the present ten gas producers have been reconstructed. For the generation of a power gas for the gas compressors a process was developed for the production of a high-B.T.U, low-hydrogen power gas of 2000 WE (large calories?) from coke by blowing and gasifying with CO₂. In pilot-plant scale tests the recovery of a high-% SO₂ for supplying the "Mepasin" plant from the after-combustion of the Claus furnace plant with ammonium sulfite liquor has been tried out.

High-pressure operations: In the pressure-water purification plant the new building capable of handling 85,000 cubic meters of crude gas was put into operation towards the end of the year. A special building has been erected for housing the Hy-hydrogen expansion engines of the hydrogen purification plant, and partly placed into operation. In the ammonia works the oil-scrubbing installation in the final circuit was replaced to the extent of 50% by a scrubber operated with liquid ammonia. The processes for the production of "Luran" and oxime were improved and the plant expanded for an output of 30 t.p.m.

Salt Works. The production of "Hoko" acid was expanded so as to reach 1500 t.p.m. The projected further expansion will not be carried out.

The output capacity of the adipic acid plant was expanded to reach 200 t.p.m. (commercial grade). Towards the middle of the year a reconversion for the production of pure adipic acid was effected.

C O N F I D E N T I A L

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Hydrogenation: War conditions made it necessary to develop a locally produced dominant-pool phase catalyst. It was adopted in Leuna and Magdeburg with great success in large-scale tests.

In the gas-phase process it was found that an improvement can be achieved in the action of catalyst No. 6434 by first removing the nitrogen compounds. For that purpose the circulating gas is scrubbed with water for the removal of any NH_3 present therein. The weak bases present in the middle oil B are to be extracted by a sulfuric acid wash.

Researches in the Experiment Station Dr. Herold:

The hydroforming process has been developed to a point such as to enable us to start the construction of a plant in Moosbierbaum. The experiments on the dehydrogenation of butane supplied data for the T-52 plant. Further development work was carried out in the alkylation field. The production of toluene from methanol and benzene has now been developed to a point such as to make it possible to build a plant in Waldenburg with a capacity of 50,000 t.p.y. of toluene. In the sulfochlorination field experiments were carried out to apply the process to mineral oil.

Newly taken up was the addition of carbon-monoxide and hydrogen to olefins ("oxo" process) for the production of high-molecular alcohols and aldehydes which are valuable as intermediates for detergents. The researches in connection with the KW-synthesis were switched over into this direction.

By the discovery of new catalysts for the production of adipic acid dinitrile, as well as by the successful synthesis of 1,5-diamino-2,4,4-trimethyl-pentane further progress was made in the field of intermediates for the synthetic-plastic industry. A 30 t.p.m.-pilot plant for the production of adipic acid dinitrile and hexamethylenediamine is being planned.

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C O N F I D E N T I A L

Power Department: Since the new producing departments came into operation faster than new boilers were procured the steam consumption was way ahead of the steam generation capacity. In spite of the procurement of increased amounts of current from extraneous sources (up to about 4000 kw) our own requirements were not fully met during the winter months so as to cause a drop in various outputs. At present 15 high-pressure boilers are in operation, 5 additional boilers in building 203 are to follow during the period January-June 1941. Any equalization between consumption and supply will be attained only when the planned new boiler house in the southern sector of the works with 6 high-pressure boilers is put into operation. Even then it will be necessary to procure rapidly additional high-pressure boilers in order to work in the proper proportion the large coal-salt deposits available which comprise two-thirds of our total coal deposits.

The water consumption during the summer months reached a figure of 34,000 cubic meters per hour, and it was just about possible to reach this figure. An expansion of the filter plant and the laying of a fifth pipeline is planned in order to satisfy the future greater summer requirements. For the abduction of the greater volume of waste waters the new waste water canal with a capacity of 10,000 cubic meters per hour was placed into operation at the end of the year.

Power gas supplies: Since the complete fractionation of the high-% gas in the hydrogenation plant, technically pure compressor gas can be produced only by admixing considerable amounts of coke-oven gas, partly by cutting down the high-H Winkler gas (and by the addition of propane or butane during the explosion strokes). A definitive improvement can be expected only with continued electrification of the compressors which

C O N F I D E N T I A L

C O N F I D E N T I A L

is being carried out in proportion as additional current becomes available in accordance with the installation of additional high-pressure boilers.

Raw material supplies: The consumption of run-of-mine lignite ("brown coal," Braunkohle) including granular fines amounted to 9.3 million tons which means an increase of 6% as against the corresponding figure for 1939. The consumption of salt coal ("Salzkohle") rose from 1.7 million tons in 1939 to 1.9 million tons; it is expected to reach 2.1 million tons in 1941. The procurement of "RBK" (= Rohbraunkohle = run-of-mine brown coal) during this winter did not cause the great difficulties as in the preceding year as a string of railway cars and two new locomotives were put into service and as several other locomotives had been borrowed from the Reich railroad administration. The procurement of fresh coke likewise was better than in the preceding year. It was possible to replenish the lowered stocks in the storage bins in the course of the summer up to their maximum capacity of, roughly, 200,000 tons. Only in December became it necessary to draw from our storage supply due to lack of railroad cars. At the end of the year about 167,000 tons were still in storage.

Iron and Steel: As to iron and steel 71700 tons, including 5200 tons for Moosbierbaum had been allocated for new installations during the year. For repairs we received 23200 tons which, however, were insufficient to make up the losses in allocation suffered during 1939.

Personnel: Due to the greater tasks imposed upon us by the war economy an extraordinary demand for labor arose, particularly for the construction and erection of the new installations which could only be partly covered. Besides this, replacements had to be found for 1000 Saarlanders. All in all, 6000 laborers were hired for the plants, 5600 laborers for the firms, and 553 employees, and 41 chemists and engineers were given positions. As against these figures, 3200 laborers left the works and 3000 left the firms.

C O N F I D E N T I A L

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A number of union members had to be called back from the armed forces so that at the end of the year only 213 employees, 1843 plant laborers and 1141 firm laborers are serving in the armed forces.

The discipline among the members of the parent union, also particularly during air raids, was exemplary. Difficulties arose among the laborers drawn from outside sources (foreigners).

The housing of the laborers drawn from outside sources was accomplished by means of two barrack towns and 30 community camps comprising 6500 beds. Foreigners serving as laborers numbered 2912 at the end of the year; among them were 270 French prisoners.

Contracts: With the firm Furstlich Pless'schen Bergwerks Akt.-Ges. a contract was entered into covering a 50% participation of the I.G. in the Fuerstengrube coal mine. With the Ruhrchemie and Henkel A.G. (Duesseldorf) a contract for the joint exploitation of the "Oxo" process patents and for the construction of a 10,000 t.p.y.-plant. With the RWIM (Reichswirtschaftsministerium = Reich Ministry of Economic Control) a sales guarantee covering 80,000 t.p.y. of "Mersol" in Leuna and Wolfen, respectively. With the RIM a 5-year sale-and-guarantee contract covering the purchase of 19,000 t.p.y. of T52. With the Wifo a contract covering the supply and processing of 120,000 t.p.y. of HF gasoline in Moosbierbaum. The procurement of "Kogasin" for the production of "Mersol" in Leuna and Wolfen was ensured by numerous contracts with the various Fischer plants. A contract was entered into with the Hermann Goering Werke and the Ruhrchemie covering the working up of liquid ammonia and aqua ammonia in Leuna and Oppau, respectively for the purpose of effecting savings in sulfuric acid.

Participations: Members of the operating staff participated in the planning, erection, and starting of plants in Luetzkendorf, Poelitz, Gelsenberg, Bruex, Wesseling, Flechhammer, and Waldenburg

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Housing Conditions: For our own company personnel 155 dwellings and 117 bungalow-type houses were constructed. In the course of construction are 270 houses and 99 bungalows. For the company housing project 18 houses and 8 apartment houses are constructed for works managers, and 24 apartment houses for foremen. All in all plans are being made for 1941 covering 628 dwellings and 100 bungalows.

Accidents: In 1940, 2796 accidents occurred, of which 2449 were subject to obligatory notification; 3 fatal accidents happened in the plants, and 3 others, likewise fatal, happened outside of the plants. The accidents subject to obligatory notification rose from 91.8 to 102.5 per 1000 members of the labor force; on the basis of 100,000 working hours there was a rise from 3.37 to 3.68. The increase is to be attributed in the main to the induction of new labor from outside sources.

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Production at Low Pressure in the Year 1940

(And Granular fines Consumption)

Total CO / H₂
for Syntheses
inclusive of
Wi-water gas
portion

Power Gas 1000 Kcal
inclusive Hy heating
gas without Sti-
Synthesis gases

TBK Production

Granular fines consumption

A-Carbon
Claus oven

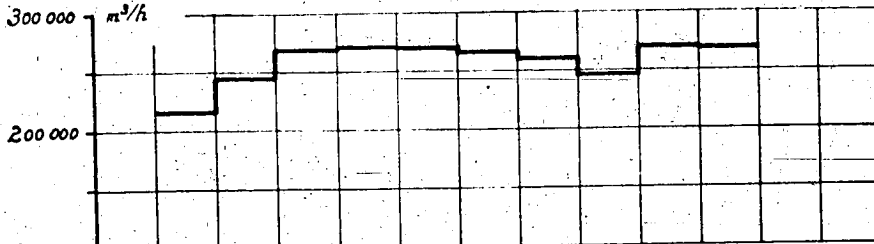
T.p.m. Sulfur

T.p.m. SO₃

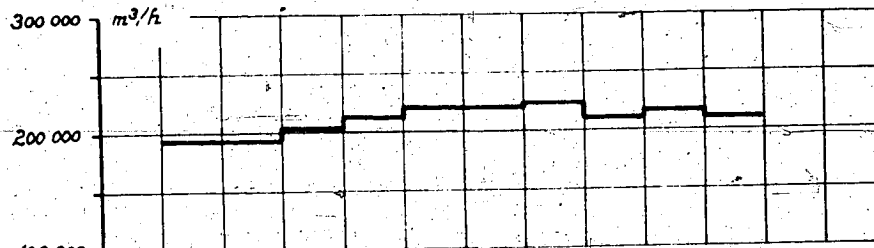
o	Jan.	Febr.	März	April	Mai	Juni	Juli	Aug.	Sept.	Okt.	Nov.	Dez
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Produktionen des Niederdrucks im Jahre 1940.

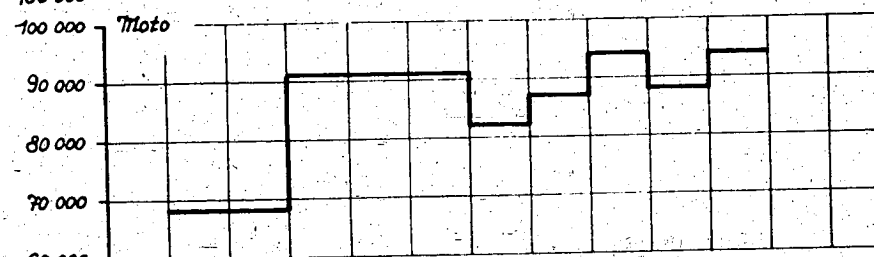
(und Grude-Verbrauch)



Gesamt CO+H₂
für Synthesen.
einschl. Wi-Wassergas-
Arbeit.



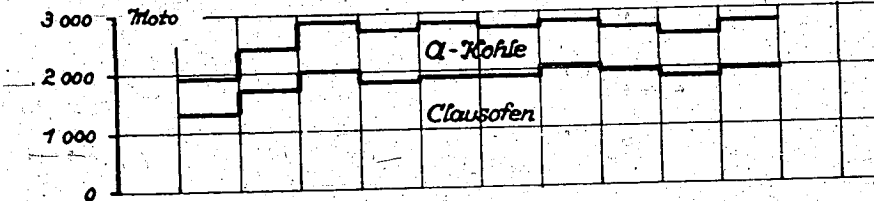
Kraftgas 1000 kcal
einschl. H₂-Reingas
ohne Si-Synthesegase



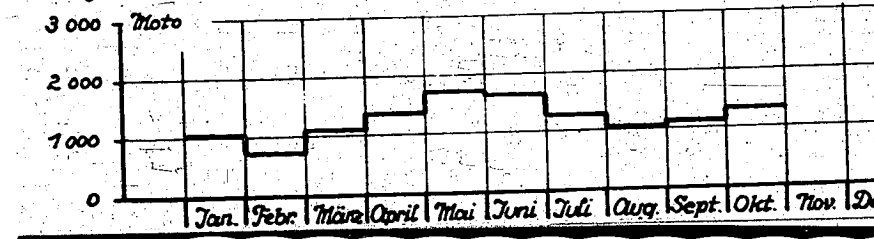
T.B.H.-Erzeugung.



Grude Verbrauch



Tons Schwefel



Tons SO₃

Jan. Febr. März April Mai Juni Juli Aug. Sept. Okt. Nov. Dez

page 6

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Graph opposite Page 6

Pure Coke/1000 cubic meters CO₂ / H₂ 1937-1940

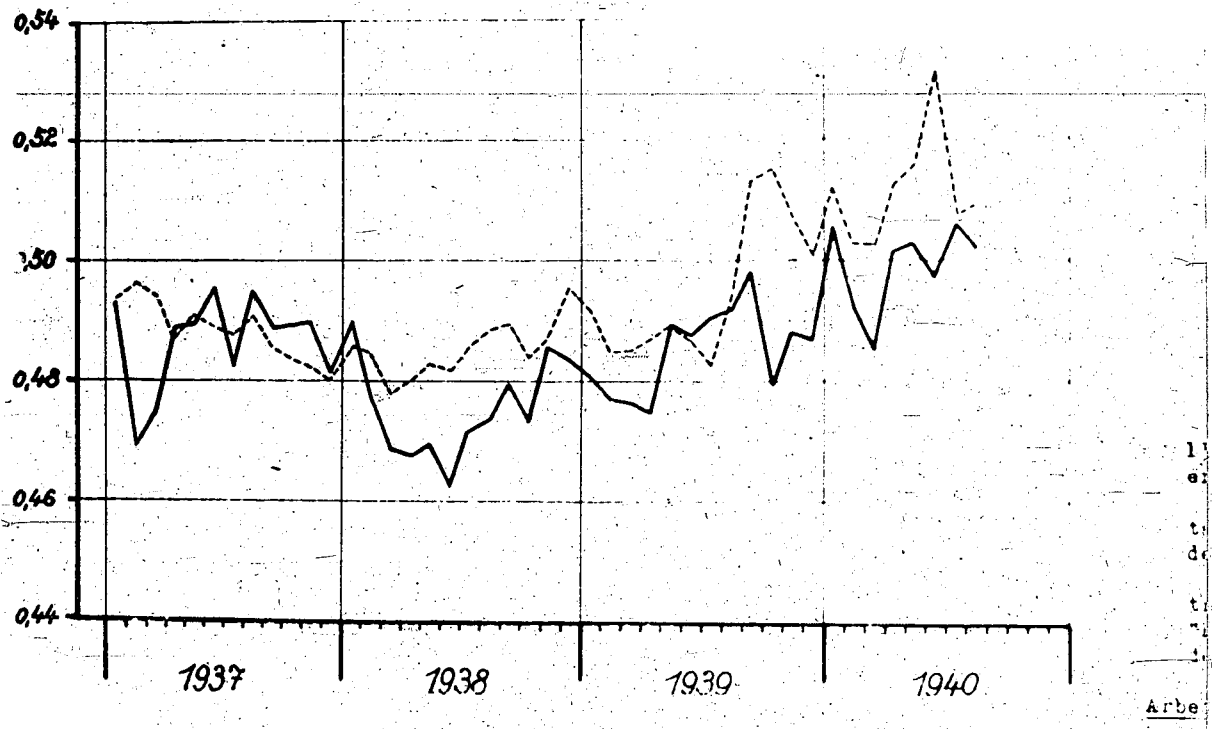
Merseburg

Oppau

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Reinkoks / 1000 m³ CO+H₂ 1937 - 1940.

— Mersaburg
- - - - - Oppau



Gas Works Me 1

Operation: Dr. Haller
Dr. Laudenklos

Repairs: DI Zeigner

Activities in 1940:

The automatization of the water gas plant was further carried out. Four generators with Pintsch-Brassert grate were put into operation anew. Of the total 8 Brassert generators present in Me 1, 6 are in operation, 2 in repair. The increase in performance, expected from enlarging the shaft diameter and going over from the 4-minute to the 3-minute gasification, was attained; the hourly production of a Brassert generator comes to about 7500 cubic meters per hour of CO and H₂ (carbon monoxide and hydrogen). The running time of the generators could likewise be further increased; thus there are in continuous operation:

Generator (Brassert roaster)	44-14 months
" " "	25-13 "
" " "	46-7 "
" " "	24-6 "

The cooling of the Brassert generators with Wofatit water was found to be advantageous; hourly about 70 cubic meters of Wofatit water are warmed from 60° to 90° and are passed on in the boiler houses.

Six generators were supplied with the new simplified gear support, one automatic gear of Pintsch was built into generator 42.

The building of the sludge bucket conveyor works was stopped due to the war (lack of fitters), new conveyor works were not built.

The specific consumption of coke in the year 1940 compared to the previous years rose further from 485 g. pure coke per cubic meter to 497 g. In part this is due to the rise in the cindering time (various types of coke), in part, however, the coke quality itself plays a role, as in Opau the specific consumption of coke rose in the same way (see graph).

(Page 6 contd.)

During the blackout period the burning of the blast gas was very disturbingly noticeable. By building in a filter plant for the sprinkler water and by increasing the bore of the sprinkler itself from 10 to 14 millimeters, the burning above the roof of the generators could be markedly retarded.

For the protection of the personnel and machines, protecting walls were erected in the machine shops of Me 1a, 135, 136, in the cellar of Me 1 and on various gear support huts.

The 16-hour alternating shift (1.4-18.10.40) with 1/2 hr. lunch period could only be carried out in the gas plant, because from the coal supplying plant for the time being 10 men helped out for 8 hours in the gas plant.

Beginning with 1940 Dr. Schmitz stepped out of the management of the coke gas plant in order to take over the development of the gas part of the project Upper Silesia Hydrogenation works "Eblechhammer." Dr. Schmitz was works director of gas plants Me 1 and Me 240 for 23 years.

Activities in 1941:

Continuation of the automatization.

Further experiments are anticipated for the production of Null(hydrogen) water gas out of hard granular fines, or salt hard granular fines and oxygen and steam in the Brassert generator.

C O N F I D E N T I A L

Page 7

Gas Works Me 240

Operation: Dr. Haller
Dr. Schroeter

Repairs: Diploma Engineer Ihlenburg
later Diploma Engineer Spichal

Activities in 1940:

Of the 10 Pintsch-rotating grate-generators at least 2-3 were running, often even 4 with wind and carbonic acid to power gas, which was needed as compressor power gas. The remaining produced Sti-water gas.

Of the 6 generators for tapping mostly 4 were in operation, two each for power gas and Null(hydrogen) water gas. The water jackets of the generators for tapping are strongly corroded by the returned cooling water of the gas works Me 240. Therefore conversion to cooling with Wofatit water is under way. This water is taken off at 40-50° and heated to 70-80° and sent to the steam plant. Two generators for tapping are already connected to Wofatit water.

The contact line for the electrical filling cranes of the generators for tapping were replaced by rubber cables, as the former corrode markedly and give rise to difficulties.

On the east side 2 of the 4 coolers of the water gas generators were connected to the power gas network. In this way the capacity of the plant for power gas was increased.

Experiments:

Experiments for production of power gas from Deubener hard granular fines with wind and carbonic acid were successfully carried out in the normal Pintsch- (rotating grate) generator as well as in the Brassert-generator. A power gas of about 1200 heat units with 4% hydrogen/1000 heat units was produced. While the normal Pintsch-rotating grate as well as the tower grate with side scrapers are not suitable for gasification of granular fines, as they do not sufficiently carry out the ashes, in the case of the Brassert grate the cinders are turned out in sufficient amount and well burned out. The Brassert generator used for these experiments has no cooling ring, but is instead walled in with Tertial stone.

C O N F I D E N T I A L

(Page 7 contd.)

In the Brassert-generator Null(hydrogen) water gas with 72% carbon monoxide and hydrogen was also produced (16-17% in oxygen-steam mixture) from Deubener hard granular fines with oxygen and steam.

An experiment was also carried out--with not altogether uniform results-- for the production of water gas from Deubener hard granular fines in alternating operation with wind blowing and gases with steam. This process, because of the great reacting capacity of the granular fines and consequent large fuel losses due to high carbon monoxide content of the blowing gases, does not come into consideration.

The gasification of Deubener hard granular fines in the normal coke generator with superheated (600-700°) steam with and without oxygen addition was tested to give a basis for the war project worked out for the end of 1939, "Gas production in Deficiency of Mineral Coal Coke." In this type of gasification the output of the Pintsch-generator amounted to 3000-3500 cubic meters of carbon monoxide and hydrogen per hour (mineral coal coke: 5000 cubic meters of carbon monoxide and hydrogen per hour). Grate and cleaning out equipment of the generator supplied with steam superheater were not yet satisfactory.

An experiment for producing hydrogen-poor, heating-powered power gas of 2000 heat units per cubic meter from coke by heat blowing with wind and gases with carbonic acid (instead of steam) is successfully carried out. The consumption of coke for the heating powered power gas 2000 heat units was only 0.620 kilograms per cubic meter of carbon monoxide and hydrogen against 0.640 kilograms per cubic meter of carbon monoxide and hydrogen with the usual coke power gas of 1000 heat units.

Activities in 1941:

Gasification experiments with salt granular fines, upon which is reported separately, are to be continued.

CONFIDENTIAL

(Page 7 contd.)

The Kandell system for waste water of the gas plant Me 240 must be improved and broadened.

With the automatization of the gear supports on the water gas generators a beginning is to be made.

CONFIDENTIAL

Drawing opposite p. 8

Preparation of Hydrogen-poor W1-power Gas

H₂-rich power gas
for manufacturing and heating gas

16% CO₂
11" H₂
25" CO
4.8" N₂

H₂-poor power gas
for compressors

14% CO₂
3" H₂
31" CO
52" N₂

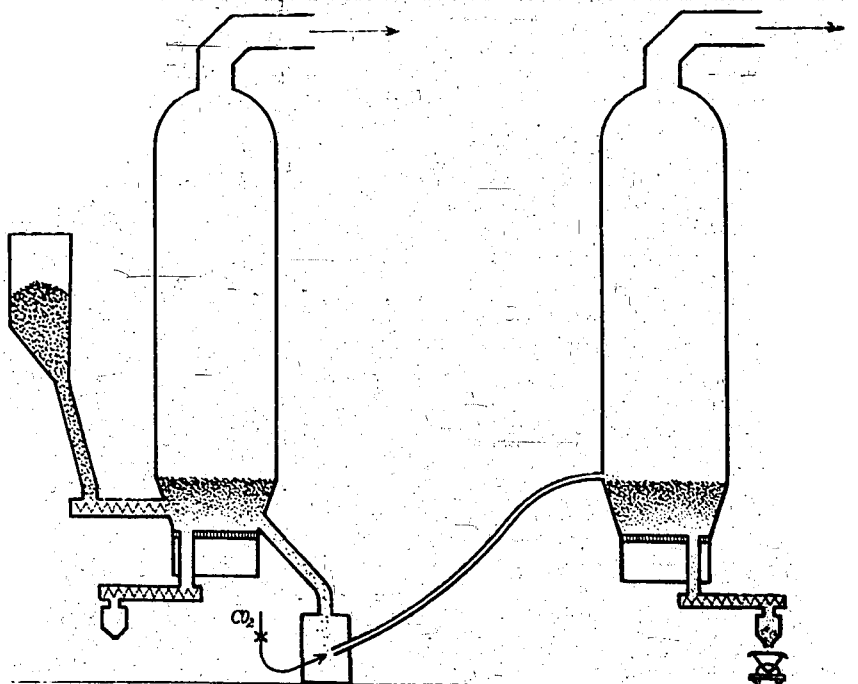
Herstellung von wasserstoffarmem Wi-Kraftgas.

*H₂-reiches Kraftgas
für Fabrikation und Heizgas*

16 % CO₂
11 " H₂
25 " CO
4,8 " N₂

*H₂-armes Kraftgas
für Kompressoren*

14 % CO₂
3 " H₂
31 " CO
52 " N₂



Winkler-Generators-Operation

Operation: Dr. Pattenhausen
Dr. Pastzold

Repairs:
January-April: Dr. Pfeiffer
May-December: Diploma Engineer Oehler

Activities in 1940:

The possibility of preparing hydrogen-poor power gas with the aid of refined granular fines in continuous operation was verified by a longer experiment.

After experiments to gasify salt coal with the aid of flux failed, a gasification experiment with Hermine coal without flux was undertaken, in which the generator temperature was kept below the sintering point of the Hermine ash. The experiment showed that in this way salt coal can be gasified in the Winkler generator, increased consumption figures and poor gas having to be taken into consideration.

In the middle of the year a modern superheater was built into waste-heat boiler 4. The manifold difficulties which the old superheater caused were thus eliminated.

For the prevention of clinker formation at the gas outlets in the generators operated with Deubener granular fines on generator 1 an arrangement was experimentally made to install on the generator ceiling a circulating gas to cut down the outlet temperature of the gas. The arrangement seems effective.

For supplementing of the inadequate reserves a new oxygen blower and a further disintegrator were erected.

The direct drawing off of the sludge water out of the receivers after the desulfurizing towers was advantageous, the conduit arrangement was thereby markedly eased. Therefore a sludge pump was also set up in receivers 3 and 5

~~CONFIDENTIAL~~

Page 8 contd.)

Activities in 1941:

Water gas preparation with the grateless generator 1.

Gasification experiments with the accumulating Winkler flying dust.

Combating clinker formation in the generator outlet.


Further improvement in water economy.


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Drawing opposite p.9

Total Sulfur Production since 1924

 Claus oven installation

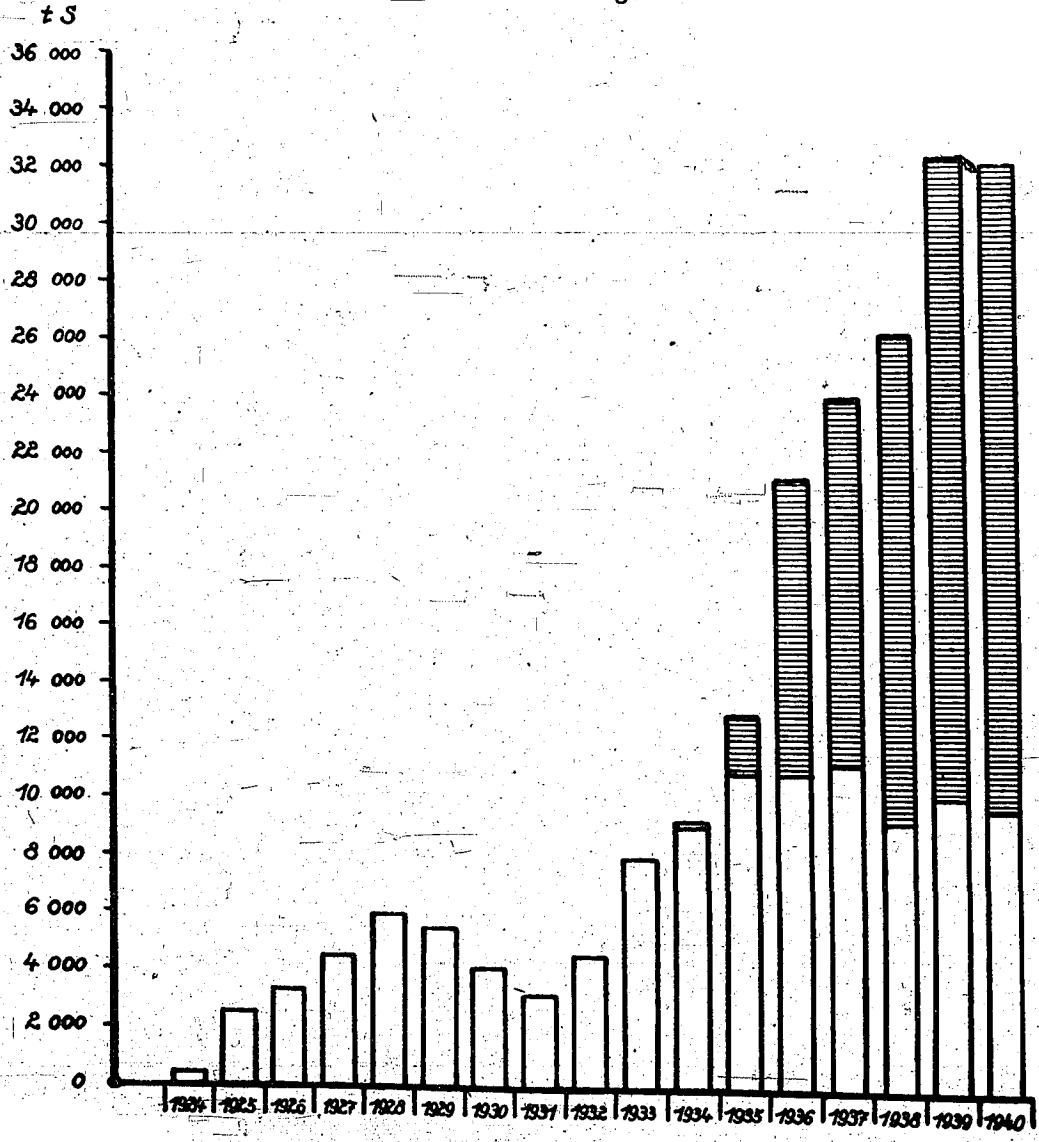
 F-coal installation

~~CONFIDENTIAL~~

früher 9

Gesamt-Schwefelproduktion seit 1924

▨ Clausofen-Anlage
□ F-Kohle-Anlage



F-Coal-Sulfur purification Me 284, 285 and 40 u Sulfur Bed

F-Coal-Sulfur purification.

Operation: Dr. Seeger
Dr. Keilig

Repairs: Diploma engineer Spichal

Operations in 1940:

The operation was heavily loaded in the previous years, at times up to 400,000 cubic meters per hour of gas. By cutting off the fine desulfurizing of the Winkler Null (hydrogen) gas by means of Alkacid the F-coal plant had to help out, which was possible in every case. Noticeable operation disturbances were not noticed.

Twice in the Sti-absorbers; marked resistance increases took place due to breaking in of dust, due to sudden high dust content of the Sti-water gas or of the Wi-power gas. The normal condition could only be reinstated by newly filling a number of absorbers. Attempts were made to avoid the objectionable breaking through of the dust through the dust boxes, in that the coke layers were subdivided by wooden compartments into individual cells and the individual layers were covered by gratings. The effectiveness of this precaution is not yet certain.

For protection from corrosion, experimentally a tower with a grate of Beton was set up and another tower was supplied in the lower part with a light walling in. Because of increase of the methanol production on June 1, the absorber was switched from Sti to methanol.

The insufficient gas pressure in front of the machines in Me 40 was increased by leading water gas to a line on the east side of Me 40, previously used for Sti-contact gas.

The Igelit walling in of the tubular coolers Me 285a was not found to be satisfactory and had to be removed.

(Page 9 contd.)

The removal of liquid ammonia from Me 12 was stopped and replaced by taking gaseous ammonia from Me 14 (April).

Experiments;

Experiments to eliminate the organic sulfur by means of granular fines were carried out on a technical scale. With Oberröbling semi-coke (coke breeze) it was possible in the turning out tower at about 100° to split the organic sulfur (200 milligrams sulfur per cubic meter of gas) to about 20 milligrams sulfur per cubic meter of gas into hydrogen sulfide.

The anticipated erection of the Wifo plant in Niedersachswerfen, the direction of which we were to take over, was the cause for carrying out laboratory and technical experiments on reduction of sulfur dioxide by means of granular fines and passing the gases over contact for production of lump sulfur. The conversion is almost complete with Oberröbling semi-coke and without particular difficulties. Production of the experimental plant was 10 t.p.m sulfur. Experiments are nearing completion.

Operations in 1941:

Continuation of experiments for fine purification of gases of dust on a technical scale. Technical experiments on utilization of iron oxide of Hy-coal for gas desulfurization.

Sulfur bed.

Operation: Dr. Seeger

Repairs: Diploma engineer Ihlenburg

Operations in 1940;

Due to difficulties due to cold in the beginning of the year and later because of importation of large amounts of foreign sulfur the exportation of lump sulfur of our manufacture fell off markedly for the time. Various necessary storage piles within the plant were a great hindrance and in April reached about 6000 tons of poured lump sulfur.

~~CONFIDENTIAL~~

(Page 9 contd.)

At the instigation and at the expense of Schwefel G.m.b.H. a stock pile was later erected west of the Reichsbahn (government highway), about 20,000 tons of lump sulfur and on October 1, 1940 was taken into operation. The sulfur was liquefied in the Claus oven and by means of a heated line taken to storage.

The Schwefel G.m.b.H. was particularly kept informed by testing and evaluation of sulfur samples.

~~CONFIDENTIAL~~

Page 10

Contact-Hydrogen-Plant

Operation: Dr. Baumann

Repairs; Diploma engineer Zeigner

Operations in 1940:

Sti-contact gas-production:

After the completion of the necessary lines the contact gas network was switched in on the larger gas container Me 524 (30,000 cubic meters), which up till that time was used for methanol gas. Since the beginning of the war as the large waterless plate gasometer (65,000 cubic meters) had to be shut off, the Sti-contact gas had to make use of gas containers which were much too small (10,000 cubic meters), first with Me 503 and then with Me 502. Me 503 underwent some big repairs and was then put into operation with methanol-contact gas. Me 502 was then put back in the gas plant.

The conversion of 10 systems, already due in the previous year, could not be completed. Only 5 systems could be converted.

One new evaporator with accessories was set up and switched in. The water lines of the cooler which was still to be built were delayed.

Three vaporizer pumps and 3 cooler pumps were newly arranged. The steam turbine on one gas blast was renewed.

The turned out finely granular contact was recommended for the hydrogenation for organic gas desulfurizing and was used there.

In the attack of the 28th to the 29th of August 1940 the bell jar and telescope of the gasometer 524 were broken in by 42 bomb splinters. The 3-day repair job made it necessary to feed in Sti-contact gas without buffering the gases before the gas compressors.

Methanol-contact gas-production:

In order to insure increased methanol production 2 pieces of apparatus were converted into double system, so that the number of methanol systems was increased to 18.

After the fire in the methanol plant on August 2, 1940 the systems were converted to Hy-contact gas. In this way the plant was kept up to temperature and in operation. On August 8, 1940 methanol gas was again fed.

Although the Null (hydrogen) water gas was well purified, noticeable attacks originating from hydrogen sulfide were observed in the oven. In one vaporizer after opening practically no objectionable bodies were any longer found. From the residue it was clear that the total content was used up by the sulfur corrosion (formation and decomposition of iron sulfide). Also the contact was mostly decomposed. More Bauxite was filled in to replenish the contact. The marked decomposition of the contact can probably be attributed to the feeding of the oven with Hy-contact gas (decomposition of iron sulfide with steam).

Hy-contact gas production:

As the capacity of the Me 241 structure is limited to 6,000 revolutions, in the case of higher requirement of gas for the hydrogenation, the 102 structure (nitrogen) must be shunted in. In that case for hydrogenation, the Winkler Null (hydrogen) gas which is desulfurized in the Alkacid purification, is fed. It is found that the resistance rises/rapidly in this very system operating with Hy. This may be attributable to the high dust and sulfur content of the Winkler gas.

On January 23, 1940 the lower part (cotton case) of a large slide valve in the contact gas line burst due to ice formation. A fire resulted which could only be put out by a partial shutoff.

Due to the increased addition of oxygen in the Null (hydrogen) water gas (for better hydrogen sulfide purification) marked corrosion occurred on the warm water lines and vaporizers. The water distributors on the vaporizers had to be renewed.

CONFIDENTIAL

(Page 10 contd.)

In the attack of the 28th to the 29th of August, 1940 the large Hy-contact gas line in IId Street was struck/smoothly by a bomb (explosion of the rails ^{through} underneath). The resulting fire was extinguished in a short time while the air attack was still going on.

Operations for 1941:

Work, due already for some years and having accumulated, is to be concluded. The following still remain to be completed: Two coolers, two vaporizers, the conversion of five systems, one blast apparatus and a third feed for the Null (hydrogen) water gas to Me 102.

CONFIDENTIAL

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Page 11

Methanol Contact Gas Purification

Operation: Dr. Baumann
Dr. Haak, from January 9 to
March 7, 1940

Repairs: Diploma engineer Zeigner

Operations in 1940:

In Me 66 for 1940 particularly a saving of sulfuric acid was visualized. The consumption dropped from 1506 tons in 1939 to 450 tons of sulfur trioxide in 1940. The success (saving) was primarily achieved by improved cooling of the filtrate and secondarily by working with carbon dioxide-acid filtrate (bicarbonate-containing). From December 5, 1939 to July 23, 1940 acid filtrate was fed. Unfortunately, however, in a short-time corrosion was found on the new tubular lines of Al coolers. Consequently less acid filtrate was again used.

In one cooler corrosion due to outside factors (flying ash) occurred on the flanges. Self cast prewelded flanges were built in.

For methanol now only one type of gas is fed. Consequently the old methanol coolers came into reserve.

When hydrogenation was dropped out (air danger), Hy-raw carbon monoxide gas mixed with Null (hydrogen) contact gas was replaced for example by Null (hydrogen) contact gas. In that case, however, the carbon monoxide content of the total gas had to be increased.

Particularly recently in gas compressor plant Me 104 corrosion was noted in the pure gas machines. The cause could not be altogether discovered. In order to eliminate the possible influence of traces of oxygen, which might be dangerous in the presence of even the smallest traces of hydrogen sulfide, the gas cooling was changed over to Pelton water. As a further possible cause for the observed corrosion, the admixture of pure gas Me 66 with Null (hydrogen) contact gas containing appreciable amounts of carbon dioxide may be mentioned.

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~~CONFIDENTIAL~~

(Page 11 contd.)

This disturbance can also easily occur during a shutoff, because no gasometer is on hand for the pressureless purified methanol gas Me 66 and therefore constantly a part of this gas must flow over into the methanol gas 2, which goes through the pressure water washer.

Operations for 1941:

The setting up of a gas container for pure gas Me 66 is becoming pressing.

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Contact gas Purification

Operation: Dr. Scheidemandel

Repairs: Diploma engineer Ihlenburg
(to June 30)
Diploma engineer Spichal
(from July 1)

Operations in 1940:

The loading of the contact gas purification amounts on an average to/1000 t.p.d. about of sulfate and thus comes to only 80% of last year's production. It has been planned after the war to convert the plant from Sti-contact gas, to methonal contact gas. In that way from the point of view of the gas Me 386 is completely utilized, the pressure water purification markedly unloaded and from the point of view of operation an appreciable saving of energy could be attained.

The specific consumption of sulphuric acid was lowered to about 40% of the former consumption by conversion of the final gas wash to filtrate acidified with carbon dioxide (bicarbonate).

The carbon for the residual desulfurization of pure gas, having become unusable because of phenol and light oil, was regenerated. At the same time by building in a calorifer (heat carrier) we have a possibility of heating the gas above the dew point. In that way the residual desulfurization was improved from 0.05-0.09 milligrams per cubic meter to 0.00-0.01 milligrams per cubic meter hydrogen sulfide-sulfur.

The overhauling of the A tower series was concluded.

The filtration of filtrate II with wood carbon in place of gravel was found advantageous. The consumption of energy when less gas was passed through, 20,000 cubic meters of gas per hour, was decreased by about 25% by conversion of the gas blast (simpler slider).

Experiments:

The circulating experiments, which, due to lack of manpower, could only be carried out to a limited extent, showed a probable steam consumption of 4 kg./cubic meter of washed out carbonic acid.

~~CONFIDENTIAL~~

(Page 12 contd.)

The plant director participated in a trip of a commission for supervisory testing of the nitrogen industry of northern France and Belgium.

Operations for 1941:

Circulation experiments with precipitation and regeneration of ammonium bicarbonate sludges and of solid salt partially for use of the process in the plant.

Study of the absorption processes in the washers with the object of freeing the towers for the circulation processes by changing the gas feed.

The possibility of widening the sulfur basis of the plant by obtaining higher hydrogen sulfide-containing gas water from the salt lattice, is to be further worked out by taking in the hydrogen sulfide plants on a broader basis.

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(Drawing opposite page 13)

Alkacid Regenerating Column
(Alkacid)

Old construction

New construction

Built in portion: metal

Built in portion: stone

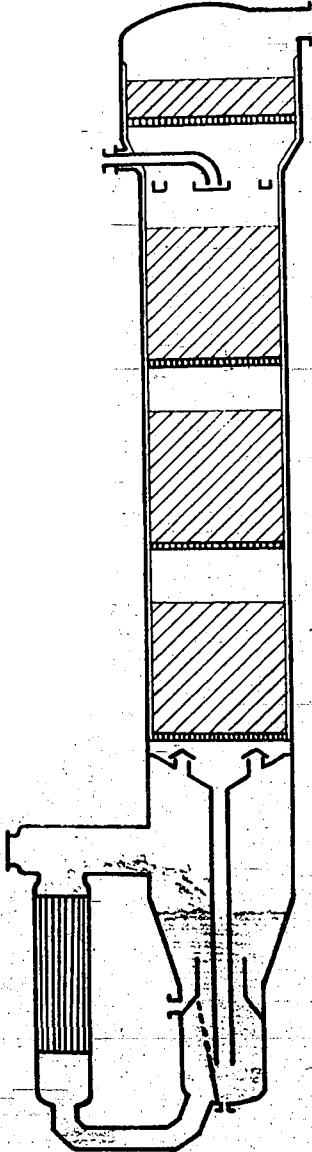
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Alkacid Regenerierkolonne.

Fig. 13

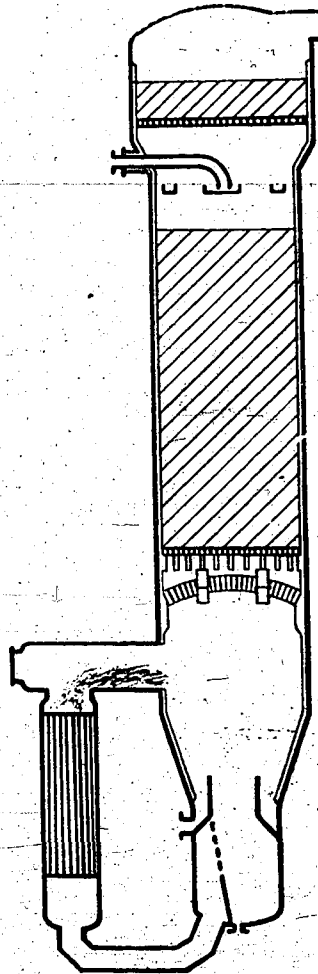
Alte Bauart

Einbauten: Metalle



Neue Bauart

Einbauten: Steine



Alkacid Desulfurization

Operation: Dr. Jeltsch
Dr. Menschick
Dr. Eyber (from May 1, 1940)

Repairs: Diploma engineer Sommer

Operations in 1940:

Hy-poor gas-desulfurization:

The desulfurization of the poor gas was now only carried out in bell jar washers. The significant superiority of bell jar washers over wash towers was verified. From May on the new Dik liquor A (di-ethyl-) was used, this apparently being superior to the old liquor (dimethyl-) in selectivity. The better selectivity was also observed during the concentration of the dilute gas.

Hy-rich gas-desulfurization:

A new simplified regenerating column was found efficient. The new column has a masonry lined dome with masonry lined grate and no built-in portions of metal. It is filled throughout with Raschig rings and is only six meters high (previous filling nine meters in three charges for each three meters).

The plant liquor was further damaged by hydrocyanic acid (from the rich gas) and by oxygen (absorption from air due to faulty liquor pumps). The plant liquor was therefore taken out of the plant in the middle of September for regeneration and in its place Dik liquor A was filled in.

For the same reason after about two years operation the M liquor of the second washing stage was also markedly contaminated.

Wi-O-water gas-desulfurization:

The rough purification was in operation on the average with 70,000 cubic meters per hour of gas, occasionally 80,000 cubic meters per hour of gas. The hydrogen sulfide content of the Winkler-O-gas amounted on an average to about 17 grams; at times it rose to 25 mg. of sulfur per cubic meter. The superiority of the Dik-liquor A in the rough stage was shown by the fact

(Page 13 contd.)

that an outlet gas was obtained with about 60% hydrogen sulfide (old Dik liquor: about 50% hydrogen sulfide).

Because of some corrosion difficulties, however, primarily because of foaming difficulties in the rough purification, the fine desulfurization had to be interrupted a number of times in order to insert the apparatus for the rough purification.

The apparatus at first on hand for half of the gas for fine purification gave 40,000 cubic meters of gas per hour and yielded a pure gas with an average of 100 mg. of sulfur per cubic meter.

The poly-sulfide preliminary wash was not in a position to wash out the main portion of hydrocyanic acid. The high carbon dioxide content of the gas disturbed the hydrocyanic washing sensitively.

The new blower apparatus for 80,000 cubic meters of gas per hour was put into operation in the middle of April, but after two weeks it had to be fitted out with a new slider (construction mistake).

Extensive precautions were taken for the timely shutting off of the hydrogen sulfide operation and the hydrogen sulfide net was installed in case of air attack. In order to be able to supply a water-free hydrogen sulfide gas in time of frost, the hydrogen sulfide collecting line was supplied with a final cooler.

Experiments:

Intensifying the poly-sulfide preliminary wash. Washing efficiency of Alkacid liquors diluted with potash. Working up Koppers potash experiments. Comparative measurements on steam consumption in the simplified and the earlier regenerating column. As in previous years projects of various plants were under consultation, particularly Wintershall-Lutzkendorf, where the plant operation of the Alkacid plant was completely in our hands from May to November, 1940.

C O N F I D E N T I A L

(Page 13 contd.)

Operations for 1941-

Completion of poor-gas- and rich-gas-plant to insure ethane production.

Setting up of two bell jar washers for W1-0-gas fine purification.

Setting up pumps for utilization of waste water from the liquor cooler as fresh water for the condensers of the outlet gas.

Preliminary degasification of Hy-gas liquors from hydrocarbons to get a pure hydrogen sulfide gas.

Utilization of hydrogen sulfide content of phenol-poor gas by concentration with Alkacid.

C O N F I D E N T I A L

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Drawing opposite page 14

Sulfur Line

Old construction:

Hot steam

Liquid sulfur

New construction:

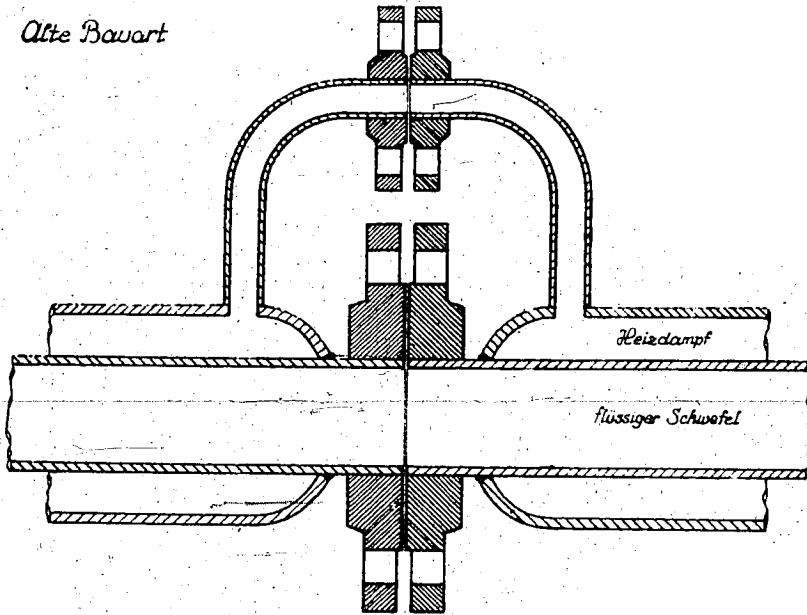
Hot steam

Liquid sulfur

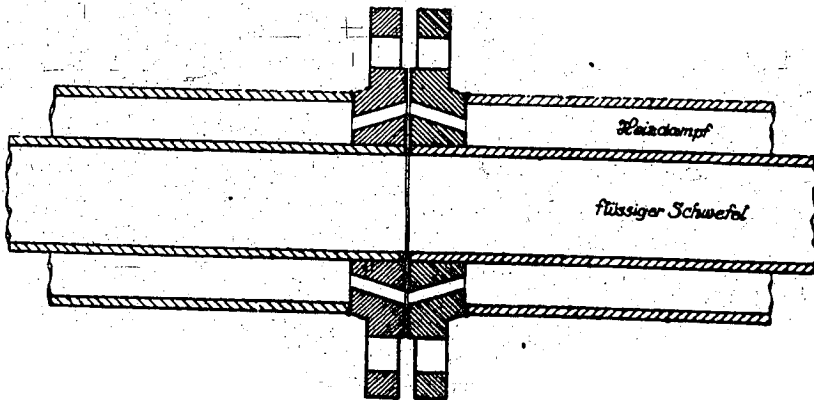
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Schwefelleitung.

Alte Bauart



Neue Bauart



Page 14

Claus Oven Operation and Alkacid Liquor Purification

Operation: Dr. Jeltsch
Dr. Grimm

Repairs: Diploma engineer Ihlenburg

Operations in 1940:

The Claus ovens were permanently overloaded. For this reason and because of impurities (contact poison) in the hydrogen sulfide gas, the yields were poor. The overloading was caused by the fact that because of low sulfuric acid production an unusually large amount of hydrogen sulfide was led to the Claus ovens. Also the condition of the kettles and ovens, because of the frequent shutoffs due to air raid alarms, was not good.

Because of the dropping out of a Claus oven vessel more hydrogen sulfide gas had to be temporarily led to the sulfuric acid plant and for this time the working up of phenol-hydrogen sulfide had to be stopped--likewise because of lack of reserve in the Claus oven operation. The setting up of the necessary reserve system, which has long been pressing, now seems to be possible, after the iron allotment for this has been made.

At the end of the year the water injection in the Claus oven was changed over from condensate to hydrogen sulfide-condensate.

At the request of Schwefel G. m. b. H. a block sulfur pile was started west of the Reichsbahn. The sulfur pumped to this pile was experimentally estimated with an oval wheel counter. The wooden roof line for the Claus oven waste gases burned down for the third time and is now being replaced by an iron line protected with heat resistant lacquer and Asplit.

Among the smaller improvements the following are noteworthy: Intensified insulators in the electro-filter 1, outwardly heated sulfur receivers, enlarged heated withdrawal cocks for liquid sulfur, heated flanges for sulfur-and gas lines, heated piston rods of the sulfur pumps (omission of the extensive new packing of the pumps).

C O N F I D E N T I A L

(Page 14 contd)

Waste gas washer:

The waste gas washer for half of the Claus oven waste gases, in the process of being built, could not be completed, and is being held back until later. The experimental washer was at the time in operation and thereby the superiority of the standing column over the packed tower was shown.

Procelein and glass were found to be the only suitable material for pump lines, slide valve, and tower casing.

Experiments, to get rid of objectionable hydrocarbons, which are impurities in the hydrogen sulfide gas, with the aid of a cracking chamber inserted before the kettle, were not uniform in their effectiveness. Also the combustion of hydrogen sulfide with oxygen or oxygen addition, did not give any better yield. Investigations on the types of Bauxite suitable as contact. Investigations on the factors which determine the yield in the Claus process. Development of analytical methods for iodine exchange.

Alkacid liquor purification:

The plant operated satisfactorily. Besides liquors from Leuna, Dik-liquors from Scholven, Gelsenberg-Nordstern were regenerated. Because of increased delivery of liquors the operation had to be expanded. Until a roof is erected and a partial wooden casing, it is possible to work only in the daytime with decreased efficiency, for reasons of air protection and partially because of the influence of weather. The regeneration requires considerable analytical control because of the variety of liquor contaminations.

Operations for 1941:

Setting up a new Claus oven system.

Gas cooling in the oven instead of water injection, which results easily in freezing, with liquefied sulfur, circulating and cooled.

Plant experiments, to improve the yield by purification of hydrogen sulfide gases.

Addition of a rotating filter to increase the capacity of the Alkacid liquor purification.

C O N F I D E N T I A L

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Drawing opposite page 15.

After-burning Furnace

M.1:100

Old setup

New setup

Decreasing corrosion of casing

Decreasing dead space

Simplification of internal structure

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Fein 15

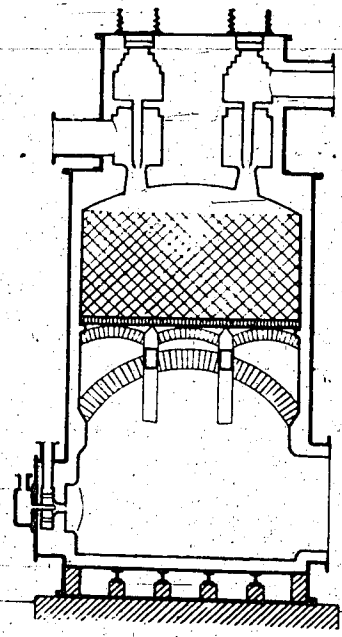
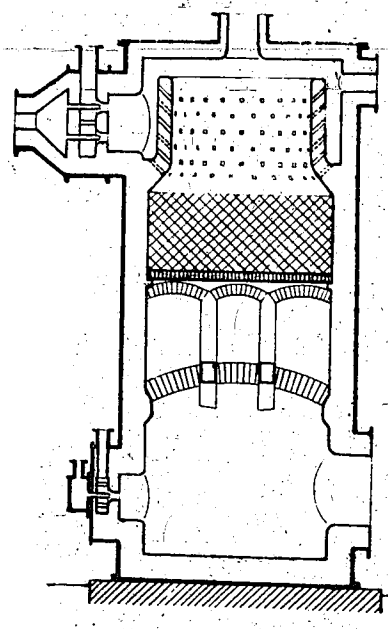
Nachverbrennungsöfen

M. 1:100

Alte Bauart

Neue Bauart

Verringerung der Mantelkorrosion
Verringerung des toten Raumes
Vereinfachung der Einbauten



Sulfuric Acid Plant

Operation: Dr. Jeltsch
Dr. Grimm

Repairs: Dr. Ihlenburg

Operations in 1940:

The running time of an after-burning kettle amounted for the first time to 1-1/2 years (formerly 4 mos.). The super tubes (see sketch in the short yearly report 1939) were therefore advantageous. Both after-burning ovens built in 1934 had to be thoroughly renewed in the course of the year. In that way considerable construction improvements were applied, these giving a longer life period of the ovens. During repair there was an opportunity to impregnate the chamotte layer which lies over the contact, whereby a conversion improvement of the after-burning is attained.

In February 1940 only Claus oven waste gases without addition of Alkacid hydrogen sulfide were worked up; the production amounted to 700 t.p.m. of sulfur trioxide (the lowest production so far, normal production: 1500 t.p.m. of sulfur trioxide).

Due to frequent shutoff during air raid alarm the cooling tower leaked and it had to be put back into condition in a five-day shutoff of the plant. The packed cooler was fitted out with satisfactory water distributors and also with Igelit nets, stretched over. In this way an appreciable saving of cooling water was attained. In the period of decided frost the Termisilid cooler was seriously damaged. For times of serious cold a circulating arrangement of the cooling water was installed.

For various parts of the plant, where the previous construction material, lead, was seriously attacked, good results were obtained by replacing with synthetic materials (Igelit, Oppanol) used to protect the iron. Igelit nozzles were, also satisfactory for the towers; porcelain nozzles were not conclusive. Igelit acid protected coatings were tried out and were satisfactory with regard

~~CONFIDENTIAL~~

(Page 15 contd.)

to the workmen. The acid pressure line to the stockpile Me 159 had a number of oscillation breaks and some damage by bomb splinters. In order to eliminate quickly difficulties due to shutoff of the plant in air attacks, two emergency lines were laid and a central switch arrangement for the pump motor was installed. The control apparatus installed in 1939 was satisfactory.

The concentration plant which had been planned, could not be started because of shortage of fitters. The necessary Pauling kettles had been delivered some time ago.

Experiments:

In laboratory experiments the denitrogenization of thick acid was investigated; on the basis of results a plant test is to be made, which is to show the possibility of plant simplification and further saving of cooling water. In the laboratory afterburning contacts were tested. The technical experiments ^{which} were done on direct concentration of acid with combustion gases, produced by hydrogen sulfide combustion, had to be stopped due to lack of fitters. Technical experiments on the continuous concentration of sulfur dioxide according to the sulfite-bisulfite process, were begun.

Operations for 1941:

Changing the large (1400 ϕ) gas canals from lead to iron lines encased with Oppanol. Setting the sprinkler lower in tower 6 in order to avoid corrosion of the ceiling.

Arranging a sulfur dioxide production plant.

Arranging a plant for obtaining ammonium disulfite liquors for Luran

~~CONFIDENTIAL~~

~~CONFIDENTIAL~~

The Lignite Drying Plant

Operations: Diploma engineer Binneweis
Dr. Wenzel II to June 30, 1940

Repairs: Dr. Pfeiffer

Operations in 1940:

Paste forming plant Me 390:

Two conveyors for delivering the dry contact dust-free were built in and resulted in a considerable decrease in dust loading. The removal bands for the wet contact were altered due to the experience of last winter. The delivery belts for transporting away the red earth contact to storage were made more efficient by rebuilding. The red earth contact delivery, which in the past was served by the coal conveyor plant, at the beginning of the year was taken over by the lignite drying plant. As the space for the two red earth contact stockpiles west of Me 108 was required otherwise, a new stockpile was arranged at the south near Me 67 and partially covered. In order to avoid the heavy loading of the hoppers containing dry contact with dust, a dustproof hopper house is being used.

Combustion gas drying plant Me 394:

The rebuilding of the chimney was carried out to the point of exchanging the Zeta tube. The exchanging of the Zeta tube cannot be carried out for the present because of operating conditions. At the request of the plant administration a new vapor chimney was planned and it is ready for building. Carrying this out was postponed to the end of the war. Complete saving of fresh water was achieved by using vapor dust removal in connection with cooling water in contact hydrogen plant. The further building of Hartmann cyclones was abandoned for reasons of safety. In the yearly report several times a daily production rate corresponding of the plant to 3000 t.p.y. of dry lignite was reached and surpassed. Because of the air situation it was necessary to shut off combustion gas drying frequently and quickly. Shutting off of the whole plant was finally accomplished in about a quarter of an hour.

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(Page 16 contd.)

Screening and Grinding Plant Me 282c:

Dust removal of the screening- and bunker-deaeration in a wash tower with sprayed oil was not satisfactory. Instead of this a suction blast was set up and all vapor removed by suction through a collecting conduit which is attached to the vapor line Me 394, going on to wet dust removal and the chimney of the combustion gas drying plant. In order to increase the coal hydrogenation the plant was further enlarged by means of a filter and crushing mill aggregate. The special freight cars necessary for dust-free transportation required development of the loading bunker, rebuilding of the freight car filling arrangements and setting up of a freight car switching machine. This work is planned and submitted for estimates.

Steam drying plant Me 279:

Working up the crude lignite coal has reached a new stage by building in two rotating screening grates. In order to increase coal hydrogenation, three drums for hydrogenation had to be available. The dry lignite for gasification was replaced by granular fines of foreign source. Dry contact delivery and bunkering as well as transport of the dry lignite after screening and grinding have been submitted for contract. Fire protection for the dry lignite bunker was extended by using an Erkalen high container with dropping lines and hose connections.

The Viag pressure water experiments were concluded (see the report of Dr. Wirth of June 4, 1940).

Operations for 1941:

Possible construction of two vapor chimneys.

Planning of wet electro-filters for waste gas vapors.

Completion of the expansion of the screening and grinding plant and enlarging the loading bunker with auxiliary equipment.

Conversion of three drums Me 279 for hydrogenation.

~~CONFIDENTIAL~~

~~CONFIDENTIAL~~

(Drawing opposite Page 17)

Power Gas Distribution

Coke consumption for power gas

T.P.M.

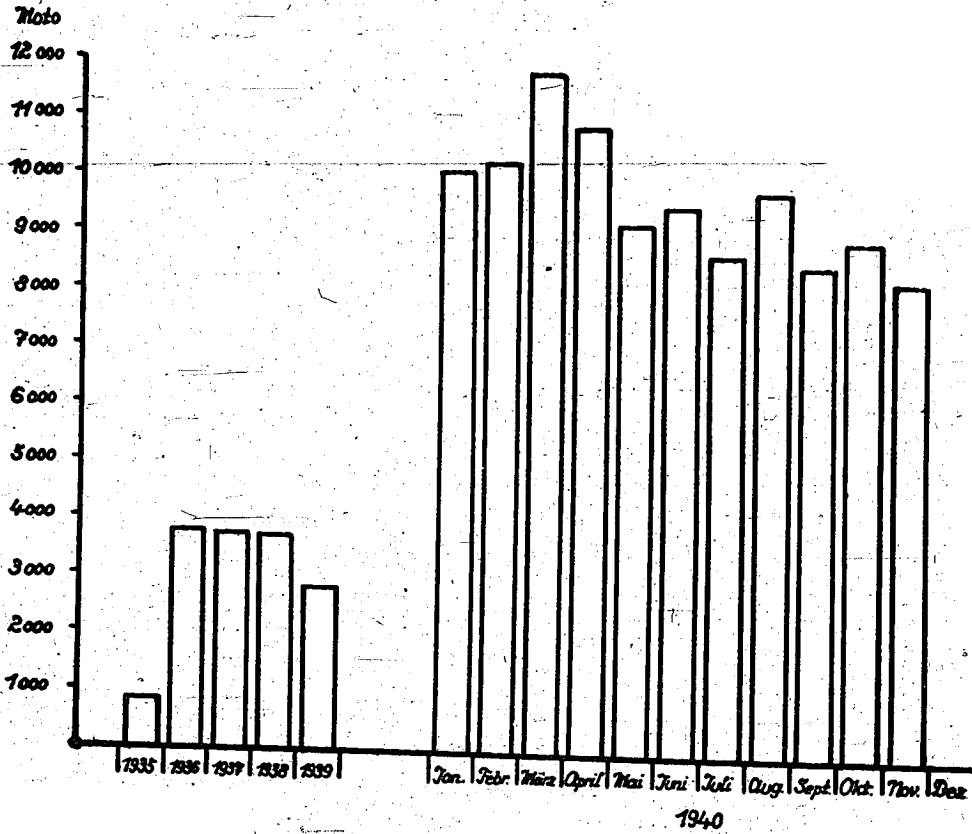
Jan. Feb. Mar. Apr. May Je. Jy. Aug. Sept. Oct. Nov. De.
1940

~~CONFIDENTIAL~~

Feig 17

Kraftgasverteilung

Koksverbrauch für Kraftgas.



