Method of Pressure Measurement

As has been already mentioned, the covers of the experimental pipes each contained a ball pressure apparatus. This consisted essentially of a steel die of 10 mm diameter in the surface of which a steel ball of 4 mm diameter is inserted. This rests on a copper cylinder 10.5 mm high and 7 mm diameter. During the explosion the ball of the die is pressed into the copper cylinder and causes an indentation of a certain depth and diameter. This device can be calibrated by means of a press and the pressure corresponding to certain sizes of indentation can be determined.

Besides the pressure measuring devices in the pipe covers, there were nine more along the pipe. These were located at distances of 0.2, 0.5, 1.0, 2.0, 5.0, 10.0, 15.0, 20.0, and 25.0 m from the front end of the pipe, i.e., from the ignition point. These devices were somewhat smaller than those in the covers. The die had a diameter of 7.9 mm; the copper cylinder a diameter of 6 mm and a height of 9 mm. These devices are generally called "Krupp'sche Messeier". In some of the experiments, especially those in which high pressures resulted, these devices were used with a compression cylinder rather than with the ball device. In the compression devices the bottom surface of the steel die is machined smooth and rests on a copper cylinder of somewhat smaller dimensions, 3x6 mm. The explosion causes compression of the copper cylinder which can be measured with a micrometer. Calibration is also done statically by means of a hand press.

In the publication "Autogene Metallbearbeitung", 1943, Vol. 13, page 213, it is stated that the ball apparatus gives usable results only in the case of true explosions and not with detonations. This is because the accuracy is dependent upon the velocity of the pressure rise and hence the speed of propagation of the process. (Decomposition or combustion) Detonations, which have a velocity of 1,000 to 3,000 m per second, cause too high values with both devices. The result can actually be twice as high as the true values. However, as to the actual deviation there is insufficient actual experience.

In order to utilise the pressure measuring devices not only as indicaters for a detonation but also in order to make their results somewhat usable, data from them were compared with those obtained by means of an electric device.

This consists of a quartz transmitter, in which a piezo-quartz causes a change in electrical charge with variations in pressure; furthermore an amplifier in which these changes in charge can be made into changes of current in amplified form; as well as a cathode ray oscillograph, in which the changes in current deflect a stream of electrons. This deflection is shown by means of a moving film. The calibration is

The experiments with the powder igniter (Experiments 9, 10, 11, and 12) gave values, which do not fit well into the general scheme, because, as already mentioned, this type igniter has too short a burning time and causes irregular ignition.

Most remarkably, a week later as the experiments were again continued, the decomposition stopped after 3 m in the pressure range of 2-4 kg/cm$^2$. Detonation was again observed at a pressure of 5 kg/cm$^2$, which started at a distance of 2 m from the ignition point. The phenomenon was due apparently to the 4° drop in temperature occurring in the meantime. This could be demonstrated by use of a short spark plug located next to the cover. Apparently the cooling effect of the pipe wall and cover plays an appreciable roll in the development of an explosion in narrow pipes of this sort.

The experiments were therefore continued with a spark plug which had electrodes 15 cm long for all pipes including this 25 mm pipe. It was shown that at a temperature of 6°C, (Table 3) a decomposition with detonation always occurred above 2.75 kg/cm$^2$. (Experiments 146, 147).

After the ball pressure devices were taken out (installed on a bar) decomposition started at 0.6 kg/cm$^2$. At this pipe cross section, this obstruction had a strongly adverse effect. Detonation began at 2 kg/cm$^2$, however.

For all the experiments in the 25 mm and 50 mm pipe the ignition took place in the pipe. In the following experiments a chamber of 200 mm I.D. and 2 m long was placed in front of the pipe and ignition took place at the end of the chamber, so that a strong explosion wave could be developed before entry into the pipe. With this arrangement, detonations occurred in the 25 mm pipe at 0.5 and in the 50 mm pipe at 0.4 kg/cm$^2$. (See Table 4) This effect was not apparent in the large pipes. A chamber of sufficient size thus has such a strong effect on the experimental conditions that detonations start in small pipes at the same pressure as in large ones.

Illustration XI shows the ignition chamber with the 25 mm pipe attached.

Experiments in the 100 mm Pipe (Table 5)

Between 0.4 and 0.6 kg/cm$^2$ lays a region, in which decomposition traverses the whole pipe without detonation occurring. The ball pressure devices showed very little indentation in this range. From 0.6 kg/cm$^2$ and above detonation occurs with final pressures between 100 and 200 kg/cm$^2$. With this series of experiments some pressure data were obtained with the help of the oscillograph, which are shown in the last vertical column.

As the pressures obtained show, between 0.6 and 0.9 kg/cm$^2$

pressures.

Then the pipe was evacuated to a pressure of some 10 mm; acetylene gas put in; again evacuated and acetylene added to the desired pressure. The acetylene averaged 99% purity. The pressure at which ignition took place lay with pure acetylene between 0.3 and 5 kg./cm$^2$ gauge pressure. Then the ignition wires were attached, the transformer hooked up, the area cleared and then ignition was brought about from a distance of 40 m.

If ignition occurred, a noise could be heard, especially with the larger pipes and at higher pressures, which became stronger the higher the initial pressure. Further characteristics of an actual decomposition were warming of the pipe, separation of soot, an odor of aromatic hydrocarbons and a pressure rise. In the case of complete decomposition the soot lay in a thick layer up to the front flange. If decomposition was not complete, the soot stopped at a certain distance from the ignition point although exactly at what point was difficult to determine, because the soot was carried on further by the explosion wave in the direction of propagation and in some cases was present in large quantities only at the end of the pipe.

After firing, a gas sample was taken and analyzed. The data are not given, because the rust layer on the inner surface of the pipe gave off lighter compounds of hydrocarbons and $N_2$, through the heat of the explosion, which caused analytical values to be false. Even after several experiments the splitting off of these gases was still appreciable.

The chief product of decomposition was hydrogen. Heavy hydrocarbons were usually present only to a few percent. Light hydrocarbons were found up to 30% by volume, which led to the observation that the quantity of light hydrocarbons is presumably greater in the case of a detonation than for a slow decomposition.

After the gas samples were taken the pipes were opened and the soot blown out through the rear flange after the forward cover was removed and the blower piping hooked up. In this way it was possible to clean out the soot in a few minutes.

Illustration X shows the soot being blown out of the 200 mm pipe.

Finally the ball pressure devices were taken out, pressures measured and the pipe made ready for a new experiment.

## Discussion of the Experimental Results

The results of the experiments are shown in the ten tables, in which they were arranged according to initial pressures.

likewise static. Since the piezo-electric measuring device is practically without inertia, it gives better accuracy than any other methods for the measurement of fast moving pressure waves.

In all experiments in which this method of measurement was used, the transmitter was installed on the front cover next to the ball pressure apparatus, while ignition occurred at the other end, so that both measuring devices were the same distance from the point of ignition. Also for some of the measurements two or three additional ball devices were installed in order to investigate the scattering of the values.

Without going into detail at this time, it might be mentioned that the ball pressure and compression cylinder devices showed a scattering of values of up to 120%. All the results suffer from this trouble. Furthermore, the results of the ball pressure device differ from those of the electrical system by a factor of 1.75 (as mean between 1.4 and 2.1), i.e. the ball pressure values must be divided by this to get the true value. Of course, this is purely an experience factor, which is good only for these experimental conditions, i.e. for ball pressure devices of this size. (The weight of the die is the primary factor). A further condition is that the device must be so built that the axis of the die lies in the direction of flame propagation, and finally the decomposition must be a detonation. In the case of ordinary decomposition, neither the weight of the die nor the position of the apparatus relative to the flame front makes any difference. Then the pressures are subject only to the usual mistakes and do not have to be divided by a certain factor.

The transition of a slow decomposition to a detonation is characterized by a very sudden increase in pressure. In the range of the transition the results of the ball pressure device are also uncertain. However, in the experiments the deviations from the true values were not determined.

## Experimental Procedure

The experiments were carried out in the following way:

The new ball pressure devices were installed in the pipe: the cover was put on, and the pressure measuring apparatus, or as the case may be, the quartz transmitter was screwed into place. The spark plug was installed in the rear cover. The spark plug was built like a normal Bosch plug except that the electrodes were increased to 15 cm. On the ends of these, three platinum wires 0.15 mm diameter were arranged, which served as the means of ignition and which were melted, depending upon the initial acetylene pressure, by a current of 15-25 Amp. As a rule, direct current was used. In some experiments an igniter of 0.2 g. of lead picrate was used instead of the platinum wires. However, this igniter had too short a burning time to cause ignition especially at low acetylene initial pressure, the start of detonation is at the end of the pipe, i.e. after a starting distance of 30 m. The start of detonation is always characterized by a very high pressure, which is higher than the pressure within the detonation wave. In such cases, viz., the start of detonation at the pipe end, destruction frequently occurs, such as breaking of the wire to the quartz transmitter, shearing of bolts, bending of the cover, tearing loose of the flanges, etc. Experiments 55 and 95 with a pressure of 350-600 kg/cm$^2$ at the end of the pipe are examples of this.

Experiments in the 200 mm Pipe (Table 6)

At 0.4 and with certainty at 0.5 kg/cm$^2$ a slow decomposition changes to a detonation. It is interesting and clearly to be seen in this series of experiments how the start of detonation with increasing pressure moves in the direction of the point of ignition. At 0.4 kg/cm$^2$ detonation starts at 25 m from the ignition point; between 0.6-1.0 kg/cm$^2$ it starts at 15 m; and between 1.33-2.0 kg/cm$^2$ at 10 m.

With the experiments in the 200 mm pipe, the duration of ignition and the explosion time were more frequently measured by use of the quartz transmitter. The ignition time when the powder charge was used was 0.004 sec. and with the platinum wires usually a few tenths of a second, sometimes considerable more. From this it follows that the latter type of ignition is effective a longer time, but so far as total strength is concerned shows some irregularities, as indicated by the fact that near the pressure limit the experiments did not give exactly the same results, i.e. the pressure limit varied some 0.1 kg/cm$^2$.

The explosion time, i.e. the time between closing the circuit and the arrival of the wave at the opposite cover was between 0.6 and 1.6 seconds for the wire fusion and became longer with falling initial pressure. With the use of the powder charge it was appreciably longer, viz., 1.56-1.84 sec., although the initial pressure was higher (1.66 and 2.0 kg/cm$^2$). The explosion time is merely a point of orientation, which primarily depends on the starting distance required before detonation starts.

Experiments in the 300 mm and 400 mm Pipe (Table 7 and 8)

Here it is very apparent that detonation occurred as soon as decomposition begins. This happened at a pressure of 0.5 kg/cm$^2$ gauge. Below this pressure only soot deposition on the spark plug took place without the deposition being propagated further.

The start of detonation lay always at or very near the pipe end and was characterized by very high pressures (350 kg/cm$^2$). Therefore in these experiments the cover, and mostly also the flanges were torn off. The breaking point of the flange was always right next to the weld. At the same time the end of the pipe was torn apart into a tulip shape, while a cloud of soot rose and a flame several meters long and lasting several seconds rose from the trench into which the end of the pipe extended. The thunderous noise could be heard at some distance.

The ties, which covered the trench, were usually blown off. However, the broken parts of the pipe were usually found in the trench.

The attached illustrations show the destruction caused by the detonations.

Illustration XII shows the cover torn off, to which parts of the flange are still attached.

Illustration XIII and XIV show a cover with the flange torn off near the weld.

In Illustrations XV and XVI are shown the ends of two pipes.

Illustrations XVII and XVIII show the condition of the trench immediately after breaking off the flanges of the 300 and 400 mm pipes.

Experiments with Acetylene-Nitrogen Mixtures in the 100 and 200 mm Pipe (Table 9 and 10)

These experiments were completed by experiments with acetylene-nitrogen mixtures to determine to what extent higher $N_2$ contents would raise the explosive limit or hinder detonation. However, only a few experiments were carried out. The 100 and 200 mm pipes were used.

The acetylene-nitrogen mixtures were produced in the following way:

Acetylene and nitrogen were each taken from a battery of cylinders in the ratio required, passed through two meters, through a mixer and to a 5 $M^3$ gas holder. From the gas holder a pipe went to a compresser which served to compress this mixture into the pipes which were first evacuated.

Illustration XIX shows the two meters on the right, the mixer in the center on the cylindrical container and the gas holder on the left. The house on the right, in front of which stand the cylinders, contains the compressers.

Before the experiments were started, the gas mixture was analyzed each time both at the gas holder and after compression into the pipe. The production of the mixture and the pumping of the same into pipes

## Zahlentafel 1

**Versuche im 25 mm Rohr mit reinem Azetylen.**

Messei 40m vom Rohrende entfernt, senkrecht zur Rohrachse.

| Vers Nr | Datum | Anfgs druck kg/cm² | Art u Stärke der Zündung | Zünddauer sec | Verbr. dauer sec | Temp. d Gas °C | Reinh. d Gas % | Ind 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | ~~25,0~~ | 30,0 | Ind 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle | | | | | | | | | | | | | |
| 136 | 18.11. | 2,00 | 3/4 Drähte 20 Amp = | — | — | 5,0 | 99,2 | — | — | — | — | — | — | — | — | — | — | — | — | — | Zündkerze verrusst. Russ bis 4m von Zündstelle. Kein fortschr. Zerfall. |
| 137 | " | 2,50 | " " | — | — | " | " | — | — | — | — | — | — | — | — | — | — | — | — | — | " " " |
| 148 | " | 2,75 | " " | — | — | " | " | — | — | — | — | — | — | — | — | — | — | 150 | 195 | — | Fortschr. Zerfall. |
| 135 | " | 3,00 | " " | — | — | " | " | — | — | — | — | — | — | — | — | — | — | 1280 | 197 | — | " |

**Versuche mit reinem Azetylen und ZS-Schleifen.**

| Vers Nr | Datum | Anfgs druck kg/cm² | Art u Stärke der Zündung | Zünddauer sec | Verbr. dauer sec | Temp. d Gas °C | Reinh. d Gas % | Ind 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | ~~25,0~~ | 30,0 | Ind 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 160 | 20.11. | 2,75 | 3/4 Drähte 20 Amp = | — | — | 5,0 | 99,0 | — | — | — | — | — | — | — | — | — | — | — | × | — | Russabscheidung bis 6-8 m. Kein fortschr. Zerfall. |
| 170 | " | 3,00 | " " | — | — | " | " | — | — | — | — | — | — | — | — | — | — | — | 140 | — | Fortschr. Zerfall, Knall hörbar. |

58

Zahlentafel 1

Versuche im 25 mm Rohr mit reinem Azetylen.

Messel [illegible] m vom Rohrende entfernt, senkrecht zur Rohrachse.

| Vers. Nr | Datum | Anfgs. druck kg/cm² | Art u. Stärke der Zündung | Zünddauer sec | Verbr. dauer sec | Temp. d. Gas °C | Reinh. d. Gas % | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle: Ind. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | ~~25,0~~ | 30,0 | Ind. 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| [illegible] | [illegible] | 2,00 | [illegible] | — | — | 5,0 | 99,2 | — | — | — | — | — | — | — | — | — | — | — | — | — | Zündkerze verrusst. Russ bis [illegible] von Zündstelle. Kein fortschr. Zerfall |
| [illegible] | ″ | 2,50 | ″ ″ | — | — | ″ | ″ | — | — | — | — | — | — | — | — | — | — | — | — | — | ″ ″ ″ |
| [illegible] | ″ | 2,75 | ″ ″ | — | — | ″ | ″ | — | — | — | — | — | — | — | — | — | — | 150 | 185 | — | Fortschr. Zerfall |
| 135 | ″ | 3,00 | ″ ″ | — | — | ″ | ″ | — | — | — | — | — | — | — | — | — | — | [illegible] | 197 | — | ″ |
| | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | Versuche mit reinem Azetylen und ZS-Schleifen. | | | | | | | | | | | | | | |
| [illegible] | [illegible] | 2,75 | [illegible] | — | — | 5,0 | 99,0 | — | — | — | — | — | — | — | — | — | — | — | × | — | Russabscheidung bis 6-8 m kein fortschr. Zerfall |
| 170 | ″ | 3,00 | ″ ″ | — | — | ″ | ″ | — | — | — | — | — | — | — | — | — | — | — | 140 | — | Fortschr. Zerfall, Knall hörbar. |

took place at the same time.

The results are to be seen in Tables 9 and 10.

According to Table 9 no decomposition took place in the 100 mm pipe with a mixture of 70% acetylene and 30% nitrogen at a pressure of 1 kg/cm$^2$, and at 2 kg/cm$^2$ a complete decomposition took place but without detonation. With mixtures in the ratio 1:1 the corresponding values are 3.0 and 4.0 kg/cm$^2$.

In the 200 mm pipe (Table 10) the same was found for mixtures in the ratio 1:1, namely that at 4.0 kg/cm$^2$ complete decomposition takes place (Experiment 150); at 8.0 kg/cm$^2$ the decomposition seems to change to a detonation. (Experiment 152). Experiments, however, in the range of 4.0-8.0 kg/cm$^2$ for the exact determination of the detonation limit are lacking.

An experiment with only 25% $N_2$ gave detonation at 2.0 kg/cm$^2$.

Zahlentafel 2

**Versuche im 50 mm Rohr.**
**mit reinem Azetylen**

**Kurze Zündkerze von unten am Ende in Rohr eingeführt.**

~~Blatt I~~

| Vers. Nr | Datum | Anfgs druck kg/cm² | Art u. Stärke der Zündung | Zünd dauer sec. | Verbr. dauer sec. | Temp. d. Gas. °C | Reinh. d. Gas. % | Jnd. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Jnd. 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle | | | | | | | | | | | | | |
| 1 | | 0,4 | 3 Pt-Drähte. 15 Amp~ | - | - | 11 | 98,7 | – | – | – | – | – | – | – | – | – | – | – | – | – | Zündkerze verrusst. Kein fortschr. Zerfall. |
| 2 | | 0,6 | ″ ″ | - | - | ″ | ″ | – | x | x | x | – | – | – | – | – | – | – | – | – | Kerze verrusst. Russ bis 1m von Zündstelle. Kein fortschr. Zerfall. |
| 3 | | 0,8 | ″ ″ | - | - | ″ | 99,3 | – | x | x | x | x | – | – | – | – | – | – | – | – | Kerze verrusst. Russ bis 15m von Zündstelle. Kein fortschr. Zerfall. |
| 4 | | 1,0 | ″ ″ | - | - | ″ | ″ | – | x | x | x | x | x | – | – | – | – | – | – | – | ″ ″ ″ |
| 5 | | 1,25 | ″ ″ | - | - | ″ | ″ | – | x | x | x | x | x | x | x | x | x | – | – | – | ″ ″ ″ |
| 6 | | 1,50 | ″ ″ | - | - | 10 | ″ | – | x | x | x | x | x | x | x | x | x | x | x | – | ″ Russ bis 14m von Zündstelle. Kein restl. Zerfall |
| 7 | | 1,75 | ″ 18 Amp~ | - | - | ″ | ″ | – | x | x | x | x | x | x | x | x | x | x | x | – | ″ Russ bis 15 m von Zündstelle. Kein restl. Zerfall. |
| 10 | | 1,75 | Zündpille | - | - | ″ | ″ | Jnd | x | x | x | x | x | x | x | x | x | x | x | – | ″ Russ nur vorn u. hinten in grösserer Menge. Kein restl. Zerfall |
| 8 | | 2,0 | 3 Pt-Drähte. 18 Amp~ | - | - | ″ | ″ | – | 22 | 27 | 19 | 27 | 22 | 200 | 130 | 98 | 140 | 93 | 106 | – | Fortschr. Zerfall. |
| 13 | 17.10 | 2,0 | ″ Akku = | 0,86 | - | ″ | ″ | Jnd | | ohne Schiene, ohne Messeier. | | | | | | | | | 9 | – | ″ ″ |
| 9 | | 2,0 | Zündpille! | - | - | ″ | ″ | 23,5 Jnd | – | 42 | 30 | 61 | 38 | 155 | 77 | 126 | 100 | 129 | 100 | – | ″ ″ |
| 11 | | 2,20 | ″ | - | - | ″ | ″ | Jnd | – | x | x | x | x | x | x | x | x | x | x | – | Kerze verrusst. Russ im ganzen Rohr, jedoch kein restl. Zerfall. |
| 12 | | 2,25 | ″ | - | - | ″ | ″ | Jnd | – | x | x | x | x | x | x | x | x | x | x | – | Kein restloser Zerfall. |
| 128 | | | | | | | | | | | | | | | | | | | | | |
| 129 | 14.11. | 2,0 | 3 Pt-Drähte 15 Amp = | - | - | 8 | ″ | – | – | Nur geringe Einschläge a.d. Messeiern | | | | | | | | | | – | Kerze verrusst. Russ bis 3m von Zündstelle. Kein fortschr. Zerfall. |
| 130 | 15.11. | 2,0 | ″ 16 Amp ~ | - | - | 7 | ″ | – | – | | | | | | | | | | | – | ″ ″ ″ |
| 125 | 13.11. | | | | | | | | | | | | | | | | | | | | |
| 126 | 14.11. | 3,0 | ″ 15 Amp = | - | - | 8 | ″ | – | – | | | | | | | | | | | – | ″ ″ ″ |
| 133 | 15.11. | 3,0 | ″ ″ | - | - | 7 | ″ | – | – | ohne Schiene, ohne Messeier. | | | | | | | | | | – | ″ ″ ″ |
| 127 | 14.11. | 4,0 | ″ ″ | - | - | 8 | ″ | – | – | nur geringe Einschläge a.d. Messeiern | | | | | | | | | | – | ″ ″ ″ |
| 134 | 15.11. | 4,0 | ″ 20 Amp = | - | - | 7 | ″ | – | – | ohne Schiene, ohne Messeier. | | | | | | | | | | – | ″ ″ ″ |
| 131 | ″ | 5,0 | ″ 18 Amp = | - | - | ″ | ″ | – | – | keine Einschläge a.d. Messeiern | | | | | | | | | | – | Zündkerze nicht durchgeschmolzen |
| 132 | 15.11. | 5,0 | ″ 25 Amp = | | | | | | | 40 | 35 | 39 | 139 | 200 | 130 | 185 | 175 | 172 | 200 | – | Fortschr. Zerfall. |

726,7

59

72 C

## Zahlentafel 3

**Versuche im 50 mm Rohr.**
**mit reinem Azetylen**

**Lange Zündkerze.** ~~Blatt II~~

| Vers Nr | Datum | Anfgs druck kg/cm² | Art u Stärke der Zündung | Zünd dauer sec. | Verbr. dauer sec. | Temp. d. Gas °C | Reinh. d. Gas % | Ind. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Ind 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 139 - 141 | 18.11. | 1,00 / 1,25 / 1,50 | 3 Pt Drähte 15 Amp. = | – | – | 6 | 99,3 | – | – | Nur geringe Einschläge a.d. Messeiern | | | | | | | | | | | Kerze verrusst. Russ bis 2-4 m von Zündstelle. Kein fortschr. Zerfall. |
| 142 - 144 | 19.11. | 1,75 / 2,00 / 2,25 | " " | – | – | " | " | – | – | | | | | | | | | | | | " " " |
| 145 / 146 | " | 2,50 / 2,75 | " " / " " | – | – | " | " | – | – | | | | | | | | | | | | " " " |
| 147 | " | 3,00 | " 10 Amp. = | – | – | " | " | – | – | 35 / " | 42 / " | 39 / " | 152 / " | 119 / " | 107 / " | 143 / " | 141 / " | 160 / " | 152 / " | - / - | Fortschr. Zerfall |
| | | | | | | | | | | **Ohne Schiene, ohne Messeier** | | | | | | | | | | | |
| 162 | 25.11. | 2,0 | 3 Pt Drähte 15 Amp. = | – | – | 5 | 99,0 | – | – | – | – | – | – | – | – | – | – | – | 1400 | – | Fortschr. Zerfall bis zum Rohrende. Schlag hörbar. |
| 163 | " | 1,0 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | x | – | Fortschr. langsamer Zerfall durch d. ganze Rohr, fahnig flockiger Russ. Kein Schlag hörbar. |
| 164 | " | 0,8 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | x | – | " " " " |
| 165 | " | 0,6 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | x | – | " " " " |
| 166 | " | 0,3 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | – | – | Kein Zerfall. |
| 167 | " | 0,4 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | – | – | " |
| 168 | " | 0,5 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | – | – | " |

Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle

## Zahlentafel 4

**Vorkammer-Versuche.**

**Versuche im 50 mm Rohr**
**mit reinem Azetylen u. vorgelegter Zündkammer (200 mm Ø, 2 m lang).**

| Vers. Nr | Datum | Anfgs druck kg/cm² | Art u. Stärke der Zündung | Zünddauer sec. | Verbr. dauer sec. | Temp. d. Gas °C | Reinh. d. Gas % | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle: Ind. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Ind. 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 156 | 22.11. | 0,3 | 3 Pt-Drähte 15 Amp = | – | – | 8 | 99,0 | – | – | – | – | – | – | – | – | – | – | – | – | – | Kein Zerfall, geringe Russpuren an der Kerze. |
| 155 | " | 0,4 | " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | >300 | – | Fortschr. Zerfall. |
| 154 | " | 1,0 | " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | 122 | – | " |

**Versuche im 25 mm Rohr**
**mit reinem Azetylen u. vorgelegter Zündkammer.**

| Vers. Nr | Datum | Anfgs druck kg/cm² | Art u. Stärke der Zündung | Zünddauer sec. | Verbr. dauer sec. | Temp. d. Gas °C | Reinh. d. Gas % | Ind. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Ind. 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 157 | 23.11. | 0,3 | 3 Pt-Drähte 15 Amp = | – | – | 7 | 99,0 | – | – | – | – | – | – | – | – | – | – | – | – | – | Kohleüberbrückung a. d. Zündkerze. Kein Zerfall |
| 158<br>159 | " | " | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | – | – | Kein Zerfall. |
| 160 | " | 0,4 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | – | – | " |
| 161 | " | 0,5 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | → ↓ | >200<br>165 | – | Vollst. Zerfall bis zum Rohrende. |

## Zahlentafel 5

### Versuche im 100 mm Rohr mit reinem Azetylen.

| Vers. Nr | Datum | Anfgs druck kg/cm² | Art u. Stärke der Zündung | Zünd dauer sec. | Verbr. dauer sec. | Temp. d. Gas °C | Reinh. d. Gas % | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle: Ind. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Ind. 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 73-82 | 4.11. | 0,25 | 3 Pt-Drähte 15 Amp = | – | – | 7 | 98,2 | – | – | – | – | – | – | – | – | – | – | – | – | – | Kerze nicht verrusst. Kein fortschr. Zerfall. |
| 52 | 29.10. | 0,3 | ″ | – | – | 5 | 99,3 | – | – | – | – | – | – | – | – | – | – | – | – | – | ″ ″ |
| 67-71 | 1.11. | 0,3 | ″ | – | – | ″ | 99,2 | – | – | – | – | – | – | – | – | – | – | – | – | – | Nur ganz wenig Russ an Kerze. Kein fortschr. Zerfall. |
| 83-92 | 5.11. | 0,3 | ″ | – | – | 9 | ″ | – | – | – | – | – | – | – | – | – | – | – | – | – | ″ ″ |
| 53 | 29.10. | 0,4 | ″ | – | – | 5 | ″ | – | – | – | – | – | – | – | – | – | – | – | – | – | ″ ″ |
| 62-65 | 31.10. | 0,4 | ″ | – | – | 4,5 | 99,1 | – | – | – | – | – | – | – | – | – | – | – | – | – | ″ ″ |
| 66 | ″ | 0,4 | ″ | – | – | ″ | ″ | – | x | x | x | x | x | x | x | x | x | x | x | – | Kerze verrusst. Russ im ganzen Rohr. Langsam fortschr. Zerfall. |
| 54 | 29.10. | 0,5 | ″ | – | – | 5 | 99,3 | – | – | – | – | – | – | – | – | – | – | – | – | – | Nur ganz wenig Russ an Kerze. Kein fortschr. Zerfall. |
| 61 | 31.10. | 0,6 | ″ | – | – | ″ | 99,1 | – | x | x | x | x | x | x | x | x | x | x | x | – | Kerze verrusst. Russ im ganzen Rohr. Langsam fortschr. Zerfall. |
| 93 | 5.11. | 0,6 | ″ | – | – | 9 | 99,3 | – | x | x | x | x | x | x | x | x | x | x | x | – | ″ ″ ″ |
| 55 | 29.10. | 0,6 | ″ | – | 8-10 | 5 | 99,3 | – | x | 18 | 25 | x | x | 19 | x | 30 | 30 | 42 | 200 | – | Fortschr. Zerfall. Nach 8-10s lauter Knall. Deckel verbogen, Rohren gerissen. |
| 94 | 6.11. | 0,8 | ″ | – | – | 8,5 | 99,2 | – | x | x | x | x | x | x | x | x | x | x | x | Ind. | Kerze verrusst. Russ im ganzen Rohr. Langsam fortschr. Zerfall. |
| 106 | 8.11. | 0,9 | Zündpille | 0,002 | – | 8 | ″ | – | | | | ohne Messeier | | | | | | | – | Ind. | Kein fortschr. Zerfall. |
| 105 | 7.11. | 0,9 | ″ | ″ | 0,66 | ″ | ″ | – | | | | ″ | ″ | | | | | | 101 / 111 | Ind. | Fortschr. Zerfall. |
| 95 | 6.11. | 1,0 | 3 Pt-Drähte 15 Amp = | 0,48 | 1,16 | 8,5 | ″ | – | x | x | 21 | 30 | 31 | 27 | x | x | 34 | 42 | 350-400 | Ind. | Fortschr. Zerfall. Indikatorenkabel abgerissen. Messung kaum auswertbar. |
| 110 | 9.11. | 1,0 | ″ | – | 1,01 | 6,5 | 99,0 | – | x | x | 20 | 28 | 17 | 31 | 200 | 105 | 122 | 111 | 185 | Ind. | Fortschr. Zerfall. |
| 120 | 12.11. | 1,0 | ″ | – | – | 7,5 | ″ | – | x | x | 24 | 20 | 22 | 21 | 200 | 155 | 93 | 79 | 190 | 96 Ind. | ″ |
| 107 | 8.11. | 1,0 | ″ | 0,48 | 0,44 | 8 | ″ | – | | | | ohne Messeier | | | | | | | 140 | Ind. | ″ Quarzgeber zertrümmert. |
| 121 | 12.11. | 1,0 | ″ | 0,73 | 0,73 | 7,5 | ″ | – | – | | | ″ | ″ | | | | | | 117 | 96 Ind. | ″ |
| 117 | 11.11. | 1,33 | ″ | – | – | 6,5 | ″ | – | x | x | 30 | 26 | 23 | 30 | 162 | 130 | 138 | 100 | 163 | Ind. | ″ |
| 118 | ″ | 1,66 | ″ | – | – | ″ | ″ | – | x | x | 31 | 32 | 31 | 31 | 190 | 144 | 127 | 93 | 135 | Ind. | ″ |
| 119 | 12.11. | 2,0 | ″ | – | 0,53 | 7,5 | ″ | – | x | x | 30 | 31 | 33 | 47 | 122 | 138 | 138 | 111 | 178 | 165 Ind. | ″ |

75

Zahlentafel 6

**Versuche im 200 mm Rohr mit reinem Azetylen.**

| Vers. Nr. | Datum | Anfgs. druck kg/cm² | Art u. Stärke der Zündung | Zünddauer sec. | Verbr. dauer sec. | Temp. d. Gas °C | Reinh. d. Gas % | Jnd 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Jnd 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle | | | | | | | | | | | | | |
| 10 | 21.10. | 0,3 | 3 Pt-Drähte 15 Amp = | – | – | 8,5 | 99,0 | – | – | – | – | – | – | – | – | – | – | – | – | – | Kerze nicht verrusst. Kein fortschr. Zerfall. |
| 33-40 | 25.10 | ″ | ″ ″ | – | – | 7,5 | ″ | – | – | – | – | – | – | – | – | – | – | – | – | – | ″ ″ |
| 43-51 | ″ | 0,35 | ″ ″ | – | – | ″ | ″ | – | – | – | – | – | – | – | – | – | – | – | – | – | Nur ganz wenig Russ an Kerze. Kein fortschr. Zerfall. |
| 14 | 18.10. | -0,4 | ″ Akku = | – | – | 10 | 99,2 | Jnd. | × | × | × | × | × | – | – | – | – | – | – | – | Kerze verrusst. Russ bis 1m von Zündstelle. Kein fortschr. Zerfall. |
| 16 | 19.10. | ″ | ″ 25 Amp = | 0,15 | – | ″ | 99,0 | – | 30 | 22 | 33 | 33 | 38 | 33 | 22 | 13 | 14 | <u>172</u> | 157 | Jnd. | Fortschr. Zerfall. |
| 22 | 22.10. | ″ | ″ 15 Amp = | 1,84 | – | 9 | ″ | – | Rohr nach dem Versuch nicht geöffnet. | | | | | | | | | | | | Kerze nicht verrusst. Kein fortschr. Zerfall. |
| 23 | ″ | 0,65 | ″ ″ | 0,58 | – | ″ | ″ | – | | | | | | | | | | | | Jnd. | ″ ″ |
| 17 | 19.10. | 0,5 | ″ 25 Amp = | – | – | 9,5 | ″ | – | × | × | × | – | – | – | – | – | – | – | – | Jnd. | Kerze verrusst. Kein fortschr. Zerfall. |
| 18 | 21.10. | ″ | ″ 15 Amp. = | 0,39 | – | 8,5 | ″ | – | × | × | × | × | – | – | – | – | – | – | – | Jnd. | ″ ″ |
| 20 | ″ | ″ | ″ ″ | – | – | ″ | ″ | – | 29 | 30 | 21 | 20 | 2C | 32 | 29 | 51 | <u>147</u> | 94 | 127 | – | Fortschr. Zerfall. Ab 20 Versuch ohne Azetonabscheider. |
| 24 | 22.10. | ″ | ″ ″ | 0,35 | 1,58 | 9 | ″ | – | 29 | 21 | 28 | 32 | 21 | 33 | 20 | 29 | <u>189</u> | 97 | 127 | 134 Jnd | ″ ″ |
| 15 | 18.10. | 0,6 | ″ 25 Amp = | 0,15 | – | 10 | ″ | Jnd. | – | 57 | 38 | 46 | 33 | 42 | 20 | 33 | <u>>230</u> | 177 | 178 | – | ″ ″ |
| 21 | 21.10. | ″ | ″ 15 Amp = | – | 1,15 | 9 | ″ | – | 28 | 33 | 24 | 38 | 28 | 31 | 30 | <u>78</u> | >200 | 132 | 116 | 91 Jnd | ″ ″ |
| 25 | 22.10. | 0,8 | ″ ″ | 0,84 | 1,01 | ″ | ″ | – | 25 | 27 | 28 | 36 | 30 | 30 | 28 | <u>190</u> | 117 | 133 | 130 | 151 Jnd | ″ ″ |
| 26 | 23.10 | 1,0 | ″ 20 Amp = | 0,17 | 0,64 | 7,5 | ″ | – | 33 | Keine Messeier | | | | 35 33 33 | 36 | <u>>250</u> | 107 | 83 | 119 | 95 Jnd | ″ ″ |
| 122 | 13.11. | ″ | ″ 15 Amp = | 0,09 | 0,578 | 8 | ″ | – | | Ohne Messeier | | | | | | | | | 125 | 146 Jnd | ″ ″ |
| 123 | ″ | ″ | ″ ″ | 0,09 | 0,025 | ″ | ″ | – | | | | ″ | ″ | | | | | | 154 | 161 Jnd | ″ ″ |
| 124 | ″ | ″ | ″ ″ | 0,0 | 0,95 | ″ | ″ | – | | | | ″ | ″ | | | | | | 165 | 210 Jnd | ″ ″ 4 Bolzen abgerissen |
| 27 | 23.10 | 1,33 | Zündpille | 0,002 | – | 7,5 | ″ | – | 23 | Keine Messeier | | | | 38 32 35 | <u>>290</u> | >200 | 87 | 96 | 119 | Jnd. | ″ ″ |
| 28 | ″ | 1,66 | ″ | ″ | 1,56 | ″ | ″ | – | 34 | 47 | 45 | 42 | 39 | 37 | <u>>290</u> | 197 | 120 | 110 | 116 | 125 Jnd | ″ ″ |
| 29 | 24.10. | 2,0 | ″ | ″ | 1,84 | ″ | ″ | – | 41 | 57 | 46 | 46 | 46 | | <u>212</u> | 170 | 170 | 125 | 144 | 147 Jnd | ″ ″ |

63

Zahlentafel 7

## Versuche im 300 mm Rohr mit reinem Azetylen.

| Vers Nr. | Datum | Anfgs druck kg/cm² | Art u. Stärke der Zündung | Zünd dauer sec. | Verbr. dauer sec. | Temp. d. Gas °C | Reinh. d. Gas % | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle: Ind. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Ind. 30,0 | Bemerkungen. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 56-58 | 30.10. | 0,3 | 3 Pt-Draht 15 Amp = | – | – | 5,5 | 99,3 | – | – | – | – | – | – | – | – | – | – | – | – | – | Kerze nicht verrusst. Kein fortschr. Zerfall. |
| 96-102 | 7.11. | 0,3 | " " | – | – | " | 99,2 | – | – | – | – | – | – | – | – | – | – | – | – | – | " " |
| 59 | 30.10. | 0,4 | " " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | – | – | Nur ganz wenig Russ an Kerze. Kein fortschr. Zerfall. |
| 103-104 | 7.11. | 0,4 | " " | – | – | 8 | " | – | – | – | – | – | – | – | – | – | – | – | – | – | " " |
| 108-114 | 8.11. | 0,4 | " " | – | – | 5,5-8 | " | – | – | – | – | – | – | – | – | – | – | – | – | – | " " |
| 115 | 9.11. | 0,5 | " " | – | – | 5,5 | " | – | – | – | – | – | – | – | – | – | – | – | – | – | " " |
| 60 | 30.10. | 0,5 | " " | – | – | " | " | – | 40 | 41 | 55 | 54 | 61 | 38 | 39 | 28 | 60 | 105 | >350 | – | Fortschr. Zerfall. Flansch abgerissen. |

