Bag 2734 Target No. 30/4.11
Bottrop - Item 5

December 15, 1938

Report of Experiments of the Conversion of Coal in Extract Chamber 12

I. Purpose and Aim of Experiments

The function of extract chamber 12 is to convert the coal, which has been stirred with solvent to form a coal paste, into a filterable extract, which is then further treated in filtration building 13.

II. Equipment of Chamber and Processing of Product

The equipment consists of:

(1) Six regenerators
Inside diameter of jacket, 185 mm
Length, each, 12 meters
Number of coils (14 x 23 mm), each, 12
Heating surface, each, 15 square meters

The regenerators are arranged in such a way that the product in forward direction (pressure side) goes through the inner nest, and, in the reverse direction (suction side), around the nest.

(2) The preheater with 40 hairpin-rib tubes
Inside diameter, 70 mm; length, each 13 meters
Total length, 1040 meters; volume 4.02 cubic meters

(3) The autoclave having 48 hairpin tubes, of which the ascending branch has a diameter of 185 mm and the descending branch a diameter of 120 mm; length, each, 14 meters; total length, 1344 meters; volume, 23.66 cubic meters.

(4) The product cooler, consisting of 2 groups of 6 trays, each 20 meters in length; inside diameter, 70 mm; average cooling surface, 60 square meters.

The product coming from building 10 goes to the paste presses (see scheme of chamber in arrangement 1). The latter were built by the Esslinger Machine Works. Each press produces, at a maximum of 12 double strokes per minute, 16 cubic meters, at a maximum of 175 atm.

The product then passes through the nest of regenerators. Originally there were 6 regenerators installed. Owing to difficulties later to be described, a number of modifications were effected. In experiment 6 there were used only 4 regenerators, which were traversed in alternate directions. The cold product flows downward in regenerator 4, upward in regenerator 3, again downward in regenerator 2, and passes out of regenerator 1 into the preheater.

The product, which has been preheated to about 250°, is flowed through the preheater at a velocity of 1.08 meters per second, and is heated to the reaction temperature of about 415° by means of 12 superimposed burners. The heating-gas requirement amounts to about 540-630 cubic meters per hour at 4400 cal. Service and adjustment of the preheater are not simple and many experiments were required before an optimum heat distribution was found. One part of the flue gas enters through 4 valves into the autoclave space. In the beginning it was impossible to heat the latter perfectly uniformly. By increasing the velocity of the 4 lower burners, it became possible to equalize the temperature perfectly. Two Schiele blowers, type 1162, each having an hourly capacity of 110,000 cubic meters at a maximum of 500° served for circulating the flue gas. The temperatures of the flue gases in the preheater amounted to 430-480° and in the autoclave to 390-490°. The blast temperature was about 375°.

The product then enters the autoclave disposed in the interior of the preheater. The autoclave consists of 48 smooth hairpin tubes, of which the descending branch has a diameter of 120 mm, while its ascending branch has a diameter of 185 mm. By this construction any gas pockets that might form should be carried along with the strongest downward current. The flow velocity amounts to 15 cubic meters per sec. in the ascending tube, and 37 cubic meters per sec. in the descending tube at 15,000 liters per hour of injection. At 15,000 liters per hr of injection the coal mixture requires about 16 minutes for flowing through the preheater, and then remains for about 92 minutes in the autoclave.

Hereupon it gives up its heat to the inflowing product since it again flows in the regenerators upwardly and downwardly around the nest.

At about 210° the crude conversion product enters the product cooler where it is cooled to 140-150°. After passing the cooler the crude conversion product is expanded by one of the two pressure-release gauges, whereupon it flows to building 13, where it is filtered.

III. Brief Summary of Experiments 1 to 5

Duration of experiments. Reasons for termination.

The experiment which is herein described in greater detail is No. 6. Since none of the preceding 5 experiments were made with coal for any length of time, they will only be briefly mentioned here.

Experiment 1, from January 7-11, 1938, had to be terminated before any coal paste whatever could be injected since the regenerators were clogged with particles of dirt from the solvent and the lines.

In experiment 2 two rib tubes were installed in the suction side to take the place of the 6 clogged regenerators. Owing to a deficiency of heating gas the necessary temperatures were not reached.

Experiment 3 ran the longest, that is, from February 1st to April 10, 1938. Four regenerators were installed instead of the rib tubes. For the most various reasons, such as the occurrence of resistances in the regenerators, a poor degree of conversion, disturbances in the filters, etc, it was very frequently necessary to change over from coal to solvent.

In this experiment, however, a very large number of observations were made which could be utilized in the later experiments. The operation continued a total of 1434 hours with solvent and 177 hours with coal.

Experiment 4 ran from July 6-18, 1938. Obstructions again occurred in the regenerators, so that it was necessary to operate 2 units alternately. Soon after changing over to coal paste a great disturbance occurred in chamber 17, so that building 12 also had to be shut down.

Experiment 5, from Aug. 28-Sept. 13, 1938, was continued only for a short time with coal because a disturbance occurred when using coal paste.

IV. Experiment 6

Experiment 6 from October 15th to November 22, 1938 was the first one that ran for a long time without difficulty. The observations made on it are described in the following sections of this report.

For the sake of a better understanding, a number of expressions which frequently recur will first be defined.

The coal is stirred with the solvent. By "solvent" is meant a mixture consisting of about 60 parts of tetraline and about 20 parts each cresol and naphthalene. The content of coal in the paste is called the "coal concentration." The ash content of the pulverized and dried coal is called the "ash in the starting coal."

The product coming from the chamber is called the "crude conversion product." The latter is separated into "pure filtrate" and "residual coal," in the filtration plant, and washed with solvent, whereby the "washing filtrate" is obtained.

The degree of conversion of the starting coal is calculated as follows:

$$\text{Degree of conversion} = 100 \cdot \left(1 - \frac{\text{ash of starting coal}}{\text{ash of residual coal}}\right) \%.$$

If it is desired to refer the degree of conversion to pure coal, then the percentual value is multiplied by

$$\left(\frac{100 - \text{ash in starting coal}}{100}\right)$$

By the "filtration time" is meant the number of seconds necessary for 100 grams of crude conversion product to filter through a suction filter 9 cm. in diameter provided with a No. 597 Schleicher and Schöll filter. During the earlier experiments the temperature was maintained at 120°. In experiment 6 it amounted to 150°, since this temperature corresponds to the conditions present in large filters.

By "charge" is meant the quantity (in metric tons) of dried crude-coal per cubic meter of autoclave space per hour. By "yield" is meant the quantity (in metric tons) of extract which is likewise produced per hour per cubic meter of autoclave space.

(a) Experiment 6 ran 811 hours or 34 days, of which 217 hours or about 9 days were required for starting and stopping. During the remainder of the time, for 467 hours or 20 days, there was injected coal paste, and, during the remaining 127 hours or about 5 days, crude conversion product; hence, a product which had already passed through the chamber.

The most important data of the experiment are listed in tables 2 and 3.

(b) The coal concentration amounted to about 31 to 33% and varied only to a very slight extent.

(c) The quantity of injection was maintained at 15,000 liters per hour, and was increased to 24,000 liters per hour only in the capacity experiment to be described later.

(d) The temperatures in the autoclave (elements 1a, 3a and 5a; see appendix 1 and tables 4, 5, and 6) were at first 22 mv or 425° at the inlet and 21 mv or 408° at the outlet. The temperature measurement was made with these elements in thermal connecting pieces which extended into the liquid stream. This method of measurement was found to be much more reliable than that in which the elements were at first merely tied but later welded to the upper tubes. By variation of the preheater and the flue-gas streams it was possible permanently to reduce and finally completely equalize the difference between the inlet and outlet temperatures (Tables 4 and 5). It was further found that it was also possible to obtain a good degree of conversion of the coal at temperatures lower than those earlier assumed. The temperature was gradually lowered to the point at which the filterability became perceptibly worse. Under the present conditions at 15,000 liters of injection the lower limit was 21 mv = 410°. This temperature was maintained with good results for 2 days (from November 5-7 1938). Since, however, the fluctuations in the heating value made it impossible to maintain this temperature precisely, the temperature was, for the sake of certainty, maintained at 21.3 to 21.4 mv = 413 to 415°. According to the observations of this experiment, therefore, this temperature seems to bring about the conversion of the coal in a reliable way (at 15,000 liters per hour of injection the product remained about 95 minutes in the autoclave and about 15 minutes in the preheater).