Power Gas Distribution

Operation: Dr. Haller
Dr. Schroeter

Repairs: Diploma engineer Ihlenburg
(to June 30)
Diploma engineer Spichal
(from July 1)

Operations in 1940:

Because of further decrease in delivery of Hy-recirculating gases and because of the further deterioration in quality of the Hy-recirculating gases (due to increase in hydrogen content) it was very difficult to supply corresponding power gas of proper specification particularly for the driving machines of the compressors, only small amounts of hydrogen being admitted.

Supply of recirculation decreased per hour on the average from 130-140,000 cubic meters 1000 heat units in 1939 to 110-120,000 cubic meters 1000 heat units in 1940. The hydrogen content of recirculating gases for the compressors increased per cubic meter 1000 heat units from 6-7% hydrogen in 1939 to 7-8% hydrogen in 1940. Therefore larger amounts of power gas from coke had to be produced as hydrogen poor (2%) power gas. 9,000-10,000 tons per month of coke (as compared to 3000 tons per month in 1939) were consumed for this.

In a longer experiment on operating scale a hydrogen-poor Wi-power gas with 3-4% hydrogen instead of the normal 7% hydrogen per cubic meter 1000 heat units was produced and added to the compressor-power gas. The setting up of a second power gas-Winkler for the production of hydrogen-poor gas was contemplated. This measure, however, can only contribute in a modest way to alleviating the power gas situation, because the portion of hydrogen, which reaches the compressor-power gas by means of the normal Winkler-power gas, amounts to only 13% of the hydrogen content of the compressor-power gas. On the other hand about 80% of the total hydrogen from Hy-recirculating gas went into the compressor gas.

(Page 17 contd.)

In order to avoid the detonation, which has become more prevalent and endangers the machines, and to maintain a safer gap away from the dangerous hydrogen limit, there was finally nothing more that could be done except to mix larger amounts of power gas (butane) than previously into the compressor gas. Because apart from the fact that with larger consumption of coking power gas corresponding amounts of hydrogenation recirculating gas would become superfluous, the capacity of the gas plant before further expansion, that is, conversion of the Brassert-generators, is insufficient to supply larger amounts of coking power gas than previously (about 60,000 cubic meters 1000 heat units per hour). The addition of butane to the Hy-recirculating gas (compressor power gas) for the improvement of the hydrogen content amounted to 460 tons per month on the average inclusive of October.

On the whole for the same reason it was not possible to carry out the measures planned to decrease the sulfur content by taking out the Wi-power gas from the heating gas and to replace it by the Hy-recirculating gas.

A way out of these difficulties would permit new production of heating power gas (higher heat content) out of the coke with 2000 heat units per cubic meter (compare gas plant Me 240). As the gas machines permit more hydrogen, the higher the heat content of the power gas (for example, 4% hydrogen per cubic meter 1000 heat units at a heating value of 1000 heat units but 7% hydrogen per cubic meter 1000 heat units at a heating value of 2000 heat units), it will be possible to make the conversion of the total coking-power gas production to the new 2000 heat unit-power gas, more hydrogen being usable. Thus the butane addition is not only decreased (saving) but also at the expense of the coking-power gas, the component of Winkler-power gas for power gas purposes is markedly increased.

Operations for 1941:

Building of the compressor power gas with the aid of high heating power coking-power gas.

Plant Waste Water Clarifying Equipment

Operation: Dr. Haller
Dr. Schroeter

Repairs: Diploma engineer Ihlenburg
Diploma engineer Spichal

Operations in 1940:

Of the Winkler-recirculating cooling water only a portion (about two-thirds of the total amount) was desulfurized for treatment with carbonic acid and oxygen. The further development of water desulfurization, anticipated for 1940, particularly the setting up of a new oxygen tower, did not take place, partly because of the shortage of manual workers and partly because the problem of material has not been sufficiently cleared up. The tower, first operated with oxygen and which was strongly corroded even at the very beginning, was lined with Igelit and was again put into operation in November. The oxygen gasifying tower, made of wood, was found to be satisfactory in a one year operation but showed some operating disadvantages.

The sludge removal of the preliminary clarification basin for the Neustadt basin, which was tied up previously with major difficulties, could be markedly improved. Now the sludge is sucked off at the deepest sections of the individual pools by means of sludge pumps. In that way at times one of the two lines of the preliminary clarification basins is taken out of operation.

In the air attack on the night of August 16-17, four clarification basins were destroyed by bomb hits. Operation disturbances and damage by the water flowing out did not occur.

Operations for 1941:

Desludging the Neustadt basin is again to be carried out as formerly with the desludging conveyors on hand. This was stopped in 1937 because of the insufficient effectiveness of the clarifying basin, coarser particles getting into the Neustadt basin, the conveyors not being efficient enough to get these out.

(Page 19)

Lignite gasification Laboratory Me 420

Operations: Dr. Kah

Repairs: Dr. Pfeiffer

Operations in 1940:

Although the current operation analyses were shortened to a minimum, the laboratory was burdened very much by having to carry out unavoidable experiments and to meet miscellaneous operating requirements (clarification of irregularities, miscellaneous analyses, etc.).

Accordingly the following experimental work was carried out:

The suitability of combustion gas drying for the preparation of iron oxide masses, prepared with Hy-carbon, for the preliminary desulfurization of gases was investigated. The purpose was to attain a high gas throughput by abandoning high sulfur loading, the desulfurizing apparatus being kept as small as possible. The experiments gave the interesting result that by keeping the moisture content of the mass between 10 and 30% water, the gas rate may be chosen to be from ten to twenty times larger than is usual. Then with unobjectionable purification of gases a loading of the iron oxide masses of about 20% sulfur (as compared to 40-50% sulfur usually) was attained. It was shown that the burning off of the sulfurized masses is ^{definitely} hindered by dampening with water or with oil, rubbed on.

With the object of improving the dust removal of ~~Winkler~~ gases, which is still unsatisfactory, extensive experiments were undertaken to carry out washing out of gas dust by means of various washing liquids. It was found that for our dusts water is still the best washing agent, also for removal of final dust particles. Besides investigations were begun on the suitability of ceramic filters for this purpose. Extraordinary purity of the gases treated was attained. Experiments are still in progress on the loading capacity and regeneration of used filters.

~~CONFIDENTIAL~~

(Page 19 contd.)

In order to obtain the previously unknown heat transfer figures for liquid sulfur, which are necessary as a basis for planning the waste heat boiler of the plunger burning process, measurements in a technical cooling aggregate were carried out. The figures found were surprisingly low. The waste heat boiler for the plunger burning process for obtaining pure sulfur dioxide must be made much larger than was formerly assumed.

Operations for 1941.

Experiments for dust removal from gases with ceramic filters.

Continuation of experiments for obtaining sulfur from anhydrite (natural calcium sulfate).

~~CONFIDENTIAL~~

Low Pressure Experimental Laboratory Me 431

(Managing personnel listed)

Operations in 1940:

Experiments for removing alkali from salt coal by means of distillation and leaching of the fines with water were concluded. The experiments showed that only such fines in earthy form, for example, in the rotating drum out of Hermine Henriette II coal, could be desalted. The ash melting point curves in this case did not show any preliminary softening. Unfavorable results were obtained from fines distilled from briquets and by washing gas distillation.

Further experiments for removal of alkali from crude lignite produced two principal methods: treatment with acid and ion exchange. Extensive experiments are progressing on this and are not yet concluded.

Obtaining concentrated sulfur dioxide from gases containing sulfur dioxide by means of ammonium sulfite-bisulfite liquor in circulating process was worked up into the technical stage. In comparison with the sulfidin process less charging is obtained but the rate of absorption is considerably larger, the undesirable operation with two non-miscible components is avoided and the recovery with a bell jar washer, plant for the xylidine carried over is saved. The rapid operation/made possible by quick absorption rate, also cuts down the sulfurization rate considerably. The liquor, taken out, can easily be worked up by pressure distillation into sulfur and ammonium sulfate.

On a broad basis experiments were taken up to improve the yield in the Claus oven process. The experiments deal with clarification of the reaction mechanism, determining the equilibrium relationship in the interesting range of temperature and preparation of efficient contacts.

Also in this year the alkali acid licenses, which became very numerous, had to be aided by many investigations. In general the liquors operated

(Page 20 contd.)

satisfactorily; most objections could be traced back to unscientific handling of the plant. Only the preliminary washers set up in the water gas plants did not hold back sufficiently the hydrocyanic acid. Extensive experiments showed that it was necessary to remain with the principle of polysulfide washing. Thiosulfate, polythionate, colloidal sulfur have no advantages. Insufficient operation of the poly-liquor depends upon a subsequent reformation, too slowly, of the polysulfide used up. The experiments showed that the formation rate of this may be increased by using sulfur, as finely divided as possible, preferably colloidal. In the same way additions of solubility improvers for sulfur are effective. With the aid of this measure the washing out could be increased from previously 40-50% to 70-80%. Pure water is also suitable to wash out hydrocyanic acid, however, in the case of the circulatory process extensive gasifying is necessary. A large number of experiments on the problem of preliminary washing are still in progress.

The possibility, on principle, for obtaining aluminum oxide from the sludge of the generator for tapping by means of sintering with lime and soda and subsequently washing this out, was experimentally worked out.

It was shown that the Winkler dust which occurred in the gasification of lignite could be converted by treatment with Mepasin waste hydrochloric acid into a medium type of active carbon. The conditions were determined under which the best possible activity was obtained and the basis for a calculation was determined.

Further experiments, still in progress, deal with improvement of pressure-carbonic acid washing, water being replaced by other liquids operating on the physical principle, utilization of gypsum, removal of hydrogen from Pelton carbonic acid and gasification of coal with liquid water under high pressure.

CONFIDENTIAL

TRANSLATION

AMMONIA WORKS MERSEBURG

ANNUAL REPORT

for

1940

(Page 21, paragraphs 1-8)

Experiments re K.W.Sy. (synthesis of hydrocarbons)
Messrs. O. I. Sabel, Dr. Wenzel, Dr. Wintzer
temporarily: Dr. Rössinger, Dr. Wirth, D. I. Spichal

Further experiments have shown that a very hard paraffin can be obtained with a high yield (70%) by means of our special paraffin catalyst from CO / H_2 . Thus, the control of the synthesis in the direction of a special product which is not an energy-providing substance, has been successful.

The synthesis of olefins from water gas and with the aid of iron catalysts was further developed.

Progress was made by lowering the reaction temperature, by a more adequate design of the tubular furnaces (20 mm. ϕ pipes instead of 15 mm.)

Important information was gained on the subject of the formation of oxygenated compounds, particularly alcohols, during the synthesis. On a fused-iron catalyst up to 30% alcohols could be obtained, on a precipitated-iron catalyst 40-50%.

It was found to be important to remove all the acid compounds and the alcohols prior to the distillations, as these give undesirable side reactions.

It is planned to continue the experiments for the production of starting materials for the "Oxo" synthesis and "Mepasin," particularly however for the production of long-chain alcohols.

CONFIDENTIAL

C O N F I D E N T I A L

One project is being studied relating to the construction on Ley Square of an experimental plant capable of converting a total of 1000 cu.m./h. in several furnaces.

Since August 1940 the research group has been attached to the experimental laboratory after it had temporarily formed part of the Organic Division from June to the middle of August.

C O N F I D E N T I A L

(Page 21 contd.)

Gasification of Salt Coal

(Personnel listed)

Experiments, to obtain water gas from the salt-containing lignite of the Hermine-Henriette pit, are in progress and were pushed forward considerably this year and led to a decided clarification of the problem. Beside many smaller experiments, a larger experiment of six weeks with 120 t.p.d. of salt coal fines from Deuben showed that prospects with a sound basis exist, showing that lumpy hard salt fines may be gasified by heating with oxygen-steam mixtures if necessary. However, the rotating grate-or Brassert-generators which must be used here must still-be-improved in structure, if the sludge problem is to be finally mastered. Also the generator for tapping seems to be suitable for gasification of salt fines with oxygen and steam mixtures.

In 1941 it is proposed to repeat the large scale experiments.
pressure
Continuous/concentration of ammonium sulfite

(Personnel listed)

With a view to larger possibilities of application in the nitrogen projects, outside of Germany, the continuous pressure throughput of ammonium sulfite into ammonium sulfate and sulfur on a technical scale was tested and worked out. It was determined that the continuous method of operation went smoothly on the plant scale and showed great advantages over the previous Chargen operation (for example, plant Japan 3).

The continuous method of operation required practically no steam for the conversion (Chargen operation: 0.6 tons of steam per ton of ammonium sulfate). The conversion takes place at low pressure (at 10 instead of 20 atmospheres). The production of a continuous autoclave is five times larger than an autoclave in the Chargen operation.

As is shown, the danger of corrosion difficulties is greater in continuous pressure concentration. Arrangements were, however, made (including the use of

(Page 21 contd.)

ceramic materials) and in part tested, which will very probably exclude the danger of corrosion.

(Drawing opposite page 22)

Ammonia Plant

Flow diagram for subsequent synthesis

325 atmospheres

Final circulation

Subsequent synthesis

Oil wash
(liquid ammonia wash)

Vom Hauptkreislauf - from the main circulation

Umlauf-Pumpe - circulating pump

Ofen - furnace

Nachschalt-Kompressor - subsequent compressor

Maulwurf-Pumpe - mole pump

Frischgas - fresh gas

Entsp-Ventile - pressure release valve

Wascher - washer

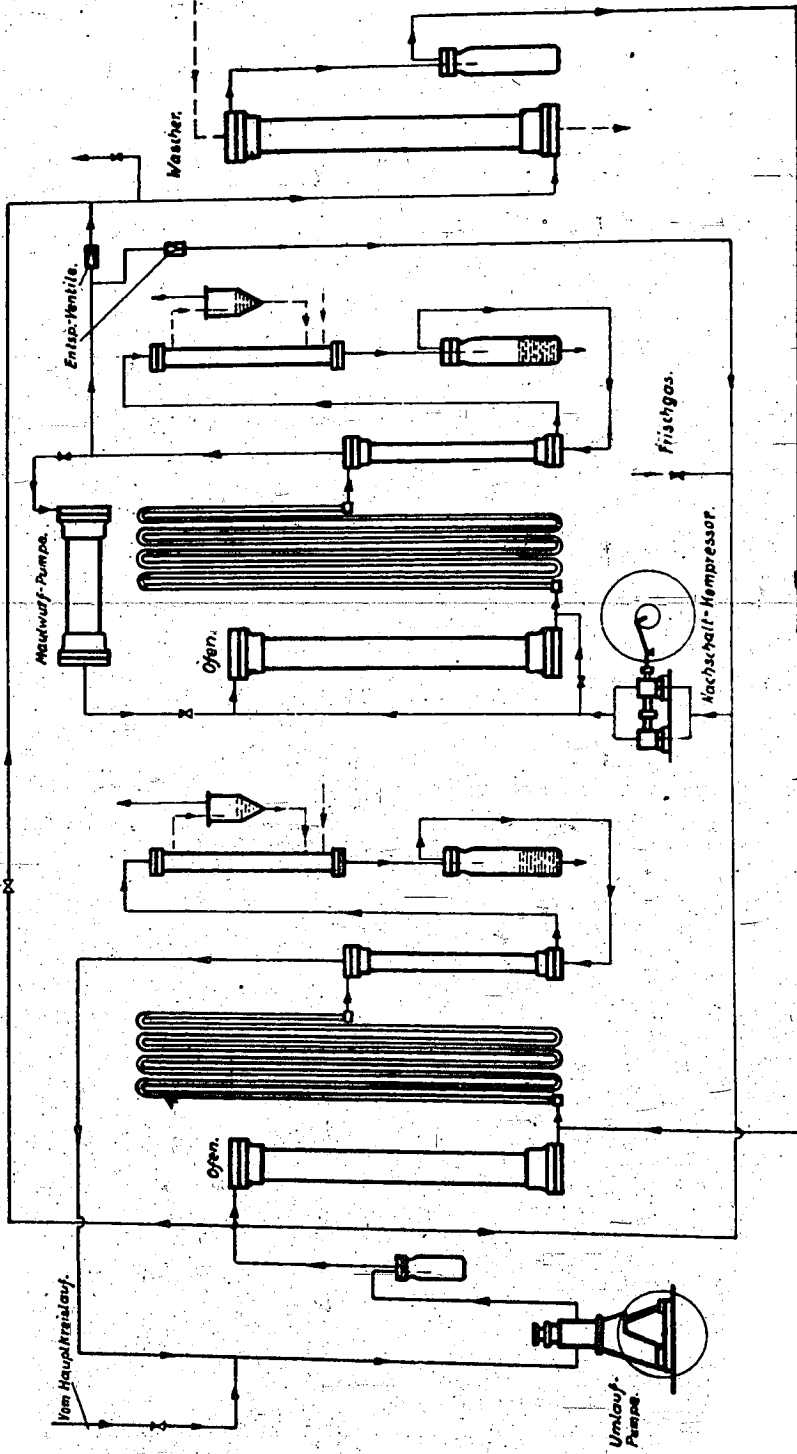
Ammoniakfabrik.

Schema der Nachschalt-Synthese.
325 atm.

End-Kreislauf.

Nachschalt-Synthese.

Öl-Wäsche.
(NH₃flüssig-Wäsche.)



fertig 22

Ammonia Plant

(Managing personnel listed)

Operations in 1940:

The substitution planned for the oil wash by means of a liquid ammonia wash with regard to the final circulating gases was partially carried out. In an 800 washer half of the final circulating gas 5000 cu.m./h. to be washed was treated with about 20 cu.m./h. of liquid ammonia. The remaining portion of the final circulating gas is still treated as before with oil.

It was not possible to set up a mole pump with a performance of 50,000 cu.m/h. for the final circulation in place of the one used before, only half utilized piston circulated pump, because of delays in delivery in the year 1940. For the same reason an increase in circulation pump capacity by setting up three further mole pumps (each of 100,000 cu.m./h.) was not possible.

The development of the preliminary purification planned for the total fresh gas through preliminary furnaces could not be carried through further, as in the operation of the full space ovens, used as preliminary purification furnaces, considerable injuries were found on the furnace charge. Because of high temperatures at the furnace regenerator inlet and the consequent nitrition of the material cracks occurred in the passage from the contact space to the regenerator and in the lower part of the contact tube. For this reason the preliminary purification of the fresh gas with M-carbon was also for the present not further developed.

In order to cut down the temperature of the contact in the preliminary furnaces, a new full space furnace construction was developed, in which in the contact space three cold gas inlets and blending to mix the gas streams were arranged. This furnace could be put into operation

(Page 22 contd.)

at the end of the year. A full space furnace with cooling tubes, corresponding to a patent of the firm Uhde, was put into operation with fresh gas at the end of 1939. As the temperature rose too high thereby, the furnace was later fed with circulating gas. Experiments with this furnace were continued.

With a new tubular furnace construction, a contact with smaller granulation (3 mm.) than before should be used to increase thruput. To decrease the resistance the contact space was subdivided into four contact tubular sections arranged in parallel with a contact layer height of 2 meters each. The furnace gave a very low resistance in operation with a separate gas circulation and yielded up to 100 tons/day N.

The experimental plant, with which the performance of a 500 furnace at 325 ats. pressure was to be determined, was in operation for four weeks. In spite of the bad condition of the charge an average performance of 23 tons/day N was attained in this way, corresponding to the guarantee figures for foreign projects. The 325 ats. plant was subsequently used to utilize the final circulation gas for further ammonia production under this pressure and at the same time to increase the inert gas picture and in that way to attain a decrease in the amount of oil- or liquid ammonia-washed final circulating gas (see after-synthesis).

Operations for 1941:

The isobutyl oil residual gas, the pressure having been previously released to one at., is in the future to be released only to 30 ats. and then subjected to a pressure conversion, considerable energy costs may be saved in this way. An apparatus for this has been worked out, which is to be set up in building 11.

(Page 22 contd.)

To increase the efficiency of the ammonia contact by electrical-aftermelting, an electric arc furnace of the AEG was set up in a place, arranged for it, next to the contact melt in kettle house, building 245.

In order to prepare for increasing the operating pressure to 250 ats. which will probably be successful in the year 1942, all those parts of equipment, especially flanges and covers, not corresponding to this operating pressure, are exchanged and supplemented.

Experiments with new furnace charge construction were continued.

(Drawing of opposite page 23)

HYDROGEN PURIFICATION

Recirculating gas washer

Kondensat-condensate

Überlaufsyphon-over flow syphon

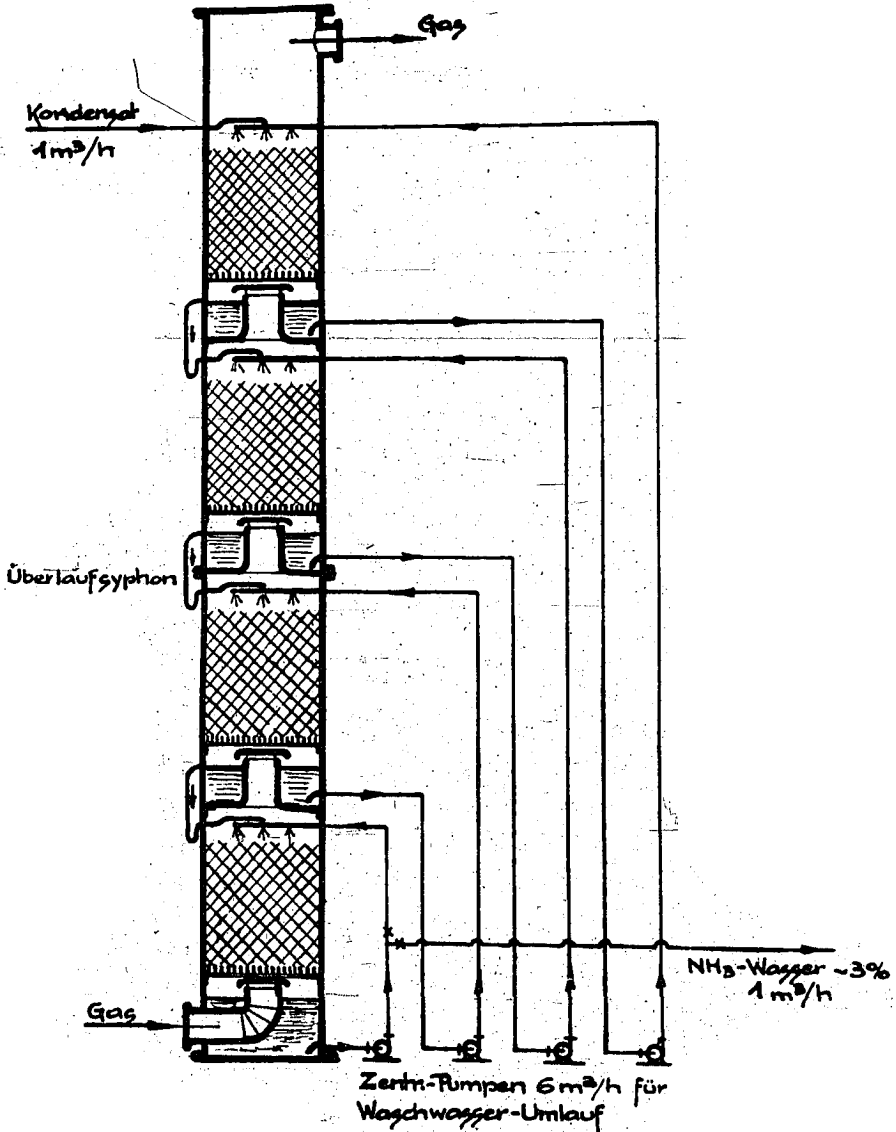
NH₃-Wasser-ammonia water

Zentr-Pumpen 6 m³/hr für Washwasser-Umlauf-
Central pumps 6m³/hr for Wash-water circulation

Wasserstoff-Reinigung

Feurig 23

Rückgas-Wascher



HYDROGEN PURIFICATION

(Managing personnel listed)

Operations in 1940:

The new construction of the pressure release machines for the building 334 is almost completed and a new pressure release machine has been set up in it and put into operation.

In building 41 and building 10 the cylinders, not without disadvantages, was replaced in each case by new cylinders in the pressure release machine.

In building 10 the recirculating gas washers were replaced by a single washing tower, in which all 4 washing stages are unified (see sketch). The improved process of washing recirculating gas resulted in a further considerable saving of material and space over the improvement which was carried out in the previous year in building 106.

The decrease in diameter of the copper washers was taken care of in the widening of 11 washers; at the same time in these washers liquor brakes were built in.

Four new carbonmonoxide recorders (meters) for the control of the washer were delivered and set up in building 106.

Further work planned for the year 1940 could not be carried out because of lack of personnel and material.

In order to prevent precipitation of salt out of the pure gas and out of the copper liquor due to cold and consequent danger of stoppage the pure gas valves as well as a part of a stripper and syphon of the washer were supplied with heating and insulation.

(Page 23 contd.)

The structures 10 and 106 were switched into Hy-gas network, so that they may be converted in the shortest possible time to purification of Hy-hydrogen.

The cooling plant for pure hydrogen was extended to 2 coolers, in order to be able to give a sufficient amount of purified gas for the increased demands of Schkopau and for the new operation in the south of the plant.

In order to decrease the impurities of the Sti-synthesis gas, enriched in the carbommonoxide recirculating gas, which in the past were carried back into the Sti-crude gas, conduit combinations were set up to lead the carbon monoxide residual gas occasionally into the heating gas instead of into the Sti-synthesis gas.

~~To determine the copper losses at all places where copper could be~~ carried out with the gases or liquids in the operation, a large number of analyses were carried out. It was found that the main losses are due to the copper liquor being carried over in the ammonia after-washer. The other copper losses are kept as low as possible by better supervision of the operation and increased control of the plant waste water.

Operations for 1941:

In structure 10, the pressure release machine of Politz, which is still in operation, is being replaced. In the new structure 334b the 2 pressure release machines taken from structure 334 and a fourth new machine are being set up.

In structures 106 and 334 the recirculating gas washer is being altered according to the pattern of structure 10.

Overcoming the decrease in diameter and building in liquor brakes into the washers are being continued.

(Page 23 contd')

In structure 10 a 500 ϕ after-washer was replaced by a 700 ϕ after-washer.

At the south of structure 10 the new building for the pressure release machines was erected. The building 41 is to be expanded to take in 4 pressure release machines toward the south.

Rebuilding of the cooler plant in structure 334 is to be continued.

As far as high pressure space for this is available, the stripping space for the gas between the copper liquor washers and the ammonia after-washers in the Sti-gas purification structures, is to be enlarged.

(Drawing opposite Page 24)

Gas Compressor Operation

Bauten - Construction

Zahl - Number

Typen Und Verwendungszweck der Kompressoren - Types and purpose of the compressors

Kompr. III im Umbau - Compressor III being rebuilt

Sti - Sti

Hy - Hy

Meth^{II} mit CO₂ - Meth^{II} with CO₂

Meth^I ohne CO₂ - Meth^I without CO₂

Sti od. Hy od. Meth. - Sti or Hy or Meth

Hy od. NH₃ fl. - Hy or liquid ammonia

Sti od. Hy - Sti or Hy

Meth I od II - Meth I or II

Elektr. Dopp. Komp. - Electric double compressor

Einh. Komp. - Uniform compressor

Wald. Komp. - Wald. compressor

Turbo - Turbo

Verteilung der Kompressorentouren - Distribution of compressor revolutions

Mögliche Gesamtouren - Possible total revolutions

Touren pro minute - Revolutions per minute

Meth-Touren - Meth-revolutions

Hy-Touren - High-revolutions

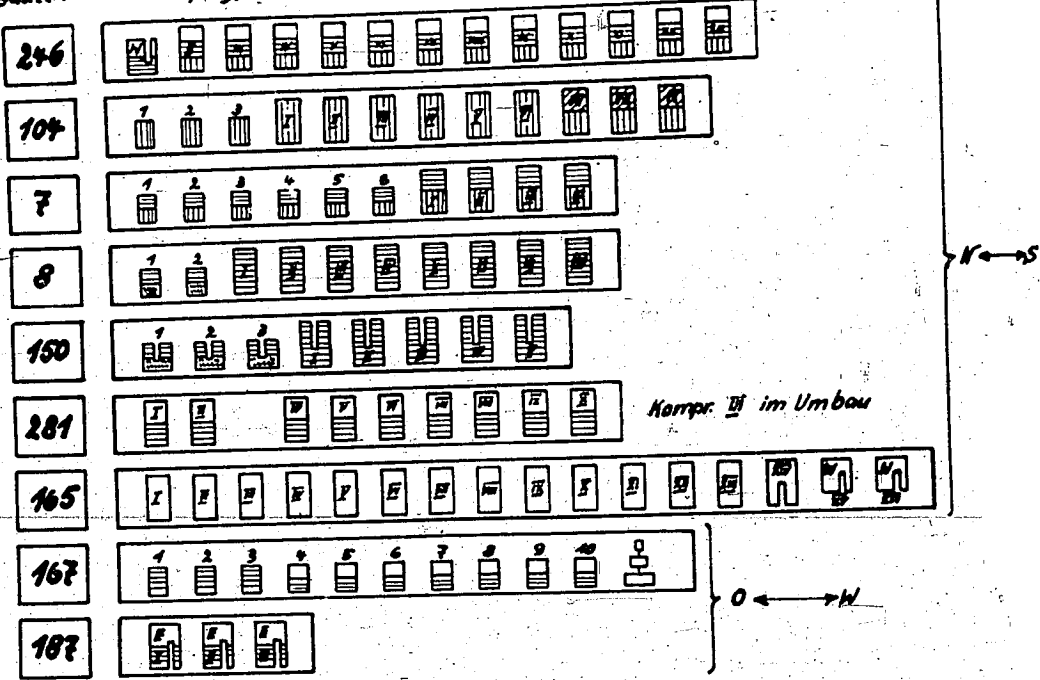
Sti-Touren - Sti-revolutions

Tatsächliche Touren - Actual revolutions

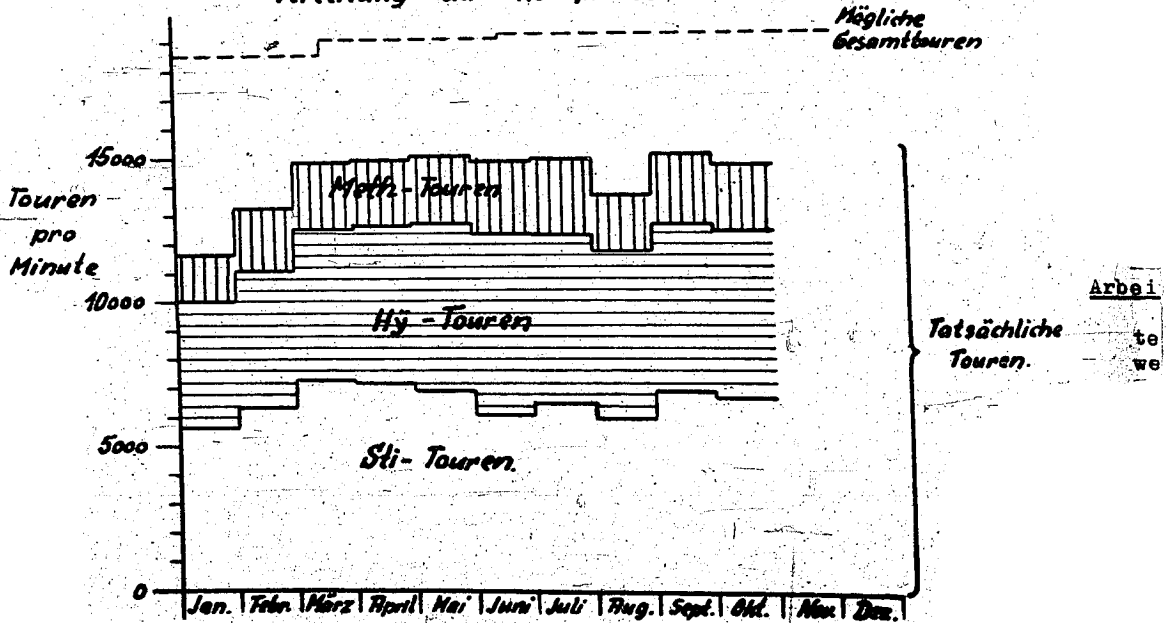
frei v. 4

Gaskompressorenbetrieb

Bauten Zahl, Typen und Verwendungszweck der Kompressoren.



Verteilung der Kompressortouren.



Gas Compressor Operation

(Managing personnel listed)

Operations in 1940:

The third uniform compressor with electric starter in building 187 was put into operation in March. Experiments (removal) for a uniform compressor showed a 5% higher performance than the guaranteed value.

Expansion of the ammonia liquefaction plant led to building a third and last additional compressor in building 150 into an electrically started double compressor. For the same reason the two additional compressors in building 8, operated with vaporizing machine, and the liquefaction in building 150 were switched in, being enlarged by setting up three tower liquefiers. The total performance for ammonia liquefaction was increased thereby from 247 to 390 tons per day.

The methanol gas compression capacity was increased, by additional insertion of all Hy-gas compressors in building 7 into the methanol gas network, by 16,000 cubic meters meth-pure gas.

The conversion possibility for Hy-hydrogen was increased by inserting the three uniform compressors with a total suction of 48,000 cubic meters on the Hy-network as well as the Sti-network.

The firmer arrangement of the shaft of the turbo compressor proved satisfactory. The turbo was in continuous operation, with the exception of being shut off for a short time for cleaning the condenser and cooler. Delivery experiments cannot be carried through just yet as, because of the war, the manufacturing firm (GHH) could not yet deliver the new turbine slider and low pressure slider.

The degree of utilization could not be as yet brought to 80% by the middle of the year because the repair condition of the gas machines was

(Page 24 contd.)

not improved because of increase in detonation. The detonation number increased when butane and ethane were taken out of the Hy-residual gas, the heating value being lowered and the hydrogen content increasing because of this. Primarily marked crack formation occurred at the outlet valves and in the case of the additional gas machines also in the cylinder heads, so that, for example, of the ten additional machines, instead of two, three machines were in constant repair. An improvement of the power gas is for the present being attained by constant addition of about 500 kg. per hour of butane.

The air addition valves were built in to overcome the detonation for Hy-hydrogen content of the power gas, in the case of 20 large machines out of 23 and for three additional gas machines out of 10. For continuous operation it was shown that the gas machines supplied with air addition valves have about half as much detonation. Great difficulties were encountered when German hot steam cylinder oil was used instead of Pennsylvania. The piston rings of the vapor machines (26 mm) which were worn down on the average by 2mm per year, were worn down 12 mm in 14 days. This danger was decreased by cutting down the vapor temperature from 320° to 260°C. and by mixing the German oil with the Pennsylvania oil. This mixture has not yet produced a definite improvement (a wearing down of 2 mm in 4 weeks). Experiments are in progress using various other oils and altering the pistons.

Experiments with large water- and dust-separators on the turbo compressor were successful. Dangerous coating of the runner wheels was thereby prevented.

Operations for 1941:

Two compressors previously operated with gas machines are being converted to a further double compressor, electrically operated. Two gas addition machines are being supplied with cooled cylinder load bush, in order to avoid the high wearing down of the cylinder bush and in order to operate the machine at a cooler temperature and free of detonation.

(Page 24 contd.)

In order to determine the cause of the detonation, which appears to be present not only in the case of a high hydrogen content of the power gas, experiments on the influence of certain gas constituents or insufficient gas mixing were continued with the high pressure laboratory.

(Drawing opposite Page 25)

Pressure Water Purification

Leading off CO₂ and Separating CO₂ and Water

Alte Ausfuhrung - old arrangement

CO₂ - Netz - CO₂ network

Abscheider - separator

Fruher Kanal - previous canal

Jetzt - now

Entspannungs-wasser - water from pressure release

Rohwasserbehälter - crude water storage tank

Neue Ausfuhrung - new arrangement

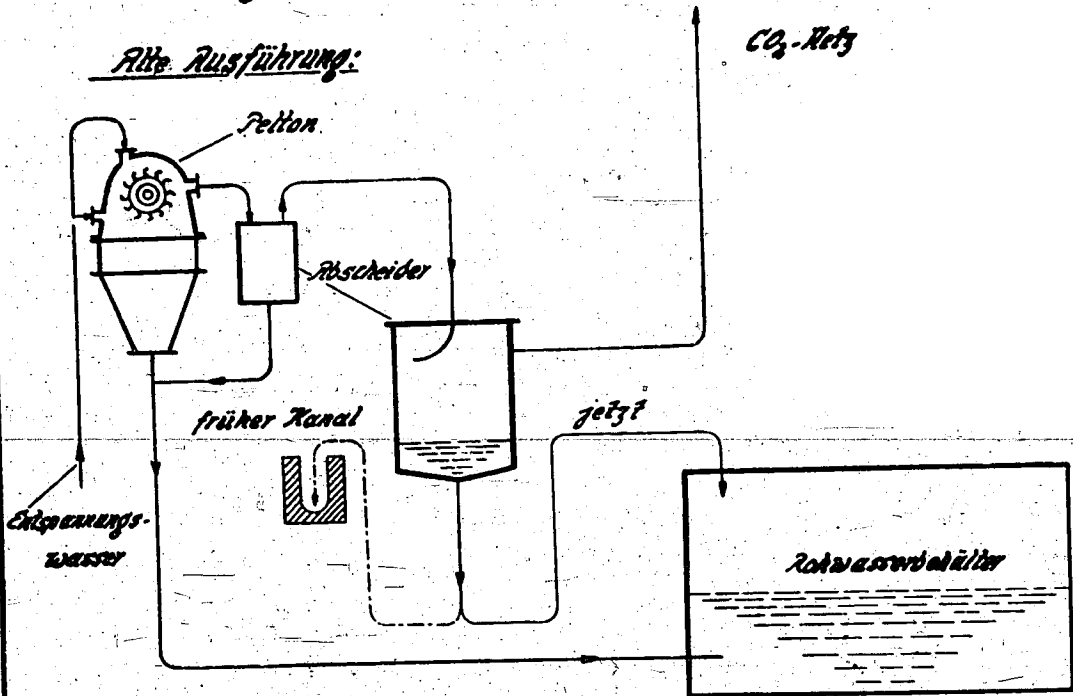
CO₂ uber Dach - CO₂ overhead

faunig 25

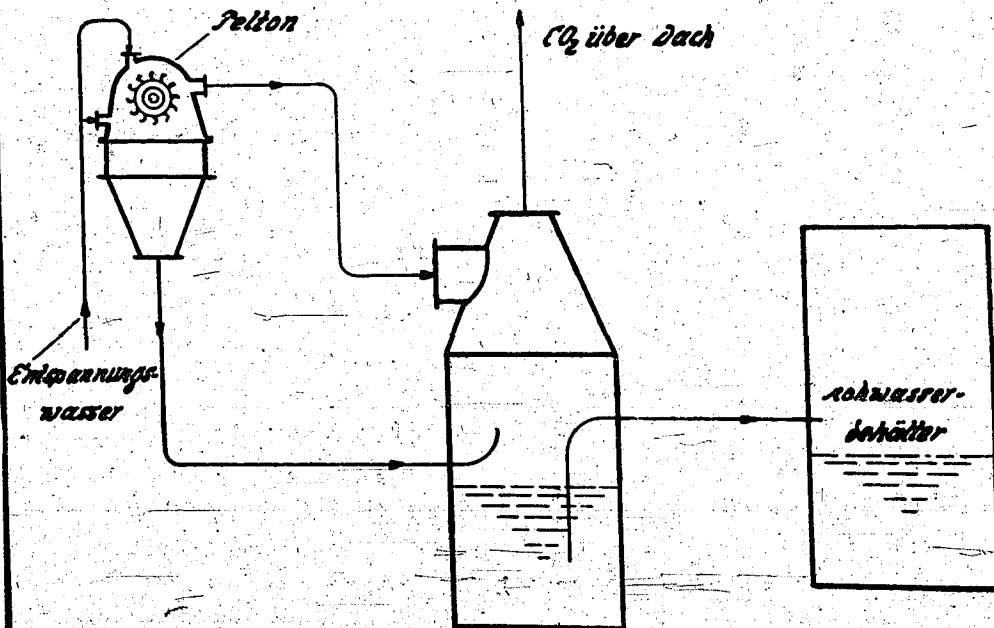
Druckwasserreinigung.

Abführung der CO_2 und Trennung von CO_2 und Wasser.

Alte Ausführung:



Neue Ausführung Bau 78:



Pressure Water Purification and Water Regeneration

(Managing personnel listed)

Operations in 1940:

A gasification plant for the south pressure water purification structure 9, which previously did not have its own water regeneration, was completed and put into operation at the middle of the year. The building and erection work of the new pressure water purification and water regeneration (structure 78) at the north of the plant was finished; the new building being put into operation in October. It can be used for the purification of the ammonia synthesis gas as well as for the Hy-hydrogen also. This new pressure water purification plant, instead of the previously used washer, 16 meters high and 1400 mm in diameter, was supplied with four large washers, 20 meters high and with 2300 mm diameter, this diameter being 2.75 times that previously used and the filled washing column being more than 3 meters higher. This increase in the height of the washer should effect a decrease in the specific water consumption and thus a considerable energy saving. Larger machine units were set up to correspond with the increased washer performance.

In contrast to the old plants, in case of the new one, the pressure water purification and water regeneration were unified into one structure, the cost of building being lowered thereby and the current cost and consumption of energy being decreased. A particular characteristic of the plant is the fact that the water of the pressure water washer and of the gasification plant is fed in closed circulation. The performance of this plant is thus independent of all disturbances in the low pressure water network. Besides, in the new pressure water purification improved Pelton turbines were set up and the water separator coming after the Pelton turbines was omitted. Previous results of operation have shown that the changes in the new structure were advantageous as compared to the old ones.

(Page 25 contd.)

The summer capacity of the structure amounts to about 70,000 cubic meters per hour of crude gas, the capacity of the total pressure water purification is thus increased by about 16% to approximately 500,000 cubic meters per hour for high water temperatures.

The capacity of the water regeneration increased by about 36% to 32,800 cubic meters of water per hour, due to the two new regeneration plants. The contemplated stocking up of the washer for a purification structure of 5 meters could not be carried out in the year 1940 as no iron for this was available.

With a higher loading of the two older pressure water purification structures, up to 200 cubic meters of water were carried over by the Pelton carbonic acid per hour, this separating from the gas only in the separator of the carbonic acid gathering line and previously passing on into the Kandel. By relaying the lines it is now led on to the water regeneration. The attached sketch shows this change in the separation of carbonic acid and water.

In the case of a Pelton turbine performance experiments were carried out with better developed runner wheels (with regard to technical flow), this permitting a better utilization of pressure release energy, to about 4%.

Operations for 1941:

Stocking up the washer of a purification plant up to 5 meters is to be carried out because of expected advantages as soon as the required iron necessary can be obtained.

In the Sti-crude gas network (25 atmospheres) the fall in pressure between compressors and pressure water purification is particularly great. As a considerable resistance in the narrow gas inlets and outlets of the larger strippers for crude and pure gas in structure 325 was found, the poured steel covers of these strippers are to be replaced by connections with larger diameter.

(Drawing opposite page 26)

Nitrogen-Oxygen Plant
(Structure 337)

CO₂-Kompr. - CO₂ compressor

Luft-Kompressoren - Air compressors

Sauerstoff-Kompr. - Oxygen compressors

Oxim-Anlage - Oxime plant

Waschturme f. l-Dr.-Luft - Wash towers for l-Dr.-air

NH₃ Kompr. fur Trockeneis - Ammonia compressor for dry ice

CO₂-Verfluss. fur Feuerwehr - Carbon dioxide liquefaction for fire prevention

NH₃-Kompr. f. Wassereis - Ammonia compressor for water ice

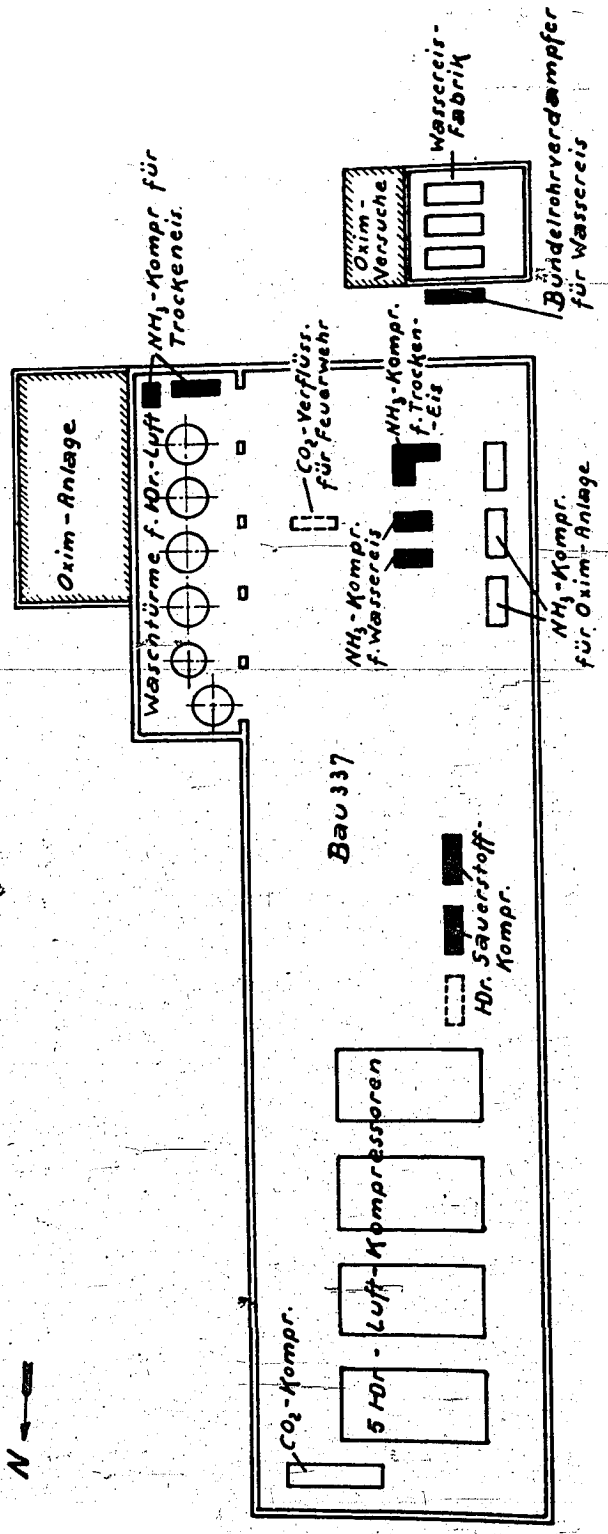
NH₃-Kompr. fur Oxim-Anlage - Ammonia compressor for oxime plant

Oxim-Versuche - Oxime experiments

Wassereis Fabrik - Water ice plant

Bundelrohrverdampfer fur Wassereis - Tubular bundle vaporizer for water ice

Stickstoff-Sauerstoff-Fabrik (Bau 337)



Jan 26

M = 1:500

Nitrogen-, Oxygen- and Krypton-Production

(Operating personnel listed)

Nitrogen- and Oxygen-Production:

Operations in 1940:

The old Linde plant in building 337 with the auxiliary high pressure air- and low pressure air-concentrators, as well as the two high pressure compressors taken over from Waldenburg in the year 1937, were torn down and turned over to Waldenburg. The ammonia concentrators remained standing and were put into operation again for the oxime production plant. In building 343 of the six high pressure concentrators present two were consequently converted from synthesis gas to air.

In order to supply the plant with sufficient protective nitrogen also during the thaw period of one of the four pieces of apparatus, which up till that time were able to supply pure nitrogen, a fifth Linde-Frankl apparatus was switched into the nitrogen system. The maximum production amounts to 50,000 cubic meters per hour of 99.5% nitrogen.

A sixth Linde-Frankl apparatus was converted so that the additional condenser could be thawed during the operating time.

The liquor wash towers for high pressure air in building 343 were again set up ready for operation for safety's sake (for four pieces of apparatus).

In order to increase the degree of utilization of the Linde-Frankl apparatuses, which is now 95-96% after the average of 120 days required for thawing, the time periods of operation between two thawing periods are to be increased as much as possible. Consequently by means of careful analysis, it was determined what increase in the content of acetylene and hydrocarbons in the main condenser of an apparatus takes place during longer periods of operation as opposed to shorter periods. Previous findings made it possible

(Page 26 contd.)

to increase the operating periods to about 170 days. The experiments are being continued for the purpose of further lengthening the operating periods.

In order to ~~keep the~~ carbonate hardness of the recirculating cooling water in structure 247 at about 6°, a plant for injecting with hydrochloric acid was put into operation.

Operations for 1941:

In order to cover the constantly increasing demand for oxygen in the plant, in structure 337 a new Linde-Frankl apparatus with a performance of 3650 cubic meters per hour of 98% oxygen was set up.

Krypton Production:

Operations in 1940:

The 25 cubic meter per hour pure Krypton column was exchanged for one of 60 cubic meters per hour. The Krypton production, as compared with 1939, was lowered from an average of about 32 cubic meters to about 29 cubic meters per month. The reason was the necessity to turn off the main apparatus during the extreme cold of January 1940 and due to the frequent turning off of the Krypton production in the line of air protection measures.

(Drawing opposite page 27)

Flow Diagram of the Water Ice Plant
(structure 242)

Before Rebuilding

Flüssig-Abscheider - Liquid separator
Kondensator - Condenser
Wasser - Water
NH₃-Kompr. - Ammonia compressor
Flüssigkeits-Sammler - Liquid collector
Eiszellen - Ice cells
Sole - Brine
Verdampfer - Vaporizer
Eis-Generator - Ice generator

After Rebuilding (fully automatic regulation)

Generators, brine pumps and ammonia compressors switch themselves in and out automatically.

Bündelrohrverdampfer - Tubular bundle vaporizer

Sole - Brine

Aut. Regul. Ventil - Automatic regulating valve

NH₃-Kompressor - NH₃ compressor

Sole-Pumpen - Brine pumps

Gesteuertes Ventil - Control valve

Thermostat - Thermostat

Eiszellen - Ice cells

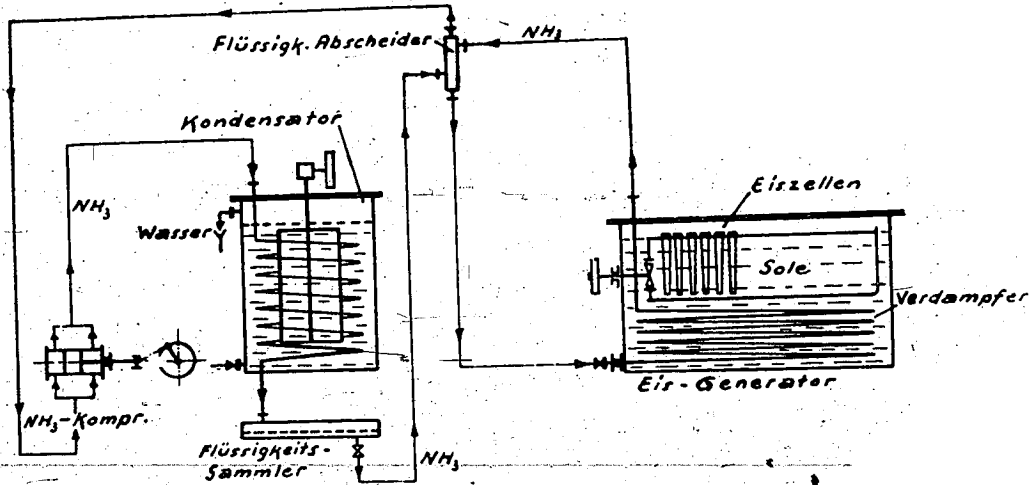
Eis-Generator - Ice generator

NH₃-Kreislauf - Ammonia circulation

Sole-Kreislauf - Brine circulation

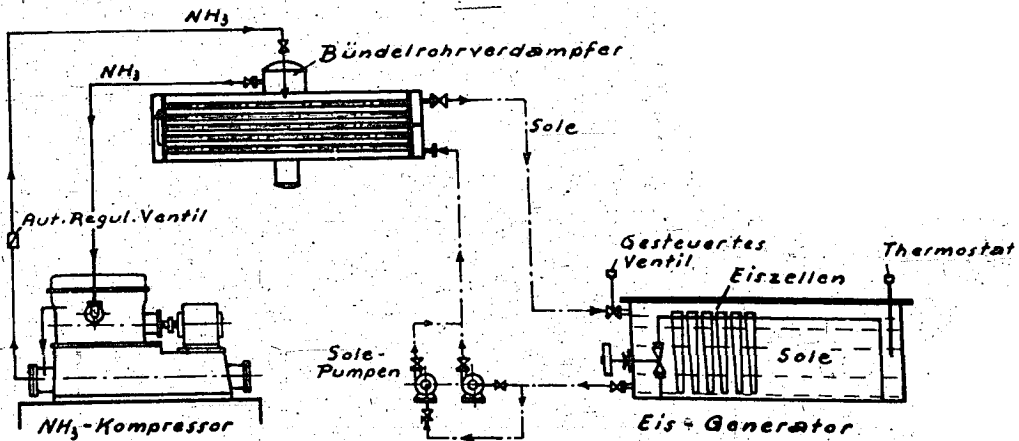
Schema der Wassereis-Fabrik. (Bau 242) *frei 27*

Vor dem Umbau.



Nach dem Umbau. (Vollautomat. Regulierung)

Generatoren, Sole-Pumpen und NH₃-Kompressoren schalten sich selbst ein und aus.



— NH₃-Kreislauf
 - - - Sole-Kreislauf

Nitrogen- and Oxygen-Plant Auxiliary Equipment, Structures 12, 247, 337

(Managing personnel listed)

Operations in 1940:

The requirement of high pressure oxygen continually increased for the production of "Hoko" acid (Hokosaure) and for the laboratory, so that the performance of the compressors was no longer sufficient. For this reason two new concentrators were set up in building 337 and put into operation. Compressor capacity at present is at a maximum of 1300 cubic meters per hour.

The need for water ice for the laboratory and plant setup further increased, so that the previous plant with a performance of a maximum of 800 sticks of ice per day was no longer sufficient. By replacing the brine vaporizer in the individual ice generators by a joint vaporizer having bundles of tubes outside the generators, the performance was increased to 1200 sticks per day. This performance could be attained by using a greater brine rate in the tubular bundle vaporizer than was possible in previous individual vaporizers. The heat throughput number "K" was thus increased from 200 to 950 kcal/m²/h/°C and the transferable coal was increased from a maximum of 90,000 to a maximum of 125,000 kcal/h. By automatically controlling the brine temperature, the brine pumps and the cooling machines, the economic efficiency of the plant was increased.

The demand for dry ice increased both for the laboratory in the Leuna plant and for the Schkopau plant, so that the capacity of the plant of 1400 kg. per day was no longer sufficient. By setting up a constant carbonic acid pressure, by pressure release of an always constant amount of inert gas by controlling the state of the liquid ammonia and the carbonic acid in the coolers by using Hampson meters, it was possible through improved drying of

(Page 27 contd.)

the carbonic acid and inserting a further ammonia cold machine, to increase the capacity of the plant to a maximum of 3200 kg. per day.

For reasons of improving operating safety the oil from the boilers of the oil purification is no longer pressed off with nitrogen, but is led through pumps to the settling container placed high.

Operations for 1941:

As the requirement of the plant for high pressure oxygen for the new operations in the south were increased by 300 meters per hour, a third oxygen compressor was set up in structure 337.

Filling liquid carbonic acid into bottles for fire fighting purposes, which formerly went to the carbonic acid works Engelsdorf, was now carried out in plant 337. For this purpose a new carbonic acid concentrator and liquefier was set up.

Because of the consumption of hydroxyl-amine salt in the plant and because of the requirements of other plants the previous experimental plant in structure 287 became too small. The capacity is to be increased in a new large scale plant from about 500 to about 2000 kg. per month.

(Drawing opposite page 28)

Phenol Plant

Phenol water extraction with triphos

Old Method of Operation

Roh-Wasser - Crude water

Entphenoltes Wasser - Water with phenol removed

Betr. - Plant

Res. - Standby

New Method of Operation

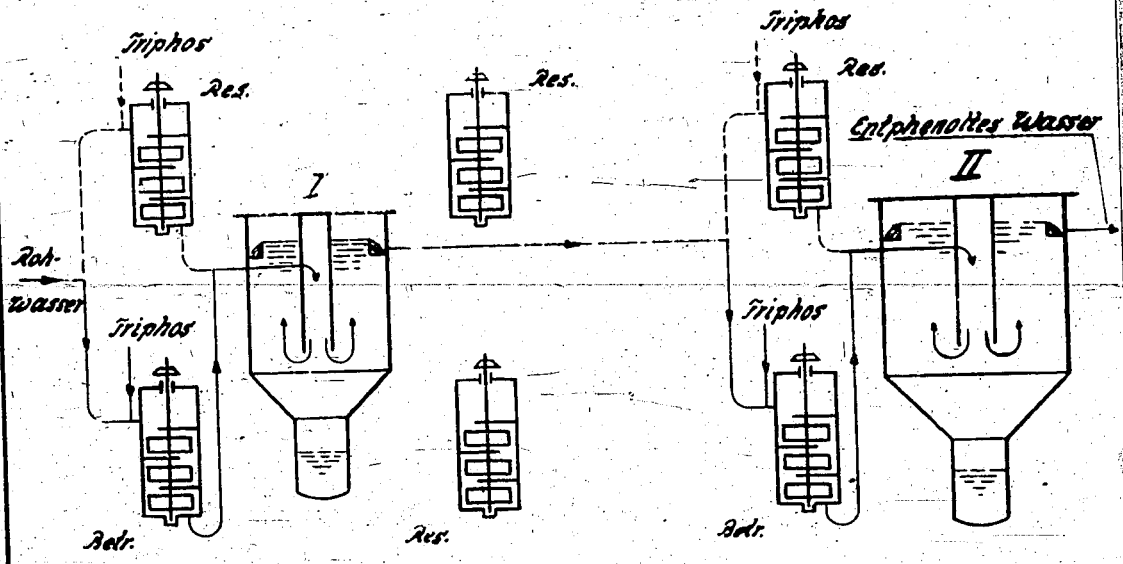
Translation of words same as above.

Jan 1888

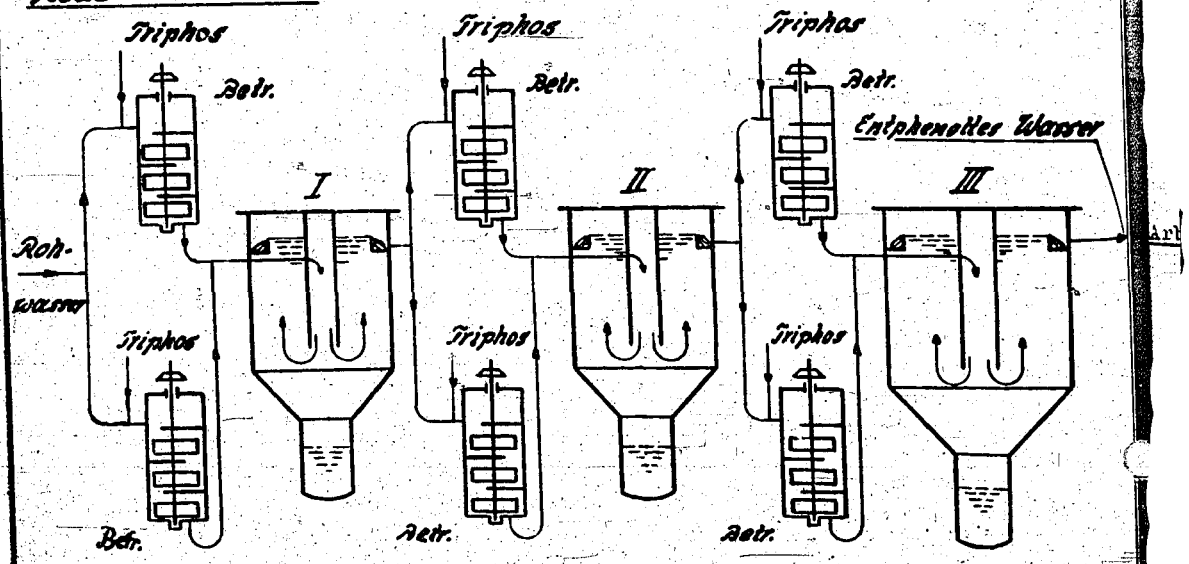
Phenolbetriebe.

Phenolwasser-Extraktion mittels Triphos.

Alle Fahrweise.



Neue Fahrweise.



Phenol Plant

(Managing personnel listed)

Operations in 1940:

A. Phenol Removal

In order to broaden a further extraction stage, a third settling container was set up, being put into operation at the beginning of 1941. By using this container and the stirring equipment on hand it is possible to work up the phenol water occurring in larger amounts and to remove the phenol to a greater extent than before.

Setting up a 4. triphos regeneration column with auxiliary equipment was delayed because of late delivery of material and was not finished until the beginning of 1941.