**Zahlentafel 8**

**Versuche im 400 mm Rohr.**
**mit reinem Azetylen**

| Vers. Nr | Datum | Anfgs druck kg/cm² | Art u. Stärke der Zündung | Zünd dauer sec. | Verbr. dauer sec. | Temp. d. Gas °C | Reinh. d. Gas % | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle: Ind 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Ind 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 30 | 24.10 | 0,3 | 3 Pt-Drähte 15 Amp. | – | – | 7,5 | 99,0 | – | – | – | – | – | – | – | – | – | – | – | – | – | Kerze nicht verrusst. Kein fortschr. Zerfall. |
| 31 | 25.10 | 0,4 | " | – | – | " | " | – | – | – | – | – | – | – | – | – | – | – | – | – | " " |
| 32 | " | 0,5 | " | – | – | " | " | – | 64 | 74 | 73 | 95 | 71 | 65 | 92 | 36 | 62 | 150 | 150 | – | Fortsch. Zerfall. Flanschdichtung verbrannt. |
| 42 | 26.10 | 0,6 | " | – | – | 6 | 99,1 | – | 70 | 65 | 78 | 81 | 72 | 49 | 68 | 83 | >343 | >315 | >257 | – | " Flansch neben Schweissnaht gerissen. |

Zahlentafel 9

**Versuche im 400 mm Rohr.**
**mit Azetylen - Stickstoff - Gemischen.**

| Vers. Nr. | Datum | Anfgs druck kg/cm² | Art u. Stärke der Zündung | Zünddauer sec. | Verbr. dauer sec. | Temp. d. Gas. °C | $C_2H_2$ : $N_2$ % | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle: Ind. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Ind. 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 175 | 28.11. | 7,0 | 3 Pt-Drähte 25 Amp. = | 0,058 | | 6,0 | 40/60 | — | x | x | x | x | x | x | x | x | x | x | 1,8 | 4,2 Ind. | Fortschr. Zerfall; Reihenfolge d. [illegible] nicht mehr feststellbar, höchst Druck 20 at |
| 171 | 26.11. | 3,0 | " 18 Amp. = | — | — | 5,0 | 50/50 | — | — | — | — | — | — | — | — | — | — | — | — | — | Kein Zerfall. |
| 172 | " | 4,0 | " " | — | — | " | " | — | 4,0 | — | — | — | — | — | — | — | — | — | 4,0 | — | Fortschr. langsamer Zerfall. |
| 173 | 27.11. | 1,0 | " " | — | — | 5,0 | 70/30 | — | — | — | — | — | — | — | — | — | — | — | — | — | Kein Zerfall. |
| 174 | " | 2,0 | " " | — | — | " | 71/29 | — | — | — | — | x | x | x | x | x | x | x | x | — | Fortschr. Zerfall. |

79

Zahlentafel 10

**Versuche im 200 mm Rohr mit Azetylen-Stickstoff-Gemischen.**

| Vers. Nr. | Datum | Anfgs druck kg/cm² | Art u. Stärke der Zündung | Zünd dauer sec. | Verbr. dauer sec. | Temp. d. Gas. °C | $C_2H_2$ : $N_2$ % | Explosionsdrucke in kg/cm² in m Entfernung v. Zündstelle: Jnd. 0,0 | 0,0 | 0,2 | 0,5 | 1,0 | 2,0 | 5,0 | 10,0 | 15,0 | 20,0 | 25,0 | 30,0 | Jnd. 30,0 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 148 | 19.11. | 5,0 | 3 Pt-Drähte 25 Amp= | – | – | 6,5 | 40/60 | – | – | – | – | – | – | – | – | – | – | – | – | – | Kein Russ an Kerze. Kein Zerfall |
| 149 | 20.11. | 6,0 | " " | – | – | " | 46/54 | – | – | 48 | 28 | 31 | 33 | 43 | 39 | 32 | 33 | 26 | 35/58 | – | Fortschr. Zerfall. Flockiger Russ. |
| 151 | 21.11. | 3,0 | " " | – | – | 6,5 | 50/50 | – | – | – | – | – | – | – | – | – | – | – | – | – | Kein Zerfall. Kein Russ an der Kerze. |
| 150 | 20.11. | 4,0 | " " | – | – | " | " | – | × | × | × | × | × | × | 30 | 15 | 21 | × | × | – | Fortschr. Zerfall, flockiger Russ. Messeier lagen verkehrt. |
| 152 | 21.11. | 8,0 | " " | – | – | " | " | – | 75 | 81 | 55 | 60 | 57 | 61 | 60 | 58 | 67 | 55 | 107/133 | – | Fortschr. Zerfall. Flockiger Russ. |
| 153 | 21.11. | 2,0 | " " | – | – | 6,5 | 75/25 | – | 106 | – | 92 | 81 | 62 | 74 | 89 | 89 | 89 | 111 | >450 | – | Fortschr. Zerfall. Alle Bolzen abgerissen. |

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Illustration I

Illustration II

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Illustration III

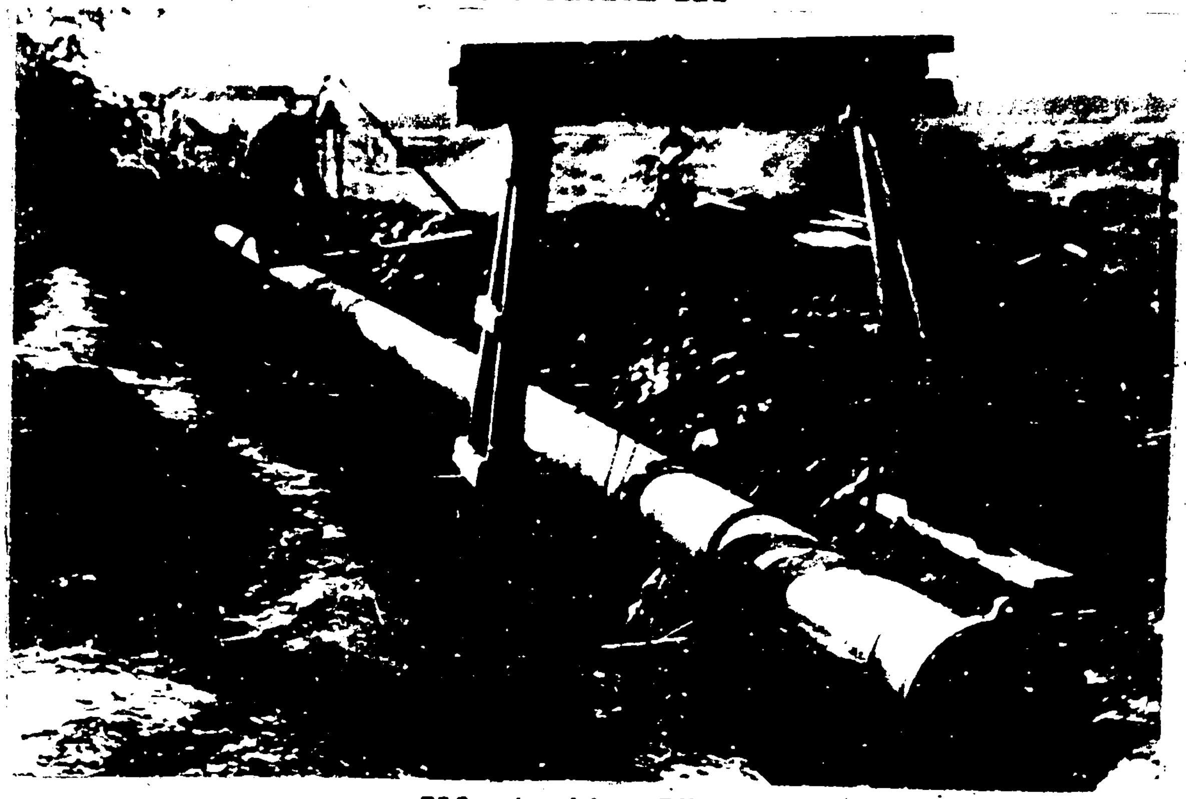

Illustration IV

Illustration V

Illustration VI

Illustration VII

Illustration VIII

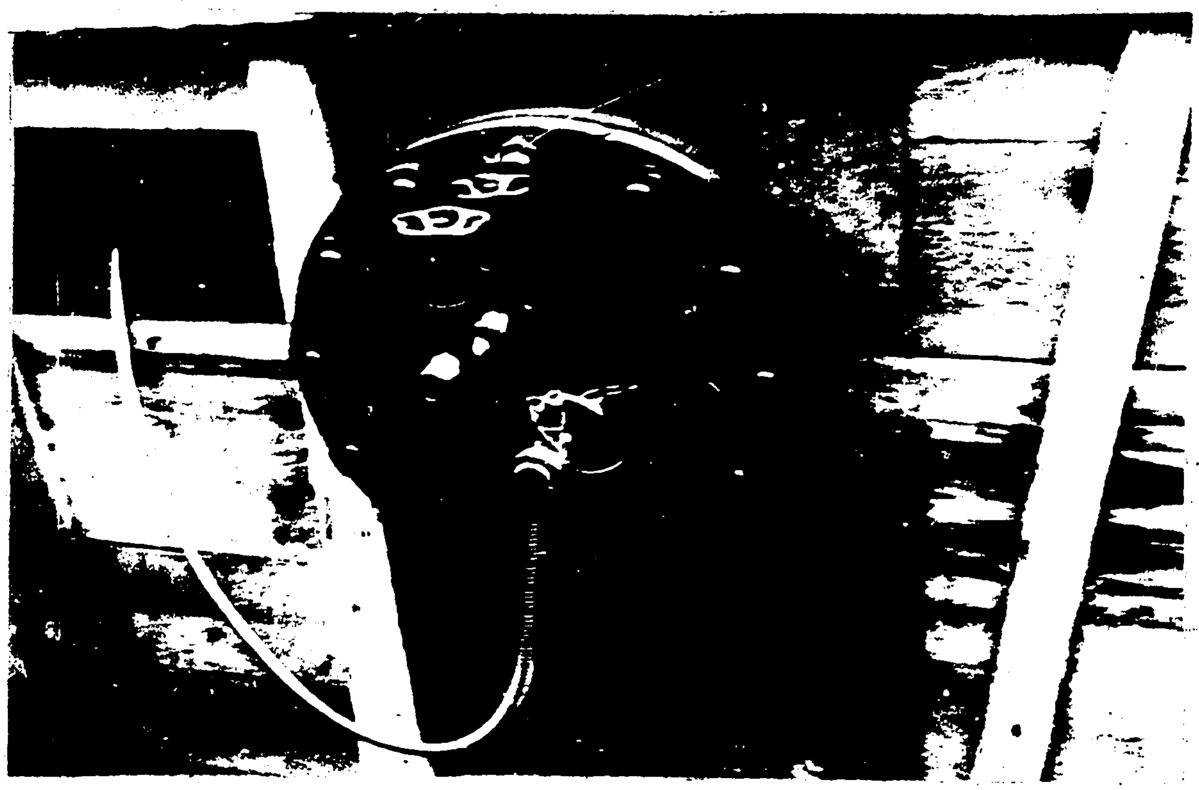

Illustration IX

85

Illustration X

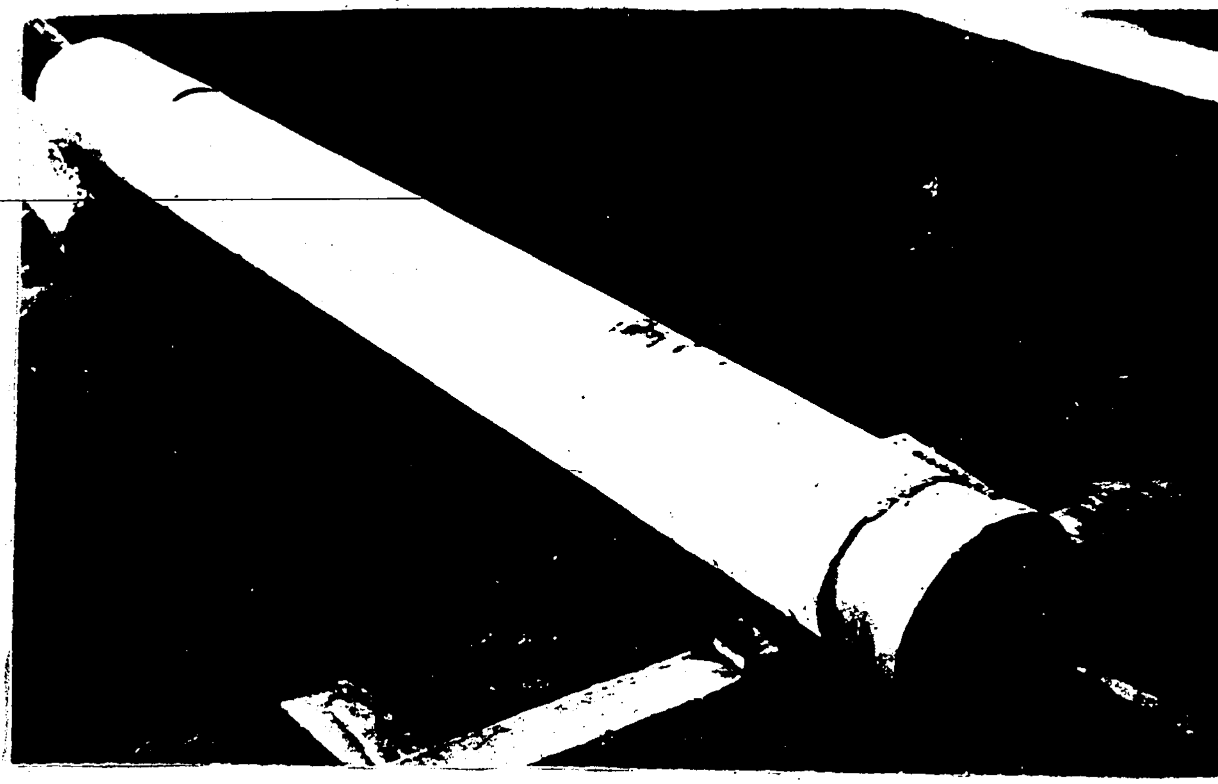

Illustration XI

Illustration XII

Illustration XIII

Illustration XIV

Illustration XV

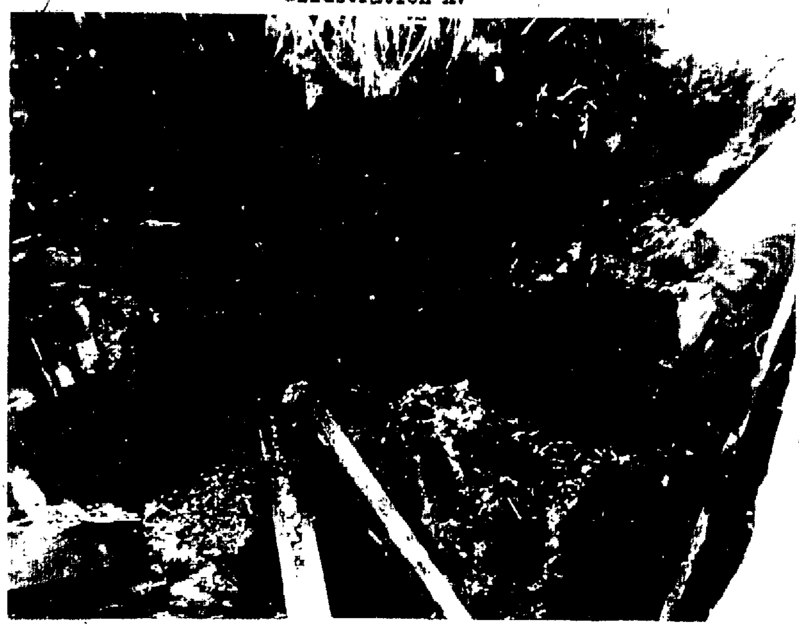

Illustration XVI

Illustration XVII

Illustration XVIII

Illustration XIX

Illustration XIX

EXHIBIT C

I. G. Farben
Physics Laboratory
Dr. Weissweiler

Ludwigshafen on the Rhine
September 24, 1942

## EXPERIMENTS BY THE PHYSICS LABORATORY, 1940-1941 TO DETERMINE THE DECOMPOSITION PRESSURE OF ACETYLENE AND THE MEANS OF PREVENTING TRANSITION OF A SLOW DECOMPOSITION INTO A DETONATION

In the past few years there has emerged a very definite acetylene chemistry. Acetylene is the raw material for a vast series of important synthetic products among the most important of which is synthetic rubber, our Buna. It is understandable that such a versatile gas must be subject to a variety of widely different conditions in the manufacturing processes. In particular, we cannot be satisfied to handle it in pipes and equipment at very low pressures as has been customary heretofore, but we must strive for appreciably higher pressures. The manufacture of butinediol by the Reppe process requires gas at pressures up to 5-6 atm. gauge. Many other Reppe processes now being developed or even already worked out require still high pressures.

The question was therefore raised as to the maximum pressures to be expected as a result of acetylene decomposition and their dependence upon initial pressure, first for pure acetylene and second for mixtures of acetylene with inert gases. ($N_2$ or $CO_2$), finally for acetylene in the presence of catalyst. So far as possible it was also desired to know something about the character of the pressure wave, i.e., whether it was fast or slow, etc. Finally, means should be found to stop a decomposition or at least to prevent a slow decomposition from changing to a detonation.

This investigation was carried out during 1940-1941 in Ludwigshafen and Griesheim. At the latter place, an experimental installation was utilized which had been designed by the C.T.R. in Berlin for the investigation of acetylene decomposition in long pipe lines. We had already made the first series of experiments in Ludwigshafen in two high pressure pipes 1 m. long and either 90 mm or 120 mm diameter, before the C.T.R. had begun their work at Griesheim. The C.T.R. wanted to clarify the problem of long acetylene gas lines, viz., to determine up to what pressure acetylene could be safely handled in long pipelines, i.e., with the assurance that a slow decomposition occurring somewhere would not be changed into a detonation. We had assumed that an explosion chamber 5.5 m. long was enough to yield sufficiently accurate information over explosion pressures and their character. Unfortunately, the experiments by the C.T.R. in Griesheim showed that this was not the case. It is necessary to go to considerably longer chambers to determine whether or not a detonation would occur in a long pipeline. Of course, it goes without saying that such long pipes for experiments are only necessary if one is dealing in actual practice with long conduits. For smaller reaction vessels and short lines, experiments on a smaller scale are sufficient.

A brief summary of the Griesheim results is as follows:

6 pipes; 30 m. long; 25, 50, 100, 200, 300 and 400 mm. I.D., were used. They were buried horizontally in the ground. Ignition was at one end by means of a spark plug, which carried three parallel platinum wires of 0.15 mm thickness. Ignition was with D.C. current of approximately 15 A. Pressure measurement was by means of a copper compression cylinder, a ball pressure measuring apparatus, and piezo-quartz. Pure acetylene from cylinders was used (99-99.2% $C_2H_2$). The results showed that in the 50 mm pipe pressures up to 2 atu. could be used without detonation occurring. With pipes of I.D. 100 mm and higher, detonation developed at 0.4-0.6 atu. (With detonations the resulting pressure is far more than 12 times the initial pressure).

These experiments are, of course, binding on all future aceylene installations which involve compressed acetylene in long pipes.

Before going into the details of the experiments themselves, we shall present a few preliminary considerations:

1. <u>Type of Ignition</u>. The probability that a decomposition started at a point by some igniter will be

propagated further, assuming, of course, sufficient pressure, is dependent upon the kind and strength of ignition. In acetylene decomposition experiments, both according to our experience as well as that of the C.T.R., a platinum wire brought to instantaneous fusion has proved very satisfactory. The C.T.R. used for their experiments three parallel platinum wires of 0.15 mm; we used one of 0.3 mm diameter. As an example of the effect of strength of ignition, data by Boesler of Oppau are shown in the following table. The experiments were carried out in a cylinderical vessel of 15.5 l. content. Ignition was at the center of the gas chamber and was by means of wire fusion:

| Molybdenum | Melting | Point | 2550° | Initial | Pressure | 1.40 ata* |
|---|---|---|---|---|---|---|
| Platinum | " | " | 1773° | " | " | 1.40 " |
| Iron | " | " | 1530° | " | " | 1.74 " |
| Copper | " | " | 1083° | " | " | 1.84 " |
| Aluminum | " | " | 658° | " | " | 2.65 " |
| Lead | " | " | 327° | " | " | 7.50 " |

The table shows that with a decreasing melting point and consequently decreasing ignition energy, the initial pressure must be increased in order to cause decomposition of the entire contents of the vessel.

It has been our experience that a flame front, such as results in an acetylene-filled pipeline, which fills the entire cross section, acts a a very strong initial igniter. It can be concluded from this that to attain a self propagating decomposition, the heat liberated must be greater than the losses through conduction, radiation and convection.

In order to compare different experiments, the same method of ignition was used at all times. In doing this, we were aware that in practice such energetic ignition would seldom occur.

* 1 atu = 1 kg./$cm^2$ gauge pressure
1 ata = 1 kg./$cm^2$ absolute pressure

2. Size and Shape of the Explosion Vessel. It is a well known fact that shape, size and material of the explosion vessel influence the explosive limits of combustible gases. The same is true of acetylene decompositions. If one disregards the possible effect of the walls in breaking up chain reactions or their accidental role as catalysts, then the effect of the explosion chamber is dependent upon the heat balance of the process. It is, of course, obvious that the heat loss decreases with increasing ratio of volume to surface. It is, therefore, correct, not to conduct experiments in too small volumes. Also, the bomb volume does not have to be too large because as experience shows, above a certain size (varies with gas in question) further increase has no effect.

Also, the location of the igniter in the explosion chamber is important. With a vertical vessel, one gets the widest "explosive range" if the igniter is at the bottom of the vessel since then convection favors the spread of combustion. The decomposition of acetylene is analogous. In very long pipes also there is the danger that a slow decomposition in acetylene can change to a detonation, i.e., the speed of decomposition reaches detonating speed (1000 - 2000 m./sec.). In our experiments with larger pipe sizes, it was not possible to install the pipes vertically, and so they were used in a horizontal position. Only at the end of our experiments, narrow pipes (nominal diameter 10, 12.5 and 16 mm) were set up perpindicularly and ignited at the base.

According to the experiments of Dr. Rimarski of the C.T.R., a pipe length of 30 m. should be sufficient to permit transition of a slow decomposition into a detonation. Our experiments proved the correctness of this assumption. During our first experiments in Ludwigshafen in a pipe of 120 mm I.D., and 5.5 m. long, no detonations were observed. However, in the pipe 30 m. long used at Griesheim, detonation definitely occurred. From this it follows that in practice, long pipe lines carrying acetylene under pressure are dangerous and should be protected. However, in the case of autoclaves, vessels, etc., containing acetylene no detonation pressure can occur unless a detonation wave enters the container through a pipeline.

3. Measurement of the Decomposition Pressure. In the experiments at Ludwigshafen, the decomposition pressure was measured most effectively with a piezo-quartz and cathode ray oscillograph made by Zeiss of Dresden. The quartz was calibrated before each series of experiments, and the sensitivity of the oscillograph so set that the pressure peaks fell on the diagram. In the Griesheim experiments, the same equipment was also used. At Griesheim, it developed that to measure very rapid pressure rises (0.001 seconds and less) the natural frequency of the quartz must be higher. In some experiments, it could not be determined whether very rapid vibrations shown by the oscillograph were actually vibrations of the gas column or of the quartz itself. (A new piezo-quartz with a natural frequency of 40,000 to 60,000 was made available by Zeiss after the Griesheim experiments were concluded.) In Griesheim, also, two additional types of apparatus were used, namely, the copper compression cylinder and the Krupp indentation apparatus at lower and medium pressures without too rapid pressure rise. These instruments gave fairly good agreement with the quartz measurements. However, in the case of a very steep wave front, they gave considerably higher pressure than the quartz. Without doubt there occurred a certain flow of the copper so that the indentation of the ball in the copper or the compression of the cylinder was too great.

4. Kind of Gas. For the experiments, cylinder acetylene with a purity of 99 to 99.2 percent was used. In some of the experiments at Ludwigshafen, gas from the Reppe butinediol process also was used. This gas consisted of 96% $C_2H_2$, 2% $H_2$, 1% CO and 1% $CO_2$.

## THE EXPERIMENTS

### I. Experiments at Ludwigshafen in Pipes 1 and 5 m. Long.

1. Experiments in 1 m. Pipe. The diameter was 90 mm. The pipe specification was 325 at. The pipe was used in a horizontal position. Ignition was accomplished by fusion of a 0.3 mm platinum wire. Pressure was by means of piezo-quartz. Initial pressures were 4 to 9 atu. Initial temperatures were 30°, 40°, 100°, 110°, 130°C. The gas was in most cases dry, although in some, saturated gas was used. In other experiments, considerable quantities of the liquid occurring in the process, namely, water solutions

of formaldehyde and butinediol were introduced into the pipe, also varying quantities of catalyst (copper acetylide on silica gel), both dry and wet, and both fresh and used.

The chief results of these experiments are as follows: In the experiments with dry gas, the maximum pressure developed was approximately 10 to 12 times the initial pressure and was reached in some 0.2 to 0.3 seconds after ignition. After the introduction of dry catalyst, final pressures of 14 times the initial were measured. This was for a temperature of 30 to 40°C. However, the condition was more favorable with moist catalyst in the bomb. Here the retarding action of the high water vapor content (1 ata and more) was very noticeable. The final pressures amounted to only 3.6 times the initial pressures. (Water vapor pressure for 30°, 110° and 130° amounts to 0.043, 1.46, and 2.75 ata respectively). With a working temperature in the butinediol process of 130°C and 6 ata total pressure, the vapor pressure of water is 2.75 ata as compared to the acetylene partial pressure of 3.25 ata. It will be noted that for a constant pressure the acetylene content by weight of the saturated gas decreases in proportion to the absolute temperature. For 30°C and 130°C, the ratio is $\frac{303}{403}$, that is an approximately 25% decrease.

All experiments in the presence of catalyst, even when dry gave maximum pressures not appreciably higher than those without catalyst. Only the time for the attainment of the maximum pressure was somewhat lower. (In these experiments the catalyst grains did not fill the entire pipe cross section). As was shown in later experiments in the 5.5 m. long pipe (120 mm I.D.), catalyst filling the entire pipe cross section acts as an explosion arrestor since in every experiment the decomposition did not pass through.

These experiments are comforting in the event that a decomposition for any reason should occur in the catalyst tower. The catalyst is comparatively harmless and in no way can be compared with well known igniters such as fulminate of mercury, etc. This is because no gas is set free by its decomposition even though heat of formation is released.

In the 1 m pipe, experiments were undertaken with $C_2H_2$-$N_2$ and $C_2H_2$-$CO_2$ mixtures. These experiments demonstrated the well known fact that small additions of inert gas do not decrease appreciably the maximum pressure reached. With an initial pressure of 6 ata and 25°C initial temperature, the final pressure for the mixtures with $N_2$ follow:

| | | | |
|---|---|---|---|
| 0 | % | $N_2$ | 62 ata |
| 10 | " | " | 61 " |
| 20 | " | " | 58 " |
| 30 | " | " | 53 " |
| 47 | " | " | 49 " |
| 48 | " | " | 43 " |
| 49 | " | " | No decomposition |

With $CO_2$ mixtures the conditions are somewhat more favorable. In this case, 42% $CO_2$ is sufficient to prevent a decomposition.

A mixture of 50% $C_2H_2$ and CO first decomposed at an initial pressure of 7 ata., however, the pressure rise was very slow and required some 0.9 to 1.3 seconds. At 21 ata initial pressure the final pressure was 185 ata, and was achieved in 1.10 seconds. All experiments with acetylene to which ethyl alcohol vapors had been added showed roughly the same action as additions of $N_2$, $CO_2$, CO, and water vapor.

2. Experiments in the 5.5 m. Pipe. All experiments carried out in the 1 m pipe were repeated in a pipe of 5.5 m length and 120 mm I.D. with practically the same results. In this pipe 50 liters of catalyst were packed, acetylene added at a pressure of 6 ata, and ignited. The decomposition stopped after penetrating the catalyst mass some 10 cm. The catalyst, filling the entire cross section, had a retarding action. In an experiment where the catalyst was distributed the length of the pipe but only filling about 1/4 of the cross section, a maximum pressure of 128 ata was attained in some 0.01 seconds.

In this 5.5 m pipe, a number of explosion arresters were tried. Originally we had set for ourselves the goal of finding an arrester which, under all conditions, would stop any explosion. In these experiments, in order to have as severe conditions as possible, pure, dry acetylene was used at room temperature and 6 ata pressure. The starting distance for the decomposition wave that is, the distance between the ignition wire and the arrester was increased from 0.5 m to 5 m. These experiments demonstrated that it is possible to quench an explosion with porous filters or with porous plates of sintered quartz. However, these arresters because of their high resistance to flow are not practical for use in the butinediol process. The compression of acetylene to the system pressure of 6 atu is accomplished by so-called Elmo compressors, which compress the gas by the centrifugal force of a water seal and present no possibility of ignition. They are not, however, capable of producing high pressures. As an arrester with less resistance, a mass of metal wool was tried. We thought for a while that we had found the desired device as the following table shows:

1. Starting distance of the explosion wave: 0.5 m.
   - 5 plugs each 376 g.: No break-through
   - 5 plugs each 115 g.: No break-through

2. Starting distance: 2.6 m.
   - 5 plugs each 334 g.: No break-through
   - 5 plugs each 283 g.: No break-through

3. Starting distance: 5.0 m.
   - 8 plugs each 350 g.: No break-through
   - 5 plugs each 350 g.: Weak break-through
   - 5 plugs each 210 g.: Strong break-through

On the basis of these experiments such steel wool plugs were also tried at Griesheim in the 30 m pipe (200 mm I.D.). However, in these experiments a very undesirable thing happened. In two experiments, in which the plugs had apparently stopped the explosion, a break-through occurred some 20 minutes after ignition with a heavy detonation which destroyed the other end of the pipe. The incandescent wires in the end of the plug nearest the ignition point continued to decompose the acetylene. The hot decomposition zone moved slowly through the plug until finally the whole mass of undecomposed acetylene detonated. After this experience we gave up the whole idea of such plugs as explosion arresters. At this point the experiments having to do with acetylene decomposition as it might be met in the Reppe process were ended. The investigation was continued at the suggestion of the C.T.R. in the 30 m pipe at Griesheim with the splendid cooperation of Dr. Holler and Ing. Schnedler. The investigation was concluded with a series of special experiments conducted at Ludwigshafen in narrow vertical pipes 30 m long.

This part of the work is reported below:

In two discussions with Dr. Rimarski, President of the C.T.R., and his co-worker, Dr. Konschak, the goal of further work was discussed. It was decided that it would be sufficient to find a device which could assure that a slow decomposition did not change to a detonation. The requirement that any decomposition be stopped was accordingly abandoned, since this seemed possible only with devices which had an impractical resistance to flow. While an apparatus cannot be protected practically against a detonation pressure, this is not the case with slow decompositions which merely result in pressures 10 to 12 times the initial pressures. Since in the Reppe butinediol process, the system pressure in the gas lines amounts to 6 to 7 ata, a maximum pressure of 70 to 80 ata would be encountered in a slow decomposition. Construction of the apparatus of material of specification 64 or 100 would offer sufficient security. Also, in practice, conditions of ignition would not be so severe as we used in our experiments and accordingly lower pressures should actually occur.

We made a series of experiments in Griesheim with steel plugs as arresters, which were installed in a pipe of 200 mm I.D. Since, however, this type of arrester was shown to be unreliable, we decided against their use as already mentioned. We, therefore, shall not discuss these experiments further in this report. In the appendix are shown some pictures of these filters.

As an arrester to prevent the transition of a slow decomposition into a detonation, the installation of a bundle of small tubes finally proved to be very effective. This arrangement fortunately causes only a small pressure drop in a pipe line. The first experiments of the C.T.R. in Griesheim had established the fact that in small pipes (1 inch and smaller) slow decomposition cannot change to detonations. For many years it has been a known fact that explosions of gas mixtures in small pipes are difficult to bring about, i.e., the explosion range becomes much narrower. The cause for this lies in the good heat transfer from the flame front to the pipe wall. (Unfavorable ratio of gas volume to surface). As exact theoretical analysis shows, the wall thickness of such a pipe actually can be very small without appreciable influence on the effectiveness of the pipes. (In this connection see Jost, "Explosions und Verbrennungsvorgange in Gasen"), Berlin, Springer, 1939, or page 123 ff or our own report, "Theory of Acetylene Decomposition: Influence of Internal Diameter and Wall Thickness of a Pipe on the Transmission of a Decomposition Wave", 6-21-41.

In Griesheim a 30 m long pipe of 100 mm I.D. was filled with a bundle consisting of 13 ½" pipes, and 6 3/8" pipes. At the suggestion of Dr. Rimarski, a free space of 30 cm. was left at each end. Ignition and pressure measurements were the same as in the experiments by the C.T.R.

The following remarks are made with respect to the pressure measurements.

In all experiments with moderate pressures of fairly long duration, all three pressure measuring devices gave results sufficiently in agreement. At very high and very rapid pressure rises (over 100 atu), however, the compression cylinder and ball indentation methods gave values 2 to 4 times as high as those determined by the piezo-quartz. The quartz gave unquestionably the better results according to expert opinion. With rapid loading of the small copper cylinder, flow of the metal takes place so that the ball indentation is too great. These values which are apparently much too high are given in Table 1 in parenthesis and are shown with question marks in the other tables.

The suitability of such a system as a means of preventing detonation was thoroughly investigated under a variety of experimental conditions. In Table 1 of the Appendix is given an extract from our field notebooks.

The fact was established that the tube bundle at smaller pressures up to approximately 3.4 ata not only prevented detonation but also stopped the explosion so that the decomposition stopped in the first half of the bundle. At higher initial pressures 4, 5 and 6 atu, breakthrough occurred. However, the maximum pressure recorded by the Piezo-quartz was 120 atu. The end of the pipe after the failure was only warm to the touch. This pressure would be easily withstood by equipment built with pipe of specification 64, because it is still far below the elastic limit of the material.

The effectiveness of the tube bundle is diminished by rust formation because of the resultant poorer heat transfer. This is shown in Table 1, Experiments 1 to 13. Therefore, if decomposition occurs in the plant, soot should be carefully removed from all pipes and equipment which are equipped with this type of device. However, even in the rusty tube bundle the decomposition at low initial pressures was so slow that the final pressure could not be measured. It was merely a very slow burning. After thorough cleaning, the effectiveness of the tube bundle returned to its original value.

In connection with the experiments on the tube bundle, experiments on two pipes of 60 m length and ½" diameter, that is, 10 mm I.D. were undertaken at pressures of 5.1 to 10 atu. (Table 1, 14 to 19; 24 ff). The ½" pipe stopped the decomposition up to 5 to 6 atu, and, with both pipes, the final pressure at the end of the pipe was less than 10 times the initial pressure.

With this 60 m pipe it was ascertained that bends in the pipe (even a large number) did not stop a decomposition. Somewhat surprising was the fact that good cooling of a pipe (10 mm I.D.), which had been provided with many bends and then laid in a cold water bath did not result in any increased effectiveness. In the report 6-24-41 mentioned above, the theoretical reason for this is discussed.

The Griesheim experiments with the tube bundles just discussed proves that we have found the desired device for prevention of detonations up to working pressures of approximately 7½ atu. It is again emphasized, however, that the Griesheim experiments were carried out under particularly unfavorable conditions, especially insofar as the method of ignition was concerned.

For the first butinediol installation (in Schkopau) the following specifications were laid down on the basis of the experiments discussed above and carried out: all pipelines with a nominal diameter of 1" or over which carry acetylene under a working pressure of several ata are equipped with such tube bundles or corresponding safety devices. The I.D. of the tubes is a maximum of ½" or less if possible. Since soot deposition diminishes the effectiveness of these small tubes, tubes as smooth as possible, should be used. Also, they should be kept as clean as possible. Since, according to our experiments, elbows are not effective as explosion arresters, they should also be provided with small tubes. If the pressure drop permits, Raschig rings or the equivalent are also satisfactory. Likewise, all dead spaces should be filled with Raschig rings or the equivalent in order to break large volumes into as many parts as possible.

So far as other safety measures are concerned, it may be mentioned that the manometer leads should be provided with orifices to damp pressure waves resulting from a decomposition. Also meters should be several times normal range and strength. For ring balance meters for flow measurement, the high pressure installation used at Oppau is recommended.

Further safety measures which are necessary for the butinediol installation are beyond the scope of this report.

## FINAL ACETYLENE DECOMPOSITION EXPERIMENTS FOR THE REPPE BUTINEDIOL PROCESS NOVEMBER AND DECEMBER 1941 IN GRIESHEIM AND LUDWIGSHAFEN

After the results of the Griesheim experiments had been discussed with the C.T.R., they requested another final series of experiments which will be reported below:

On September 24 and 25, 1941, the following was agreed to at the Chemische Technische Reichsanstalt and the Reichswirtschaftsministerium.

1. In Griesheim additional decomposition experiments were to be made in a horizontal pipe 30 m long of 100 - 120 mm I.D., which was to be provided with a tube bundle of approximately 10 mm tubes.

2. Further experiments were to be made in a horizontal pipe 30 m long of 100-120 mm I.D., filled with Raschig rings of steel and later with porcelain.

In both series of experiments, the pressure was to be gradually raised until it was certain that a detonation occurred at the other end of the pipe. The experiments, however, were not to be carried to the ultimate destruction of the pipe.

3. In Ludwigshafen, ignition experiments were to be carried out on a 30 m long pipe of approximately ½" I.D., installed vertically, in which the ignition would take place at the bottom end. Vertical pipes are much more favorable to propagation of an explosion if ignited at the bottom end. The pressure was to be increased until there was no doubt that a detonation had occurred at the upper end. In the course of the experiments, a vessel of approximately 2 liters volume was to be attached to the bottom end of the pipe and it was to be determined to what extent this chamber favored the formation of a detonation wave. These experiments were carried out in November and December 1941 at Griesheim and in Ludwigshafen. Data sheets are given at the conclusion of this report.

## DISCUSSION OF RESULTS

1. Measurements, Table II, Experiments 1 to 5

Pipe length 30 m; I.D. 120 mm; horizontal; filled with 50 tubes of 10 mm I.D. and 2 tubes of 8 mm I.D. The ends of these tubes were welded together and the bundle welded to the inside surface of the large pipe. Chambers 30 cm long filled with steel Rashig rings 35 x 35 mm were provided at the beginning and end of the large pipe. Ignition as usual was with a 0.3 mm platinum wire 10 mm in length. Pressure measurement by means of a copper compression cylinder; in some experiments by means of a piezo-quartz set in the end flanges of the pipe. The pressure was increased from 7 to 10 atu. The copper cylinders in the end chamber showed 195, 178, 234 atu.

It is demonstrated here again that the ball pressure apparatus gives much too high values at high pressure. Of course, our piezo-quartz was also not too reliable because of the very rapid pressure rise involved (some 1/1000 second) and the very steep wave front. We are at the moment discussing with Zeiss the possibility of obtaining special apparatus more suitable for such measurements. At any rate, the 120 mm pipe after installation of the tube bundle withstood the stress set up in spite of the fact that it was not a high pressure pipe (specification 64.)