(e) The filterability was always good, except in 2 cases, and varied between 15 and 30 seconds for 100 cc of crude conversion product at 150°.

(f) The content of residual coal was usually between 6.5 and 7.5%.

(g) The degree of conversion calculated on pure coal varies between 83 and 86%.

The gasification in the crude conversion amounted to 20.3 cubic meters per ton of coal or 1.7% of the charged dried coal. The average analysis was: CO_2 plus H_2S - 2.5%, O_2 - 0.2%, CO - 2.0%, H_2 - 18.0%, hydrocarbons 75%, N_2 - 1.0%; C value = 1.6; heating value, 9180 kg. Cal; specific gravity, 0.814.

(h) The only great variations in this experiment occurred in the chamber back pressure. Even on the first day of the injection of coal paste the back pressure of the chamber, which had for such a long time amounted to 13 atm, rose to 32 atm. Measurement showed that the back pressure was always present only on the suction side of the two regenerators. On being demounted the latter were found to be extensively clogged with thin scales, which consisted mostly of iron sulfide. Regenerators I and III were completely clogged on the suction side, while regenerators II and IV still had a narrow passage.

The back pressure of the autoclave fluctuated slightly during the experiment between 2 and 3 atm. The deposits were slight. Large deposits, (known as Sparrow's nests) such as had been observed in the upper bends in the previous interruptions were not present, but only thin residues consisting of individual "warts."

Analyses of Deposits in Regenerators

	Solids %	Ash in Solids %	Iron Calc. on Solids %	Sulfur Calc. on Solids %
Regenerator I, suction side, top (inlet)	69.0	59.5	40.2	19.2
Regenerator I, suction side (middle)	70.0	35.0	15.7	8.6
Regenerator I, suction side, bottom (outlet)	86.0	40.6	19.0	10.1
Regenerator II, suction side, bottom (inlet)	75.0	48.8	24.0	13.5
Deposit in autoclaves	70.6	15.6	1.6	

The high content of iron sulfide would make it advisable to examine the equipment for corrosion after a long experiment.

In the preheater the back pressure amounted to 2 atm and slowly rose to about 4 atm. Most of the back pressure was present in the hottest part. On demounting it was found that some of the bends had large deposits here and there.

In summary it can be stated with regard to deposits that the latter were present practically only in the hottest part of the preheater and autoclave. Since their thickness amounted to a maximum of 6-8 mm, usually less, they could scarcely cause any disturbance. The hottest preheater tube was taken out and drilled. The contents amounted to about 2-3 kg.

At the outlet of the autoclave to regenerator I there was found a very curious phenomenon. The tube was completely filled up from the valve of the autoclave outlet to regenerator I with solid balls of about 2 cm in diameter. The surface of these bodies was partly smooth and partly notched, the interior structure was shell-like and arranged in layers around a core.

Instead of the first supposition that it was a question of fragments which had been broken off and deposited by the stream, it seems that it is caused by aggregations of residual coal which assume a spherical form on the way through the autoclave (solid-material, 90-92%; ash, 27-28%).

On releasing the pressure in the chamber a back pressure was found ahead of pressure reducing gauge 1, so that gauge 2 had to be connected. On demounting it was found that there were present ahead of the latter pressure-reducing valve fragments similer to those ahead of regenerator I (solid material, 72-85%; ash, 20-26%).

The regenerators are the most sensitive part of the whole installation. In the earlier experiments the regenerators usually became clogged (see report on experiments 1 to 5). In particular the greatest back pressure was found on the suction side. The product passes through the nest of tubes into the autoclave and then around the nest. If any kind of solid particles form in the preheater or autoclave, they are deposited between the tubes, particularly at the inlets and outlets. Since these intermediate spaces are very narrow only a handful of small shell fragments is sufficient to bring about a considerable increase in the back pressure.

Even on the first day of experiment 6 such a disturbance occurred a few hours after the injection of the coal paste, and the differential pressure increased more than 30 atm (on October 26, 1938). It was possible, however, slowly to remove the obstruction by flushing with solvent. Frequent conversions to crude conversion product had a very good influence on the back pressure and nearly always led to a decrease in the chamber differential pressure.

On November 14, 1938, however, the differential pressure increased to 62 atm, so that destruction of the tube of nests in the regenerators was feared. Operation without regenerators was therefore attempted. It was found that this was possible without any difficulty at 15,000 liters of injection (table 6). The consumption of heating gas increased about 30-40% (from 600 to 900 cubic meters). The product cooler was sufficient to cool to about 150°. It was only in the capacity experiment that the failure of regeneration was disturbingly manifested.

(i) Up to the last day 15,000 liters per hour were injected into the chamber, or 0.21 metric ton of dried crude coal per cubic meter of autoclave volume per hour. In this case the yield in metric tons of extract amounted to 0.17.

For the purpose of establishing the maximum output, on November 21, 1938, the operation was started with 20,000 liters per hour of solvent at 21.8 mv at elements 1a and 3a, and then changed over to 20,000 liters per hour of coal paste. The quantity was converted satisfactorily, and the filtering time amounted to about 20 seconds. Then it was slowly increased to 24,000 liters per hour. At 23,000 liters, or 0.32 metric ton, of charge the conversion was still good; however, the preheater was no longer sufficient to bring this quantity to the desired reaction temperature of 21.8 mv. The temperature dropped to 21.0 mv, and the charge had to be reduced to 20,000 liters per hour. As the result of this experiment it can be stated that it is entirely possible to put through 25,000 liters per hour. It also seems that the charge can be increased to 30,000 liters per hour and higher. This can, however, only be done by having a larger preheater or by providing a suitable regeneration. Instead of the nest regenerators heat exchange by jacket tubes or double coils should probably be considered.

(k) No noteworthy defects were found in the mechanical arrangement of chamber 12. The paste presses have been operating without giving any trouble since the beginning of the experiments. The load of presses 1 and 2 has usually been 5,000-10,000 liters per hour, although for a few days it was 15,000 liters per hour. There was no leakage from the packed boxes in the case of coal, although a slight amount of leakage occurred in the case of crude conversion product and particularly with solvent.

In the earlier experiments it had been found that the introduction of flushing oil to the measuring points was very important. If this is not done, clogging soon occurred. Originally only a Bosch injection pump for Deisel motors was available as the flushing-oil pump. Ordinary solvent was used for flushing. This injured the very precise grinding of the plunger, and the pump became unusable. The three-stage pump from the old Meiderich Tar Works was much better. Its maximum capacity amounted to about 2700 liters per hour but only 400 liters per hour were needed. The 2 small two-stage pumps of the Muller Company of Esslinger, which had previously served as flushing-oil pumps of chamber 17, were connected as reserves.

In the beginning of the experiment there was available only a flushing-oil tank of 5 cubic meters, which was connected with all of the paste presses and the flushing-oil pump. With the frequent changeovers from coal paste to solvent which occurred at that time, it was impossible to prevent a little coal paste from being formed into the flushing-oil line. The result of this was constant clogging in the capillaries of the measuring lines. Moreover, the tank was much too small to enable the chamber to be properly flushed with solvent under a serious disturbance. For this reason a larger tank of 25 cubic meters was installed. The smaller tank serves as a suction tank for the flushing-oil pumps, and is filled with pure tetraline. The large tank is permanently connected only to coal press 3. The flushing-oil lines to the two other presses are closed off. These two expedients have proven entirely satisfactory. During the whole time of experiment 6 no clogging of the measuring lines occurred. The wear on the washing-oil pumps was also much less. Except in starting and stopping, no solvent is pumped out of the large tank. There were practically no change-overs from coal to solvent. Whenever it was necessary for some reason, crude conversion product was again flowed through the chamber. This procedure has also been found satisfactory.

After terminating the experiments, the whole apparatus was filled with pure tetraline for the purpose of preventing obstruction by deposits of naphthalene.