The impure triphos from circulation is now worked up with phosphoric acid instead of with fuming sulfuric acid. Compared with the fuming sulfuric acid operation, the new method of working is very markedly simpler. Further, the total losses of triphos are thereby cut down from about 300 grams per cubic meter of phenol water worked up to about 150 grams per cubic meter.

Regenerating the impure triphos by extraction with light gasoline in aqueous methyl alcohol solution was carried out successfully in plant experimental apparatus. Laboratory findings could be verified.

The extraction of phenol with ether (Phenosolven process) was next tried out in an experimental apparatus for one cubic meter per hour of hydrogenation waste water. Because of favorable results a large experimental plant for the phenol removal from distillation water was set up at the distillation plant Deuben. The experiments are being carried out by Leverkusen (Dr. Dierichs). The plant was enlarged for a throughput of 5-6 cubic meters per hour and gives a preliminary picture for large scale installation (for example Politz)

B. Phenol Preparation

Two new horizontal containers of 20 cubic meters capacity each were set up for storing R-phenol oil, as the previously used Intze vessel had to be given up.

Buna rubber was found to be a suitable material to resist the marked corrosive action of the acidic refining residue. A residual line was coated with rubber, the residue being led through it for combustion in kettlehouse 103.

The M-oil caused a marked corrosion in the hydrogenation apparatus, during its distillation, because of the high temperature fuel, for this reason larger amounts of M-oil had to be prepared in the distilling bulbs of the phenol preparation, because no corrosion occurs here as careful warming with low pressure steam is used in combination with circulatory heating.

C. Phenol Oil Decomposition (separation)

In the circulation of cooling water for the condensers a used tubular heater was built in to avoid freezing of the condensers during marked frost.

Operations for 1941:

For phenol removal an overflow centrifuge is to be set up with firmly fixed revolving drum. In this centrifuge a partial stream from the regenerating column of the triphos, flowing out, is to be hurled and thus separated from the suspended impurities. It would appear that then the triphos regenerating system would have to be cleaned less often of asphalt-like precipitations.

The combustion of the refining residue, separating out in phenol preparation, in kettlehouse-103 is linked with high repair costs because of the strongly corrosive effect of the residues. An attempt is to be made to get rid of the residue in some other way.

(Page 28 contd.)

In the phenol oil separation plant a 30 mm diameter bell bottom column is to be set up and with it, it is to be determined whether continuous separation, instead of the discontinuous method previously used, is possible. The continuous method of operation is much superior to the other.

(Drawing opposite page 29)

Cyclohexanol Production

Flow diagram for the distillation

Von Phenol-Hydrierung - From phenol hydrogenation

Rohhexanol-Behälter - Crude hexanol container

Vorlaufkolonne - Preliminary column

Rohprodukt - Crude product

Dampf - Steam

Cyclohexan u. Wasser - Cyclohexane and water

Kühler - Cooler

Vorlauf - First runnings

Hauptkolonne - Main column

Zwischenprodukt - Intermediate product

Phenolhaltiger Rückstand - Phenol containing residue

Rückstandsbehälter - Container for residue

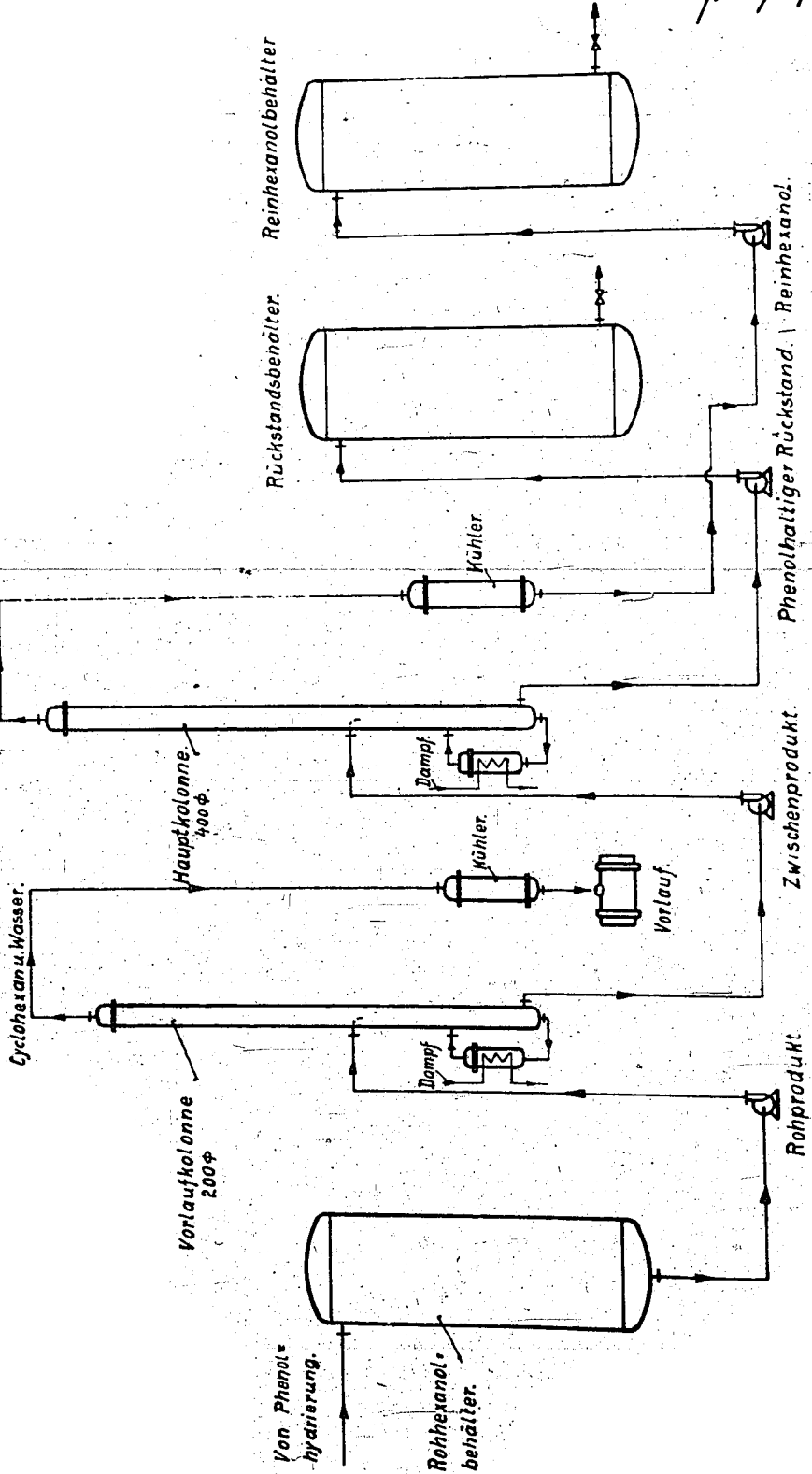
Reinhexanol - Pure hexanol

Reinhexanolbehälter - Container for pure hexanol

Cyclohexanolfabrikation.

Schema der Destillation.

frei 29



Cyclohexanol Plant

(Managing personnel listed)

Operations for 1940:

The expansion visualized for 1940 for cyclohexanol production to be increased to 500 tons per month was not carried out, because on the one hand not enough phenol was freed for the production of adipic acid (linoleum manufacture) and on the other hand the requirement for Luran production did not reach the extent anticipated.

At the end of 1940 two new furnaces of 800 mm. l.W. were put into operation, having been set up in the course of the year, operating according to the same principle as the former tubular furnaces of 300 mm. l.W., two of these still being in operation at the time.

The capacity of the furnace plant amounts at the time to about 220 tons per month. The total plant was simplified and completely rebuilt in connection with setting up the two 800 mm. l.W. furnaces.

Steam consumption was decreased by the fact that the steam produced in the reaction furnaces was used mainly to heat the hydrogen to 150°, so that no outside steam was required except to cover the small amount of remaining operation.

For cyclohexanol production, since the middle of the year the specially purified Hy-hydrogen from installation 334 for Schkopau was especially used. Further purification of this gas was carried out by converting the remaining carbon monoxide into methane at 320° over a nickel contact.

Since March 1940 the distillation plant, at the southwest corner of installation 11, including the storage containers, was put into operation. A first run distillate, consisting of water and cyclohexane, was separated in a preliminary column of 200 mm. l.W., while the cyclohexanol was

(Page 29 conti.)

distilled off at 158-161° in the main column. The phenol-containing hexanol occurring in sludge of this column is subjected once more to hydrogenation. The planned theoretical performance of 300 tons per month was not attained in the plant. The monthly capacity amounts to about 220 tons with the required purity of a maximum of 0.05 weight per cent of phenol in the pure hexanol.

Beginning with the middle of December the two small hexanol furnaces (300 mm. l.W.), which are still in operation, were used at the request of the organic division for the hydrogenation of tiglinic aldehyde to amyl alcohol (about 15 tons per month). From the point of view of the gas the furnaces are connected ^{after} as they were before in the hexanol-hydrogen circulation.

Operations for 1941:

Three furnaces with 800 mm. l.W. are to be additionally set up and to be put into operation about the middle of the year. The furnace capacity then invested in installation 11, about 500 tons per month, will, in the course of the year, meet the rising demand of hexanol until the time that the new plant in the south of the factory can take over this production.

A small experimental plant was erected in installation 11, information to be gathered there on obtaining cyclohexanol at ordinary pressure. At the same time it was to be determined whether in place of the hydrogen, the ammonia residual gas, free of impurities but with considerable content of inert gas, could be used for the hydrogenation. This plant is to be put into operation at the beginning of 1941.

The plant for preliminary hydrogenation of crude phenol oils, to be substituted for sulfuric acid refining, and scheduled for being put into operation in the year 1940, could not yet be made ready for operation because of lack of personnel and material. It should be in operation beginning about April 1941.

(Drawing opposite page 30)

Luran Production

Flow Diagram for Oxime Conversion

Oxim - Oxime

15% Vorlage f. neuen Ansatz - 15% receiver for new section

85% zur Neutralisation - 85% for neutralization

Ansatz behälter - Attachment container

Grobverteilung - Course separation

Feinverteilung - Fine separation

Verdränger - Displacer (remover)

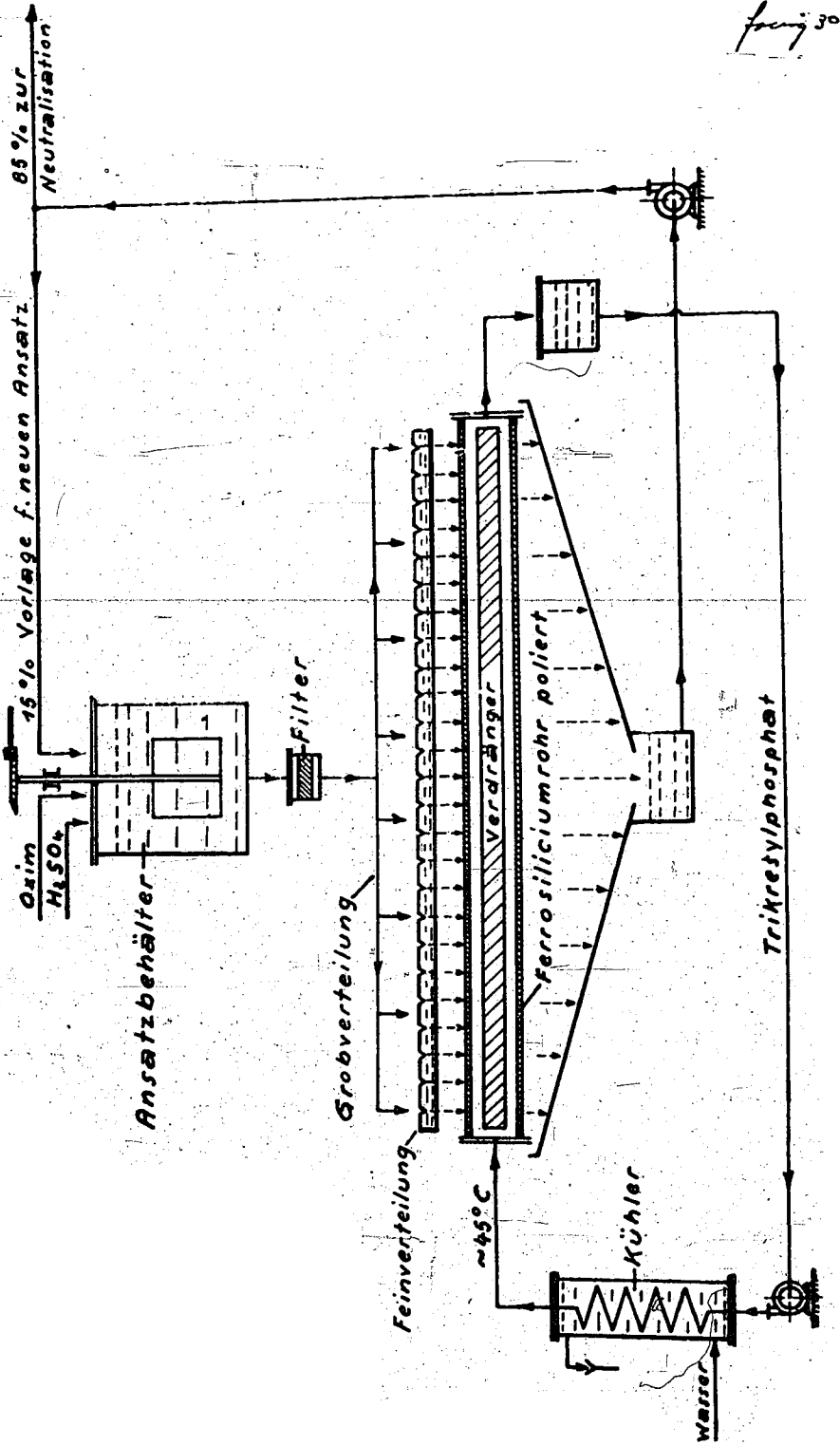
Ferrosiliciumrohr poliert - Ferro-silicon tube, polished

Kühler - Cooler

Wasser - Water

Trikresylphosphat - Tricresyl phosphate

Luran-Fabrikation.
Schema der Oxim-Umlagerung



Luran Production

(Managing personnel listed)

Operations in 1940:

For the preparation of aminocaproic acid-Laktam (Luran ML as intermediate products for the production of Perluran) a new process was worked out in 1939, operating apparatus being built for it in structures 337 and 245 and put into operation at the beginning of 1940. Production increased to 15 tons per month in the course of the year. In the meantime the plant was expanded so that its capacity was increased to about 30 tons per month. This expansion will go into effect probably at the beginning of 1941. For the two production steps the following changes and supplementary procedures were made:

1) Cyclohexanone-oxime production

The preparation of ammonium nitrite from calcium nitrite and ammonium sulfate solutions was converted for continuous operation.

The separation of the ammonium nitrite solution from the precipitated gypsum is accomplished at first through centrifuging. However it was very hard to get the gypsum out of the centrifuge; this was therefore replaced by a continuously operating rotating filter.

The continuous operation in laboratory experiments for production of ammonium bi-sulfite, was carried out and proved so favorable that now the continuous separation of ammonium bi-sulfite is to be used on plant scale.

The pumps, which feed the strong sulfuric acid solution of the hydrolysate as well as the gypsum-containing solution of ammonium nitrite, and which were made of lead or were lead covered, were largely worn down and therefore frequently unusable. In place of these, pumps made of substitute material, especially Durax pumps, proved satisfactory.

(Page 30 contd.)

2) Preparation of aminocaproic acid-Laktam

In the conversion of sulfuric acid oxime ester, the yield is increased from 65% to 85% by shortening the reaction time and lowering the conversion temperature. This was accomplished by having the conversion tube made of ferro-silicon and polishing it on the outside and cooling it on the inside by circulating tricresyl-phosphate. Besides this sulfuric acid in excess and 15% of the already converted product was added to the sulfuric acid oxime mixture.

In the saponification of the Laktam ester with ammonia the ammonia is no longer used in excess, but the reaction solution is kept slightly acidic. By this method of saponification a better separation of the oily layer from the aqueous ammonia sulphate solution takes place, the extraction of the crude oil with methylene chloride is considerably simplified and the salt obtained from the solution of ammonia sulphate is considerably purer and lighter than previously.

In the vacuum distillation the efficiency was increased by building in separators, containers, larger receivers, etc.

The final product was discolored in the beginning during storage and when it was worked up further into Perluran. By adding phosphoric acid to the first distillation and sodium hydroxide solution to the second distillation light-stable products are obtained, yielding light polymerization products.

For particularly high demands for polymerization, a Laktam is required which must be recrystallized after vacuum distillation. For recrystallization from propyl-ether a suitable apparatus is set up.

Operations for 1941:

Structures in buildings 337 and 245 are being expanded for a production of about 30 tons/mo. This expansion will be put into operation about the beginning of 1941.

(Page 30 contd.)

A new plant for 300 tons/mo. of Luran is being erected at the south of the works, this is scheduled to go into partial production of 150 tons/mo. in Summer of 1941.

High Pressure Experimental Laboratory

(Managing personnel given)

Operations for 1940:

The main work of the experimental laboratory was investigation on preparation of cyclohexanol and its homologs by nuclear hydrogenation of the phenols.

In 18 small experimental furnaces, each with a 100 cc. contact volume, various contacts were tested at 20 ats. on the one hand, for their life period, especially those being tested which were to be used in operation; on the other hand using the same contact, phenol oils, of both domestic and foreign source, were studied for their suitability in nuclear hydrogenation.

Two experimental furnaces were operated with the residual gas containing about 40% hydrogen (from ammonia production), this forming a cheap source of hydrogen free of contact poisons. It was shown that the contact in the hydrogenation of MD-carbolic acid or cresols, in the application without pressure, of very pure residual gas, showed the same performance as in the case of hydrogenation at 20 ats. with purified H₂-hydrogen in circulating processes.

In three additional furnaces at 20 ats., homologs of cyclohexanol, especially methyl and dimethyl cyclohexanol, were prepared in amounts of 50-200 kg. for further preparation. Thus the process for partial vaporization was found very satisfactory for saturation of the hydrogen, for phenols and cresols, for the purpose of holding back the contact poisons. For xylenols it was found less effective.

For the ammonia synthesis domestic and foreign contacts were studied with regard to the amount needed and compared with one another. Investigation of the refining effect of ammonia contact in the preliminary furnaces showed that with the retardation of ammonia formation the de-poisoning effect with regard

(Page 31 contd.)

to conversion of carbon monoxide and hydrocarbons into methane also decreased markedly. This is even the case when the contact is given electrical subsequent heating. On the other hand de-poisoning action was unchanged for many months in an experimental furnace, if the ammonia formation is avoided and the fresh gas is led over the contact at a temperature of only about 200°. If the water, resulting from the conversion of carbon monoxide or oxygen-containing organic compounds, is subsequently removed by drying the gas, (for example with liquid ammonia), then an ammonia furnace, operating with the gas, thus preliminarily purified, gave the same degree of conversion as with the use of the purest synthetic gas, prepared by splitting out liquid ammonia.

The experiments showed the possibility that fresh gas for the ammonia production may cause difficulties, which have not yet been overcome, requiring extensive preliminary purification, without the use of ammonia-forming preliminary furnaces, with their frequent change of contact and construction complications.

In an experimental furnace various contacts for the pressure conversion of isobutylene expansion gas was investigated with regard to conversion, contact temperatures required, formation of precipitates, etc. In case of absolute freedom from sulfur of the gas to be converted, among others very sensitive contacts may also be used, with which particularly high conversion is attained.

Gas analyses investigation--determination of unsaturated compounds by their absorption in concentrated sulfuric acid and subsequent oxidation-- showed space and time variations in the composition of the power gas and relationships of these changes with occurring detonations. A poor distribution of olefins over the diameter of the power gas line is in parallel relationship with the frequency of detonation in structure 165, while in the case of uniform distribution of the gas in line in front of structure 281 only very few detonations

(Page 31 contd.)

occurred. This important observation led to cooperative experiments with the gas compression plant.

Operations for 1941:

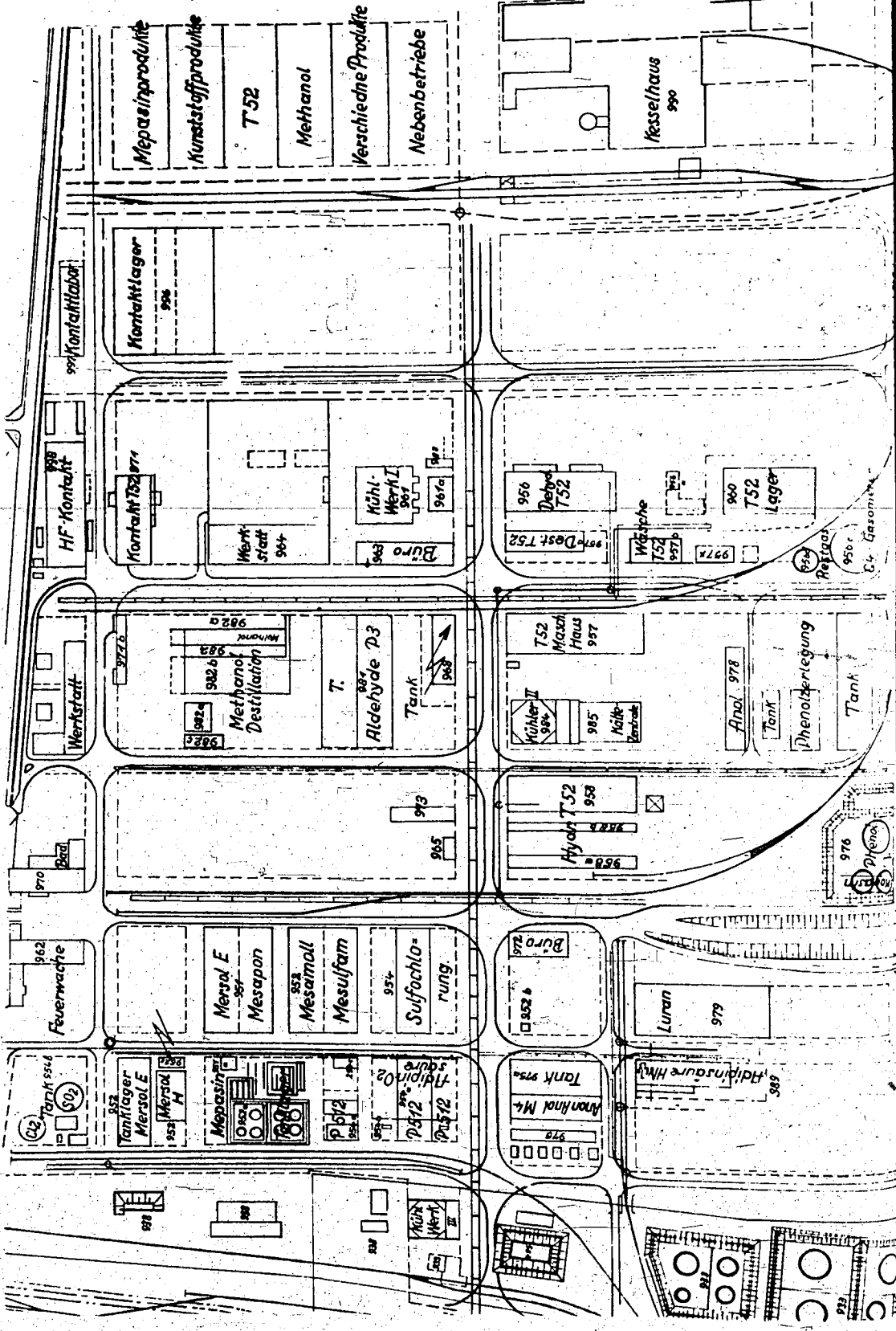
The ability to be hydrogenated, of xyleneols, is to be improved by a preliminary treatment, for example, with contacts.

The contact experiments for the pressure conversion for isobutylene expansion gas are to be carried out on a somewhat larger scale.

The power gas investigations are being continued according to an experimental program set up in cooperation with the gas compressor plant, with the object of decreasing the number of detonations in the gas machines and to determine their cause.

(Drawing Opposite Page 38)

(Gives a floor plan of the Organic Division)



Organic Division

(Managing personnel listed)

The addition of new plants and growth of old production caused us to divide the operations into two groups, this being already done in 1938. The group I, west of the older plant and including production developed in connection with the older work was under Dr. Hanisch. Dr. Korn took over the new production, including preliminary production of Mersol, phenol and plastics. The subdivision is as follows:

Group I

Production and distillation of methanol and isobutyl and alcohol refining

Amine production

Contact production

Ethylene-, SS-oil production

ET 100- and T 52-production

Aldehyde-, Fg-, carbonic acid plant

Group II

Mersol- and Mesapon-plant as well as P 512

Chlorethyl production

Plastic preproduction plant

Phenol distillation

(A paragraph discusses production increase and price regulation)

(This paragraph discusses the plant layout to the south and the west.

Some projects were worked out for countries outside Germany, such as for amine, methanol, toluol, polymerization.)

(This paragraph tells of difficulties in connection with getting material and office help and discusses cooperation necessary, gives names of managers, and discusses extent of work done by different divisions.)

(This paragraph gives figures on the number of men employed as compared to 1933.)

(This paragraph discusses the age of the employees and the quality of work done.)

(Page 38 contd.)

(This paragraph discusses technical and business employees and the amount of night work done.)

(This paragraph takes up the death of Dr. Hasselblatt and the splendid work he did. His place was taken by Dr. Sackmann).

Methanol- and Isobutyl-Plant, Laboratory

(Managing personnel listed)

Methanol Plant:

Operations in 1940:

(This paragraph says the war required increase in production and this ^{be} would/made possible by an electric circulating pump in Me 330.)

(This paragraph discusses a small fire and a large fire which made a brief shutdown necessary. Damages caused by the fire are taken up. The fire caused some loss in production.)

~~An experiment was carried out using the furnace with internal regenerator.~~
The purpose was to get a higher initial temperature in the furnace. The performance of the furnace was not satisfactory. Two experiments, to put into operation a methanol furnace together with an iron N_5 -regenerator, was not satisfactory due to poor regeneration. A fire occurred in the second experiment and therefore a repetition of the experiment was postponed. Two further chambers for methanol were built.

Operations for 1941:

Experiments on internal regeneration furnace are to be continued. A number of operation control instruments are to be specially built.

Isobutyl Plant:

Operations in 1940:

(Further increase in production, requiring more double chambers. Fourteen oven plates are available at the close of the year. Maximum furnace performance is now 62 tons per day.)

(Large fire, August 2-3, more damage in isobutyl plant than methanol plant. Production difficulties caused by fire, electrical preheating therefore not built in, water washing for expansion gas not carried out, high pressure work not completed.)

(Page 39 contd.)

Alcohol experiments were continued. A special circulation is suitable. Small scale experiments could not up until now be reproduced. A number of larger experiments in a 500 furnace were carried out. Two types of furnaces were used; a normal furnace with cold gas rings and an outside regenerator, and a tubular furnace built like the ammonia furnace. Neither was satisfactory as the temperature could not be controlled. With the cold gas furnace operating periods up to 12 days could be reached. By charging a gas mixture of nitrogen and hydrogen the dangerous period of carbon monoxide-carbonyl formation could be suppressed.

Preparation of A-gasoline from the alcohol fraction 120-180° was reduced to the 1939 level as the alcohol was used elsewhere. One gasoline furnace was used for hydrogenation of white oil.

Operations for 1941:

Alcohol experiments were continued. A special circulation combination is to be set up. A number of operation control instruments are to be installed.

(Drawing opposite Page 40)

Flow Scheme for Isobutyl-Preparation 1940.

Entatherung v. Isobutylol - De-etherizing isobutyl oil
Methanol-abtreibung aus Rohol - Driving off methanol from crude oil
Destillation v. Wasser ohaltig - Distillation of water, oil-containing
Entwässerung v. Gesamtöl - Dehydration of total oil
Propylalk.-Destillation - Propyl alcohol distillation
Isobutylalk.-Dest. - Isobutyl alcohol distillation
~~Vakuum Dest. Hexyl-Heptylalk.-Gemisch. - Vacuum distillation of hexyl-heptyl-
alcohol mixture~~
Abgas - Waste gas
Methyläther - Methyl ether
Rohol - Crude oil
Rohol entathert - De-etherized crude oil
Vorlaufmethanol - Preliminary distillate of methanol
Wasser - Water
Vorlauföl - Preliminary distillate oil
Wasser ohaltig - Water, oil-containing
Isobutyron ob. Schicht - Isobutyron upper layer
Isobutyron u. Schicht - Isobutyron lower layer
Gesamtöl - Total oil
Vorlauf, benzinhaltig - Preliminary distillate, gasoline-containing
C₁, C₂, C₃ Alkohole - C₁, C₂, C₃ alcohols
Abwasser in Kandel - Waste water in Kandel
Isobutylalk.-Frkt. - Isobutyl alcohol fraction
Rückstand \bar{u} . 140° - Residue above 140°

(Drawing opposite page 40, contd.)

Propylalkohol-Vorlauf - Preliminary distillate of propyl alcohol

Propylalkohol roh - Crude propyl alcohol

Rückst. " 108° - Residue above 180°

Isobutylalkohol-Vorlauf - Preliminary distillate of isobutyl alcohol

Isobutylalkohol roh - Crude isobutyl alcohol

Amylfraktion - Amyl fraction

Rückst. " 120° und 140° - Residue above 120° and 140°

Hexyl-Heptylalkohol-Gemisch. - Hexyl-heptyl-alcohol mixture

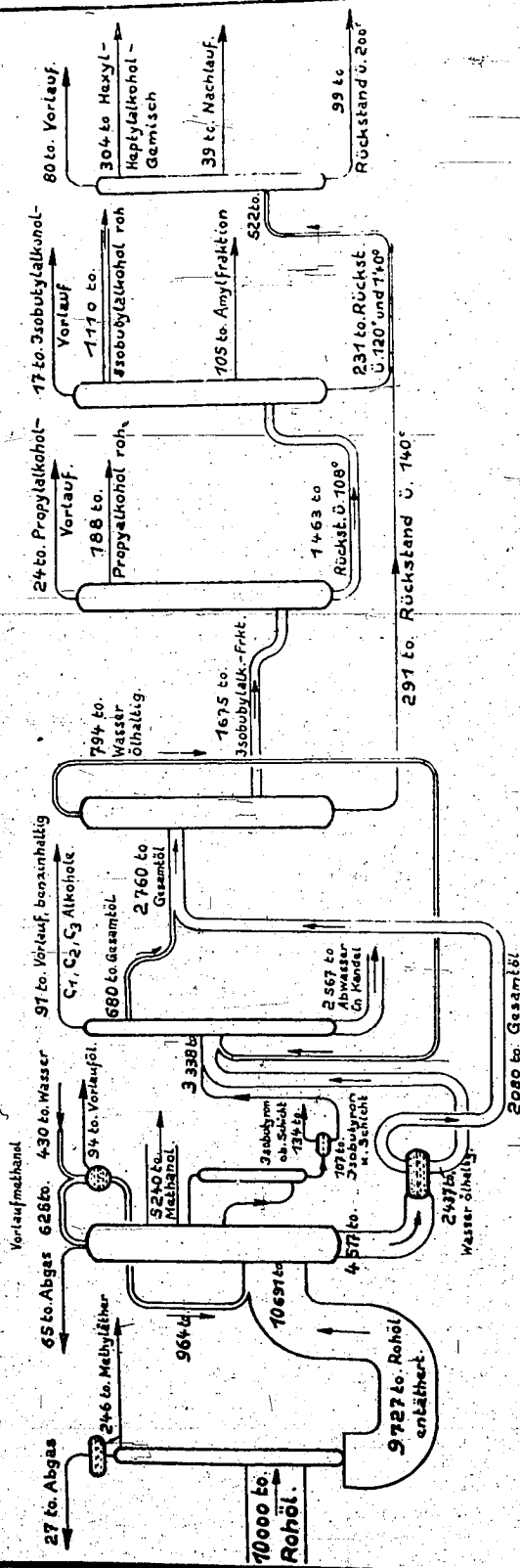
Nachlauf. - Second runnings

Vorlauf. - Preliminary distillate

Rückstand " 200° - Residue above 200°

Fließschema der Isobutyl-Aufarbeitung 1940.

Entätherung Methanol- Destillation Entwässerung Propylalk.- Isobutylalk.- Vakuum Dest.
 v. Isobutylalk. abtreibung v. Wasser v. Gesamtl. Destillation. Dest. Hexyl-Heptylalk.-
 aus Rohöl. öhhaltig. Gemisch.



Kol. 6 u. 7 Kol. 3 Kol. 1 u. 2 Kol. 5 Kol. 6 Kol. 7 Kol. 5 Kol. 5
 Me. 477 Me. 490 Me. 490 Me. 490 Me. 490 Me. 490 Me. 490 Me. 477

Freitag 40

Isobutyl Oil Distillation

(Managing personnel listed)

Operations for 1940:

A 1200-diameter column being freed in Me 417 by shutting off methanol recovery, was rebuilt contrary to original planning, for vacuum distillation of higher alcohols and was put into operation in the late spring, so that the previously used plant unit, a 2000-diameter column in Me 490 could be used for a sharper separation of normal propyl alcohol from isobutyl alcohol. A big demand for normal propyl alcohol made this change in operation essential.

Odor deficiency of normal propyl alcohol was overcome by changed operation.

Two 2000-diameter bell bottom columns were made exchangeable in Me 490, and alteration of the third is being prepared. By taking over a 2000 cubic meter tank as a storage container for isobutyl oil, the danger of a sudden shutting off of the high pressure operation in case of shutting off the column in Me 490 is avoided. As the formation of emulsion in the isobutyl residue is hardly noticeable any longer, the setting up of a new separator is not necessary at present. The waste gases of the methanol columns were collected in a gasometer since the beginning of the year and pumped into the heating gas.

Operation of the columns could be simplified by taking into use various regulators. By using cooling water after the two columns, cooling water was saved.

There is always a shortage of normal propyl alcohol and isobutyl alcohol. The greater part of amyl alcohol, hexyl alcohol, and heptyl alcohol fractions could be used in other I.G. plants. Complete utilization of the highest boiling alcohols (from C₈ on) will only be possible when the planned expansion of the carbonic acid plant has been concluded.

(Page 40 contd.)

Operations for 1941:

The gradual conversion of distillation to a performance of 15,000 tons per month of crude oil, already visualized at the beginning of the year, makes the conversion of Me 490 and technical improvements of the columns on hand essential. For obtaining amyl alcohol and the highest boiling alcohols, as well as for dehydration of isobutyron, two small bell bottom columns and a Raschig column were ordered, being attached to operating plant Me 490. Likewise some new containers will have to be procured. After increasing the number of columns in Me 490, this installation will take over exclusively the separation of the crude alcohols, thus setting free installation Me 33, in so far as the large production increase of pure alcohol is thus met. Only after the pure methanol distillation was transferred to the south of the plant could a 2500 bell bottom column in Me 33 be made available for additional distillation of the crude oil and thus relieved the present stoppage in the ability to work up the crude oil.

In connection with expanding Me 490 it was necessary to increase the height of the office building as in Me 490 the testing room was needed.

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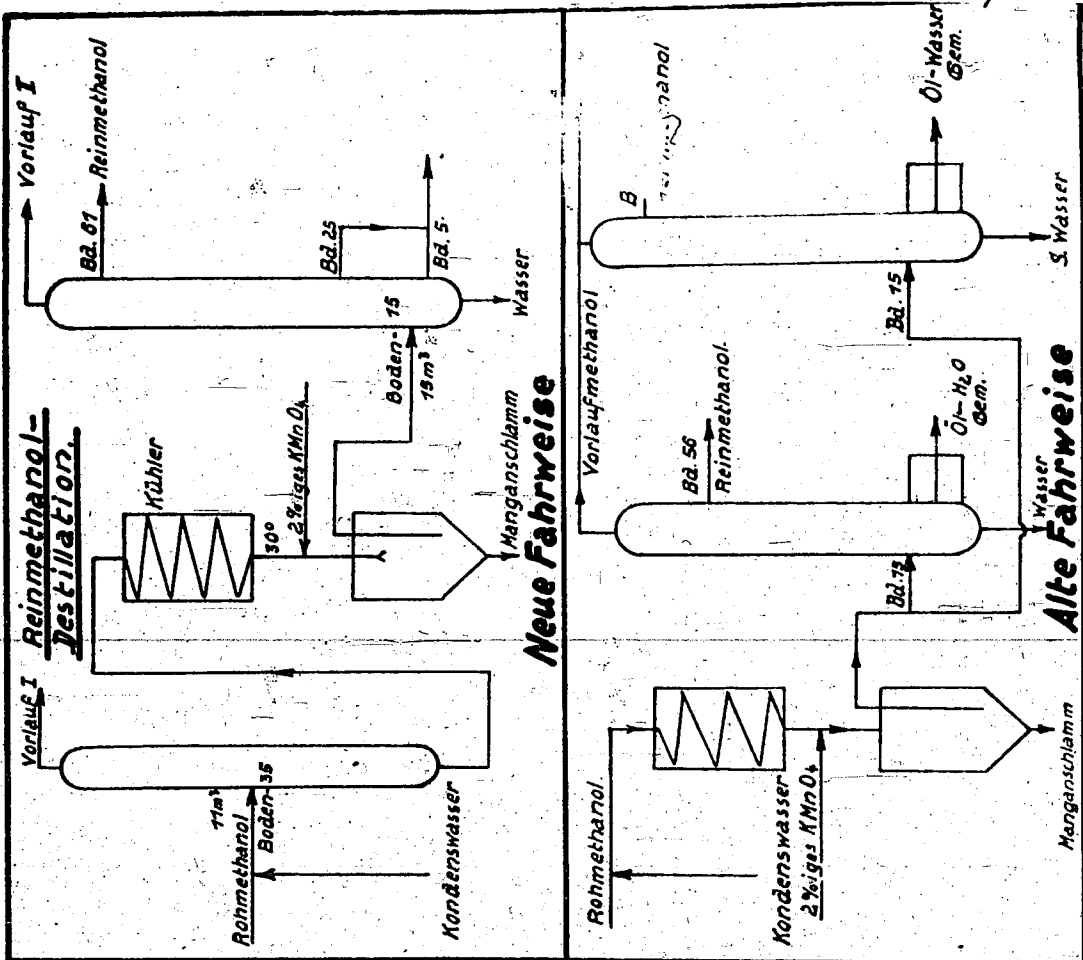
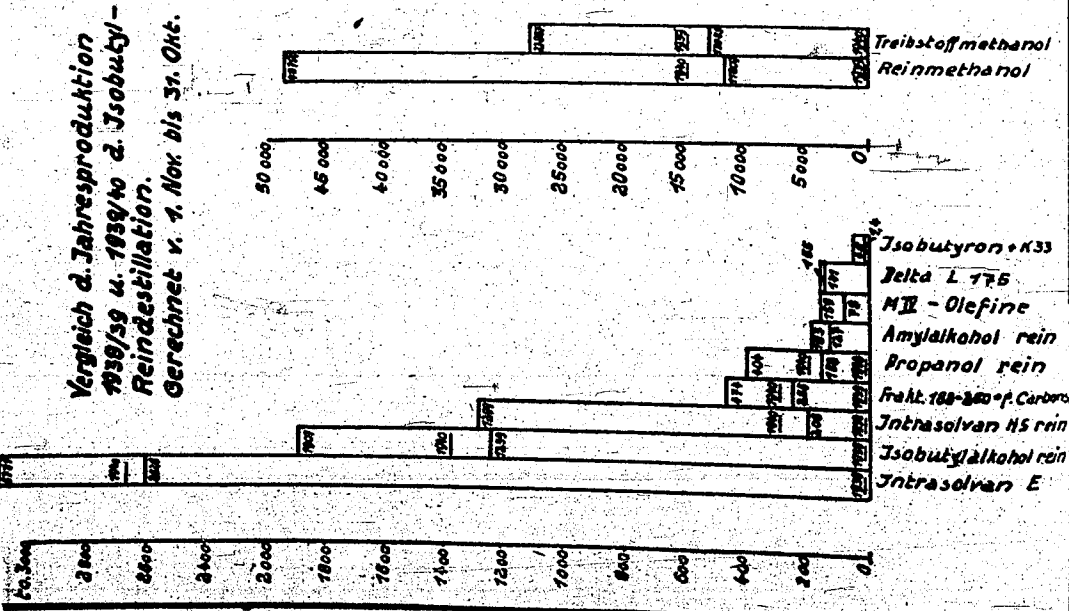
(Drawing opposite Page 41)

Comparison of the Yearly Production 1938/39 and 1939/40 of the Isobutyl Distillation Unit

Gerechnet	-	Computed
Intrasolvan E	-	Same
Isobutylalkohol rein	-	Pure isobutyl alcohol
Intrasolvan ES rein	-	Same
Frakt. 180-250°F Carbons	-	180°-250° fraction for carbons
Propanol rein	-	Pure propanol
Amylalkohol rein	-	Pure amyl alcohol
M IV Olefine	-	M IV olefins
Delta L 175	-	Same

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Vergleich d. Jahresproduktion
1939/40 u. 1938/39 d. Isobutyl-
Reindestillation.
Gerechnet v. 1. Nov. bis 31. Okt.



facing 41

Methanol and Deetherization 1940

I. Methanol deetherization

The sales of pure ether were similar to those in the previous year so that they could be covered completely out of recoveries from crude methanol.

A very large part of the crude ether had to be diverted to plant gas or to the high pressure synthesis of isobutyl oil depending upon the plant situation. Therefore, the construction of a pressure wash plant for the purification of isobutyl ether was not necessary. By constructing a larger vaporizer it was possible to increase the capacity of a 700 mm. diameter ether column 25%.

By exchange of information with Hoechst it was found that an even sharper fractionation of the ether permitted the production of a special quality which yielded a nearly water white dimethyl sulfate.

Condenser leaks temporarily reduced the output of two columns.

II. Isobutyl oil deetherization:

A pressure column formerly used for methanol recovery was used for deetherization of isobutyl oil after revisions which resulted in an ether free oil for nearly all of 1940. The ether was used primarily in plant gas because its light hydrocarbon content did not make its use in the high pressure furnaces desirable. The prior removal of the ether facilitates the methanol recovery from crude oil especially at high column load. One column was out of service temporarily for repairs and for a period replaced a defective methanol deetherization column.

III. Tiglic Aldehyde (C₅H₈O).

The new plant for the production of Tiglic aldehyde from propionic aldehyde and acetaldehyde was started up and included into the Me 417 plant.

(Page 41 contd.)

After overcoming start-up troubles the plant operated at the guaranteed throughput of 35 tons per month of crude tiglic aldehyde (60%).

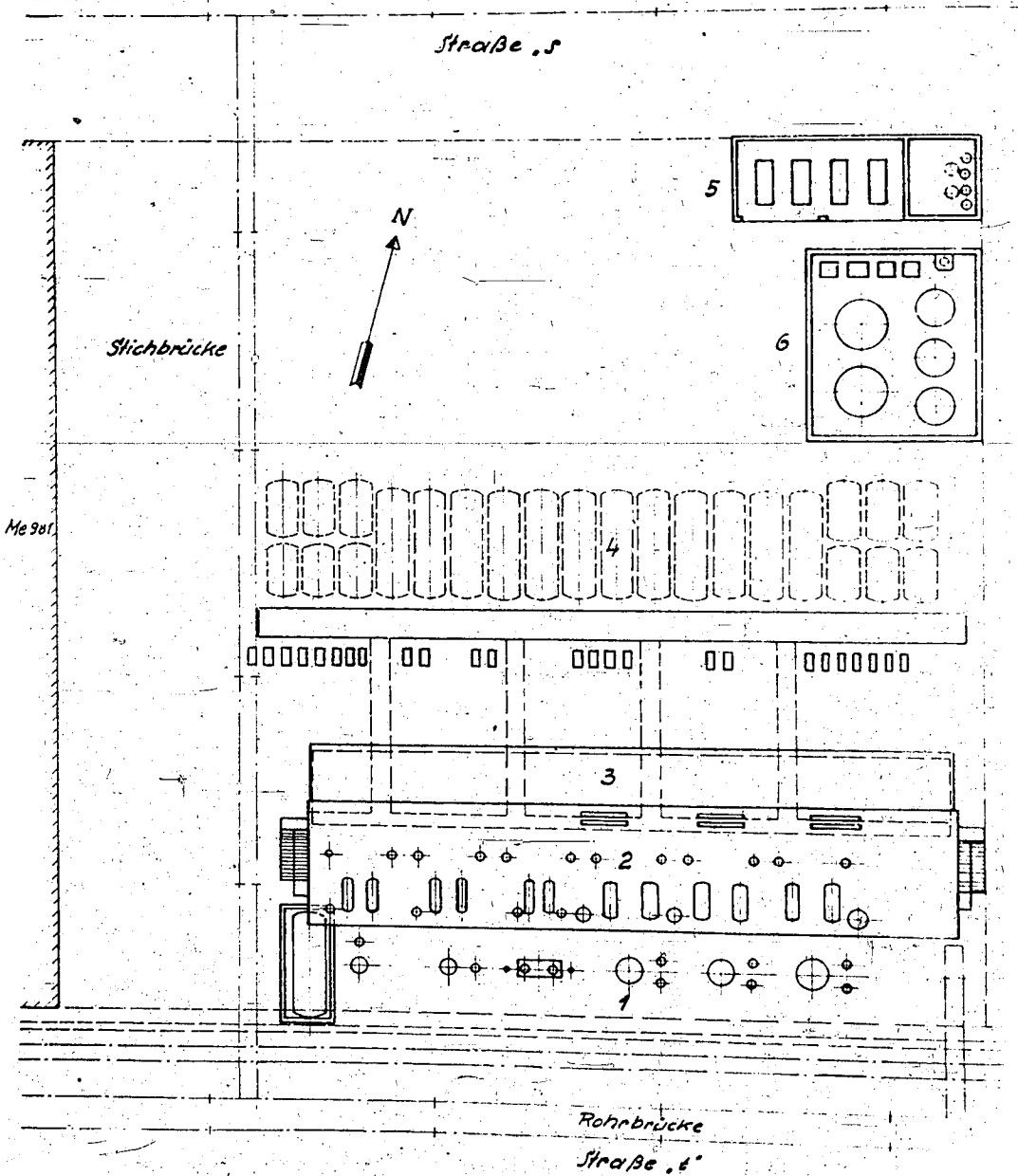
Plans for 1941:

The large crude methanol deetherization column is expected to be erected early in 1941. The isobutyl oil deetherization column throughput has to be increased by the addition of larger vaporizers.

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Rein-Methanol-Destillation Me 982
Maßstab 1:500



- 1 Destillationskolonnen
- 2 Kondensatoren u. Kühler
- 3 Bedienungsraum

- 4 Tanklager
- 5 Permanganatanlage
- 6 Absatzbehälter

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(Drawing opposite Page 42)

Pure Methanol Distillation Me 982

Masstab	-	Scale
Strasse	-	Street
Stichbrücke ["]	-	?
Rohrbrücke ["]	-	Pipe alley
Destillations Kolonnen	-	Fractionating columns
Kondensatoren u. Kähler ["]	-	Condensers and coolers
Bedienungsraum	-	Control house
Tanklager	-	Tank farm
Permanganatanlage	-	Permanganate plant
Absatzbehälter ["]	-	Product storage

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(Page 42)

Pure Methanol and Isobutyl Distillation
Propyl ether and M₄ Production

Pure Methanol Distillation:

Work in 1940:

The pure methanol production was quadrupled because of the war demands. At the same time the product purity was improved considerably which was found necessary because Ludwigshafen had difficulties with the quality. This improvement was achieved on the same columns by a change in flow without decreasing capacity appreciably. At the same time the use of permanganate, which was available in limited quantities only, was reduced to half.

Work for 1941:

Because of the increasing demand (particularly for storage purposes) a new distillation plant with a slightly higher capacity than the present is to be started up early in 1941. The methanol light tops which are presently disposed of as water free methanol or as charge to the isobutyl plant, will be processed for pure methanol production. Experiments are in progress to increase the plant capacity by decreasing water addition.

Isobutyl-Distillation.

Work in 1940:

The production of pure products from isobutyl oil has increased steadily during the war. The production of pure HS for palatinol and of "intrasolvan F" for solvent increased particularly. The production of propanol was below requirements part of the time. The portions of isobutyl oil which were not useful and which were converted to gasoline have decreased considerably and it is hoped that their degradation can be eliminated completely. Two new products were marketed: Solvent K 33 (a broader butyrene fraction) and

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(Page 42 contd.)

Dioprol (diisopropyl carbinol, $(C_3H_7)_2CHOH$). Particularly dioprol, which is recovered from the light ends of the HS fraction and by hydrogenation of the isobutyrene fraction, is in demand and all production was sold. Besides, the olefin produced from it can be dimerized to the desirable polymer M VII for the production of "Igepal."

Progress was made in distillation so that pure amyl alcohol and propyl alcohol are now produced continuously. The processing of the ketone fraction was also simplified. By means of a preliminary water wash in Me 490 the water and soluble alcohol content were reduced so much that a separate water removal distillation could be eliminated. Distillation was made continuous.

Work for 1941:

It is planned to fractionate pure HS continuously in a new 1000 mm bubble tray tower. The M IV production shall be increased to 80 tons per month for the time being. A new plant will be built for this. Columns C and D in Me 33 shall be doubled in capacity by the addition of a larger vaporizer. After starting up the new distillation plants in Me 982 the distillation capacity for pure products will be nearly doubled which in turn will result in a large reserve capacity for the distillation of crude isobutyl oil in Me 33. Experiments are under way to convert the ketone fraction, of which 70% has been going to gasoline, to valuable products (Dioprol and M-VII olefins besides K 33).

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(Page 43)

Amyl Alcohol Production

(Managing personnel listed)

Work in 1940:

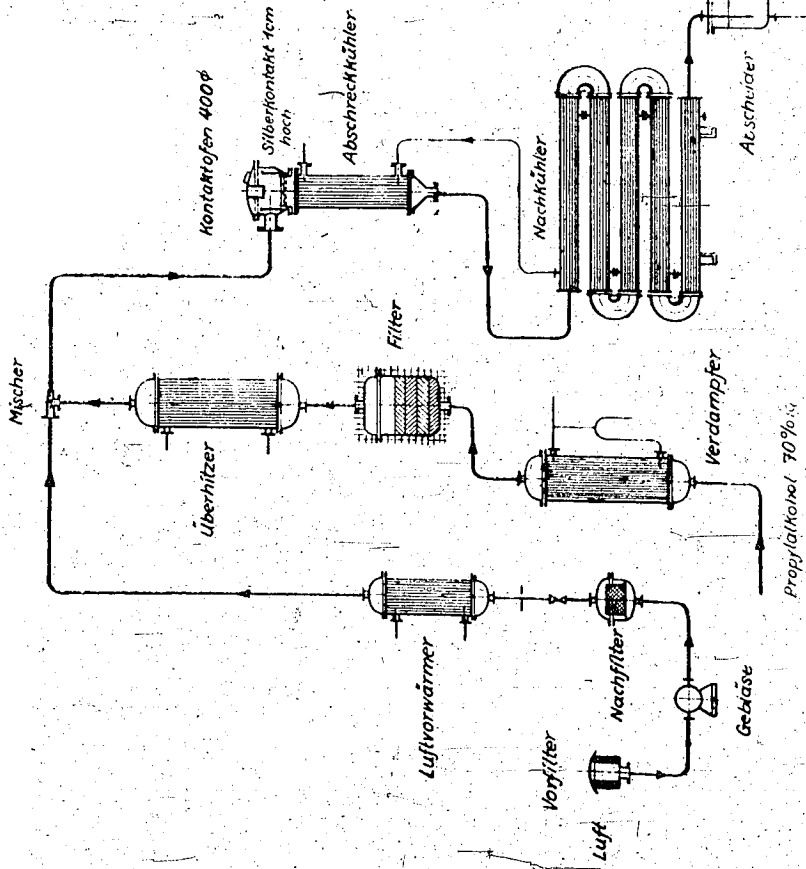
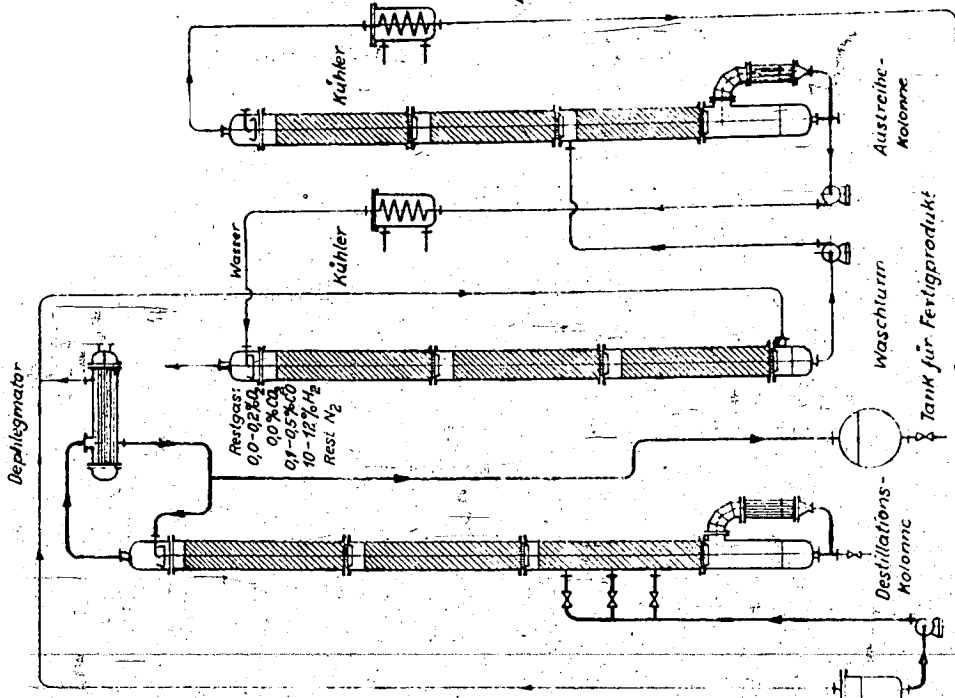
The production of tiglic aldehyde ($\text{CH}_3\text{CH}_2\text{C}(\text{CH}_3)\text{CHO}$) by condensation of acetaldehyde and propionic aldehyde was carried on without excessive difficulties. Lack of phosphoric acid required the use of dilute sulfuric acid for neutralization; the danger of corrosion in the vaporizer coil will be eliminated by the substitution of a lead coil. The hydrogenation of tiglic aldehyde to amyl alcohol at a pressure of 10-15 atm. in Me 13 was carried out without difficulty but low reactor output. It had to be discontinued when the circulating pumps had to be transferred to the Me 12 Kogasin hydrogenation plant.

Hydrogenation of tiglic aldehyde at atmospheric pressure over nickel- Al_2O_3 and NiCO_3 on pumice in the commercial plant Me 924, which uses former acetaldehyde hydrogenation reactors, was not as successful as pilot plant results. Conversion was incomplete. The cause was partly the disintegration of the nickel-pumice catalyst which plugged the reactors and partly too low a reduction temperature. Since there is a possibility of carrying out the tiglic aldehyde hydrogenation in the phenol hydrogenation reactors of the ammonia plant Me 11, no further experiments will be carried out in Me 924. The fractionation of the hydrogenated product was temporarily done at Griesheim because of lack of equipment. However, it will again be carried out at Leuna after the completion of Me 490 and Me 33.

Work for 1941:

The separation of the tiglic aldehyde from the unreacted acetaldehyde and propionic aldehyde in column IV must be improved by the addition of a subcooler and a pressure controller. The adequacy of the reactors in Me 11 for the tiglic aldehyde hydrogenation must be tested.

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Schema der Propylaldehydanlage nach dem Oxydationsverfahren

Ammoniakwerk Merseburg G.m.b.H.

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(Drawing opposite page 44)

Flow Sheet of the Propionic Aldehyde Plant by the Oxidation process

Luft	-	Air
Vorfilter	-	Pre-filter
Gebälse	-	Blower
Nachfilter	-	After-filter
Luftvorwärmer	-	Air pre-heater
Verdampfer	-	Vaporizer
Überhitzer	-	Superheater
Mischer	-	Mixer
Kontaktofen	-	Reactor
Silberkontakt	-	Silver catalyst
Abschreckkühler	-	Quench cooler
Nachkühler	-	Aftercooler
Abscheider	-	Separator
Destillations-Kolonne	-	Distillation column
Dephlegmator	-	Partial condenser
Restgas	-	Tail gas
Wasser	-	Water
Waschturm	-	Wash tower
Austreibekolonne	-	Stripping column
Tank für Fertigprodukt	-	Finished product tank

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(Page 44)

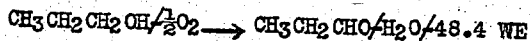
Aldehyde Production

(Managing personnel listed)

Work in 1940:

The preparation of propionic aldehyde by the dehydrogenation of propyl alcohol in the new annular space (ringraum) reactor in which heat can be added better is still unsatisfactory. At the high temperatures (about 420°C.) which are necessary for dehydrogenation, both the preheater and the reactor coke up after a short period. The poor heat conductivity must be offset by higher wall temperatures which in turn increase coke formation. After 4-6 weeks the preheater and reactor were plugged with coke. The difficulties with materials of construction have not been solved yet.

The new pilot plant for the production of propionic aldehyde by the oxidation process was started. According to the process, which was developed last year, propionic aldehyde is produced in good yields by passing aqueous propyl alcohol-air mixtures over a silver catalyst which is maintained at 450-500° by the oxidation of hydrogen.



Less than 1% of the propyl alcohol is burned to carbon dioxide or carbon monoxide. The aldehyde yield was 85% or over; several test values have been obtained which were over 90%. On starting the plant considerable operating difficulties occurred, particularly because of the uneven vaporization. On the basis of this a tubular vaporizer was developed which vaporizes the injected propyl alcohol water mixture (70%) uniformly. Since pure propyl alcohol was not available for aldehyde production, because of its use as a solvent, the oxidation of propyl alcohol was carried out with the impure product. Under these conditions the silver catalyst had a life of from 2-3 days. By the inclusion of filters with a layer of activated charcoal, the life of the

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(Page 44 contd.)

catalyst could be increased to 22 days when using impure propyl alcohol. The silver catalyst was improved.

The aldehyde loss in the tail gas was reduced from 6% to 1.5% by the inclusion of a wash tower. The best absorption medium is water which is continuously concentrated to 5% and then stripped.

Difficulties in the distillation are caused by propionic acid and formic acid (3% and 1% of the propyl alcohol respectively) which appear as by-products. Since at the present time no corrosion resistant materials are available, an attempt is being made to neutralize the acid with diluted caustic, even though this results in a reduction of the aldehyde yield.

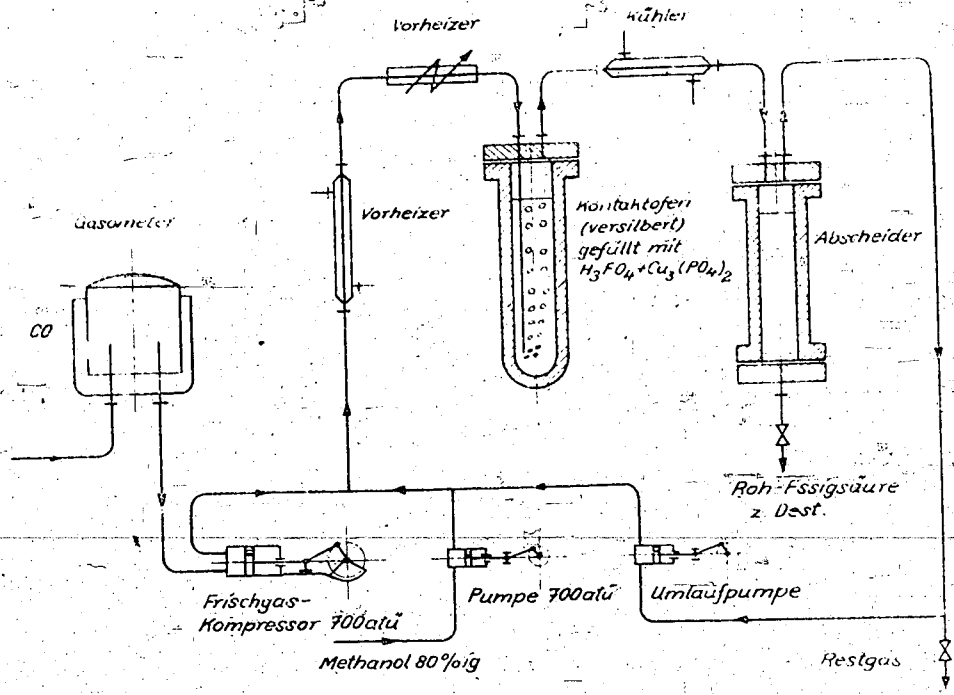
Work for 1941:

Improvement of the oxidation process, solution of the corrosion difficulties by neutralization.