For the last experiment of this series, the chamber at the ignition point was also reduced from 20 to 6 centimeters in depth. This time no Raschig rings were added. Pressure 9 atu.

The reduction in size of the first chamber had the expected action. The final pressure was appreciably smaller. In actual plant practice, empty spaces are to be avoided so far as possible or should be filled with Raschig rings or equivalent. The following series of measurements present further information on this subject.

2. <u>Measurements. Table II, Experiments 6 to 17</u>

Pipe length 30 m; I.D. 120 mm; horizontal; filled with steel Raschig rings 35 mm x 35 mm. The rings were made of strips of metal bent to a cylinderical form; sheet thickness was some 0.5 mm. Since in the C.T.R. experiments of the previous year detonations occurred at pressures of 0.6 to 1 atu, an initial pressure of 0.8 atu was used at first and then was gradually raised to 6 atu. The Raschig rings had a very definite retarding action on the progress of the detonation wave, so that at 1.4 atu there was no failure. At 2 atu there was a very weak break-through. At 3 atu there was no failure, and at 4 and 6 atu failure again occurred. At 6 atu pressure, the apparatus in the end flange gave the value of 320 atu which was much too high; the Krupp instruments gave 303, 81 and 85 atu. After these experiments, we cut open the pipe and found out that the Raschig rings had been pressed together and empty spaces of up to 1 m had resulted. The pipe was reasonably free of soot because of the thorough cleaning given it each time. At any rate, however, this comparatively rough installation exerted a strong retarding action on the propagation on the decomposition wave. So strong in fact that at an initial pressure of 6 atu no damage occurred to the pipe. If flow resistance presented by a pipe filled with such rings is unimportant, these rings can be used with assurance as protection against detonations.

The 120 mm pipe including the 2 chambers at each end was then filled with procelain Raschig rings 25 x 25 mm. Porcelain rings of the other size were not obtainable. In the first three experiments at 3.5 and 6 atu pressure, no break-through occurred. After opening the pipe it was found that the Raschig rings had been hurled almost 6 m from the ignition point agains the end of the pipe and pulverized to a considerable extent. The pipe was filled again and pressures of 7 and 8 atu were used. In both cases break-through occurred. In the last experiments a gasket became loose but the pipe, itself, was completely intact. The copper cylinders ahead of the end flange showed impressions corresponding to 268, 230 and 275 atu. These values are certainly too high.

The experiments with Raschig rings demonstrated that such rings are a good protection against detonations. Particularly, they can be used to fill large irregular spaces such as elbows. Raschig rings of metal are preferable to ceramic, since the latter can be destroyed easily by an explosion wave. It should, of course, not be forgotten that such pulverization causes stoppage of the pipeline and may very well stop the explosion wave in this way. However, this action is likely to be uncertain.

3. Measurements. Table III, Experiments 1 to 44

(a) 30 m pipe; vertical; I.D. 10 mm; specification 325, ignition at the bottom; pressure measurements with piezo-quartz. At 5 and 6 atu pressure there was no break-through and at 7 atu only a very weak one. The highest pressure rise was recorded as 327 atu at an initial pressure of 14 atu. At 15 atu pressure the oscillograph showed a pressure rise of 278 atu. These values are also certainly too high since the pressure rise is extremely rapid.

(b) 30 m pipe vertical; I.D. 12.5 mm; specification 64; ignition at the bottom; pressure measurement with piezo-quartz. With this pipe a break-through occurred at 5 atu. At a pressure of 6 atu a pressure rise of 81 atu was measured by the quartz, at 10 atu pressure a final pressure of 199 atu. The ball pressure device gave for all pressures of 5 to 10 atu final pressures between 200 and 300 atu, obviously much too high. The data were considerably scattered; however, results of experiments 26 to 21 looked more regular.

(c) 30 m pipe; vertical; I.D. 10 mm; specification 325 atu; ignition at the bottom; pressure measurement with piezo-quartz; also in this pipe failure occurred at a pressure of 5 atu. The initial pressure was raised to 12 atu and a pressure rise of 315 atu was recorded. The pressure rise in this pipe was also very steep. Because of the very short time in which the quartz is loaded (some 0.01 to 0.001 seconds), the values are certainly too high.

(d) Experiments with Entrance Chamber. 30 m pipe; vertical; I.D. 10 mm; specification 325; a chamber at the bottom end; length 500 mm; diameter 70 mm; volume 1.9 liters; ignition at the bottom end of the entrance chamber.

In these experiments the pressure rises were naturally too high. Whereas the same pipe without an entrance chamber, with an initial pressure of 10 atu gave a pressure rise of 160 atu, the same experiment with entrance chamber gave a pressure rise of 275 atu. These experiments were discontinued at 10 atu to protect the piezo-quartz. These experiments demonstrated definitely as expected that unprotected empty spaces which are large as compared to the pipeline cannot be tolerated. It is remarkable that the pressure rise with pressures 5 to 10 atu is almost linear. Whereas, the 10 mm pipe with entrance chamber showed no failures at 5 and 6 atu (the decomposition wave was extinguished in the pipe), the same pressures with entrance chamber gave final pressures of 144 and 185 atu.

Results of the Final Experiments

The investigation of the safety of acetylene pipelines against acetylene detonations through installation of tube bundles or use of Raschig rings was extended to 15 atu. The necessary pressures for the Reppe butinediol synthesis are 5 to 10 atu. For initial pressures of 5 and 6 atu, as was already known from our earlier experiments, very low pressure rises developed at the end of a pipe protected with a tube bundle. In an experiment with Raschig rings, it was found that at low pressures, the decomposition is extinguished and that even at 6 to 8 atu the violence of the explosion is so lessened that rings can very well be used as a protective device. At higher pressures the small pipes show a sharp pressure rise and as a matter of fact the more violent as the pipe diameter is increased. For higher pressures, the tube bundle must be made out of smaller tubes.

Above all, large empty spaces are to be avoided. They make the best tube bundle ineffective in that they permit detonations to occur even in small pipes.

This experimental investigation concludes the work requested by the C.T.R. over protection of the Reppe butinediol process against the danger of acetylene detonations.

Weissweiler

Tabelle I

| Tag 1941 | lfd. Vers Nr. | N.W. mm | Rohrlänge m | Sicherung | Lufttemp. °C. | Barom mm Hg | Fülldruck atü | Stromstärke Amp. | Druckanstieg atü | Quarz atü | Druckmeßapparat atü: an der Zündstelle | Druckmeßapparat atü: entgegengesetzt zur Zündstelle | Meßeier (atü): an der Zündstelle Nr. 13 | Nr. 17 | Nr. 21 | Meßeier (atü): entgegengesetzt zur Zündstelle Nr. 26 | Nr. 28 | Nr. 30 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 12.5. | 1 | 100 | 30 | Röhrenbündel V2ᵃ | + 6,5° | 759 | 1,0 | - | - | - | - | - | - | - | - | - | - | - | Kein Durchschlag |
| " | 2 | " | " | " | " | " | 2,0 | - | - | - | - | - | - | - | - | - | - | - | " " |
| " | 3 | " | " | " | 2,5 | " | 3,0 | 24,5 | 3 | - | - | - | - | - | - | - | - | - | " " |
| 13.5. | 4 | " | " | " | 3 | " | 4,0 | 23,5 | 4,8 | - | 29 | (120) | - | - | - | - | . | 70 | Durchschlag |
| " | 5 | " | " | " | 7 | " | 5,0 | " | 6 | - | 61 | (238) | 55 | 43 | 39 | 96 | 98 | 96 | " |
| 14.5. | 6 | " | " | " | 6 | 760 | 6,0 | 24 | 7 | - | 61 | (273) | 64 | 61 | 58 | 136 | 103 | 111 | " |
| " | 7 | " | " | " | 4 | " | 3,8 | 23,5 | - | - | - | - | - | - | - | - | 41 | 22 | " |
| " | 8 | " | " | " | 9 | " | 3,6 | 24 | 3,8 | - | 24 | 27 | 27 | 29 | 17 | 19 | 19 | 23 | " |
| " | 9 | " | " | " | 10 | " | 3,5 | " | 3,5 | - | - | - | - | - | - | - | - | - | Zündung am entgegengesetzten Rohrende. Durchschlag. |
| 13.5. | 10 | " | " | " | 8,5 | 759 | 3,4 | " | 3,4 | - | - | - | - | - | - | - | - | - | Kein Durchschlag |
| 15.5 | 11 | " | " | " | 5,5 | - | 3,4 | 23,5 | 3,4 | - | - | - | - | - | - | - | - | - | Zündung am entgegengesetzten Rohrende. (Durchschlag nach etwa 2 Minuten) |
| " | 12 | " | " | " | " | - | 3,3 | 21 | 3,2 | - | - | - | - | - | - | - | - | - | langsamer Zerfall im ganzen Rohr. |
| | 13 | " | " | " | 8 | - | 3,2 | 24 | 3,1 | - | - | - | - | - | - | - | - | - | " |
| 13.5. | 14 | V2ᵃ | 60 | - | 17 | 761 | 5,1 | 21,5 | - | - | - | - | - | - | - | - | - | - | Zerfall kommt erst nach 5 Minuten [illegible] |
| " | 15 | " | " | - | 19 | " | 5,1 | 20 | - | - | - | - | - | - | - | - | - | - | Zerfall kommt schon im Rohranfang zum stehen. |
| " | 16 | " | " | - | 20 | " | 5,2 | 21,5 | - | - | - | - | - | - | - | - | - | - | " |
| 16.5 | 17 | " | " | - | 16 | 758 | 6,8 | 20 | - | - | - | - | - | - | - | - | - | - | " |
| " | 18 | " | " | - | 18 | " | 8,0 | 19,5 | - | - | - | - | - | - | - | - | - | (158) | Durchschlag; ohne Knall |
| " | 19 | " | " | - | 19 | " | 7,5 | 21 | - | - | - | - | - | - | - | - | - | (200) | " ; Zischen. |
| 26.5. | 20 | 100 | 30 | Röhrenbündel V2ᵃ | 16 | 747 | 5,0 | 45! | 5,8 | - | 63 | (400) | - | - | - | - | - | - | Durchschlag |
| " | 21 | " | " | " | 19 | " | 4,0 | 24 | 4,3 | 76 | 60 | (239) | 24 | 17 | - | 105 | 82 | 88 | " |
| " | 22 | " | " | " | 21 | 740 | 6,0 | 24 | 7,5 | 120 | 76 | (400) | 60 | 57 | - | 195 | 140 | 160 | " |
| " | 23 | " | " | " | 16 | " | 5,0 | 24 | - | 95 | 59 | (235) | 62 | 46 | - | 83 | 95 | 76 | " |

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Tabelle I

| 19.. | lfd. Vers Nr. | N.W. mm | Rohrlänge m | Sicherung | Lufttemp. °C. | Barom. mm Hg. | Fülldruck atü | Stromstärke Amp. | Druckanstieg atü | Quarz atü | Druckmeßapparat atü: an der Zündstelle | Druckmeßapparat atü: entgegengesetzt zur Zündstelle | Meßeier (atü) an der Zündstelle Nr. 13 | Nr. 17 | Nr. 21 | Meßeier (atü) entgegengesetzt zur Zündstelle Nr. 26 | Nr. 28 | Nr. 30 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 12[illegible]3. | 1 | 100 | 30 | Röhrenbündel 1/2" | + 6,5° | 759 | 1,0 | - | - | - | - | - | - | - | - | - | - | - | Kein Durchschlag |
| | 2 | " | " | " | " | " | 2,0 | - | - | - | - | - | - | - | - | - | - | - | " " |
| | 3 | " | " | " | 2,5 | " | 3,0 | 24,5 | 3 | - | - | - | - | - | - | - | - | - | " " |
| 1[illegible]3. | 4 | " | " | " | 3 | " | 4,0 | 23,5 | 4,8 | - | 29 | (120) | - | - | - | - | - | 70 | Durchschlag |
| | 5 | " | " | " | 7 | " | 5,0 | " | 6 | - | 61 | (238) | 55 | 43 | 39 | 96 | 98 | 96 | " |
| 1[illegible]3. | 6 | " | " | " | 6 | 760 | 6,0 | 24 | 7 | - | 61 | (273) | 64 | 61 | 58 | 136 | 103 | 111 | " |
| | 7 | " | " | " | 4 | " | 3,8 | 23,5 | - | - | - | - | - | - | - | - | 41 | 22 | " |
| | 8 | " | " | " | 9 | " | 3,6 | 24 | 3,8 | - | 24 | 27 | 27 | 29 | 17 | 19 | 19 | 23 | " |
| | 9 | " | " | " | 10 | " | 3,5 | " | 3,5 | - | - | - | - | - | - | - | - | - | Zündung am entgegengesetzten Rohrende. Durchschlag |
| 1[illegible]3. | 10 | " | " | " | 8,5 | 759 | 3,4 | " | 3,4 | - | - | - | - | - | - | - | - | - | Kein Durchschlag |
| 1[illegible]3. | 11 | " | " | " | 5,5 | - | 3,4 | 23,5 | 3,4 | - | - | - | - | - | - | - | - | - | Zündung am entgegengesetzten Rohrende. Durchschlag nach etwa 2 Minuten! |
| | 12 | " | " | " | " | - | 3,3 | 21 | 3,2 | - | - | - | - | - | - | - | - | - | langsamer Zerfall im ganzen Rohr. |
| | 13 | " | " | " | 8 | - | 3,2 | 24 | 3,1 | - | - | - | - | - | - | - | - | - | " |
| | 14 | 1/2" | 60 | - | 17 | 761 | 5,1 | 21,5 | - | - | - | - | - | - | - | - | - | - | Zerfall kommt erst nach 5 Minuten ans Rohrende, dieses nur handwarm. |
| | 15 | " | " | - | 19 | " | 5,1 | 20 | - | - | - | - | - | - | - | - | - | - | Zerfall kommt schon im Rohranfang zum stehen. |
| | 16 | " | " | - | 20 | " | 5,2 | 21,5 | - | - | - | - | - | - | - | - | - | - | " |
| [illegible]5. | 17 | " | " | - | 14 | 758 | 6,8 | 20 | - | - | - | - | - | - | - | - | - | - | " |
| | 18 | " | " | - | 18 | " | 8,0 | 19,5 | - | - | - | - | - | - | - | - | - | (158) | Durchschlag; ohne Knall |
| | 19 | " | " | - | 19 | " | 7,5 | 21 | - | - | - | - | - | - | - | - | - | (200) | " ; Zischen. |
| [illegible]5. | 20 | 100 | 30 | Röhrenbündel 1/2" | 16 | 747 | 5,0 | 45! | 5,8 | - | 63 | (400) | - | - | - | - | - | - | Durchschlag |
| | 21 | " | " | " | 19 | " | 4,0 | 24 | 4,3 | 76 | 60 | (239) | 24 | 17 | - | 105 | 82 | 88 | " |
| | 22 | " | " | " | 21 | 748 | 6,0 | 24 | 7,5 | 120 | 76 | (400) | 60 | 57 | - | 195 | 140 | 160 | " |
| | 23 | " | " | " | 16 | " | 5,0 | 24 | - | 95 | 59 | (235) | 62 | 46 | - | 83 | 95 | 76 | " |

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| Tag 1941 | lfd. Vers. Nr. | N.W. mm | Rohrlänge m | Sicherung | Lufttemp. °C. | Barom. mmHg. | Fülldruck atü | Stromstärke Amp. | Druckanstieg atü | Quarz atü | Druckmeßapparat atü an der Zündstelle | Druckmeßapparat atü entgegengesetzt zur Zündstelle | Meßeier (atü) an der Zündstelle Nr. 13 | Meßeier (atü) an der Zündstelle Nr. 17 | Meßeier (atü) an der Zündstelle Nr. 21 | Meßeier (atü) entgegengesetzt zur Zündstelle Nr. 26 | Meßeier (atü) entgegengesetzt zur Zündstelle Nr. 28 | Meßeier (atü) entgegengesetzt zur Zündstelle Nr. 30 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 27. | 24 | 10 | 60 | Schlangenanordnung | 16 | 748 | 10 | 20 | 12,6 | - | - | - | - | - | - | - | - | (191) | Durchgehender Zerf |
| bis | 25 | " | " | " | 17 | " | 9 | 20 | 10,5 | - | - | - | - | - | - | - | - | (162) | " |
| 31.5. | 26 | " | " | " | 21 | " | 8,5 | 20 | 9,8 | - | - | - | - | - | - | - | - | (176) | " |
| " | 27 | " | " | " im Wasserbad | 20 | 747 | 10,1 | 20 | 11,5 | - | - | - | - | - | - | - | - | (218) | " |
| " | 28 | " | " | " | " | " | 8,5 | 22 | 9 | - | - | - | - | - | - | - | - | (153) | " |
| " | 29 | 1/2" | " | Schlangenanordnung | 21 | " | 7,5 | 20 | 9 | - | - | - | - | - | - | - | - | (218) | " |
| " | 30 | " | " | " | 13 | 748 | 7,0 | 35! | 8 | - | - | - | - | - | - | - | - | (181) | " |
| 8.5. | 31 | " | 30 | " mit Vorkammer 200Ø | 16 | 761 | 3,2 | 21 | - | - | - | - | - | - | - | - | - | (152) | " |
| 12.5. | 32 | " | 60 | " | 17 | " | 5,1 | 22 | - | - | - | - | - | - | - | - | - | (143) | " |

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I. Vorsuche in Griesheim (120er Rohre, waagerecht)

Tabelle II

| Tag | lfd.Nr. | Nennweite mm | Sicherung | Luft-temp. °C | Barom. mm | Fülldruck atü | Druck-anstieg atü | Strom-stärke Amp. | Druckmeßzylinder atü an der Zündstelle | Druckmeßzylinder atü entgegengesetzt zur Zündstelle | Meßeier atü Nr. 26 | Meßeier atü Nr. 28 | Meßeier atü Nr. 30 | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 5.11.41 | 1 | 120 | Röhrenbündel | +2,5 | 755 | 7,0 | über 11 | 24 | 94 | 85 | - | - | - | Durchschlag |
| " | 2 | (30 m lang) | 50 Rohre $\frac{10}{8}$ ⌀ 1.W. | +2,4 | " | 8,0 | " | 25 | 78 | 153 | - | - | - | " |
| 6.11.41 | 3 | " | Vorkammer 30cm lang gef.m.Raschigringen | +2,3 | 757 | 9,0 | " | " | 80 | nicht meßbar | +195 | +178 | +234 | Oszillogr. 258 atü |
| " | 4 | " | " | +4,5 | " | 10,0 | " | " | 94 | " " | Ø223 | Ø187,5 | Ø175 | " 250 " |
| 10.11.41 | 5 | " | Wie oben aber 6 cm Vork. ohne Raschigringe | +2,5 | 747 | 9,0 | " | 24,5 | 96 | " " | Ø180 | Ø170 | Ø170 | Durchschlag |
| 7.11.41 | 6 | " | Raschigringe, Eisen 35 X 35 mm | + 6 | 750 | 0,8 | - | - | kein Eindruck | kein Eindruck | - | - | - | kein Durchschlag |
| " | 7 | " | " | + 8 | " | 1,0 | - | - | " | " | - | - | - | " " |
| " | 8 | " | " | + 8 | " | 1,4 | 2,5 | 25 | " | 21 | - | - | - | Schwacher Durchschlag unverbranntes $C_2H_2$ |
| 8.11.41 | 9 | " | " | + 4 | 754 | 2,0 | 2,3 | 25 | " | 47 | 25 | 38 | 35 | Durchschlag |
| " | 10 | " | " | + 4 | 754 | 3,0 | 3,2 | 24,5 | 23 | kein Eindruck | - | - | - | kein Durchschlag |
| 10.11.41 | 11 | " | " | -1,5 | 747 | 4,0 | 5,9 | " | 57 | 105 | 47 | 47 | 83 | Durchschlag |
| " | 12 | " | " | +2,5 | " | 6,0 | 9,4 | " | 74 | 320? | 303? | 81 | 85 | " |
| 27.11.41 | 13 | " | Raschigringe aus Ton 25 X 25 mm | + 5 | 761 | 3,0 | 3,3 | 25 | kein Eindruck | kein Eindruck | - | - | - | kein Durchschlag |
| " | 14 | " | "(außen) | + 5 | " | 5,0 | 5,5 | 25 | 28 | " | - | - | - | " " |
| 28.11.41 | 15 | " | " | +3,5 | " | 6,0 | 6,8 | 25 | - | " | - | - | - | " " |
| 9.12.41 | 16 | " | "(keine Füllung) | + 2 | 751 | 7,0 | 10,2 | 20 | 71 | nicht meßbar | 147 | 95 | 103 | Durchschlag |
| " | 17 | " | " | + 3 | " | 8,0 | 10 | 20 | 76 | " | 268 | 230 | 275 | " |

ohne Zeichen und: + Kugeldruck
Ø Stauchzylinder

Tabelle III

## II. Ludwigshafener Versuche (Senkrechte Rohre; 10; 12,5 und 16 mm l.W.)

| Tag | lfd.Nr. | Nennweite mm | Rohrlänge | Lufttemp. °C | Barom. mm | Fülldruck atü | Stromstärke Amp. | Kugeldruck-Meßapparat atü unten (Zündstelle) | Kugeldruck-Meßapparat atü oben (7m vom Ende) | Drucksteigerung (Oszillograph) atü | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 4.11.41 | 1 | 10,0 | 30 | + 2,0 | 753 | 5,0 | 25 | - | - | - | kein Durchschlag; Flamme erlischt etwa nach 1/2 m. |
| " | 2 | " | " | + 2,0 | 757 | 5,0 | " | - | - | - | " " " " " " " " |
| " | 3 | " | " | + 3,0 | 757 | 6,0 | " | - | - | - | " " " " " in 1/4 Rohrlänge |
| 12.11. | 4 | " | " | + 7,0 | 752 | 7,0 | " | - | - | - | Sehr schwacher Durchschlag; Oszillograph nicht auswertbar! |
| " | 5 | " | " | + 8,0 | 752 | 9,0 | " | - | - | - | Schwacher Durchschlag. |
| " | 6 | " | " | + 8,5 | 752 | 11,0 | " | 280? | 350? | Störung! | Durchschlag |
| " | 7 | " | " | + 8,0 | 752 | 12,0 | " | 350? | 350? | " | " |
| " | 8 | " | " | + 8,0 | 752 | 13,0 | " | 350? | 350? | " | " |
| 20.11. | 9 | " | " | +11,0 | 753 | 9,0 | " | - | - | 133 | " |
| " | 10 | " | " | +10,2 | 753 | 10,0 | " | - | - | 160 | " |
| 19.11. | 11 | " | " | + 5,3 | 755 | 11,0 | " | - | - | 185 | " |
| " | 12 | " | " | + 5,4 | 755 | 12,0 | " | - | - | 225 | " |
| 20.11. | 13 | " | " | +10,0 | 753 | 13,0 | " | - | - | 215 | " |
| " | 14 | " | " | +10,0 | 753 | 14,0 | " | - | - | 327 | " |
| " | 15 | " | " | +10,0 | 753 | 15,0 | " | - | - | 278 | " |
| 14.11. | 16 | 12,5 | 30 | + 4,5 | 755 | 5,0 | 25 | 59 | 200 | - | Durchschlag |
| " | 17 | " | " | + 4,0 | " | 6,0 | " | 76 | 295 | 81 | " |
| " | 18 | " | " | " | " | 7,0 | " | 76 | 305 | 80 | " |
| " | 19 | " | " | " | " | 8,0 | " | 102 | 260 | 97 | " |
| 15.11. | 20 | " | " | + 0,8 | 750 | 9,0 | " | 265? | 280? | 117 | " |
| " | 21 | " | " | " | " | 10,0 | " | - | - | 173 | " |
| 17.11. | 22 | " | " | +11,4 | 754 | 5,0 | " | 57 | 207 | 67 | " |
| " | 23 | " | " | + 9,0 | 753 | 6,0 | " | 74 | 230 | 79 | " |
| " | 24 | " | " | " | 753 | 8.0 | " | 74 | 287 | - | " |
| " | 25 | " | " | +11,4 | 754 | 8,0 | " | 120 | 280 | 117 | " |
| 21.11. | 26 | " | " | + 2,5 | 756 | 5,0 | " | - | - | 80 | " |
| " | 27 | " | " | " | " | 6,0 | " | | | 107 | " |
| " | 28 | " | " | + 6,5 | " | 7,0 | " | | | 123 | " |

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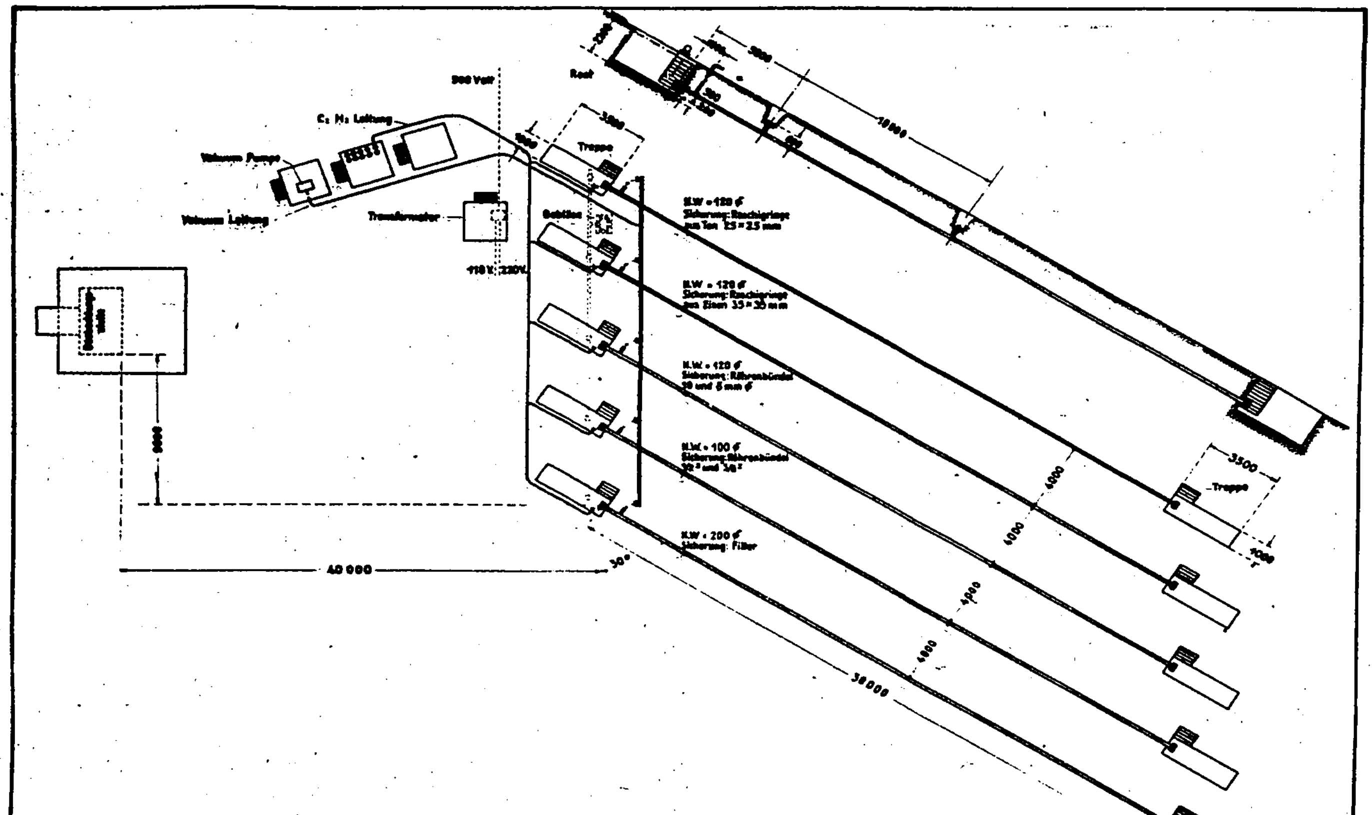
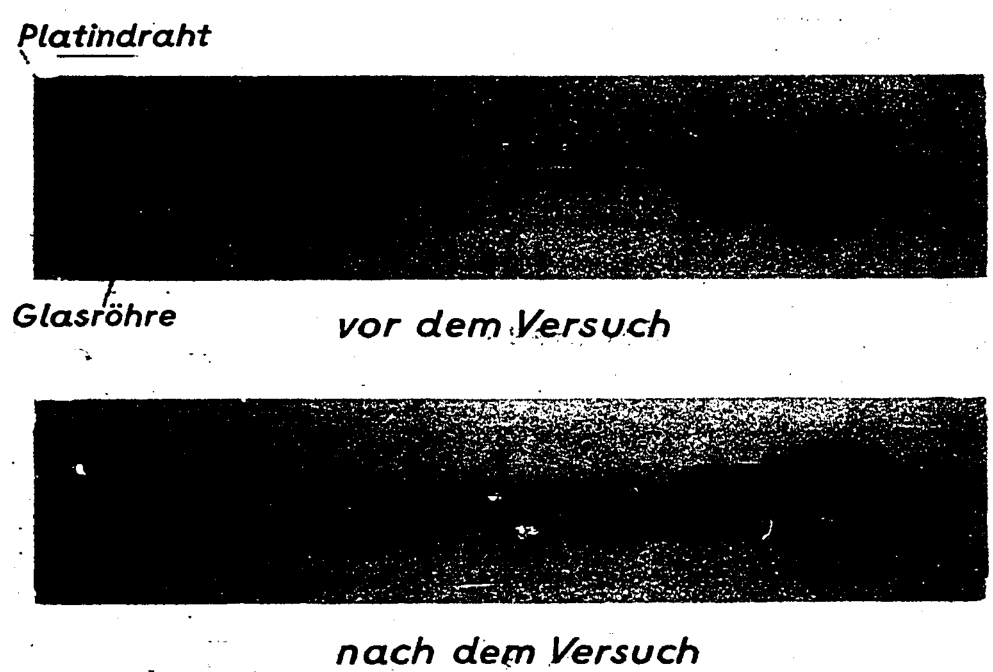
## II. Ludwigshafener Versuche (Senkrechte Rohre; 10; 12,5 und 16 mm l.W.) Forts.

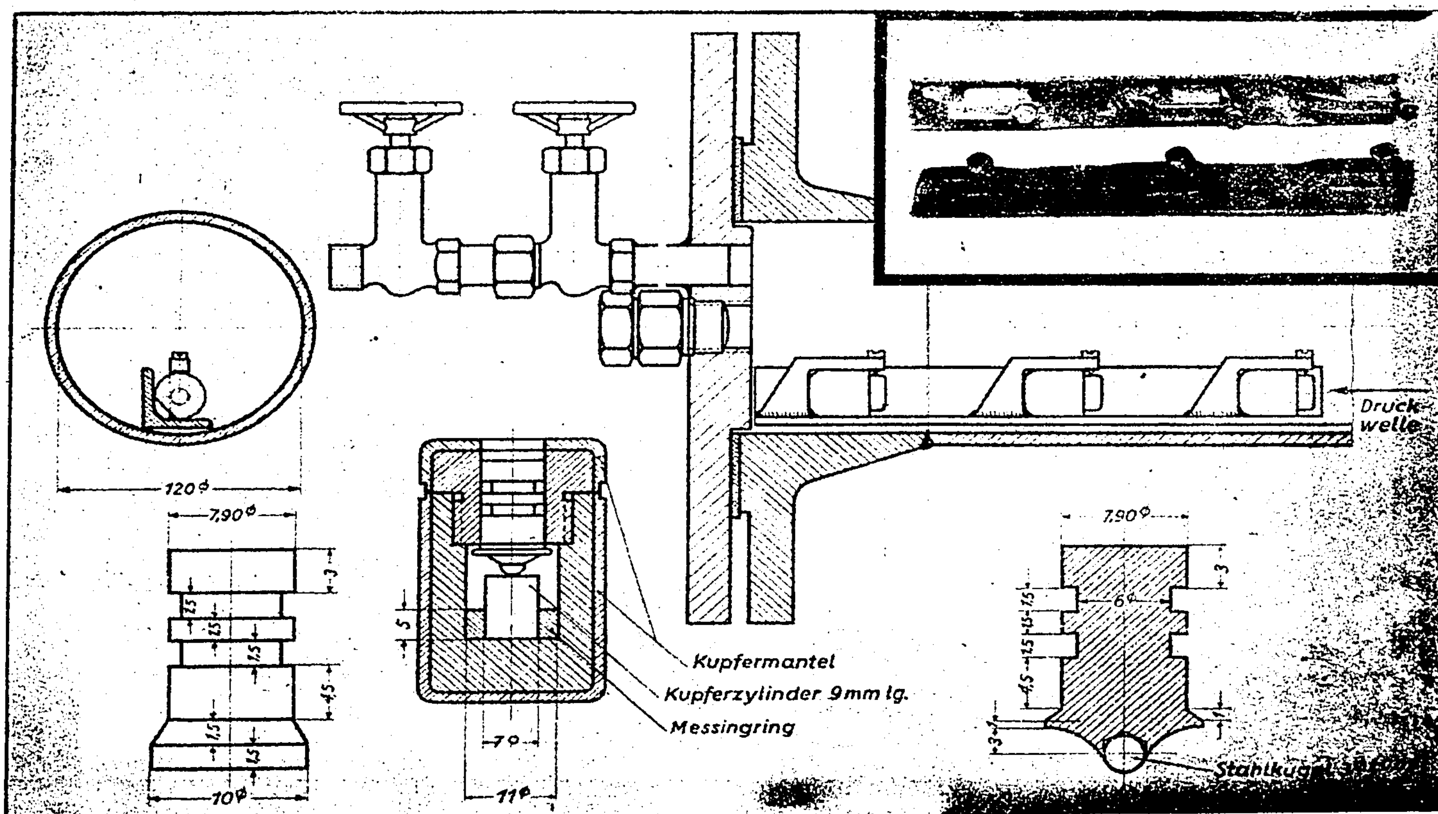
| Tag | lfd.Nr. | Nennweite mm | Rohrlänge m | Lufttemp. °C | Barom. mm | Fülldruck atü | Stromstärke Amp. | Kugeldruck-Meßapparat atü unten (Zündestelle) | Kugeldruck-Meßapparat atü oben (7m vom Ende) | Drucksteigerung (Oszillograph) atü | Bemerkungen |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1.11.41 | 29 | 12,5 | 30 | + 6,8 | 756 | 8,0 | 25 | - | - | 140 | Durchschlag |
| " | 30 | " | " | + 7 2 | " | 9,0 | " | - | - | 155 | " |
| " | 31 | " | " | " | " | 10,0 | " | - | - | 199 | " |
| 2.12.41 | 32 | 16,0 | 30 | - 2,2 | 768 | 5,0 | 25 | - | - | 20 | Durchschlag |
| " | 33 | " | " | - 1 | " | 6,0 | " | - | - | 125 | " |
| " | 34 | " | " | + 1,3 | " | 7,0 | " | - | - | 130 | " |
| " | 35 | " | " | + 1,0 | " | 8,0 | " | - | - | 160 | " |
| " | 36 | " | " | " | " | 9,0 | " | - | - | 174 | " |
| " | 37 | " | " | + 1,6 | " | 10,0 | " | - | - | 277 | Wegen sehr steiler Wellenfront Messung unsicher. |
| 2.12.41 | 38 | " | " | + 1,6 | 769 | 11,0 | " | - | - | 256 | " |
| " | 39 | " | " | + 1,6 | " | 12,0 | " | - | - | 315 | " ; Versuche abgebrochen. |
| **Versuche mit Vorkammer ( Ø = 70 mm; l = 500 mm; V = 1,9 Liter)** | | | | | | | | | | | |
| 3.12.41 | 40 | 10,0 | 30 | + 4,6 | 764 | 5,0 | 25 | | 180 | 144 | Durchschlag |
| " | 41 | " | " | + 4,8 | " | 6,0 | " | | 224 | 185 | " |
| " | 42 | " | " | + 5,0 | " | 7,0 | " | | 257 | - | " |
| " | 43 | " | " | + 6,0 | " | 8,0 | " | | 317 | 220 | " |
| " | 44 | " | " | + 6,0 | " | 10,0 | " | | nicht auswertbar | 275 | " |



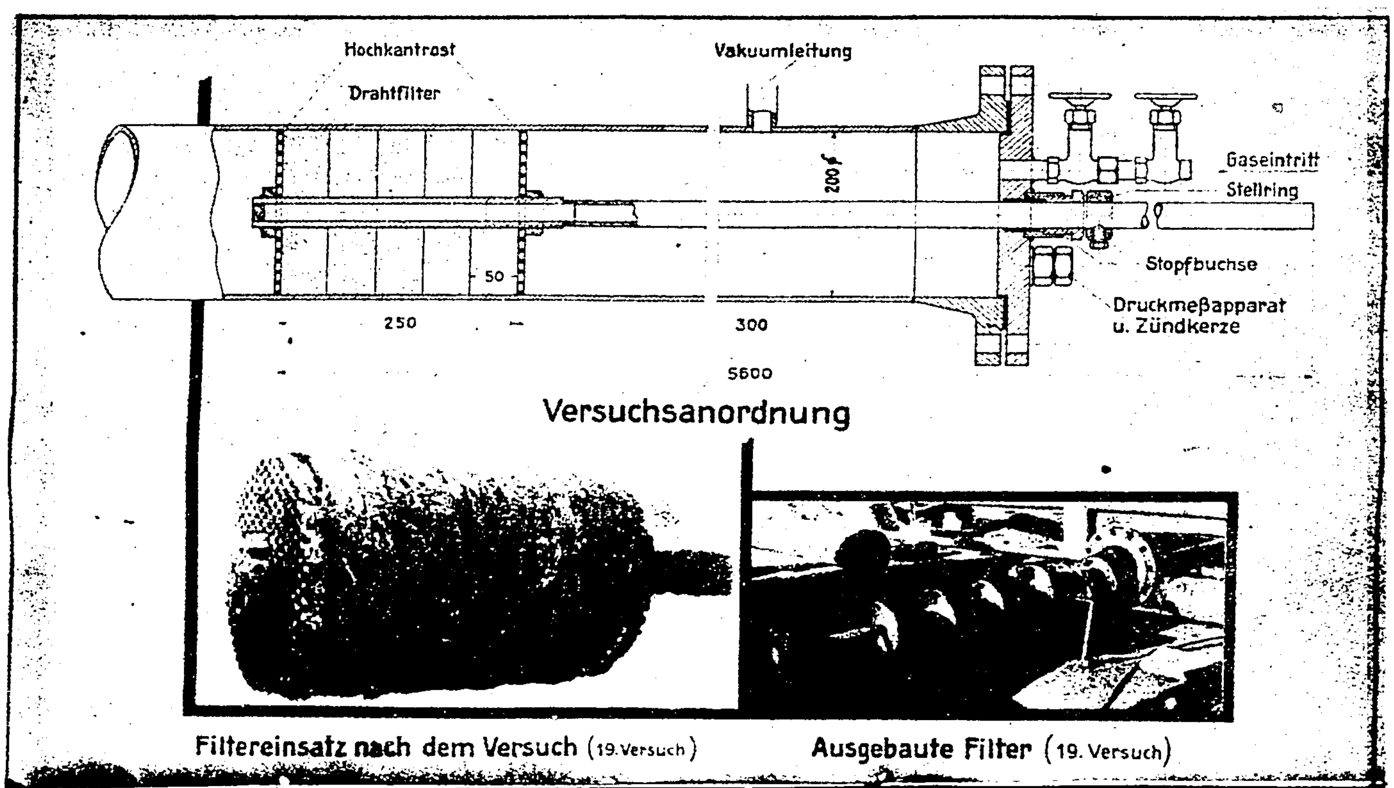

Griesheimer Versuchsanlage
( Anordnung der Versuchsrohre )