The temperature measurement of chamber 12 was improved in several ways. In the beginning the elements were connected at the upper bends of the tubes of the preheater and autoclave. Since this method of measurement was too inaccurate, the thermoelements were later inserted in welded-on tubes. For the purpose of establishing the actual temperature in the liquid stream, 3 elements in thermotubes were installed in the middle and at the ends of the autoclave. In doing so it was found that in the earlier experiments the temperatures had actually been measured about 20° too low. This also explains why deposits and obstructions occurred so frequently in the first experiments.

The pressure-release valves were demounted after experiment 6. In valve 1 the nozzle was badly worn, although the cone was in good conditions. Valve 2 did not exhibit any injuries.

Summary

On the basis of a number of preliminary experiments it has been found that coal can be treated with solvent in continuous operation. Experiment 6 ran for 811 hours from October 10th to November 22, 1938.

Stopping & starting, 217 hours
With coal paste, 467 hours
With crude conversion product, 127 hours

In the case of 15,000 liters per hour of injection of an approximately 30-33% coal paste the optimum temperature was 413-415°.

The filterability amounted to about 15 to 30 seconds for 100 cc of crude conversion product.

After various small modifications the mechanical arrangements operated satisfactorily. However, back pressures frequently occurred in the suction side of the regenerators, which finally led to the regenerators being omitted entirely. This is possible at 15,000 liters per hour of injection; above 20,000 liters per hour, however, the capacity of the preheater is no longer sufficient. A short experiment on the capacity showed that the chamber can be charged with 23,000 liters per hour or 0.32 metric ton of coal per cubic meter of autoclave volume per hour.

Additional experiments are required to determine to what extent a higher charging is possible, and what limit can be attained with suitable regeneration.

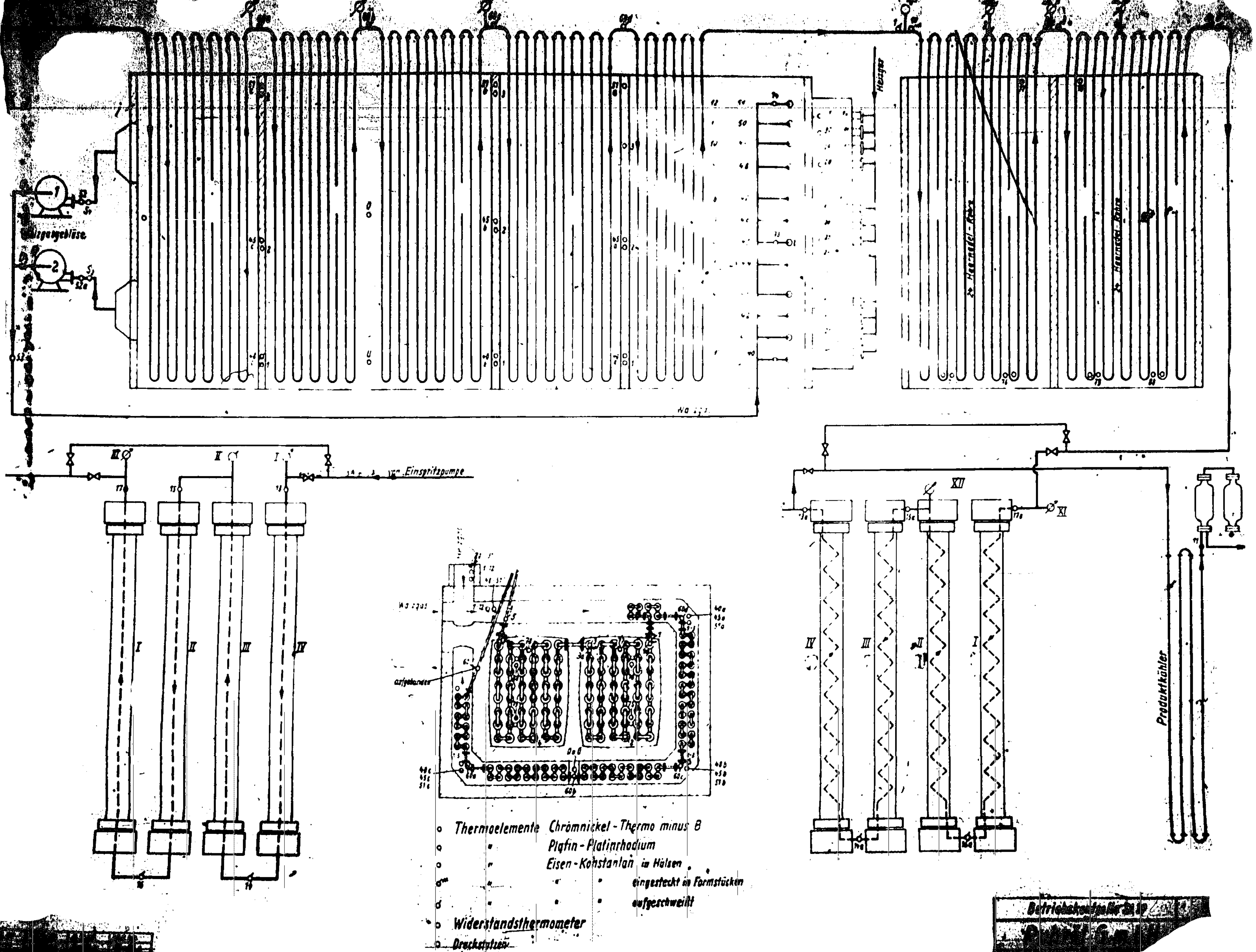
In experiment 6 there were introduced a total of:

2420 metric tons of dry coal
5462 metric tons of solvent.

Enclosures:

(1) Drawing of chamber showing location of thermocouples
(2) and (3) Graphs of important data
(4) (5) and (6) Graphs ~~of~~ showing temperature distribution