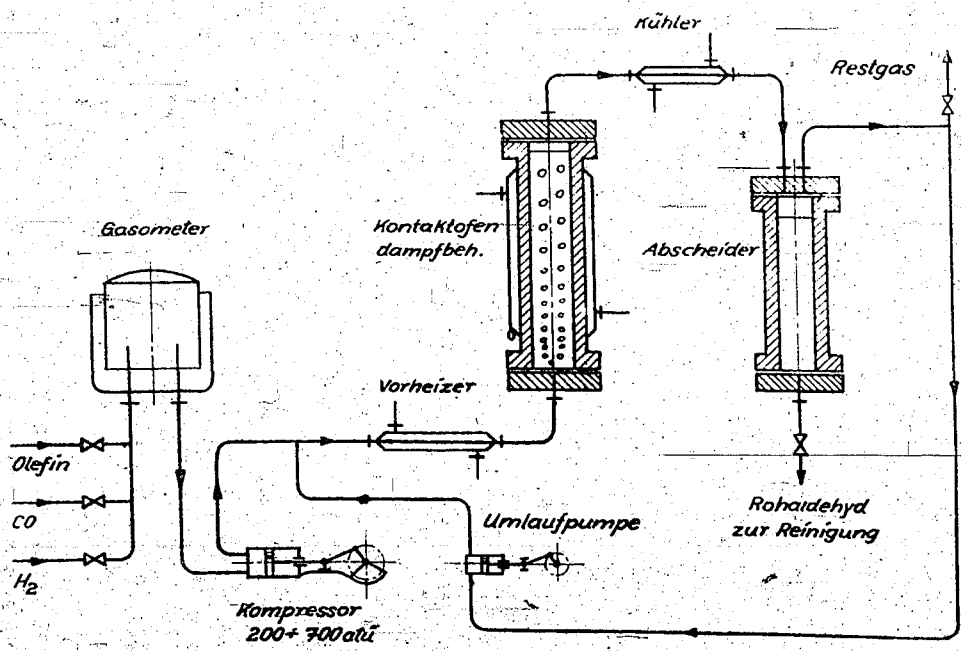
The construction of a plant for the production of 200 T/M of propionic aldehyde in Me 981 shall be completed in April 1941.

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Ammoniakherstellung ... Schema der Essigsäure-Anlage 700atü
 $CH_3OH + CO \rightarrow CH_3COOH$



Schema der Oxo-Anlage zur Herstellung von Propionaldehyd und Isobutylaldehyd aus Äthylen bzw. Propylen



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(Drawing opposite page 45)

Flow Sheet of the Acetic Acid Plant
700 atmospheres

Gascmeter	-	Gas-holder
Frischgaskompressor	-	Fresh gas compressor
Vorheizer	-	Preheater
Kühler	-	Cooler
Abscheider	-	Separator
Kontaktoven	-	Reactor
Roh-Essigsäure z. Dest.	-	Crude acetic acid to distillation
Umlaufpumpe	-	Circulating pump
Restgas	-	Tail gas

Flow Sheet for the Oxo Plant for the Preparation of
Propionic Aldehyde and Isobutyric Aldehyde from
Ethylene and Propylene Respectively

Rohaldehyd zur Reinigung	-	Crude aldehyde to purification plant
Kontaktoven dampfbeh	-	Steam-jacketed reactor

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(Page 45)

Technikum Me 13a and Very High Pressure Experiments Me 238

(Managing personnel listed)

Work in 1940:

Acetic acid experiments

The preparation of acetic acid from methanol and carbon monoxide is carried out best at 700 atmospheres with liquid phosphoric acid and 2% copper phosphate at 300-320°C. The yields of 50-60% of theoretical which have been obtained so far and which can be increased further by the recovery of volatile side products such as dimethyl ether and methyl acetate, indicate that the process is economical. Somewhat more than one gram of acetic acid is obtained from 1 gram of methanol. Experiments were made both in an autoclave and in a continuous apparatus. In spite of the use of silver, the corrosion problem has not been solved adequately. A unit for the production of 100 kg. acetic acid per day is nearly completed.

Alcohol experiments

In the synthesis of ethyl alcohol, the effect of pressure, temperature, rate, and gas composition on the synthesis of various alcohols over the present catalyst (a five-component catalyst), was investigated. Increasing pressure favors the formation of methanol which becomes the chief product at 700 atmospheres. For the synthesis of ethanol the best pressures are between 150-250 atmospheres. Lower pressures increase the rate of methane formation very much. The best temperatures lie between 370-420°C. Lower temperatures favor the formation of methanol and higher ones, the formation of methane. Increasing the partial pressure of carbon monoxide increases ethanol formation and at the same time the production of propyl alcohol is increased. With 40-60% carbon monoxide, propyl alcohol can be formed preferentially but the catalyst life has been low so far.

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Oxo experiments

The preparation of aldehydes from olefins and carbon monoxide-hydrogen is possible with cobalt catalyst, copper cobalt catalyst with only little cobalt, and with cobalt carbonyl. The preparation of propionic aldehyde and isobutyric aldehyde from ethylene and propylene respectively, was investigated. The process appears promising particularly with pure low molecular weight olefins. A pilot plant for the production of propionic aldehyde from ethylene is being started up at the present time.

Nitration of aliphatic hydrocarbons

Good yields have been obtained in the nitration of ethane, propane and butane which is carried out at atmospheric pressure and at 380-420°C. in the vapor phase. These nitro-paraffins of medium boiling points are good solvents and also important starting materials for further chemical processing. With mineral acids the corresponding carboxylic acids and hydroxylamine salts are obtained from 1-nitro-paraffins in 90% yields. In the condensation of 1-nitro propane with formaldehyde, dimethylol-nitropropane is obtained in over 95% yields. Experiments for the use of these products are in progress.

Preparation of aldehydes in ketones

Good yields of the following compounds can be obtained by oxidation of the corresponding alcohol with air over a silver catalyst; acetaldehyde, propionic aldehyde, isobutyric aldehyde, acetone, cyclohexanone, methyl-cyclohexanone, acrolein, and others. The preparation of formaldehyde from crude methanol appears to be possible and economical by the use of mercury lamps and pre-filters.

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(Page 45 contd.)

Work for 1941:

Continuation of experimental work

Construction of a 300 T/M plant for the production of acetic acid from methanol and carbon monoxide. Development of a better catalyst for the preparation of aldehydes from olefins plus carbon monoxide plus hydrogen.

Construction of a larger apparatus for the preparation of nitro-paraffins.

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Amine Plant Me 107 and 13

(Managing personnel listed)

Work in 1940:

In the beginning of this year several experiments were made to obtain a good separation of the olefin products with the plate pressure column (flachbodendruckkolonne) which was delivered by Heckmann and erected in Me 107a. The column alone was, however, not sufficient. It was shown that it was necessary first to separate water and methanol from amine and ammonia in two existing columns and then to separate the trimethylamine-ammonia azeotrope in the plate column, from ammonia in the upper trays while mono- and dimethylamine remain in the bottom. These were transferred from there to another column for further separation. By the use of the plate column the former ammonia column was made available so that the capacity of the plant was increased considerably. It was shown several times that the ammonia in the reactor mix was not sufficient to separate all trimethylamine as the azeotrope; therefore, a perforated pipe was installed in the still of the mono- dimethylamine column through which ammonia was injected in the liquid phase by means of a pump. The large reactor I serves for the production of methylamine while the old reactor II, and the small experimental reactor III, were also used to split monomethylamine, for the preparation of high purity trimethylamine, and for investigation of new catalyts. For the purpose of designing a new amine plant several new catalyts had to be investigated on a commercial scale. The experiments were carried out in two directions, for the purpose of lowering the reaction temperature and for the change of the mono-di-tri-ratio in favor of the mono and di by maintaining only partial conversion. A temperature which was 2-3 MV at the same thruput and the same yield was possible.

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with the aid of a catalyst which was first obtained from Ludwigshafen and was later manufactured in the local catalyst plant in a different way. It was further possible to decrease the production of the tri compound by partial conversion.

In chamber 3 Me 13 the preparation of stearic amine has been further developed so that a saleable product is now obtained.

Since the process which has been used for the preparation of diisobutylamine in the last few years by direct conversion of isobutanol with ammonia yielded only 50%, this process was discontinued this year and replaced by one with aldehyde as a starting material. For this changed operating procedure the stearic amine apparatus could be used. The yields were 75-80% based on crude products.

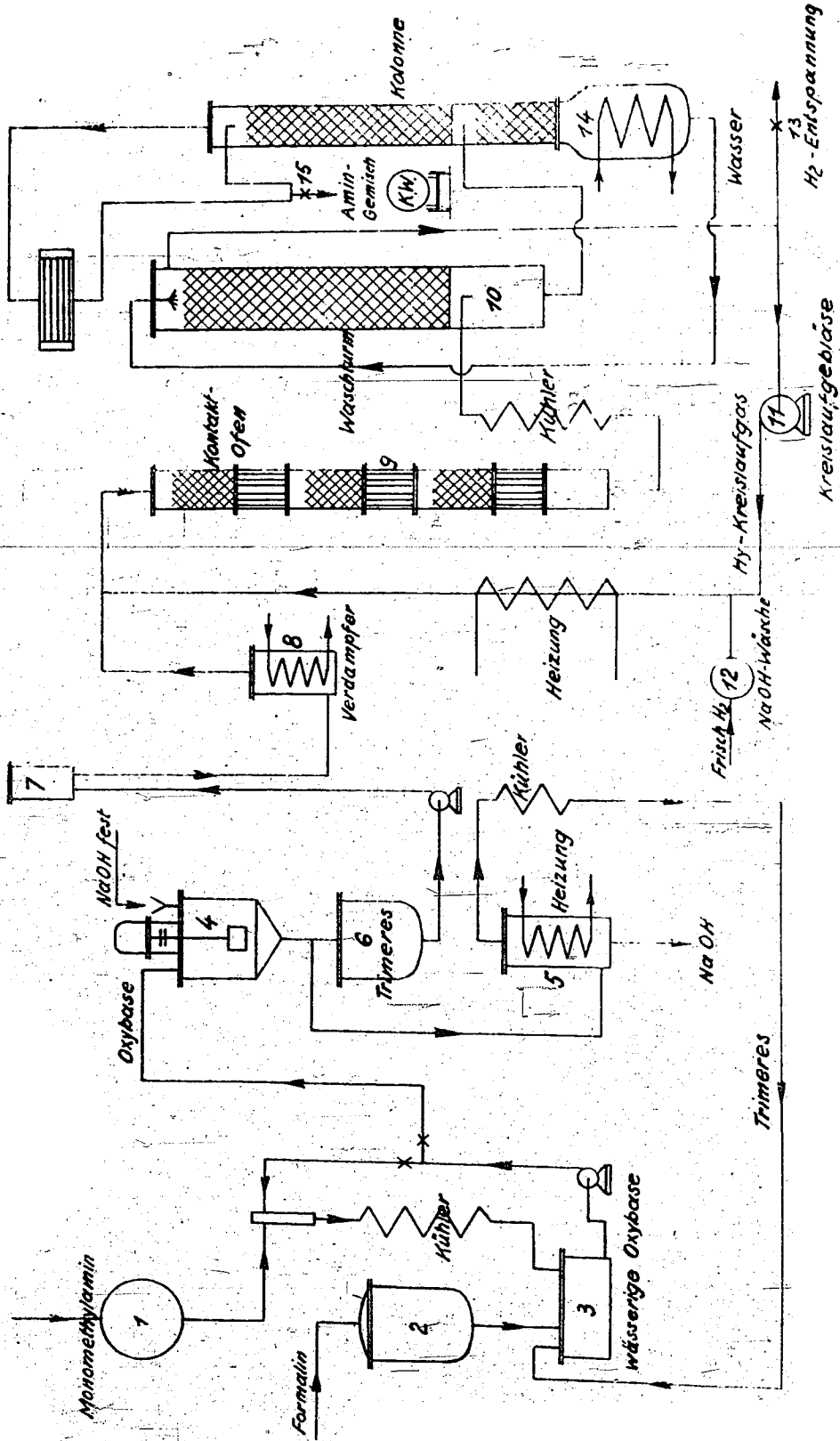
Work for 1941:

Lately the requirements for dimethylamine have increased, so that not sufficient use was found for the monomethylamine by-product. Therefore a process has been worked out in the laboratory Me 127 and a plant has been built in Me 924f to convert monomethylamine to dimethylamine with the aid of formaldehyde. (See Organic Laboratory Me 127 and Dimethylamin Production Me 924f) The crude solution is to be fractionated in Me 107 to pure dimethylamine, so that its production will be considerably increased.

In a newly developed reactor, stearic amine is to be produced with the aid of fine grained catalyst.

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Dimethylamin - Fabrikation



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(Drawing opposite Page 47)

Dimethylamine Plant

Formalin	-	40% formaldehyde in water solution
Kühler	-	Cooler
Wässrige Oxybase	-	Aqueous oxybase
NaOH fest	-	Solid NaOH
Heizung	-	Heater
Verdampfer	-	Vaporizer
Frisch H ₂	-	Fresh H ₂
NaOH-Wäsche	-	Caustic wash
Kontaktoven	-	Reactor
Hy-Kreislaufgas	-	Hydrogen recycle gas
Kreislaufgebläse	-	Recycle compressor
Waschturm	-	Wash tower
Amin Gemisch	-	Amine mixture
K.W.	-	Hydrocarbon
Kolonne	-	Column
Wasser	-	Water
H ₂ -Entspannung	-	H ₂ pressure reduction

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(Page 47)

Dimethylamine Plant Me 924f

(Managing personnel listed)

Work in 1940:

On the basis of a process worked out by laboratory Me 127 for the production of dimethylamine from monomethylamine and formaldehyde, a plant was built in Me 924 during the last half of 1940 with a capacity of 300 tons/year. It also serves as a model for the construction of an outside commercial plant for the production of 4000 tons/year of dimethylamine.

The plant was completed in 1940. On starting, certain changes were found necessary so that operation will commence in 1941.

The process flow is as follows (see drawing):

Monomethylamine from storage (1) and formaldehyde from storage (2) are mixed and cooled in vessel (3). The oxybase produced is dehydrated in the cooled mixer (4) by the addition of solid caustic soda. The aqueous caustic is removed as the lower layer in vessel (5) and separated there from trimers by heating, which are recycled to vessel (3). The trimers recovered from the oxybase by treatment with caustic is collected in vessel (6) and from there charged to the vaporizer (8) by way of the charge tank (7). Together with recycle hydrogen the vaporized trimers pass through the reactor (9) where the trimer is dissociated to the monomer which in turn is hydrogenated to a mono-di-trimethylamine mixture containing about 70-90% dimethylamine. This mixture is dissolved in water in the washtower (1) from where the unused hydrogen is recycled by way of blower (11). A small amount of makeup hydrogen is added to the recycle gas from which CO₂ is removed completely by means of the caustic wash 12.

Work for 1941:

Since it was found in laboratory Me 127 that the aqueous oxybase can be hydrogenated directly to dimethylamine, the plant is to be revised later on

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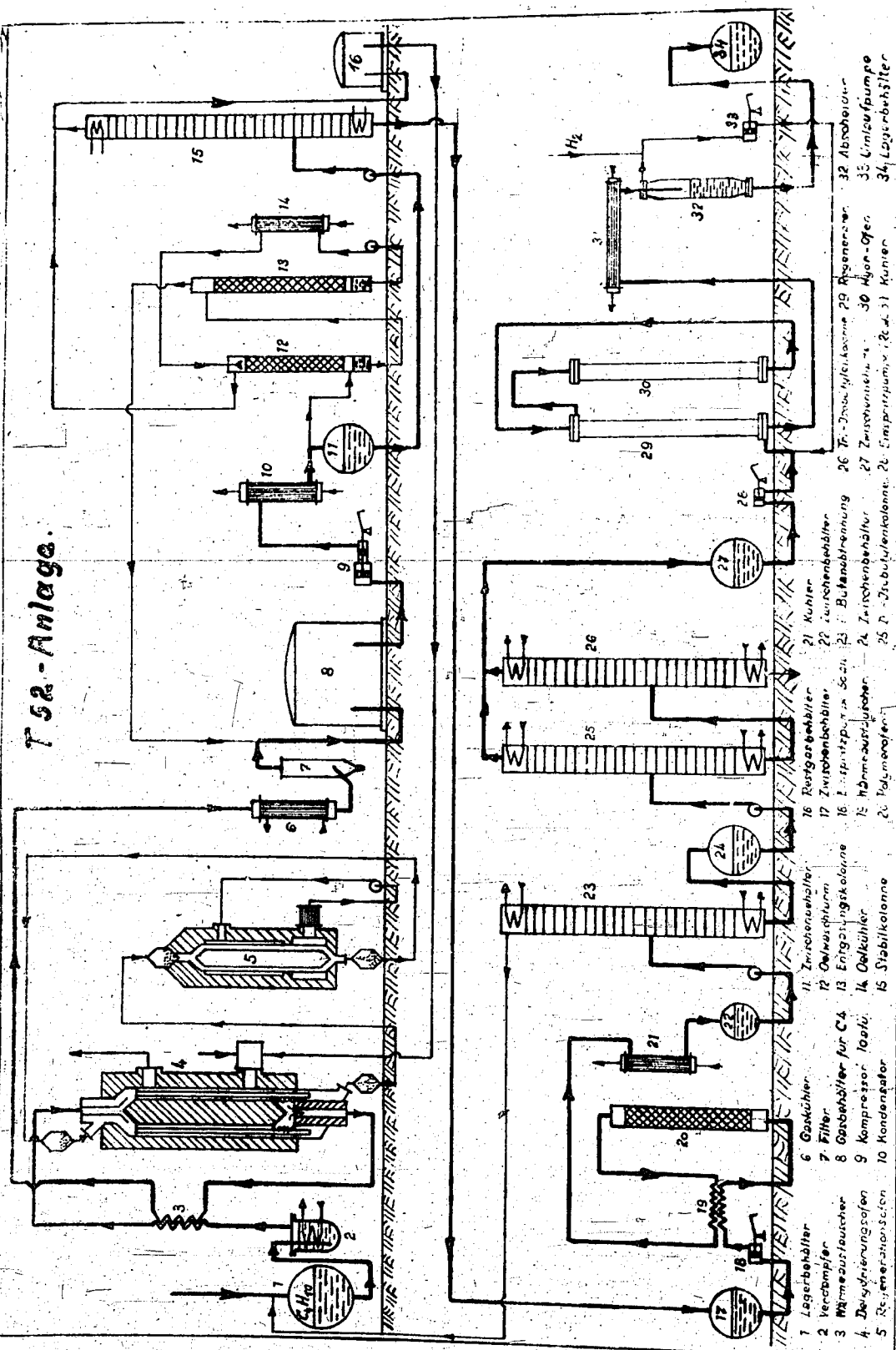
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(Page 47 contd.)

accordingly. By this simplification a considerable reduction of cost of the finished product can be realized. Before revising the plant its operability of the original process is to be tested first for several months.

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T 52 - Anlage.



1 Lagerbehälter
 2 Verdampfer
 3 Wärmetauscher
 4 Desydrationssofen
 5 Reagenzienstufen

6 Gaskühler
 7 Filter
 8 Gasbehälter für C₄
 9 Kompressor laub.
 10 Kondensator

11 Zwischenbehälter
 12 Ölwaschkolonne
 13 Eingestüblte Kolonne
 14 Gaskühler
 15 Stabkolonne

16 Restgasbehälter
 17 Zwischenbehälter
 18 Einspritzpumpe
 19 Hämmerauslöser
 20 Polymereisen

21 Kuehler
 22 Zwischenbehälter
 23 Butanbehälter
 24 Zwischenbehälter
 25 P. - Druckkolonne

26 Kuehler
 27 Zwischenbehälter
 28 Zwischenbehälter
 29 Agensenzwe.
 30 Hoch-Ofen

31 Kuehler
 32 Umwälzpumpe
 33 Lagerbehälter
 34 Lagerbehälter

35 Abscheider

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(Drawing opposite Page 48)

T 52 Plant

1. Storage tank
2. Vaporizer
3. Heat exchanger
4. Dehydrogenation furnace
5. Regenerating furnace
6. Gas cooler
7. Filter
8. Gas tank for C₄
9. Compressor 10 atm.
10. Condenser
11. Intermediate tank
12. Oil scrubbing column
13. De-gassing column
14. Oil cooler
15. Stabilization column
16. Residual gas tank
17. Intermediate tank
18. Injection pump 50 atm.
19. Heat exchanger
20. Polymerization furnace
21. Cooler
22. Intermediate tank
23. Isobutane separator
24. Intermediate tank
25. Diisobutylene column
26. Triisobutylene column
27. Intermediate tank
28. Injection pump 220 atm.
29. Regenerator
30. Hydrogenation furnace
31. Cooler
32. Separator
33. Circulating pump
34. Storage tank

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TRANSLATION

A M M O N I A W O R K S M E R S E B U R G

ANNUAL REPORT
for
1940

(Page 48)

The production of iso-octane (ET110)

Operation:

Messrs. Dr. Fischer
Dr. Jagemann
Dr. Kahr

Repairs:

Messrs. von Lom
Wenk
Gruettner

Activities in 1940.

Dehydration (chemical) of isobutyl alcohol

The gas-heated dehydration furnace built in the operation year 1939 packed with a catalyst charge of 400 l. (liters) was started and has come up to expectations as regards throughput capacity. It was able to stand a catalyst load 3.75 times the normal load with a 99% conversion. The electrically heated furnaces no longer needed for splitting off water served as standbys for the gas-heated furnaces. In a 4-month plant run it was found that the crude isobutylene can be used as such for the polymerization without distillation, and without impairing the quality of the ET110. By an additional spray injection of water behind the furnaces it is possible to scrub the non-reacted isobutyl alcohol out of the reaction gases and thus to keep the isobutyl alcohol content of the crude isobutylene at as low a figure as possible. The wash water together with the reaction water is worked up for the recovery of isobutyl alcohol which amounts to about 0.5-1.0% of the processed amount of alcohol. Due to the direct utilization of the crude isobutylene 25 t.p.m. of higher hydrocarbons are no longer available.

The bell plate column I heretofore used for distillation purposes thus becomes available for other purposes. Smaller operational activities with the equipment on hand were as follows: The splitting-off (chemical dehydration) of water from iso-amyl alcohol and secondary butanol, furthermore the dehydrogenation of sec. butanol and cyclohexanol, each in batches of several tons (metric).

Polymerization of isobutylene.

The active life of the A-carbon catalyst containing 25% of H_3PO_4 was 5 months in the case of expert handling, as compared with 3-4 months in the preceding year. More extensive plant runs showed that isobutane polymerizes together with isobutylene to the extent of 5% of the latter. Thus there also takes place alkylation on fixed catalysts to a certain extent. However, the O.Z. (Oktananzahl ?, octane number ?) of the alcohol (?) does not reach the corresponding figure for the pure polymerizate. The production capacity of the polymerization plant is at present 8000-9000 t.p.y. of ET110.

Columns.

The column II in which the unconverted isobutylene is distilled from the polymerizate has been the bottleneck of the plant up to the present, corresponding to about 7000 t.p.y of ET 110. Due to the fact that column I has become available and can be used conjointly with II the distillation equipment is ample for a production of 10,000-12,000 t.p.y. of ET 110. In the course of erection are one 1 cu.m.-still for the distillation of specialties and one 800 mm.-Raschig column for the distillation of the alcohol-containing water.

Tri-cracking (?) ("Tri-Spaltung").

There was no need of restarting the tri-cracking plant as the Air Force ("Luftwaffe") continues to accept the Tri-containing ET 110.

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Hydrogenation of diisobutylene.

The life of catalyst No. 5076 is three years up to the present; any recession of its activity has not been observed up to now. The output capacity ranges from 10,000 to 12,000 t.p.y. of ET 110.

Losses.

Due to the direct utilization of the crude isobutylene for the polymerization the overall losses have dropped from 7 to 5%, and as a result thereof only 1.39 ton of isopropyl alcohol are consumed per ton of ET 110 instead of the 1.42 ton as heretofore.

Production.

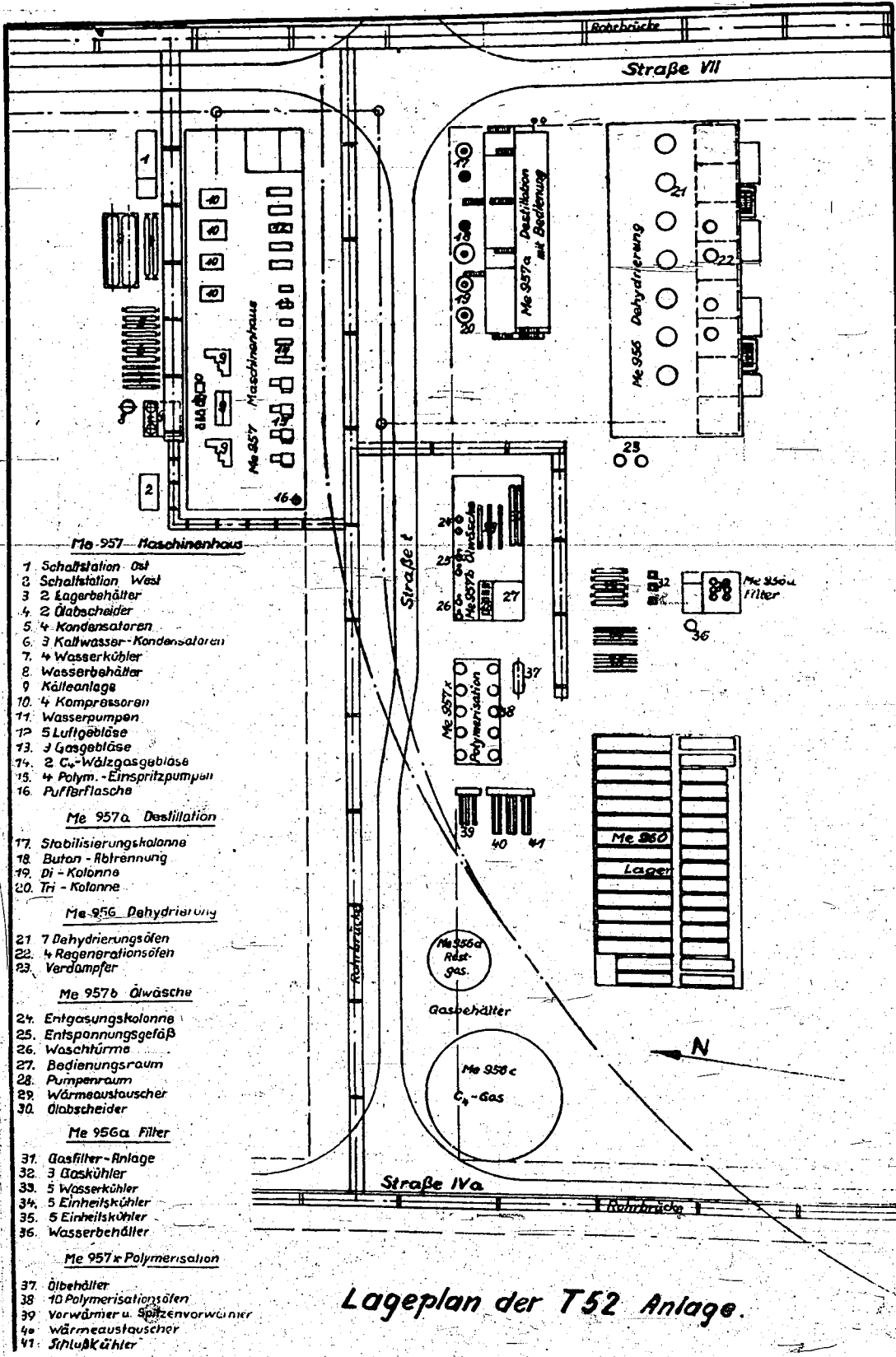
The output of ET 110 during the operational year amounted to, roughly, 6000 tons, about 100 tons of which originated in the T-52 pilot plant.

In the first of August the operations had to be stopped due to a fire which broke out in the isobutyl plant, and again on August 17 during six weeks due to bomb damage. If these incidents had not occurred the annual production would have reached 7000 tons.

Activities for 1941.

The experimental work relating to the splitting-off of water and polymerization in a single operation is to be continued. New plant equipment comprising two 800 mm. silver-lined (?) ("Silberoefen") furnaces are to be started. Occasionally products with octane numbers as high as 95 are obtained, but these values are as yet not fully reproducible. The influence of pressure, temperature, water content, time of contact (rate of flow) must be investigated some more.

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Me 957 - Maschinenhaus

- 1. Schaltstation Ost
- 2. Schaltstation West
- 3. 2 Lagerbehälter
- 4. 2 Übscheider
- 5. 4 Kondensatoren
- 6. 3 Kaltwasser-Kondensatoren
- 7. 4 Wasserkühler
- 8. Wasserbehälter
- 9. Källeanlage
- 10. 4 Kompressoren
- 11. Wasserpumpen
- 12. 5 Luftgebläse
- 13. 3 Gasgebläse
- 14. 2 C₂-Wälzgasgebläse
- 15. 4 Polym.-Einspritzpumpen
- 16. Puffflasche

Me 957a. Destillation

- 17. Stabilisierungskolonne
- 18. Butan - Abtrennung
- 19. Di - Kolonne
- 20. Tri - Kolonne

Me 956 Dehydrierung

- 21. 7 Dehydrierungsöfen
- 22. 4 Regenerationsöfen
- 23. Verdampfer

Me 957b Ölwäsche

- 24. Entgasungskolonne
- 25. Entspannungsgefäß
- 26. Waschtürme
- 27. Bedienungsraum
- 28. Pumpenraum
- 29. Wärmeaustauscher
- 30. Ölabscheider

Me 956a Filter

- 31. Gasfilter-Anlage
- 32. 3 Gaskühler
- 33. 5 Wasserkühler
- 34. 5 Einheitskühler
- 35. 5 Einheitskühler
- 36. Wasserbehälter

Me 957x Polymerisation

- 37. Ölbehälter
- 38. 10 Polymerisationsöfen
- 39. Vorwärmer u. Spitzenvorwärmer
- 40. Wärmeaustauscher
- 41. Strömungskühler

Lageplan der T52 Anlage.

The working up of isobutane (T52 plant)
(Page 49)

Operation:

Messrs. Dr. Fischer
Dr. Straetz
Dr. Jagemann

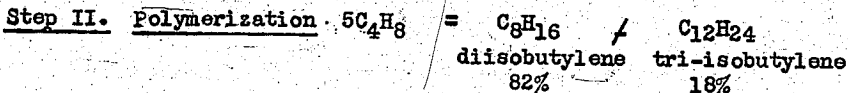
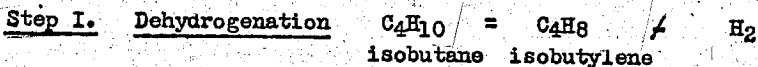
Repairs:

Mr. D. T. Adolphi

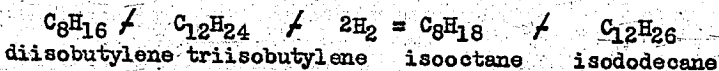
Activities in 1940

The process for the dehydrogenation of isobutane to isobutylene and the following polymerization of the latter to di- and tri-isobutylene as developed in the experimental laboratory has reached a point of development such that a plant with an output capacity of 19,000 t.p.y. of T52 was planned and constructed within the confines of the new southern sector of the works. In connection therewith it is intended to work up in addition to the isobutane produced in the Leuna hydrogenation plant, also the isobutane recovered in the Boehlen and Magdeburg hydrogenation plants. Simultaneously a 26,000-ton plant carrying out the same process is being built in Scholven, and a 12,000-ton plant in Poelitz.

The process comprises the following steps:



Step III. Hydrogenation



The plant equipment is shown in the accompanying diagrammatic layout plan. The isobutane (C_4H_{10}) is led from the storage tank via a vaporizer and regenerator at ordinary pressure and at 550-600°C. through a dehydrogena-

C O N F I D E N T I A L

tion furnace continuously charged with a chromium oxide-aluminum oxide catalyst. Back of the dehydrogenation furnace is a likewise continuously operated revivifying furnace the function of which is to reactivate the carbon-laden catalyst by passing air over it at 600°C. The conversion to isobutylene in the dehydrogenation furnace amounts to about 20%. The isobutane now carrying this isobutylene content is collected together with the liberated hydrogen in a gas tank. After the compression to 10 atmospheres (superatmospheric) the liquefied hydrocarbons separate from the hydrogen which is freed from the last traces of the hydrocarbons in an oil-fed-scrubber at 45°C. and is passed on into a waste gas tank. The wash oil is freed in a degasifying tower by heating from the C₄ hydrocarbons which are again expanded in the reaction gas tank. The liquefied C₄ hydrocarbons are freed in a stabilizing column from small amounts (0.5-1.0%) of C₂- and C₃-hydrocarbons and methane which owe their origin to side reactions in the dehydrogenation furnace. These, too, are passed into the waste-gas tank. The waste gas is used for heating the hydrogenation furnace. Of the pure C₄-hydrocarbons, viz., isobutane and isobutylene, collected in the sump part of the stabilization column in a ratio of 80:20%, the isobutylene is next polymerized to the extent of about 82% to disobutylene and to the extent of about 18% to triisobutylene by means of an asbestos-phosphoric acid catalyst at 50 atmospheres (abs.). A column connected in series has the function to separate the unconverted isobutane from the polymerizate. The former is again returned into the isobutane tank. In two other columns purification of the di- and tri-isobutylene mixture to free it from small amounts of higher polymers is effected. The thus purified polymerizate is hydrogenated at 200 atm. (abs.) with hydrogen in a cyclic process to the end product T52 (= ET 110).

C O N F I D E N T I A L

The accompanying plant layout sheets show how the buildings required in this production fit into the general survey plan of the southern sector of the works.

Activities for 1941

Further experimental work with active catalysts in the pilot plant Me 52, and start of the new plant.

T-52 Plant layout diagram (facing page 49)
(translation of terms)

Ma 957 Powerhouse

1. Electric switch cabinet (east)
2. " " " (west)
3. 2 storage tanks
4. 2 oil separators
5. 4 condensers
6. 3 cold-water condensers
7. 4 water coolers
8. Water tank
9. Refrigerating machine
10. 4 compressors
11. Water pumps
12. 5 air blowers
13. 3 gas blowers
14. 2 blowers for recirculating C_4 gas
15. 4 injection pumps for handling polymerizate (?)
16. Gas cushion flask

Me 957a Distillation

17. Stabilization column
18. Butane separator
19. Di-column
20. Tri-column

M 956 Dehydrogenation

21. 7 dehydrogenation furnaces
22. 4 reactivation furnaces
23. vaporizers

Me 957b Oil-scrubbing plant

24. Degasifying column
25. Expansion tank
26. Scrubbers
27. Office
28. Pump room
29. Heat exchangers
30. Oil separator

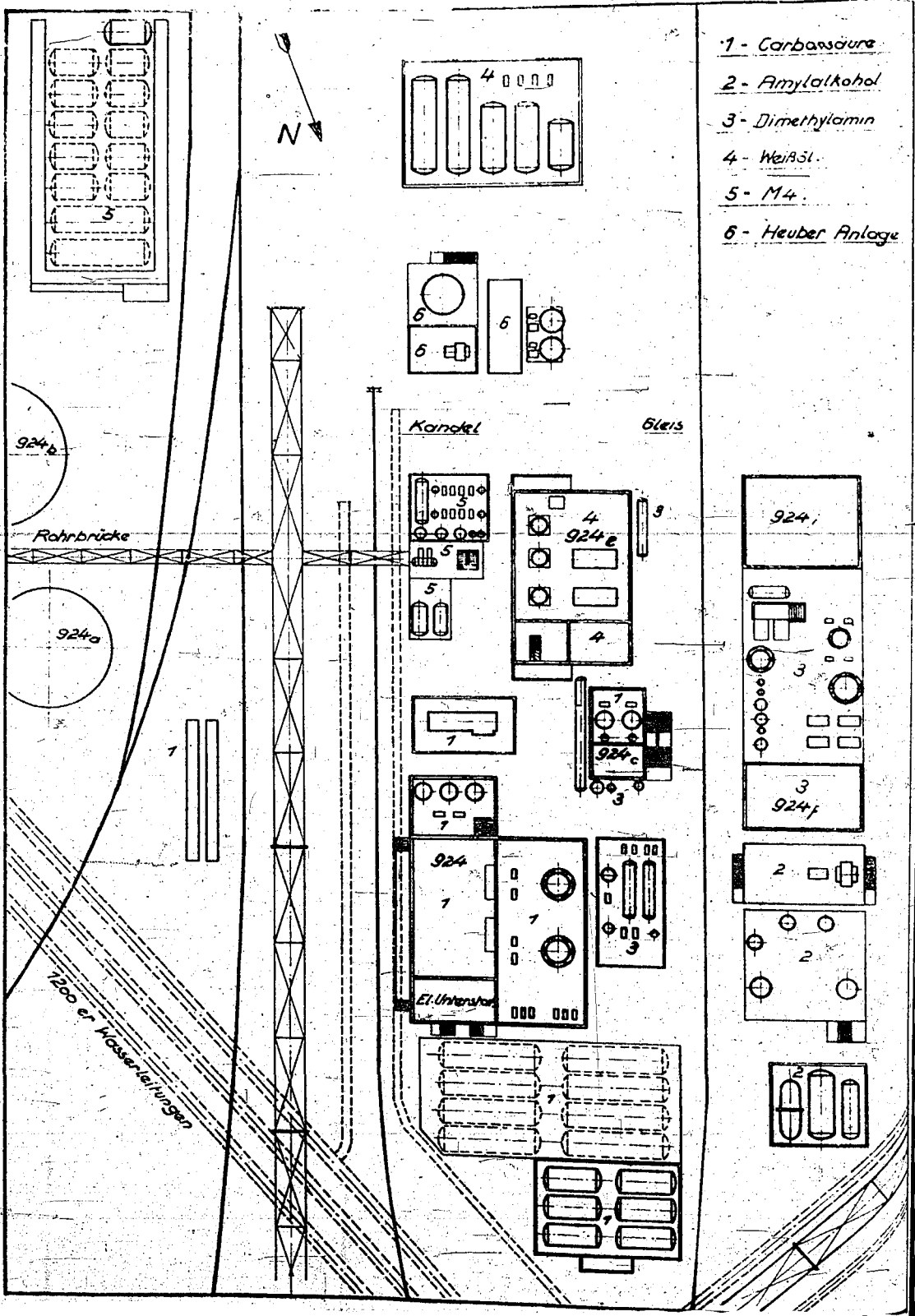
Me 956a Filtration installation

31. Gas filter installation
32. 3 gas coolers
33. 5 water "
34. 5 unit "
35. 5 " "
36. Water tank

Me 957x Polymerization

37. Oil tank
38. 10 polymerization furnaces
39. Preheater
40. Heat exchanger
41. Final cooler

- 1 - Carbonsäure
- 2 - Amylalkohol
- 3 - Dimethylamin
- 4 - Weisöl
- 5 - M4
- 6 - Heuber Anlage



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(Drawing opposite Page 50)

Plot Plan

Carbonsäure	-	Carboxylic acid
Amylalkohol	-	Amyl alcohol
Weissol	-	White oil
Anlage	-	Plant

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(Page 50)

Production of Carboxylic Acids

(Managing personnel listed)

Work in 1940:

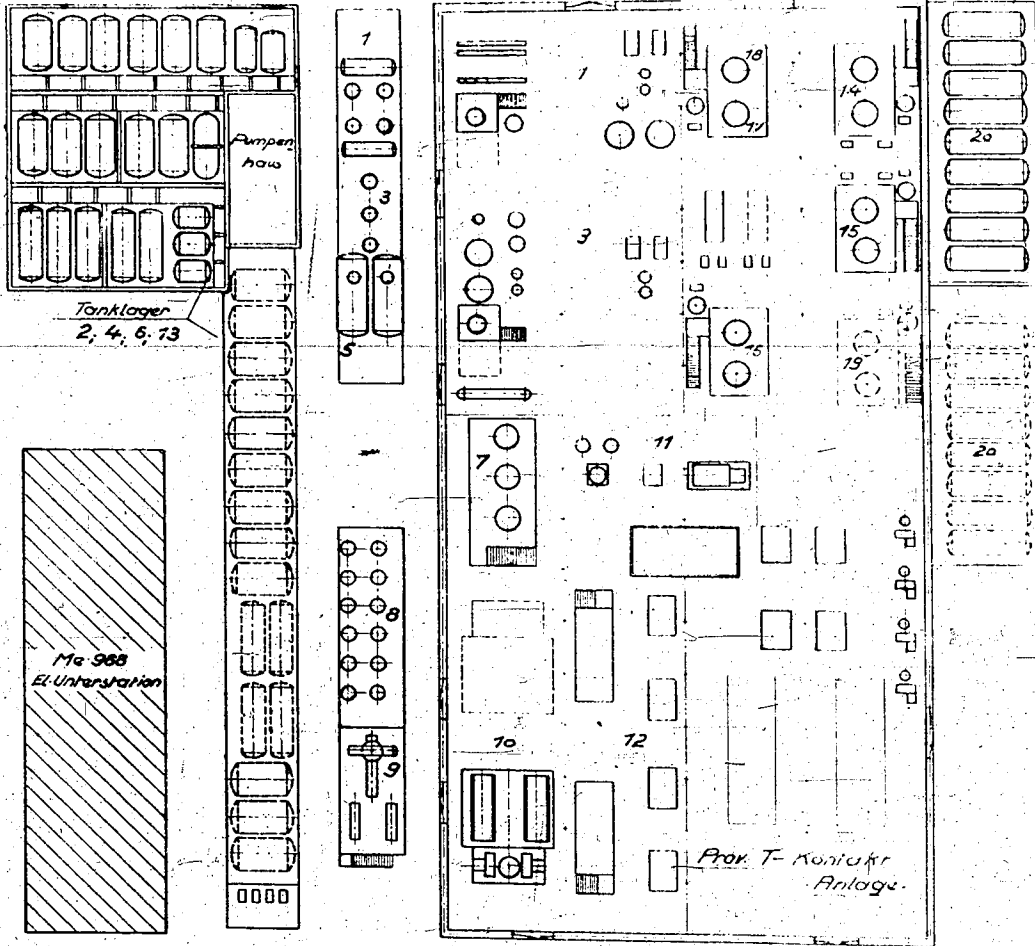
The increase in capacity predicted in 1939 has not yet been carried out south of the plant, but in plant Me 494. An atmospheric pressure process developed by Dr. Löwenberg was used which permits the replacement of autoclaves with an agitator for the conversion of alcohols with caustic soda. The new agitator has a volume of 8 cubic meters and was to produce 60 tons per month of carboxylic acid. It was started in October and is presently producing 20 tons per month since operating difficulties still have to be eliminated. A leaking jacket interrupted operation of the autoclave on November 1, which resulted in 6 weeks loss of production. Otherwise the autoclave operated without trouble. The 1940 production was about 250 tons.

Shortage of naphthenic acid caused Griesheim to increase its use of Leuna carboxylic acid to 40 tons per month. The plant is loaded at present in order to produce this amount. In 1940 a total of 250 tons was shipped to Griesheim. Some lower molecular weight acids were sent from the laboratory to interested parties.

Work for 1941:

Since the demand of Griesheim will increase considerably, a new unit will be built next year. Design is in progress and the layout is shown on the attached plot plan. In Me 924 one atmospheric pressure agitator of 20 cu.m. volume will be built which is to produce 150 tons per month of carboxylic acid. A second agitator and vacuum column is planned. For the production of lower molecular weight acids an existing autoclave will be erected. This unit also includes three acidification vessels, vacuum distillation, vessels for preparing the water and several storage vessels. The units will be built in the buildings which house the amyl alcohol, dimethylamine, white oil, K-4, and Häuber plants.

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1-6 Aldehyde:

- 1 = Formaldehyd
- 2 = Tanklager zu 1
- 3 = Propionaldehyd
- 4 = Tanklager zu 3
- 5 = Propionsäure
- 6 = Tanklager zu 5

7-13 P₂-Anlage:

- 7 = Kondensation
- 8 = Extraktion
- 9 = Destillation
- 10 = Maischung u. Zentrifugierung
- 11 = Rückstandaufarbeitung
- 12 = Sublimation
- 13 = Tanklager

14-20 T-Anlage:

- 14 = Veresterung
- 15 = Waschung
- 16 = Destillation
- 17 = Raffination
- 18 = Mischung
- 19 = S-Produkt
- 20 = Tanklager

Ammoniakwerk Merseburg G.m.b.H.

Lageplan.

M. 7:500

Me. 981

21.11.40. Korbmann Jr.

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(Drawing Opposite Page 51)

Plot Plan Me 981

Rohrbrücke	-	Pipe alley
Tanklager	-	Tank farm
Pumpenhaus	-	Pumphouse
El. Unterstation	-	Electrical substation
Anlage	-	Plant
Propionsäure	-	Propionic acid
Mischung	-	Mashing
Zentrifugierung	-	Centrifuging
Rückstandaufarbeitung	-	Workup of residue
Veresterung	-	Esterification
Waschung	-	Wash
Raffination	-	Treating
Mischung	-	Mixing

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(Page 51)

Production of Trimethanol Ethane and its Ester

(Managing personnel listed)

Work in 1940:

The equipment for the condensation of formaldehyde with propionic aldehyde and caustic to trimethanolamine, which was described in the 1939 report, has been completed. The extraction section was started up. The estimated capacity of 20-30 tons/month was reached and current production is a ton of crude product per day. However, this performance could only be realized lately when improvements were made which gave an 80% yield. A total of 120 tons was produced in 1940 and sent to Uerdingen for conversion to "Alkydale."

Further developments were made by cooperation of the plant laboratory with the organic laboratory primarily in the field of purification of the crude product by sublimation. A series of experiments were made with the Gersthofen camphor sublimier. These led to the conclusion that the crude product could be sublimed in Gersthofen and that Leuna should install its own facilities with the aid of Gersthofen. The Leuna sublimier is being brought up at present.

In the plant laboratory an increase in yield to 80-90% was reached by maintaining a definite temperature and suitable charge rates of the aldehydes. The condensation to the aldol and its catalytic hydrogenation to trimethyl-ether mentioned in the last report, will be taken up again as soon as the necessary staff is available.

Work for 1941:

At the request of RWA and on the basis of the guaranteed demand by Uerdingen, the construction of a new unit in the southern part of the works (Me 981) is planned with a capacity of 150 tons/month. The attached sketch shows the location of the new unit in Me 981.

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(Page 51 contd.)

Production of Esters for Special Lubricants

(Managing personnel listed)

Work in 1940:

The production of low temperature lubricants was further investigated by the organic laboratory. Several such lubricants were developed from chemicals produced in the plant (trimethanolethane, Leuna acids, Leuna alcohols, and adipic acid) which were considered so favorable by authoritative sources that larger quantities had to be prepared for experiments. This was carried out in the carboxylic acid plant and in a new experimental plant in Me-126.

Work for 1941:

In order to be prepared for the demand which we have to count on, a commercial plant was built. It will be erected in the northern part of the works (Me 981). The attached sketch shows the plot plan.

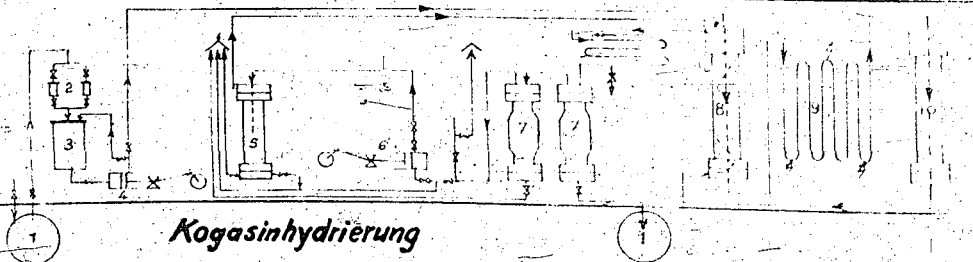
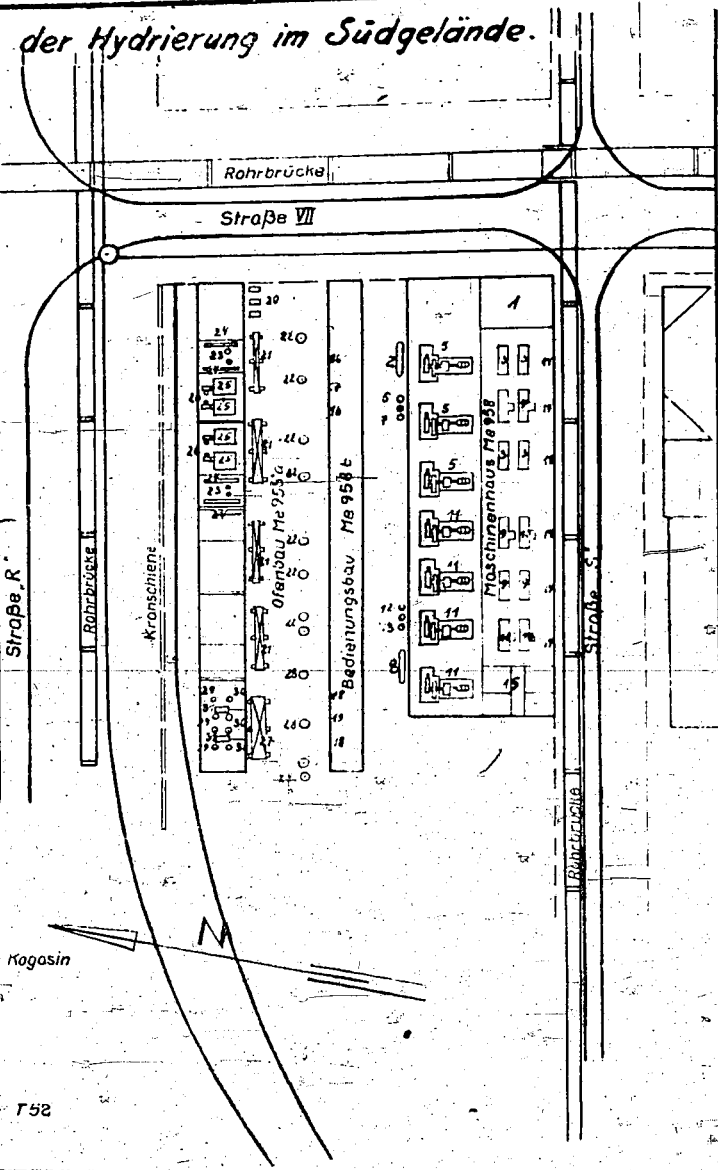
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Lageplan der Hydrierung im Südgelände.

- Ma 958 Maschinenhaus**
1. Schallstation
 2. Ansaugbehälter
 3. Einspritzpumpe 2,5 m³
 4. Einspritzpumpe 50 m³ } für Kogasin
 5. Umlaufpumpe
 6. Abscheideflasche
 7. Pufferflasche
 8. Ansaugbehälter
 9. Einspritzpumpe 2,5 m³
 10. Einspritzpumpe 50 m³ } für T52
 11. Umlaufpumpe
 12. Abscheideflasche
 13. Pufferflasche
 14. Kühlgebläse für Motoren
 15. Meisterzimmer

- Ma 958 b Bedienungsbau**
16. Bedienungsstand } für Kogasin
 17. Tasterkasten
 18. Bedienungsstand } für T52
 19. Tasterkasten

- Ma 958 a Ofenbau**
20. Kaltgasgebläse
 21. Kühler
 22. Produktflasche } für Kogasin
 23. Hochdruckofen
 24. Regenerator
 25. Gasbeheizter Spitzenvorheizler
 26. Wälzgasgebläse
 27. Kühler
 28. Produktflasche } für T52
 29. Hochdruckofen
 30. Regenerator
 31. Dampfspitzenvorheizler



Kogasinhydrierung

1. Zwischenbehälter
2. Filter
3. Ansaugbehälter
4. Einspritzpumpe
5. Abscheider
6. Umlaufpumpe
7. Abscheiderflaschen
8. Regenerator
9. Elektr. Spitzenvorheizler
10. Vorheizler

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TRANSLATION

Hydrogenation of Kogasin to Mepasin

(Page 52)

Operation:

Messrs. Dr. Fischer
Dr. Jagemann
Dr. Kahr

Repairs:

Messrs. Adolph
Wink
Gruettner

Activities in 1940

The Kogasin supplied by various Fischer synthesis plants must be freed from oxygen and olefinic bonds prior to being processed to Mersol, in order to prevent sulfochlorination in the carbon chain. The Kogasin is received with a 5-10% content of hydroxyl-containing and unsaturated compounds, and after the hydrogenation of the latter it is called "Mepasin". Hydrogenation is effected in a closed hydrogen cycle at about 320°C. and under a superatm. pressure of 200 atmospheres in a plant sketched out on the accompanying sheet. The product is passed under nitrogen pressure via a filter and a metering vessel into a force pump by which it is forced into the reaction furnace under 200 atm. (abs.) through a regenerator and an electrically heated peak preheater. The required hydrogen is cyclically conveyed by a circulating pump in an amount equivalent to 1000 cu.m. per cu.m. of Kogasin injected and is separated behind the cooler from the finished Mepasin in the product tank. The equipment is located in building Me 493 and comprises a 500 mm ϕ -furnace holding a 900-liter catalyst charge (2MS. WS₂) and a 300 mm. ϕ -reactivator. The plant is run with 3-3.5 times the catalyst load (?) and has a maximum output capacity of 55 tons of Mepasin per day = 1650 t.p.m. = 20,000 t.p.y. It has been possible to lower the content of oxygenated and hydroxyl-containing impurities to 0-0.5%. The plant was put in operation in

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April 1940 and up to the end of the year (with the December production figure estimated) has produced 5,500 tons of Mepasin.

In plant-scale test runs carried out for Wolfen it was found that the hydrogenation is also possible at 25 atmospheres (abs.), but in this case the content of OH-containing and unsaturated compounds rises to 0.5-0.9%. Even though a product of this composition appears to be still utilizable for the production of Mersol, provision has been made at the Wolfen works for a 200 atm. hydrogenation in view of certain mineral oil fractions to be worked up there later on.

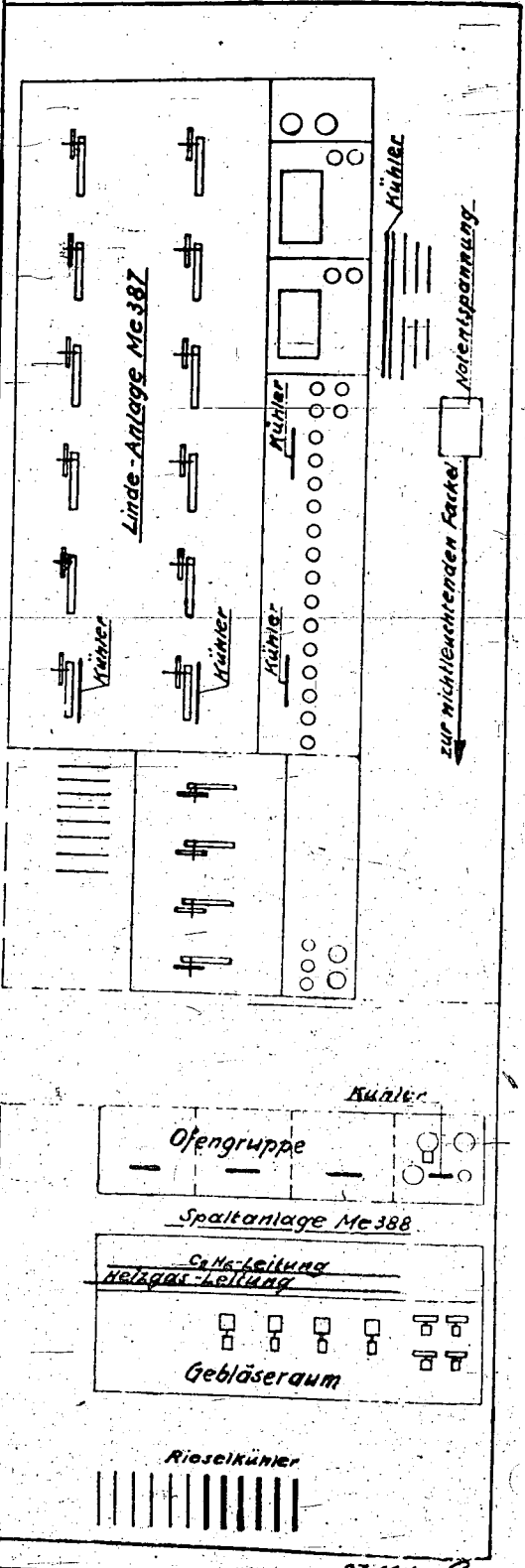
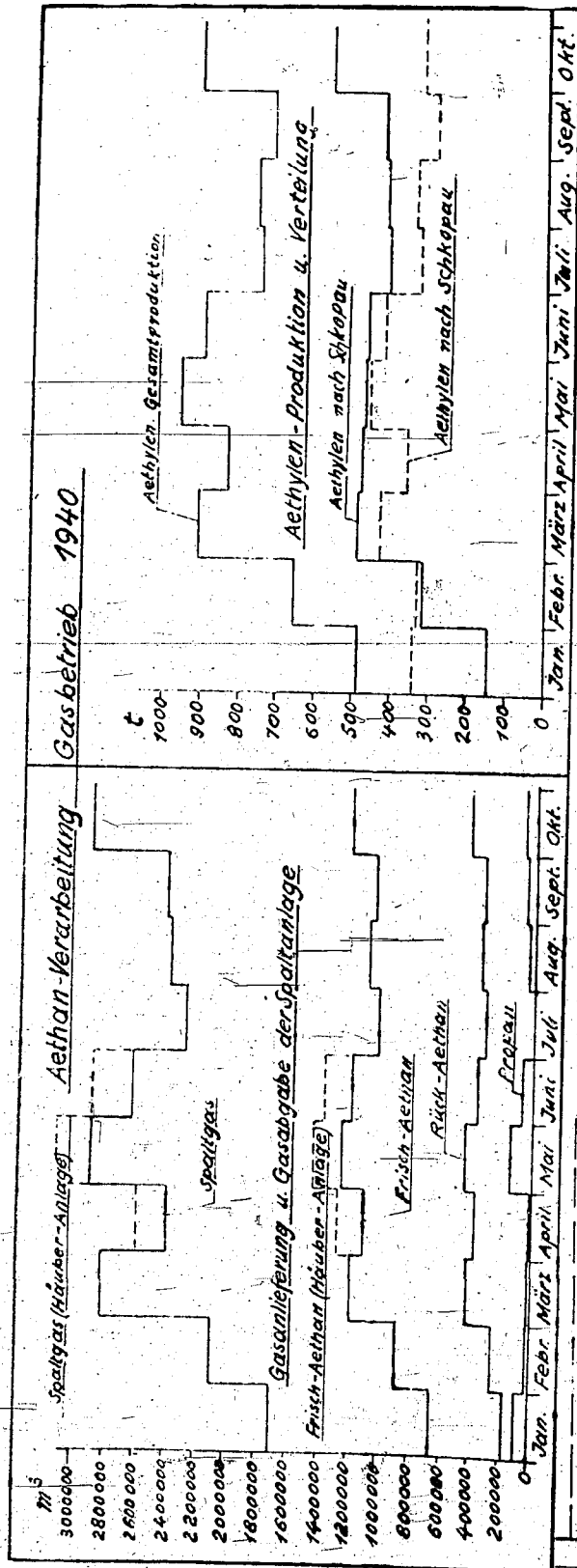
Activities for 1941.

Since the production of Mersol at Leuna has originally been planned for 60,000 t.p.y. the stocks of Kogasin required therefor, viz., 40,000 t.p.y. are to be hydrogenated in a new plant to be situated in the southern sector of the works (Me 958). The first furnace therefor is already being erected.

Translation of terms of accompanying flow sheet
Kogasin hydrogenation.

1. Intermediate tank
2. Filters
3. Suction line tank
4. Injection pump
5. Separator
6. Circulating pump
7. Separating flasks
8. Reactivator
9. Electric peak preheater
10. Preheater

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(Drawing opposite Page 53)

Ethane Processing - Gas Plant 1940

Spaltgas (Häuber-Anlage)	- Cracked gas (Häuber plant)
Spaltgas	- Cracked gas
Gasanlieferung u. Gasabgabe der Spaltanlage	- Gas lines to and from cracking plant
Frisch-Aethan	- Fresh ethane
Rück-Aethan	- Recycled ethane
Propan	- Propane
<hr/>	
Aethylen Gesamtproduktion	- Total ethylene production
Aethylen-Produktion u. Verteilung	- Ethylene production & distribution
Aethylen nach Schkopau	- Ethylene (Schkopau)
Linde-Anlage Me 387	- Linde Plant Me 387
Kühler	- Coolers
zur nichtleuchtenden Fackel	- To non-flare torch
Notentspannung	- Emergency release
Ofengruppe	- Reactor group
Spaltanlage Me 388	- Cracking plant Me 388
Leitung	- Line
Heizgas-Leitung	- Firing gas line
Gebäseraum	- Blast room
Rieselkühler	- Drizzle cooler

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(Page 53)

Ethane Processing, Gas Plant

(Managing personnel listed)

Work in 1940:

Cracking Plant:

The gas production was limited throughout by the delivery of ethane and propane from the hydrogenation plant. Heavy frosts caused trouble in January and February. Pipe enlargement (ethane-crude gas; heating gas) and inclusion of heaters at particularly exposed points should eliminate the freezing up of lines in the future. The cracking reactors were in service without interruption. In order to take care of high gas rates, water spray coolers were added. All tubular coolers after the reactors were equipped with ceramic protection on the inlet side since this measure was found good in an experimental case. The enlargement of the unit for the acetylene hydrogenation of all the cracked gas was successful after initial catalyst trouble. The acetylene content was lowered from 0.4-0.8% to 0.1-0.2%

Scrubbing-Linde-Plant:

The enlargement of the scrubbing plant in several sections last year, caused by the increased gas rate, was successful. Besides, the two alkacid scrubbers and the corresponding alkacid regenerators were piped up in such a way that they could be operated in parallel. Thus, each scrubber operates with low gas throughput. The carbon dioxide removal is improved. Foaming and carryover of alkacid solution has not occurred since then. For the stabilization of the increased production (particularly in the summer) coolers were added; after the crude gas compressor, in the oil scrubber, in the alkacid scrubbers and prior to the Linde unit. In this way the output of a Linde unit was increased to 700-750 m³/hr. compared to 550-600 m³/hr. last year. The Linde plant can produce about 12,500 tons/year of pure ethylene compared to an original capacity of 6500 tons/year after deduction of 10% for compressor repair and cleanout time.

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(Page 53 contd.)

The purity of the ethylene averages 97%. The delivery to Schkopau was satisfactory. For fire protection water sprays were installed. In the attached drawing changes have been shown by heavy lines.