**Zündkerzen.**




# Anordnung der Krupp'schen Meßeier

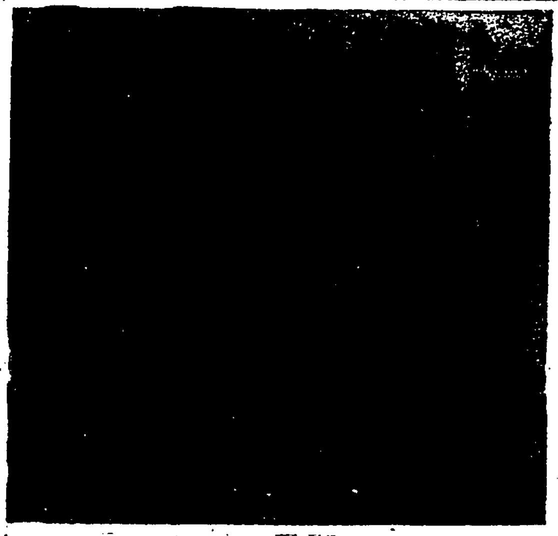


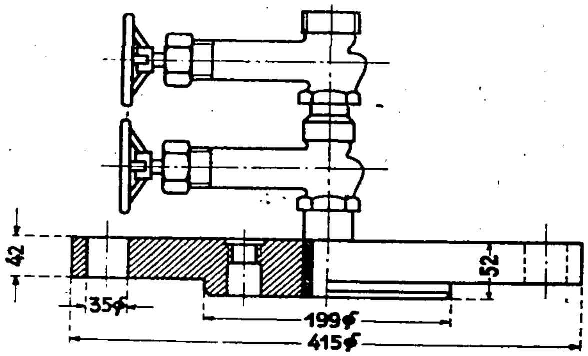

Versuchsanordnung

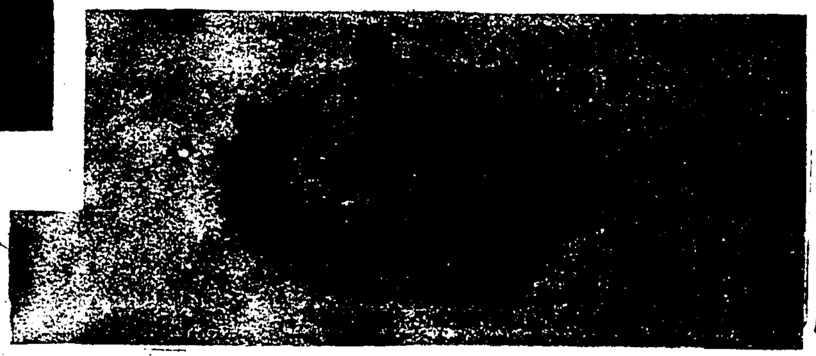
Filtereinsatz nach dem Versuch (19. Versuch)

Ausgebaute Filter (19. Versuch)

Versuchsleitung von 200 mm⌀ mit eingesetzten Drahtfiltern (Bauart Griesheim)

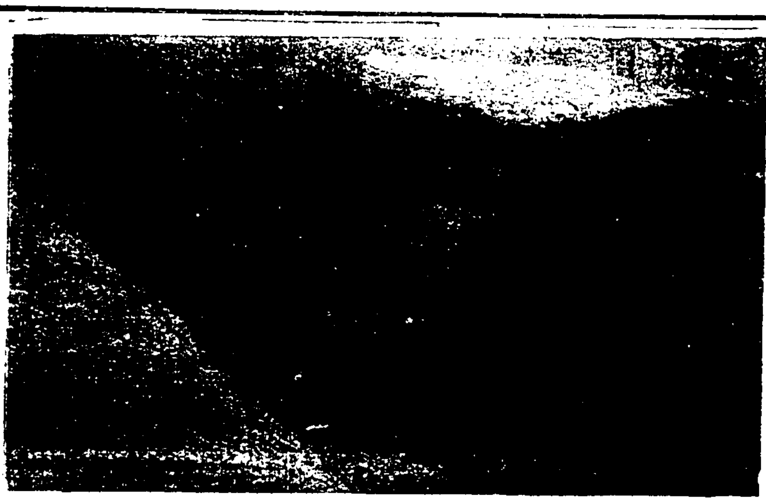


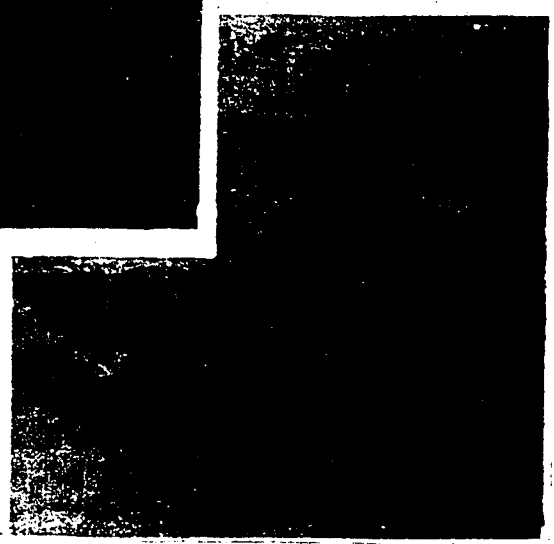


Rohrdurchmesser: 200 mm NW.
Fülldruck: 4,0 atü.

**Abschlußdeckel d. Versuchsrohres nach der Detonation beim 21. Versuch**

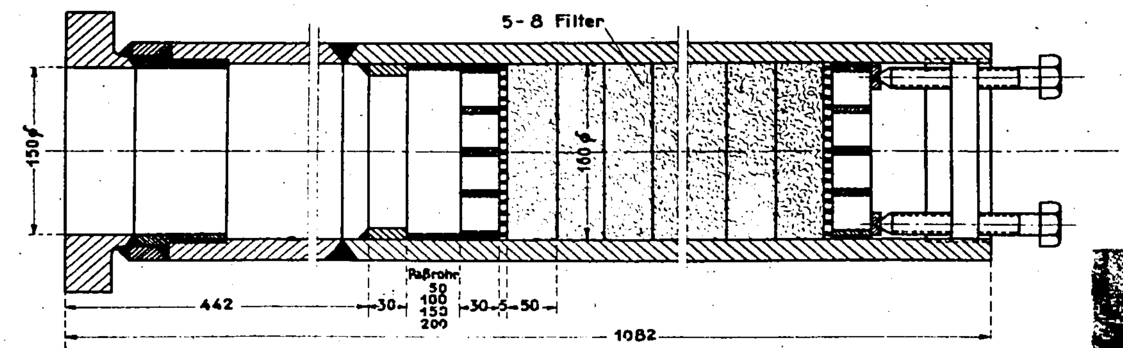




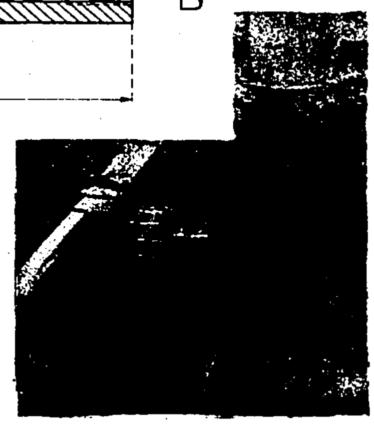
Versuchsanordnung:

Rohrdurchmesser: 200 mm NW
Sicherung: Drahtfiltereinsätze (Bauart Ludwigshafen) 8 Drahtfilter
Fülldruck: 4,0 atü

Rohrdetonation beim 21. Versuch




Einsteckvorrichtung



Versuchsrohr mit Einsteckvorrichtung

Ausgebaute Drahtfilter nach dem Versuch

## Versuche mit Drahtfiltereinsätzen (Bauart Ludwigshafen)

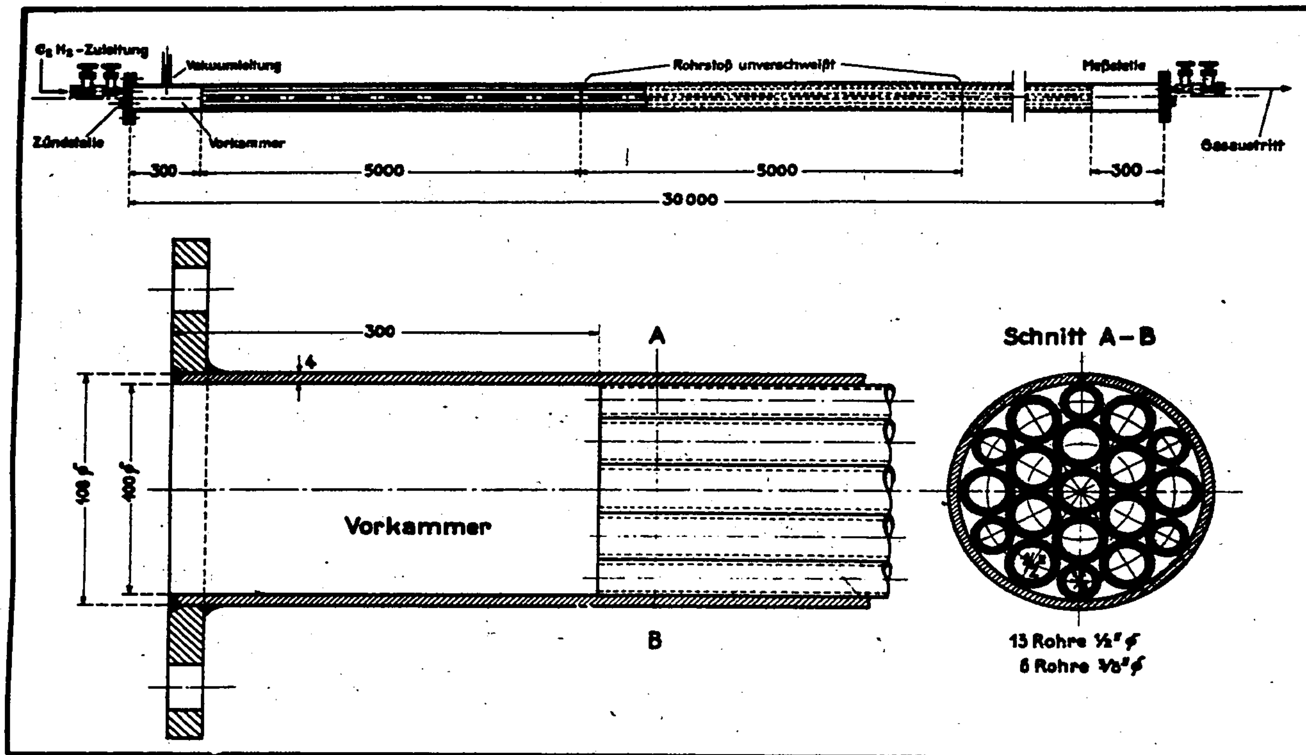
**Versuchsanordnung:**

Rohrdurchmesser: 200 mm NW
Fülldruck: 4,0 atü
Sicherung: 5 Drahtfilter
(Anordnung Griesheim)

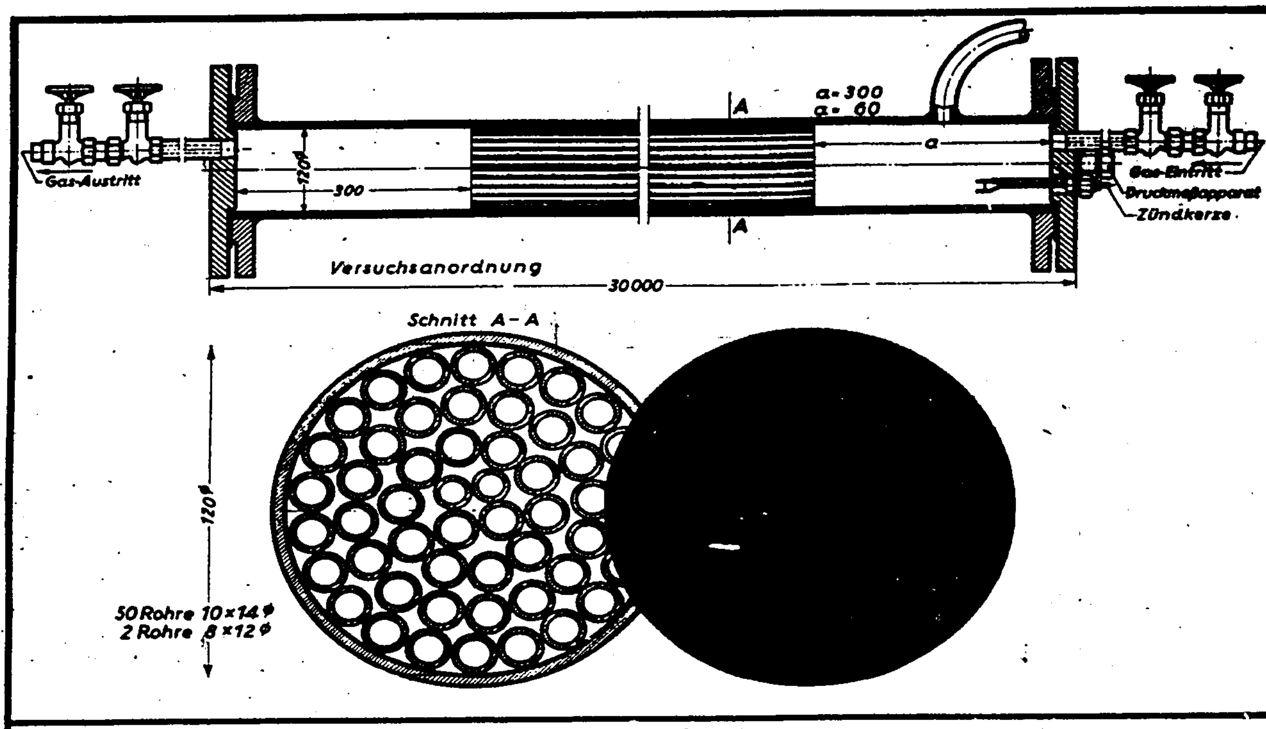
**Rohrende zerstört**

Abschlußdeckel mit abgerissen Absperrventilen, Bolzen und Teile des Vorschweißflansches.

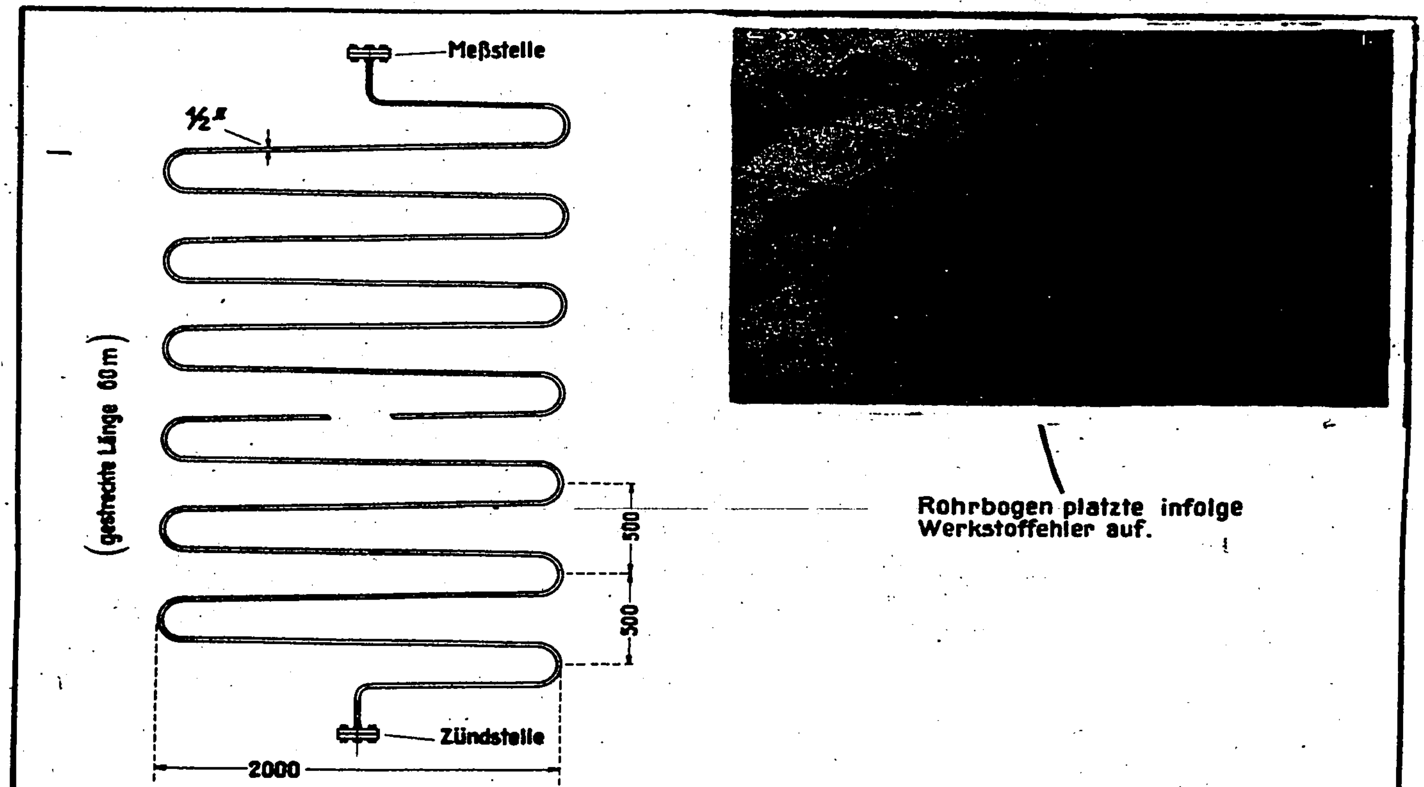
## Rohrdetonation beim 20. Versuch



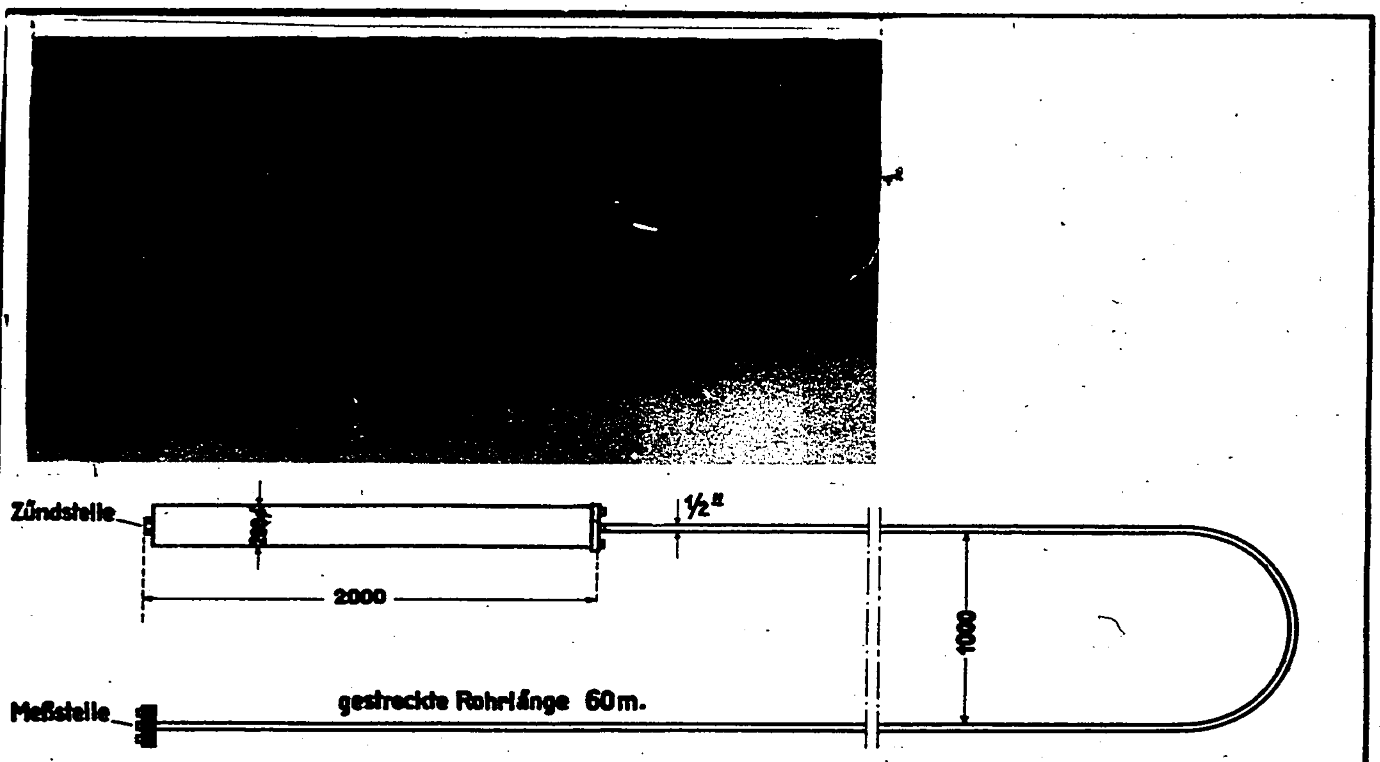

**Versuchsleitung von 100 mm ϕ mit eingezogenem Röhrenbündel**



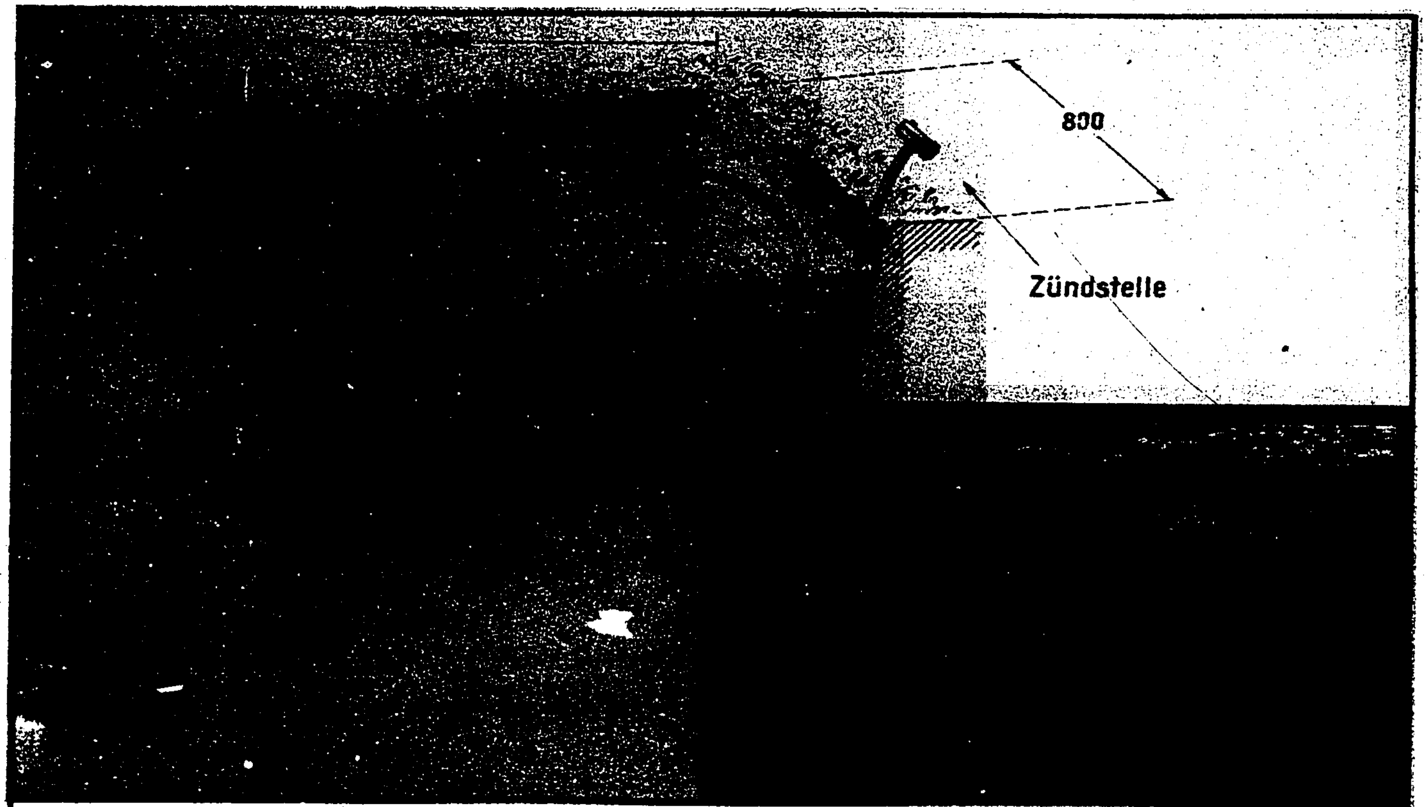

***Versuchsleitung von 120 mm ϕ mit eingezogenem Röhrenbündel.***



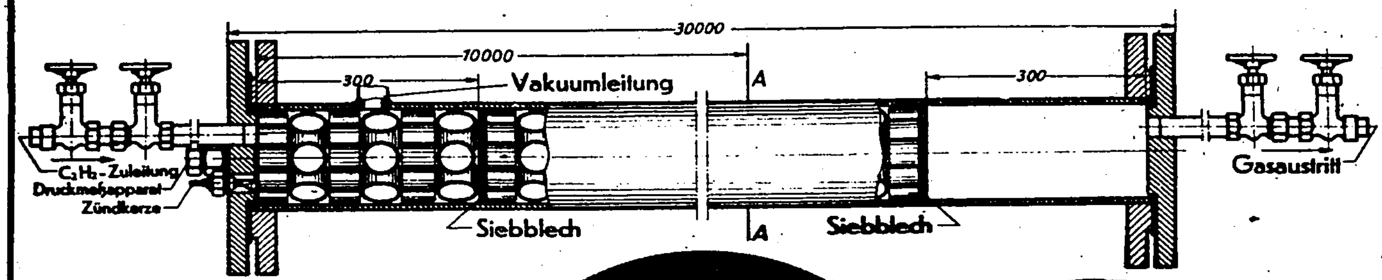

**Versuchsrohre in Schlangenanordnung**
NW = ½" und 10 mm ø

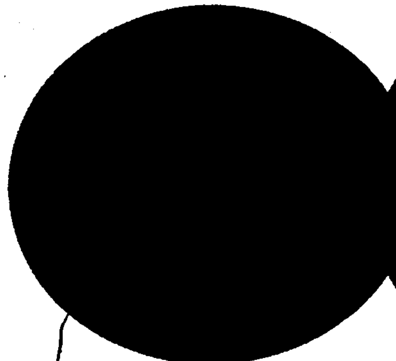



**Versuchsrohr mit vorgesetzter Zündkammer**
NW = ½"

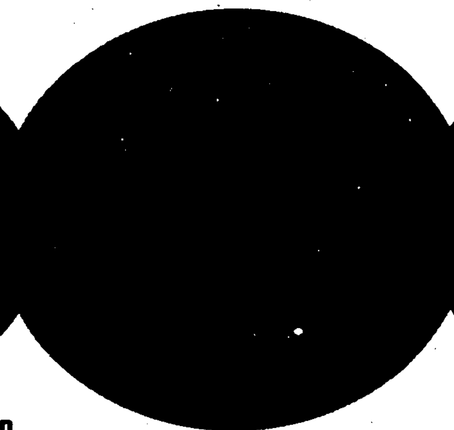



**Versuchsrohre von 10mm N.W. schlangenförmig angeordnet im Wasserbad**

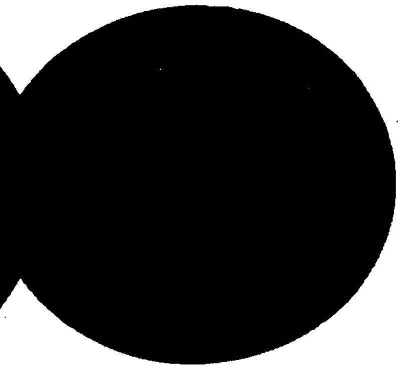


Zündkammer mit Raschigringen (Eisen)

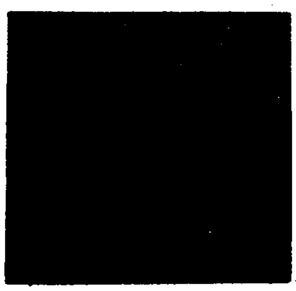


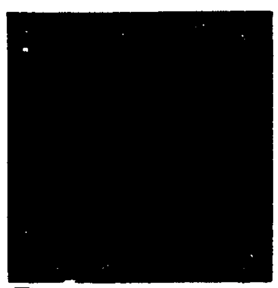
Zündkammer mit Raschigringen (Ton) nach dem Versuch mit 6,0 atü Fülldruck



Schnitt A—A
Rohrleitung nach sieben Versuchen an der Stelle „A" aufgeschnitten

Versuchsleitung von 120 mm Ø mit eingebrachten Raschigringen

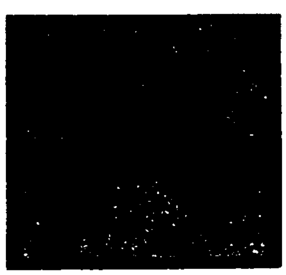




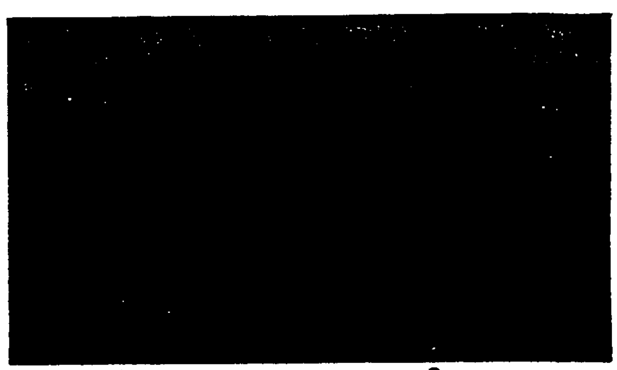
unbenutzte Raschigringe aus Eisen

Raschigringe (Eisen) nach sieben Versuchen

unbenutzte Raschigringe aus Ton



Raschigringe (Ton) nach dem ersten Versuch



Reste von Raschigringen (Ton), die nach mehrmaligen Versuchen aus den Rohrleitungen entfernt wurden

EXHIBIT D

PRINTS OBTAINED AT HUELS

The following prints were obtained and will be available at the office of the Rubber Subcommittee, J.I.O.A. in Washington.

1. Mengen pro Monat, Betrieb der Lichtbogen Mit Hy Gasen.
This is a block flow sheet showing a material balance for the entire plant and indicating the number of operating units.

2. Ofengaswäsche Hä 442; Nr. 2
Flow sheet showing schematic lay-out of water scrubbing system and subsequent flashing to obtain concentrated acetylene. Included are equipment sizes and performance requirements of pumps; gas flows and pressures.

3. Tiefkühlanlage; Nr. 3
Flow sheet showing low temperature refrigeration system for removing higher homologues from acetylene stream. Gives equipment sizes, flows, etc.

4. Lichtbogenanlage - Russfabrik - N.D.G.R.; Nr. 1
Flow sheet showing arc operation, carbon black production, and low pressure gas purification.

5. Oelkreislanf der N.D.G.R. bei Verwendung von Kogasin als Waschoel Nr. 1a.
Flow sheet showing low pressure purification when using special oil.

6. Ölwäsche Hä 442; Nr. 2a
Flow sheet showing high pressure oil scrubbing.

7. Koksofengas - Wäsche Hä 442; Nr. 2b
Flow sheet showing removal of acetylene from coke oven gas.

8. Gaskompression in Bau 442; Hilfsmachinen; Nr. 2d
Tabulation of number and performances of auxiliary compressors.

9. Fluss - Schema von Gaszerlegung; Nr. 4
Flow sheet of Linde gas separating plant.

10. Hä 460, Gesamtschema der Wasserstoffboxen 1-5; Nr. 4a

11. Gaskompression in Bau 460; Hilfsmaschinen; Nr. 4d.
List of auxiliary compressors with performance characteristics.

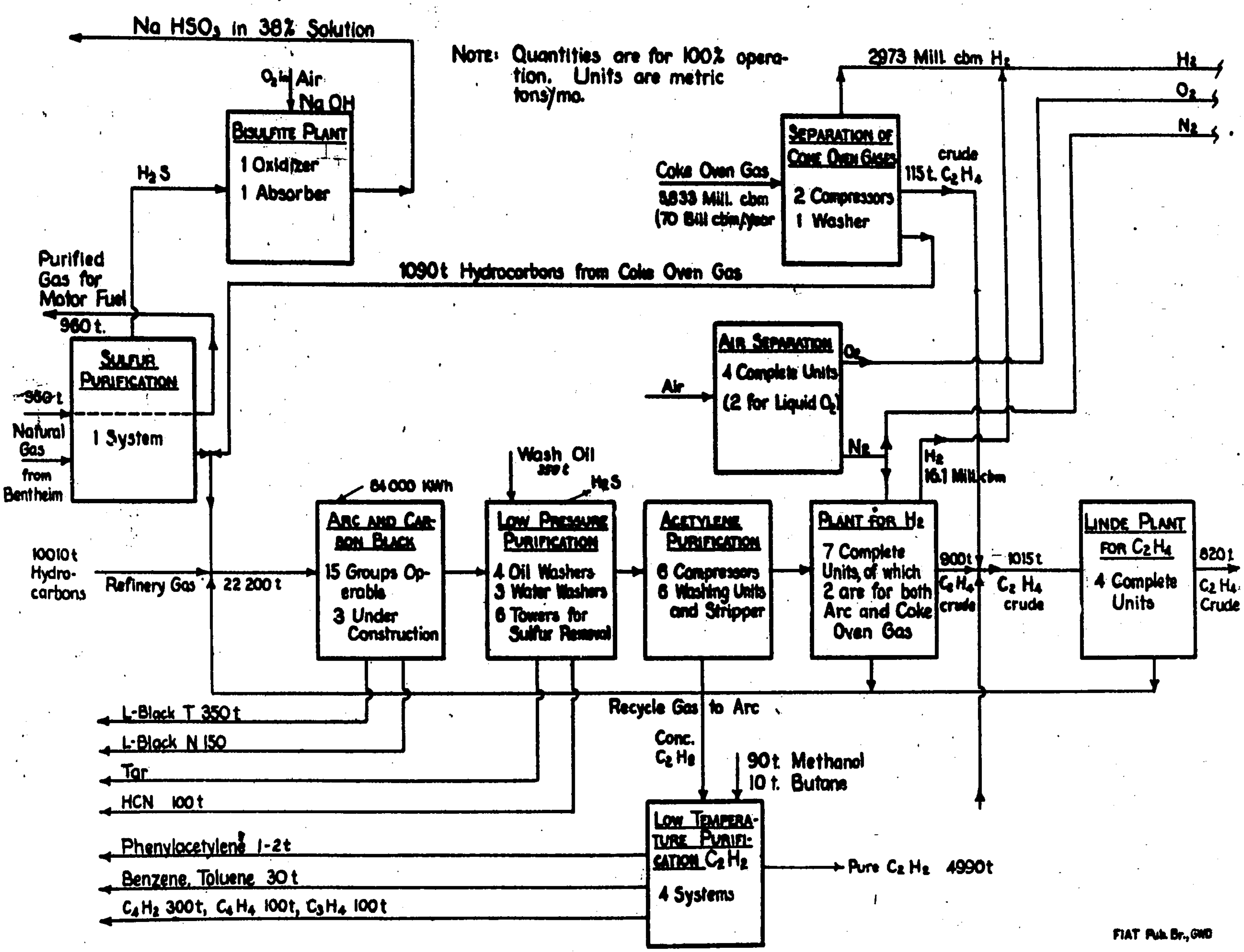
12. Schema; Athylen - Anlage Huels; Nr. 4b.
Flow sheet of ethylene refrigerating machine.

13. Schema; Linde - Anlage; Nr. 4c
Flow sheet Linde Liquefaction plant.

14. Aldehyde Synthesis; Distillation; Acetylen Ruckgewinnung; Aceton Distillation; Kontaktregeneration. Nr. 5.
Flow sheet of acetaldehyde manufacture.