…stellen-Schema für die Rohaufschluß-Kammer St. 12



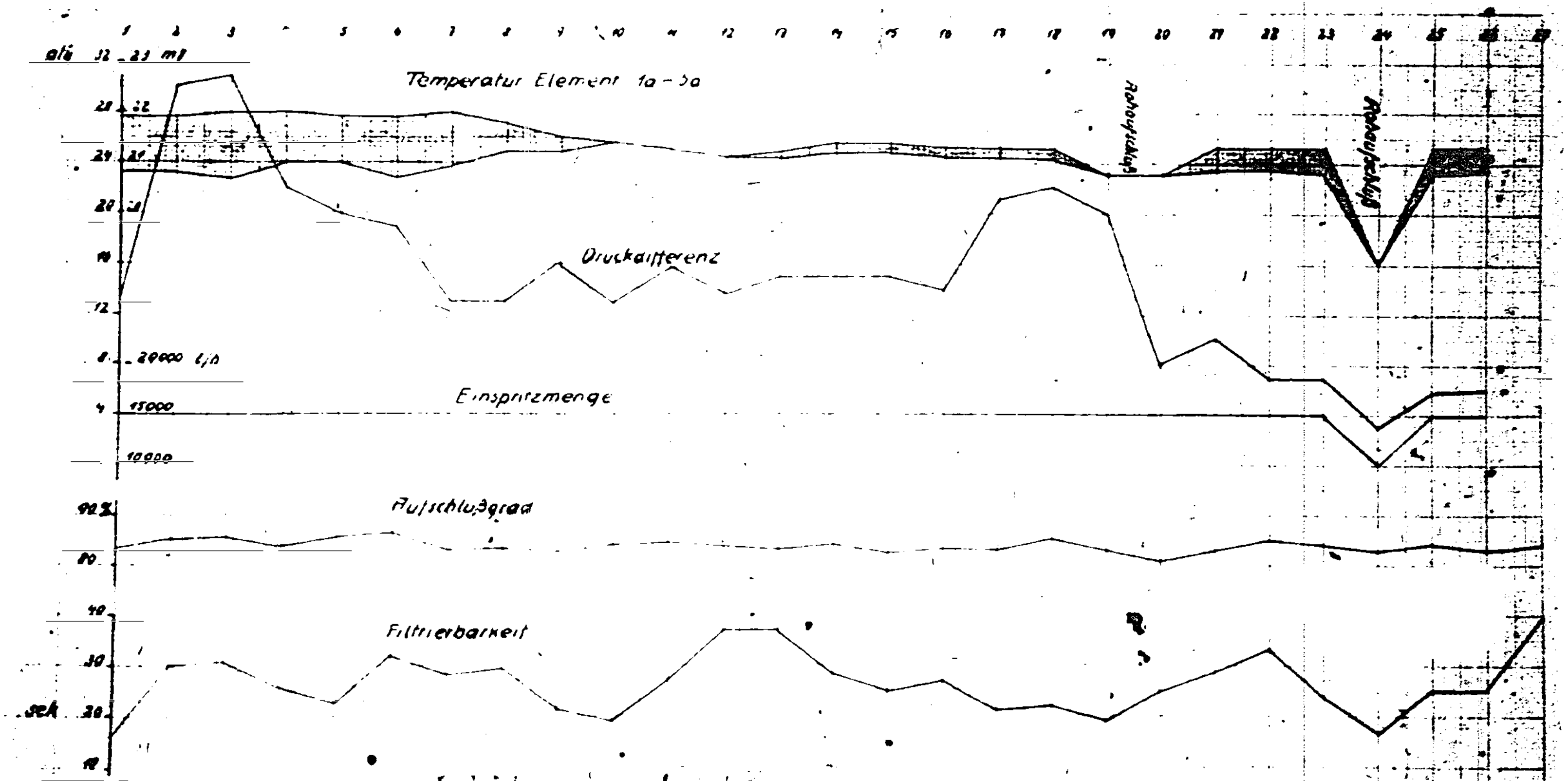


Tabelle 2 zum Bericht über Rohaufschlußkammer 42

9. III. 34

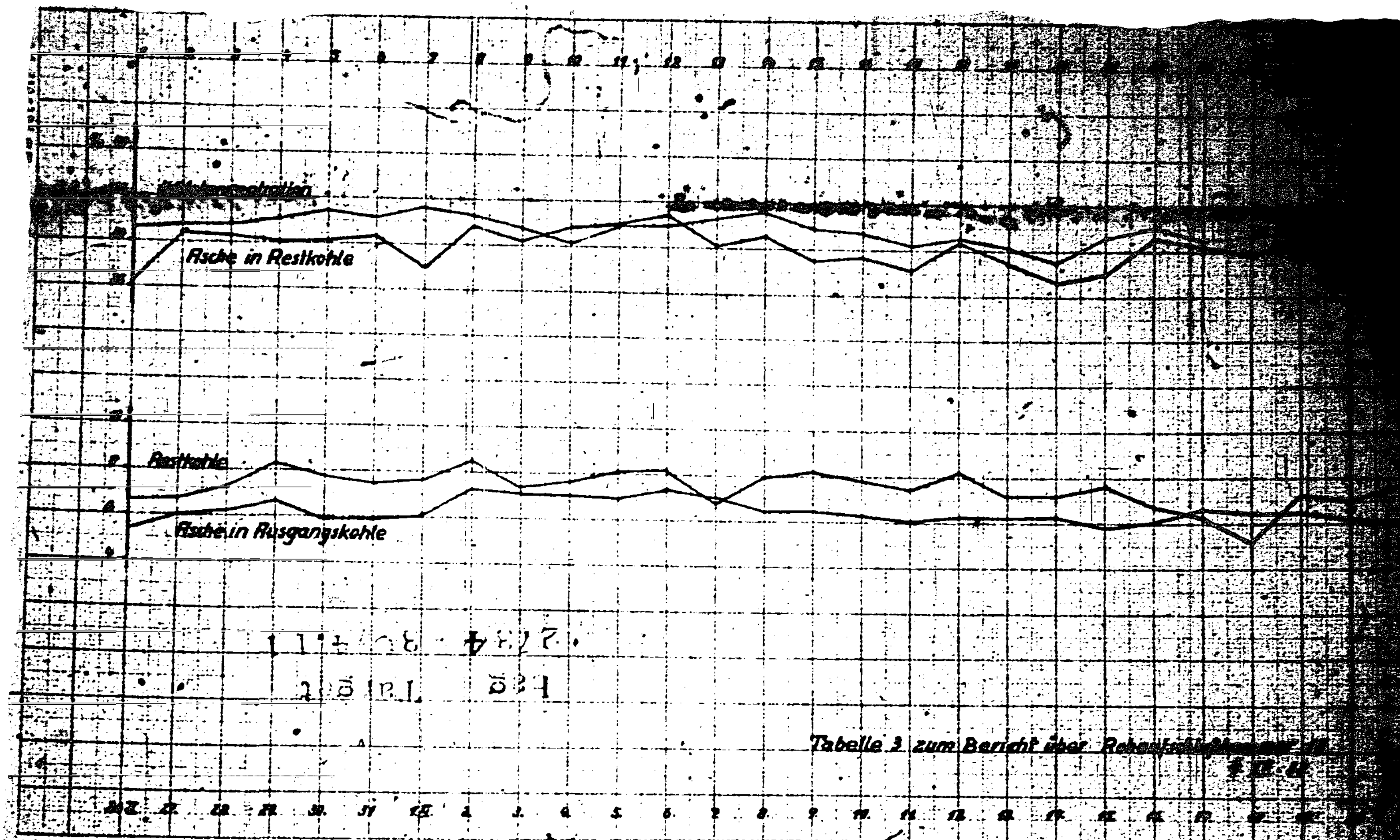
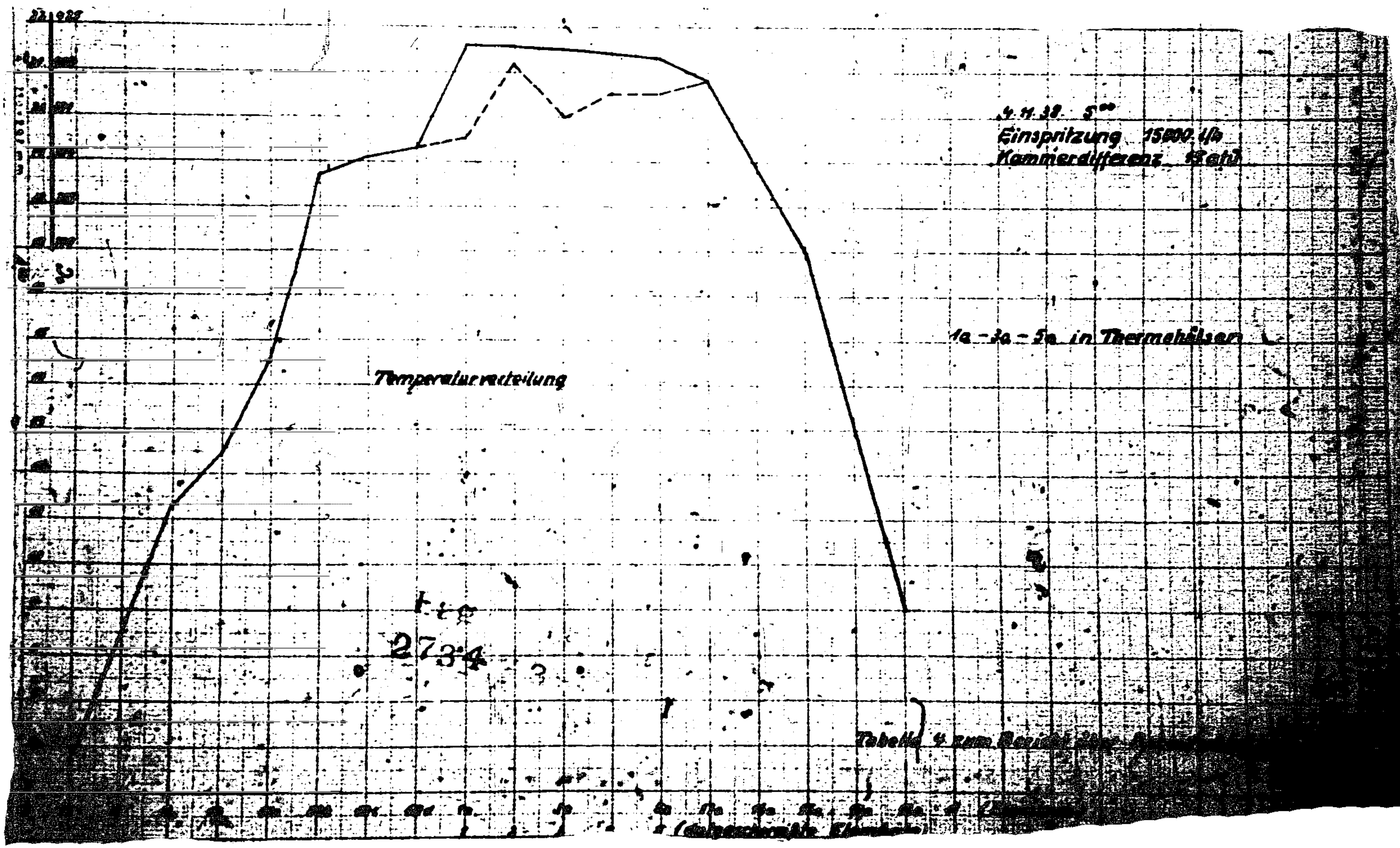