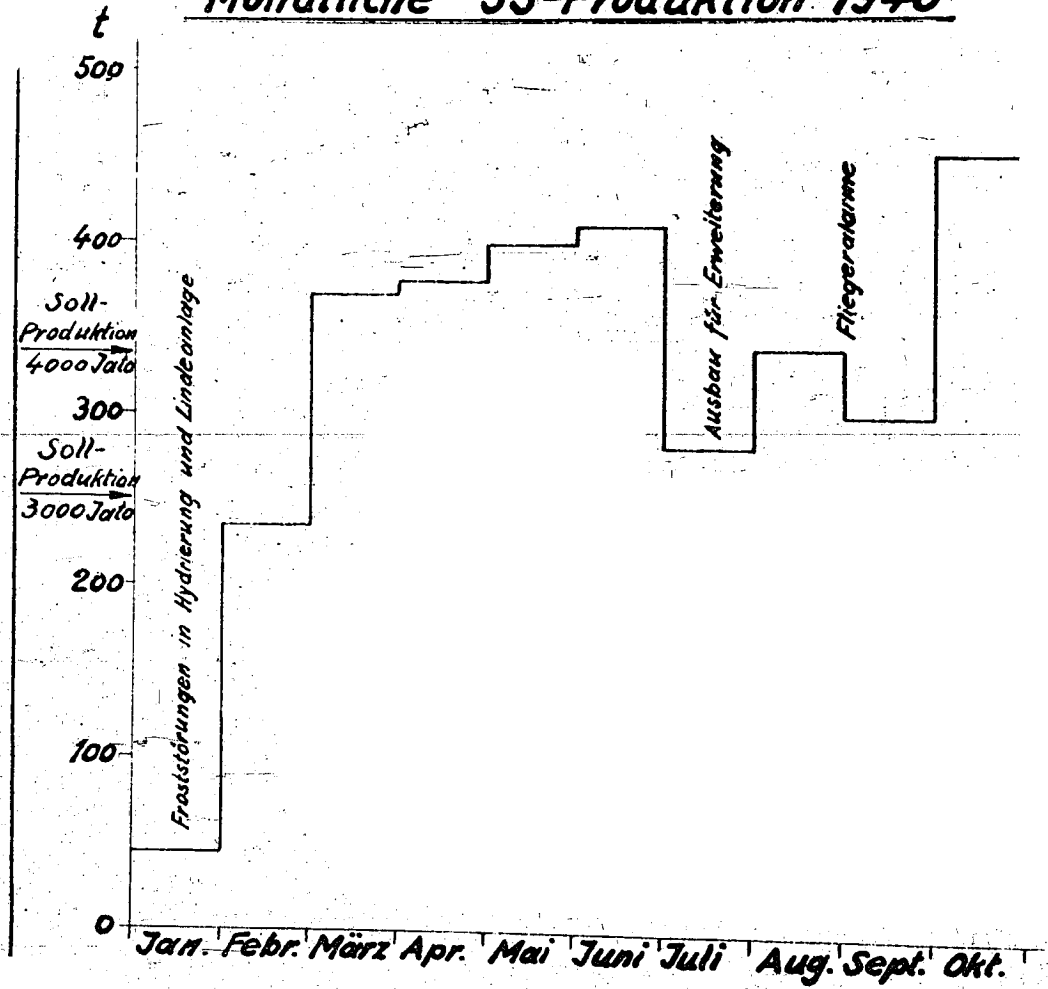
Work for 1941:

Installation of a compressor for crude ethane in order to increase the pressure before the reactors. This will provide more capacity in the present reactors. Installation of two crude gas compressors to insure the higher production in the Linde plant.

Installation of a non-visible flare (air raid precaution) for the disposal of 30×10^6 calories/hour. The flare under construction will burn gas mixtures which must be vented in case of emergency from the cracking plant, Linde plant, and all gas tanks.

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Monatliche SS-Produktion 1940



22.11.40 *Reger*

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(Drawing opposite Page 54)

Monthly SS Production 1940

Soll Produktion	-	Scheduled production
Froststörungen	-	Frost interference
Ausbau für Erweiterung	-	Removal for expansion
Fliegeralarme	-	Air raid alarms

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Ethane Processing, Oil Plant

(Managing personnel listed)

Work in 1940:

The SS-oil plant (synthetic lube) which was built for 3600 tons/year was increased to 4000 tons/year by the addition of a predecomposition unit, a filter press, and operating improvements. This production could even be surpassed in the revised plant. Some loss of production was caused by frost and air raid alarm. A further expansion to 10,000 tons/yr. is under way. Construction is finished and the setting up of equipment and machines has progressed sufficiently that the plant will produce 6000 tons/year by the end of the year.

The following improvements were made:

Polymerization

Catalyst addition was changed. The former weighing and handfilling were replaced by a mechanical device which has the following advantages: one man can do the job quickly and easily; no weighing and deterioration of $AlCl_3$; no dust and odor nuisance.

Decomposition

The old method of sludge separation and decomposition was eliminated and replaced by this new and improved procedure.

The autoclave content is depressured in a 2 m³ agitator and there treated with a certain quantity of methanol which converts the sludge into an easily separable form. The depressuring in this pre-decomposer and the removal to the centrifuge (Schälzentrifuge) is done continuously. After the crude polymer has passed through two centrifuges in series where most of the sludge is separated it contains only small quantities of dissolved catalyst. For complete purification the oil is charged to the main decomposition vessel of 10 m³ volume where it is treated with methanol and lime and then cleaned by filtration.

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(Page 54 contd.)

The advantages of this revised final process are: the cleaning and maintenance of the Laval centrifuges formerly used for catalyst sludge separation were very time consuming and costly. The filter cake is now dry, therefore easily removed and recovered practically oil free by extraction. The PC filter cloths are easier to clean.

Distillation.

Column 3 which was installed for the case that the air ministry required delivery of SS 903, was used for SS 906. In order to prevent corrosion in the coolers, diluted caustic injection was used successfully for condensation.

The caustic was circulated. The column formerly used in this service was transferred to white oil distillation after revisions for vacuum operation.

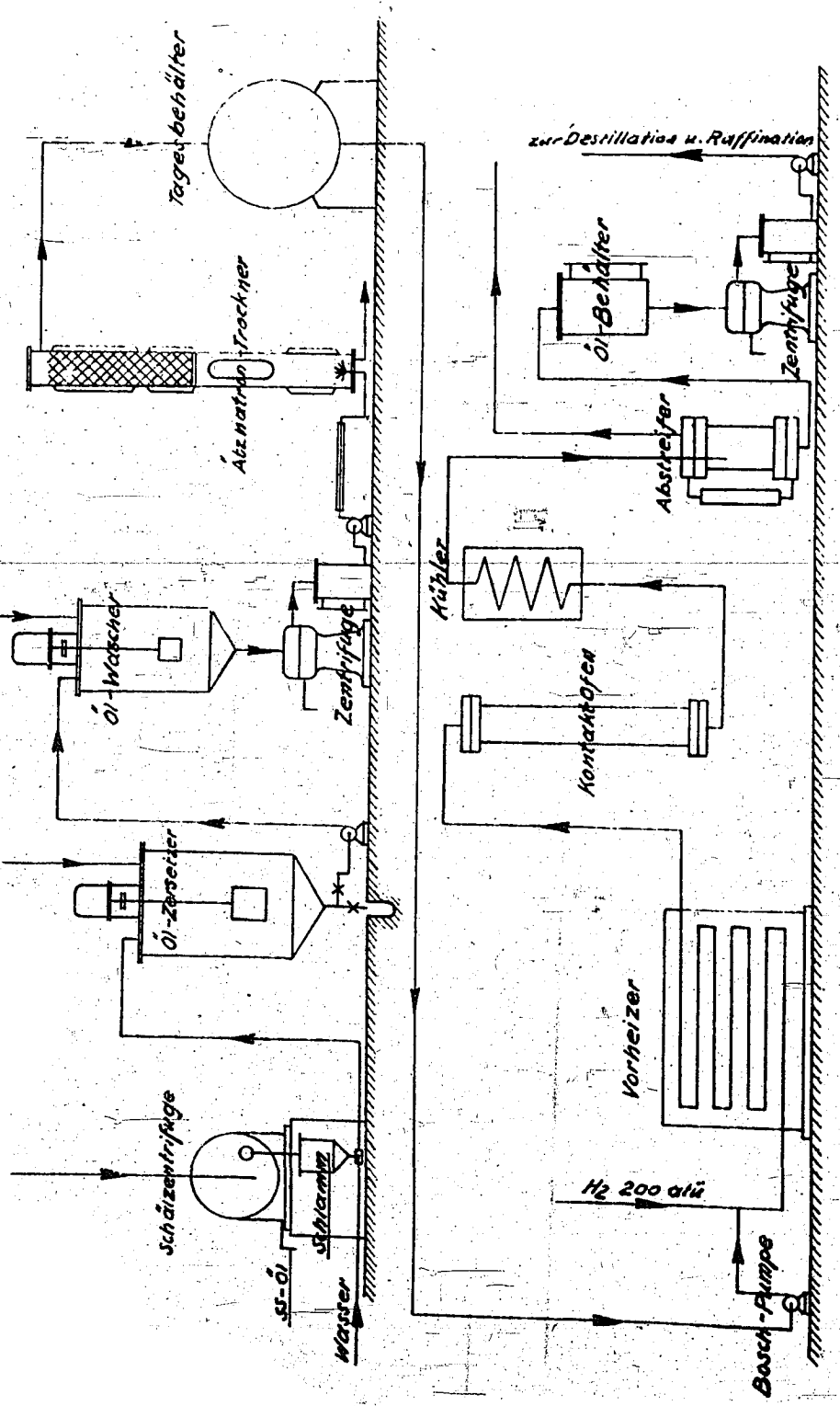
Work for 1941:

The enlargement of the plant will be continued. The production of the complete plant shall be increased to 10,000 tons/year by mid-1941.

Beyond the enlargement in progress a new pipestill column will have to be installed as a reserve.

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R-Öl Fabrikation



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(Drawing opposite page 55)

R-Oil Production

Schälzentrifuge	-	Peeling centrifuge
Schlamm	-	Sludge
Wasser	-	Water
" Öl-Zersetzer	-	Oil decomposer
" Öl-Wascher	-	Oil washer
" Ätznatron-Trockner	-	Caustic dryer
Tagesbehälter	-	Daily storage
Bosch-Pumpe	-	Bosch pump
Vorheizer	-	Preheater
Kontaktofen	-	Reactor
" Kühler	-	Cooler
Abstreifer	-	Separator
" Öl-Behälter	-	Oil storage
Raffination	-	Treating

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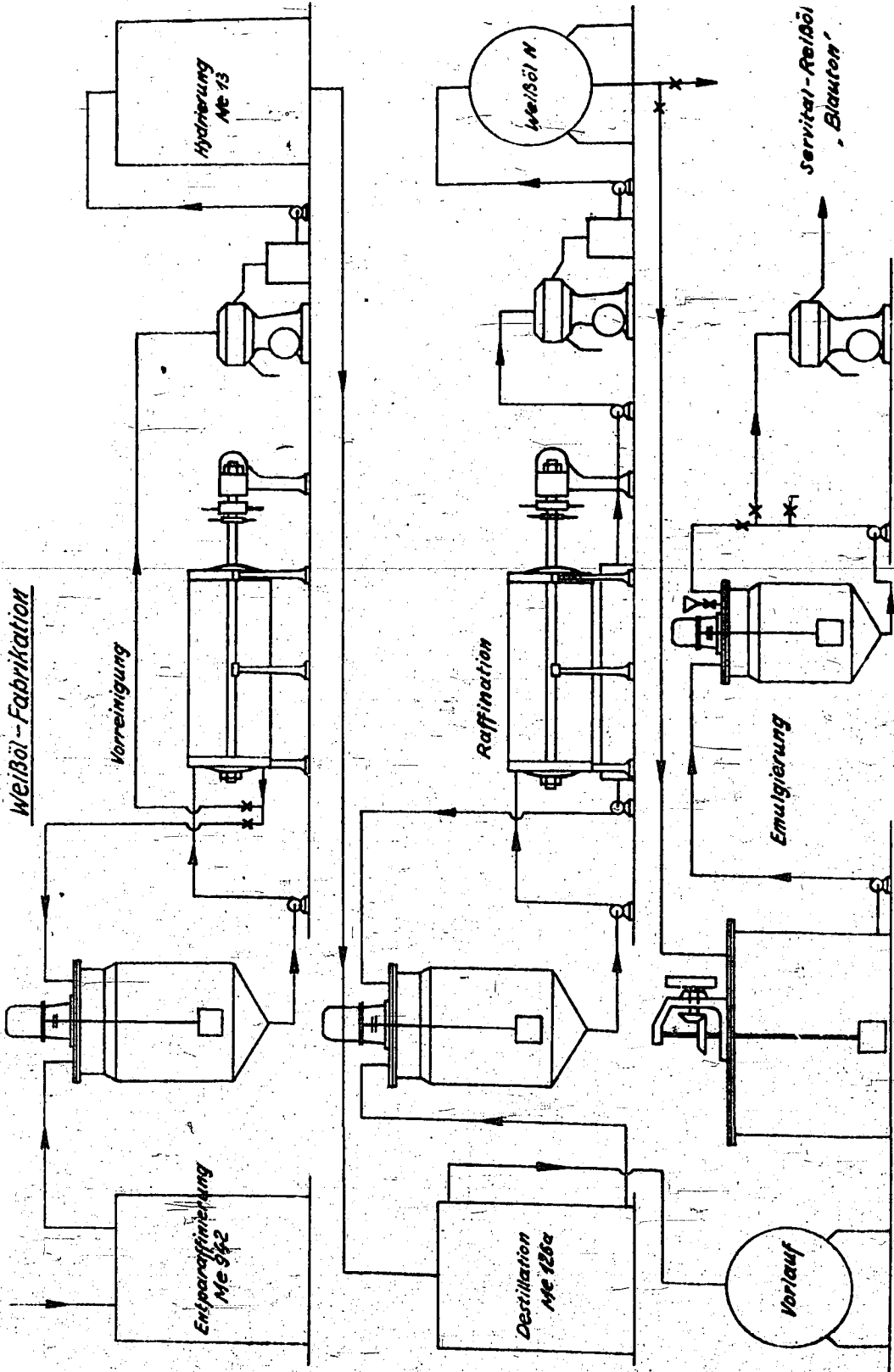
R-oil production

(Managing personnel listed)

Work in 1940:

The catalyst sludge which is recovered in the centrifuges of the SS oil (synthetic lube) production is an addition compound of aluminum chloride and highly polymerized olefinic hydrocarbons. By conversion with water a residual oil was recovered from the sludge which is sent to the hydrogenation plant at the present time. Experiments showed that a very valuable product could be produced by hydrogenation which could be used as lubricating oil or for several special purposes, depending upon operating conditions. The quantity of residual oil recovered in the SS oil production is at present 20 T/M. After the expansion of the lubricating oil plant has been completed about 50 T/M of R-oil will be produced. A plant for the recovery of crude residual oil from the polymerization sludge and for the hydrogenation of the recovered oil is under construction. The flow sheet, according to which the polymerization sludge is converted to R-oil, is shown in the attached drawing.

Weißöl-Fabrikation



(Drawing opposite page 56)

White Oil Plant

Entparaffinierung	-	De-waxing
Vorreinigung	-	Pre-purification
Hydrierung	-	Hydrogenation
Raffination	-	Treating
Weißöl	-	White oil
Vorlauf	-	Light cut
Emulgierung	-	Emulsification

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White Oil Production

(Managing personnel listed)

Operations in 1940:

The process to produce white oils from spindle oils by hydrogenation which was worked up by the main laboratory, was used in a new plant with a capacity of 150 T/M. The oil will be used in the textile industry. Since the laboratory experiments were based on wax Salzbergener oil and since this oil is not available in large quantities the only available crude spindle oil, from Lutzkendorf, had to be used. Because of the widely differing properties from the Salzbergener oil and because of its much more unfavorable behavior, new experiments had to be performed with the Lutzkendorf oil and changes in the process had to be made. As an example, the oil had to be de-waxed because of its insufficient pourpoint.

De-waxing:

The de-waxing was carried out in Me 942 by the dichloroethane process. A pourpoint of -17°C . was obtained.

Hydrogenation:

The de-waxed oil was hydrogenated in Me 13. The hydrogenation was carried out at 18 MV in a hydrogen stream at 200 atmospheres.

Distillation:

The hydrogenated product was steam distilled under vacuum in a bubble plate column in Me 126a (column II of the SS oil plant).

Treating:

Treating of the distillation residue with Fuller's earth and clay was carried out in Me 96a with some equipment from the SS oil plant. Since the product delivered from Lutzkendorf was contaminated with salts and high boiling materials, difficulties were often experienced during hydrogenation

RESTRICTED

(Page 56 contd)

because the electric preheater became coated with a poor heat conducting material which caused the preheated temperature to rise too high. Another difficulty was that the exit became plugged. In order to eliminate these difficulties purification of the crude oil was resorted to. This required the installation of an agitator and a filter press. Because of the expansion of the lubricating oil production a separate installation was required for white oil. Also a new mixing vessel was required for the production of Servital-Reissöl "Blauton" at Leuna. These three installations were put into the empty building Me 924a and started up. The yield is 70% on the basis of spindle oil with the following removals or losses:

2-5% in pretreating
15% " de-waxing
2-5% " hydrogenation
10-15% " distillation

The sales so far have been 630 tons.

Work for 1941:

Further improvements of the process in order to carry out the hydrogenation step without trouble.

RESTRICTED

(Page 57)

Methanol Catalyst Plant

(Managing personnel listed)

Work in 1940:

The drying drum for the regeneration of used isobutyl catalyst, which was ordered on the basis of experiments, was started up. The performance of the drum is up to expectations. The operating costs particularly are very low. The capacity could be doubled so that the greatly increased production of isobutyl catalyst could still be operated with regenerated catalyst. The difficulties in obtaining materials were at times very grave. The use of nitrates had to be reduced greatly because of storage reasons. The necessary salts were largely made from the corresponding metals in our own plant, which increased the purity of the raw material. The new catalyst introduced last year continued to be produced. To this were added considerable quantities for HF (hydroforming?) and T52. In 1940 13 types of catalysts were made, a total of 572 tons. Much time had to be spent on preparation for the production of HF and T52 catalyst. The commercial production of dehydrogenation catalyst in the form of 5-6 mm. spheres has caused particular difficulties which have not been overcome. The spherical shape is necessary since the catalyst is moved continuously through the reactor and regenerator. In order to form the catalyst spheres it is planned to squeeze the kneaded catalyst mass by means of vacuum presses into strands and to roll these into balls in a Frankoma rolling machine.

Two electric Tammann melting furnaces were installed in which alloys can be molten at over 2000°C. The furnaces had formerly been used for the production of Raney nickel.

Work for 1941:

The already finished experiments for substituting hydraulic double presses by Weglin and Hübner for briquet presses have led to a solution,

(Page 57 contd)

by which the mixing of the methanol and isobutyl catalyst paste, which was formerly done by hand, has been largely mechanized. Such a press has been ordered and is to be delivered in March. The completely insufficient equipment for the preparation of five compounds catalyst and similar ones for the ethanol synthesis will be replaced by a decomposition drum which is also expected to be installed in the beginning of the year.

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YELLOW OCHER CATALYST PLANT

(Managing personnel listed)

Work in 1940:

The yellow ocher catalyst plant was used for several months in 1940. Because of the possible substitution of low tungsten content catalyst for catalyst 5058 only yellow salt was produced which can be included in all new catalysts under consideration so far. Considerable quantities of used 5058 catalyst were delivered by Zeitz for regeneration. Since the roasting worm conveyor was not needed for the decomposition of yellow salt, catalyst 3076 which was needed for the various T52 plants and for Kogasin hydrogenation was sulfurized. The pressure filter for drying of yellow salt was not purchased. The decision about this installation will be deferred until the catalyst program has been clarified.

Work for 1941:

Leuna has the job of building a plant for the production of 50 T/M of purification compound 6434 (diluted catalyst) or 6719 (iron catalyst). The plant will probably be started up in the second quarter of 1941. Besides that, purification compound 5058 will again have to be produced since stocks are low. The roasting plant in Me 244 will be transferred to Me 245 in connection with the return of building Me 244 to the power plant. In connection with this a roasting drum with dust chamber will be installed. Its capacity is 30-40 T/M so that in the future all used returned purification compounds can be completely roasted and reworked.

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Catalyst Experiments

(Managing personnel Listed)

Work in 1940:

About 700 catalysts were prepared.

I. Work was done primarily on the improvement of the five compound catalyst for ethanol production from CO and H₂ which was developed last year by varying the relative quantity of the components, the manufacturing method, the shaping and the aftertreatment. It was also attempted to affect the catalytic activity by the addition of many oxides, metals, and specially treated earths for the purpose of increased ethanol yield and for the formation of other desirable products. Finally experiments were conducted on a broad basis to determine the active principle for the ethanol formation in the five compound catalyst in order to determine guides for future development.

All these projects are very time-consuming since the possibilities for variation in a multi-component catalyst are very great and therefore most of the investigation cannot be considered completed. Up to now the most important result has been the development of a variation of the five component catalyst with which the propanol formation can be obtained in at least the same order of magnitude as the ethanol formation with the original catalyst. The ethanol content of the products is approximately half, butanols appear only in traces. Furthermore, production methods for such catalysts were determined (denitration of the concentrated solution of the starting material in a rotating heated drum) which eliminates the equipment difficulties encountered with molten nitrates and also yields a far more homogeneous product in a considerably shorter time. This process is at present being changed to a larger scale. Finally a method of preparation for the five component catalyst was tested, in which the used catalyst was brought back in a simple way to the original activity.

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(Page 59 contd.)

II. The following other catalyst problems were worked on or solved;

For the preparation of dimethylamine from monomethylamine and formaldehyde a new copper catalyst was developed. The process is at present being changed to large scale. The preparation of Raney nickel was further worked on and brought to a satisfactory point. In connection with the further development of the methanol and isobutyl catalyst, the experiments for the replacement of chromic acid with a sufficiently active chromoxide were continued. They could not be completed yet. However, experiments for the elimination of hydraulic pressing which is very undesirable on the basis of cost and hygiene, have progressed sufficiently far that this method can soon be eliminated. Furthermore, a series of experimental catalysts were prepared for the dehydrogenation of alcohols to aldehydes and ketones for the Oxo process and for the very high pressure synthesis of acids. Finally a number of hydrogenation catalysts were prepared for various purposes.

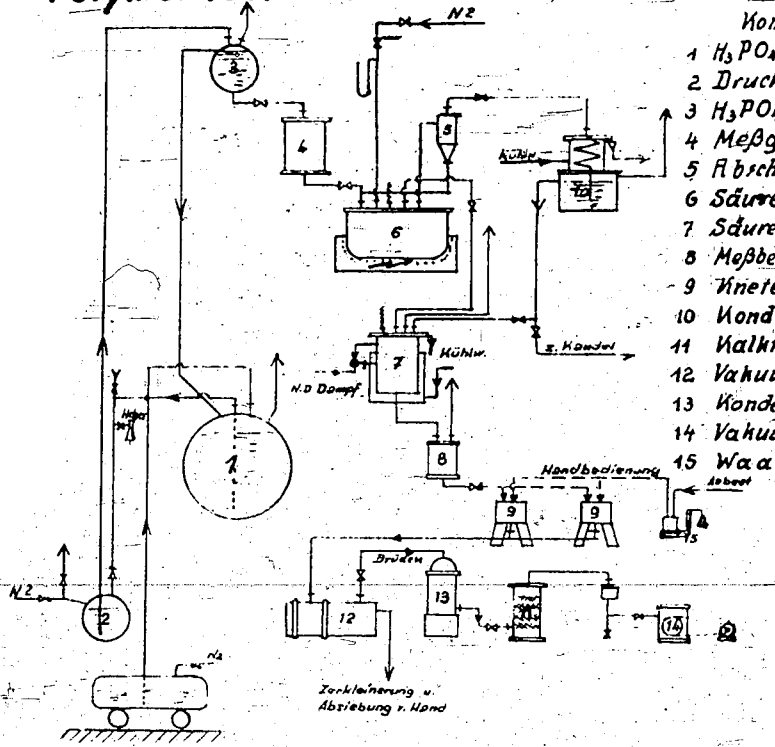
III. A considerable part of the time and personnel had to be used this year for plant experiments. At one time the raw material situation forced the use of raw materials of doubtful purity in most catalysts and this required considerable testing work. In particular, however, was it necessary to develop catalysts for the isobutane, HF, and T-catalyst production which had only been developed very roughly in the experimental laboratory and the optimum conditions for their commercial production had to be worked out.

Work for 1941:

After taking care of the purely production experiments and besides taking care of several special catalyst problems, the chief interest next year is to be placed on the alcohol synthesis from CO and H₂ and in particular the development of specific catalysts for the formation of individual alcohols or limited groups of alcohols.

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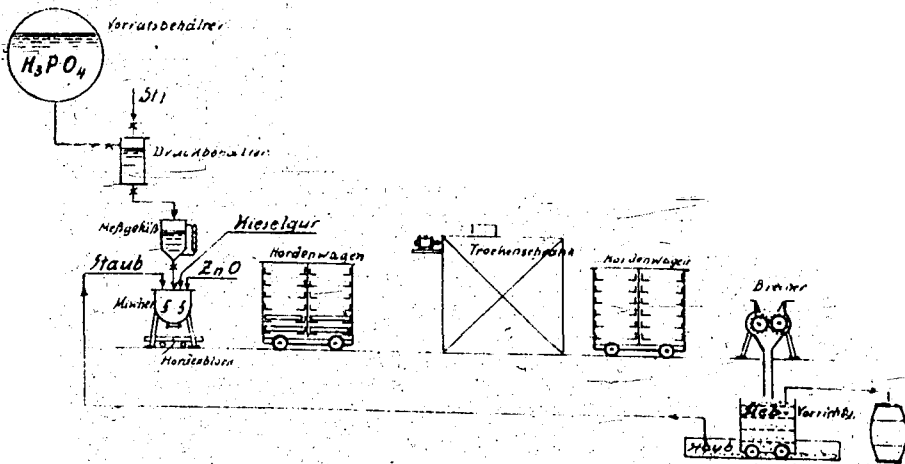
Polymerisations Kontakt für T 52.



Kontakt II Me. 971

- 1 H_3PO_4 Säurebehälter
- 2 Druckbehälter
- 3 H_3PO_4 Hochbehälter
- 4 Meßgefäß
- 5 Abscheider
- 6 Säurehoher [elektr. beh.]
- 7 Säurekühler
- 8 Meßbehälter
- 9 Knieter
- 10 Kondensator
- 11 Kalkturm
- 12 Vakuumtrockenschrank
- 13 Kondensator
- 14 Vakuumpumpe
- 15 Waage

T-Kontakt [Totusol Anlage] Me. 981 später Me 971



C O N F I D E N T I A L

TRANSLATION

T catalyst plant
(Page 60)

Operation: Dr. Kratz

Repairs: Dr. Herele

Dr. Hill II
(from Oct. 1, 1940)

Activities in 1940

The Waldenburg toluene plant is to be provisionally supplied with catalyst from the Leuna works. According to plans made for the plant in May a catalyst requirement of about 220 t.p.m. is to be reckoned with. The equipment needed for the production of the catalyst were ordered at once due to the urgency. The further development of the project made it however advisable not to install the entire equipment at first due to changes made in the preparation of the catalyst. For that reason the construction of a separate plant building was desisted from and production initiated as a temporary expedient in building Me 981 with a daily output capacity of about one ton. In doing this various defects became apparent which, however, could be remedied by changes in the equipment. A plant type catalyst is now being produced which as regards activity, life and form stability comes up to all the requirements.

Activities for 1941

Since the building Me 981 is to be used for its original destination it becomes necessary to shift the plant elsewhere. Provision has been made to install in building Me 971 as much equipment as is needed for a capacity of about 60 t.p.m. Up to the present no figures regarding the magnitude of the catalyst requirements are on hand as no decision has as yet been taken regarding the production capacity of the Waldenburg plant.

C O N F I D E N T I A L

Rec'd JUL 19 1945

Flow sheets facing page 60
Polymerization catalyst for T 52

Catalyst II Me 971

1. H₃PO₄ tank
2. Pressure tank
3. Overhead H₃PO₄ tank
4. Metering vessel
5. Separator
6. Acid boiler (electrically heated)
7. Acid cooler
8. Measuring vessel
9. Dough mixers
10. Condenser
11. Lime tower
12. Vacuum drier chamber
13. Condenser
14. Vacuum pump
15. Scale

Heber: Syphon

Kuehler: Cooler

N.D.Dampf: Low-pressure steam

Handbedienung: Manual operation

Brueden: Water Vapor

T catalyst (toluene plant) in building Me 981, later on in Me 971

Vorratsbehälter: Storage tank

Sti: Abbreviation for nitrogen

Druckbehälter: Pressure tank

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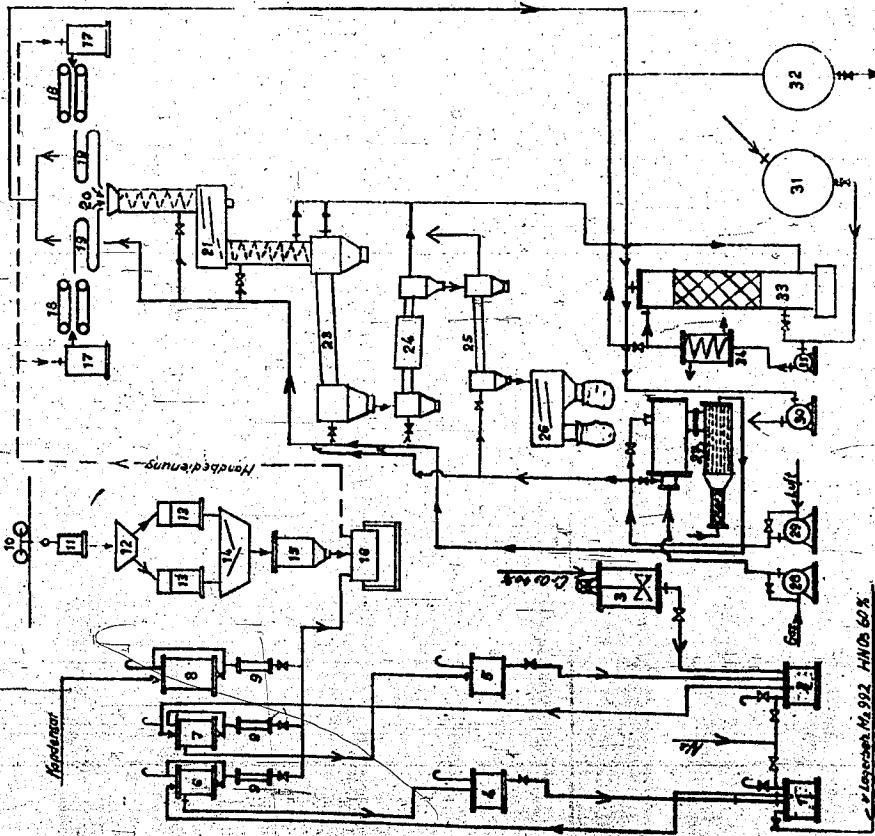
Kieselguhr: Infusorial earth
Messgefäess: Measuring vessel
Staub: Fines (dust)
Mischer: Mixer
Hordenblock: Shelf
Hordenwagen: Tray-carrying truck
Trockenschrank: Drying oven
Brecher: Crusher
Sieb-Vorrichtung: Screening device

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Dehydrier-Kontakt für Isobutan.

Kontakt I Nr 971

1. Druckgefäß HNO_3
2. Druckgefäß Cr-O_2
3. Ansaufgefäß Cr-O_2
4. Rücklaufbehälter
5. Rücklaufbehälter
6. Hochbehälter HNO_3
7. Hochbehälter Cr-O_2
8. Kondensatbehälter
9. Meßgefäße
10. Handlaufkran
11. Transportbehälter
12. Bunker
13. Kugelmühle
14. Zwischenbunker
15. automatl. Waage
16. Krieter
17. Strangpresse
18. Rollmaschine
19. Transportband
20. Wendetrutsche
21. Wüchlsieb
22. Wendetrutsche
23. Trockenhammer
24. Glühtrammel
25. Kühltrammel
26. Schüttelstiel
27. Luftvorwärmer
28. Heizgasgebläse
29. Luftgebläse
30. Saugzuggebläse
31. Frischluftbehälter
32. Rillluftbehälter
33. Waschlurm
34. Laugekühler
35. Laugepumpe



Layebach, Nr. 971, HNO₃ 60%

TRANSLATION

Isobutane Catalyst Plant
(Page 61)

Operation: Dr. Kratz
Dr. Hill
(from Oct. 1, 1940)

Repairs: Dr. Herele

Activities in 1940

For the T-52 plants in course of construction Leuna supplies the necessary catalysts for the dehydrogenation, polymerization, and hydrogenation. Since already considerable amounts of the catalyst are in demand it became necessary to start producing the catalyst in the methanol catalyst plant. In this way it will become possible to build up a stock supply pending the completion of the isobutane catalyst plant, so that both plants together can cover the initial requirements. The new catalyst plant will come into operation - at least partly - at the end of the year. The dehydrogenation catalyst is prepared as follows:

Hydrated aluminum oxide ground dustless in six ball mills is worked up into a stiff homogeneous paste with nitric acid and an aqueous solution of chromates. From this doughy mass a uniform elastic bar is extruded in eight vacuum extrusion presses, and this bar is guided over a conveyor belt into one of the Frankoma rolling mills. In these mills the bar is squeezed into small cylindrical slugs which are then molded into balls of 5-6 mm. ϕ by rolling them between oppositely rotating rollers. The still moist balls slide onto a conveyor belt 10.5 m. long heated to about 100°C., on which they are preheated to such an extent as to permit of freeing them from fines by screening and completing their drying in a V2A drum drier. The dry balls are discontinuously denitrated in a drum calciner at 450°C. whence they are dumped into a drum cooler. After a second screening the finished catalyst is packed into barrels.

C O N F I D E N T I A L

In the polymerization catalyst we have to deal with a mixture of ortho-
an pyrophosphoric acid mounted on asbestos. For its preparation
commercial ortho-phosphoric acid is dehydrated in a silver pan by heating
it to about 220°C. to such an extent as to form a mixture of equal
parts of ortho-phosphoric acid and pyro-phosphoric acid. These mixed
phosphoric acids are cooled and formed with asbestos into a dough in
dough mixers. The resulting dough is dried in vacuum driers at about
100°C. and at 15 mm. Hg pressure. The dry cakes are broken up and
the desired grain size screened out as the finished catalyst. The fines
are returned to the dough mixers.

Activities for 1941

The plant must remain in full operation during 1941. A production
of 360 tons of dehydrogenation catalyst and 200 tons of polymerization
catalyst is expected. Experiments are being made of increasing the
activity of the dehydrogenation catalyst by using precipitated aluminum
hydroxide instead of the commercial hydrated alumina. The tests are to
be continued on a pilot plant scale.

Flow sheet-accompanying page 61
Isobutane dehydrogenation catalyst.

Catalyst I in building Me 971

1. Pressure transfer tank for HNO_3
2. " " " " CrO_3
3. CrO_3 solution tank
4. Reflux tank
5. " "
6. Overhead tank for HNO_3
7. " " " CrO_3
8. Condensate tank

C O N F I D E N T I A L

C O N F I D E N T I A L

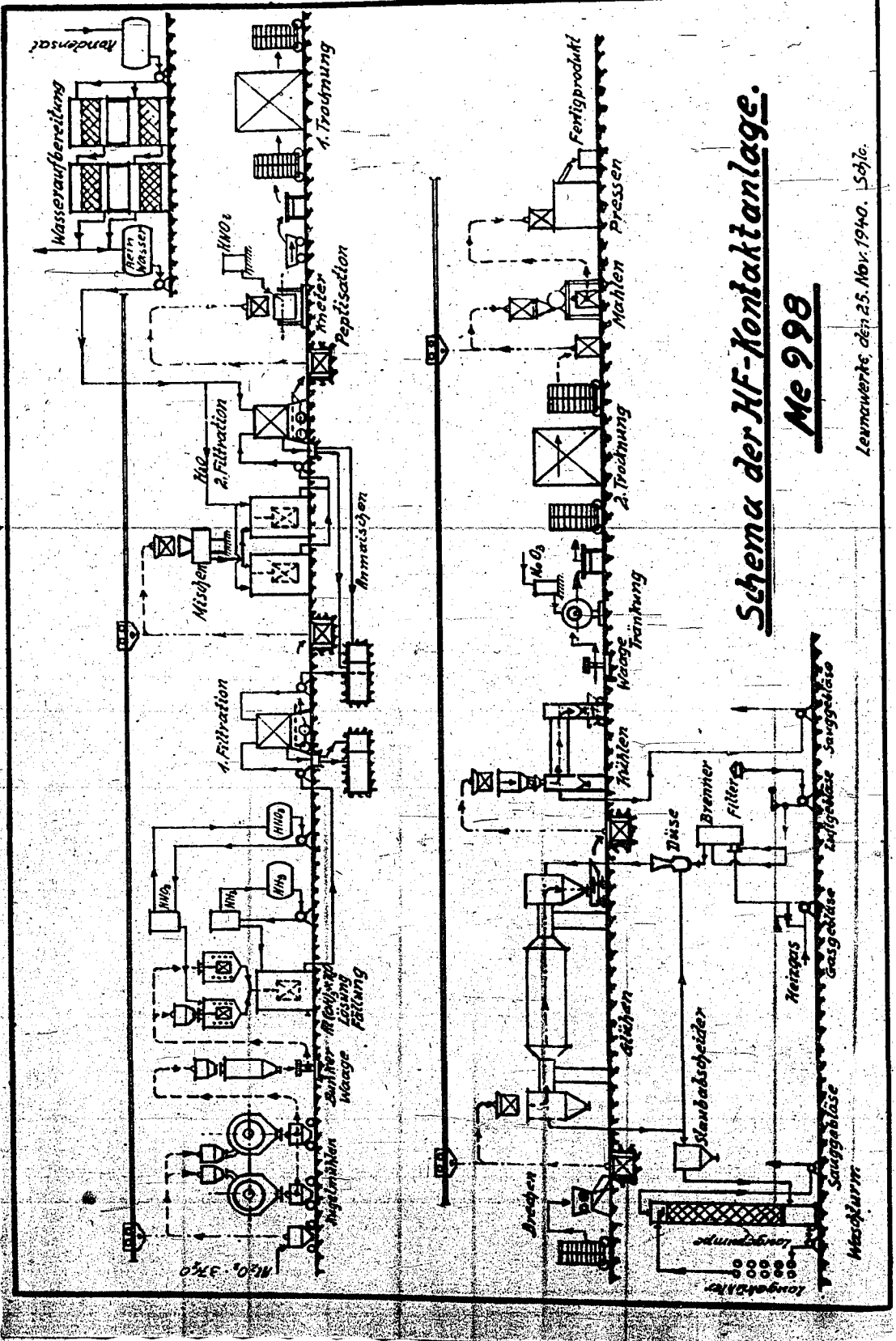
9. Measuring vessels
10. Overhead crane
11. Small transfer bin
12. Hopper
13. Ball mills
14. Intermediate hopper
15. Automatic scale
16. Dough mixer
17. Bar extrusion press
18. Balling machine
19. Conveyor belt
20. Helical slide
21. Screen (shaking screen?)
22. Helical slide
23. Drum drier
24. Drum calciner
25. Drum cooler
26. Shaking screen
27. Air preheater
28. Blower (for heating gas)
29. Blower (for air)
30. Exhaust fan
31. Fresh lye tank
32. Spent lye tank
33. Tower scrubber
34. Lye cooler
35. Lye pump

C O N F I D E N T I A L

~~C O N F I D E N T I A L~~

Kondensat: Condensate
Handbedienung: Manual operation
Luft: Air
Gas: Gas

~~C O N F I D E N T I A L~~



Schema der HF-Kontaktanlage.

Me 998

Lehrwerke, den 25. Nov. 1940. Schlc.

C O N F I D E N T I A L

TRANSLATION

HF CATALYST PLANT
(Page 62)

Operation: Dr. Kratz
Dr. Hill II
(from Oct. 1, 1940)

Repairs: Dr. Herele

Activities in 1940

According to the contract the catalyst for the Moosbierbaum HF (hydroforming) plant must be supplied by Leuna. The amount of this precipitation catalyst (about 20 t.p.m.) exceeds the output capacity of the methanol catalyst plant for catalysts of this type, to such an extent as to render it necessary to build a new plant (20 x 75 meters) in order to install the numerous equipment pieces required. This installation was completed within six months. It will be put into operation partly in the early part of February 1941. Several pieces of plant equipment provisionally taken from other works departments will be replaced upon the arrival of the new equipment. The preparation of the catalyst is as follows:

Commercial hydrated alumina is ground in ball mills to dust fineness and is then dissolved in boiling nitric acid. From this solution hydrated alumina is precipitated by ammonia under certain conditions, repeatedly washed until it is free from salt, whereupon the filter cake is peptized with nitric acid and dried at about 100°C. The dry material is denitrated, saturated with a solution of the activator, dried once more, and formed into pellets by pressing.

The following equipment pieces were procured:

- 2 Ball mills, each with a charging capacity of 200 kg.
- 2 V2A-solubilizing tanks, each holding 1.5 cu.m.
- 1 6cu.m-precipitation tank

C O N F I D E N T I A L

C O N F I D E N T I A L

- 2 Agitators, each of 6 cu.m. capacity
- 1 Petzoldt mixer
- 6 Filter presses 1200 64 frames
- 7 " " 1450 50 "
- 4 Seemann dough mixers, each with a capacity of 400 liters
- 6 Dual drying ovens, each with 80 sq.m. heating area
- 1 Drum calciner V2A with washing equipment
- 2 Dragee boilers
- 2 Alpine mills
- 2 Kilian pelleting machines (from Me. 860 c)

Activities for 1941

During the first months of 1941 the plant for supplying Moosbierbaum will be started. In the event that the next projects for HF-gasoline or DHD-gasoline, as the case may be, are not to be supplied as yet, then it is intended to make in the plant catalysts for splitting off water (amines) or starting material for highly active dehydrogenation catalysts (T 52). The NH_4NO_3 -contg. wash water obtained in the washing operation which originally is run off into the Saale river, is to be enriched in a Wofatit plant and disposed of to the salt plants as a high-% ammonium nitrate liquor. Experimental work with respect thereto is already in course of progress.

Flow sheet of the HF-catalyst plant Me 998
(facing page 62)

Kugelmuehlen:	Ball mills:
Bunker:	Hopper
Waage:	Scale
Loesung:	Solubilization
Faellung:	Precipitation
Mischer:	Mixing

C O N F I D E N T I A L

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Aimaischen:	Slurry preparation
Kneten:	Dough mixer
Trocknung:	Desiccation
Reines Wasser:	Pure water
Wasseraufbereitung:	Water conditioning
Kondensat:	Condensate
Brechen:	Crushing
Gluehen:	Calcination
Kuehlen:	Cooling
Waage:	Scale
Trankung:	Impregnation
<hr/>	
2 Trocknung:	2nd drying operation
Mahlen:	Grinding
Pressen:	Pelleting
Fertigprodukt:	Finished product
Langekuehler:	Lye cooler
Waschturm:	Tower scrubber
Laugepumpe:	Lye pump
<hr/>	
Staubabscheider:	Dust separator
<hr/>	
Heizgas:	Heating gas
Gas geblaese:	Gas blower
Sauggeblaese:	Exhaust fan
Duse:	Nozzle
Brenner:	Burner
Luftgeblaese:	Air blower

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Ethyl Chloride Plant

(Managing personnel listed)

Work in 1940:

Product quality improvement.

The quality of the ethyl chloride produced up to now is decreased by low boiling components (propane, propylene, butane, and vinyl chloride) which cause undesirable pressure increases during the conversion to tetraethyl lead in the autoclave. The current control of the products showed considerable variation of the specific gravity depending on the composition of the ethane charge from the propane plant of the hydrogenation unit which, on the average, contained 88-90% ethane. The plant was therefore changed to the much purer ethane tail gas of the Linde plant Me 387. The quality of the ethyl chloride has since been much more uniform. Interference by the small acetylene content of the tail gas has not occurred.

Further experiments to remove the still present low boiling material by redistillation in column III have not been completely successful but are being continued.

Reactor.

The lower part of the funnel-shaped reactors of V2A are subject to considerable corrosion due to the action of chlorine. The use of silicon carbide lining as a protection against chlorine was not successful. A further experiment with carbon-containing material ("carbon stones") is being prepared.

By-products.

Nearly the total production of 32% hydrochloric acid can be used in the works and sold to outside customers. The dichloroethane was bought by tar plants as a diluent.

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(Page 63 contd.)

Plant expansion.

The capacity of the plant was raised in the course of the year from 50 to 100 tons per month. A further expansion to 120 tons per month is in progress. The installation of new vessels increased the storage for chlorine to 75 tons and for ethyl chloride to 80 cubic meters. Also added were two chlorine vaporizers, one larger ethane charge preheater and one additional reactor with cyclone. The expansion of the hydrochloric acid absorption plant to six 150 mm. glass tubes with water jacket cooling which was started in the spring was finished in November. A second compressor was installed and the partial condenser of column I was converted to ammonia cooling in order to increase the duty. Column III, used for the fractionation of the product, was enlarged by four meters.

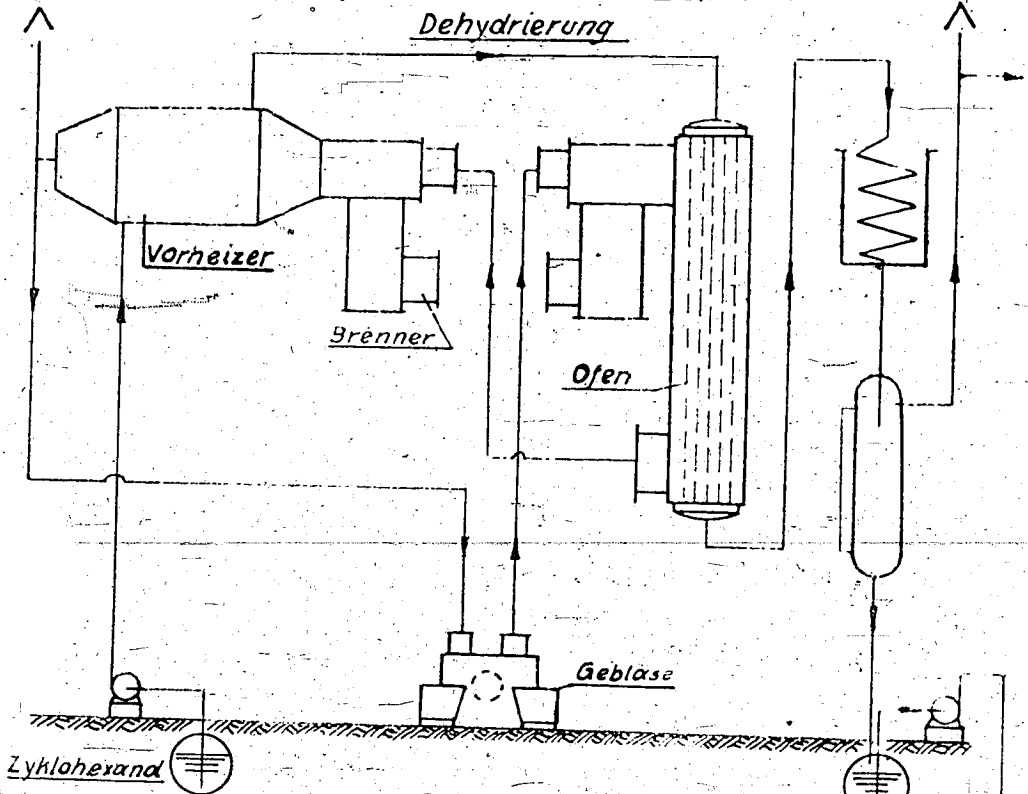
Work for 1941:

For further purification of the product a fourth column is to be installed. The experiments for a ceramic liner for the reactors is being continued.

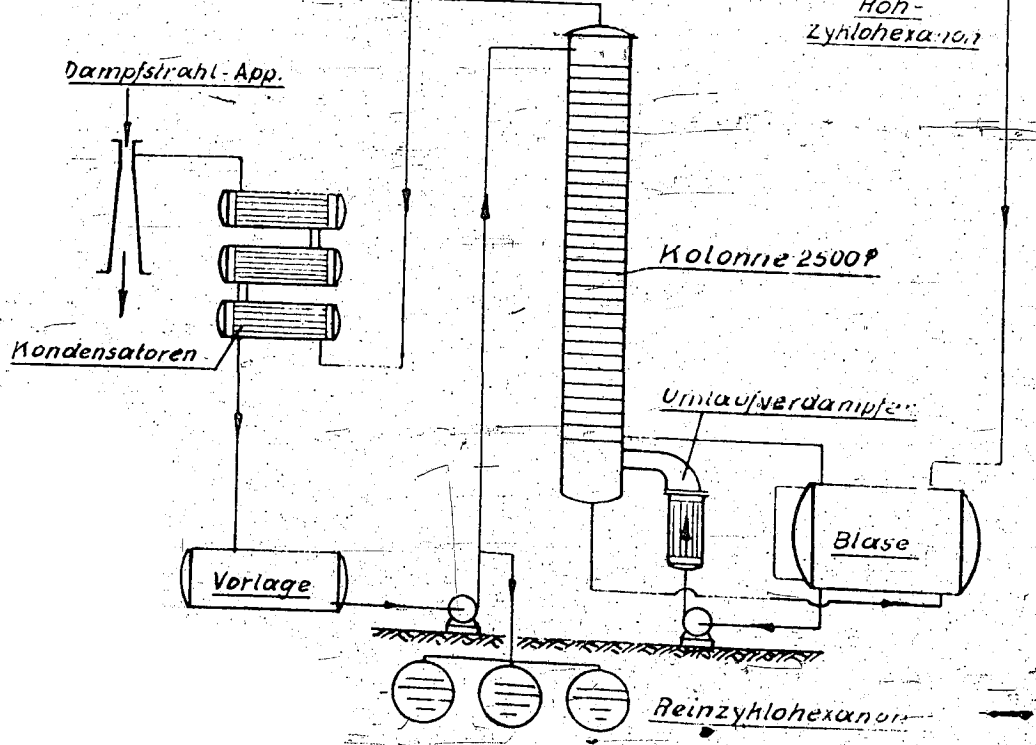
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Zyklhexanon-Anlage

Dehydrierung



Destillation



RESTRICTED

(Drawing opposite Page 64)

Cyclohexanone Plant

Dehydrierung	-	Dehydrogenation
Vorheizler	-	Preheater
Brenner	-	Burner
Ofen	-	Reactor
" Geblase	-	Compressor
Dampfstrahl App.	-	Steam eductor
<hr/>		
Kondensatoren	-	Condensers
Roh Zylohexanon	-	Crude cyclohexanone
Vorlage	-	Accumulator
Kolonne	-	Column
Umlaufverdampfer	-	Reboiler
Blase	-	Still
Reinzylohexanon	-	Pure cyclohexanone

RESTRICTED

(Page 64)

Hexanone Plant

(Managing personnel listed)

Work in 1940:

A new plant was built in Me 975 for the production of cyclohexanone which is used as a starting material for plastics. It was originally planned only for the dehydrogenation of cyclohexanol to cyclohexanone but is used now also for the dehydration of higher boiling alcohols (HS-fraction).

The plant consists of six reactors of which two are completed and one fractionating column which is also ready.

The reactors are tubular and heat is added in the preheater and reactor which is gas fired with circulation.

For the fractionation a bubble plate column of 2.5 meter diameter and 60 trays is available. Fractionation of the reactor product, a mixture of cyclohexanone, cyclohexanol, a little water, phenol and cyclohexene, has to be carried out in a vacuum which is obtained with steam eductors. Fractionation is carried out batchwise.

The production of cyclohexanone has not been started yet since there are still difficulties in obtaining catalyst. An iron-zinc alloy is used.

HS Dehydration

HS fraction is being dehydrated in one of the completed reactor systems since November 1940. The product is fractionated in the bubble plate column and the olefin mix is sent to Me 33 (M₄-production).

Work for 1941:

The startup of one reactor for cyclohexanone is planned for the end of December 1940 or early in January 1941. Four more reactor systems are under construction and will be completed and one reactor with a larger catalyst volume is planned. Furthermore, the installation of one reactor is planned for the

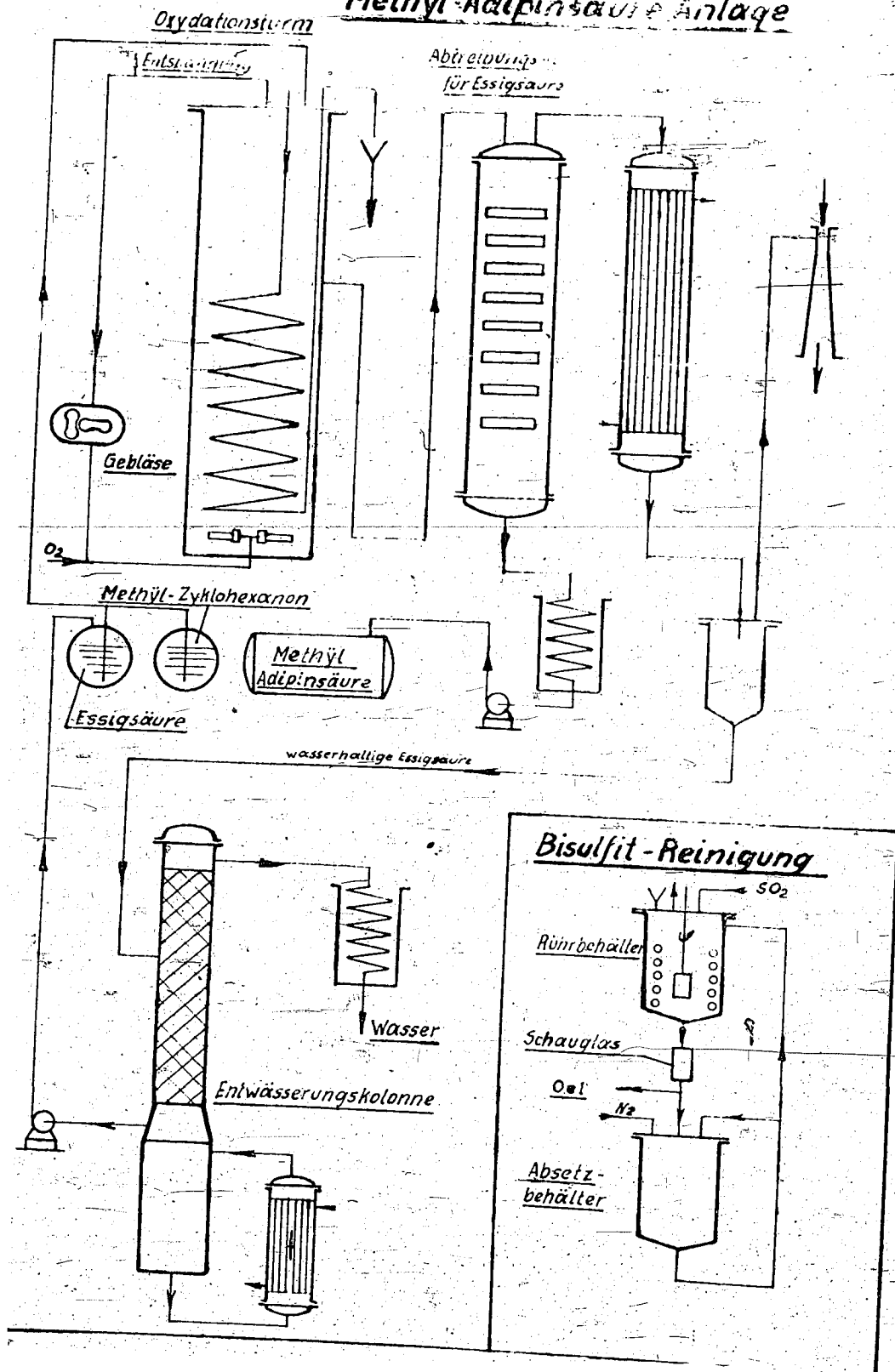
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(Page 64 contd.)

catalytic oxidation of cyclohexanol over silver catalyst. Since the fractionating column will be used later on for the fractionation of crude cyclohexanone, the installation of a packed column (Raschig rings) for the fractionation of the products from dehydration of HS is planned.

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Methyl-Adipinsäure-Anlage



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(Drawing opposite Page 65)

Methyl Adipic Acid Plant

Oxydationsturm	-	Oxidation tower
Entspannung	-	Depressuring
" Gebläse	-	Compressor
Abtreibungskolonne	-	Stripping column
" Essigsäure	-	Acetic acid
" Methyl-Adipinsäure	-	Methyladipic acid
Wasserhaltige	-	Aqueous
Wasser	-	Water
" Entwässerungskolonne	-	Water removal column

Bisulfite Purification

" Rührbehälter	-	Agitator
Schauglas	-	Sight glass
Öl	-	Oil
" Absetzbehälter	-	Separating drum

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Methyladipic Acid

Work in 1940:

Experiments have been conducted on the preparation of ketones from cyclic hexanols and the catalytic oxidation of the ketones to adipic acids.

Dehydrogenation of (methyl) cyclohexanol to (methyl) cyclohexanone

For the catalytic dehydrogenation of cyclohexanol at about 400°C, metallic zinc has been found to be the best catalyst. Because of its low melting point it is used in the form of alloys with nickel or iron. The alloys must not contain any free zinc as mixed crystals. This is eliminated by the addition of 15% iron or nickel. The ketone yield is 80-90%. The separation of cyclohexanol and cyclohexanone is carried out best in a vacuum tower.

Purification of methyl-cyclohexanol with bisulfite

Mixtures of methyl-cyclohexanone and methyl-cyclohexanol are more difficult to separate by fractionation. It was found that the methylketone-bisulfite compound could be decomposed by heat. Dissolved hexanols separate out at low temperature, pure ketones at higher ones (see drawing).

Oxidation of (methyl) cyclohexanone to (methyl) adipic acid

The oxidation of o-methyl-cyclohexanone with oxygen in the liquid phase in the presence of metal salts and fatty acids was investigated. An acid mixture is formed which contains, among others, adipic and methyl adipic acid.

It was found that cyclohexanol and methyl-cyclohexanol can be converted directly to dicarboxylic acids in the presence of aliphatic or hydroaromatic ketones and aldehydes. As an example, a mixture of cyclohexanone and cyclohexanol or a mixture of cyclohexanol and propionic aldehyde, can be oxidized so that the dehydrogenation of the hexanols to hexanones can be largely or completely eliminated.

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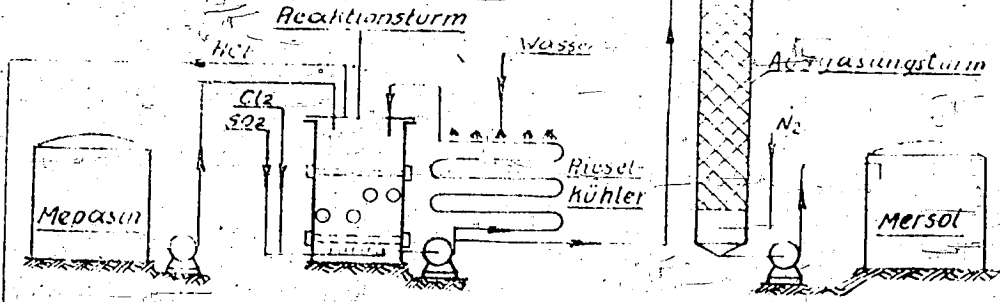
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Work for 1941:

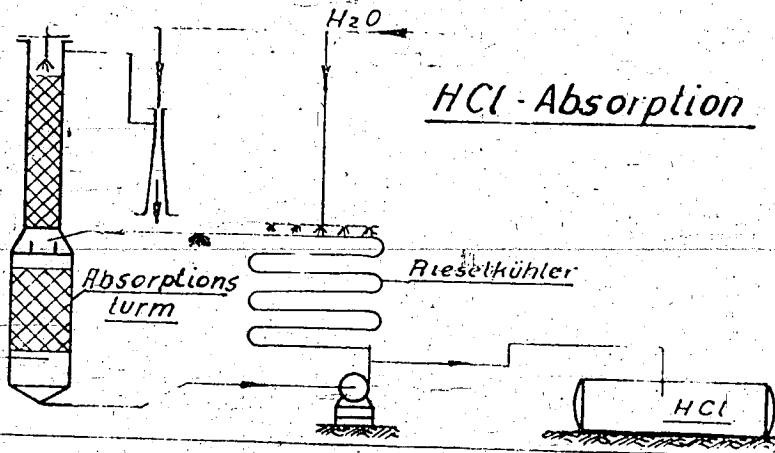
The installation of a large pilot plant with a 6 cubic meter oxidation tower for the production of methyl adipic acid is being prepared (see drawing).