Part of D-1

# MATERIAL BALANCE OF $C_2H_2$ MANUFACTURE & PURIFICATION - HÜLS



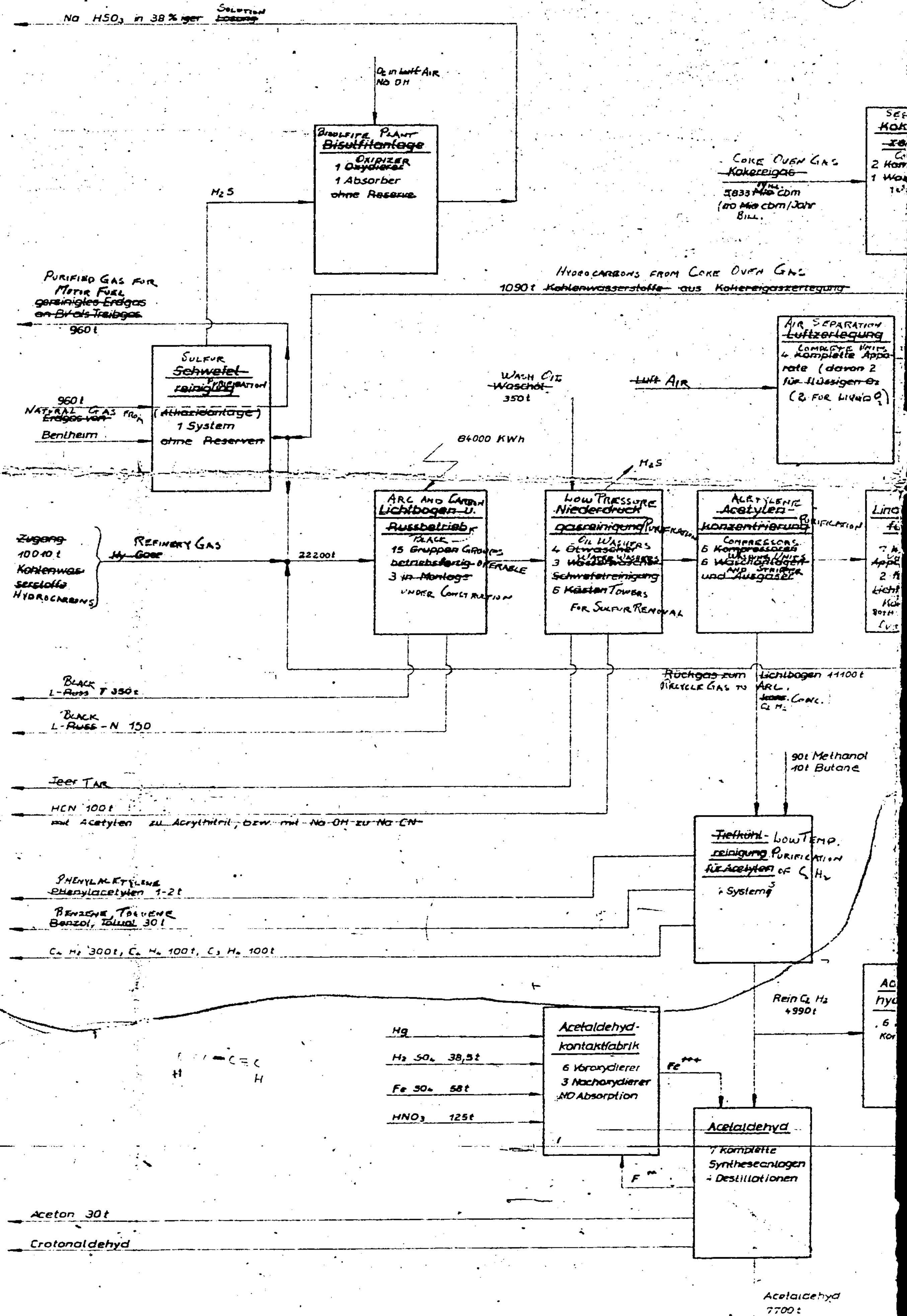
Bau Nr. | Betrieb | Gegenstand | Anmerkungen

To ... in ... to ...

# ~~Mengen pro Monat, Betrieb der Lichtbogen mit Hy Gasen~~

MATERIAL BALANCE OF $C_2H_2$ MANUFACTURE AND PURIFICATION - HÜLS

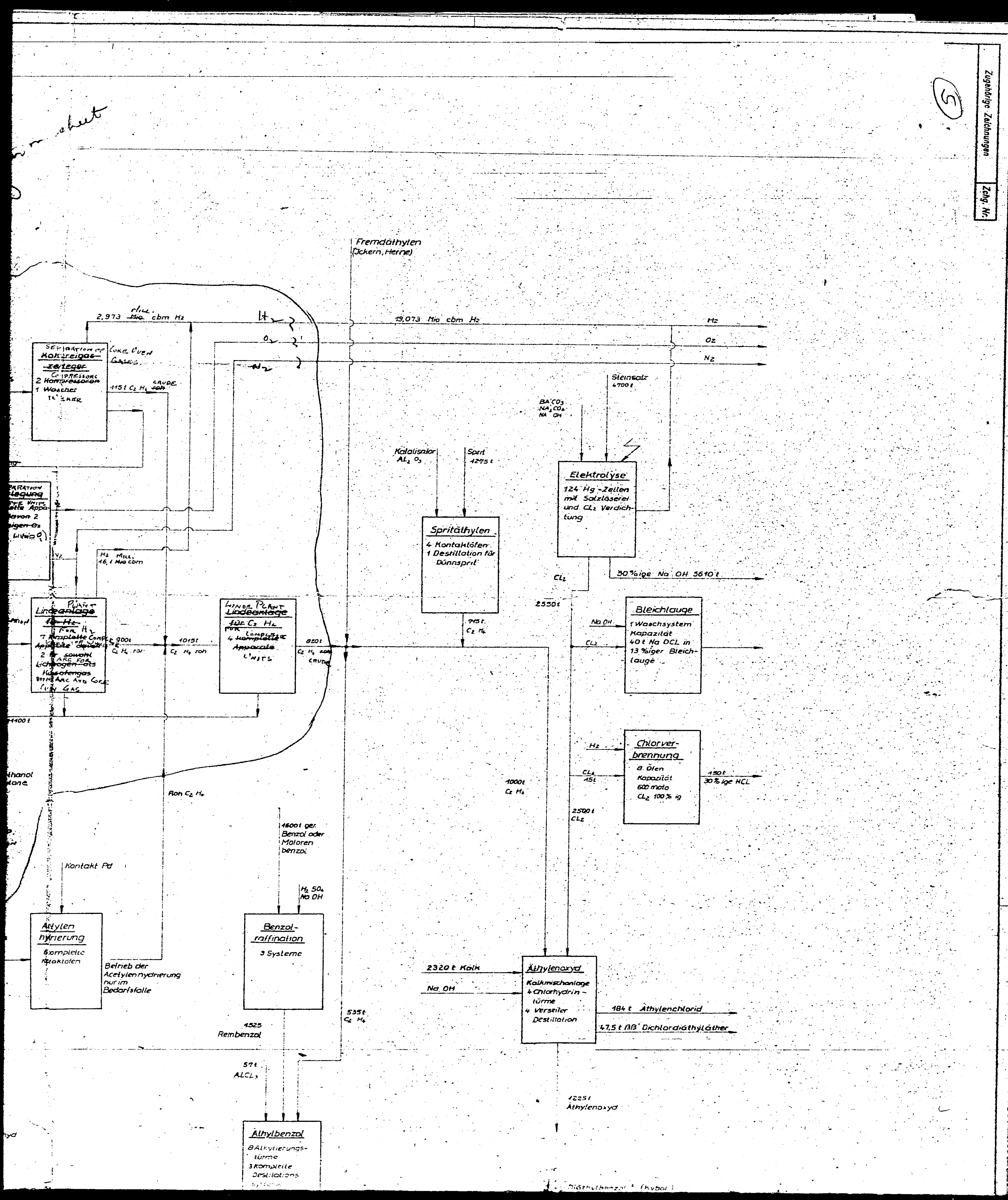
Note:- QUANTITIES ARE FOR 100% OPERATION. UNITS ARE METRIC TONS/MO.




Center ...

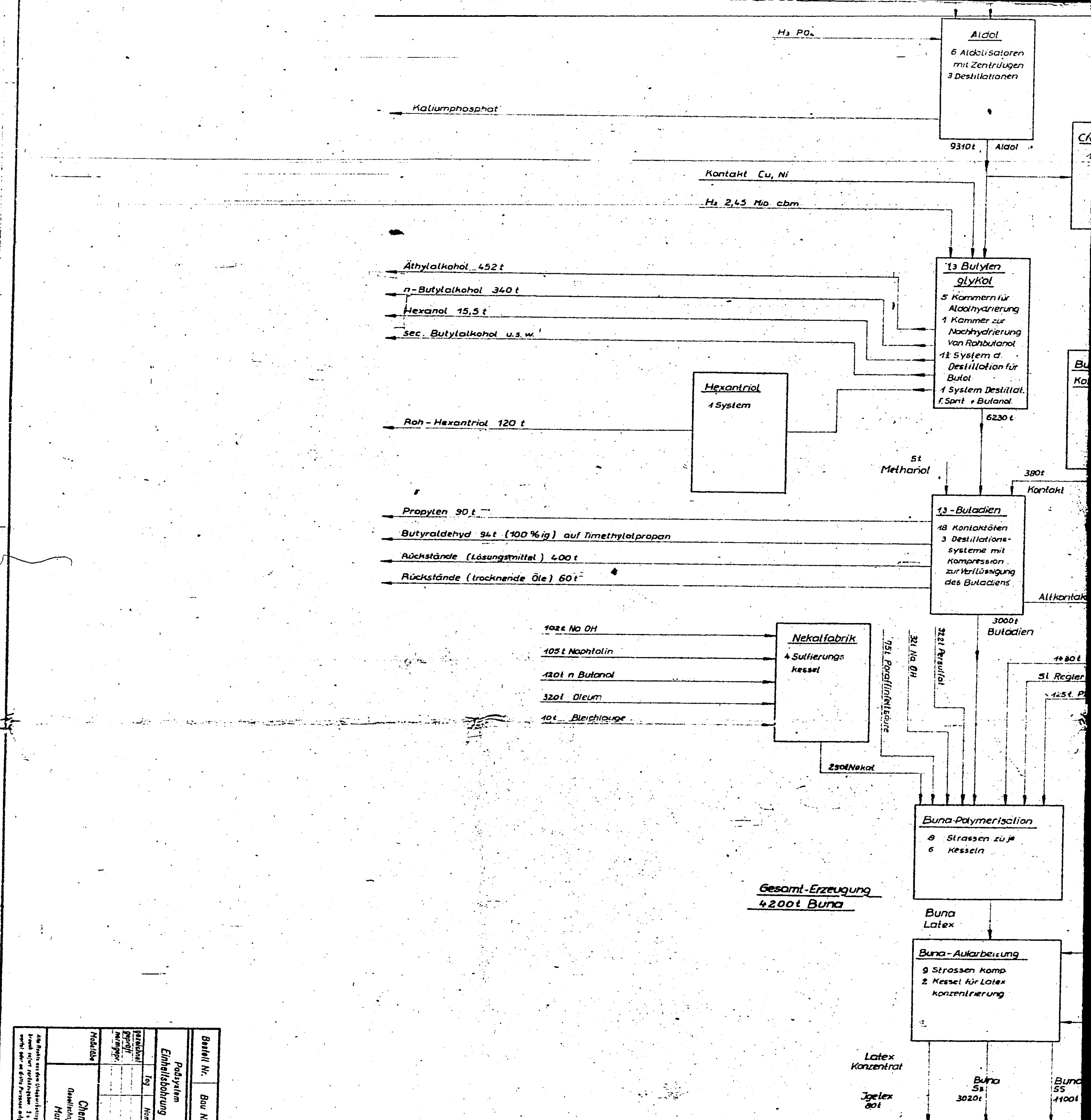
PB 20078

I-11

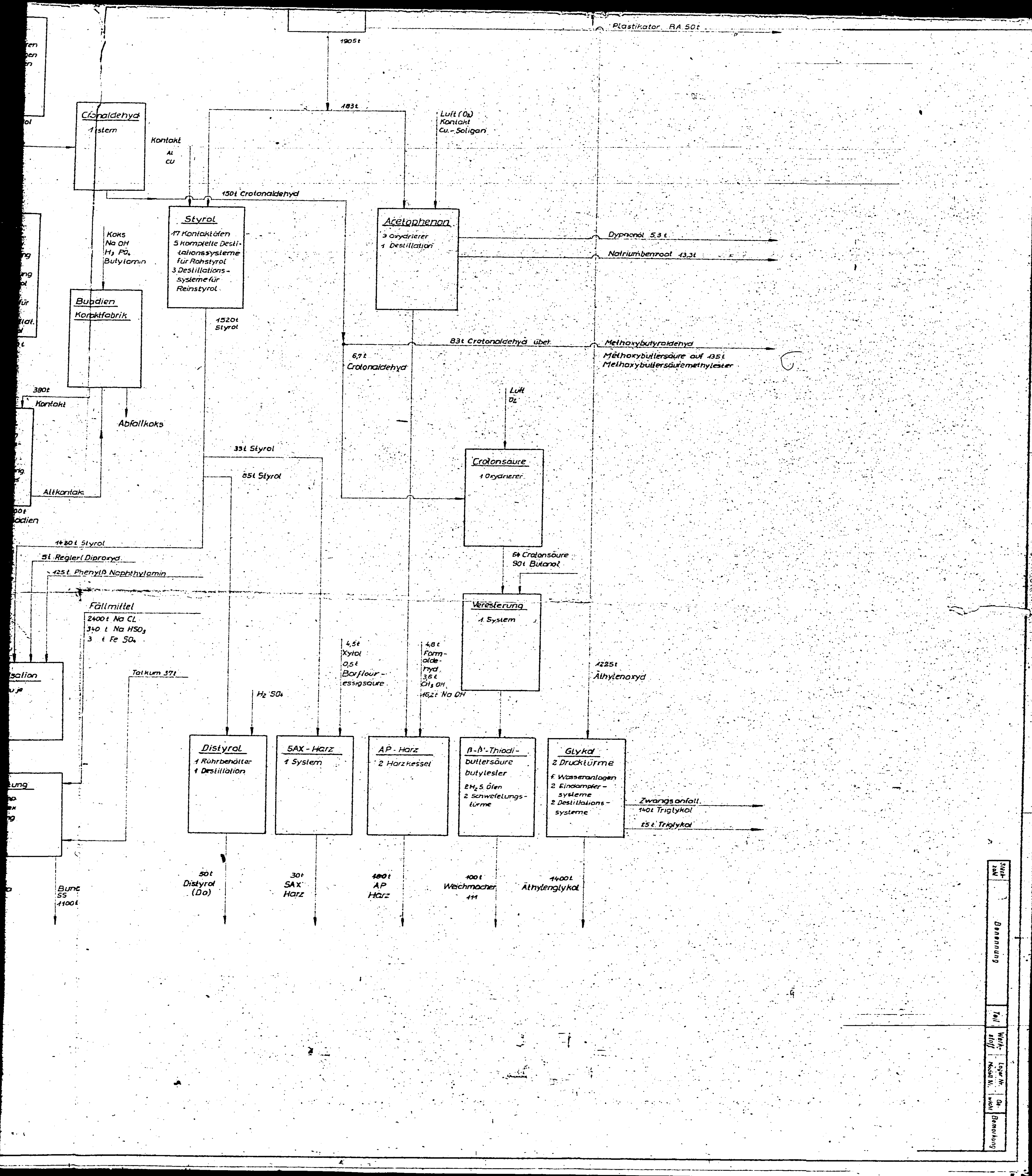


Fremdäthylen
(Ickern, Herne)
2,973 Mio cbm H2
13,073 Mio cbm H2
H2
O2
N2
Kokereigas-zerleger
2 Kompressoren
1 Wascher
1151 t C2 H4
Steinsalz
4700 t
BA CO3
NA2 CO3
NA OH
Katalysator
AL2 O3
Sprit
4275 t
Elektrolyse
124 Hg-Zellen mit Salzlöserei und CL2 Verdichtung
Spritäthylen
4 Kontaktöfen
1 Destillation für Dünnsprit
H2 16,1 Mio cbm
30 %ige Na OH 5610 t
CL2
2550 t
Lindeanlage für H2
Lindeanlage für C2 H4
900 t C2 H4 roh
1015 t C2 H4 roh
820 t C2 H4
915 t C2 H4
Bleichlauge
1 Waschsystem
Kapazität 40 t Na OCL in 13 %iger Bleichlauge
Na OH
CL2
Chlorverbrennung
8 Öfen
Kapazität 600 moto
CL2 100 % ig
H2
CL2 15 t
150 t
30 % ige HCL
1400 t
Roh C2 H4
1000 t C2 H4
2500 t CL2
1600 t ger. Benzol oder Motoren benzol
Kontakt Pd
H2 SO4
Na OH
Äthylenhydrierung
6 komplette Kontaktöfen
Betrieb der Acetylenhydrierung nur im Bedarfsfalle
Benzolraffination
3 Systeme
2320 t Kalk
Na OH
Äthylenoxyd
Kalkmischanlage
4 Chlorhydrintürme
4 Verseifer
Destillation
184 t Äthylenchlorid
47,5 t ßß' Dichlordiäthyläther
1525 Reinbenzol
535 t C2 H4
57 t ALCL3
1225 t Äthylenoxyd
Äthylbenzol
8 Alkylierungstürme
3 komplette Destillations systeme
Diäthylbenzol (Kybol)


Zugehörige Zeichnungen
Zchg. Nr.

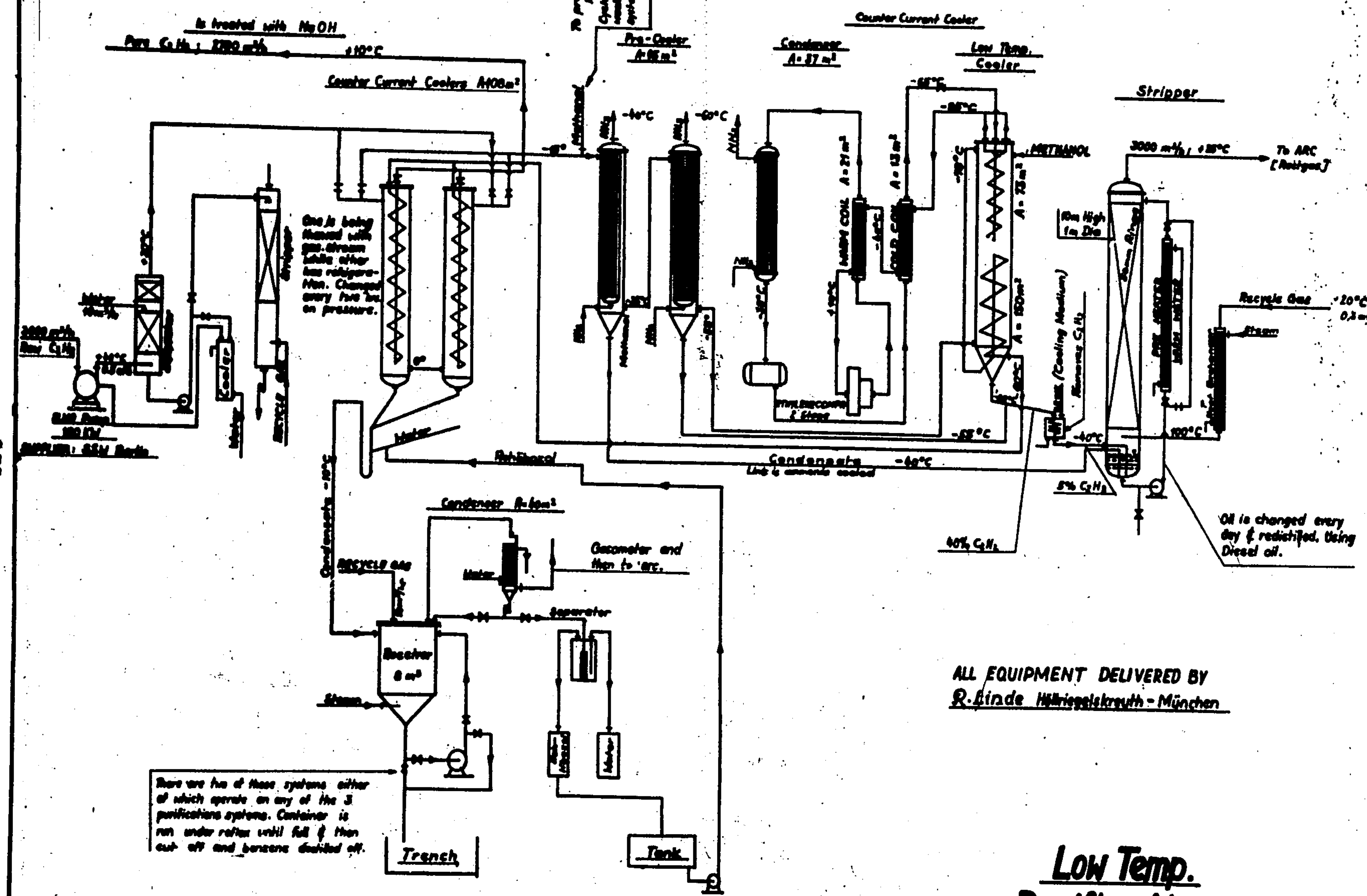


Aldol
6 Aldolisatoren mit Zentrifugen
3 Destillationen
$H_3PO_4$
Kaliumphosphat
9310 t
Aldol
Kontakt Cu, Ni
$H_2$ 2,45 Mio cbm
13 Butylenglykol
5 Kammern für Aldolhydrierung
1 Kammer zur Nachhydrierung von Rohbutanol
1½ System d. Destillation für Butol
1 System Destillat. f. Sprit + Butanol
Äthylalkohol 452 t
n-Butylalkohol 340 t
Hexanol 15,5 t
sec. Butylalkohol u.s.w.
Hexantriol
1 System
Roh-Hexantriol 120 t
6230 t
5 t Methanol
380 t
Kontakt
13-Butadien
18 Kontaktöfen
3 Destillationssysteme mit Kompression zur Verflüssigung des Butadiens
Propylen 90 t
Butyraldehyd 94 t (100%ig) auf Trimethylolpropan
Rückstände (Lösungsmittel) 400 t
Rückstände (trocknende Öle) 60 t
Altkontakt
3000 t Butadien
Nekalfabrik
4 Sulfierungskessel
102 t Na OH
105 t Naphtalin
120 t n Butanol
320 t Oleum
10 t Bleichlauge
250 t Nekal
75 t Paraffinfettsäure
32 t Na OH
32 t Persulfat
1480 t
5 t Regler
Buna-Polymerisation
8 Strassen zu je 6 Kesseln
Gesamt-Erzeugung 4200 t Buna
Buna Latex
Buna-Aufarbeitung
9 Strassen komp.
2 Kessel für Latexkonzentrierung
Latex Konzentrat
Igelex 80 t
Buna S3 3020 t
Buna SS 1100 t
Bestell Nr.
Bau Nr.
Bestellung
Besteller
Jahr
Paßsystem Einheitsbohrung
Betriebsdruck
Probedruck
atü
gezeichnet
geprüft
Tag
Name
Maßstab
Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Kr. Recklinghausen
Ersatz für
Ersatz durch



Plastikator RA 50t
1905t
183t
Crotonaldehyd
1 System
Kontakt
Al
Cu
Luft ($O_2$)
Kontakt
Cu.-Soligan
150t Crotonaldehyd
Styrol
17 Kontaktöfen
5 komplette Destillationssysteme für Rohstyrol
3 Destillations-Systeme für Reinstyrol
Acetophenon
3 Oxydrierer
1 Destillation
Dypnonol 5,3t
Natriumbenzoat 43,3t
Koks
Na OH
$H_3PO_4$
Butylamin
Butadien
Kontaktfabrik
1520t
Styrol
83t Crotonaldehyd über
Methoxybutyraldehyd
Methoxybuttersäure auf 435t
Methoxybuttersäuremethylester
6,7t
Crotonaldehyd
380t
Kontakt
Abfallkoks
Luft
$O_2$
35t Styrol
65t Styrol
Crotonsäure
1 Oxydrierer
Altkontakt
1430t Styrol
5t Regler (Dipr oxyd)
425t Phenyl β Naphthylamin
64 Crotonsäure
90t Butanol
Veresterung
1 System
Fällmittel
2400t Na CL
340 t Na $HSO_3$
3 t Fe $SO_4$
4,5t
Xylol
0,5t
Borflour-essigsäure
4,8t
Formaldehyd
3,6t
$CH_3OH$
46,2t Na OH
1225t
Äthylenoxyd
Talkum 37t
$H_2SO_4$
Distyrol
1 Rührbehälter
1 Destillation
SAX-Harz
1 System
AP-Harz
2 Harzkessel
β-β'-Thiodibuttersäure butylester
2 $H_2S$ Öfen
2 Schwefelungstürme
Glykol
2 Drucktürme
1 Wasseranlagen
2 Eindampfersysteme
2 Destillationssysteme
Zwangsanfall
140t Triglykol
25t Triglykol
50t
Distyrol
(Do)
30t
SAX
Harz
480t
AP
Harz
100t
Weichmacher
411
1400t
Äthylenglykol
Bunc
SS
1100t
Stück-zahl
Benennung
Teil
Werkstoff
Lager-Nr. Modell-Nr.
Gewicht
Bemerkung

D-3

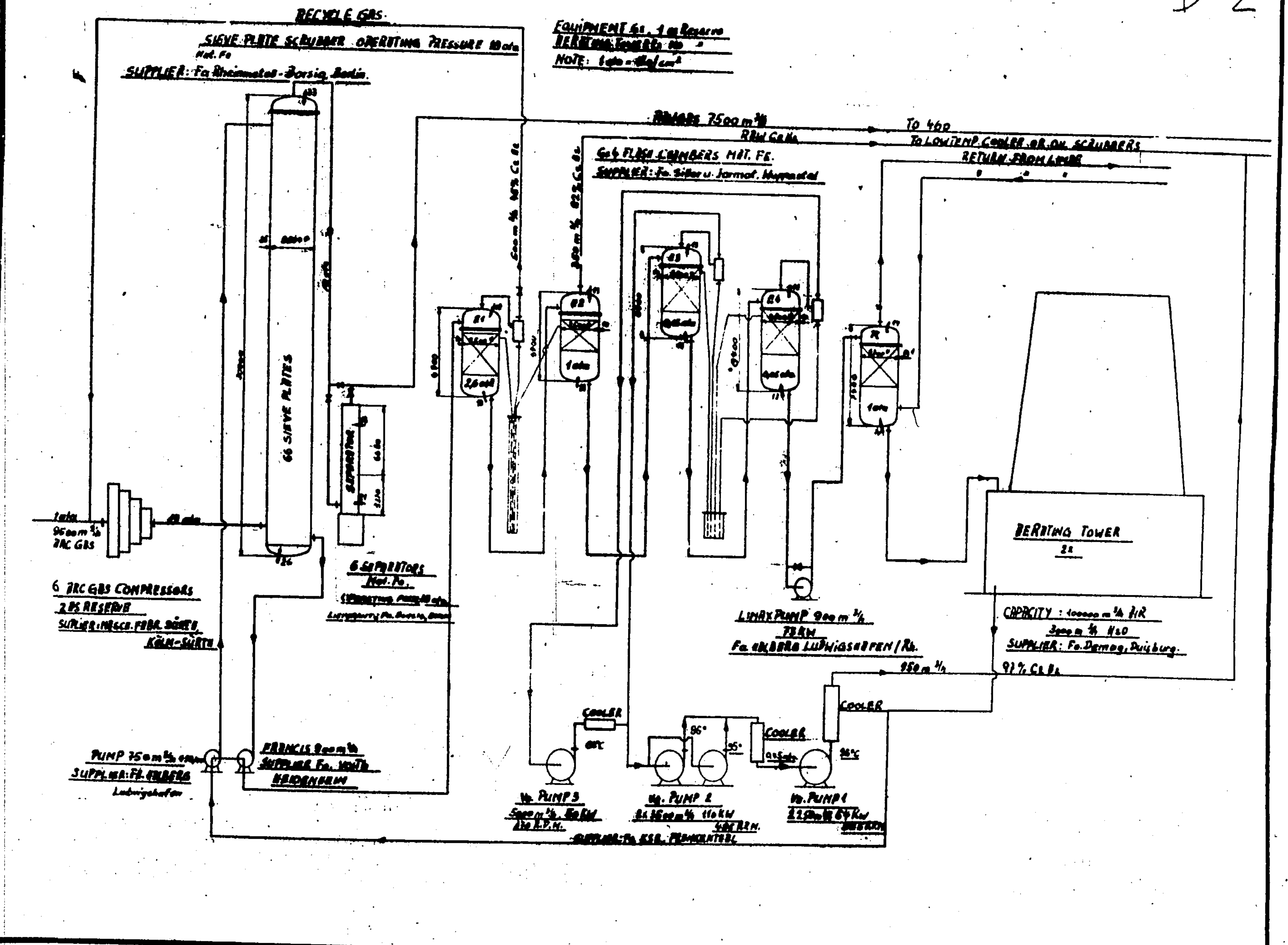


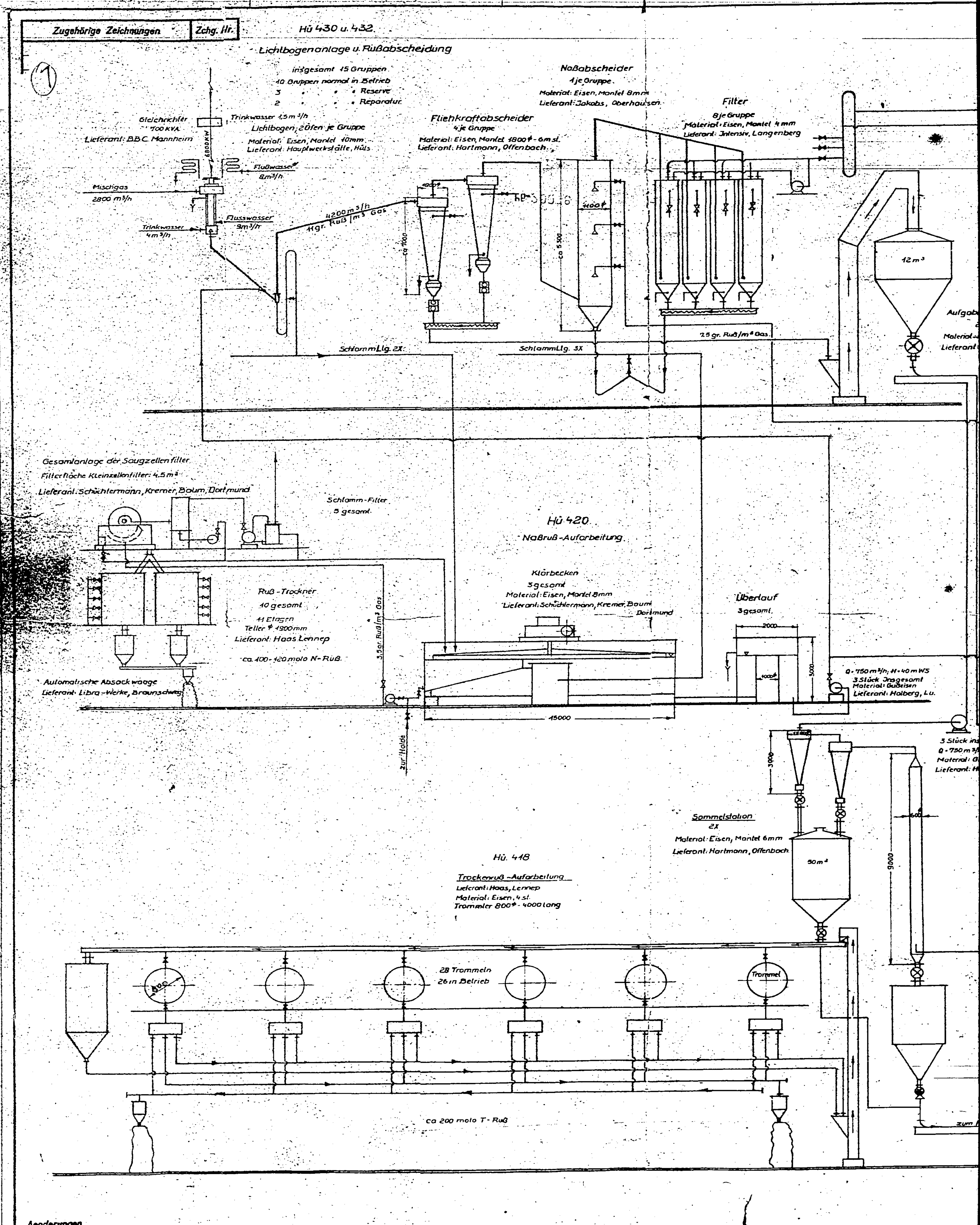

ALL EQUIPMENT DELIVERED BY
R. Linde Höllriegelskreuth-München

## Low Temp. Purification

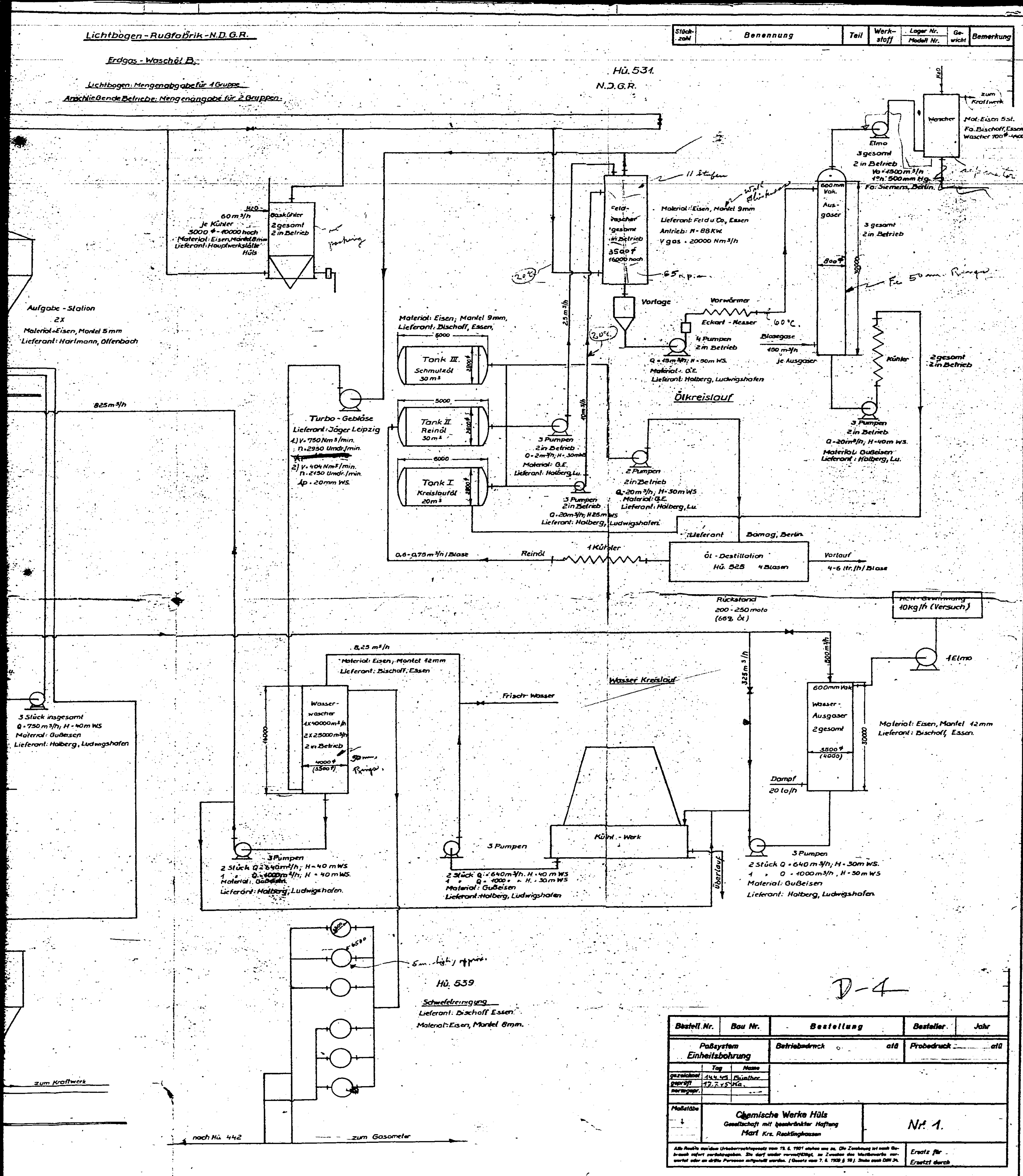
# ARC GAS SCRUBBING

D-2



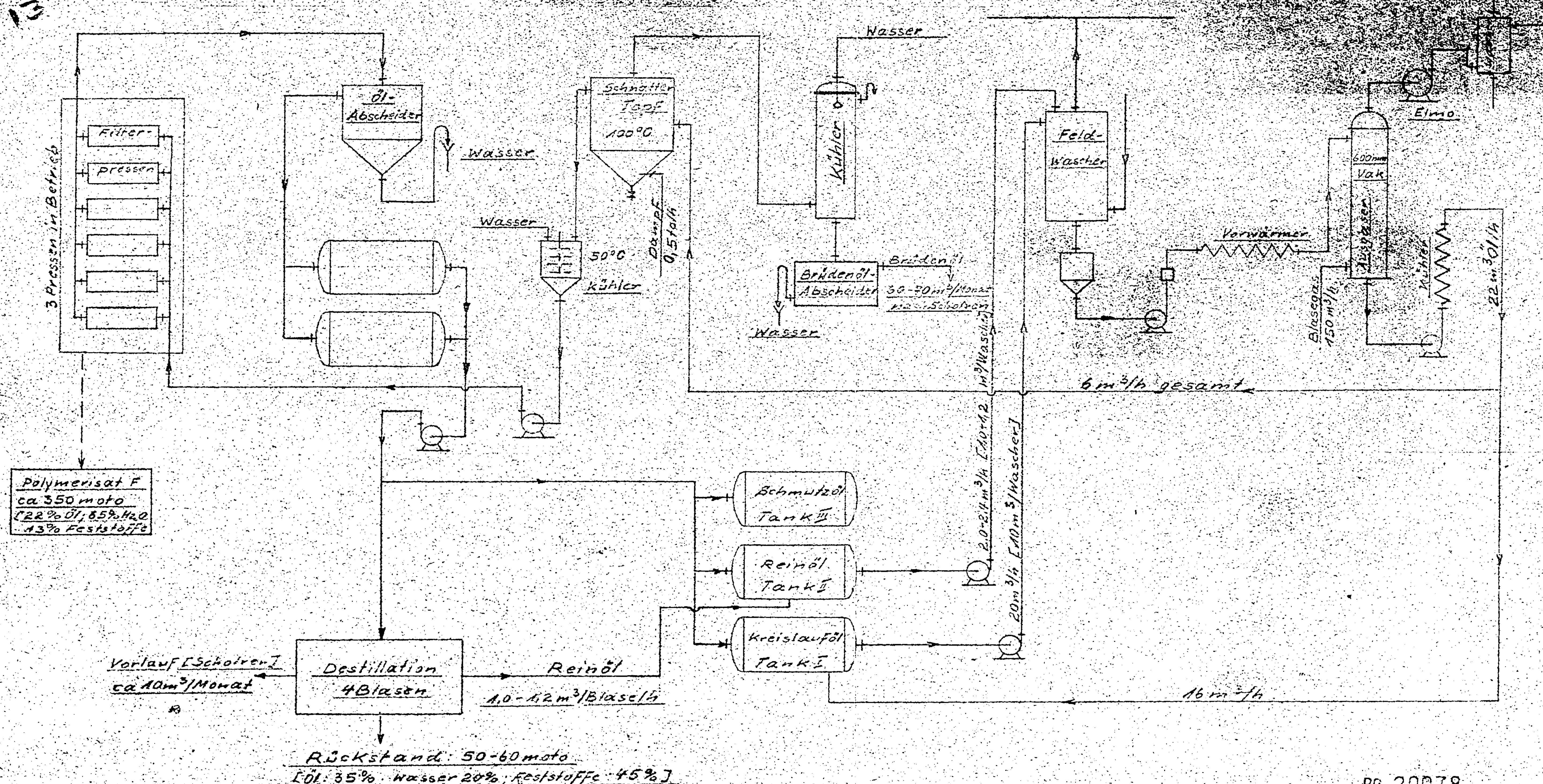


Zugehörige Zeichnungen
Zchg. Nr.
Hü 430 u. 432.
Lichtbogenanlage u. Rußabscheidung
insgesamt 15 Gruppen
10 Gruppen normal in Betrieb
3 " " Reserve
2 " " Reparatur
Gleichrichter
700 KVA
Lieferant: BBC Mannheim
Trinkwasser 1,5 m³/h
Lichtbogen, 20 Öfen je Gruppe
Material: Eisen, Mantel 10 mm
Lieferant: Hauptwerkstätte, Hüls
Flußwasser
8 m³/h
Mischgas
2800 m³/h
Trinkwasser
4 m³/h
Flusswasser
9 m³/h
4200 m³/h
11 gr. Ruß/m³ Gas
Fliehkraftabscheider
4 je Gruppe
Material: Eisen, Mantel 1800 ϕ - 6 m sl.
Lieferant: Hartmann, Offenbach
Naßabscheider
4 je Gruppe
Material: Eisen, Mantel 8 mm
Lieferant: Jakobs, Oberhausen
Filter
8 je Gruppe
Material: Eisen, Mantel 4 mm
Lieferant: Intensiv, Langenberg
12 m³
2,5 gr. Ruß/m³ Gas
Schlamm Ltg. 2X
Schlamm Ltg. 3X
Gesamtanlage der Saugzellenfilter
Filterfläche Kleinzellenfilter: 4,5 m²
Lieferant: Schüchtermann, Kremer, Baum, Dortmund
Schlamm-Filter
5 gesamt
Hü 420
Naßruß-Aufarbeitung
Ruß-Trockner
40 gesamt
11 Etagen
Teller ϕ 4900 mm
Lieferant: Haas, Lennep
ca. 400-420 moto N-Ruß
5,5 gr. Ruß/m³ Gas
Klärbecken
3 gesamt
Material: Eisen, Mantel 8 mm
Lieferant: Schüchtermann, Kremer, Baum Dortmund
Überlauf
3 gesamt
Q = 750 m³/h; H = 40 m WS
3 Stück insgesamt
Material: Gußeisen
Lieferant: Halberg, Lu.
Automatische Absackwaage
Lieferant: Libra-Werke, Braunschweig
45000
zur Halde
Sammelstation
2X
Material: Eisen, Mantel 6 mm
Lieferant: Hartmann, Offenbach
50 m³
Hü 418
Trockenruß-Aufarbeitung
Lieferant: Haas, Lennep
Material: Eisen, 4 st.
Trommeln 800 ϕ - 4000 lang
28 Trommeln
26 in Betrieb
Trommel
ca 200 moto T-Ruß
Aenderungen