Tabelle 3 zum Bericht über


4.11.38. 5⁰⁰
Einspritzung 15000 l/h
Kammerdifferenz
1a - 3a - 5a in Thermohülsen
Temperaturverteilung
2734
Tabelle 4 zum Bericht

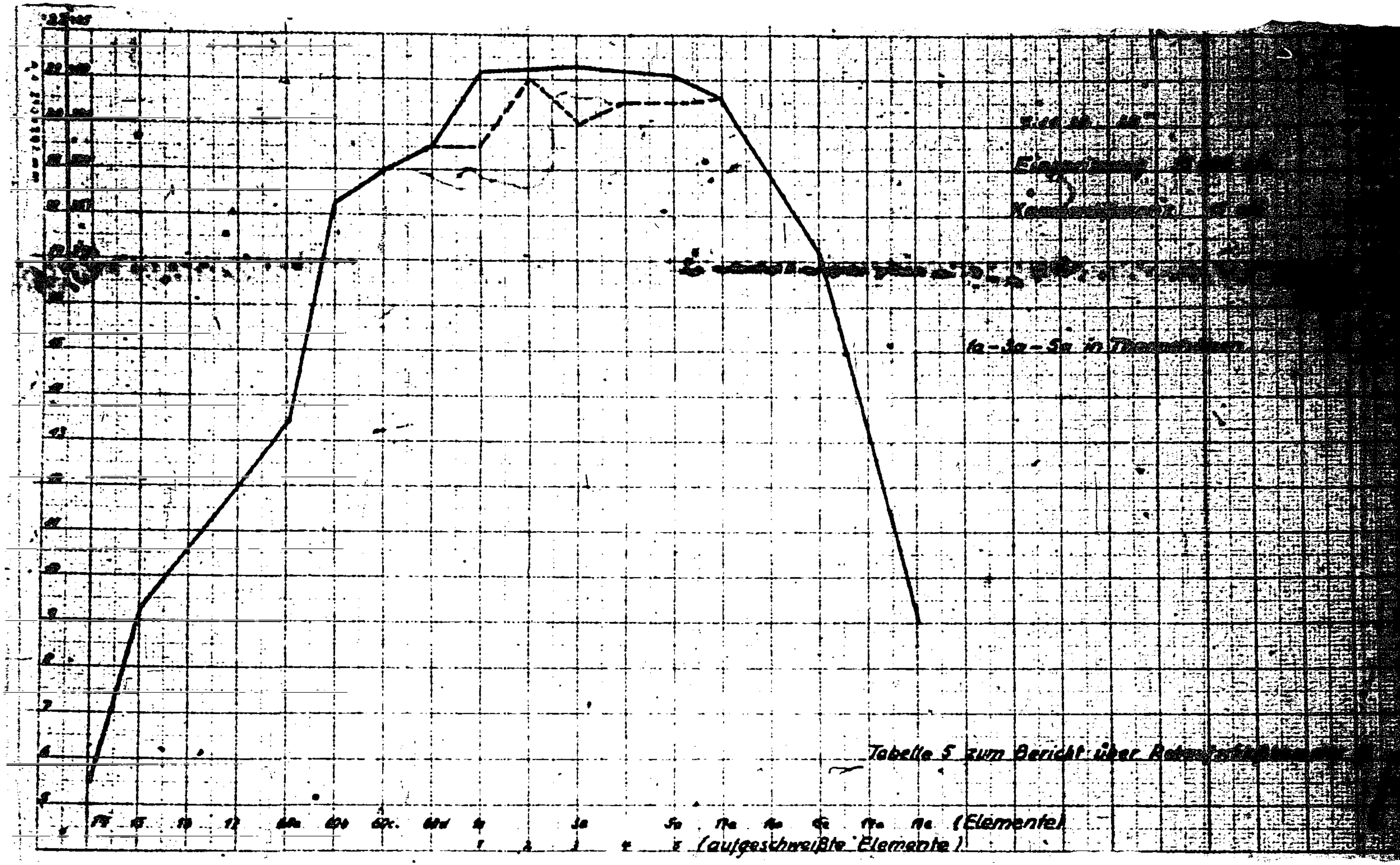


Tabelle 5 zum Bericht über

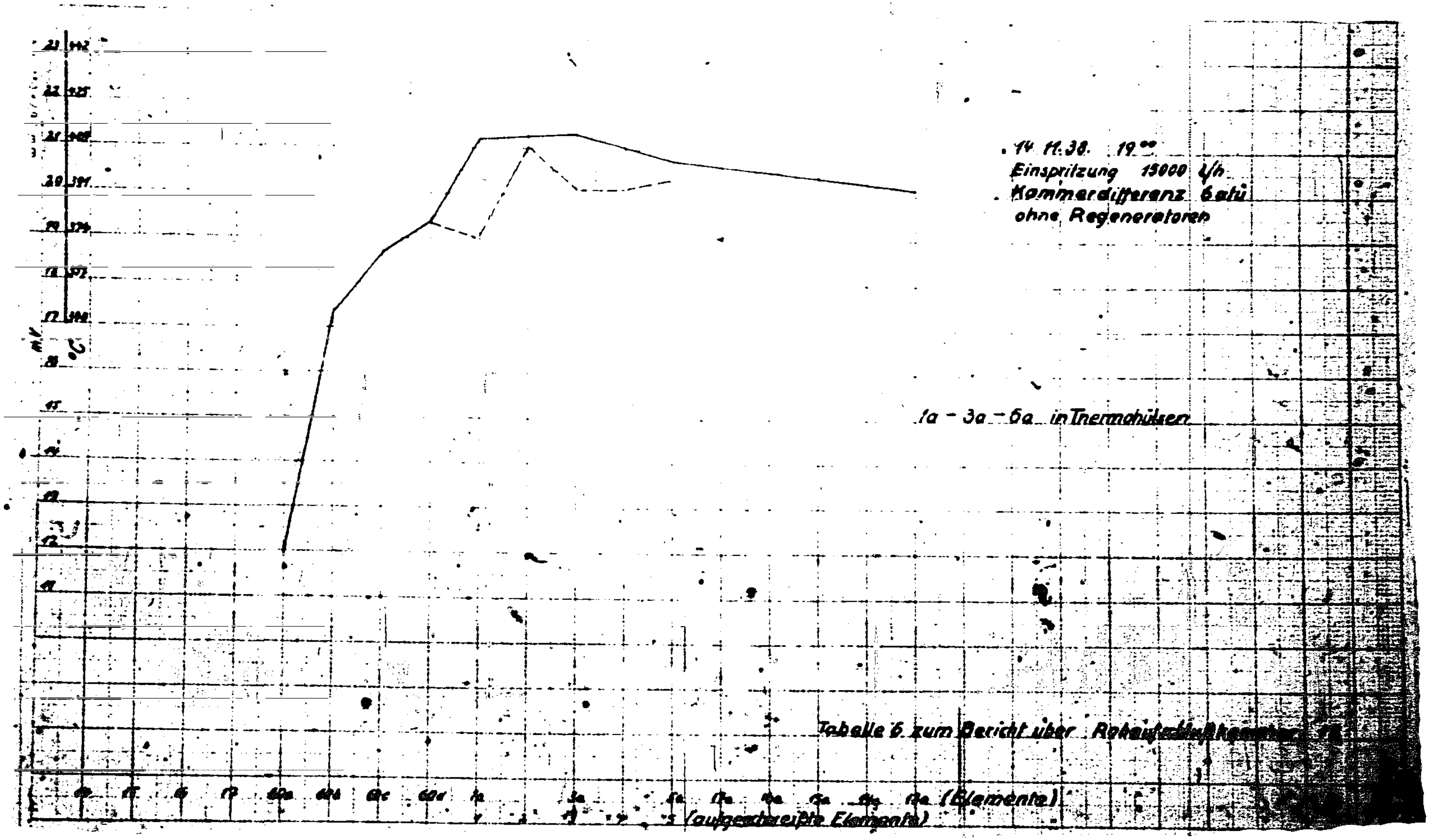


Tabelle 6 zum Bericht über

Production Analyses

Investigation of Extraction-Middle Oil: Average of samples from March to August 1943.