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Sulfchlorierung

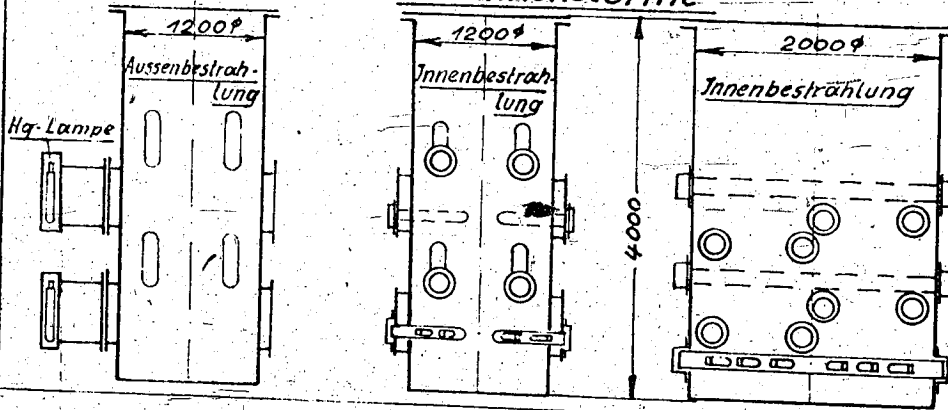


vom Reaktionsturm

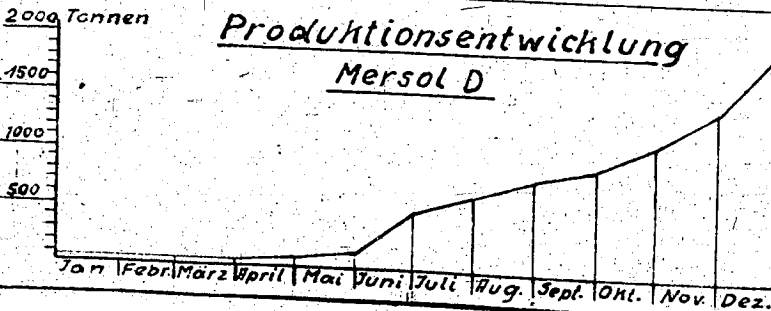


HCl-Absorption

Reaktionstürme



Produktionsentwicklung Mersol D



(Drawing opposite Page 66)

Sulfochlorination

Rieselkühler - Spray cooler
Ausgasungsturm - Gas stripper

HCC Absorption

Absorptionsturm - Absorption tower

Reaction Towers

Innenbestrahlung - Internal radiation
Aussenbestrahlung - External radiation

17

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Mepasin Processing

(Managing personnel listed)

Work in 1940:

A plant for the production of mepasin sulfochloride (Mersol) was built in Me 954 on the basis of experience in the pilot plant Me 947.

Before the war the plant had been planned for 10,000 tons/year of Mersol, for the production of softeners (Mesamoll) and sulfonate (Mesapon).

After the beginning of the war, the difficulties of obtaining raw materials for the soap industry resulted in an expansion of the Mersol production first to 40,000 tons/year (30,000 at Leuna and 10,000 at Wolfen) and then to 80,000 tons/year (50,000 at Leuna).

Plant Me 954 was started up in April 1940 and was producing 2000 tons/mo. by the end of the year. Start-up and operating difficulties were caused by breakage of the ceramic spray coolers and "Igelit" pipes. The mercury-vapor lamps, especially the exterior ones, often fall off because of breakage of the sockets and terminals. The stuffing box problem on the "Durax" pumps has not yet been solved satisfactorily.

Reaction towers were originally used for the sulfochlorination which had a capacity of 80 tons/month with a diameter of 1200 mm and a height of 4 m. Installation of 2000 mm towers increased the capacity to 220 tons/month/tower. At the present time ten 1200 mm and five 2000 mm towers produce 24,000 tons/year of mersol.

Internal illumination has replaced the original external one. The advantages are greater light utilization and safety.

The gas mixture and reacting liquid were processed in concurrent as well as countercurrent flow.

Difficulties were encountered in the disposal of the tail gases consisting of hydrogen chloride, sulfur dioxide, and some nitrogen and chlorine. Hydrochloric

RESTRICTED
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(Page 66 contd.)

acid recovered by absorption could not be disposed of because of SO₂ content. Most of it was dumped in Me 139 after neutralization with lime. Electrolysis of the hydrochloric acid is planned.

Some 50 soap firms were consulted about the processing of mersol.

Work for 1941:

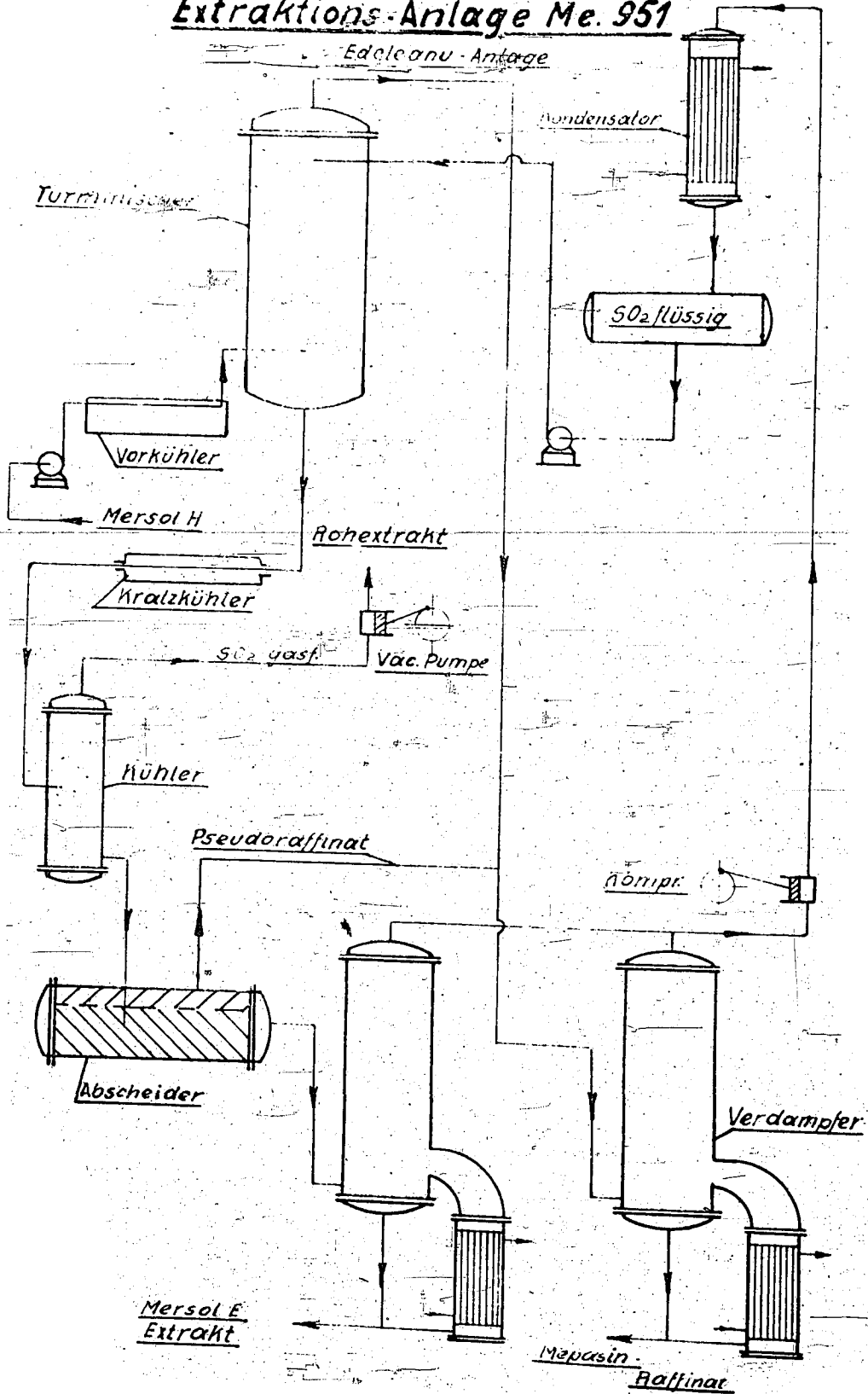
Replacement of the small by large towers is planned. For the final production of 50,000 tons/year, ten 2000 mm and ten 2400 mm towers are planned.

A salable hydrochloric acid of low sulfur dioxide content is planned by hot absorption from the tail gas. It would be desirable to replace the Igelit and ceramic pipes with internally protected (lacquered) steel. Glass coolers have been installed experimentally.

An important goal is to make the Mersol production continuous.

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Extraktions-Anlage Me. 951



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(Drawing opposite Page 67)

Extraction Plant Me 951

(Edeleanu Plant)

Turmmischer	-	Tower mixer
Kondensator	-	Condenser
SO ₂ flüssig	-	Liquid SO ₂
Vorkühler	-	Precooler
Rohextrakt	-	Crude extract
Kratzkuhler	-	Scrape cooler
Vac. Pumpe	-	Vacuum pump
SO ₂ gasf.	-	SO ₂ vapor
Kühler	-	Cooler
Abscheider	-	Separator
Pseudoraffinat	-	Pseudo raffinate
Kompr.	-	Compressor
Verdampfer	-	Vaporizer

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(Page 67)

Mersol Extraction
(Edeleanu Plant)

(Managing personnel listed)

The purpose of this plant is to accomplish a separation of the neutral oils from Mersol H. This is done by liquid SO₂ extraction whereby the sulfochlorides are dissolved in contrast to the neutral ones. A sulfochloride rich lower layer is formed "extract" and sulfochloride poor upper layer "raffinate." From both layers SO₂ is recovered by stepwise vaporization (simplified scheme).

Work in 1940:

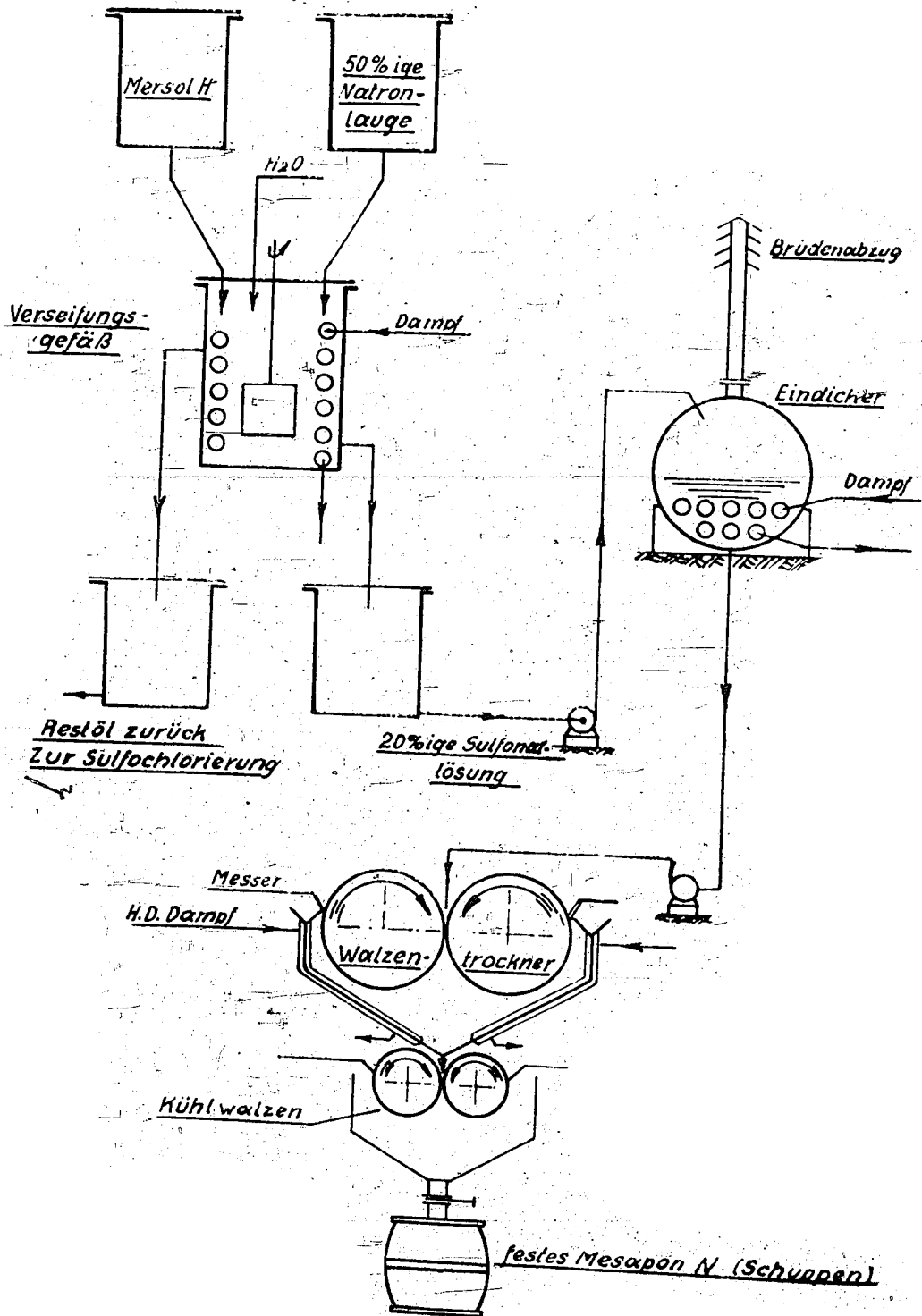
Experiments in semi-commercial scale plant in Me 333a were followed in May 1940 by the construction of a commercial plant by the Edeleanu Company (Berlin) in Me 951. Completion of this plant has been greatly retarded by material shortages. The major part of the machinery and processing equipment, however, is now installed. Some pumps, regulators, and piping are still missing and some insulation and electrical installations have to be made.

Work for 1941:

The plant is scheduled for completion and start-up in February 1941. Planned improvements include inclusion of dryers for Mersol H as well as multi-stage extract coolers for the improvement of extract quality. Filter presses are planned for a possible bleaching of Mersol.

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Mesapon-Anlage



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(Drawing opposite Page 68)

Mesapon Plant

50% Natronlauge	-	50% caustic solution
Verseifungsgefäß	-	Saponification vessel
Dampf	-	Steam
Restöl zurück zur Sulfochlorierung	-	Unreacted oil returned to sulfochlorination
20% sulfonatlösung	-	20% sulfonate solution
" Brudenabzug	-	Vapor removal
Eindicker	-	Concentrator
Messer	-	Knife
H. D. Dampf	-	High pressure steam
Walzen	-	Rollers
Trockner	-	Dryer
" Kühlwalzen	-	Colling rollers
festes Mesapon N	-	Solid mesapon N
Schuppen	-	Flakes

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Mesapon Plant

(Managing personnel listed)

Work in 1940:

The design of the commercial Mesapon N plant in Me 951/952 was based on the experiences gathered in the pilot plant in Me 947.

The quantity of Mesapon required for the textile and soap industry has not been definitely determined. The saponification plant was planned for 250 tons/month of Mesapon N for the time being. Some of the ordered equipment was only delivered after considerable delays or not at all. Therefore makeshift equipment was put up.

The saponification of the half sulfochlorinated mepasin (Mersol H) with 5% caustic was carried out first in an agitator at the sulfate plant. Trouble with iron impurities resulted in a switch to wooden vessels which had not been available originally.

For the concentration of the resulting 20% sulfamate solution a circulating iron concentrator was available. In order to avoid iron equipment, two brick-lined, closed horizontal cylindrical vessels were used to preconcentrate the solution to 35-40%. Excessive foaming in the vaporizer could not be appreciably reduced by blowing with air.

The planned rotating pressure dryer (9-11 atm.) has not been delivered. Therefore a rotating dryer from Wolfen was used which can be used only with steam up to 6-7 atm. The temperature obtained is insufficient for satisfactory drying of Mesapon; therefore electric steam superheaters were added.

The production of Mesapon N could not be carried on fully because of lack of manpower and the dryer revisions.

Work for 1941:

The circulating concentrator, which has not yet been installed shall be lined with baked lacquer. It will contain a special foam-breaking mixer.

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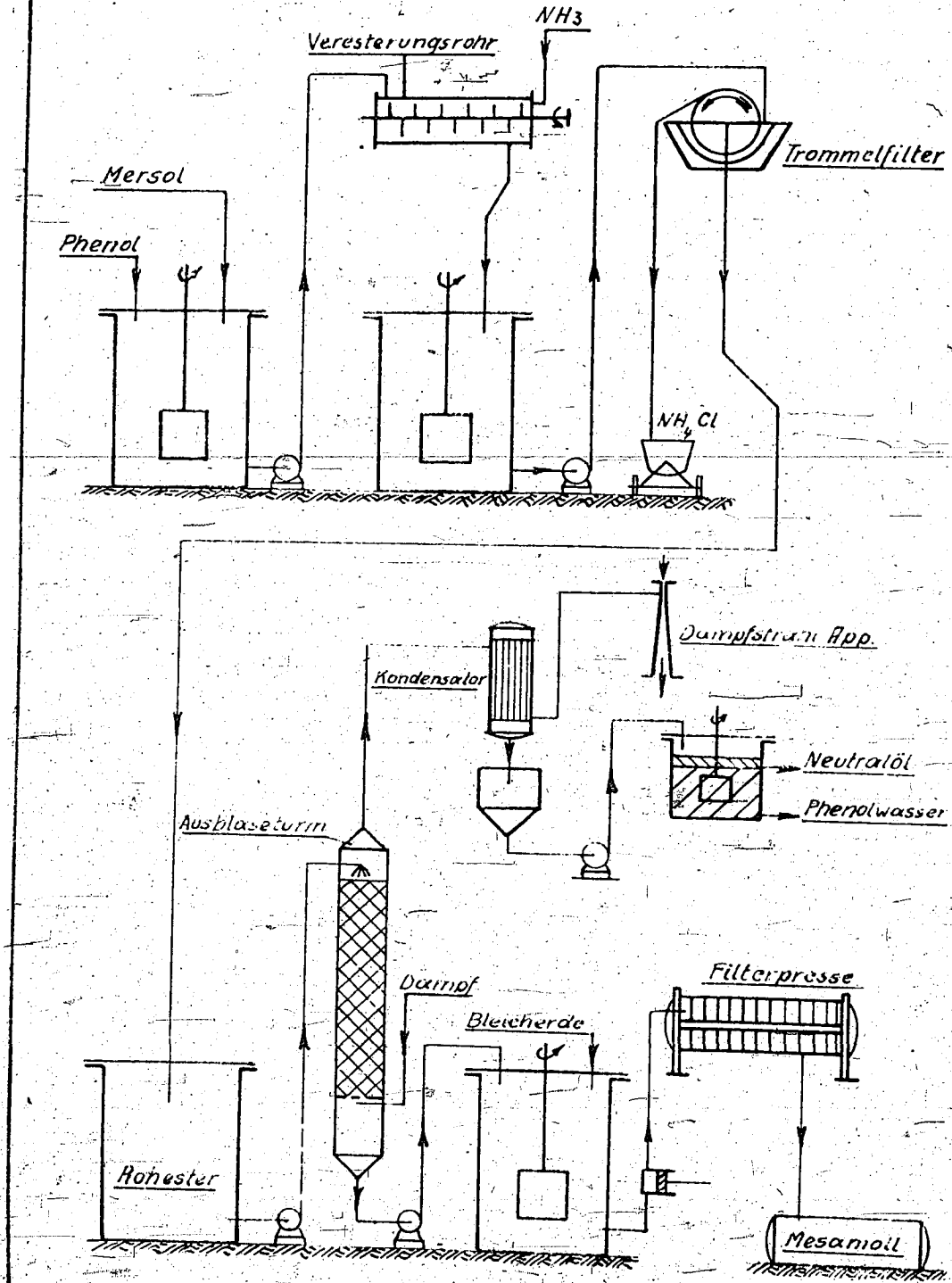
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The planned rotary dryer shall be installed as soon as delivered.

A condenser for the recovery of oil vaporized in the concentrator and in the dryer will be necessary. The water-mepasin mixed condensate will be used in the saponification section.

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Mesamoll-Anlage



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(Drawing opposite Page 69)

Mesamoll Plant

Veresterungsrohr	-	Esterification tube
Trommelfilter	-	Drum filter
Dampfstrahl App.	-	Steam eductor
Kondensator	-	Condenser
Rohester	-	Crude ester
Ausblaseturm	-	Stripping tower
Dampf	-	Steam
Bleicherde	-	Fuller's earth
Filterpresse	-	Filter press
Neutralöl	-	Neutral oil
Phenolwasser	-	Phenolic water

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Mesamoll Plant

(Managing personnel listed)

Work in 1940:

Development of the plant.

The process had been worked out so far in the pilot plant in Me 947 by spring 1940 that the construction of a plant for the commercial production in Me 952 could be started. The plant was started at the end of April with two esterification tubes, 2 drum filters, 2 stripping towers and one filter press.

Two more esterification tubes were added in June and July as well as a second filter press. Thus a capacity of 180-200 tons/month was reached. Since the installed V2A rotary pumps were unable to remove the crude ester from the high vacuum strippers, iron Sihn pumps were installed. No other starting difficulties occurred.

Product Quality.

The following measures improved the product quality which was not satisfactory at first:

- 1) Replacement of Mersol D with 12.5% chlorine to Mersol with 10.5% chlorine as charge and decrease of the esterification temperature from 70 to 40°C.
- 2) Use of mepasin free of cracked material as a charge for Mersol production.
- 3) Two stage stripping of the crude ester.

These revisions permitted the reduction of the viscosity of the product from 90 to 35-40 degrees Engler and to improve its ability to gel.

Waste and Byproducts.

A separator was installed for the clarification of the water and removal of the neutral oils from the stripper mixed condensate containing phenolic water and neutral oil (see drawing). The clarified phenolic water which used to be shipped by tank car for phenol recovery, will be pumped to Me 820 through a newly completed line.

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(Page 69 contd.)

Equipment is under construction for the purification of oily ammonium chloride obtained in the filtration of crude ester.

Work for 1941.

In order to increase the capacity of the stripping section a third tower will be put in operation.

Completion and startup of the salt purification plant.

Experiments for the further purification of Mesamoll are being continued.

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(Page 70)

Isobutyl Laboratory No 19a

(Managing personnel listed)

Work in 1940:

Women generally replaced men departed for the army, successfully.

Analyses:

The analytical section for the control of finished products was expanded. Semimicro elemental analysis was taken up successfully, as well as for sulfur and nitrogen. Refractometric investigation and potentiometric titration were introduced.

Experimental:

Methanol:

Purification methods were investigated which resulted in considerable reduction of water required for methanol distillation.

Isobutyl oil:

Attempts to isolate and purify additional products from isobutyl oil were continued successfully and resulted in the expansion of the HS fractionation as well as the introduction of dioprol and solvent K 33. The latter is a 115-130°C. cut of the ketone fraction of the crude isobutyl oil. This fraction was released by the lacquer commission for sale as a solvent. From a ketone oil from carbonization water sent to us, acetone and methyl ethyl ketone were isolated and suitable purification methods worked out. The experiments have not been concluded but indicate that a usable and cheap recovery process for these compounds from ketone oil is feasible.

Experiments on the dehydrogenation of secondary butanol were completed. Good yields were obtained and a methyl ethyl ketone was obtained which is satisfactory as a solvent.

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(Page 70 contd.)

Olefins:

It was attempted to broaden the olefin basis of the mixed polymer M IV and to prepare a useful dimer, M VII, from the olefin of the diisopropylcarbinol (dioprol).

Esters:

The experiments on the esterification of higher alcohols with Leuna carboxylic acids, started last year, have been concluded. A process was found which permits the commercial production of these esters.

Sulfur containing organic compounds:

Several preparations were made and one, suited for special purposes, was prepared from Leuna alcohols.

Work for 1941:

Besides production control and the experimental projects, experiments for the purification of ketone oil will be continued. Other projects serve the purpose of recovering valuable products from plant materials.

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(Page 71)

Laboratory Me 127 and Experimental Laboratory Me 96b for
the Organic Division

(Managing personnel listed)

I. Lubricating Oil

a) Ethylene lube oils:

The production of lubricating oils from ethylene which in turn is prepared from alcohol, was investigated. It was found that satisfactory ethylene for polymerization could only be obtained if CO formation is avoided in the alcohol dehydration step and if the crude gas is carefully washed with water, caustic and Palatinol HS. This ethylene is 99% pure and yields 80% polymer oil of about 100 V.I. In no way was it possible to improve this value. Therefore the production of ethylene lubricating oil from alcohol is not yet possible. It was shown, however, that this ethylene can be added to Leuna ethylene up to 50% without harm to the product SS 906.

It was further found that the quality of SS 900 lube oil could be improved with respect to life and low temperature behavior by the addition of an ester.

In cooperation with Prof. Dr. Heidebrock, Dresden, it was found that highly polymerized SS 900 products had excellent lubricating properties for gears.

b) Propylene lube oils:

This work was continued. The practical tests of these oils for cable insulating oils have not been concluded.

c) Lube oil inhibitors:

It was found that the tin containing di-isobutyl phenolsulfide-inhibitor, developed last year, could be improved with respect to hydrocarbon solubility if the remaining free hydroxyl group is esterified with Leuna carboxylic acids.

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(Page 71 contd.)

The following projects listed under d) to f) in the mineral oil field are based on the knowledge that the esters, which were primarily prepared and developed by Dr. Löwenberg, would form valuable lubricating oils partly alone and partly in admixture with mineral oils.

d) Railroad axle oils:

The Reichsbahn needed oils stable at low temperatures. Investigations of all those used at present indicated that a blend is more satisfactory than the individual oils with respect to low temperature which can be even further improved by the addition of 12% of an adipic acid ester. Such mixtures are being tested.

e) Special weapon lube oil:

Such low temperature resistant oils are being tested.

f) Automotive lube oil:

It was found that a valuable hydrocarbon lube-oil with a V.I. of 80 could be obtained by hydrogenating $AlCl_3$ -hydrocarbon sludge. Addition of 5% of an adipic acid ester yielded an automotive oil which gave good results. It was further found that by the addition of 20 parts SS 906 and 5 parts of an ester excellent automotive oil could be made from ordinary machine oil. This mixture has been tested.

II. Fuel investigations

Several fuels based on ET 100 were tested at Oppau. Further work was carried out on the development of an ignition point lowering additive for diesel oils. Work was started on the mixed-polymerization (?) of all gaseous olefins with saturated C_3 and C_4 hydrocarbons and the preparation of a high molecular safety fuel.

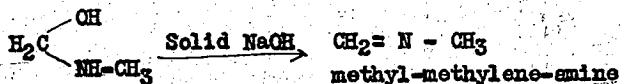
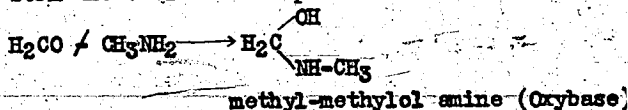
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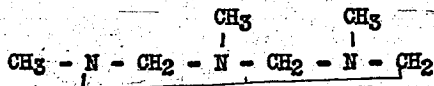
III. Amine projects.

Work was continued on the preparation of vulcanization accelerators by condensation of various amines with Crotonaldehyde. The practical tests at Leverkusen have not yet been completed.

On the basis of the reaction between formaldehyde and monomethylamine, a process for the preparation of dimethylamine was developed. The following reactions form the basis of the process:



This monomer immediately polymerizes to 1,3,5 trimethyl-trimethylene-1,3,5-amine



This trimer is converted to the monomer above its boiling point of 167°C. and it was found that it could be hydrogenated to dimethylamine at atmospheric pressure and 230-240°C. over copper-magnesiumoxide catalyst. The yield is 80%. The remaining 20% consist of equal portions of mono and trimethylamine. It was also possible to hydrogenate the oxybase directly to dimethylamine over the same catalyst with the same yield. A plant for the production of 25 tons/month of dimethylamine is being started up.

IV. Aldehyde condensation:

a) The work on the catalytic reduction of dimethylol propionic aldehyde to trimethylolmethane was discontinued since good yields were not possible and no catalyst of sufficient life could be found. The work on the production of trimethylolmethane from propionic aldehyde and formaldehyde in the presence of

RESTRICTED

(Page 72 contd.)

alkali were carried on in cooperation with Me 494. It was attempted particularly to make the process continuous. The reaction was also tested on other aldehydes. The work on antifoaming agents was continued. A small experimental unit was put up to satisfy the current demand.

b) Work on "Polyaldehyde condensation was continued. Advances were made in the condensation and hydrogenation step.

V. Acids:

Alkali fusion of the high boiling (over 200°C.) alcohols of the isobutyl oil was successful at atmospheric pressure. The yield of carboxylic acids were the same as under pressure. This process was taken over by Me 494.

VI. Esters:

Work was continued on the relationship between chemical structure of esters and their lubricating oil properties. It was found the esters of trimethylolethane and Leuna carboxylic acids as well as the ones of adipic (or methyladipic) acid and isobutyl oil alcohols have valuable lubricating properties. A small experimental unit was built and a design prepared for a 2000 tons/year plant for such esters.

Work for 1941:

Work on the topics discussed above will be continued.

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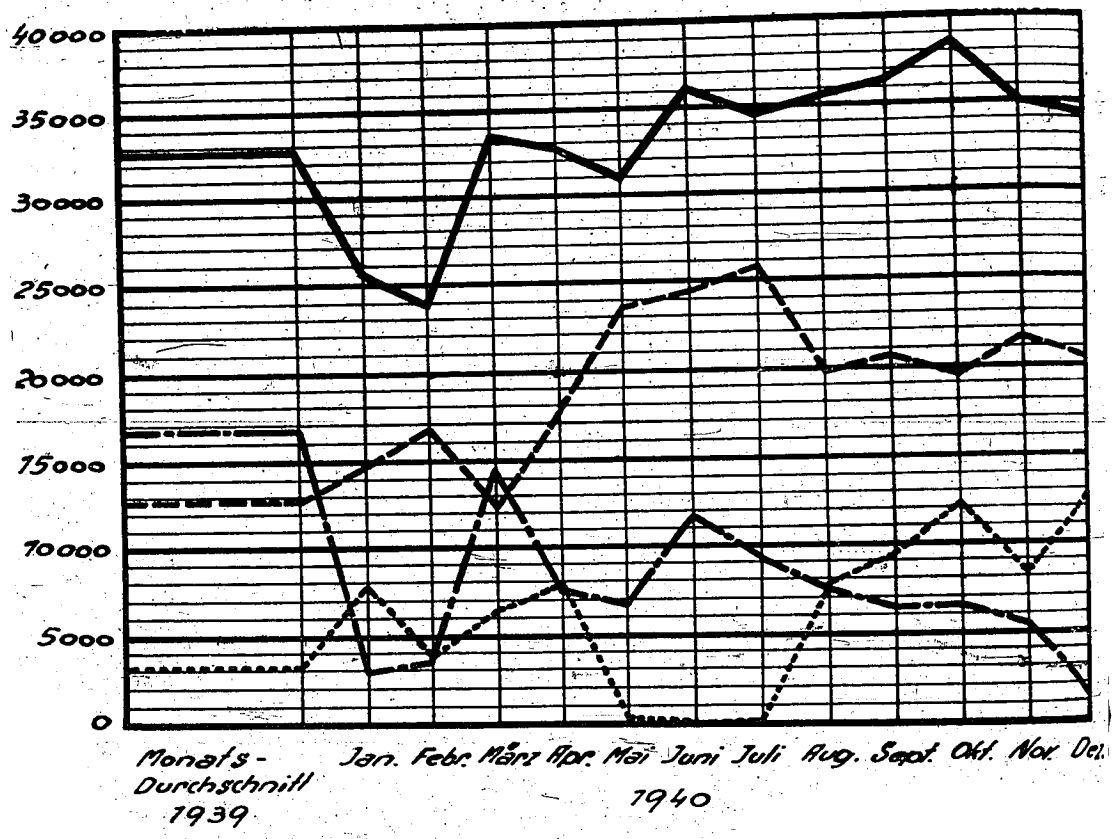
(Page 73)

Lacquer Laboratory

(Nothing worthwhile in this section.)

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Benzin- und Dieselöl- Erzeugung.



- Gesamt
- - - - - Auto-Benzin
- · - · - · Sonder-Benzin
- · · · · Diesel-Öl

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(Drawing opposite page 74)

Production of Diesel Oil and Gasoline

Monats-Durchschnitt 1939	-	1939 monthly average
Gesamt	-	Total
Auto-Benzin	-	Automobile gasoline
Sonder-Benzin	-	Special gasoline
Diesel-Oel	-	Diesel oil

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(Page 74)

Hydrogenation

The increase in the 1940 production of gasoline for motor vehicles and aircraft, and of Diesel oil, as compared with 1939, was only the difference between 393,000 and 397,500 tons; the aviation gasoline factor, however, did show a decided rise, and there was a consequent augmentation in the yield of propane, butane and liquefied petroleum gas. When this additional production is taken into consideration, the over-all increase of hydrogenation production over 1939 is seen to be 6.2%. The products derived from coal showed an increase of from 68.5 to 70% of the total production.

Of the licensed establishments, those in Böhlen, Magdeburg and Zeitz operated without encountering any difficulties. In Magdeburg our new liquid-phase catalyst was tried out and found satisfactory. The paraffin and lubricating oil installation in Zeitz was placed in operation during 1941. The Lützendorf hydrogenation plant will be running from December 1940 on, operated by the Winterhall A.G., after it has been operated for a few months under our own supervision.

Aside from certain interruptions due to enemy air action, the Scholven and Gelsenberg coal hydrogenation plants operated satisfactorily. Processing of coal tar pitch continued in Welheim. This plant and the one in Scholven were enlarged. The Pölitz hydrogenation works came into operation during the second half of 1940. What interruptions were suffered here were likewise due mostly to enemy action.

The Brück hydrogenation plant is under construction, as is also the Rheinbraun plant in Wesseling. Both will start operating some time during the second half of 1941.

The Blechhammer hydrogenation plant of the Oberschlesisches Bergwerk A.G. is being planned.

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(Page 74 contd.)

No further reports have been received, since the outbreak of war, from the Italian installations in Bari and Leghorn.

Persons connected with hydrogenation operations participated in the planning, setting up and putting into service of the works in Lütakendorf, Pölitx, Gelsenberg, Brück, Wesseling and Blechhammer. . . . (Mention of personnel engaged in this work at the places named) . . .

In Leuna, 152 men were made familiar with operations, in a total of 12,800 man-days of training.

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(Page 75)

Reaction-Chamber Operation

(Managing personnel listed)

a) Coal chambers (total of 8)

Work in 1940:

The 27 cu. m. per hour throughput of the reactor, attained in the previous year only as an experiment, was however the normal figure. Indeed, the injection has temporarily been increased to as much as 30 cu. m. an hour.

The life of the reactors could not be increased any further over the maximum achieved in the previous year, because of the serious salt deposits they suffered due to the deterioration of the raw coal; this was not eliminated until recently, by a change made from one area to another in the v.d. Heydt Mine and a reversal of the ratio of v.d. Heydt coal to Elise coal.

The reactor I type of de-sander has now been installed in all reactors and given good satisfaction. The operating time is more or less equal to that of the corresponding chambers (300 days). The automatic device for the removal of the sand produced has been installed in one of the chambers and is planned for all of them. By mixing the sand sludge with normal sludge there was a reduction of the clogging and wear in the sludge pumps.

The automatic device for constant-level control of the sludge separator has now been very successfully introduced into all the chambers.

The two devices--reactor I type of de-sanding and automatic constant-level control--have contributed most decisively to uniformity of operation.

To effect a more reliable maintenance of temperature in reactor I, a device for adding cold gas was installed between the preheater and reactor I, and the first element in this reactor was also equipped with an alarm for maximum temperature.

RESTRICTED

RESTRICTED

(Page 75 contd.)

The tests with Rhenish brown coal at 650 atm. were concluded. By an addition of sulfur it was possible to reduce the formation of residue, which occurred only during the first days of operation.

Work for 1941:

1) Continued experiments to increase throughput and extend the life of the chambers. To exclude the possibility of serious erosion, tests with smaller quantities of gas are planned.

2) Installing automatic de-sanding equipment in all chambers.

3) Increasing the sensitivity of the temperature measurement at the opening of reactor I to keep the temperature constant. Installing an automatic regulator for the cold gas at that place in order to prevent coking at the bottom of reactor I.

4) Using N5 for the liners in the sludge separator, so as to be able to use them over again after disassemblage.

b) Tar chambers (1)

Work in 1940:

As molybdenum is now hard to get, the molybdenum catalyst used in the operation was successfully replaced with one of iron, this being in line with the results derived from experience and from research done in laboratory 869. The other hydrogenation plants may follow suit.

After thorough tests made in the distillation outfit for a way to preserve the fireproof masonry in the combustion chamber of the preheater, a burner was installed which has an opening through which a circulating gas can be added to the hydrogen fuel gas and by which the temperature of the flue gas in the combustion chamber can be held below 1400°. This has proved eminently satisfactory.

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(Page 76)

The effect of the higher cut point of the middle oil A, and of the higher temperature in the coal chambers due to the increase in throughput, has been to do away with the excess of heavy oil which used to exist; there is in fact too little grinding oil. Since in Lema heavy oil is brought into the hydrogenation process by the tars, it can be utilized as a grinding oil and the shortage of grinding oil be thereby compensated for. The amount of heavy oil from tar that is furnished to the coal liquid phase means that much easing of the tar reactor and its freeing for the reception of other products. Since new products are however not to be had on the market, the chamber is being operated on a temporary basis.

The three compartments of the furnaces were made interchangeable.

- c) Gasoline chambers (9 - i.e., 5 with 5058 catalyst,
4 with 6434 catalyst)

Work in 1940:

Chambers with 5058 catalyst.

An addition of water made possible a further lengthening of the life of the regenerators. Corrosion in the lines leading to regenerator II, caused by concentrated ammonium chloride solution, made it necessary to again abandon the injection of water onto the lower tube sheet of regenerator I, but an improved nozzle has been introduced to supply water to the upper tube sheet of regenerator II.

The efficiency of the 5058 chambers is limited by the resistance offered. The following measures were accordingly resorted to in one of them to reduce resistance: changing the direction of the current of gas in the furnaces to a downward one, replacing the 500 mm. regenerators with 600 mm. ones, and, for the first time, equipping the electric preheater with tubes of 120 mm. diameter to replace the 90 mm. ones thus far used.

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(Page 76 contd.)

Whether or not this arrangement is successful will not be apparent until the next catalyst re-charge, as the present catalyst, now several years old, had been causing additional resistance in the reactors owing to formation of dust.

Chambers with 6434 catalyst.

Small-scale experimentation had shown that ammonia and organic bases have an unfavorable effect upon the catalyst, as was confirmed by tests on a large scale, in which the ammonia was washed out of the gas entering a 6434 chamber, to below 0.1 mg. to the cubic meter. The middle oil B utilized to wash the expansion gases out of the coal chamber takes up ammonia and organic bases in the process, with the result that it also impairs the 6434 catalyst, as was demonstrated by a further large-scale test. In addition to the scrubbing with water of the recycle gas, an attempt is to be made to free of bases all middle oils that have to pass over 6434 catalyst, by washing them with water or dilute acids. To free the 6434 catalyst a heavier middle-oil A fraction will be used in place of the middle oil B for gas scrubbing, and it will then be refined over 5058 catalyst.

Chamber 13, operated thus far as a pre-hydrogenation chamber with an electric preheater, was equipped with a gas preheater and arranged for 6434 catalyst.

Work for 1941:

- 1) Erection of a water scrubbing outfit, in which only the gas for the 6434 chambers is to be scrubbed. This will reduce the loss of pressure for the 5058 chambers and make possible a larger throughput with it.
- 2) Further tests for freeing the middle oils B from ammonia and organic bases by scrubbing with water or acid.

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RESTRICTED

(Page 76 contd.)
(and Page 77.)

3) Development of an automatic regulating device to keep certain elements constant in the reactors.

d) Low-temperature hydrogenation chambers (1, up to June 1940)

Work in 1940:

The tests were continued and concluded. The chamber is being employed for 5058 gasoline production.

e) Miscellaneous

Work in 1940:

In the gas-phase cycle a by-pass has temporarily been installed around the scrubber, and a separator in the purified-gas unit, the result being to reduce the resistance to flow, bring about a satisfactory blending of the re-cycle gases with the fresh gas, and protect the circulating pumps against entrained liquid.

Safety valves were installed in the fresh-gas lines, controlled by oil pressure, to prevent any pressure release in the gas lines connecting with the chambers in the event of a violent break in those lines.

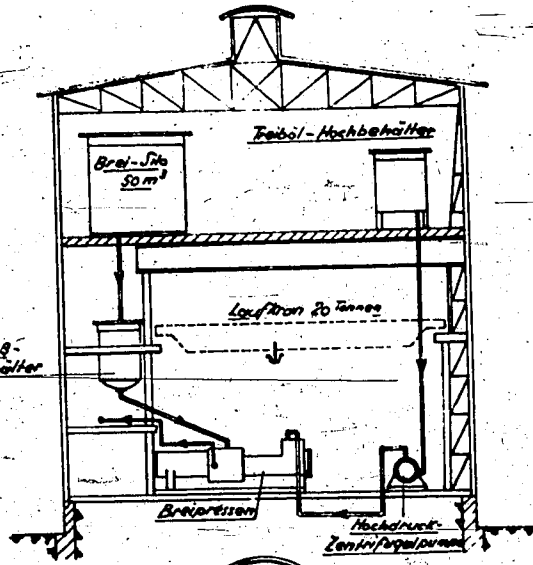
The central release for the coal and gasoline separators were combined and enlarged in Me 809.

Work for 1941:

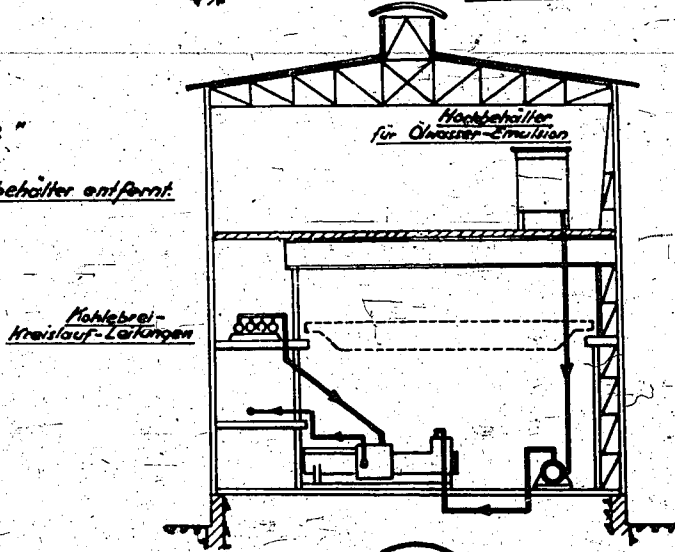
Installation of further electrically-controlled gate valves in the circulation lines.

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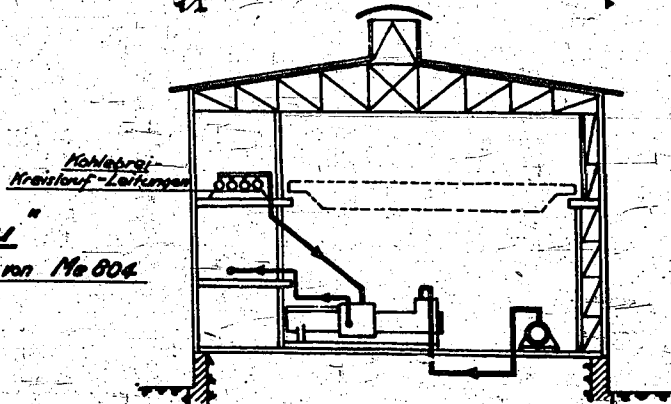
"alt"
mit Silo & Meßbehälter



"alt"
Silo und Meßbehälter entfernt



"Neubau"
des zerstörten Teiles von Me 804



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(Drawing opposite page 78)

<u>"alt"</u> <u>mit Silo & Messbehälter</u>	-	<u>Old building,</u> <u>with charge storage and meas. tank</u>
Brei-Silo 50 m ³	-	50 cu. m. paste tank
Treibol-Höchbehälter	-	Elevated power-oil tank
Messbehälter	-	Measuring tank
Laufkran 20 Tannen	-	20-ton overhead crane
Breipressen	-	Paste injectors
Hochdruck-Zentrifugalpumpe	-	High-pressure centrifugal pump
<hr/>		
<u>"alt"</u> <u>Silo und Messbehälter entfernt.</u>	-	<u>Old building,</u> <u>storage bin and meas. tank removed</u>
Hochbehälter für Ölwasser-Emulsion	-	Elevated storage tank for oil-and-water emulsion
Kohlebrei-Kreislauf-Leitungen	-	Circulation lines for coal paste
<hr/>		
<u>"Neubau"</u> <u>des zerstörten teiles von Me 804</u>	-	<u>New building</u> <u>to replace destroyed part of Me 804</u>
Kohlebrei-Kreislauf-Leitungen	-	Circulation lines for coal paste

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Paste Injectors, Scrubbing Plant, Tail-Gas from h.p. Hydrogenation, Circulation Pump

a) Paste injectors

Work in 1940:

A new electrically-driven injection pump was put into operation, also another centrifugal pump for liquefied-petroleum power oil.

To save metal there was further replacement of metallic stuffing boxes with soft packings made of cellulose and buna.

It was possible to procure and install new and more efficient rotors and control devices for the high-pressure centrifugal pumps required to operate the paste injectors by hydraulic power, with consequent 10% increase in the production of liquefied-petroleum power oil by the close of the year.

During the attack in the night of the 28th to the 29th of August a bomb hit a middle-oil storage tank on the top platform of building 804 and set it afire; about half the building was destroyed, the fire being fed continually by the destruction of further stores of oil.

To eliminate in the future the possibility of damage due to the burning of inflammable liquids stored in the building, the plan of reconstruction of building 804 will provide for the middle-oil charge tanks and screening unit to be erected outside and for the products to be conveyed from there to the paste injectors. Similar arrangements are to be effected in buildings 803 and 805.

Special devices will have to be adopted in connection with the direct delivery of coal paste, in view of the absolute necessity to prevent its standing for any length of time in any one section of pipe; otherwise it settles, and the line is rendered inoperative. Coal paste must therefore be run in a "cycle"-- the steam pumps bringing it out of the storage tank must send it in such a way as to by-pass the paste injectors and insure part of it getting back into the storage tank. Each paste injector draws the required amount of paste from the recycle circuit, through a tap line. Measuring vessels are no longer needed.

(see drawing)

RESTRICTED

RESTRICTED

(Page 78 contd.)
and Page 79.)

The middle oil B has for quite a while been run to the pumps without any charge tanks being connected in between. Corresponding lines for the middle oil A are provided. Technically, the direct pumping of middle oils to the injection pumps offers no difficulties.

As a measure of protection against air attack, the power oil used to drive the paste injectors was replaced by water to which is added 5% of "Muzin" (an anti-corrosion agent with an emulsifier). The results have been very satisfactory

After a necessary 7-day complete shutdown of building 804 the injection of middle oil was resumed, and after a fortnight it was possible to put the plant back into full operation. Within another week part of the coal-paste injection mechanism could be set operating again. By resort to these quick measures and by radical restrictions effected with respect to low-temperature hydrogenation, hot circulation and elimination of tar chambers, it was possible to keep the fall-off in production extraordinarily low--to something like 13% in 12 days.

Work for 1941:

1) Shutdown of the remaining charge tanks used for tar, coal paste and catalyst sludge-removal. Installing of circuit lines from which the pumps draw the injection product. As an emergency reserve for grinding oil and as stock tanks for fresh product and catalyst sludge-removal, 4 tanks are provided, in direct connection with building 805.

2) Installation of more new centrifugal pump outfits, with consequent further 20% increase in the total production of power oil.

b) Scrubber plant.

Work in 1940:

The cylinders of another of the depression outfits were strengthened with reinforcing rings.

RESTRICTED

RESTRICTED

(Page 79 conti.)

To one of these outfits was attached an automatic safety device to prevent the wash oil and recycle gas from getting out of the high-pressure system in the event of the breakage of a cylinder.

On one of the depression outfits there was a change made from ordinary valves to slide valves; this does away with the stuffing boxes, the operation becomes cleaner, and the objectionable odor is no longer present. The slide-valve arrangement already used for copper liquor has so far given a good account of itself also for wash oil.

The water-scrubbing outfit for the gas-phase recycle gas was set in operation.

A start was made on equipping the high-pressure scrubbers with electric level indicators.

Work for 1941:

- 1) The third and last depression outfit to be equipped with reinforcing rings.
 - 2) Conversion to slide-valve equipment on the other depression outfits.
 - 3) Equipment of all high pressure scrubbers with electric level indicators.
- c) High-pressure tail-gas installation

It was found impossible to deliver as yet the third lot of blowers figured on.

An unenclosed burner designed to make possible the invisible combustion of hydrogenation recycle gas, up to 15 million heat-units per hour, was developed and delivered to the plant. Further tests will be made, to determine whether to build a large number of such burners, to burn up all this gas without any visible flare in the event of an air attack.

Work for 1941:

Setting up the third lot of blowers.

- d) Gas-circulation pumps

RESTRICTED

RESTRICTED

(Page 79 contd.)

Work in 1940:

The ninth gas-circulation pump, the first electrically driven, was put in operation. This electric drive has proven satisfactory. Owing to the absence of the connection with a second electric plant, safety considerations made it necessary to run the pump on the gas circuit at only 50% capacity.

The lower stuffing box, used for the first time, has so far given satisfaction.

The new building, to be used for further gas-circulation pumps, is under construction.

Three shatter-proof walls have been erected in the old building as a protection to machinery and attendants.

Work for 1941:

- 1) Connecting the electric circulation pump to the second electric plant.
- 2) Getting new building ready and setting up two new circulation pumps.
- 3) Transfer of one of the North circulation pumps into the annex.
- 4) Erection of a double fire-wall in the ^{old} building.

e) Hydrogenation recycle gas

Work in 1940:

To prevent the gas by which the compressors are operated from getting into the hydrogen-rich gases, the expansion-poor gas from the gasoline separator was fed directly to the hydrogenation fuel-gases instead of being delivered to the power gas distributor. With CO₂ it is brought to a calorific power of 2,200 heat units.

RESTRICTED

RESTRICTED

(Page 80)

General, for h.p. hydrogenation

Work in 1940:

. . . (Details of safety measures adopted for equipment and personnel in high-pressure hydrogenation plant under emergency conditions. Increased air-raid shelters, protective walls, etc.) . . .

RESTRICTED

RESTRICTED

(Page 81.)

Me 869 Small-Scale Experimentation

(Managing personnel listed)

Work in 1940:

I. Gas-phase experimentation with small reactors.

Continuous investigation of materials utilized in the operation (catalysts and products).

Investigations to determine the utilizability of new catalysts under local operating conditions. Miscellaneous carrier-catalysts were used as pre-hydrogenation catalysts, with better results than with 6719 (7525, 7675, 7360); the tests are still in progress.

It was found that the water scrubbing of the injection product, as well as of the recycle gas, is advantageous for 6434.

A number of different high-boiling petroleums and lubricating oils of (Politz, Zeitz) were used for gasoline production with 5058 with good results.

A series of removed catalysts were investigated, for the Brabag, to determine whether they could be used over again.

II. Experimentation with kilo reactor

Continuous investigation of products and catalysts for use in the tar and coal reactors.

The possibilities and conditions with respect to "Brixⁿ tar" in the liquid phase were investigated. Owing to its sensitivity to temperature the tar cannot be distilled and so has to be run without being topped. Bad iron incrustations were observed last year; these are considerably reduced by an addition of 0.5% of sulfur to the injection product. The yield is high.

Low-temperature hydrogenation paraffin, which was unsuitable for oxidation to fatty acid, was hydrogenated with 5058 at low temperature to a satisfactory degree of purity.

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(Page 81 contd.)

To avoid the use of molybdenum for the liquid phase, the initial experiments were again effected with iron. There was confirmation of the results therein observed, according to which the iron catalyst (generator dust impregnated with FeSO_4) was found inferior to molybdenum. It is however possible to achieve still further improvement by adding the equivalent quantity of caustic soda on occasion of impregnation. So far the efficiency obtained has been 85-90% of that of the molybdenum catalyst. The tests in question were carried out for both Leuna and Böhlen, under the corresponding operating conditions.

An investigation of the temperature sensitivity of various tars was made, and certain rather surprising determinations reached which will be followed further, for both the low-temperature hydrogenation process and the distillation of tar.

By hydroforming, larger quantities of 5058 gasoline were prepared for the testing laboratory, with a view to further processing; larger amounts of unsaturated Rumanian Diesel oils were also hydrogenated for the preparation of "Mepasin."

Work for 1941:

- 1) Further prosecution of tests now in progress.
- 2) Processing, in the gas phase, of the "Brux middle oil" obtained in the liquid phase.
- 3) Performance of the experiments originally planned for 1940.
- 4) Investigation of sulfur-treated swamp-ore from Me. 431, for coal hydrogenation.
- 5) Experimentation to improve the catalyst used for tar and coal hydrogenation.

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Me 870 Oil-Manufacture Laboratory

(Managing personnel listed)

Work in 1940:

Assignments.

Observation and supervision of operations and of small-scale experimentation; investigation of products yielded.

With respect to operational activities, investigations were carried on in the following fields:

- 1) During the past two years an increase has been observed in the amount of alkali salts and magnesium salts, together with calcium carbonate, deposited in the connecting lines from reactor I to reactor II of the coal chambers. These phenomena were found to be related to the rise in the alkali content of the v.d. Heydt coal. The worst crusting observed was during and shortly after the time when the alkali content of the v.d. Heydt coal had reached its maximum. There was a reduction again after the alkaline coal was replaced by a v.d. Heydt coal with only a small alkaline content.
- 2) As a substitute for the iodometric H_2S determination, an equivalent titration method was worked out, based on the oxidation of the hydrogen sulfide in alkaline solution with perhydrol, to sulfate, and on the acidimetric determination of the excess of alkali. This method, with the proper modifications, is now being applied to gases, liquids and solid substances.
- 3) The float-and-sink sand determination in brown coals gave too high a figure in the case of coals mixed with purifying agents. The method was improved by a subsequent varied chemical treatment with concentrated sulfuric acid, caustic soda solution and dilute hydrochloric acid.
- 4) The centrifuge residue from Böhlen tar sludge was investigated in the laboratory with a view to recovery of the molybdenum by the sulfuric-acid process. An 80% regeneration was found to be possible.

RESTRICTED

RESTRICTED

(Page 82 contd.)

5) The sensitivity of the 6434 catalyst to the action of nitrogen compounds as observed at other plants was confirmed. To keep as low as possible the de-activation of the 6434 catalyst due to such nitrogen compounds,

- a) the recycle gas is refined by an inserted water scrubber,
- b) the B product used as wash oil for liquid-phase waste gases is freed, also by washing with water, from organic nitrogen bases and ammonia, before it passes over the catalyst.

These measures were preceded by the necessary preliminary studies, etc.

RESTRICTED

RESTRICTED

(Page 83)

(& P. 84)

Plans and Calculations for Distillation and Gas-Separation
Plants

. . . (Mention of establishments which were consulted, or with which orders were placed, in connection with items of equipment for various processes) . . .

RESTRICTED

RESTRICTED

(Page 85)

Coal De-Sludging, Centrifuging, Distillation

(Managing personnel listed)

a) Centrifuging

Work in 1940:

There was a continuation of the conversion operations on the Sweden centrifuges, begun some years ago.

Of the 47 existing machines, 39 have cyclo gearing (2 new ones in 1940)

46 have planetary gear and large-sized injection rings (12 new ones in 1940)

28 have synthetic-resin bearings (6 new ones in 1940)

To enlarge the plant 12 machines were installed in Me 884. The motors have not yet been delivered.

Experiments were continued with the recording density indicator used for the sludge from the coal chambers. The float type of gauge was replaced by a pneumatic device.

There were test-circuit experiments with the two-stage centrifuges. In the first stage the material entering with 12% solids was run to 20% residue. In the second stage this residue was concentrated to 45%. On the average, a centrifuge oil containing less than 1% of solids was obtained. The character of the residue however still leaves something to be desired.

Walled-in places were provided as a protection to the operators in the event of air attack.

Work for 1941:

- 1) Continued conversion of Sweden centrifuges.
- 2) Continued experimentation with the recording density indicator.
- 3) To prevent interruptions due to increased precipitation of sand from the dilute sludge, two new de-sanding tanks are to be installed and the present manual loading arrangement for the separated sand is to be made automatic.

RESTRICTED

RESTRICTED

(Page 85 conti.)

(and Page 86.)

b) Distilling

Work in 1940:

. . . (Bad falling off of production due to severe strain placed upon distilling plant, aging of parts, persistent hard frost, etc.; difficulties overcome, however, by speed of repairs made, careful supervision and thorough-going renewal of worn materials, with the result that by the 1st of March 1940 full production had been achieved; comparison showed a marked increase over the same period of last year) . . .

The operating time of the parts of the outfit is substantially lengthened, over what it was the preceding year: the lower coils were operated 73 days, the upper ones 263 days (average 115).

The numerous shutdowns that had to be effected during air-raid alarms were tolerated without noticeable impairment of the outfit.

Wattmeters were installed on 6 of the motors driving the reactors, to detect in time any resistance developing.

After a series of lubrication tests with calypsol and superheat steam oil, 6 of the cyclo gears were fitted with an engine-oil circulation system; this was entirely successful.

A flow controller was installed on reactor I and gave good satisfaction. Even after 80 days operation the error was only 4%.

The direct distillation of the sludge was resumed with success, to reduce the amount of asphalt brought back over the grinding oil into the coal chambers. At times as much as 30% of sludge was distilled directly.

Owing to the serious impairment of the quality of the conveyor belts, repairs had to be made on it quite a number of times during the year.

RESTRICTED

RESTRICTED

(Page 86 contd.)

No satisfactory results were achieved with the experimental distilling of sludge in the test reactor, even effected after a reduction in the incline. Scholven grinding bodies were therefore used instead of the rods. The test reactor was also equipped with a dry discharge arrangement.

Two new reactors (21 and 22) are to be put in readiness shortly after the first of the year.

Several bomb-proof stations for operatives were arranged, and the building housing the telephones and transformers was surrounded with a wall.

Work for 1941:

- 1) Attachment of further regulators for the charging of the worm reactors.
- 2) Starting work on erection of reactors 23 to 26.
- 3) Alterations in oil-vapor lines of reactors 9 to 20.
- 4) Experiments to lengthen life of grinding devices in worm reactors.
- 5) Tests with dry discharge device.
- 6) Distillation of sludge in experimental reactors using Scholven grinding bodies.

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Preparation and Distillation of Tar

(Managing personnel listed.)

Work in 1940:

Of four Haubold centrifuges taken over from the Welheim hydrogenation works, three were set up and put into service. The fourth still lacks the motor.

The Haubold waste-oil centrifuge was equipped with a larger-sized shaft; however, the expectation that this would result in a satisfactory functioning of the outfit at full speed was not entirely fulfilled.

The present air-tight arrangement, on all centrifuges, using carbon dioxide protection, does away with a condition which is frequently a cause of fire.

A new and more powerful suction unit for the ventilation of the centrifuge was put into operation on the south side of the building, with resulting substantial improvement in this respect.

The depositing of residues in the raw-tar storage tanks, which caused clogging and consequently damaged the agitator, has been entirely eliminated by using the pump to draw from the bottom of the tank. The two tanks have now been operating uninterruptedly for 200 days without any trouble developing.

An average of 950 tons a month of Koppers tar (fines content 3.4 to 8.4%) from the Schwarzheide Brabag plant was processed. The centrifuges without acid did not work satisfactorily. In dilution with 1 to 2 parts of other distillation tars, and after an addition of 0.2 to 0.6% of SO_3 , it was possible to centrifuge as much as 0.15-0.3% of dust.

Using the two outside centrifuges there was no difficulty experienced in processing the quantity of waste oil (1050 tons a month), which was substantially above the 1939 average of 890.

Work for 1941:

1) Improvement in design is expected to insure satisfactory waste-oil centrifuge operation at full speed and even with products of lesser specific gravity.

RESTRICTED

RESTRICTED

(Page 87 contd.)

2) Better caulking of the four Welheim Haubold centrifuges; satisfactory storage arrangements for these.

3) Installing a mechanical removal device for the residue in all five of the Heine centrifuges.

4) Acid-proof coatings to protect the raw-tar and waste-oil storage tanks against corrosion. Installing acid-resistant pumps.

b) Tar filtration.

Work in 1940:

Filtration experiments with filter tubes have shown that the effect of a protracted exposure to the open air is to cause the continued further formation, from a filtered tar, of gasoline-insoluble solid polymers which act to reduce the filtration speed considerably.

It is not as yet possible, from the results of analyses, to draw any conclusions as to the filterability of a tar from its composition or physical characteristics.

Large-scale tests aimed at the thorough removal of solids by passing even the centrifuged normal tar, including Koppers tar, through the filter press, with a view to economizing catalyst in high-pressure reactor hydrogenation, have not so far proved rewarding. A tough "skin" a few millimeters thick very soon forms on the paper covering and effectually prevents any further passage.

Work for 1941:

Further tar-distillation experiments.

RESTRICTED

Catalyst Manufacture

(Managing personnel listed)

Work in 1940:

a) Catalyst for liquid-phase reactor

A special plant was erected for the preparation of iron catalyst 10927, which was made of ferrous sulfate and caustic soda and which has been used in place of the molybdenum catalyst since March 1, 1940 in the tar liquid phase. This makes it possible to prepare both catalysts simultaneously. The Brabag-Werk Magdeburg was also supplied with catalyst 10927.

The requirements of the Böhlen, Magdeburg, Welheim and Lützkendorf works were taken care of with dry catalyst 11002.

A loading outfit for wet catalyst 11002 for the Pölitz hydrogenation plant was completed and put into operation.