Lichtbogen - Rußfabrik - N.D.G.R.
Erdgas - Waschöl B.
Lichtbogen: Mengenabgabe für 1 Gruppe
Anschließende Betriebe: Mengenangabe für 2 Gruppen.
Hü. 531.
N.D.G.R.
Stückzahl
Benennung
Teil
Werkstoff
Lager Nr. Modell Nr.
Gewicht
Bemerkung
$H_2O$
zum Kraftwerk
Wascher
Mat: Eisen St.
Fa. Bischoff, Essen
Wascher 700⌀ 4400
Elmo
3 gesamt
2 in Betrieb
Vo = 4500 m³/h
600 mm Vak.
Aus-gaser
Fa. Siemens, Berlin
3 gesamt
2 in Betrieb
800⌀
30000
11 Stufen
60 m³/h
je Kühler
5000⌀ - 10000 hoch
Material: Eisen, Mantel 8 mm
Lieferant: Hauptwerkstätte Hüls
Gaskühler
2 gesamt
2 in Betrieb
Feld-wascher
1 gesamt
1 in Betrieb
3500⌀
16000 hoch
Material: Eisen, Mantel 9 mm
Lieferant: Feld u. Co., Essen
Antrieb: N = 88 KW.
V gas = 20000 Nm³/h
Vorlage
Vorwärmer
Eckart - Messer
60 °C
4 Pumpen
2 in Betrieb
Q = 15 m³/h; H = 50 m WS.
Material: G.E.
Lieferant: Holberg, Ludwigshafen
Blasegase
150 m³/h
je Ausgaser
Kühler
2 gesamt
2 in Betrieb
Ölkreislauf
3 Pumpen
2 in Betrieb
Q = 20 m³/h; H = 40 m WS.
Material: Gußeisen
Lieferant: Holberg, Lu.
Aufgabe - Station
2 X
Material: Eisen, Mantel 5 mm
Lieferant: Hartmann, Offenbach
Material: Eisen; Mantel 9 mm,
Lieferant: Bischoff, Essen,
6000
Tank III.
Schmutzöl
30 m³
5000
Tank II.
Reinöl
30 m³
6000
Tank I.
Kreislauföl
20 m³
825 m³/h
Turbo - Gebläse
Lieferant: Jäger Leipzig
1.) V = 750 Nm³/min.
n = 2950 Umdr./min.
2.) V = 404 Nm³/min.
n = 2150 Umdr./min.
Δp = 20 mm WS.
3 Pumpen
2 in Betrieb
Q = 2 m³/h; H = 30 m WS
Material: G.E.
Lieferant: Holberg, Lu.
3 Pumpen
2 in Betrieb
Q = 20 m³/h; H = 25 m WS
Lieferant: Holberg, Ludwigshafen.
2 Pumpen
2 in Betrieb
Q = 20 m³/h; H = 30 m WS
Material: G.E.
Lieferant: Holberg, Lu.
Lieferant Bamag, Berlin.
Öl - Destillation
Hü. 525   4 Blasen
Vorlauf
4-6 ltr./h / Blase
0,6 - 0,75 m³/h / Blase
Reinöl
1 Kühler
Rückstand
200 - 250 moto
(66% Öl)
HCN - Gewinnung
10 kg/h (Versuch)
8,25 m³/h
Material: Eisen, Mantel 12 mm
Lieferant: Bischoff, Essen
Wasser-wascher
1 X 40000 m³/h
2 X 25000 m³/h
2 in Betrieb
4000⌀
(3500⌀)
Frisch-Wasser
Wasser Kreislauf
325 m³/h
800 m³/h
1 Elmo
600 mm Vak.
Wasser-Ausgaser
2 gesamt
3500⌀
(4000)
30000
Material: Eisen, Mantel 12 mm
Lieferant: Bischoff, Essen.
Dampf
20 to/h
5 Stück insgesamt
Q = 750 m³/h; H = 40 m WS
Material: Gußeisen
Lieferant: Holberg, Ludwigshafen
Kühl - Werk
Überlauf
3 Pumpen
2 Stück Q = 640 m³/h; H = 40 m WS.
1 „ Q = 4000 m³/h; H = 40 m WS.
Material: Gußeisen
Lieferant: Holberg, Ludwigshafen.
3 Pumpen
2 Stück Q = 640 m³/h. H = 40 m WS
1 „ Q = 4000 „ H = 30 m WS
Material: Gußeisen
Lieferant: Holberg, Ludwigshafen
3 Pumpen
2 Stück Q = 640 m³/h; H = 30 m WS.
1 „ Q = 1000 m³/h; H = 30 m WS
Material: Gußeisen
Lieferant: Holberg, Ludwigshafen.
Hü. 539
Schwefelreinigung
Lieferant: Bischoff Essen.
Material: Eisen, Mantel 8 mm.
zum Kraftwerk
nach Hü. 442
zum Gasometer
D-4
Bestell Nr.
Bau Nr.
Bestellung
Besteller
Jahr
Paßsystem Einheitsbohrung
Betriebsdruck atü
Probedruck atü
Tag
Name
gezeichnet 14.4.45 Günther
geprüft 17.7.45 Ka.
normgepr.
Maßstäbe
Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen
Nr. 1.
Ersatz für
Ersetzt durch

13



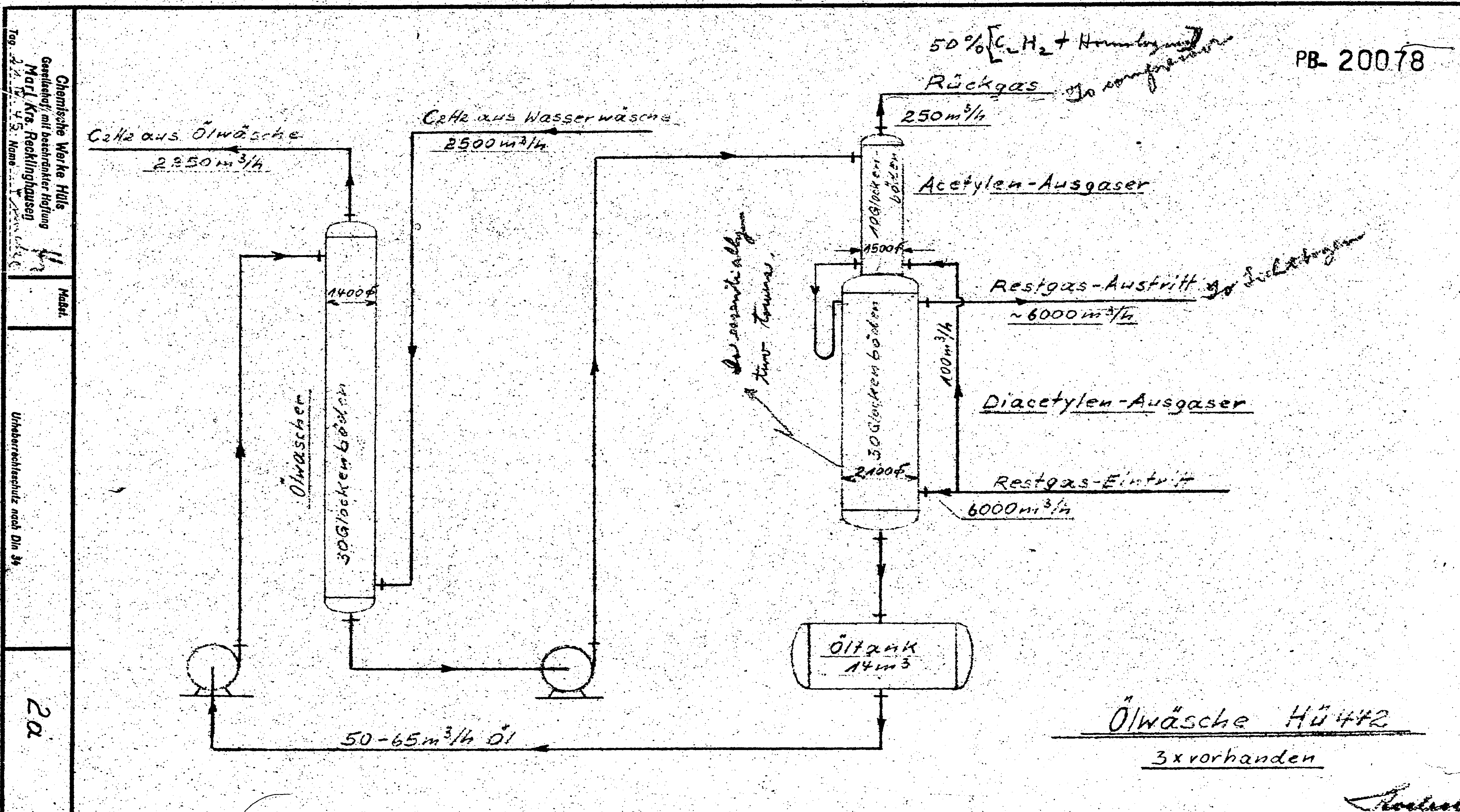

PB-20078

Ölkreislauf der NDGR bei Verwendung von Kogasin als Waschöl

| | | | |
|---|---|---|---|
| Chemische Werke Hüls Gesellschaft mit beschränkter Haftung Marl Kr. Recklinghausen | Maßst. | Urheberrechtsschutz nach DIN 34 | 11.5.45 1a |
| Tag 17.4.45 Name [signature] | | | |

D-6

PB- 20078

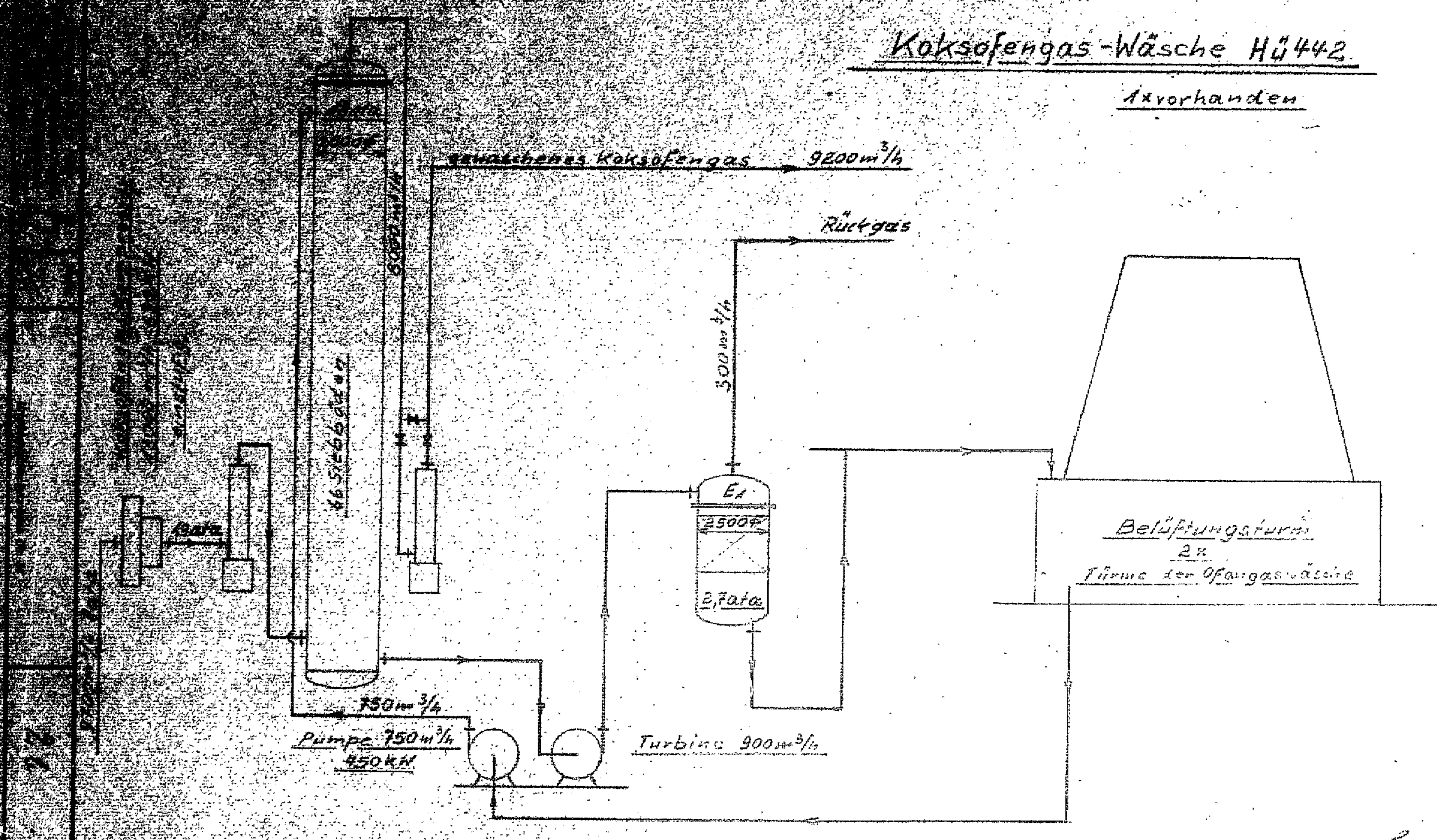


Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Kr. Recklinghausen
Maßst.
Urheberrechtsschutz nach Din 34
2a
C2H2 aus Ölwäsche
2350 m3/h
C2H2 aus Wasserwäsche
2500 m3/h
1400 Ø
30 Glockenböden
Ölwäscher
50 % [C2H2 + Homologues]
Rückgas
250 m3/h
10 Glockenböden
1500 Ø
Acetylen-Ausgaser
Restgas-Austritt
~6000 m3/h
100 m3/h
30 Glockenböden
2100 Ø
Diacetylen-Ausgaser
Restgas-Eintritt
6000 m3/h
Öltank
14 m3
50-65 m3/h Öl
Ölwäsche Hü 442
3 x vorhanden

PB-20078

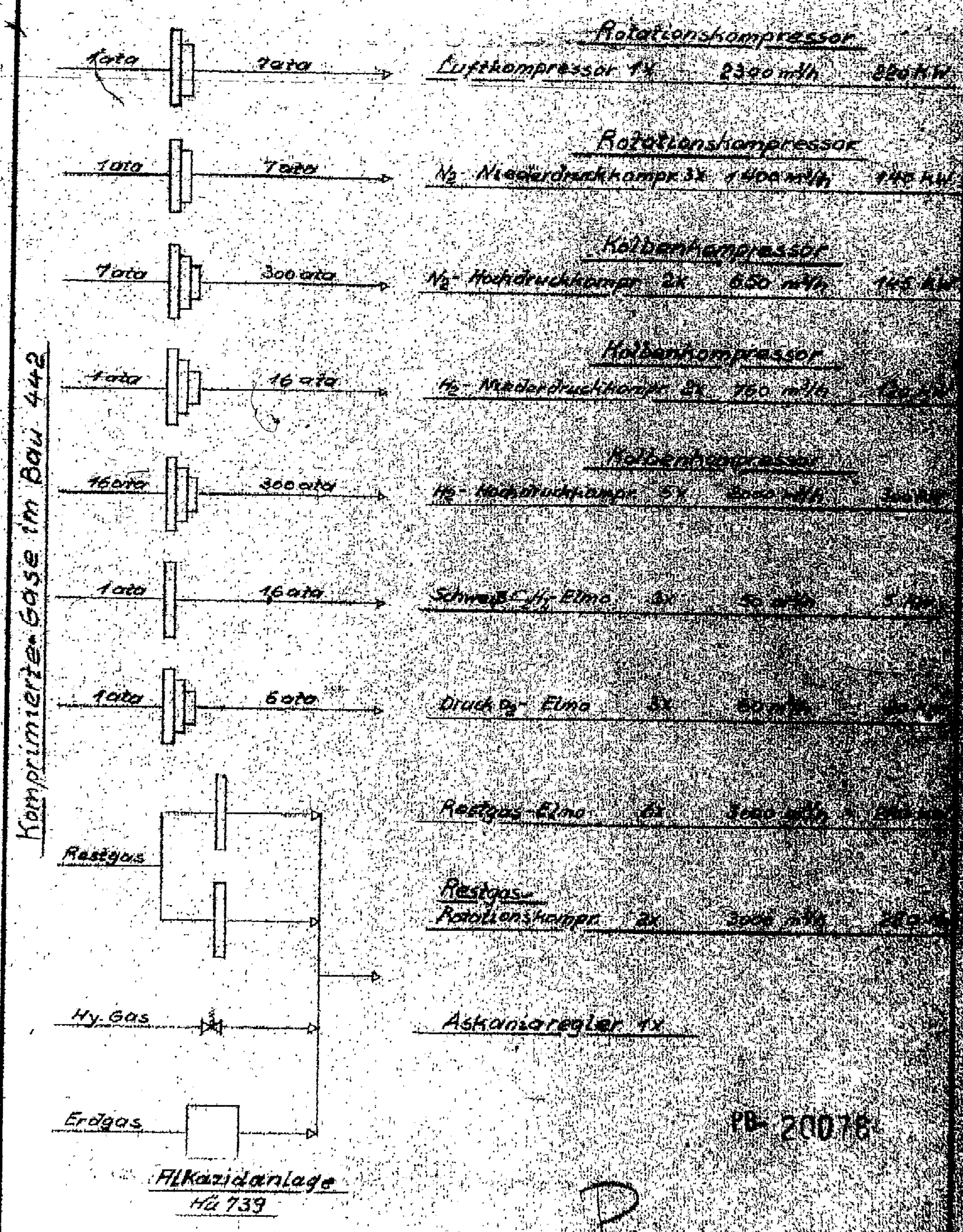
# Koksofengas-Wäsche Hü442

1x vorhanden




D-7

(14)




PB 20078

# Gaskompression im Bau 442

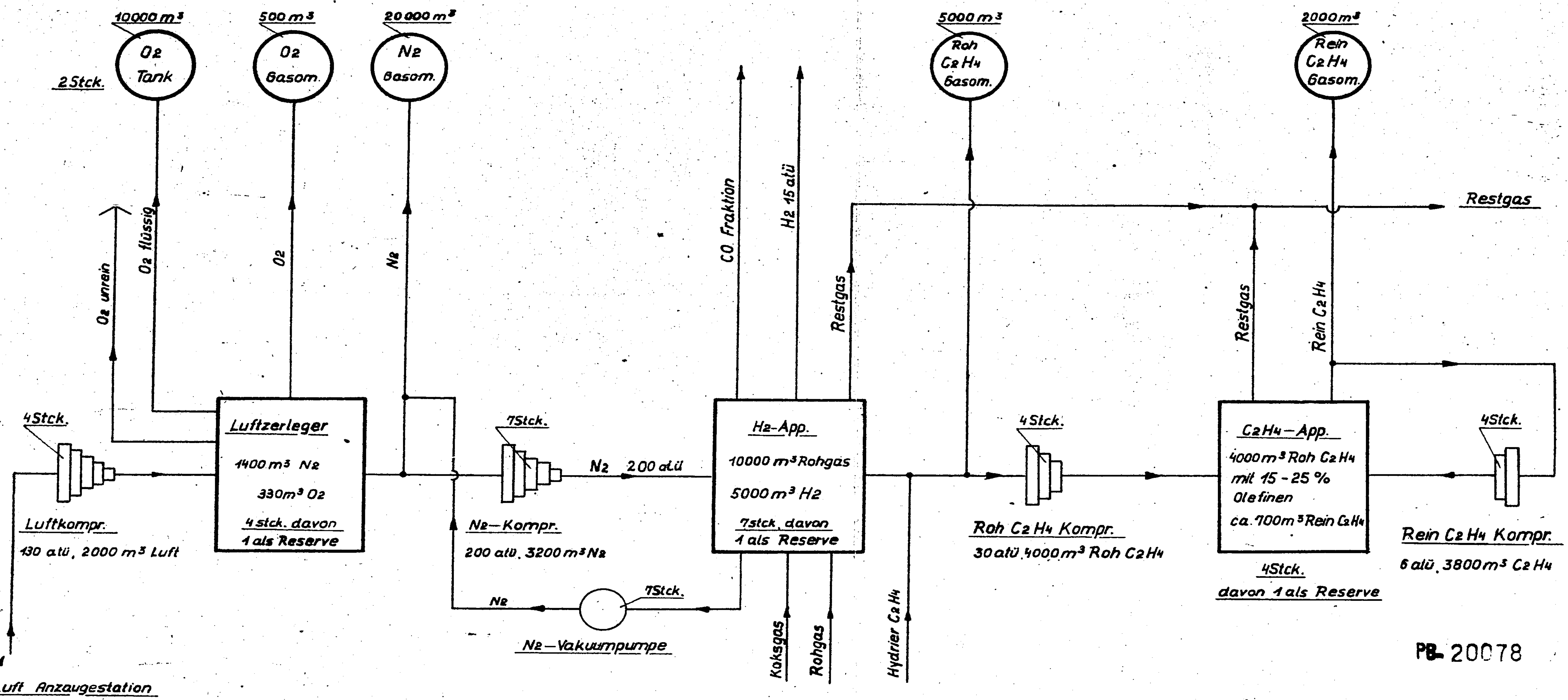
## Hilfsmaschinen

Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen
Tag: 12.4.45 Name:

Urheberrechtsschutz nach DIN 34

24

## Fluss-Schema oder Gaszerlegung




PB-20078

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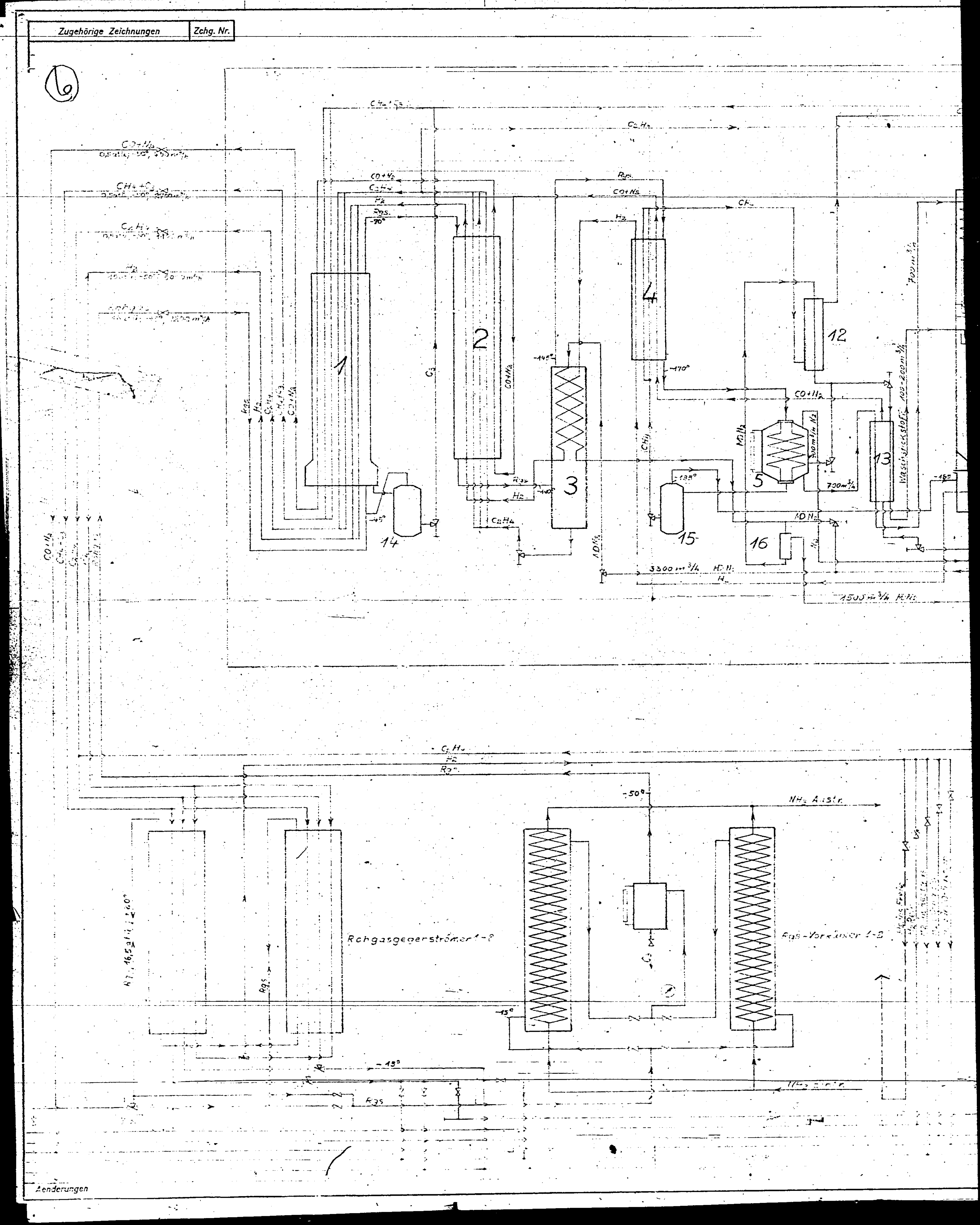
Die Apparaturen wurden Komplett von der
Fa. Linde Höllriegelskreuth b. München geliefert
Einzelheiten über die Ausführung sind unbekannt.

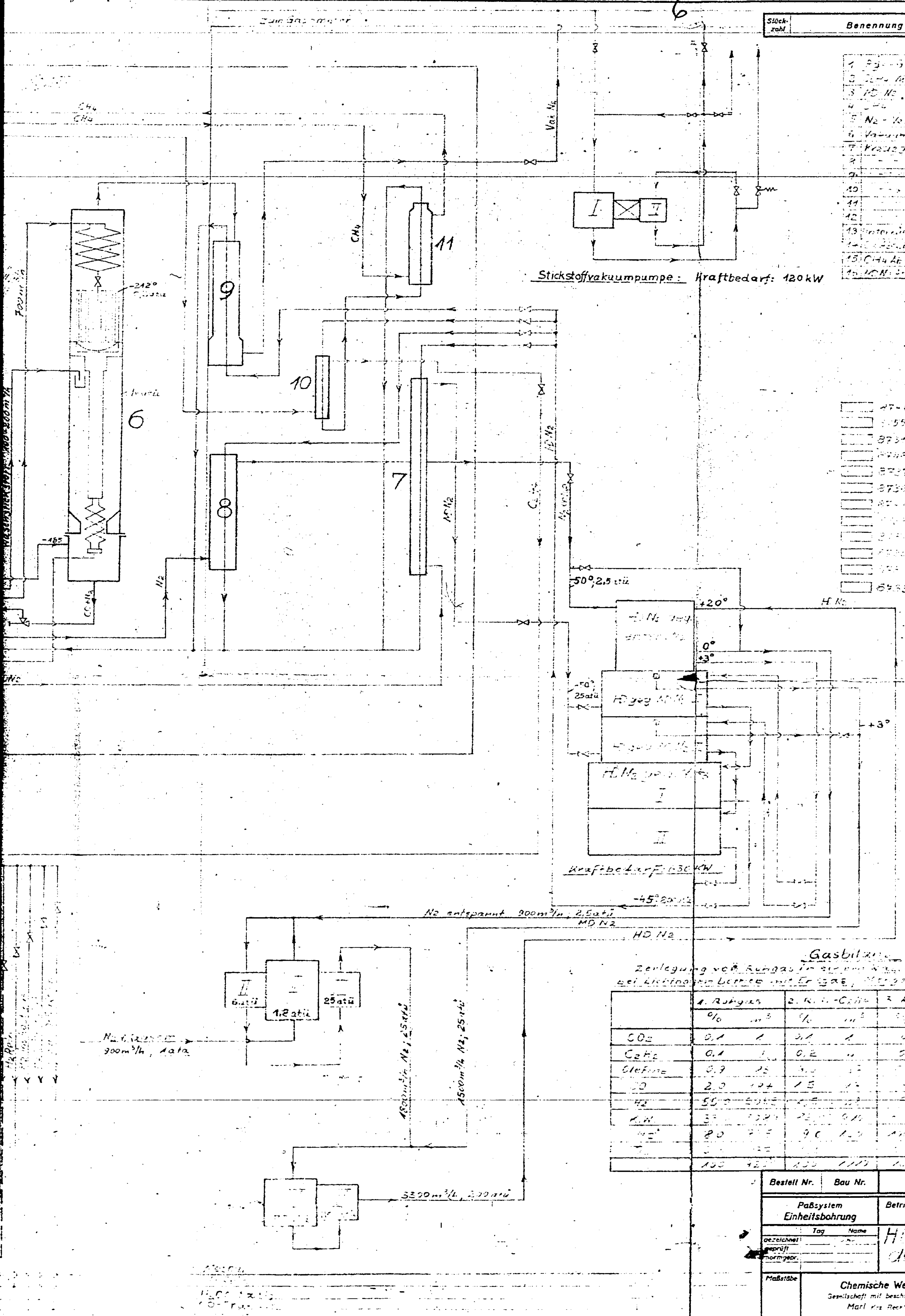
Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Kr. Recklinghausen
Tag 21.4.45 Name Nickel
4.7.45

Maßst.

Urheberrechtsschutz nach DIN 34

Nr. 4


Zugehörige Zeichnungen
Zchg. Nr.
1
2
3
4
5
12
13
14
15
16
CO+H2
C2H4
CH4
H2
Rgs.
-70°
-145°
-170°
-135°
-50°
-15°
700 m³/h
100-200 m³/h
Waschstickstoff
3300 m³/h HD-N.
NH3 Austr.
Rohgasgegenströmer 1-8
Aenderungen




| Stückzahl | Benennung | Teil | Werkstoff | Lager-Nr. / Modell Nr. | Gewicht | Bemerkung |
|---|---|---|---|---|---|---|
| | 1 [illegible] | | | | | |
| | 2 [illegible] | | | | | |
| | 3 HD $N_2$ [illegible] | | | | | |
| | 4 [illegible] | | | | | |
| | 5 $N_2$ - [illegible] | | | | | |
| | 6 [illegible] | | | | | |
| | 7 [illegible] | | | | | |
| | 8 [illegible] | | | | | |
| | 9 [illegible] | | | | | |
| | 10 [illegible] | | | | | |
| | 11 [illegible] HD $N_2$ - $CH_4$ | | | | | |
| | 12 [illegible] $CH_4$ | | | | | |
| | 13 [illegible] | | | | | |
| | 14 [illegible] | | | | | |
| | 15 $CH_4$ [illegible] | | | | | |
| | 16 HD $N_2$ [illegible] | | | | | |

## Gasbilanz

Zerlegung von Rohgas in einem Wasserstoffapparat bei Lichtbogenbetrieb mit Erdgas, Mengen in Nm³/h

| | 1. Rohgas | | 2. [illegible] | | 3. Restgas | | 4. CO-Fraktion | | 5. Wasserstoff | |
|---|---|---|---|---|---|---|---|---|---|---|
| | % | m³ | % | m³ | % | m³ | % | m³ | % | m³ |
| $CO_2$ | 0,1 | 1 | 0,1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| $C_2H_2$ | 0,1 | 1 | 0,2 | [illegible] | 0 | 0 | 0 | 0 | 0 | 0 |
| Olefine | 0,9 | 25 | [illegible] | [illegible] | [illegible] | [illegible] | 0 | 0 | 0 | 0 |
| CO | 2,0 | [illegible] | 1,5 | [illegible] | [illegible] | [illegible] | 11,0 | 50 | 0 | 0 |
| $H_2$ | 55,0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| K.W. | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 0 | 0 |
| $N_2$ | 8,0 | [illegible] | 9,0 | [illegible] | [illegible] | [illegible] | [illegible] | 322 | 2,9 | 145 |
| [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 0,1 | [illegible] |
| | 100 | [illegible] | 100 | [illegible] | [illegible] | [illegible] | [illegible] | 450 | 100 | [illegible] |

| Bestell Nr. | Bau Nr. | Bestellung | Besteller | Jahr |
|---|---|---|---|---|
| | | | | |

| Paßsystem Einheitsbohrung | Betriebsdruck atü | Probedruck atü |
|---|---|---|
| | | |

| | Tag | Name |
|---|---|---|
| gezeichnet | | |
| geprüft | | |
| normgepr. | | |