	P 61	P 62	P 64
Number of samples	16	20	21
Specific gravity	1.006	1.057	1.012
Initial distillation	210°	230°	208°
Vol. % distilled over at 300°	39.3	41.3	89.5
Distillation end point	98% 318°	88.7% 360°	98.5% 319°
Residue	1.3% Wt.	11.4% Wt.	1% Vol.
Melting point	-11.1	-5.0	-10.3
Pour point	+ 1	+4	- 0.7
Phenol	3.6	1.6	2.9
Pyridine	4.2	4.5	4.1
Carbon	90.13	90.15	89.9
Hydrogen	7.88	7.22	7.96
Sulfur	0.102	0.232	0.160
Chlorine	0.0014	0.023	0.0028

Investigation of Crude Conversion Product from Bldg. 12: (P 55) Average of Samples from August and September 1943

Contact	Na	Mo	Ni
Number of samples	18	19	19
Filtration time	18"	16"	15"
Residual coal	8.3	8.2	8.3
Ash in residual coal	29.89	30.79	32.11
Ash in starting coal	7.37	6.57	6.76
Extract in pure filtrate	28.7	28.6	27.9
Extract in crude conversion product	26.3	26.2	26.0
Degree of conversion:			
Calculated on ash:			
Ash-containing basis	77.4	78.6	78.5
Ash-free basis	83.4	84.4	84.2
Calculated on extract:			
Ash-containing basis	76.7	76.6	75.9
Ash-free basis	81.6	82.1	81.4

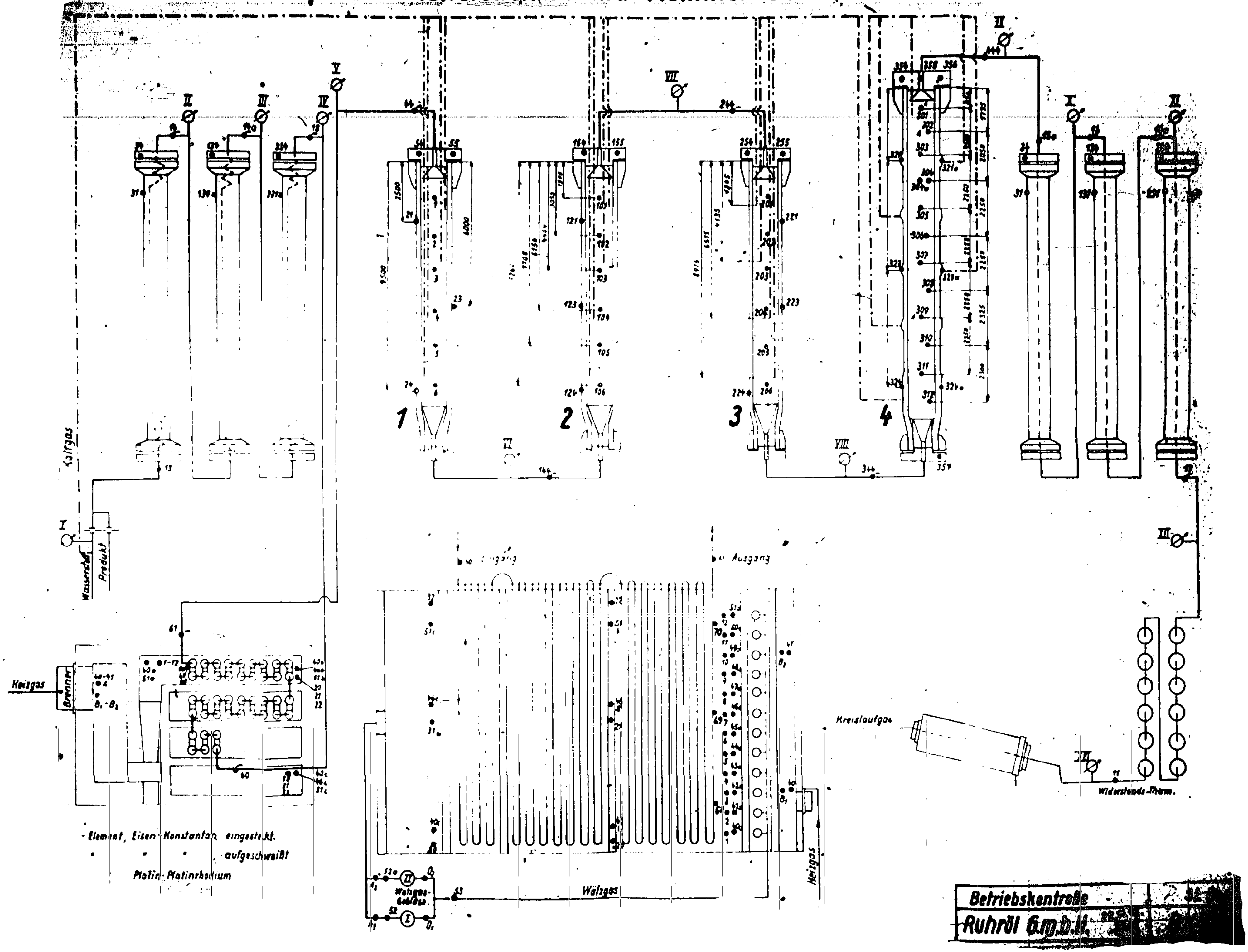
Investigation of Pure Filtrate from Bldg. 15 (P 56): Avg. of samples from April to July 1943

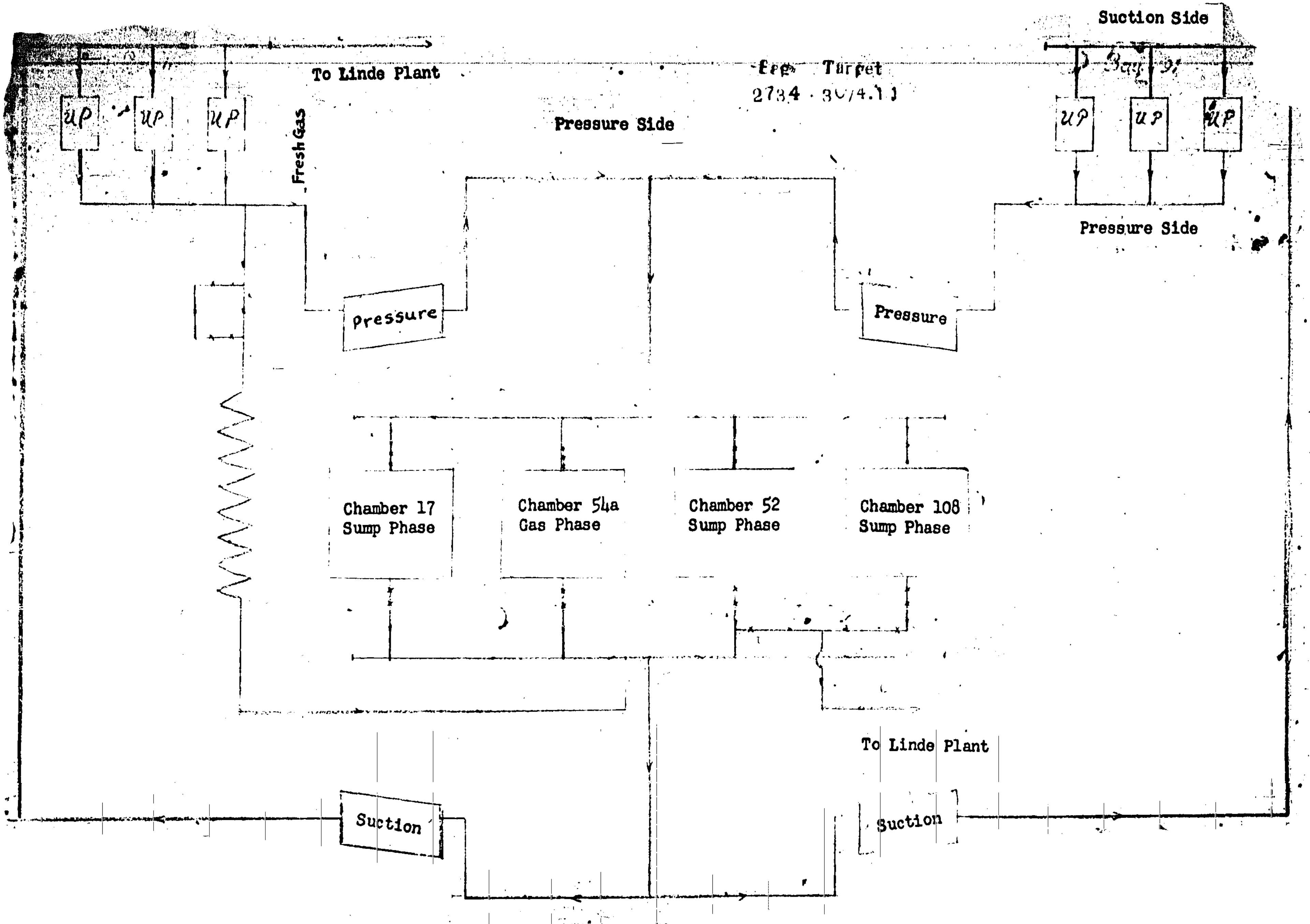
	Specific Gravity	Extract	In Extract M. P.	In Extract Ash
48 samples	1.089	24.7	225	0.06