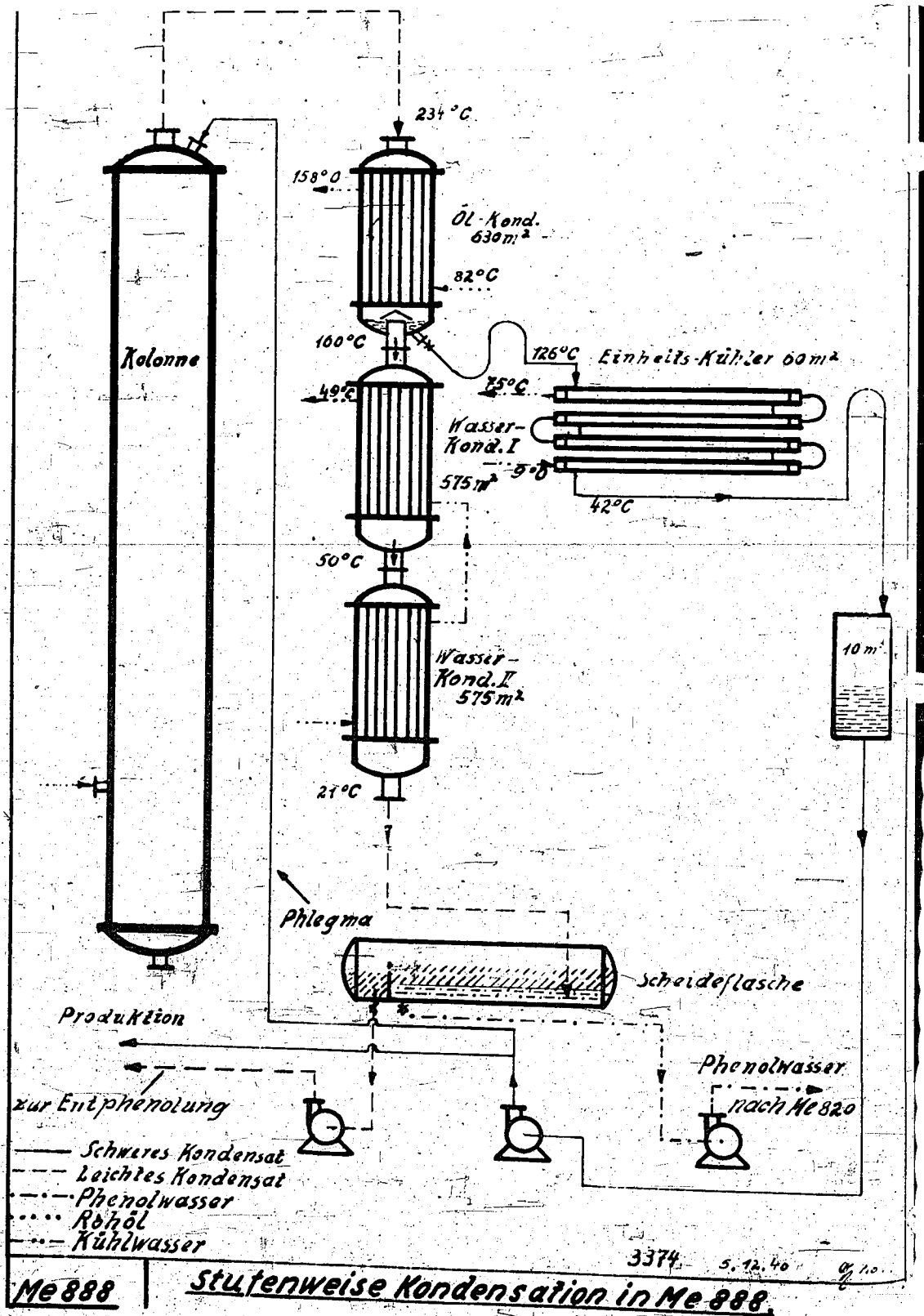
b) Pellet presses

The two large Kilian presses were delivered to catalyst factory Me 22b in October.

Work for 1941:

- 1) Erection of a new 100-ton stock bunker for catalyst 11002, made necessary by increased drawing off of supplies for new hydrogenation plants.
- 2) Erection of a fourth mixing worm (with rubber).
- 3) In view of the absence of any storage facilities as yet for catalyst 10927, the present 25-ton stock bunker for 11002 is to be converted.
- 4) If necessary, erection of a third drying oven.

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(Drawing opposite page 89)

<u>Me 888</u>	-	<u>Staged Condensation in Me 888</u>
Kolonne	-	Column
" ⁿ Öl-Kond.	-	Oil cond.
Wasser-Kond. I	-	Water Cond. I
Wasser-Kond. II	-	Water Cond. II
Phlegma	-	Reflux
Produktion	-	Production
Einheits-Kühler 60 m ²	-	60 m ² tube
Scheideflasche	-	Separating vessel
Phenolwasser nach Me 820	-	Phenol water going to Me 820
zur Entphenolung	-	For phenol removal
Schweres Kondensat	-	Heavy condensate
Leichtes Kondensat	-	Light condensate
Phenolwasser	-	Phenol water
" Rohöl	-	Raw oil
" Kühlwasser	-	Cooling water

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Tar and Coal Separator Distillation

(Managing personnel listed)

a) Tar and liquid-phase reactor separator distillation

Work in 1940:

Two of the six old gas preheaters (formerly hot-water units), with the parallel arrangement of the oil channel which was difficult to regulate and was the cause of clogging, were taken out, and a modern gas preheater, with serially connected hairpin tubes and blower unit, is under construction.

The antiquated power-gas supply device, which was hard to take care of, was rebuilt. A second intake device was brought here from the northern plant.

The waste gas from both raw-oil stock tanks, which have hitherto opened directly into the hydrogenation gas system, was connected with a gasometer functioning as a buffer, an arrangement which eliminated breakage of the seals, caused by shocks from the liquid-phase separator line.

Work for 1941:

- 1) Completion of the gas preheater with circulating air blower.
- 2) Setting up a larger-sized tower, with modern inside equipment.
- 3) Installation of tubes made of special anti-corrosion material, in the first water condensers.

b) Coal-separator distillation

Work in 1940:

In both gas preheaters the first six hairpin tubes (the hottest), so far made of S2 material, were replaced by tubes of N8.

To improve the phenol removal operation (i.e., get a better ratio of usable to unusable phenols in the light condensate) there was put into service in the more westerly of the two plants an arrangement to fractionally distill the top distillate (middle oil A) into a light and a heavy condensate.

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This sort of conversion having given a good account of itself in actual operation, the same fractional-condensation equipment will be built into the other plants; most of the preparation for this has been completed. Bi-metallic tubes have replaced the iron ones on the first water condenser of the east plant.

It was possible to increase the throughput in one plant to 146 cu. meters an hour, and even under such a load the cuts were quite satisfactory.

Despite the increase in throughput (the 1939 monthly average was 75,500 tons, that of 1940, 79,300), no serious trouble was experienced.

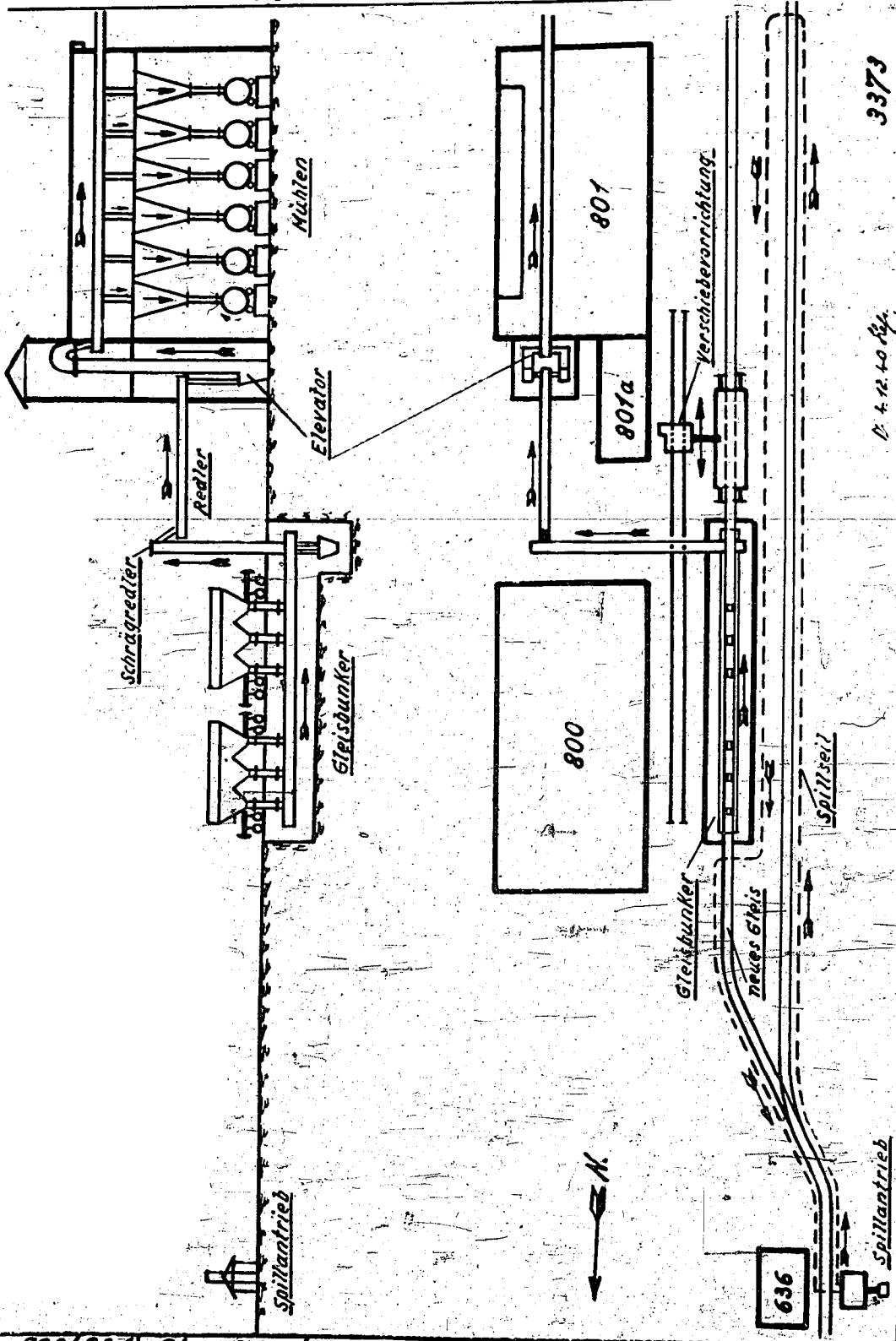
The out of middle oil A was raised from 315 to 325°.

The bottleneck in the way of further increases in throughput is the raw-oil pumps, which have given out at 146 cu. meters an hour.

Work for 1941:

- 1) Erection of one standby flue-gas blower for each preheater.
- 2) Erection of raw-oil pumps of greater capacity, in the event of increase in production.
- 3) Installation of regulating valves with lengthened conical plungers as raw-oil release valves.

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Ne 800/801 Staubfreie Entladung für Trockenkohle.

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(Drawing opposite page 90)

Me 800/801

Dust-Free Loading Arrangement for Dry Coal

Spillantrieb	-	Drum
" Schrägedler	-	Inclined conveyor
Redler	-	Conveyor
Gleisbunker	-	Hopper
Elevator	-	Elevator
" Mühlen	-	Mills
neues Gleis	-	New track
Spillseil	-	Cable
Verschiebevorrichtung	-	Switch

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Preparation of Coal

(Managing personnel listed)

Work in 1940:

. . . (Chart showing quantities of coal processed in 1938, 1939, and 1940 in Me 801). . .

To make possible the accomplishment of the task assigned to this installation, namely, the smooth preparation of something like 95 tons of TB coal an hour, with the finest possible grinding, the following improvements or enlargements were undertaken in the course of the year 1940:

1) A start was made on the construction of the central control plant for all the conveyors.

2) Conveyor belt 4 has been equipped with a double ash conveyor, since when there has been no more of the formerly frequent breakdowns due to snapping of the chain.

3) After approval of "Me-Kraft IV," construction work was started on the 9th mill (to be completed by the end of 1941).

The equipment for the screening and storing of coal paste is to be removed from the paste-pump buildings Me 803 to 805 and a second screening outfit will be set up south of the 9th mill; the individual paste pumps are to be supplied through circular paste tubes.

4) For the reduction or elimination of the dust given off when the quantities of coal are discharged, and to obviate the attendant risks, work was started on a dust-free loading installation west of Me 801, the effect of which will be to provide a second means of approach and a third set of loading facilities for coal. To be ready by the end of 1941.

The new plant has also been protected against the possibility of water getting in when pipes burst, something not entirely practicable with the former loading bunkers.

RESTRICTED

(Page 90 contd.)

Work for 1941:

. . . (Pump and mill items to be covered by program of replacement and improvement during year.) . . .

RESTRICTED

RESTRICTED

(Page 91)

Obtaining of Phenol by Middle-Oil Extraction

(Managing personnel listed)

Work in 1940:

The installation for the production of phenol from middle oil from the coal separator was put into operation during last year, and the temporary extraction plant in Me 906 with a capacity of 400 tons a month was continued. Despite a large demand for phenols it was impossible to take full advantage of the capacity of the old plant, for the reason that during the first few months it lacked a suitable distilling unit for raw phenol. The completion and putting into operation of the new plant was held up until March owing to the severe cold weather. However, production had to be more or less held down in the following months too, partly because the possibility of air attack made it necessary to lose no time in using up the large supplies of raw phenol oil and partly because of the occasional bottleneck constituted by the need to regenerate the caustic-soda liquor used for phenol extraction.

. . . (Following by the month-by-month figures for throughput and production) . . .

The above phenol distilling unit is a pipestill plant operating under atmospheric pressure. This is rather a new departure in the field of phenol distillation, as it has in general been the practice so far to use vacuum stills for the purpose. At the beginning, trouble developed frequently, due to the fact that the phenol oil, at the high operative temperatures, caused an unexpectedly serious amount of corrosion in the valves and clogged up the preheating tubes. By a lowering of the temperatures, which had to result in a reduction of the separation and yield of distillate, and by the use of special materials at points subject to particular danger, it was possible to eliminate the difficulties to a substantial degree; however, still further improvements in the safety aspects

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(Page 91 contd.)

of the plant are to be effected by converting the distillation columns to vacuum operation and by pre-refining the oil that is to be distilled. In spite of the trouble experienced with the pipestill outfit, the distillation of phenol constituted no production bottleneck, since in an emergency it was possible as a supplementary measure to distill a corresponding portion of the oil in the Me-103 phenol-production operation.

Work for 1941:

- 1) Installation for the obtaining of phenol from tar middle oil (Me-906) by the caustic-soda process.

- 2) Installation for the causticization and carbonization of the phenolate liquor.

RESTRICTED

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(Page 92)

De-Waxing Experimentation

(Managing personnel listed)

Work in 1940:

1) Propane de-waxing of low-temperature hydrogenation residue.

Two series of experiments were carried on during the year in Me 942. The outstanding result developed therefrom is that the paraffin yielded by a suspension of paraffin residue in cold propane is as satisfactorily freed of its oil as is the case when the process of dissolution and subsequent precipitation is used, so that the paraffin distillate obtained in both cases can be oxidized, after a final distillation, to a satisfactory yield of utilizable fatty acids. This fully confirms on a larger scale the results arrived at in the small-scale experimentation on quality of paraffin.

. . . (Results found, showing practically the same specific gravity, melting point and wax content for the paraffin obtained by the two processes) . . .

. . . (Experiments discontinued owing to danger from air attack) . . .

2) Final paraffin refining.

Instead of the aluminum chloride process for the final refining of the paraffin distillate, it is possible to employ a hydrogenation process, carried out under suitable conditions, using catalyst 5058.

3) Spindle-oil de-waxing for manufacture of white oil.

The throughput quantities have so far been:

Raw oil consumption	1,200 tons
Yield of slack paraffin	224 "
Yield of de-waxed spindle oil	976 "

The solvent content of the de-waxed oil was 0.05% on the average. . .

4) Experiments on use of propane containing propylene instead of pure propane, for the de-waxing of lubricating oil.

The tests showed that in place of pure (95%) propane it is possible to use a propane containing a high percentage of propylene (35% propane, 44% propylene)

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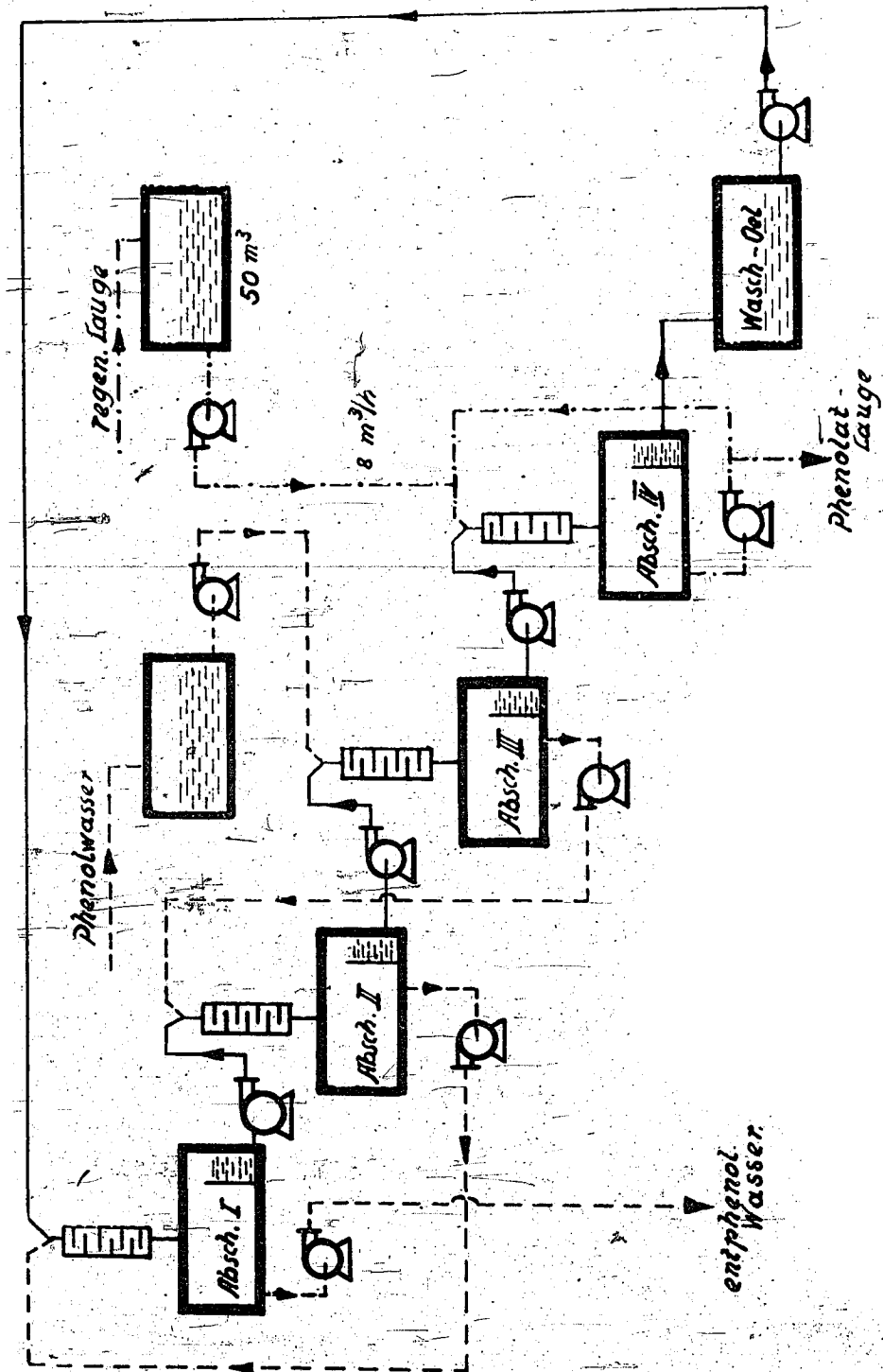
both for the oil refining (removal of asphalt and of resin) and for the de-waxing, and this without the expectation of any change in yield or quality.

Work for 1941:

- 1) Putting into operation of the paraffin plant at the Zeitz Erabag Works.
- 2) In view of the very great demand for fatty acids and of the fact that the Oppau paraffin oxidation plant is ready to start working, the experimental plants existing in Leuna will be devoted to paraffin production . . .
- 3) Gasification tests on dried and briquetted hydrogenation residue from

Me 883.

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4. 12. 40. Mann

Me 802 Schema der Wasserentphenolung durch Ölwäsche.

Me 802 Flow Diagram for Removal of Phenol from Water by
Scrubbing with Oil

Phenolwasser	-	Phenol water
regen. Lauge	-	Regenerated liquor
entphenol. Wasser.	-	De-phenolized water
Phenolat Lauge	-	Phenolate liquor

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Experimental Laboratory Me 907

(Managing personnel listed)

Work in 1940:

1) Removal of phenol from water by washing with oil.

Suitable fractions from brown-coal tars and coal-liquefaction oils are better adapted to the removal of phenol from waste water than is the raw benzene ordinarily utilized for this purpose in the Ruhr industries. The knowledge of this fact resulted in the elaboration of a phenol-removal method for use on the waste water from the distillation and hydrogenation processes, in which the phenol solvents used were, in principle, products specially adapted to such operation. By the use of comparatively large quantities of solvent (50 to 200% of the water, by volume), and by inserting a multistage counterflow washing device, a very thorough de-phenolization of the water can be achieved--down to a phenol content of as low as 50-200 mg. to the liter, which means that it is 98-99% effective. The solvent charged with the phenols from the waste water is in turn de-phenolized with caustic soda and used over again for extraction from the water, and thus moves in a closed circuit.

The experiments, started in a continuously-operating laboratory outfit, were carried further in a semi-industrial plant with a water throughput of 100 liters of water an hour, and in a one-month operation the favorable results of the small scale tests, with respect to degree of phenol removal and low solvent loss, were given confirmation. The process lends itself well to combination with that for the production of paraffin from phenol-containing oils, and on that account may be considered either as a possible substitute for the tricresylphosphate process in Leuna or as something to use in new hydrogenation

RESTRICTED

(Page 93 contd.)

plants. The laboratory staff has accordingly planned a large-scale test, and the necessary installation has already been set up in Me 906x . . . In the meantime the laboratory and semi-industrial experiments are being carried on to see whether the quantity of solvent in circulation might be reduced.

2) Alkylation tests.

Experimentation was carried on, under varying reaction conditions, on the reaction of isopentane and propylene with H_2SO_4 . It was found that the main products resulting were hydrocarbons with an octane index of only about 70; nor did the investigators succeed in arriving at any practicable determinations with regard to the consumption of propylene and sulfuric acid. The tests were therefore discontinued.

3) Experiments to obtain ketones from hydrogenation products.

The tests disclosed that certain phenol waters and light distillates from coal-reactor separator products and distillation tars contain not inconsiderable quantities of ketones. Altogether something like 450 tons a month of ketones--half of them acetone--are present in the hydrogenation products obtained in Lima. . . . (Easy possibility of obtaining ketones by steam distillation and of purifying the acetone by a simple method.) . . .

RESTRICTED

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(Page 94)

4) Separation of oils by use of selective solvents.

The purpose of the experiments is to obtain chemically homogeneous fractions from petroleum. Emphasis is at present placed on isolating a refined product that can be used as a substitute for Mepasin. After a series of solvents had been investigated, tests were carried further with phenols (with and without an addition of water), in which, in a 5-stage continuous counterflow reaction, some very decided separation effects were achieved, so far as this can be inferred from the physical constants of raffinate and extract. A final judgment as to whether such extraction is workable or not can only be arrived at by a long-continued working-up to sulfonates, and this is being done by the experimental laboratory.

The extraction tests are being continued, with resort to all known extraction aids--e.g., stepwise increase in selectivity by regulating temperature and degree of concentration of solvent.

Work for 1941:

- 1) Plant to obtain phenols from strongly phenolized water by washing with oil, in replacement of the trioresylphosphate process.
- 2) Obtaining acetone and higher ketones and alcohols from hydrogenation products.
- 3) Obtaining ammonia and hydrogen sulfide from phenol water.

RESTRICTED

RESTRICTED

(Page 95)

Laboratory Me 885

(Managing personnel listed)

Work in 1940:

1.) Investigation of paste samples from Elise and v.d. Heydt TB coal with respect to the curve of the viscosity of the paste in the range from 100 to 200°C. The viscosity-temperature curves for the paste samples made with the Elise product are low and practically without variation, while the v.d. Heydt samples showed in every case higher viscosities (up to as much as 7 times those of the Elise samples), and the coarser the grain to which the paste has been reduced in the grinding, the higher the viscosity of the samples made with v.d. Heydt coal.

2) Comparative investigation of Elise and v.d. Heydt TB coal samples to determine respective humic acid content: not much difference between the two kinds in this respect--the average content of 14% by weight for the Elise being in general somewhat higher than that for the v.d. Heydt.

3) Tests made on purifying agent 11002 of varying water content to ascertain the corrosive effect on wrought iron (for Politz); with up to 20% water content--i.e., to upper limit of resistance of an extremely dry powdered catalyst grain--no noticeable amount of corrosion. When on the other hand the catalyst particles are given an external wetting by a further increase in the addition of water, the rise in the amount of corrosion is in proportion to the increase in that part of the total water content that no longer combines by adsorption.

4) Prevention of further formation of resin and asphalt (Hirschfelde tests with Koppers tar): It was not possible to entirely prevent this aging. The tendency of Koppers tar to age could be brought down to the figure observed in the case of normal distillation tars, by an addition of practically asphalt-free tars or by a treatment with Tonsil impregnated with 5% by weight of concentrated phosphoric acid.

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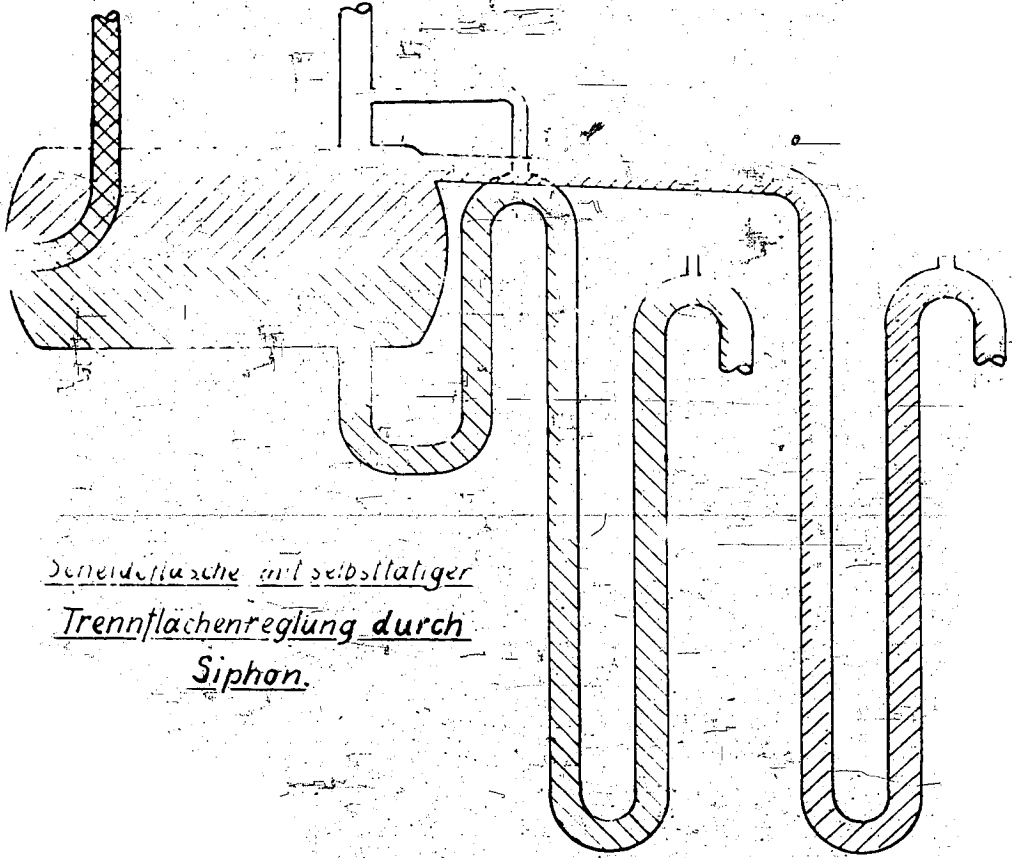
5) Tests on ketone mixtures obtained from hydrogenation waste-products to determine their suitability as de-waxing agents for crude petroleums, by comparison with methylethyl ketone: neither the low-boiling nor the high-boiling ketone mixtures show the proper selectivity. There is no possibility of replacing methylethyl ketone or any other de-waxing agent with fractions of these mixtures of ketones.

6) Experiments on the de-waxing of low-temperature hydrogenation residue with ethylidene chloride, as compared with ethylene chloride (dichlorethane): Equally effective in paraffin yield. The paraffin obtained with ethylidene chloride, however, in no case comes up to the degree of purity of the product obtained with dichlorethane; and, correspondingly, the setting points of the de-waxed oil are higher.

7) Further researches had to do with the utilizability, for petroleums, of the wax-determination methods worked out for hydrogenation products; the replacement of the analytical methods using iodine by methods not using it, and the preparation of a new type of absorption filter . . . etc.

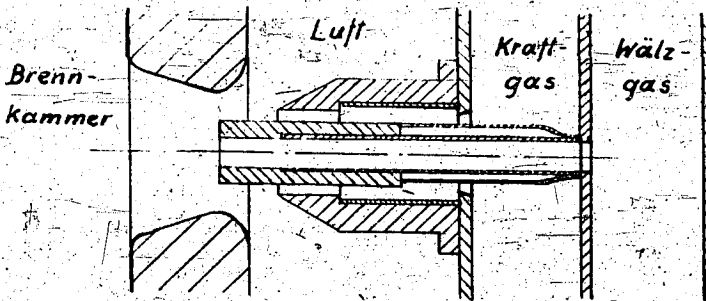
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Benzin - Abstreifer - Destillation



Benzinflasche mit selbsttätiger
Trennflächenreglung durch
Siphon.

Brenndüse mit Wälzgas - Abstimmung.



(Diagram opposite Page 96)

Gasoline-Separator-Distillation

Scheideflasche mit selbsttätiger Trennflächenreglung durch Siphon	-	Separator equipped with automatic interface regulator by means of siphon
Brennduse mit Walzgas-Abstimmung	-	Fuel nozzle modified for use with circulating gas
Brennkammer	-	Combustion chamber
<hr/>		
Luft	-	Air
Kraftgas	-	Fuel gas
Walzgas	-	Circulating gas

RESTRICTED

(Page 96)

Gasoline Separator-Distillation
Gasoline Washing
Intermediate Tank Farm

(Managing personnel listed)

Work in 1940:

A multi-tipped fuel nozzle was developed and installed in unit No. 3 whereby circulating gas is conducted to each nozzle. The advantage of this construction is easy regulation and relative independence from calorific value of fuel. The temperature in the combustion space is thus maintained below 1400°C. Flue gas temperatures from preheater are still high enough to prevent corrosion.

. . . (Paragraph deals with plant detail) . . .

The separators of units 1, 2, and 4 were equipped with automatic interface regulators using principle of siphon as indicated by sketch on opposite page. Suitably adjusted overflows for gasoline and for phenol water permit maintenance of the interface at constant level; variations in pressure within separator are compensated for by an equalizing connection with phenol water discharge.

A maximum quantity of 950 tons per day of aviation gasoline were manufactured experimentally. It was necessary to cut the pre-hydrogenation-separator and the 6434-separator at different levels (temperatures) and to distill separately in order to avoid a sacrifice in octane value and in order to retain sufficient knock resisting components in the gasoline.

Because of the high aviation gasoline production there is a surplus of absorption gasoline. The latter was utilized for manufacture of serviceable motor gasoline by blending with heavy gasoline from the B-middle oil from the aviation gasoline separator and with small amounts of 5058 prehydrogenation gasoline.

Distillation of product from gasoline separator is conducted in "gaps." Although energy costs are higher the advantages are increased yield of knock

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(Page 96 contd.)

Resisting components. Furthermore, this also results in a reduction of costs in high pressure plant operation because of less vaporization (gasification) and improved load factors (see distillation curves next page).

The production of aviation gasoline, Diesel fuel, and motor gasoline, varied with wartime requirements from time to time.

Depending upon quantities of Diesel fuel required the initial boiling points had to be adjusted accordingly. Motor gasoline production was similarly affected.

. . . . (The next six paragraphs deal with plant detail and anti-aircraft protective measures.)

Work for 1941:

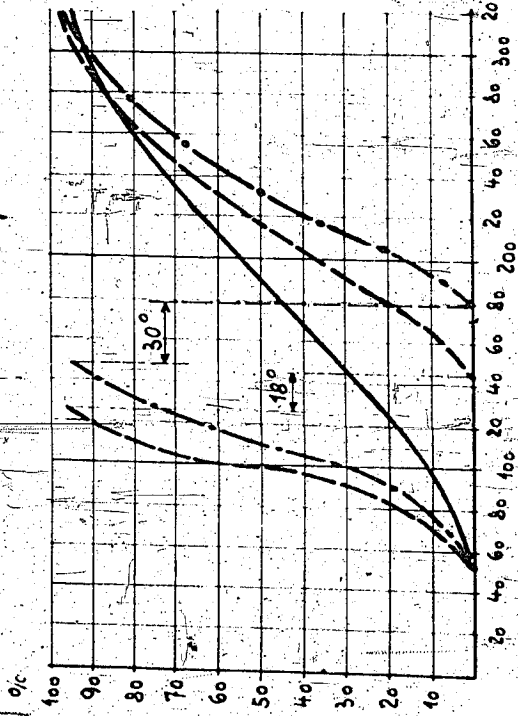
- 1) Installation of a larger crane.
- 2) Construction of unit No. 6 for distillation of superfuels.

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Benzinabstreifer - Destillation. Fahren auf Siedelücke.

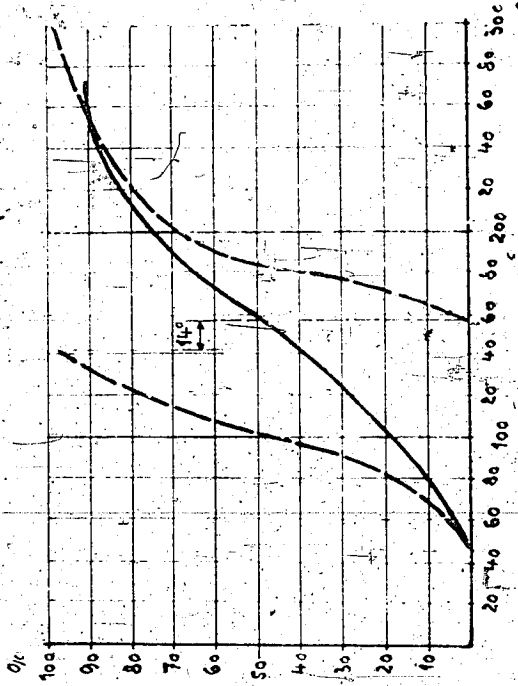
Vorhydrierungs-Abstreifer.

18° bzw. 30° Siedelücke
zwischen Benzin-Siedeschluß
und Rückstand-Siedebeginn.



Ø434-Abstreifer.

14° Siedelücke
zwischen Benzin-Siedeschluß
und Rückstand-Siedebeginn.



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(Diagram opposite page 97)

Gasoline Separator-Distillation "Gap"-Distillation Operation

Vorhydrierungs-Abstreifer	-	Prehydrogenation-separator
18° bzw. 30° Siedelücke zwischen Benzin-Siedeschluss und Rückstand-Siedebeginn	-	18°C. and 30°C. gap between gasoline endpoint and initial of bottoms
6434 - Abstreifer	-	6434 Separator
14° Siedelücke zwischen Benzin-Siedeschluss und Rückstand-Siedebeginn	-	14°C. gap (in boiling points) between gasoline endpoint and initial of bottoms

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(Page 97)

Caustic Wash

(Managing personnel listed)

Work in 1940:

In order to avoid dirtying of the regenerators during depropanizing with phenolic solutions, water washing was installed behind caustic washing in the manufacture of aviation gasoline.

For better separation of gasoline (from phenolic solutions) an additional separator drum was installed.

Depropanizing and Separation of Absorption Gasoline

(Managing personnel listed)

Work in 1940:

. . . (Deals with anti-aircraft measures.)

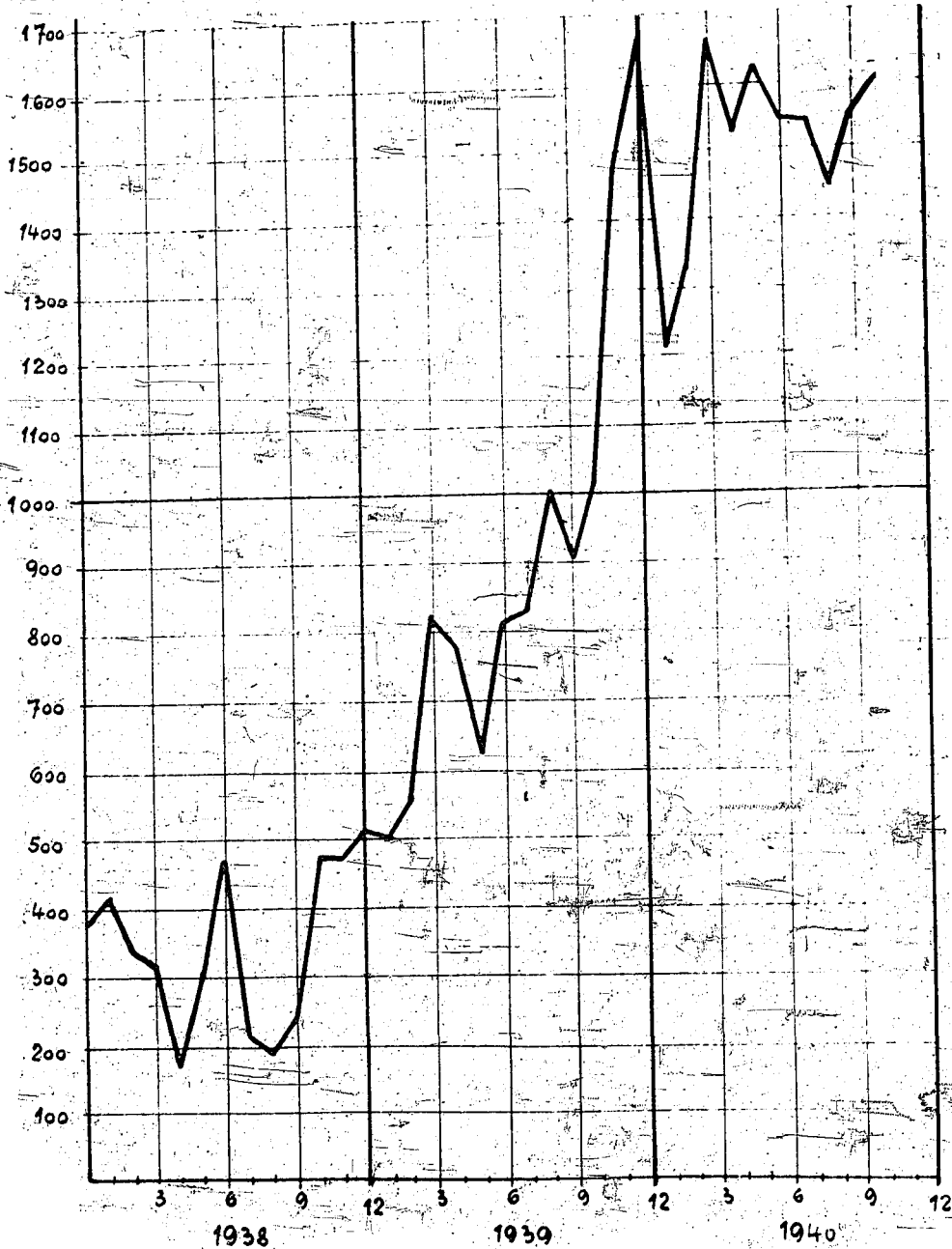
Work for 1941:

Construction of a large depropanizing unit becomes more imperative inasmuch as there are no reserves.

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Gasbenzin Niederdruck I Ethan-Erzeugung.

Moto



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(Diagram opposite page 98)

Absorption Gasoline Low Pressure No. 1

Ethane-Manufacture

Moto

Tons per month

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Absorption Gasoline
Low Pressure - No.1

(Managing personnel listed)

Work in 1940:

. . . (Deals with plant details) . . .

The replacement of iron bundles with aluminum bundles in the regenerators of the Alkacid-plant was not completed owing to supply difficulties.

In order to reduce corrosion of iron in towers and heat exchangers in Alkacid unit No. 936, arsenous acid was added (0.6 kg/cu.meter). This expedient was completely unsuccessful. Replacement of iron with aluminum is consequently imperative.

Electrical level indicators were installed in Alkacid units in order to get away from bursting of sight glasses (aircraft damage) and to get relief from soiling of sight glasses.

Corrosion of gasoline cooler by water from river Saale in the absorption plant caused leaks and necessitated replacement with "bimetallic cooler."

It has been found that GHH-compressors suffered from frequent stoppage caused by air raid alarms (34 times). They are therefore no longer stopped during air raids.

. . . (Deals with plant detail) . . .

Work in 1941:

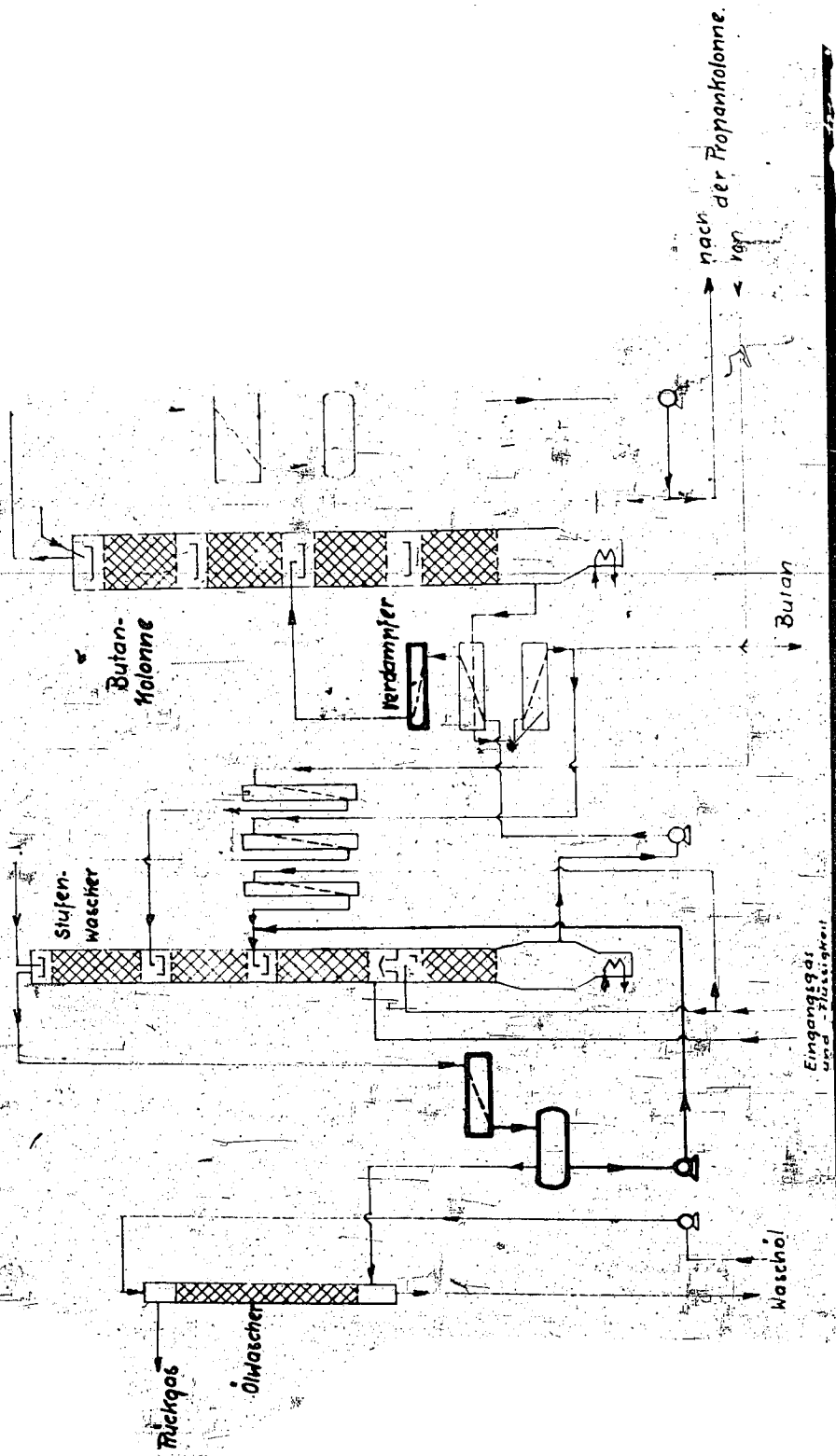
1. Attempts to remove by filtration the iron sulphide which is carried in circulation in the Alkacid aqueous solutions.
2. Construction of gas compressor No. 4.
3. Installation of a recycle cooler (Kreislaufkuehler) at the wash tower in order to attain better scrubbing of gases with Alkacid solution and to save caustic solution.

Gasbenzin Niederdruck II

Me 914

Verbesserung an der Reichgaszerlegungs-Anlage.

(nach Gerlach)



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(Drawing opposite page 99)

Low-Pressure Tail-Gas Plant II

Me 914

Improvements effected on rich-gas separator unit (Gerlach type)

Rückgas	-	Re-circulated gas
"		
Ölwascher	-	Oil scrubber
Stufen-wascher	-	Step-wise scrubber
Butan-Kolonne	-	Butane tower
Verdampfer	-	Vaporizer
"		
Waschol	-	Wash oil
Eingangsgas und-Flüssigkeit	-	Gas and liquid charge
Butan	-	Butane
nach der Propankolonne	-	to propane tower
von		from

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(Page 99)

Low-Pressure Tail-Gas Plant II

(Managing personnel listed)

Work in 1940:

As planned, a dephlegmator was installed at the outlet of the staged scrubber, and a preheater at the entrance to the butane tower, with the following resultant increases in yield, ethane from 86 to 94%, propane from 94 to 98%, butane from 90 to 100%. The improvement in the quality of the butane was from 88 to 95% . . . (Mention of other improvements, etc.) . . .

The preheaters are now connected in series instead of in parallel. A speeding-up of the gas results in better heat transfer, and the temperatures are thereby reduced from about 22° to about 10°. This means more water taken out by the silica gel in the driers and an avoidance of the danger of serious icing of the ethane condenser.

To bring about a better separation of n- and isobutane, the mixture was subjected to pre-distillation, with resultant removal of the propane contained. The amount of isobutane then obtained in the second distillation is 90 to 95%.

The installations were fitted with Bollux density indicators.

. . . (Measures taken to avoid risks due to freezing) . . .

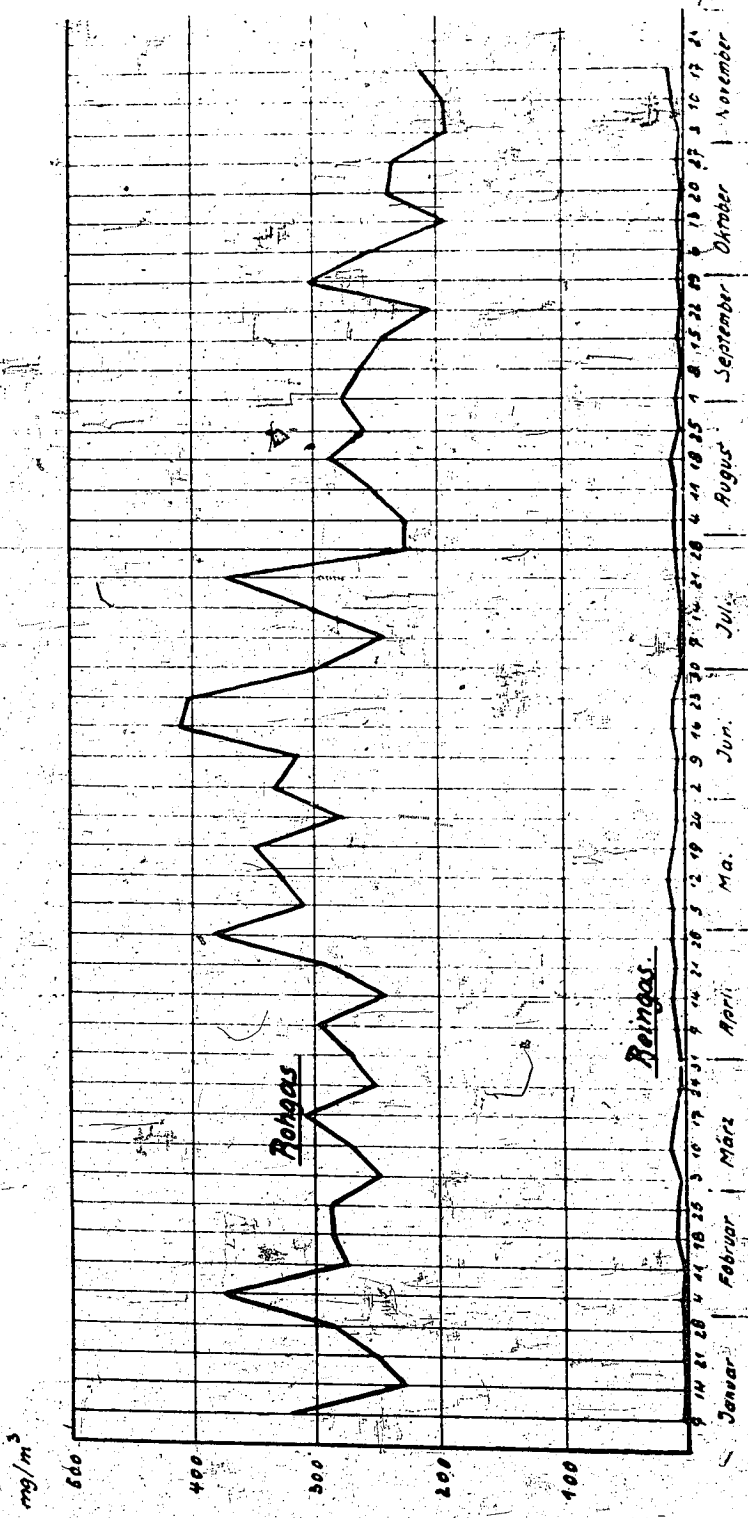
Work for 1941:

- 1) Providing the larger butane-isobutane separation plant.
- 2) Arrangements to expand liquid gases in a tank of 50 cu.m. (safety measure).

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Sumpphase Reingas - Reinigung:

Merkmalegehalt im Roh- u. Reingas.



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(Drawing opposite page 100)

Purification of Liquid-Phase Rich Gas

Mercaptan content (mg. to cu.m) in raw and refined gas

Rohgas	-	Raw gas
Reingas	-	Refined gas

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(Page 100)

Purification of Liquid-Phase Rich Gas

(Managing personnel listed)

Work in 1940:

. . . . (Providing a catalyst suction device. Rapid exhaustion of catalyst due to heavy load; two reactors therefore operated in parallel.) . . .

As shown by the drawing, there is a satisfactory removal of organic sulfur (mercaptan) from the gas. It has not been possible until this year to make a quantitative check of the COS content . . .

Work for 1941:

Providing a third catalyst chamber.

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(Page 101)

Shipment of Gasoline

(Managing personnel listed)

Work in 1940:

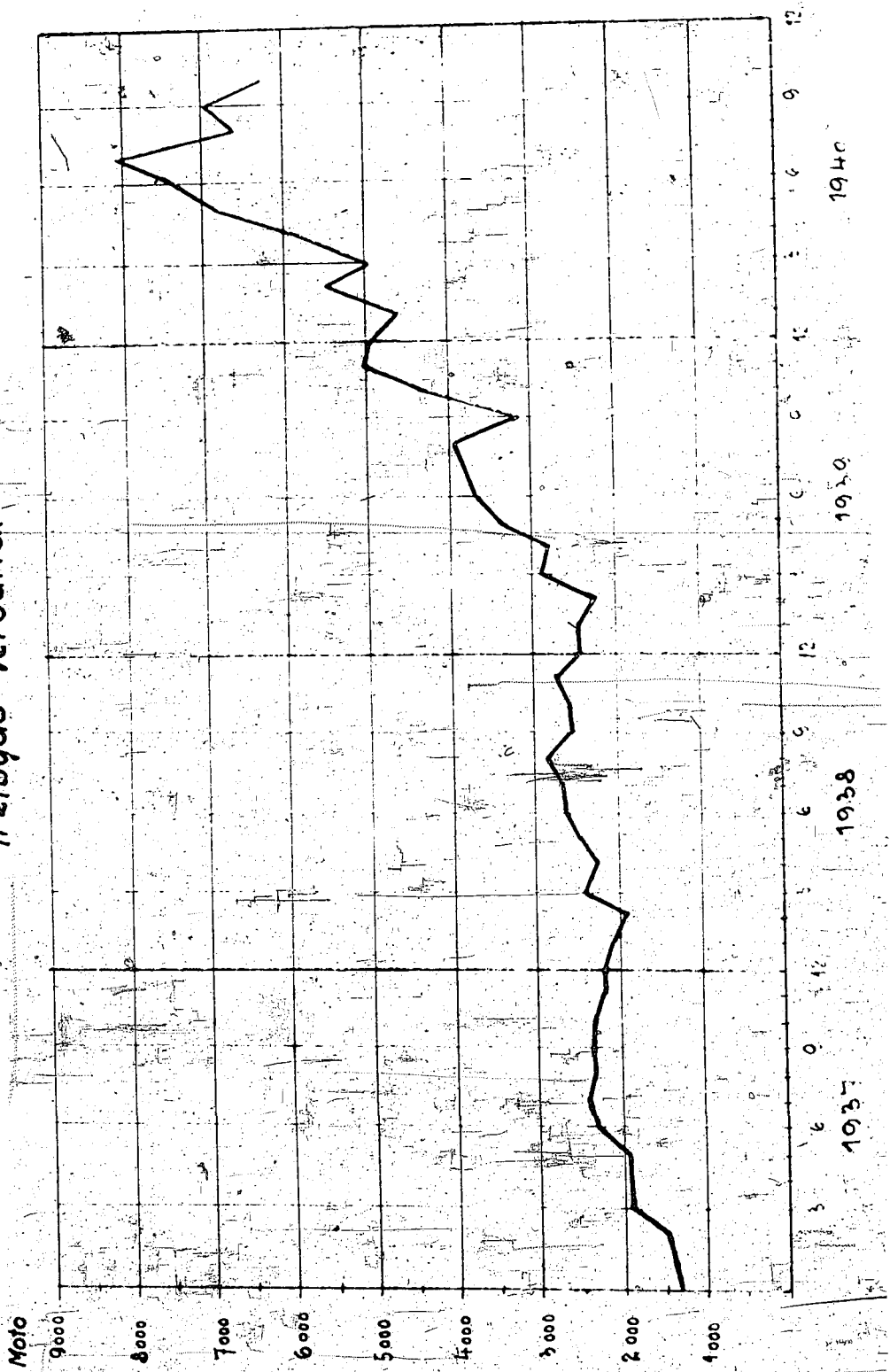
. . . (Improvements made in installations, to reduce fire risks, etc., in the event of air raids.) . . .

Work for 1941:

The planning of new projects has been held up in consequence of war conditions; this includes the tank-truck loading arrangements.

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Kohlenwasserstoff-Flussig-Versand
Treibgas-Versand.



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(Drawing opposite page 102)

Liquid Shipment of Hydrocarbons

Curve of shipments of power gas

Moto

-

tons per month

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Storage and Shipment of Liquid Gas

(Managing personnel listed)

a) Storage of liquid gas

Work in 1940:

. . . (Various measures taken in regard to tanks, etc., for air-protection reasons and otherwise.) . . .

Work for 1941:

Work has already started for the increase in the ethane storage to 20,000 cu. m.

b) Shipment of liquid gas

. . . (Improvements and enlargements in connection with packaging installation. Chart on opposite sheet shows increase in volume of shipments of power gas) . . .

c) Liquid gas operations

Work in 1940:

. . . (Increase in stock of bottles makes possible a sale of 80,000 tons per annum of power gas. Sale of propane has risen despite shortage of bottles.) . . .

Work for 1941:

The delivery of 25 new tank cars is expected.

RESTRICTED

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(Page 103)

Laboratory, Technical Institute, Motor Test Installation

(Managing personnel listed)

Work in 1940:

All investigations required for the proper checking of operations were effected with raw, intermediate and finished products, and further progress made in the quantitative and qualitative analysis of the raw gases and of the ethane, propane and butane derived from them.

Committee and government cooperation was curtailed by war conditions, but was maintained.

. . . (Mention of additional items of equipment acquired for the engine-testing shop.) . . .

In addition to the intermediate and final operational results achieved in hydrogenation, tests were likewise carried on, over a large field, on power fuels emanating from the organic section and from the main-laboratory experimentation, and these fuels were evaluated.

Work for 1941:

Better facilities will have to be provided for the growing number of women employees. New projects, especially in regard to laboratory enlargements, have had to be held up.

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Division for Economy Investigations

Dr. Langheinrich

Dr. Richter

Dr. Fritsche, Dr. Groger, Dr. Klockmann, (from 1/5/40), Dr. Kolb (from 12/2/40), Dr. Kranepuhl, Dr. Munch, Dr. Rohre (sick), Dr. Schulz (to 1/6/40), Dr. Weber (from 9/8/40), Dr. Hayek (to 1/7/40), Dr. Brandl (to 1/6/40), Dr. Eckhard (to 12/2/40)

The development of the process for synthetic gas production from the various fuels was more carefully studied in general according to technical and economic viewpoints and in particular for Leuna for producing Mepasin, likewise the gasification of lump and hard fines as well as mineral coal semi-coke. Further the synthesis for Mepasin-crude oil was worked up according to calculations by various processes in Leuna, as well as the complete H₂ gas utilization for Leuna by the Linde separation and separation with active carbon. Obtaining further amounts of granular fines for the gasification in this place by purchase from foreign plants was arranged and obtaining these in a retorting plant of our own was further investigated. (Fritsche)

In the field of gas purification, the further licensing of the Alkaid- and Claus process at home and abroad was practiced together with the industry and the Dir. Buro d. Sp.-I. The German sulfur market situation was followed currently, especially in its effect on Leuna and the I.G. The question of obtaining sulfuric acid was repeatedly investigated. For the production of Krypton and dry ice various cost estimates were made. (Richter)

In the field of phenol the economic questions of production according to various processes, refining, calculation and especially the purchase of crude phenol and the concluding of contracts (among others Phenosolvan) were worked on centrally in the AWP. (Langheinrich, Groger, Weber)

In the field of the organic products the increase in production and market, utilization of by-products, etc., were looked after by working with manufacturing plants and sales division of the I.G. as well as with the appropriate government authorities and economic organizations in purchase

C O N F I D E N T I A L

(Page 110 contd.)

of raw materials and making sure of markets, contract - price - and financial - transactions. In the foreground in this connection were particularly the Mepasin products for soap substitutes, textile auxiliary materials and softeners, further the cyclohexanols made from phenols, adipic acid, Luran as well as product 512, white oil, synthetic toluol and the oxo-process. For orientation on the constantly growing field a statement on the production value and obtaining of organic final and intermediate products of Leuna and the Sparte I was set up. (Langheinrich, Gröger, Munch, Weber)

The development of cracking and dehydrogenation processes for the preparation of ethylene, propylene, iso- and normal-butylene, production of lubricating oil from ethylene and paraffin cracking products, T 52, Di 1000, polymer-, alkylate- and hydroforming-gasolines were followed technically and economically and amicable regulation for Leuna and Moosbierbaum with RIM, Wifo and Donau Chemie carried out. (Langheinrich, Eckhard/Kolb)

The obtaining of mineral oil in the widest range, such as by hydrogenation, retorting, catalytic cracking, Fischer synthesis in its various modifications (also for obtaining Mepasin products) was thoroughly worked on. The cooperation of some of our men in the "Reichsamt für Wirtschafts-aubau" (Government Office for Economic Development) and in the cooperative work for hydrogenation, synthesis and retorting was continued. (Langheinrich, Kranepuhl, Brandl/Klockmann)

The regulation of the production and distribution of the hydrogenation contacts for various German hydrogenation plants was carried out centrally. (Langheinrich, Hayek/Klockmann, Kolb)

For the I. G. generally testing of the economic possibilities of Politz was taken-up, contract transactions for coking gas- and coke deliveries of Ferdinands Nordbahn to Heydebreck were authoritatively carried out and participation in such for the securing of coal pits in upper Silesia.

C O N F I D E N T I A L

Page 111

The collective working on technical problems for the South European Branch of the I. G. was continued in the Berlin bureau of the AWP. The technical and economic investigations, partly on the spot, extend chiefly to Slovakia (Dynamit Nobel, Pressburg), Jugoslavia (Ruse, Jaice, Zorka, Sabo) and had as their aim the development of the I. G. interests (artificial silk, chemicals). (Langheinrich, Munch, Hayek)

For putting back quickly into operation the chemical and particularly the nitrogen industry of North France and Belgium after the occupation, after a number of investigations, plans were set up for the RWIM and OKW. (Fritsche)

The works in Brazil for utilizing the technical I. G. findings had to be stopped because of certain circumstances. Dr. Schulz took over the technical direction of the Elektrolyse Fluminense obtained from the I.G. (Schulz)

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(Page 112)

Experimental Laboratory including Technikum

Only the most important investigations are listed, those that have been completed or which are in an advanced state. The projects cover the following fields:

- 1) Liquid and vapor phase hydrogenation
- 2) Catalytic cracking
- 3) Hydroforming (HF)
- 4) Preparation of very high knock rating fuels from the hydrogenation gas fraction
- 5) Chlorination of hydrogenation gases and oils
- 6) Phenolic oils
- 7) Amines and charge stocks for synthetic materials
- 8) Oxo process
- 9) Catalyst and physical-chemical projects

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Vapor Phase Hydrogenation

In order to find additional charging stocks for the Lützkendorf hydrogenation plant, a gas oil ($d_{20} = 0.890$, boiling range 110-325°C.) from cracked residus was tested in the vapor phase at 600 atm. over catalyst 6434. At 390° and a space velocity of 0.7 v/v/hr. a gasoline of 200°C. E.P. and 71 octane number (research method) was obtained. The gas yield was 10%. In spite of the high olefin content of the charge no harmful effect on the catalyst was noted.