Hü 460 Gesamtschema der Wasserstoffboxen 1-5

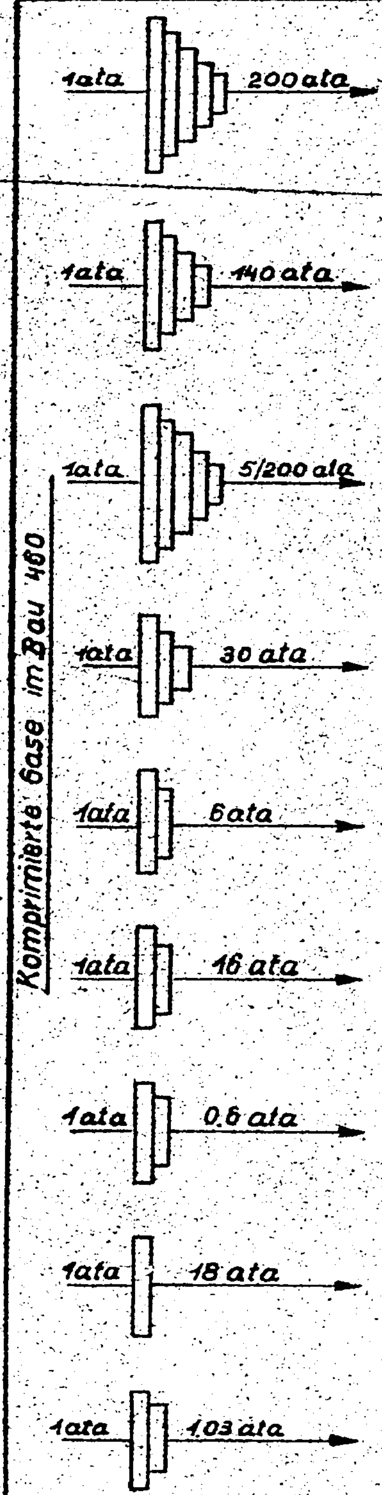
Maßstäbe

Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen

4a

Ersatz für
Ersetzt durch

1 ✓




**Kolbenkompressor**

$N_2$ Kreislauf-Kompr. 7x 500 $m^3/h$ 630KW

Lieferant: Masch.-Fabr. Sürth, Köln-Sürth

**Kolbenkompressor**

Luft-Kompr. 2x 2050 $m^3/h$ 500KW

Lieferant: Masch.-Fabr. Sürth. Köln-Sürth

**Kolbenkompressor**

Luft-Kompr. 2x 2050 $m^3/h$ 300KW

Lieferant: Masch.-Fabr. Sürth. Köln-Sürth

**Kolbenkompressor**

Roh-$C_2H_4$-Kompr. 4x 4050 $m^3/h$ 700KW

Lieferant: Masch.-Fabr. Sürth. Köln-Sürth

**Kolbenkompressor**

Rein-$C_2H_4$-Kompr. 4x 3700 $m^3/h$ 320KW

Lieferant: Masch.-Fabr. Sürth. Köln-Sürth

**Kolbenkompressor**

$NH_3$ Verb. Kompr. 5x 249000 kcal/h (+675000) 490KW

Lieferant: Masch.-Fabr. Eßlingen. Eßlingen

**Kolbenkompressor**

$NH_3$-Zus. Kompr. 2x 450000 kcal/h 70 KW

675000 kcal/h 170 KW

Lieferant: Masch.-Fabr. Eßlingen. Eßlingen

**Kolbenkompressor**

Koksgas-Kompr. 2x 13000 $m^3/h$ 630KW

Lieferant: Masch.-Fabr. Sürth. Köln-Sürth

**Rotationskompressor.**

$N_2$-Rot.-Vak.-Pumpe R.C. 580; 6x 4750 $m^3/h$ 118KW

Lieferant: Fa. Demag. Duisburg

PB-20078

D-11

## Gaskompression im Bau 460

## Hilfsmaschinen

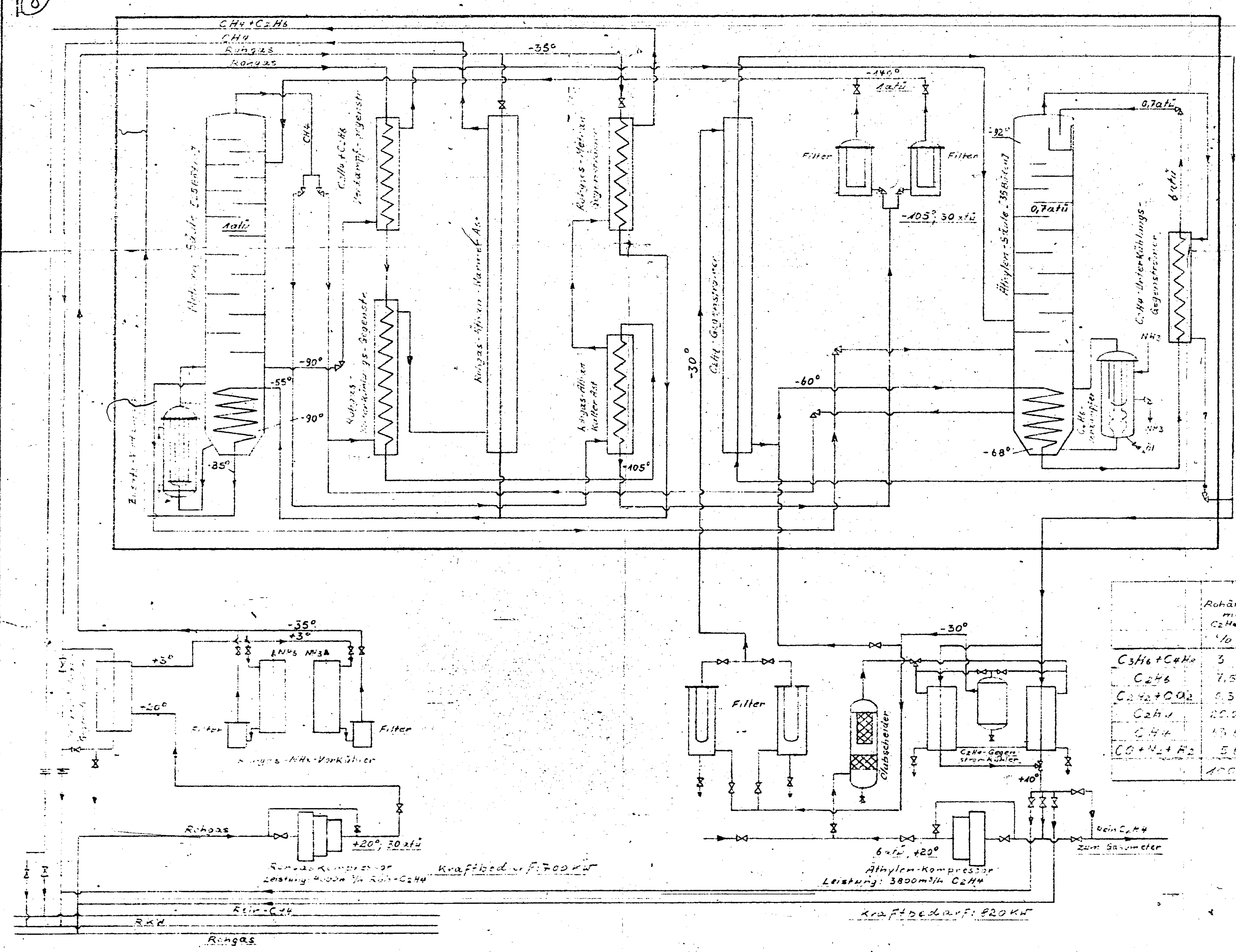
| | | | |
|---|---|---|---|
| Chemische Werke Hüls<br>Gesellschaft mit beschränkter Haftung<br>Marl Kreis Recklinghausen<br>Tag: 25.6.45 Name: Brandmann | Maßst.: | | Nr. 4d |

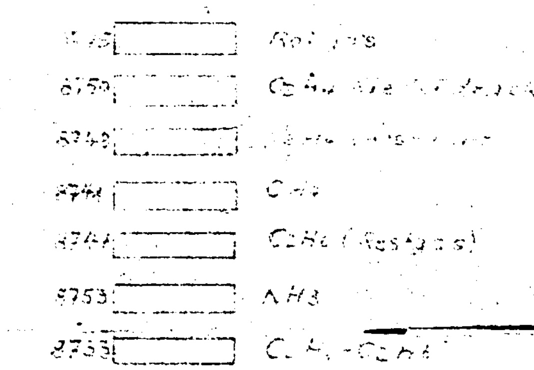
5.7.45 Stephan Kö

| Zugehörige Zeichnungen | Zchg. Nr. |
|---|---|
| | |

| Stückzahl | Benennung | Teil | Werkstoff | Lager Nr. / Modell Nr. | Gewicht | Bemerkung |
|---|---|---|---|---|---|---|
| | | | | | | |

(8)




D-12

PB- 20078

## Gasbilanz

### Für einen Äthylenapparat

Mengen in $Nm^3/h$

| | 1. Rohäthylen-Gemisch vor $C_2H_4$ Apparat | | 2. Reinäthylen | | 3. $CH_4$ | | 4. $C_2H_6$ | | 5. Restgas | |
|---|---|---|---|---|---|---|---|---|---|---|
| | % | $m^3$ | % | $m^3$ | % | $m^3$ | % | $m^3$ | % | $m^3$ |
| $C_3H_6 + C_4H_8$ | 3 | 120 | – | – | – | – | 26,7 | 120 | 3,7 | 120 |
| $C_2H_6$ | 7,5 | 300 | 2,6 | 19 | 0,1 | 3 | 62,0 | 278 | 8,8 | 281 |
| $C_2H_2 + CO_2$ | 0,3 | 12 | 1,6 | 12 | – | – | – | – | – | – |
| $C_2H_4$ | 20,0 | 800 | 95,0 | 740 | 0,3 | 9 | 11,3 | 51 | 1,8 | 60 |
| $CH_4$ | [illegible] | 2544 | 0,8 | 5 | 91,5 | 2539 | – | – | 78,8 | 2539 |
| $CO + N_2 + H_2$ | 5,6 | 224 | – | – | 8,1 | 224 | – | – | 6,9 | 224 |
| | 100 | 4000 | 100 | 776 | 100 | 2775 | 100 | 449 | 100 | 3224 |

| Bestell Nr. | Bau Nr. | Bestellung | Besteller | Jahr |
|---|---|---|---|---|
| | | | | |

Paßsystem Einheitsbohrung | Betriebsdruck ........ atü | Probedruck ........ atü

| | Tag | Name |
|---|---|---|
| gezeichnet | | |
| geprüft | | |
| normgepr. | | |

Schema
Äthylen-Anlage Hüls

Maßstäbe

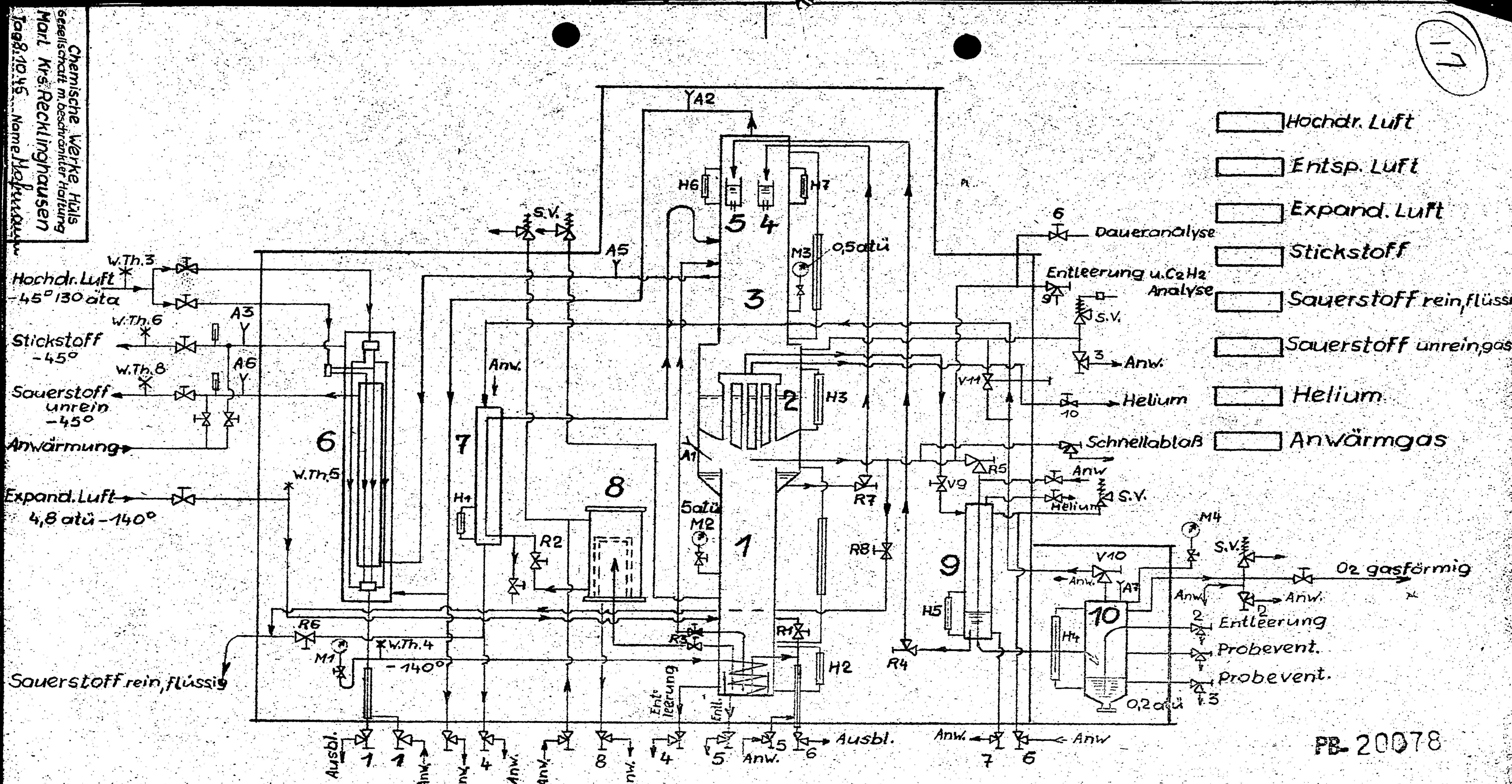
- Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Krs. Recklinghausen

4 b



Ersatz für
Ersetzt durch

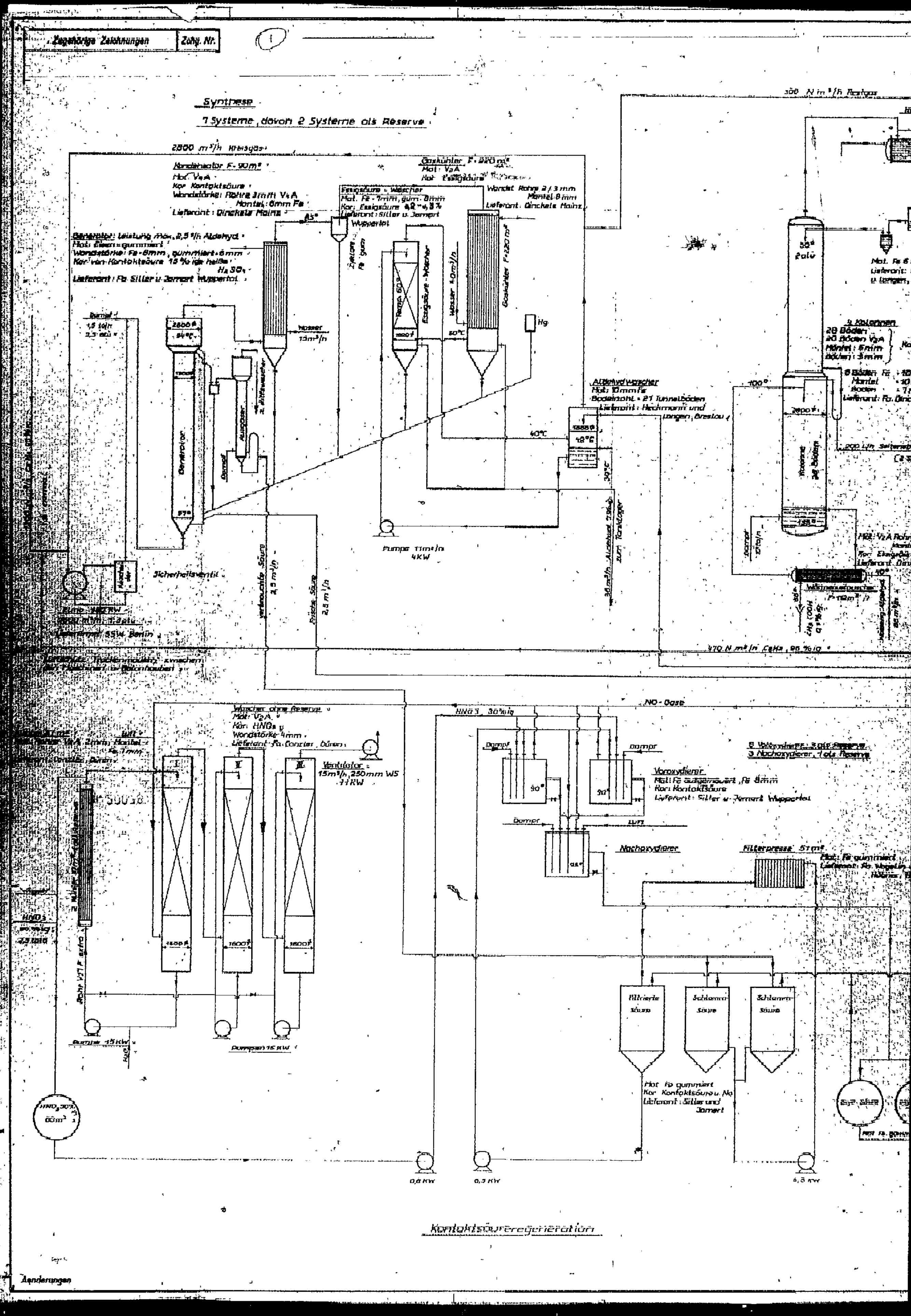
Aenderungen:



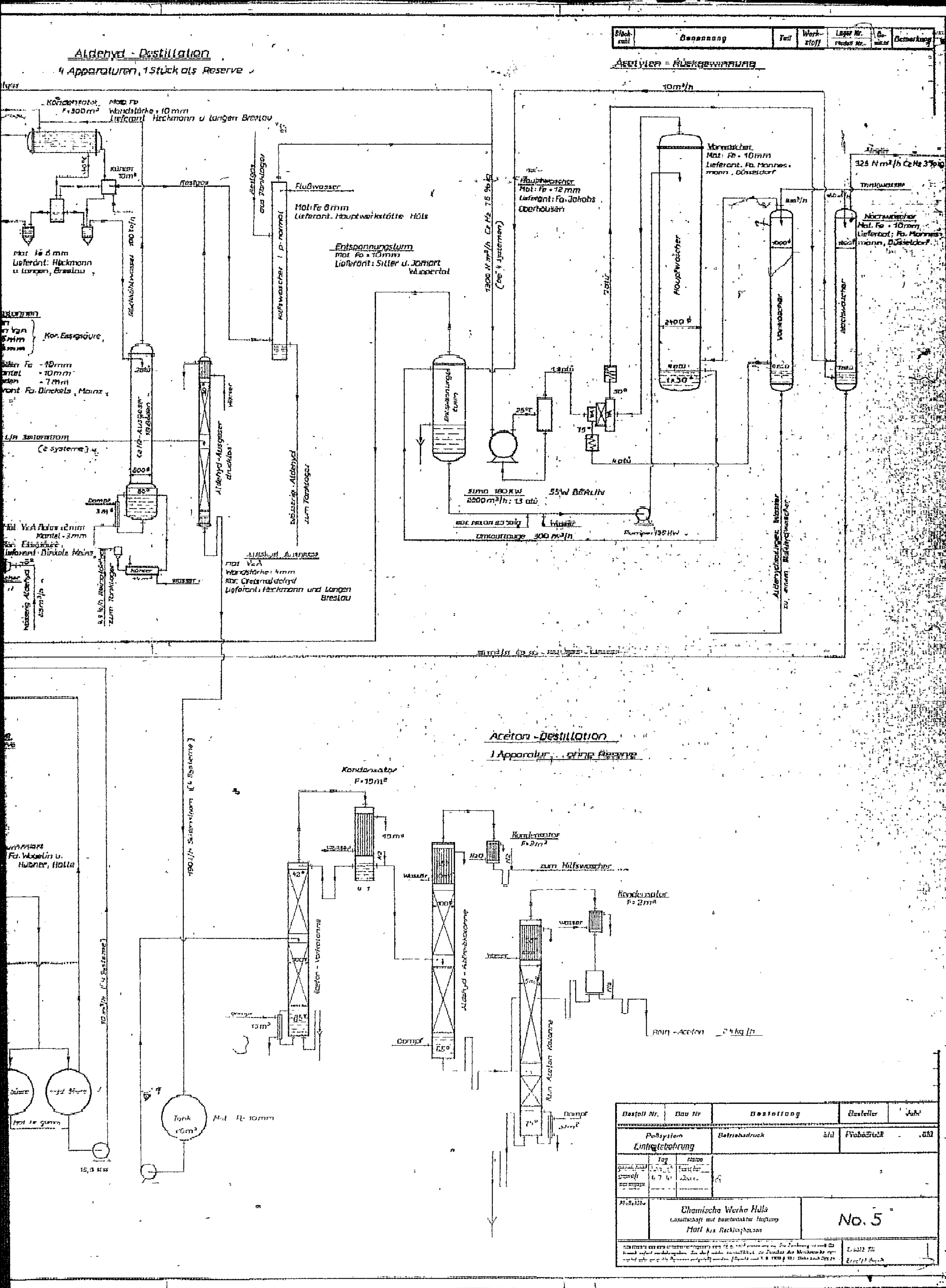

1 Drucksäule
2 Kondensator
3 Obere Säule
4 N2 Meßgefäß, Drucksäule
5 N2 Meßgefäß-Zusatzkond.
6 HDr. Luft-N2-O2 unrein Kr. Gegenströmer
7 O2-Verflüssigungs-Kr. Gegenströmer
8 Filtertopf für Flüss. O2
9 Zusatz-Kreuzstrom-kondensator
10 Azetylen-Abscheidung

D-13

4c



Zugehörige Zeichnungen
Zchg. Nr.
Synthese
7 Systeme, davon 2 Systeme als Reserve
Kondensator F-90m²
Gaskühler F-220 m²
Essigsäure - Wascher
Generator
Pumpe 11m³/h 4KW
Sicherheitsventil
Aldehydwascher
Bodenzahl = 21 Tunnelböden
4 Kolonnen
Wärmeaustauscher
verbrauchte Säure
Frische Säure
NO-Gase
Wascher ohne Reserve
Ventilator
15m³/h, 250mm WS
Voroxydierer
Nachoxydierer
Filterpresse
Filtrierte Säure
Schlamm Säure
Mat. Fe gummiert
Kor. Kontaktsäure u. $N_2$
Lieferant: Sitter und Jamert
0,6 KW
6,3 KW
Kontaktsäureregeneration
Aenderungen



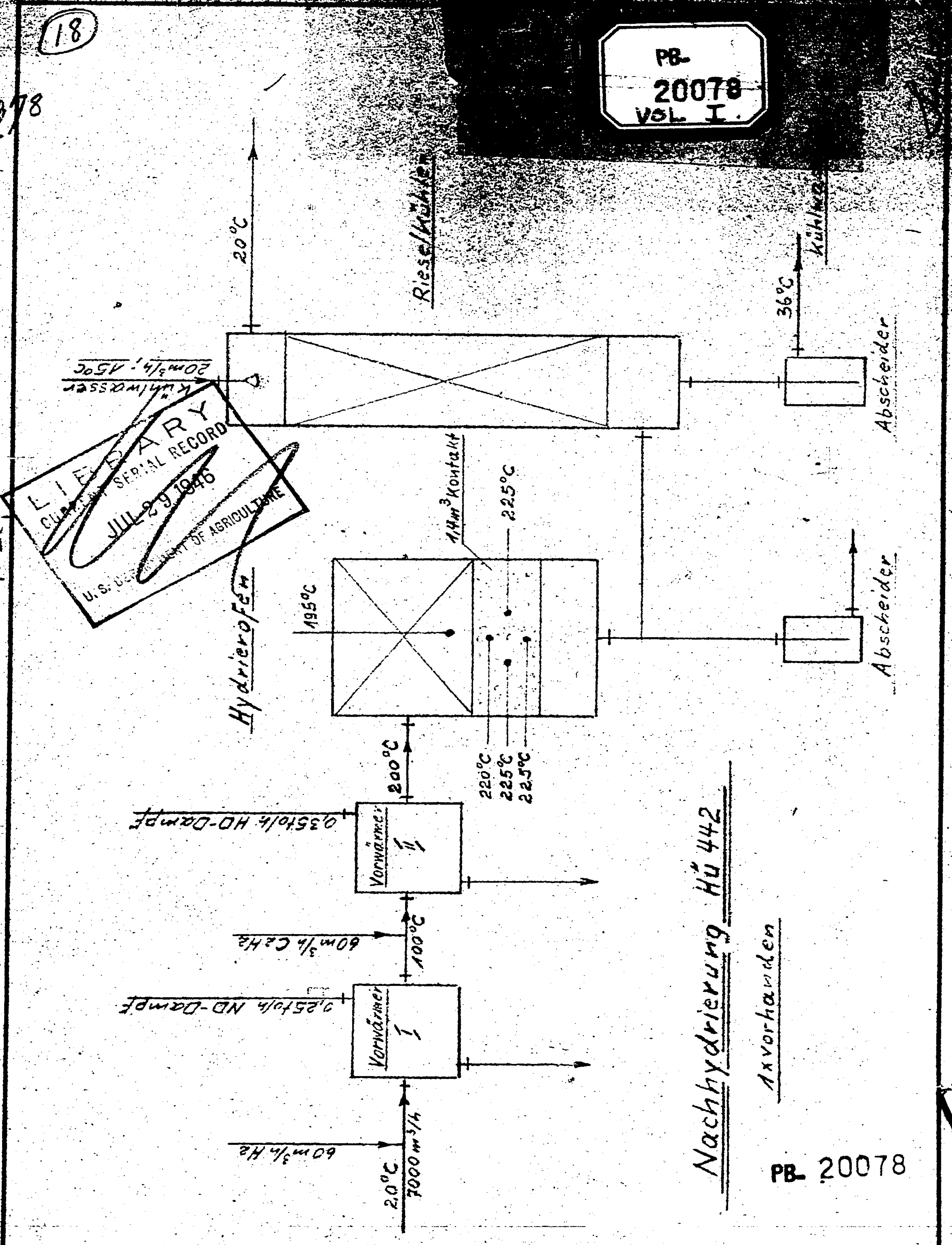

| Bestell Nr. | Bau Nr. | Bestellung | Besteller | Jahr |
|---|---|---|---|---|
| Paßsystem Einheitsbohrung | Betriebsdruck | atü | Probedruck | atü |

Chemische Werke Hüls
Gesellschaft mit beschränkter Haftung
Marl Kr. Recklinghausen

No. 5

(18)










| Chemische Werke Hüls<br>Gesellschaft mit beschränkter Haftung<br>Marl Krs. Recklinghausen<br>Tag 21.4.45 Name [illegible] | Maßst. | Urheberrechtsschutz nach Din 34 | 2c |
|---|---|---|---|

## EXHIBIT E

### PROCESS DESCRIPTIONS OBTAINED AT HUELS

The following process descriptions prepared by Dr. Baumann's staff were obtained:

1. Betriebsbeschreibung der Lichtbogenanlage unit Russabscheidung und - aufarbeitung.
   Describes arc process and carbon black production.
   Includes gas analyses, flows, etc.

2. Arbeitsweise der Gleichrichtersteuerung bei den Lichtbogengruppen.
   Explains electrical control system for maintaining stable arc.

3. Betriebsbeschreibung der Niederdruckgasreinigung.
   Describes low pressure gas purification.

4. Betriebsbeschreibung der Gaswäsche Hä 442.
   Description of water scrubbing of arc gas and concentration of acetylene.

5. Beschreibung der Tiefkühlanlagen Hä 462.
   Description of low temperature cooling of acetylene for removal of homologues.

6. Betriebsbeschreibung des Aldehydetriebes.
   Describes aldehyde production.

7. Betriebsbschreibung der Gaszerlegung (Linde-Anlage) Bau 460.
   Operation of Linde Plant.

Copy 1

# FIAT FINAL REPORT No. 809

# FERROCYANIDES AND SULFUR FROM GAS WORK RESIDUES

McBerty, Ford H.

## OFFICE OF MILITARY GOVERNMENT FOR GERMANY (US)



## FIELD INFORMATION AGENCY TECHNICAL

OFFICE OF MILITARY GOVERNMENT FOR GERMANY (US)

FIAT FINAL REPORT NO. 809 27 May 1946

# FERROCYANIDES AND SULFUR FROM GAS WORK RESIDUES

BY


FORD H. McBERTY

TECHNICAL INDUSTRIAL INTELLIGENCE BRANCH

U.S. DEPARTMENT OF COMMERCE





FIELD INFORMATION AGENCY, TECHNICAL

# A B S T R A C T

Potassium ferrocyanide for making Prussian blues is made by way of the calcium salt and the insoluble calcium-potassium salt. The former is leached from gas works spent oxides after mixing with lime. The sulfur content of the spent oxides is first extracted with carbon disulfide. Both operations are described.

TABLE OF CONTENTS.

## INTRODUCTION

### Objective:

The objective of this investigation was to obtain the process by which the Chemische Fabrik Wesseling A.G. works up the spent oxides from gas works purification operations to secure the potassium ferrocyanide they require for manufacture of Prussian blues. Information was also obtained concerning an incidental sulfur recovery step.

### Evaluation:

The ferrocyanide process has been in use for some fifty years. It is simple and inherently inexpensive, although involving the handling of large amounts of low value commodities to extract a small amount of valuable product. The extraction of sulfur is carried out in a new installation very well designed. The combining of the two steps seems obviously advantageous.

### Guide to the Reader:

General comments concerning the nature of the raw materials used are followed by an outline of the methods of recovering ferrocyanides and sulfur. There is then described in some detail, first, the process and equipment used for extracting sulfur, and next, the ferrocyanide operation and equipment. A final section outlines the methods of analysis used to evaluate raw materials.

## GENERAL OUTLINE

In the manufacture of city gas and the like, the use of some form of iron oxide or hydrate to remove sulfur and cyanogen compounds is well known. The resulting spent oxide is a highly variable material, both as to chemical composition and as to physical properties. In general, it contains the original iron oxide, plus any shavings or other material used as a carrier or admixture to facilitate gas flow. To this is added from the gas stream variable amounts of ammonia and ferrocyanide radicals, sulfur, and tar-like compounds.

The residues purchased by Chemische Fabrik Wesseling are in two broad categories: those containing 35 to 50% sulfur, average about 40%, but having a low ferrocyanide content, usually 1 to 2%; and those containing about the same amount of extractable sulfur, but containing also from 4 to 10% of ferrocyanide, expressed as $Fe_7(CN)_{18}$. The second group may be divided into two further classes: those containing not more than 0.6% of ammonia liberatable by lime; and those containing more ammonia. The former represent the majority, for which separate extraction of ammonia is not considered justified.

## Process Outline:

The spent oxide mass as received, if it contains sufficient sulfur, is first extracted with carbon disulfide at about 30°C. The resulting sulfur is cast in a tile lined "pond". It is of gray to greenish yellow color and is sold or used without further purification.

Spent mass containing more than 4% of "$Fe_7$ $(CN)_{18}$" and more than 0.6% of ammonia is first extracted with water to remove soluble ammonia compounds from which the ammonia is subsequently liberated with lime. The extracted mass then goes directly to the ferrocyanide operation.

Mass containing more than 4% of "$Fe_7$ $(CN)_{18}$" and not over 0.6% ammonia is processed for ferrocyanide. The operation can handle material regardless of sulfur content.

In the ferrocyanide operation, the mass is first mixed with a small amount of sawdust, averaging 2 to 3% of the weight of the raw mass. This serves to facilitate the leaching step by providing a mix through which the leach liquor can more readily percolate. The mass is then mixed with a quantity of quick lime, finely ground, sufficient in amount to liberate the ammonia and form calcium ferrocyanide. The mix is dumped into leach tanks, in which it is leached with water at a temperature not exceeding 60° C. The leaching is countercurrent, batch; the material remains in the leach tank about 14 days, being extracted each day with a more dilute liquor than on the preceding day, and finally for about five days, with water.

The strongest calcium ferrocyanide liquors from the leaching step usually about 5° Baume are heated to a boil and settled overnight to precipitate tarry materials. The clear liquor is treated with enough potassium chloride to form the insoluble potassium-calcium ferrocyanide. The precipitate is filtered and washed, then treated with an excess of potassium carbonate, in water. The slurry is filtered and the filtrate and wash liquors are used to treat additional quantities of the calcium- potassium ferrocyanide until the strength of the potassium ferrocyanide solution has been built up to 20° to 25° Baume, and the greater part of the potassium carbonate has been converted to ferrocyanide. The liquor is then concentrated to 33° Be. and allowed to crystallize. The successive mother liquors are concentrated finally to as high as 37° Baume because of the accumulation of impurities. Only the large clean crystals are used; the fines and muds in the crystallizing tanks are re-worked through the double salt step.

The sodium ferrocyanide is not produced because the absence of an insoluble double salt makes necessary a prohibitive amount of evaporation. Some ammonium ferrocyanide is made by the same process; the ammonium-calcium double salt is more soluble than the calcium-potassium compound, but still sufficiently low in solubility to permit the process to be carried through. The ammonium double salt is used chiefly or solely to make a pure calcium ferrocyanide, which is used by Italien and Spanish wine makers to improve the color of their product by removing some of the iron content.

## SULFUR EXTRACTION

For sulfur extraction, the mass is loaded into extraction baskets, which are then placed in tall extraction tanks, through which carbon disulfide is circulated in closed circuit with a still and condenser, until the liquid leaving the extractor contains no appreciable sulfur, Sulfur accumulates in the still. After extracting some five batches, the contents of the still is evaporated down and the molten sulfur is run out to a cooling basin, tile lined, outdoors. The sulfur is finally broken into lumps.

The extraction baskets are of cast iron, in sections. Each section is 2 meters diameter and 450 mm deep. The section consists of a cast iron cylindrical portion, with an internal flange on which rests a perforated steel plate reinforced with angle iron ribs. The sections are recessed around the periphery, top and bottom, to receive bolts to join the sections together. There are eight such bolts for each section. Each section has lifting lugs to permit lifting after filling, and also to permit lifting the assembled basket consisting of six sections bolted together.

The individual sections are filled by hand with mass brought by monorail and bucket from the nearby outdoor spent mass stock pile. The filled and assembled basket is moved by crane to the extractor, of which four have been installed. After extracting, the basket is removed and the bolts are taken out. The basket sections are then dumped one at a time by a power operated rotary dumping device which discharges the mass into a hopper from which it can be dropped into monorail buckets for further disposition.

The extractors are vertical steel cylinders 2-1/2 meters diameter and 4-1/2 meters deep. The covers are removable by the crane that handles the baskets. Graphited cotton gaskets are used. The extractor runs nearly full of carbon disulfide. The distilled disulfide enters at the top. The disulfide leaves the extractor through a bottom connection and flows upward through a riser to an overflow, thence to the still. A sampling connection is provided at the overflow. The rate of flow is determined primarily by the number of extractors being operated and by the distillation rate. The steam consumption when running four extractors was stated to be 5 metric tons per hour. This includes however the steam used for steaming out the charge after extraction. The extractors have heat insulation outside.

The extraction cycle is as follows:

1. The basket containing a fresh charge of mass to be extracted is placed in the extractor and the cover is bolted on. Preferably,

the air is then displaced by nitrogen, though in recent years this step has been dropped since nitrogen was not available; no explosions or other troubles have resulted from the omission, to date.

2. The extractor is filled with carbon disulfide, and regular flow to and from the still established. Flow continues until a sample coming through the riser from the bottom of the extractor has the same specific gravity as pure carbon disulfide - namely 1.250 at 30° C. The extraction is then considered finished.

3. The carbon disulfide in the extractor is blown out with steam, and steam is blown through the extractor for about twelve hours, or until a sample condensed in cold water shows no carbon disulfide.

The average extraction cycle is about 24 hours, but varies considerably with the quality of spent oxide being processed. The usual spent oxide charge per extraction basket assembly is seven to eight metric tons of mass which would on the average include about three tons of extractable sulfur. The consumption of carbon disulfide is 26 kilos per metric ton of sulfur produced. The steam consumption at an average of 5 metric tons per hour would be 10 metric tons per metric ton of sulfur produced.

The carbon disulfide still is a vertical tank about 3 meters inside diameter and 3-1/2 meters high, covered with heat insulation. It is heated by coils at the bottom, which operate with steam at 90lbs gage pressure. The capacity of the still is six cubic meters of distilled carbon disulfide per hour. Both shell and coils are of steel, but after two years operation, the coils have failed by pitting and must be replaced. The shell is said to be undamaged.

The still is operated continuously until five extraction batches have been processed. The contents of the still is then boiled down until a temperature of 120° C. is reached in the charge in the still. The charge is then run out to the sulfur cooling area.

Vapors from the still pass first to a foam separator, which is an empty steel tank 1-1/2 meters diameter and 3-1/2 meters long. The vapors enter and leave the foam catcher from the top, passing thence to the condenser, which consists of iron coils in a tank of water.

Carbon disulfide is stored under water. The entire area containing the extractors and stills is so constructed that any leaks or spills would immediately flow to the ground level. The entire ground floor of the building is a pool of water some six or eight inches deep, so that any carbon disulfide reaching this area is immediately covered by water. The ground floor area is covered by wooden removable grids above the surface of the water.

The present installation consists of four extractors and one still with accessories. Provision is made in the building for a complete duplicate unit, in which the same crane would serve an additional four extractors.

Sulphur production in this extraction plant would be nominally about 12 tons per day. Actual monthly productions, in metric tons, for a representative period in 1944 were as follows:

| Month | Metric Tons Sulfur Produced | Month | Metric Tons Sulfur Produced |
|---|---|---|---|
| March | 215 | July | 396 |
| April | 272 | August | 346 |
| May | 365 | September | 244 |
| June | 291 | | |

## FERROCYANIDE PROCESS

**Mix Preparation:**

Spent oxide, either from the incoming material pile, or from sulfur extraction or from ammonia extraction, is carried to the mix shed in buckets on the overhead monorailsystem. The spent oxide used can if desired be that received from the gas works; it is not necessary to extract suflur before processing for ferrocyanide. The material should however contain at least about 4% of ferrocyanide, "$Fe_7 (CN)_{18}$", otherwise the recovery of ferrocyanide is not considered worth while.

At the mixing shed, there is added if considered necessary a quantity of sawdust to make the mix porous to facilitate leaching. The quantity of sawdust added averages 2 to 3% of the weight of spent oxide.

The mix with sawdust next goes to the grinding and screening operation, where it is passed through a low speed hammer mill and through a revolving screen with openings 8 mm in diameter.

If the mix contains more than 0.6% ammonia, it goes from this step to the ammonia leaching operation, described later. Otherwise it is ready for the extraction of ferrocyanides.

The mix goes next to the shaking conveyor that carries it into the mixer. At this point there is added ground quick lime (ground in a hammer mill in the adjoining building), in amount equal to 0.7% of the weight of the spent oxide for each 1% of $Fe_7 (CN)_{18}$ that it contains. This assumes that the charge contains not more than 0.6% of ammonia, which is the maximum ammonia content that can be tolerated in the mixer area.

From the shaking conveyor, the mix drops into a 2 meter diameter cylinder, open at both ends. The cylinder is 6 meters long and revolves at 20 R.P.M. It is equipped with lengthwise lifting scoops.

It is important that after mixing with quick lime the mix be not allowed to stand in a pile for an appreciable length of time, since it tends to heat up and fire.

Extraction:

There are six extraction "systems" each consisting of 14 to 18 extraction tanks.

Each extraction tank is 2-1/2 meters square in plan and 2 meters deep. One and a half meters below the top of the tank is the surface of the filter bed. The filter bed is carried on wood supports and a wood grid, and is built up of straw, sawdust, and a surface of cloth. The space above the filter surface receives a charge of 8 to 9 tons of "heavy" mass, from which the sulfur has not been removed, or 6 to 7 tons of "light" mass from which the sulfur has been extracted. The outside of the extraction tanks are sheathed with wood. The tanks have been in use for some fifty years.

Monorails for the charge buckets run above the extraction tanks, so that the buckets can be dumped directly into the tanks. Plank walkways are provided above the extraction tanks. Spent charge is shovelled over the side into buckets carried on monorails. A launder sloping towards a collecting tank runs lengthwise of each group of leach tanks. Each tank has a steam jet syphon or steam jack by which the filtrate collected in the bottom can be transferred to the launder or to an adjacent tank.

Each extraction system of the six, is operated independently on a cycle of about fourteen days, such that one tank is emptied and receives a fresh charge each day. A tank is ready to empty when for five successive days it has shown 0° Baume filtrate. The extraction is in general countercurrent, such that tanks that are almost exhausted are leached with fresh water, and fresh mix is used to bring the liquor to maximum concentration before further processing. The surface of the charge in the leach tanks is kept covered with liquid.

The concentration of the strong calcium ferrocyanide liquor depends somewhat on the ferrocyanide content of the fresh charge. For charge containing 5% "$Fe_7(CN)_{18}$", the strong liquor will run 5 to 6° Baume.

Processing:

The leached liquor is sent to one of five rectangular iron boiling tanks, each of 9 cu. meters capacity, 2 meters by 3 meters in plan, 1-1/2 meters deep. These tanks are heated by vertical pipes between horizontal headers, the bottom header being some distance off the bottom. In these tanks the liquor is boiled 2 to 3 hours, and is then allowed to settle overnight. Tarry sludges separate.