Investigation of Residual Coal: Average of samples from Jan. to June 1943

	No. of Samples	Residual Coal	Ash of Res. Coal	Extract	Solvent
P 57	28	66.8	27.93	6.6	26.6
P 58	28	96.7	29.51	2.6	0.8

Temperatur-Meßstellenschema Kammer 54a.

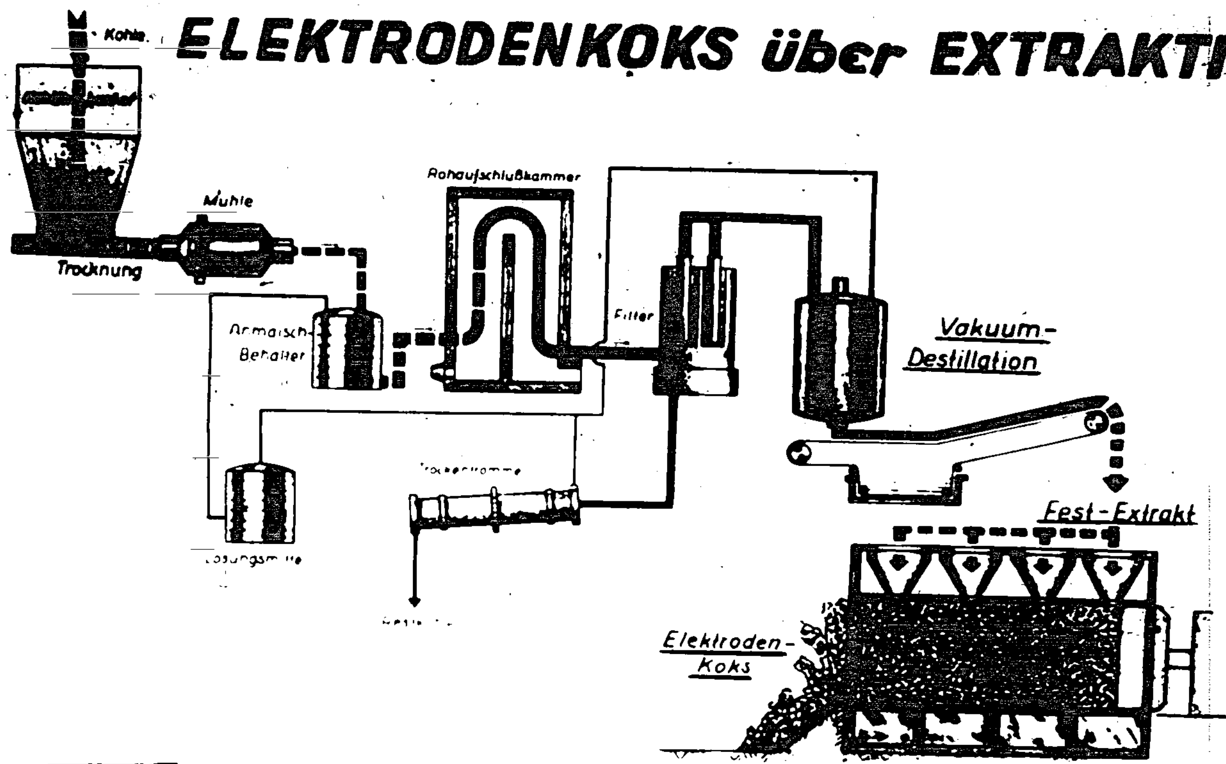




Flow Diagram for Hydrogenation at Weilheim Carried out at 700 Atmospheres 11-30-41

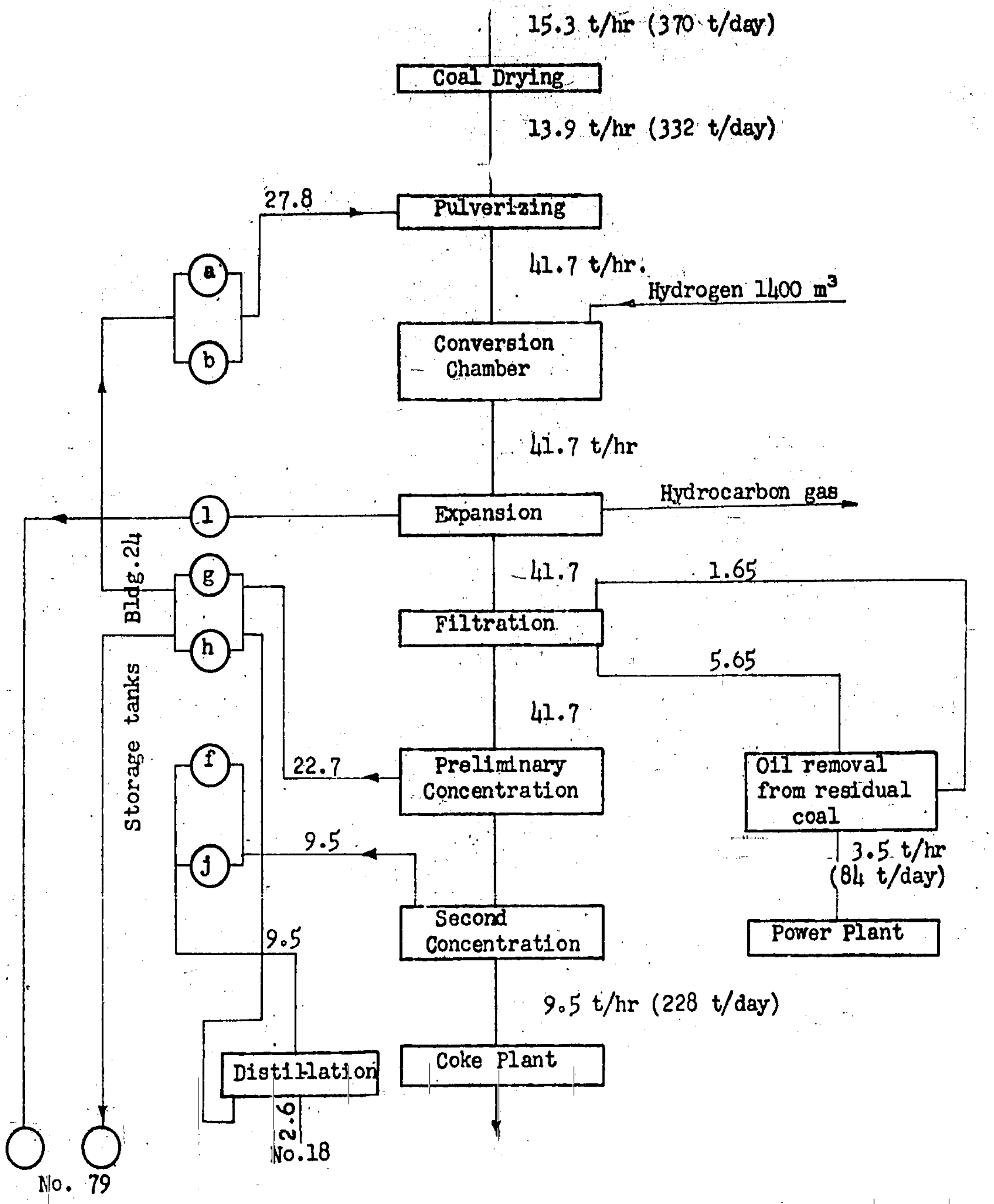
Bag 2734 Target No. 30/4.11
Bottrop - Item 7