This concludes preliminary experiments for Lützkendorf whose vapor phase reactor is now in operation.

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C O N F I D E N T I A L

TRANSLATION

CATALYTIC CRACKING

(Page 114)

In charge of Messrs. Dr. Kaufman, Dr. Welz, Dr. Mueller,
Dr. Hartmann (from Oct. 1, 1940)

Activities in 1940

The cracking experiments were exclusively devoted to the production of aviation gasoline with an end pt. of 165°C. Since this has heretofore been possible only in a no-pressure operation over a fixed catalyst the work with pulverulent catalyst (no pressure in the gas phase and curved pressure in the liquid phase) has temporarily been stopped.

For the production of aviation gasoline low temperatures (400-420°C.), short reaction periods (15-30') with rates of flow (?) of 0.6-1.5 v/v/h, and the utilization of aluminum hydrosilicate catalysts are requisite conditions. The form stability and reproducibility of the catalysts have been improved. Best catalyst aluminum hydrosilicate ($\text{Al}(\text{OH})_3 \cdot \text{SiO}_2 = 1:4$) from $\text{Al}(\text{OH})_3$ peptized with HNO_3 and silica gel precipitated at $\text{pH} = 3.5$. As a starting material a paraffin-base Roumanian middle oil was used predominantly, which is also to be used in the future in the Pressburg experimental plant.

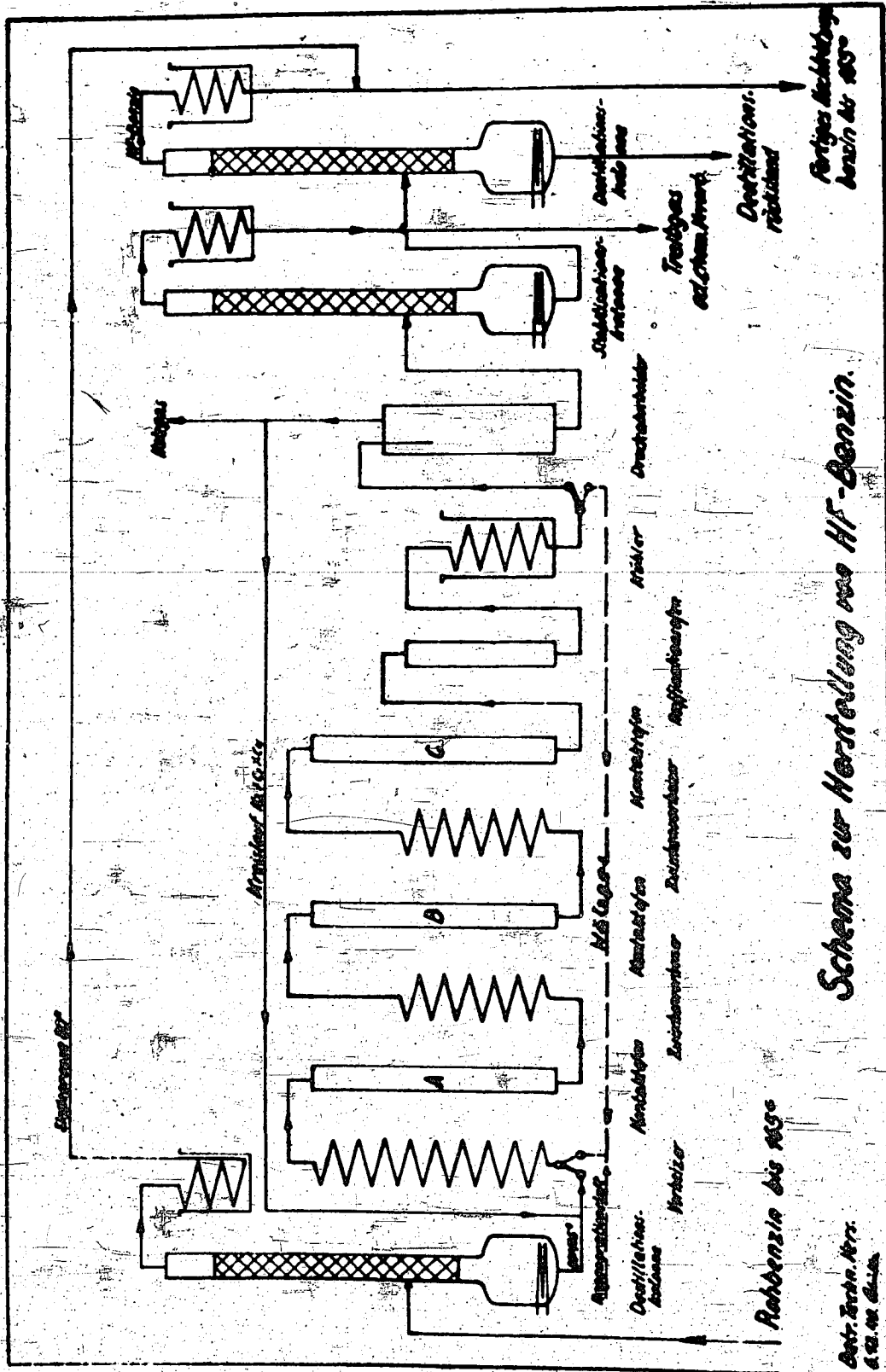
Most favorable conditions for Roumanian middle oil ($d_{20} = 0.862$, boiling range 200-400°C.: Temperature 420°C., rate of input 1.5 v/v/h, reaction period 20 minutes.

Yield: 22% (by weight) of aviation gasoline (octane no. 76, olefin content 6%).

Activities for 1941

Experimental work on the production of aviation gasoline by no-pressure cracking even with a pulverulent catalyst. Placing into operation the experimental pilot plant at Pressburg (catalyst charge 1 cu.m.).

C O N F I D E N T I A L



Schema zur Herstellung von HF-Benzin.

Rohbenzin bis 105°

Dr. Ing. H. v. ...
 1912

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(Drawing opposite Page 115)

Flow Sheet of Plant for the Production of HF-Gasoline

HF-Benzin	-	Hydroformed gasoline
Kreislauf	-	Recycle gas
Regenerationsstoff	-	Regeneration material
Destillationskolonne	-	Fractionating column
Rohbenzin	-	Gasoline charge
Vorheizer	-	Preheater
Kontaktoven	-	Reactor
Zwischenvorheizer	-	Intermediate preheater
Raffinationsofen	-	Aftertreating reactor
Kühler	-	Cooler
Stabilisationskolonne	-	Stabilizer
Treibgas	-	Fuel gas
Destillationsrückstand	-	Bottoms
Druckabscheider	-	Pressure separator

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(Page 115)

HF Process (Hydroforming)

(Managing personnel listed)

Work in 1940:

The HF process permits the production of high quality gasolines which have particularly good properties (high rich mixture rating) for airplane motors. Since the charge situation is more favorable for this process (hydrogenation gasoline and Rumanian or Russian S.R. gasolines) during the war than for catalytic cracking, experiments in this field were pushed strongly.

The investigations started with systematic catalyst and product studies as well as the most favorable operating conditions in externally heated reactors (catalyst volume 1.2 l, 4 l, and 25 l, electrically heated). These were supplemented by experiments on a semi-commercial scale (tubular and single large space reactors) which yielded data that made it possible to start construction of a commercial plant in the East (Moosbierbaum) with a capacity of probably 100,000-150,000 tons/year which had been planned along with the experimental work. A 140,000 ton/year plant for Leuna has also been considered.

The best catalyst had a composition of 90% Al_2O_3 - 10% MoO_3 . The product investigation included primarily Rumanian S.R. gasoline and Leuna prehydrogenation gasoline. Optimum conditions depend largely upon the charge. The following optimum conditions were determined:

Naphthenic gasoline (prehydrogenation at Leuna): 480-500°C., 30 atm., 500 vol. circulating gas per volume of oil, 1v/v/h, catalyst on stream 24 hours.

Paraffinic and mixed base natural gasoline (Rumanian): 490-500°C., 15 atm., 500 vol. circulating gas per vol. of oil, 0.6-0.75 v/v/h., catalyst on stream 10 hours.

RESTRICTED

RESTRICTED

(Page 115 conti.)

Under these conditions the following yields were obtained:

Prehydrogenation gasoline: 84% (50% aromatics, 78-80 octane number)

Romanian gasoline: 80% (50% aromatics, 80-82 octane number)

2 reactor systems were investigated on semi plant scale.

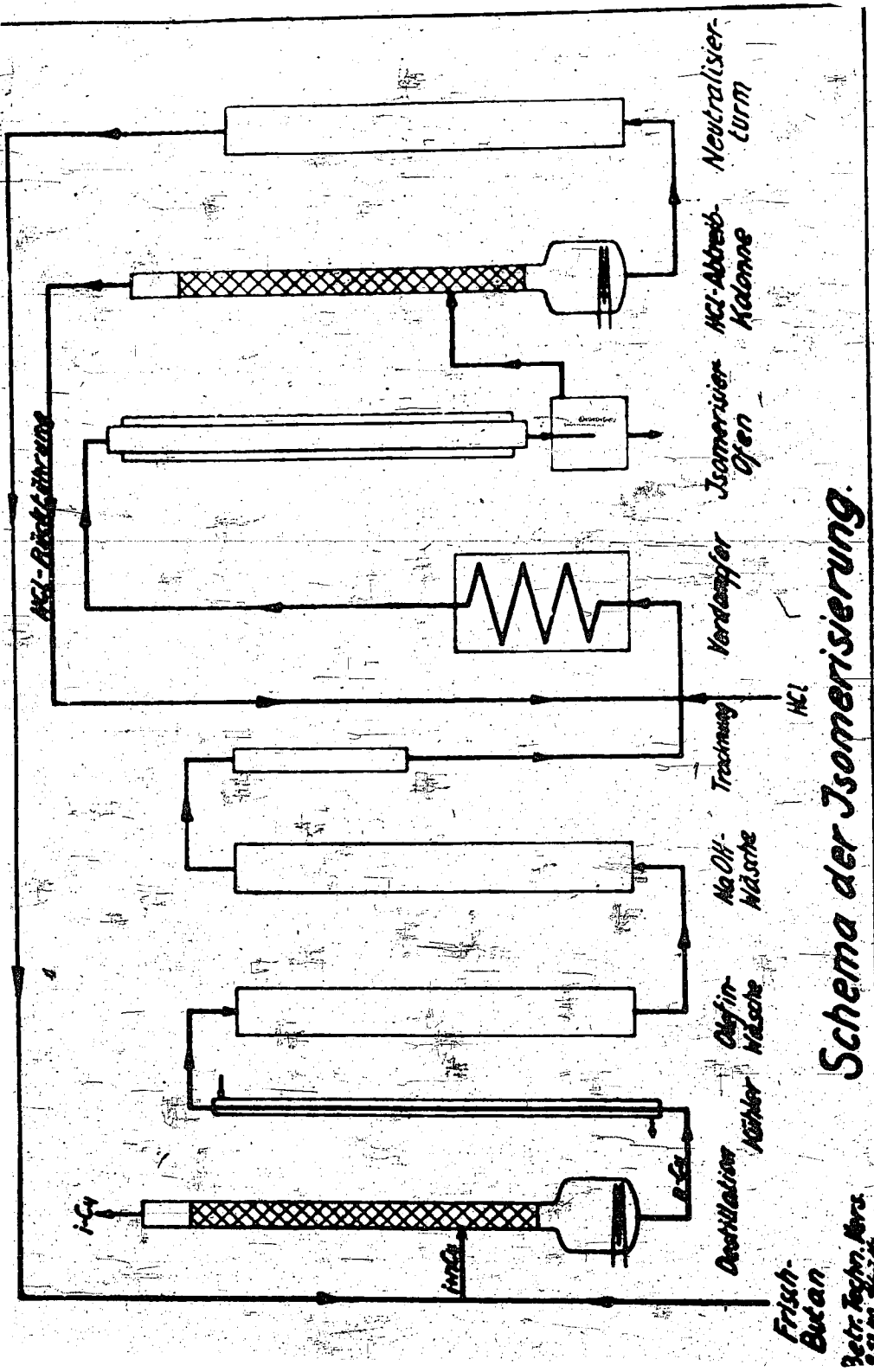
- 1) Unheated large reaction vessels consisting of three insulated reactors of 50 l. capacity with heaters between them (used in Meesbierbaum)
- 2) Gas-fired tubular reactor (5 tubes, 90 mm. diameter, 25 l. catalyst volume)

The first and cheaper system yields 5% less gasoline so far.

Work for 1941:

Continuation of experiments, particularly on semi plant scale to supply data for the commercial plants.

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Schema der Isomerisierung.

Frish-Butan
 Petr. Techn. Nov. 1924

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(Drawing opposite Page 116)

Isomerization Flow Sheet

HCl-Rückführung	-	HCl recycling
Frisch Butan	-	Fresh butane
Destillation	-	Fractionating
Kühler	-	Cooler
Olefin Wasche	-	Olefin scrubber
NaOH Wasche	-	Caustic scrubber
Trocknung	-	Drying
Verdampfer	-	Vaporizer
Isomerisierofen	-	Isomerization reactor
HCl Abtreibkolonne	-	HCl separator column
Neutralisierturn	-	Neutralizing tower

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(Page 116)

Preparation of very high Octane Fuel
from the Gas Products of Hydrogenation

(Managing personnel listed)

Work in 1940

a) Dehydrogenation of isobutane

Small scale experiments

The purpose of these investigations was to develop a more selective and more active catalyst which was abrasion resistant. This was accomplished with active aluminum hydroxide soaked in chrome salts and alkalinized. However, the life of these catalysts is only a few hundred hours. Thorough catalyst reactivation experiments were used as a basis for the construction of a semi-commercial regenerator.

Large scale experiments.

The slucice process was carried out on semi-commercial scale in a tubular bundle reactor with a catalyst residence time of 4-8 hours. At a one thousandfold gas load 22-24% of the isobutane were converted on the present commercial catalyst with an isobutylene yield of 80-85%. The technical difficulties in the removal of catalyst sludge were overcome. Abrasion was within reason. The catalyst regeneration was carried out in a ceramic slucice reactor continuously with a circulating gas containing 2-3% oxygen. By only partial regeneration it was possible to reduce the regeneration time. Formerly the regeneration was carried out discontinuously by raising the oxygen concentration gradually from 2-12%.

b) Dehydrogenation of normal butane as well as mixtures of normal and isobutane.

The dehydrogenation of normal butane and mixed butanes has become more important in view of the increased demand for high octane fuels. In the laboratory the same catalyst was used as for isobutane. For normal butane at 530-560°C., 26% conversion at 90-93% yield was obtained for over 200 hours with 4-hour reaction periods. The dehydrogenation of mixed butane gave even

RESTRICTED

RESTRICTED

(Page 116 contd.
and Page 117)

more favorable results. Using 4-8 hour reaction periods a 30% conversion was reached at 90-95% yield and a space velocity of 1000 volumes butane/v/h. In a 25 liter reactor an aluminum hydroxide (Oppau) impregnated with ammonium bichromate was used for over 200 hours with normal butane. The conversion was 22-24% and the yield 84-90%. The abrasion was quite high because of the irregular catalyst shape.

c) Dimerization of isobutylene

Additional catalyst experiments were carried out for the improvement of the asbestos-phosphoric acid plant for commercial operation.

d) Isomerization of normal butane over tungsten sulphide

The isomerization over tungsten sulphide is carried out best at a temperature of 415°, a pressure of 100-200 atmospheres, and a hydrogen partial pressure of 40-50%. The life of the catalyst is approximately six months. At a space velocity of 0.5 the conversion is 30-40% isobutane and the yield of isobutane is 85-90%.

Isomerization of normal butane over aluminum chloride

Operation in the vapor phase is preferable at a temperature of about 100°C. and a pressure of 15-17 atmospheres. It is necessary to maintain an HCl partial pressure of 10-20%. The aluminum chloride consumption is 1-2% of the isobutane formed at a conversion of 40-50% and yields of about 95%. The reactor is a tube filled with aluminum chloride and packing (Raschig rings, etc.) and heated with steam. A semi-commercial scale plant has been built in Me 52 and will be started up shortly.

e) Alkylation of isobutane with butylenes using sulphuric acid.

After the laboratory had established the chief operating conditions such as high isobutane concentration and good mixing of the reactants a small pilot plant was built in Me 244. The reactor consists of an agitator.

RESTRICTED

RESTRICTED

(Page 117 contd.)

Good mixing and removal of the heat of reaction (290 Kcal/kg. of alkylate) were attained at a reaction temperature of about 0°C. and auto-refrigeration. The sulfuric acid consumption is 5-7% of the alkylate formed. With charging stocks containing normal butane the reaction mix should contain about 40% isobutane. Further experiments are being made to study higher than theoretical yields which have been obtained. The alkylate has an octane number of 95. 80-95% boils below 170°C. A 2.5 T/D pilot plant has been built in Me 52.

Work for 1941:

1) Dehydrogenation

Further development of very active catalyst, particularly for normal butane and mixed butane.

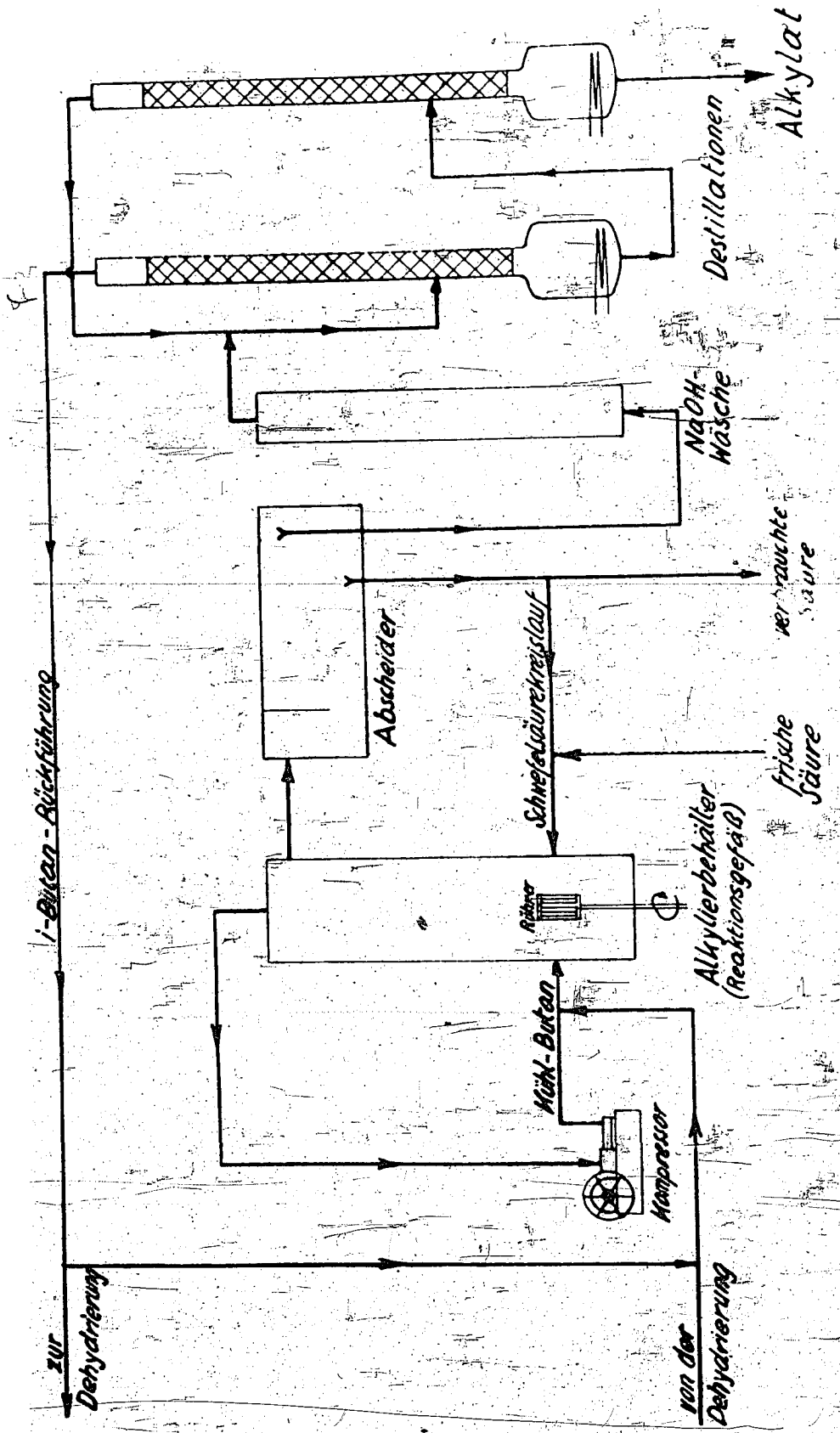
2) Isomerization

Application of small scale experiments to pilot plant scale.

3) Alkylation

Further study of operating conditions and work on pilot plant scale.

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Schema der Alkylierung

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(Drawing opposite page 117)

Flow Sheet for Alkylation

Dehydrierung	-	Dehydrogenation
Rückführung	-	Recycle
Kompressor	-	Compressor
Kühl-Butan	-	Butane refrigerant
Rührer	-	Stirrer
Alkylierbehälter	-	Alkylation reactor
<hr/>		
Frische Saure	-	Fresh acid
Schwefelsaurekreislauf	-	Sulfuric acid recycle
Verbrauchte Saure	-	Spent acid
Abscheider	-	Separator
NaOH Wasche	-	Caustic scrubber

RESTRICTED

RESTRICTED

(Page 118)

Chlorination Experiments on Gases and Oil from Hydrogenation

(Managing personnel listed)

Work in 1940:

a) Vapor phase chlorination

The pilot plant for the production of propane- and ethanesulfochloride was operated in order to gather further experience for the commercial plant. The present operating technique has proved successful. By the use of Osram burners (HgHS 1000-260 watt) it was possible to replace the jacketed quartz tubes by ordinary hard glass tubes. Ethanesulfochloride serves as a dye intermediate at Höchst. A commercial plant for the production of 40 T/M of propanesulfochloride is under construction. In the laboratory the sulfochlorination of low molecular chlorinated hydrocarbons was investigated. However, only small yields of the desired chlorosulfonic acid chlorides were obtained. Furthermore the sulfochlorination of methane was carried out at 10 atmospheres. In contrast to operation at atmospheric pressure where only traces of methanesulfochloride were obtained, yields of 40% were reached based on chlorine used. The work on the determination of the structure of the products obtained in sulfochlorination of light hydrocarbons was concluded successfully.

b) Oil chlorination.

Most work continued to be done on sulfochlorination, particularly of Mepasin. The process developed in the pilot plant has been successful on a commercial scale. The sulfochlorination of hydrogenated natural petroleum became important because of the expansion of Mersol production which completely exhausted the charge stock available from Kogasin. A large number of hydrogenated petroleum fractions were sulfochlorinated and sulfonates were obtained from various oils which have properties similar to Mesapon N from Mepasin. Special interest has been placed on the use of chlorinated bottoms. Experiments are under way to use these as fuel in Diesel motors.

RESTRICTED

RESTRICTED

(Page 118 contd. and
Page 119)

Experiments on their dechlorination, hydrogenation and re-use for sulfochlorination have not been completed.

Experiments for carrying out the sulfochlorination process continuously have shown that this method does not give higher yields than the discontinuous method.

Experiments to free the chlorinated hydrocarbons produced in sulfochlorination of chlorine dioxide and sulfur dioxide show that the former can be removed by passing the gases over charcoal and the latter by scrubbing the gases with selective solvents such as 90-95 sulfuric acid or xlenol.

The extraction of Mepasinhemisulfochloride with liquid SO_2 were carried out on pilot plant scale. The temporary plant did not yield the highly concentrated Mersol E expected on the basis of laboratory experiments. This problem was handled in cooperation with the Edleanu Company which will construct the commercial plant.

By the action of chlorine and thionylchloride in the presence of light on paraffinic hydrocarbons, chlorinated sulfinic acid chlorides were obtained. The production of Mepasinsulfamid (Mesulfam) was carried out on pilot plant scale and the former laboratory results were confirmed. Mesulfam is important as a soap raw material and as an intermediate product for textile aids. A 60 T/M plant was planned.

For special purposes (emulsion polymerization and shampoos), the production of a salt-free Mesapon N was investigated. At the present time the most promising process appears to be the extraction of Mesapon N with methanol.

The Mesamoll production was applied to commercial scale. Since Mesamoll can be used only to a limited extent in the "Kaschierung" of jute, experiments were started to improve its gelation ability by the choice of special Mepasin fractions as charge.

RESTRICTED

RESTRICTED

(Page 119)

Work for 1941:

Vapor phase chlorination.

a) Participation in planning the construction of a plant for the sulfochlorination of saturated gaseous hydrocarbons.

b) Further work on the pressure sulfochlorination of methane.

Oil chlorination.

a) Further development of natural petroleum sulfochlorination possibly under pressure.

b) Participation in the planning and starting of the Mesulfam and Mersol E plants.

c) Further developments in the production of salt-free Mesapon N

d) Development of sulfonic acid ester types as softeners.

RESTRICTED

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(Page 120)

Work on Phenolic Oils

(Managing personnel listed)

Work in 1940:

In connection with the purification of phenolic oil it was possible to make the phenolic oil M usable for Mesamoll production by catalytic treatment with air. The problem of purifying the soda containing M-phenolic residues was solved by washing them with a sodium acid sulphate solution.

For the purpose of producing phenol it was attempted to demethylate cresols catalytically with hydrogen. The results were however less favorable than with the previously investigated xylenol demethylation experiments. While in that case the reaction was largely the splitting off of methyl groups the demethylation and the reduction of hydroxyl groups appear to take place at about the same rate in the case of cresols.

For the synthesis of alkyl phenols with long side chains, cresols were reacted with high molecular alkyl chlorides over zinc dust with good yields. The chlorides were obtained in the desulfurization of Mersol E.

Catalysts based on zinc alloys for the dehydrogenation of cyclohexanol and methyl hexanol were further developed. By the addition of metals such as nickel, cobalt, manganese, and iron, the reaction temperature could be lowered to as low as 420°C. Galvanized iron was surprisingly good. The yellow mixture of dicarboxylic acids (R acids) produced in the oxidation of cyclohexanol to adipic acid could be made colorless by coupling with diazonium salts and by recrystallization another 25% pure adipic acid was separated.

In the synthetic resin field copolymers of propane disulfamide, formaldehyde and phenols were prepared. The products showed a certain water sensitivity which could be improved by increasing the phenol content of the charge. The resins from "Methylanon" which are similar to the AW2 resins are particularly interesting because of their solubility in alcohol.

RESTRICTED

RESTRICTED

(Page 120 contd.)

The alkylation of aromatic hydrocarbons was thoroughly investigated particularly in view of the production of toluene from benzene and methanol. A number of catalyst improvements could be made on the process which was taken over last year by the "Benzolverband." Numerous experiments were made in connection with the development of phosphoric acid, diatomaceous earth and zinc oxide catalyst whose life is about two months. By a daily reversal of the direction of flow in the reactors the loss of phosphoric acid catalyst was reduced and corrosion by the volatilized phosphoric acid was eliminated by passing the gases through lime. At a benzene methanol mole ratio of 4-1 and at 240°C., 60% of the methanol was converted to toluene. The rest formed alkylated benzene, dimethyl ether, and methane.

For the purpose of producing a high octane safety fuel it was attempted to react the alcohols of the isobutyl oil (C₆-C₁₁) with benzene. Similar catalysts were used as for the toluene synthesis. Only 25% of the alcohols charged were converted to alkyl benzenes, the remainder was dehydrogenated to olefins.

Work for 1941:

- a) Phenol purification
- b) Dealkylation of high boiling phenolic oils
- c) Dehydrogenation of "Dimethylanolen"
- d) Resins from "Dimethylanon"
- e) Benzene alkylation with higher alcohols in a 1 kg. reactor
- f) New methods for making adipic acid from benzene
- g) Purification of R acid

RESTRICTED

RESTRICTED

(Page 121)

Amines and Plastic Raw Materials

(Managing personnel listed)

Work in 1940:

a) Amination of carboxylic acid.

Catalyst tests for the amination of stearic acid were carried on in the pilot plant. It was found that only a 3 NiS.WS₂ catalyst prepared by precipitation yielded an Octodecylamine of 97% purity, while the use of a catalyst prepared by dry digestion yielded an amine of less than 90% purity. This catalyst also has a shorter life.

A precipitation catalyst which is first pulverized, then pressed into blocks, and then brought to the correct grain size by breaking, yielded an octodecylamine of about 90% purity. Here, too, the activity declined rapidly.

b) Preparation of diamine

The preparation of aliphatic diamines required for plastics was carried out in the laboratory in the following ways:

Diamines from aromatic compounds

The process for the preparation of adipic nitrile from adipic acid and ammonia was improved by the development of more active catalyst from tungstic acid and on the basis of these experiences a plant for the production of 30 T/M of adipic nitrile has been planned. The dinitrile will be used for the production of hexamethylenediamine necessary for polyamide plastics.

Diamine from aliphatic compounds

The following method was investigated further: isobutylene to isobutanyl chloride to diisobutanyl chloride to dichlorooctane to 1,5 diamino 2,4,4 trimethylpentane. The particular phases investigated were the dimerization of the isobutanyl chloride and the conversion of the dichloride to the corresponding diamine. Both

RESTRICTED

(121 contd.)

dichlorides can be converted with good yields to diamine by means of ammonia in the presence of water under pressure in silver autoclaves. The diamine obtained is being investigated for its use as a superpolyamide component.

A second method for the preparation of amines is being investigated in which sulfochloride is converted via desulfurization of the chlorides obtained from it to amines and similarly from disulfochlorides to diamines.

c) Higher glycols

By the reduction of methyladipic acid dimethyl ester in the presence of a copper-bariurchromite catalyst a 60% yield of methyladipol was obtained which is important as a component of the newly developed poly-urethane plastics and as a plasticizer for polyamide resins. Difficulties are still being encountered in the use of this reaction on a larger scale.

Work for 1941:

- a) Continuation of catalyst experiments.
- b) Completion and startup of the dinitrile plant as well as nitrile hydrogenation experiments on a larger scale. Carrying out of the diamine synthesis from aliphatic compounds in larger autoclaves.
- c) Pilot plant experiments and catalyst studies for the preparation of methyladipol.

RESTRICTED

(Page 122)

Oxo Experiments

(Managing personnel listed)

Work in 1940:

The process developed by Ruhrchemie for the addition of carbon monoxide and hydrogen to olefins with the formation of aldehydes in the presence of metallic catalysts, such as iron, and particularly cobalt, was investigated on the basis of the Oxo contract (I.G., Ruhrchemie, Henkel). The aldehydes obtained can be reduced to alcohol or oxidized to fatty acids, whereby valuable soap raw materials are obtained if high molecular olefins are used as charge.

Beyond the known facts developments were made in the catalyst field through the production of low cobalt, non-carbonyl forming catalysts. Particularly useful was a low cobalt copper catalyst. Experiments in the laboratory and other plant investigations, which covered catalyst studies and the use of various olefins for the determination of the structure of the reaction product as well as the reaction mechanism, were used to gather information for the design of a large-scale pilot plant for the production of 500 kg. Oxo products per day.

Work for 1941:

Design and operation of the pilot plant.

Experiments on the extraction of Oxo aldehydes from mixtures with paraffin hydrocarbons.

Further oxidation experiments with Oxo aldehydes.

RESTRICTED

RESTRICTED

(Page 123 contd.
and Page 124)

eliminate the harmful sticking together of the catalyst particles during processing.

e) Hydrocarbon synthesis catalyst

The zinc oxide- aluminum oxide- cobalt catalyst was further developed by varying the components during the production so that a good yield of high melting paraffins could be obtained. In the case of the iron precipitation catalyst aluminum oxide has so far proved to be the best additive. Preliminary work for the production and investigation of metallic and oxide sintered and melt catalyst are in progress.

f) Demethylation of cresol

The best catalyst is cobalt on bleaching clay. The same catalyst with other oxide additions (Cr_2O_3 , WO_3) was not improved. Further work is being done on a cracking catalyst with small cobalt addition.

g) Oxo process

A low cobalt copper catalyst was developed which gives optimum aldehyde yields at the lowest carbonyl formation. This was followed by studies on suitable carriers as well as exchangeability of other metals for copper. Investigations on the production and reduction conditions of the catalyst are under way.

h) Miscellaneous organic-chemical catalytic reactions

An active catalyst based on tungstic acid was developed for the production of adipic acid dinitrile from adipic acid and ammonia.

A useful catalyst was developed (though with a rather short life) for the hydrogenation of diisobutyl chloride, by the reduction of nickel oxide with electrolytic hydrogen.

A copper-barium chromite catalyst was developed for the reduction of methyl adipic acid ester to methyl adipol which has been satisfactory in small

RESTRICTED

RESTRICTED

(Page 123)

Catalyst and Physical Chemical Experiments

(Managing personnel listed)

Work in 1940:

Catalysts were further developed or newly produced for the following fields.

a) Hydroforming

For the improvement of chrome oxide, aluminum oxide catalyst, more active catalysts were prepared using molybdenum oxide and aluminum oxide where particular attention was placed on the precipitation of the aluminum oxide hydrates. In order to reduce coke formation other oxides were added but so far with no noticeable effect. A partial or complete replacement of the aluminum oxide by titanium dioxide, zinc oxide or magnesium oxide will be attempted.

b) Catalytic cracking.

Of the silica gel aluminum hydroxide catalyst the one with a Si:Al ratio of 9:1 was found to be the most active. The activity was improved by peptization of the aluminum hydroxide with a less than theoretical quantity of nitric acid. The mechanical stability of the catalyst was improved by slow drying at up to 200°C. followed by heating to a maximum of 450°C.

c) Dehydrogenation of normal and isobutane.

After a thorough investigation of the precipitation and peptization, a highly active aluminum oxide catalyst was developed for the commercial production in the catalyst plant. Besides this, several precipitation and impregnation catalysts with molybdenum-, chrome and other oxides on alumina were tried at various alkali contents. This work is still in progress.

d) Toluene synthesis.

A preliminary commercial catalyst was developed which is composed of zinc phosphate, diatomaceous earth and phosphoric acid with a small amount of binder. In the further development of this catalyst it will be attempted to

RESTRICTED

RESTRICTED

(page 124 contd.)

scale experiments. Here, too, the activity of the catalyst, which has already been prepared in larger quantities, decreases rather rapidly.

1) Physical experiments.

An apparatus was developed for the determination of the degree of whiteness of textiles, which is used at present for whiteness measurement of washed textiles.

Work for 1941:

Development of the catalysts described under a) to h).

RESTRICTED

Analytical Laboratory

Dr. Zepf
Dr. Turk, Dr. Berns, Dr. Eckhardt, Dr. Heinemann, Dr. Reinert, Dr. Markus,
Dr. Mayrhofer, Dr. Ohle, Dr. Steudemann, Dr. Wetzel

The occupation (keeping-busy) of the labor was consistently good. The personnel stability was somewhat increased. In the second half year current investigations had to give way in the interest of development work. To further these works, which shifted more and more to the organic field, Dr. Eckhardt and Dr. Markus stepped out of the experimental laboratory into the analytical laboratory.

The inorganic group attempted to clarify the nature of the chemical bonding of alkali, sulfur and lime in coal. A rapid method for determining and separating ammonia and urea nitrogen in solutions and dung water was worked out. Methods of determination for alkali, phosphoric acid, polysulfide sulfur among others were checked and applied for special cases. Fusion relationships of brown coal and mineral coal ashes were studied and suggestions for improvement of the same by corresponding flux were made.

In the spectral analysis the methods were further pursued for the determination of the smallest amounts of lead in biological material. The refractometric determination of isobutane in butane gave satisfactory values in pure gases. Work is in progress on the determination of small amounts of carbon monoxide in the blood.

The organic group, besides developing works analyses, worked out procedures and methods for phenol determination in waste waters, of lower fatty acids together and for investigation of Mersal, Mesapon N and mesapon-containing soap products. The determination of the hydrogenation iodine number, a suggestion of the Anako, was tested out in its application to di-isobutylene and other products.

CONFIDENTIAL

(Page 125 contd.)

The gas analysis group was kept very busy due to putting into operation new plants, thus for example, in the preparation of dimethylamine, and worked out methods for determining by-products arising thereby. For the safeguarding of the air, test methods and filters were suggested, these finding application in fogging experiments. For the new hydrogenation works laboratory workers were constantly trained.

The hygienic group carried out investigations for the Frohse, Gapel and Leuna works. Methods were further developed with application of graded photometer. In connection with this an exchange of experience with institutes abroad took place.

For the control of waste waters and air outside observations were also carried out this year with the Wabolu-Berlin. Difficulties and a number of complaints about damages by waste waters, waste gases and flying dust were received by us. These were investigated. In three cases considerable damages had to be paid.

CONFIDENTIAL

RESTRICTED

(page 126)
and 127

Main Shops

General discussion of the activities at the various shops including machine shop, boiler shop, welding shop, blacksmith, steel erection, foundry, tinshop.

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(Page 128)

Material Testing

Work in 1940:

The substitution of Austenitic steels by ferritic chrome steels is possible in many cases. Experiments showed, however, that several weldable types on the market are not resistant to intercrystalline corrosion for plate thicknesses below 3 mm. Above 10 mm. the properties of the plates are indefinite so that only thicknesses between 3 and 10 mm. remain useful. Even then no heavy mechanical duty should be put on the welding seams.

The suggestion to use zinc in the form of pipes and valve stems in place of brass was in general not applicable in our case. However, we have studied the corrosion of zinc in water.

In the very high pressure steam plant very considerable and strange corrosion troubles were found on the heating elements of the Schmidt kettles which seem to be connected with the complete decarbonization of the pipe material below the point of corrosion. The occurrence of corrosion took place at the same time as the change of feed water from condensate to salt-containing water of low soda number. The explanation may be that the copper in the water initiates corrosion by a certain local activation of the pipe surface.

In the fight against intercrystalline corrosion the use of the "Izettier" process has been a considerable step forward. The process could be shortened considerably. The rapid "Tupfel" process, without which control in the shops and in the plant is not thinkable any more, was completed by the development of newer methods.

The necessity for working with substitute metals brought about a demand for protective coatings. This has given an impetus to the work on baked (Einbrenn) lacquers. The experiments with "Luphenen" of the I.G. Leuna are promising. They

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are being considered for the protection of condenser tubes. Heat conductivity measurements have been favorable.

The asbestos cement insulation of the hydrogenation reactors, which has been very good, had to be discontinued because of a shortage of asbestos.

It was possible to prepare a satisfactory insulation from refractory bricks with alumina cement mortar. The cement coating process which has been used on a very large scale for the protection of the inside surface of fuel containers could be improved by a testing method which measured the regularity and quality of the application.

Thorough experiments have shown that the granular slag from Me 240 could be used as a cement-like binder. In order to use all the available slag experiments are under way for the formation of bricks.

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Schwimmer-Höhenstand-Regulierung.

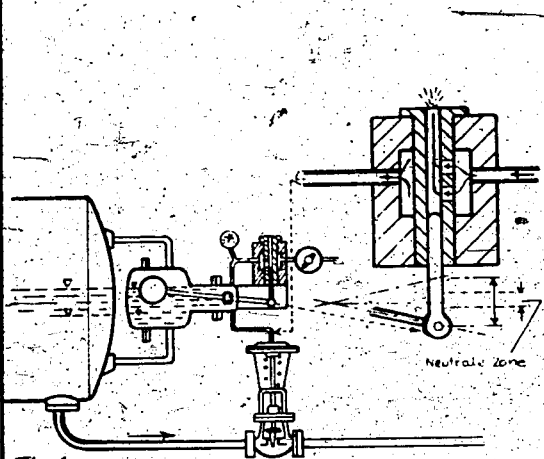


Fig. 1.

Pneumatischer Trennflächenregler

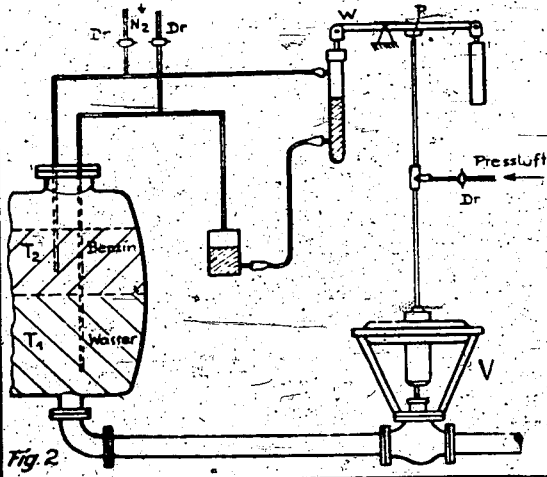


Fig. 2.

Elektrisch-pneumatischer Trennflächenregler

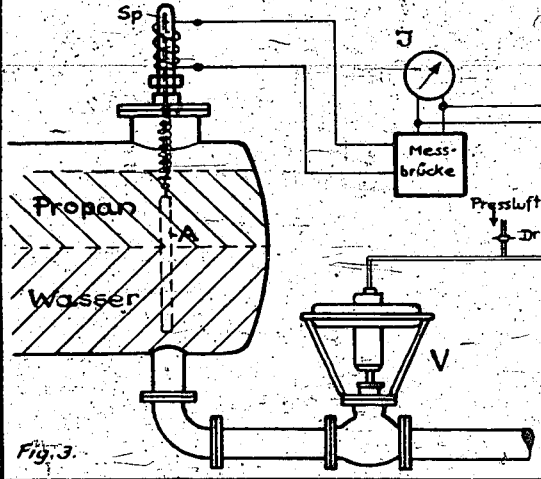


Fig. 3.

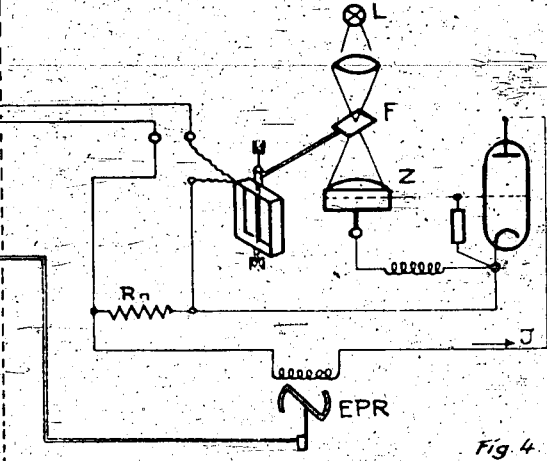


Fig. 4.

Elektrischer-Diff.-Druckmesser

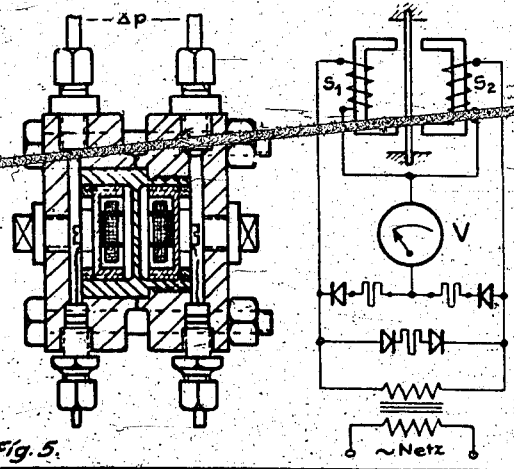


Fig. 5.

Schnellanalysiergerät für Phenolspuren in Zyklohexanol.

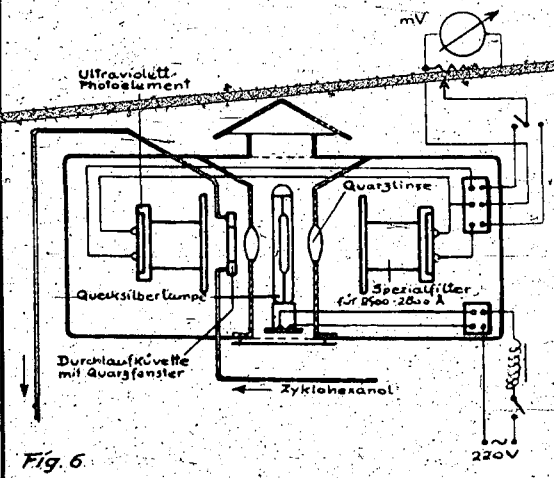


Fig. 6.

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(Drawing opposite page 129)

Fig. 1: Float Type Level Control

Fig. 2: Pneumatic Interfacial Level Regulator

Benzin - Gasoline

Wasser - Water

Pressluft - Compressed air

Fig. 3
and Electric-Pneumatic Interfacial Level Regulator

Fig. 4: Propan - Propane
Messbrücke - Measuring bridge

Fig. 5: Electric Differential Pressure Meter

Fig. 6: Rapid Analyzer for Traces of Phenol in Cyclohexanol

~~Ultraviolet Photoelement~~ ~~Ultraviolet photoelectric~~
cell

Quarzlinse - Quartz lens

Quecksilber lampe - Mercury lamp

Durchlaufkuvette mit
Quarzfenster - Cell for flowing sample
with quartz windows

Spezialfilter - Special filter

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Plant Control

Instrumentation:

A pneumatic interfacial level regulator was developed (see figure 2). Pressure difference of submersed tubes T_1 and T_2 affects plate P on the scale W and thereby the diaphragm valve V.

An electric pneumatic regulator (see figure 3) was developed. Float A affects the self induction of the coil Sp and thus the meter I. An electric pneumatic regulator R (see figure 4) controls the diaphragm valve V. The regulator uses photoelectric compensation and is universally applicable in all cases where something is regulated by the readings of an electric meter such as temperature measurements. The photoelectric compensation current I activates an electric-pneumatic relay EPR.

Laboratory measurements:

~~For measurements of differential pressure with the use of mercury, an~~ electric indicating diaphragmmeter has been developed for high pressures. Diaphragm movements of 0.03 to 3 mm can be transmitted linearly to the electric indicator. In order to indicate the square root directly from the differential pressure for the purpose of measuring gas flow rates, experiments were made. It will be necessary to maintain the plant voltage within 0.2% by means of voltage regulators, particularly in the case of complex installations.

A rapid analyzer was developed for the continuous measurement of 0.005 to 0.1% phenol in cyclohexanol. The spectrographic absorption instrument operates in the range of 2500 to 2800 μ A. A sufficiently selective filter was assembled so that measurements can be made with an ultraviolet sensitive photo element and an ordinary millivoltmeter (see figure 6).

Structural catalyst investigations by means of X-rays have been used extensively, particularly for the degree of oxidation and reduction of used

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(Page 129 contd.)

and fresh catalysts. An assumed copper cobaltite structure was found not to be existing in this way. In montmorillonite production a continuous control of the formation of rhombic swellable structures was used.

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(Page 130)

Electrical Plant

General discussion of motor repair shops and telephone system.

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Overhead Expenses and Inventory

1913 - 1940

Inventory

800

26400

24000

24600

19200

16800

14400

12000

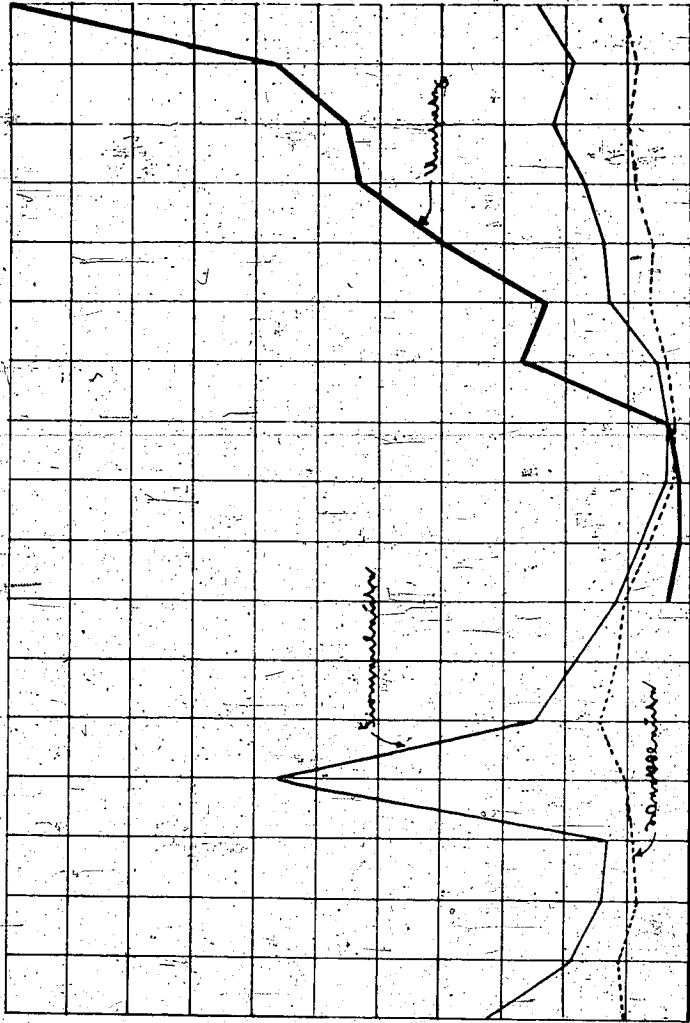
9600

7200

4800

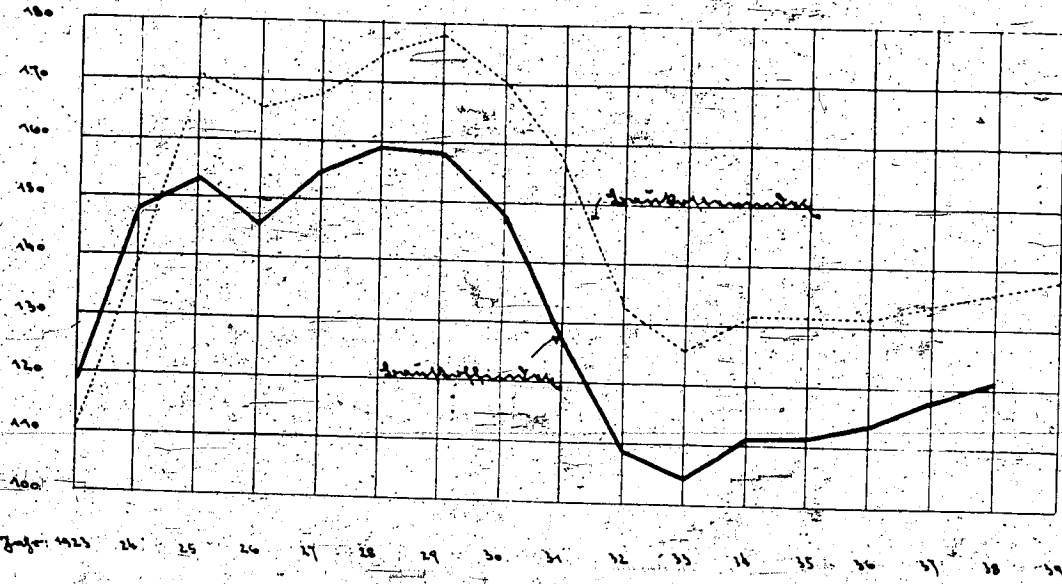
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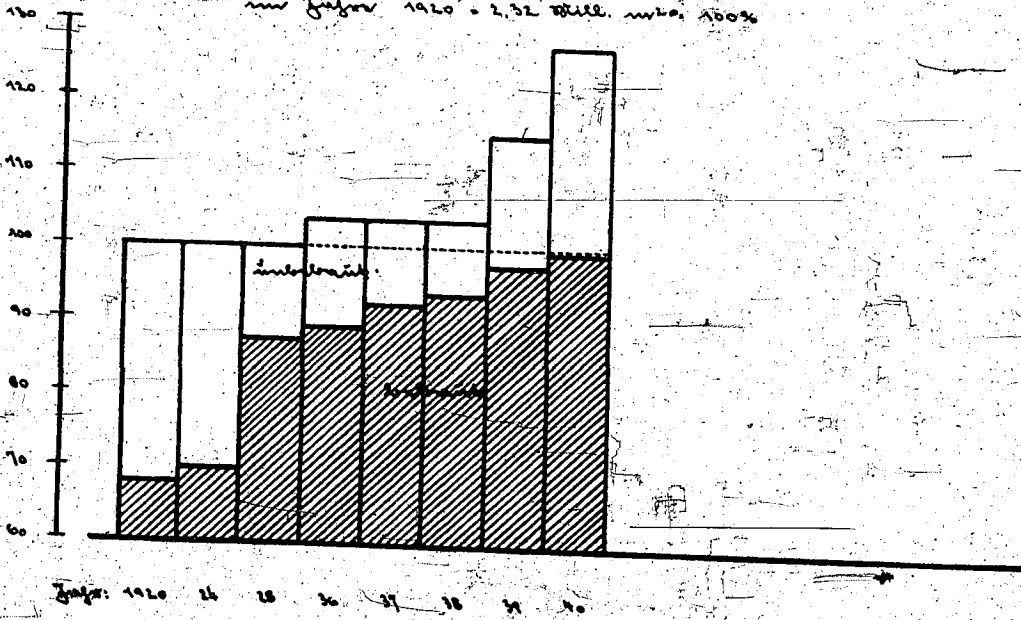
Year: 1913 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40

Lufttemperatur - und Luftfeuchtigkeit 1923 - 1939



Gesamtfläche innerhalb der Markungsgrenzung

im Jahr 1920 = 2,32 Mill. m² = 100%



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(Pages 131 and 132)

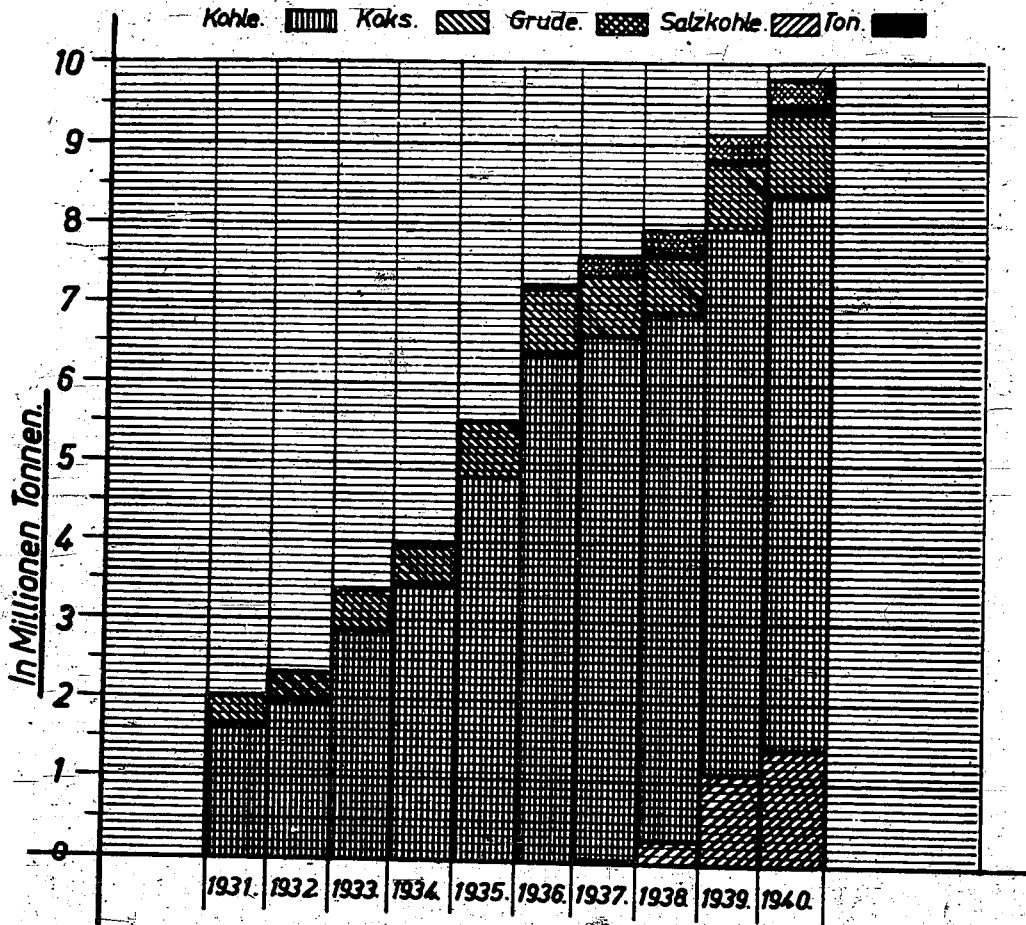
Construction Section

General discussion of construction problems with special emphasis on substitution for scarce materials. Includes two graphs.

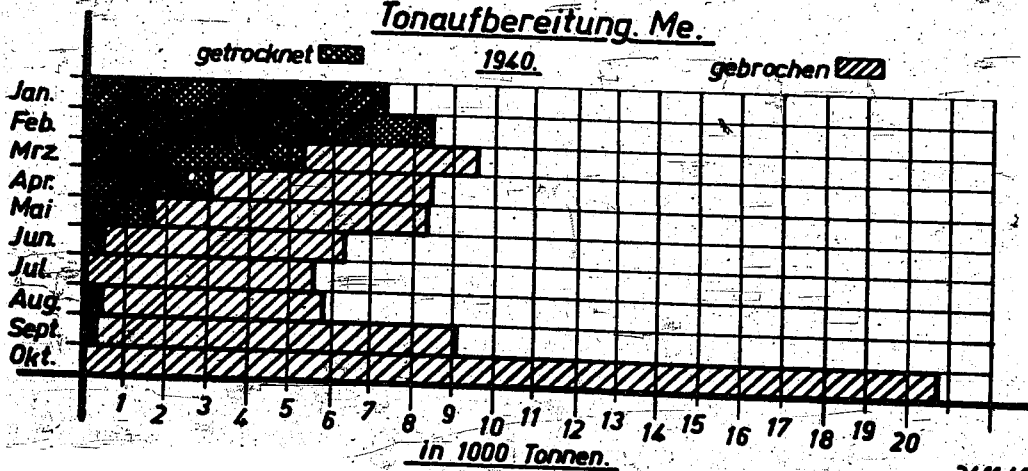
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Kohlen- u. Koks-Förderanlagen Me.

Förderleistungen 1931 - 1940



Ton aufbereitung. Me.



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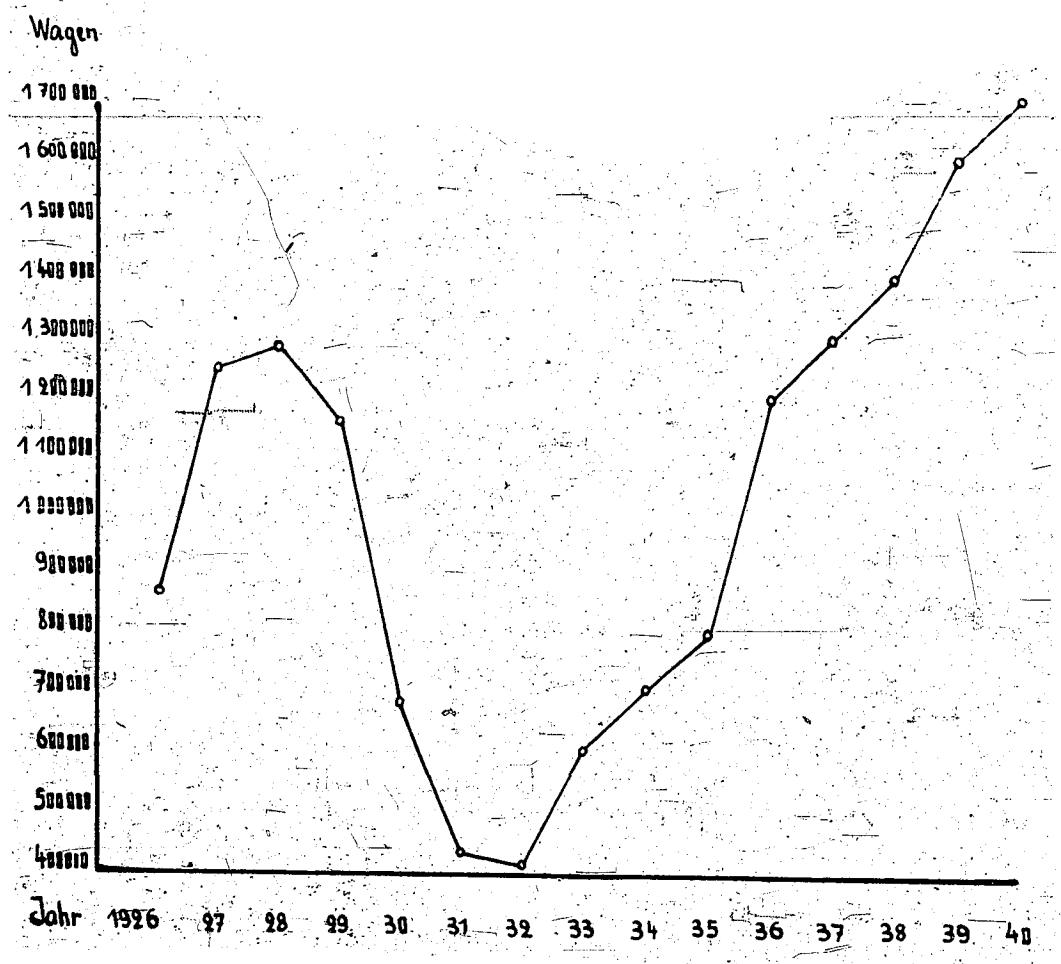
(Page 133)

Coal and Coal Mines

General discussion of transportation problems and statistical data on the production and disposition of coal, coke and clay. Includes graphs.

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Gesamtwagen - Umlauf



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(Page 134)

Railroad Operation

General discussion of railroad problems. Chiefly statistical data (including one graph) on the use of cars and addition of cars, locomotives, and auxiliary equipment.

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(Page 135)

Construction Office

Statistical data on personnel, amount of work done and the number of new processes put in operation.

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