The clear liquor is dropped to the precipitation mixer, of which two are provided but **only one is said to be required. The** precipitation mixer consists of a semicircular horizontal tank with sides extended upwards. In the tank is a horizontal agitator shaft running at 20 R.P.M. The agitator shaft carries a number of arms ending in paddles, so arranged as to work the pulp back and forth. The trough is 2 meters diameter, 2 meters high and 2-1/2 meters long; the mixer capacity is 7-1/2 cubic meters.

The clear liquor when dropped into the precipitator is at 70° C. There is added a quantity of potassium chloride of 150% of that theoretically required to form the double salt $CaK_2Fe(CN)_6$. This compound separates as an insoluble precipitate that settles rapidly. The clear liquor is decanted. The solids are discharged through a bottom outlet to an adjacent nutsch.

The nutsch is 2 meters square in plan, and provides a space 300 mm deep above the filter cloth which is carried on a wood grid. The tank is of steel. The filtration is aided by vacuum below the filter surface.

After the cake has formed, there is added water to a depth of 30 mm. The cake is sucked dry; it then contains about 50% moisture. The nutsch must be filled twice to empty the precipitation mixer.

The filter cake from the nutsch is transferred to the decomposition mixer, which is identical with that used for the precipitation of the double salt. Two such mixers are provided for the decomposition step.

In the decomposition mixer, a quantity of the double salt is pulped with weak liquor, and potassium carbonate is added in substantial excess such that 25 cc of clear liquor requires for neutralization 25 cc of normal (N/1) sulfuric acid. There is next added a further quantity of the double salt filter cake, and the pulp is brought to a boil with open steam. The pulp is then filtered in an adjacent vacuum nutsch like that described above, and the cake washed as explained below. The filtrate is returned to the mixer, where a further quantity of double salt filter cake and some additional potash is added. By continuing this procedure, there is finally obtained a strong liquor of 20 to 25° Baume, containing no appreciable excess of potassium carbonate. The calcium carbonate residue in the nutsch is washed free of ferrocyanide, the weak filtrate being used for fresh batches. The calcium carbonate is discarded.

The strong liquor goes to the evaporators, of which there are five. There are steel tanks, 2 meters by 3 meters in plan, 1 meter deep. The evaporators are heated by steam coils that are about 8" above the bottom of the tank on I beam supports. In these, the "virgin" liquor is concentrated to 33° Baume (hot). The liquor is then dropped to crystallizers, of which some 40 are provided. The crystallizers are steel tanks, 1 meter by 1-1/4 meters in plan and 1-1/2 meters deep.

Crystallization requires about 10 days. The crystal crop from each crystallizer tank is 900 to 1000 kilos of "prime" crystals and a quite small amount of dirty crystals which may be reworked.

The mother liquor from the crystallization is reconcentrated, and again crystallized. Each successive reconcentration is carried to a somewhat higher density, finally reaching 37° Baume, hot.

General:

The capacity of the plant is stated to be 500 tons per year of potassium ferrocyanide crystals. No appreciable quantity of ferrocyanide is used in any other form: even for making their own Prussian blues, high grade crystal product is used, the liquors being considered too impure and variable in composition.

When the raw material contains more ammonia than can be tolerated at the mixer ahead of the leaching step, the raw material is given a preliminary leach to extract ammonia compounds, which are then treated with lime and distilled. The ammonia thus recovered is used at the chamber sulfuric acid plant or converted to sulfate and sold, or used to make alumina hydrate.

During the leaching operation, it is important that the temperature to which the material is exposed shall not exceed 60° C, if it contains substantial amounts of sulfur; otherwise the CNS radical will be formed. In general, the leaching step is carried out at temperatures below 60° C, for this reason, regardless of the sulfur content of the mass.

The leached iron oxide is sold to blast furnaces.

Before the war, small amounts of calcium ferrocyanide were made. To make this product, the crude ferrocyanide was precipitated as the comparatively insoluble calcium-ammonium double salt by adding at the precipitation step ammonium chloride instead of potassium chloride. The precipitate after washing is then treated with calcium hydrate and the ammonia boiled off. The resulting calcium ferrocyanide is concentrated and crystallized in the usual way.

## METHODS OF ANALYSIS OF SPENT OXIDES

Moisture:

Thirty grams of the mass is dried to constant weight at 70° C. The loss in weight is calculated as water.

Ferrocyanide - $Fe_7(CN)_{18}$.

The dried material from the moisture test is ground very fine. A 10 gram sample is put in a 250 cc volumetric flask, and 50 cc of 10° Baume caustic potash, KOH, is added, with vigorous stirring.

Potassium ferrocyanide dissolves. The contents is settled 12 hours at 20° C. There is now added just sufficient lead carbonate to precipitate all the sulfur, as determined by an outside test with sodium nitroprussiate. (Before taking out the sample for this test the contents of the flask is made up to the 250 cc mark).

The contents of the volumetric flask is next filtered. In a porcelain evaporating basin of 300 cc capacity is placed 20 cc of hydrochloric acid (1 part concentrated hydrochloric to four parts water) and 5 cc of a 10% solution of ferric chloride. To this is added 100 cc of the filtrate. A blue precipitate of $Fe_7(CN)_{18}$ is formed. This is filtered and washed with hot water. The filter paper with the precipitate is then returned to the 300 cc evaporating basin and is pulped with 20 cc of 10° Baume KOH. The pulp is boiled, then transferred to a 250 cc volumetric flask, and made up to the mark. The contents of the flask is filtered, and 100 cc of the filtrate is titrated with standardized copper sulfate solution, of such strength that 1 cc equals 0.0100 grams of $Fe_7(CN)_{18}$. The end-point of the titration is determinded by a spot test on filter paper, in which a drop is brought adjacent to a drop of ferric sulfate solution. When no blue color is formed, the-end point has been reached.

<u>Sulfur:</u>

Ten grams of the finely powdered dry material is extracted with carbon disulfide in a Soxhlet apparatus, until no more sulfur is dissolved, which requires 30 to 45 minutes. The extract contains sulfur and tars. It is shaken in a separatory funnel for ten minute periods with successive 20 cc portions of a sulfuric acid mix of 4 parts 66 sulfuric acid and 1 part 40% oleum. After each addition and shaking, the acid is drawn off. The treatment is continued until the carbon disulfide solution is very light in color - this usually requires two or three additions of acid. The clear solution is put in a 500 cc volumetric flack and made up to volume with carbon disulfide at room temperature. After allowing to settle, 250 cc is drawn off into a tared Soxhlet flask. The carbon disulfide is distilled off. The gain in weight is reported as sulfur. The difference between the weight of the 10 gram sample of dryoxide before and after extracting is the weight of the tars plus sulfur; by subtracting the weight of sulfur, the weight of tar is obtained.

Materials containing excessive amounts of tar are not considered suitable for extraction of sulfur; they are better used directly to make sulfuric acid.

<u>Ammonia:</u>

To 25 grams of the original mass in a 200 cc porcelain evaporating basin there is added 150 cc of water. The mix is brought to a boil and boiled one minute. The material is then rinsed into a

250 cc volumetric flask, filled to the mark, shaken, and filtered, Fifty cc of the filtrate is put in a 700 cc Erlenmeyer flask; 250 cc of water is added, and 50 cc of 20° Baume caustic soda. The contents is distilled through a Liebig condenser into a small Erlenmeyer containing 20 cc of N/1 sulfuric acid, plus enough water to cover the end of the condenser tube. The flask is boiled 20 minutes, after which the excess sulfuric acid is titrated with N/1 caustic soda with methyl orange indicator. The difference is ammonia.

These methods give results that are of practical significance for the actual manufacturing operations.

## APPENDIX NO. 1

## LIST OF GERMAN PERSONNEL INTERVIEWED

| Name | Position | Location |
|---|---|---|
| Mr. Otto Pfister | | Chemische Fabrik Wesseling A.G. Wesseling, near Cologne |
| Mr. Josef Ink | Control Chemist | Chemische Fabrik Wesseling A.G. Wesseling, near Cologne. |
| Mr. Josef Schumacher | Sulfur Extraction Foreman | Chemische Fabrik Wesseling A.G. Wesseling, near Cologne. |

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# FIAT FINAL REPORT 621

## FUEL INJECTION WITHOUT INJECTION PUMP

Madle, A. M.

## OFFICE OF MILITARY GOVERNMENT

## FOR GERMANY (US)



## FIELD INFORMATION AGENCY TECHNICAL

OFFICE OF MILITARY GOVERNMENT FOR GERMANY (U.S.)
FIELD INFORMATION AGENCY, TECHNICAL

FIAT FINAL REPORT NO.621 — 17 December 1945

# FUEL INJECTION WITHOUT INJECTION PUMP

BY

A. M. MADLE

Joint Intelligence Objectives Agency



FIELD INFORMATION AGENCY, TECHNICAL

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## 1. PREVIEW

In the following is presentedaa development of a fuel injection system, not employing a fuel pump nor a nozzle, which, although still inhibited with certain shortcomings, had been advanced to an operative stage.

Another development of a pumpless injection, although of simpler scope, reveals insight into the basic problem.

On hand of these experiences it seems possible that a complete solution of the problem of pumpless injection can be worked out.

## 2. INTRODUCTION

The idea of pumpless fuel injection seems to have originated with Dr. Ing. Prosper L'Orange, who had patents granted on his first concept in the early thirties. The first reliable test of such an engine, however, did not take place until 1940.

About 1941, the Hirth Motoren G.m.b.H. (later Ernst Heinkel - Werk Hirth Motoren) developed pumpless injection in connection with a bi-fuel en ine.

During the war there seems to have been very little other development in this matter.

Recently Dipl. Ing. Rudolf L'Orange and Dr. Stiebens (the Trustee of the Gebrueder L'Orange Company) started setting up a test engine at Stuttgart-Zuffenhausen, Markgroeninger Strasse 50, which, however, is reported to be the same engine that was tested by Prof. Kamm in 1940. Dipl. Ing. Rudolf L'Orange also issues a pamphlet, inviting the engine industry to give him sample orders for engines with pumpless injection.

3. The L'Orange System of Pumpless Injection

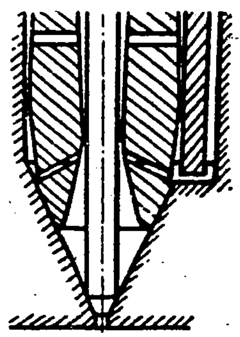
Dr. Prosper L'Orange explains fully the development of the high-speed Diesel engine, and the reasoning leading up to the injection without injection pump, as well as a description of the system in his paper "Die Entwicklung des raschlaufenden Dieselmotors bis zum Kleinstmotor ohne Einspritzpumpe" (The Development of the High-Speed Diesel Engine up to the Smallest Sizes without Injection Pump), printed in M.T.Z. (Motoren Technische Zeitschrift) Heft 3 of June 25, 1939.

To explain the fundamentals of the L'Orange system an abstract of the pertinent parts of this paper is presented in the following:

The essential premise for the combustion of heavy fuels in an engine resides in the extremely short timed, fine atomization of the fuel into the compressed, hot air, so that momentary ignition results.

This requirement was satisfied by the atomization of the fuel by compressed air, a process which has been improved by Hesselman, who effects the fuel atomization and injection by the differential pressure of inrushing air. (Figure 1)

Figure 1



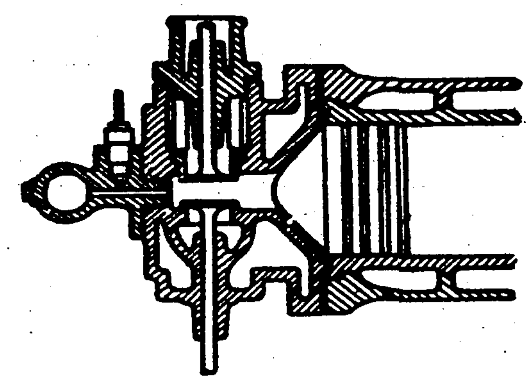
A still better atonization could have been attained by injecting the fuel into the airstream by a pump, the compression stroke of which falls entirely within the period, during which the air is



in motion. This, however, was at that time, practically impossible to attain, since injection pumps operated by excentrics and sealed by means of stuffing boxes do not lend themselves to such short injection periods.

The replacement of the compressed air as atomizing agents by the gases of an initial explosion in an after-chamber, in fact, necessitated the adoption of cam operated pumps with lapped-in plungers. Since, however, the injection took place in the connecting canal, an open nozzle and a comparatively low injection pressure was sufficient. The atomization of such an after-chamber engine was, a priori, excellent.

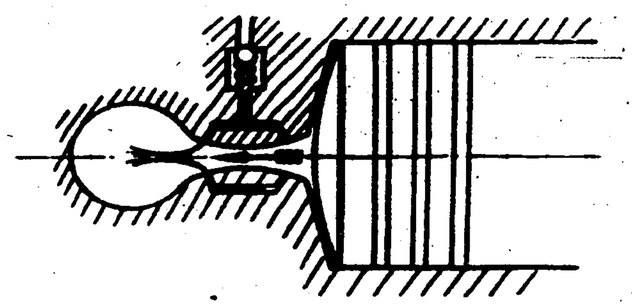
Figure 2



It was now Dr. L'Orange's idea to utilize the fundamentally sound structure of the after-chamber engine to work out further simplifications. For that purpose the after-chamber engine, illustrated in Figure 2 was modified "to effect the injection by the pressure difference* which always appears when gas flows from one chamber to another".

* This conception of the effect is faulty as borne out by the feasibility of the later discussed Hirth system, where obviously the pressure theory fails.

Figure 3



In the new arrangement, illustrated in Figure 3, the fuel, contained in a side channel, is injected into that chamber, towards which the air flow is directed.

The different phases of the process are the following (see Figure 4): (The following is a literal translation of the respective part of the paper).

Figure 4

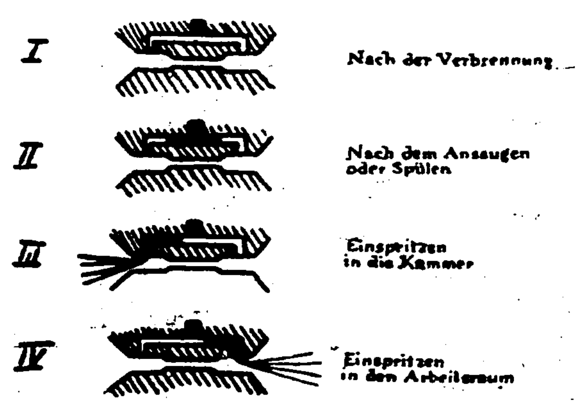



Bild 4. Schematische Darstellung des neuen Verfahrens

"1. During the suction stroke a small amount of fuel (at first controlled by a small valve) is drawn into an intermediate chamber "a".



"2. During the following compression stroke a part of this fuel, and always approximately the same quantity, is injected by the pressure difference between compression and side chamber into the latter and is there vaporized by the inrushing air. By the atomization of the fuel in the side chamber and by the compression heat, the mixture is ignited and since combustion with fixed volume occurs, a high pressure will result.

"3. By this high pressure, the rest of the fuel in the intermediate canal is injected into the combustion chamber and will be atomized by the combustion gases blowing into it.

"4. By the atomization of the fuel in the combustion chamber and with outward movement of the piston, the combustion, occurring there, will be approximately with constant pressure."

In conclusion of the paper, Dr. L'Orange presents test data and observation, gained with a 300 cc displacement, 4-cycle Diesel engine, and he claims that these results have been reproduced.

1. Power output (continuous)............4HP at 2400 RPM
   B.M.E.P.............................5 kg/sq.cm(71.2 psi)at 1262 ft/mm piston velocity
2. Maximum Speed.......................3000 RPM
3. Power output (peak).................5 HP at 2250 RPM
   B.M.E.P.............................6.7 kg/sq.cm(95.4 psi)at 1185 ft/mm piston velocity.
4. Absolutely clean exhaust by normal load and part load, and only a small soot content at overload, so that at normal RPM a clean jet can be expected.
5. An assured normal economy of 250 g/HP/hour and an optimal economy of 230 g/HP/hour.

The compression ratio, including the ignition chamber was 19:1. The pressure vs. time diagram is presented in Figure 5.

Figure 5

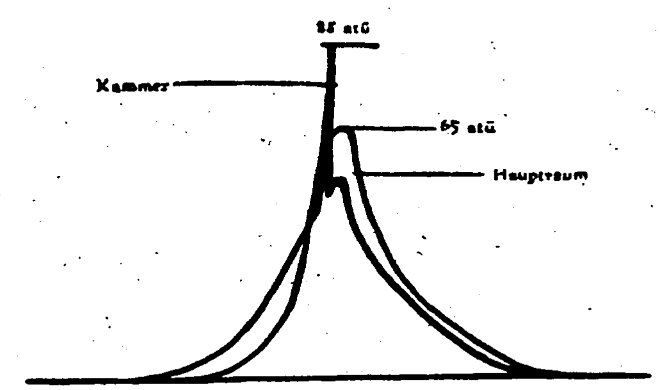



Diagramme mit der Viertaktversuchsmaschine 300 cm³
$n$ = 2300 U/min $p_{me}$ = 6 kg/cm²

From this diagram it may be seen that the initial explosion in the side chamber, as combustion with equal volume, produces a high pressure, namely 85 at (rel), while the pressure in the combustion chamber of 65 at (rel) peak, compares with that of a Diesel engine with conventional solid injection.

4. Prof. Dr. W. Kamm's Test Report

In 1939, Prof. Dr. W. Kamm, director of the F.K.F.S. (Forschungsinstitut fur Kraftfahrwesen and Fahrzeugmotoren - Technische Hochschule Stuttgart.....Institute for Research on Automotive Vehicles and Engines - Technical University Stuttgart) investigated apparently the same engine, as referred to in Dr. L'Orange's paper, for performance with various kinds of fuel and under various operating conditions and submitted a report - FKFS #366 of August 20, 1940 - on his findings.

In the report, referred to, it is pointed out that the economy vs. speed characteristic of the L'Orange engine, shows a fundamental deviation from that of a Diesel engine with pump injection, in the following expression:

"While the conventional injection pump with fixed adjustment delivers at any speed a nearly constant volume per stroke, therefore shows a fuel consumption per time unit approximately proportional to the speed, the fuel consumption of the L'Orange engine falls off at higher speed. The shape of power vs. speed curve in this speed range is therefore influenced by the mentioned peculiarity of the fuel supply."

The maximum of the power output and of the specific fuel consumption are found at the point of greatest fuel consumption per hour.

In the report it is furthermore pointed out that the full-load points were obtained with full open fuel supply valve and were approximately at smoke limit.

The starting was easy with a highly ignitable fuel (Kogasin II - Cetane Rating 78) and proportionally more difficult with a fuel of Cetane Rating 30. The latter fuel, also, showed a tendency of clogging the passages with coke, just as it was experienced with this fuel on standard Diesel engines.

The economy as well is much better with Kogasin II, when values of 260 to 270 g/HP/hour were scored, while with a poorly ignitable fuel of Cetane Rating 30 the economy was between 280 and 310 g/HP/hour.



The summary of this report is quoted:

"The investigated small Diesel engine type L'Orange has a peak power output of 5.3 HP at 2200 to 2700 RPM, which corresponds to 17 HP/liter of displacement. The maximum B.M.E.P. of 7 kg/sq.cm (99.6 psi) was found at 2000 RPM. With part loads of 3.5 to 4.5 HP an economy of 260 g/HP/hour was determined."

The data on this engine brought into the usual form are as follows: Bore: 70mm - 2.76"; Stroke: 80mm - 3.15"; Displacement: 307cc - 18.9 cu. in.; Compr. Ratio: 19:1; Ignition Chamber Displacement: 22 cu. in. 4-cycle, Diesel.

| HP | RPM | B.M.E.P. psi | Ft/min. | HP/sq.in. Piston Area | HP/1 | Economy lbs/HP/h |
|---|---|---|---|---|---|---|
| 5.3 | 2700 | 82.2 | 1420 | .832 | 17.25 | .670 |
| 5.3 | 2400 | 92.5 | 1262 | .882 | | .715 |
| 4.75 | 2000 | 99.6 | 1050 | .792 | | .670 |

The report, however, fails to point out the rather poor full load economy with a fuel of Cetane Rating 60.

5. L'Orange Development During the War

Dipl. Ing. Rudolf L'Orange, the son of Dr. L'Orange, enumerated a few engines with pumpless injection, that are supposed to have been developed during the war.

Among them was a 700 cc, 4-cycle Deutz Diesel engine and a converted 2 liter, 2-cycle engine with crankcase compression. No definite data were supplied on the performance of these engines, except the unsupported statement that with the L'Orange system of fuel injection they matched their own performance as Diesel engine with conventional injection.

A 1 liter Hirth, one cylinder engine was equipped with the L'Orange system for gasoline injection and allegedly tested in Berlin. No results of these tests are available.

As mentioned before, Dipl. Ing. L'Orange intends to install the 300 cc test engine, subject to the test of 1940, on the test block and duplicate its previous performance.

In conversation, Dipl. Ing. Rudolf L'Orange gave the impression that he is either reluctant to part with any new information or that he has nothing to impart, but in view of his promotional activities, does not want to have his lack of knowledge become too apparent.

6. <u>The Hirth Development of Pumpless Injection</u>

In 1941 the Hirth Motoren A. G. started a development program, on Government orders, purporting to the replacement of electrical ignition on gasoline engines for aircraft.*

The solution of this problem was sought in the employment of two kinds of fuel - the usual gasoline - air mixture and a small percentage of a fuel that will ignite with a Compression Ratio of 8:1. This special ignition fuel was developed by the I. G. Farben under the name of "R - Stoff".**

The main fuel - the air-gasoline was supplied in the conventional way by carburetor and supercharger, while Hirth selected pumpless injection for the supply of the ignition fuel.

Due to the fact the two fuels of different nature are used, the original L'Orange system is obviously not applicable. Dr. Bentele, who was in charge of this development, apparently recognizing L'Orange's faulty conception of the system's operation, designed an injection system with a single discharge channel with satisfactory performance and thereby proved that it is suction by gas flow through a Venturi tube, that injects the fuel. (Figures 6 and 7, attached).

Experiments were made with a 1 liter aircooled and with 3 liter water-cooled cylinder. Furthermore, in connection with the aircooled cylinder, a water-cooled and aircooled injection device respectively was used.

No difference in performance could be found traceable to the method of cooling either of the cylinder or of the injection device.

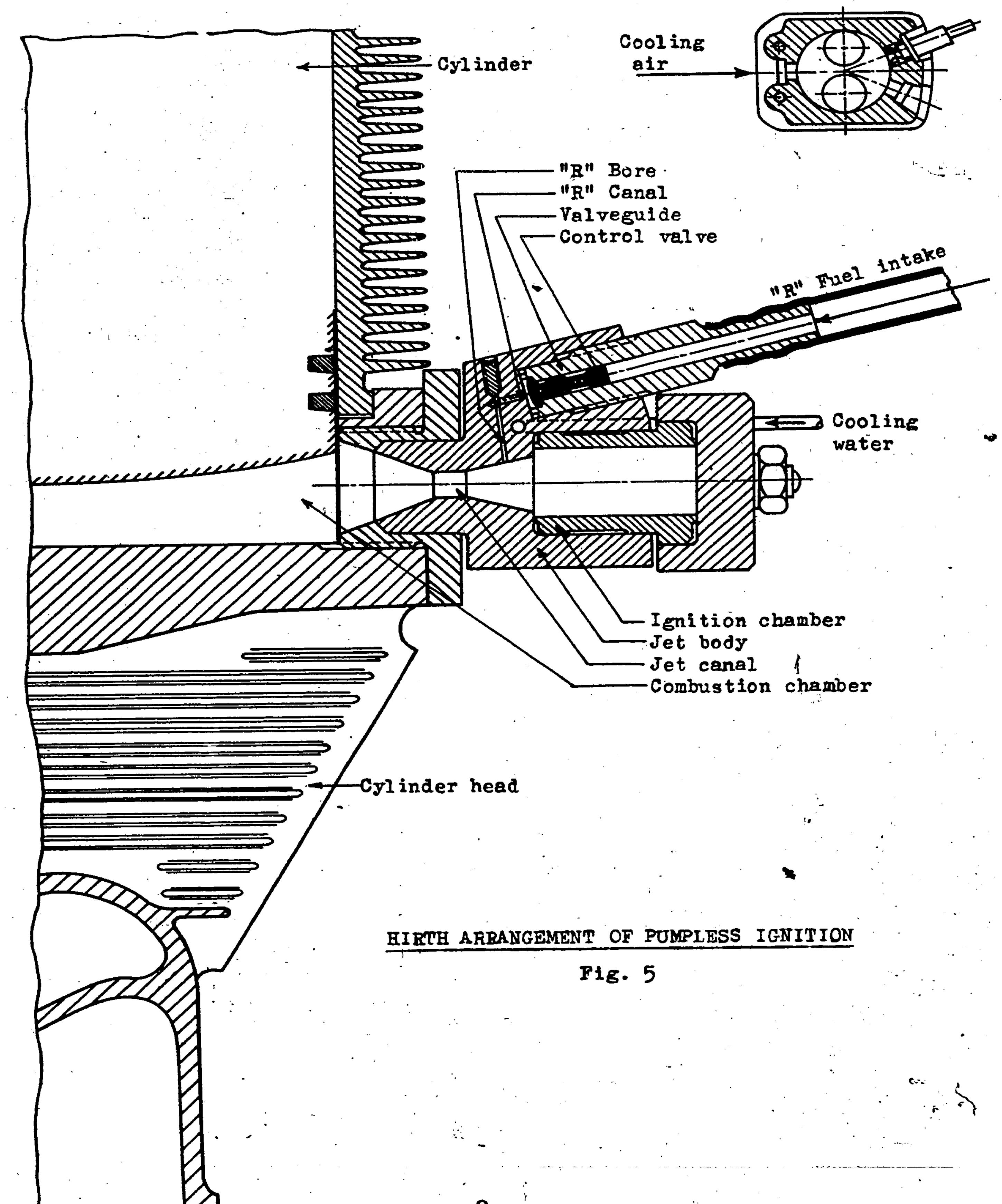
In a report of the Hirth Motoren G.m.b.H. of December 6, 1941, the following summary is given:

"After preliminary experiments with pumpless injection produced

* At that time the German airplanes had a ceiling of only 8000 m due to magneto failuresat higher altitudes. This seems to have been overcome, however, by improving the magneto.

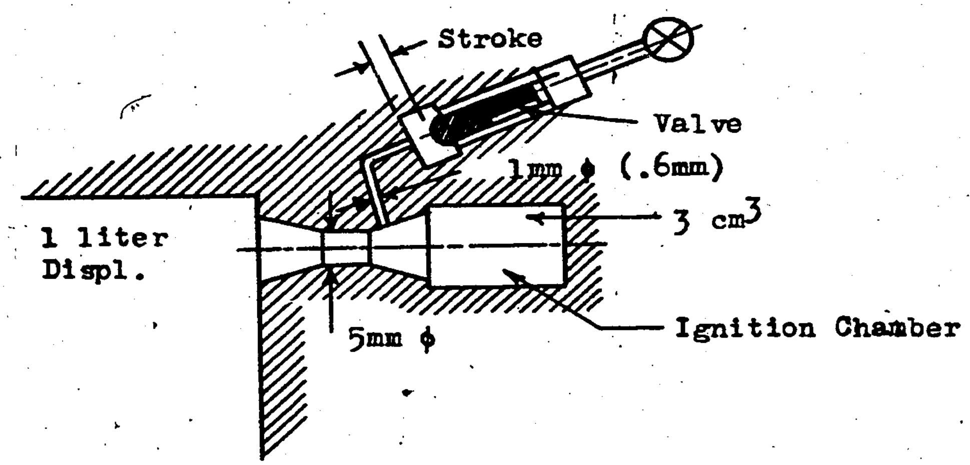
** The nature of this fuel was apparently a military secret, and nothing is known about it to the personnel of the Hirth Motoren A. G. It was found, however, that Prof. Dr. Wilke, of I. G. Farben, presumably at the experimental station Oppau, had an interest in the development and probably could supply this information.



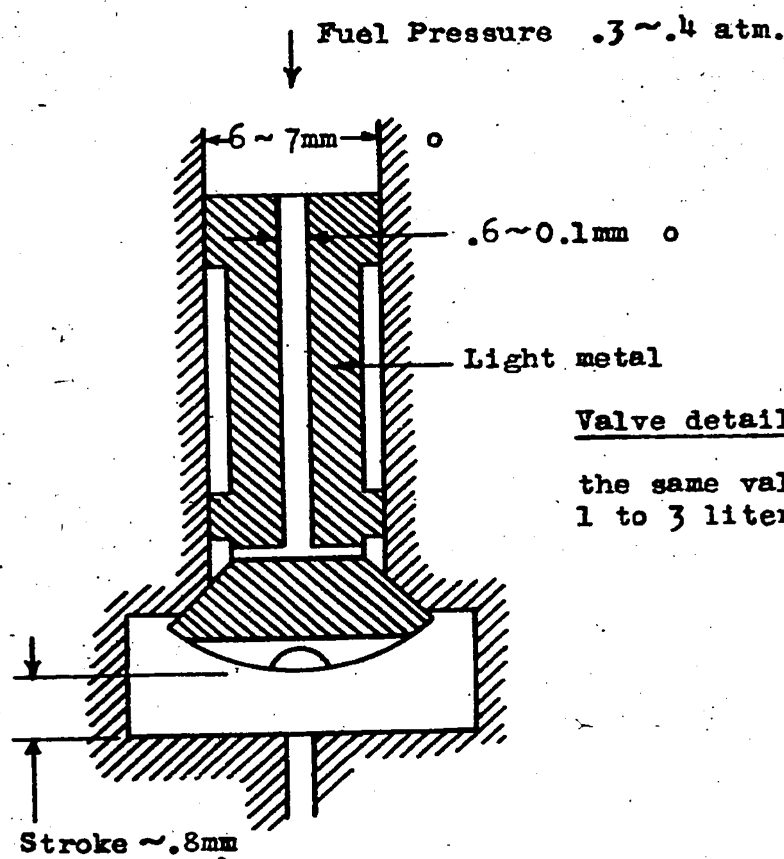



<u>HIRTH ARRANGEMENT OF PUMPLESS IGNITION</u>

Fig. 5




Schematic arrangement of the Hirth System of pumpless injection.




**Valve detail.**

the same valve was used for 1 to 3 liter displacement.

Drawn after freehand sketches by Dr. Bentele.
Fig. 6



remarkable results, it was possible, with an improved arrangement, to further increase the power output and speed to a satisfactory degree.

The regulation of the "R-fuel" quantity is simple; the R-quantity per stroke is determined by the dimensions of the components of the injection system and keeps automatically constant over a great speed range. On the test runs the engine operated smoothly and quietly with a minimum amount of R-fuel.

A report, dated December 10, 1942, sums up as follows:

"The pumpless injection of the R-fuel, compared with direct injection by pump and nozzle has in addition to improved economy also the advantage of better operating conditions. With the 1 liter cylinder, both methods are approximately equivalent in respect to power output and economy. While, however, with direct-injection the most favorable quantity of the R-fuel varies to a great extent with the charging pressure, and the air and cylinder temperature, with pumpless injection this quantity, apparently due to the heat accumulating effect of the ignition chamber, remains constant over wide pressure and temperature ranges.

"The control of the R-fuel quantity is possible by varying the supply pressure. This quantity automatically increases with lower speed.

"It is expected that the smaller and more constant R-fuel quantity improves the knock-conditions. Further investigations into this phase of the problem are in process."

It is understood that shortly thereafter this development at Hirth was terminated by the military authorities.

## CONCLUSION

It can be easily visualized that a fuel injection system not employing the expensive conventional injection equipment would make the small, high-speed Diesel engine an economic possibility.

Although the L'Orange system, in its present state, is not acceptable because of the high specific fuel consumption, there seems to be a way indicated to further improvements by the Hirth development.

The Hirth development proves that the proper control of one phase of the injection is possible and furthermore, strongly indicates that the shortcoming of the L'Orange system resides in the common control of both phases.

It seems to be within the range of possibility that a complete separation of the two functions of the system, namely injection into the ignition and into the combustion chamber, may permit full controll of the process in both phases and may thereby provide satisfactory performance of such an engine.

The design of the individual control means, could possibly follow the Hirth design, which seems to provide ample flexibility for adjustment to correct conditions.

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### AUTHORS

HOLLINGS - WITHERS (B.I.O.S.)
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| | B.I.O.S. Final Report Number | Item No. |
|---|---|---|
| HOLLINGS, H., HOPTON, G. U., SPIVEY, E. Lurgi high pressure gasification. (1946?) 75 p., incl. illus. diagrs. | 521 | 30 |
| HOWES, D. A. Interrogation of A. Bollhorn, E. Grages, and Dr. Gross of Deutsche Erdöl A.G., Berlin. (1946) 31 p. tables (part fold.). | 326 | 30 |
| HOWES, D. A. Kaiser Wilhelm-Institut für Strömungsforschung Abteilung Reibungsforschung, Berlin. Interrogation of Dr. Ing. G. Vogelpohl. August 11th - 14th, 1945. (1946) 16 p. | 325 | 30 |
| JONES, H. F., WEST, H. L. Wirtschaftliche Forschungsgesellschaft m.b.H., Hitzacker Depot. Investigation of the installation and interrogation of the personnel. July 8th-11th, 1945. 41 p., incl. illus., diagrs. (part fold.) | 120 | 30 |
| MINISTRY of Fuel and Power and British Intelligence Objectives Sub-Committee. Technical report on the Ruhr coal field, by a mission from the Mechanisation Advisory Committee of the Ministry of Fuel and Power... London: His Majesty's Stationery Office, 1946. Vol. I - (Vol. I: vi, 61 p., XXI tables (part fold.)) | 394 | |
| MORLEY, R. J. Winkler generators for manufacture of water gas, etc. (1946?) 40 p. illus. diagrs. | 333 | 30 |
| MURRAY, C. A. Chemische Fabrik Weyl A. G., Sandhoferstr. 96 - 106, Mannheim - Waldhof. 20th September 1945. 10 p. diagrs. | 332 | 30 |

| | B.I.O.S. Final Report Number | Item No. |
|---|---|---|
| PERRYCOSTE, W. B. C. German carbon electrode manufacture at Griesheim (I.G.F.) 1946. 14 p. | 338 | 21,22, 31 |
| PERRYCOSTE, W. B. C. German graphitising furnaces at Meitingen (Siemens Plania). 1946. 10 p. diagr. | 337 | 21,22, 31 |
| PURDON, F. F. Carbon electrodes, I. G. Farben, Griesheim. (1946) 8 p. | 258 | 22 |
| RANKINE, A. O. Developments of geophysical prospecting in Germany during the war. (1946) 7 p. | 334 | 30 |
| ROSE, J. D. Cyclopolyolefines. Miscellaneous report compiled from interviews with Dr. Reppe, Dr. Schlichting and Dr. Kroper (I.G.Farben, Ludwigshafen.) (1946) 6 p. | 352 | 22 |
| ROSE, J. D. Preparation of 5-diethylaminopentanol-2 by the Reppe process. Translation of a report by Dr. Hecht and Dr. Gassenmeier, Ludwigshafen. (1946) 6 p. | 353 | 22 |
| ROSE, J. D. Products formed by interaction of acetylene and amines. (1946) 5 p. | 359 | 22 |
| ROSE, J. D. Propargyl alcohol dehydration and oxidation to hexadiinediol. Interview with Dr. von Kutepow, Haup laboratorium, I. G. Farben, Ludwigshafen - 19th November, 1945. (1946) 7 p. diagrs. | 357 | 22 |
| ROSE, J. D. Recent advances in the chemistry of carbon monoxide. Translation of lecture by Dr. W. Reppe, Ludwigshafen. (1946) 18 p. diagrs. | 355 | 22 |
| ROSE, J. D. The synthesis of intermediates for polyamides on an acetylene basis (Translation of a report by Dr. W. Reppe, Ludwigshafen). (1946?) 19 p., incl. diagrs. (part fold.) ("Summary Report by Dr. W. Reppe, Ludwigshafen, 18.10.41," cf. p. 4) | 350 | 22 |
| SABEL, "Ten years of oxygen - gasification at Leuna" by Oberingenieur Sabel. Translated by D. G. Fraser and R. J. Morley. 1945. (1946) 36 p. diagrs. | 199 | 30 |

| | B.I.O.S. Final Report Number | Item No. |
|---|---|---|
| THOMAS, W. H., WITHERS, J. G. Deutsche Gasolin A. G., Hamburg - Germany. Lubricants. October 2nd - 10th 1945. (1946) 50 p. | 524 | 30 |
| THOMAS, W. H., WITHERS, J. G. Schlafhorst Chemische Werke G.m.b.H., Hamburg - Germany. Lubricants. October 2nd-10th (1945). (1946) 17 p. | 512 | 30 |
| WALLER, C. J. Dr. F. Raschig, G.m.b.H., Chemische Fabrik, Ludwigshafen: Coal tar distillation; chlorinated phenols; phenol-formaldehyde resins; synthetic phenols. (1946) 25 p. diagrs. | 507 | 22, 30 |
| WALLER, C. J., MURRAY, C. A. Gesellschaft fur Teerverwertung m.b.H., Duisberg - Meiderich, and Castrop - Rauxel. (1946?) 58 p. illus., diagrs. | 450 | 30 |
| WATT, W.; CLEWS, F. H.; MILLER, R. B.; WALKER, J.; WHEATLEY, C. H. High temperature refractories and ceramics. (1946) 53 p. | 465 | 21, 22 |
| WEST, H. L. Wirtschaftliche Forschungsgesellschaft m.b.H. Interrogation of Herr Helmut Plote of the Construction Department (evacuated to Hitzacker), July 8th - 11th, 1945. 22 p. | 122 | 30 |
| WITHERS, J. G., THOMAS, W. H., ROBINSON, E. B. Samples of petroleum products collected from the Hamburg, Hannover, Bremen and Kiel areas. September 31st - October 29th, 1945. (1946) 40 p. | 510 | 30 |
| WITHERS, J. G., WEST, H. L. Ruhr-Chemie A. G., Sterkrade, Holten. Interrogation of Dr. O. Reolen at Wimbledon, November 15th and December 20th, 1945. (1946) 27 p. diagr. (Contents: "Translations of three documents prepared by Dr. Roelen," translated by K. G. C. Kirkpatrick.) | 511 | 30 |

| | FIAT Final Report Number |
|---|---|
| BOGUE, L. E., HOERTZ, Norman. Air filters and oil filters for engines. 1946. 15 p. diagrs. | 600 |