ELEKTRODENKOKS über EXTRAKTION



RUHROL G.M.B.H.
HUGO STINNES WERKE BOTTROP
M.T.A.: Film u. Foto
Bild-Nr. 669 Bau-Nr. 0

Flow Diagram for Treatment of Extract (Pott-Broche Process)

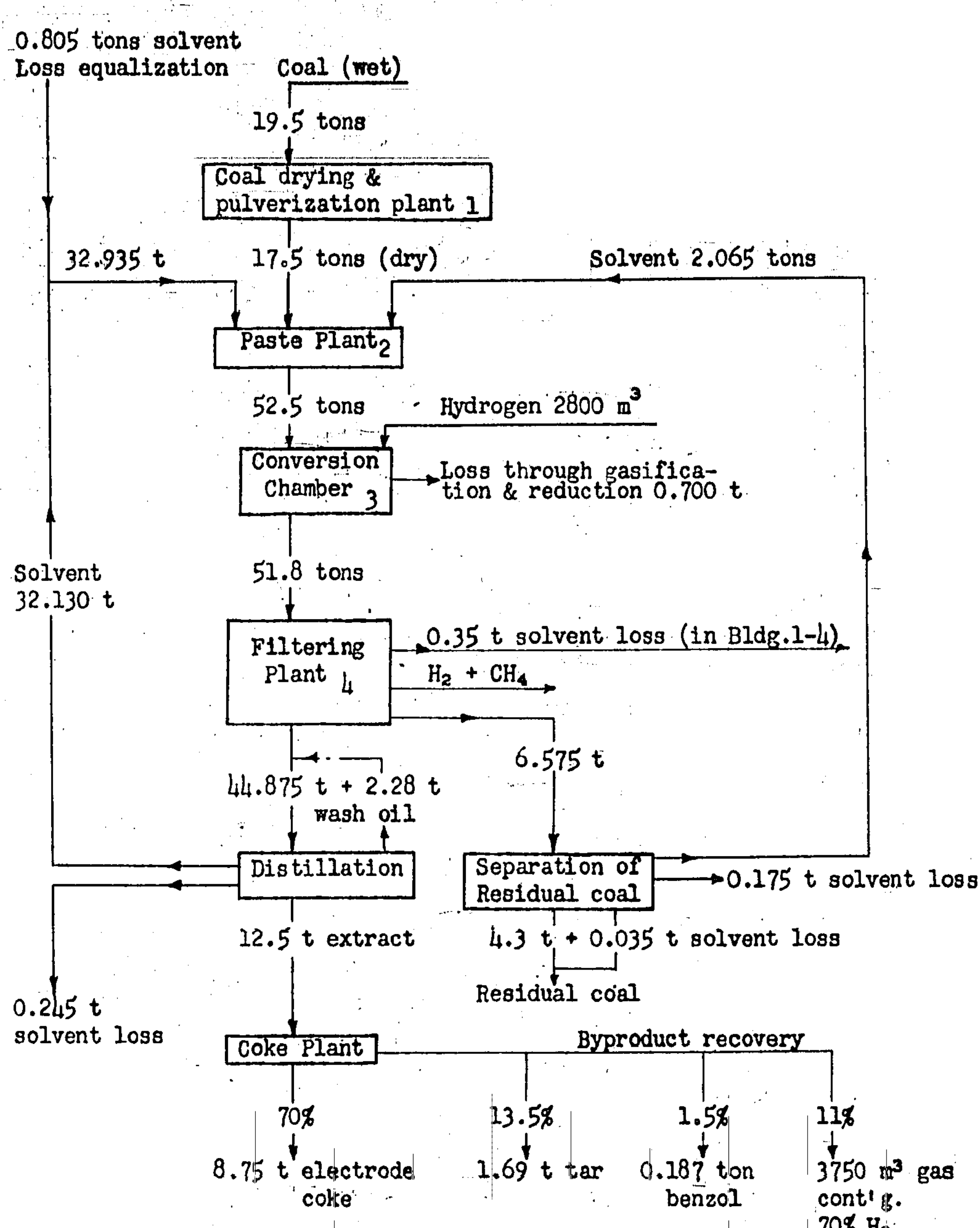


300 Operating days = 7200 hours
Oil balance per day: 27.8 x 24 = 665 tons
20% thereof is fresh: 133 tons
= 40,000 tons per year Middle Oil.

Bag. 2734 Target No. 30/4.11 Bottrop, Item 9 (Diag. 34)

June 26, 1942.

Flow Diagram of Pott-Broche Process for Manufacture of 100,000 tons per year Extract - 70,000 tons per year electrode coke (12.5 tons per hour extract - 8.75 tons per hour electrode coke).



Ruhröl G.m.b.H.
Bottrop, Aug. 4, 1941

Bag 2734 Target No. 30/4.11
Bottrop, Item 9.

Summary of Operating Period of Chamber 12 from Feb. 9, 1940 to Jan. 18, 1943 (Pott-Broche Process)

Date	Hours	Charge 1/hr.	Max. Temp. mv.	Solvent	Filter Time in Seconds	% Conversion calc. on R.K.(Pure coal ?)
2/9-13/40	4	18,000	21.7	A Product	25	83
3/11-19/40	49	18,000	21.7-21.9	A Product	18-24	83
3/25-4/1/40	38	18,000	21.7-21.9	20%-35% A Product	20-25	81
4/2-6/40	35	15,000	21.7-21.9	No A product; 80% of the solvent flowed through the chamber 4 times, 300 m^3/h H_2	20	81
4/1/41- 3/11/41	39	15,000	21.8	A Product	22	79
11/9-27/41	81	15,000	21.8	A Product	19	82
12/8/41 -	48	15,000	21.8	6 days in cycle	10	74
1/2/42		15,000	21.8	A Product	18	83
	93	15,000	21.8	20% A Product	12	75
	68	15,000	21.8	25% A Product	13	75
	62	15,000	21.8	30% A Product	13	75
1/12-17/42	-	15,000	21.8	A Product/ 600 m^3/ h H_2	-	-
1/19-4/2/42	112	15,000	21.8-22	A Product	16	83-85
2/5-26/42	158	15,000	21.8	30% A Product	14	78
2/27-3/14	170	15,000	21.8	45% A Product	15	78
3/24-4/42	113	15,000	21.9-22	30%-45% A Product/300 m^3/h H_2	18	80
	56	15,000	22.4	60% A Product / 300 m^3/h H_2	16	80
4/17-5/15	61	15,000	22.4	Pure product (A)	20	81
	74	15,000	22.3	20% A Product	16	73
	79	15,000	22	30% A Product	20	77
	133	15,000	22.3	25% A Product	14	78
	74	15,000	22.3	35% A Product	14	77
5/16-7/8/42	823	15,000	22.6	35%-40% A Product	18	77
8/21-25	55	8,000	22.9	A Product/250 m^3/h H_2	16	81
9/8-21/42	185	16,000	22.8	45%-55% A Product without H_2 & with 200-300 m^3/h H_2	15-25	81
10/7-16/42	145	17,000	22.4	40% A Product	18	82
10/21-8/11	226	16-17,000	22.4-22.5	50% A Product	20	80
11/21-12/11	255	17,000	22.5-22.7	40% A Product	15	81
12/13-1/18/43	567	17,000	22.8-23	40%-50% A Product	10	76

Bag 2734 Target No. 30/4.11 - Bottrop, Item 9