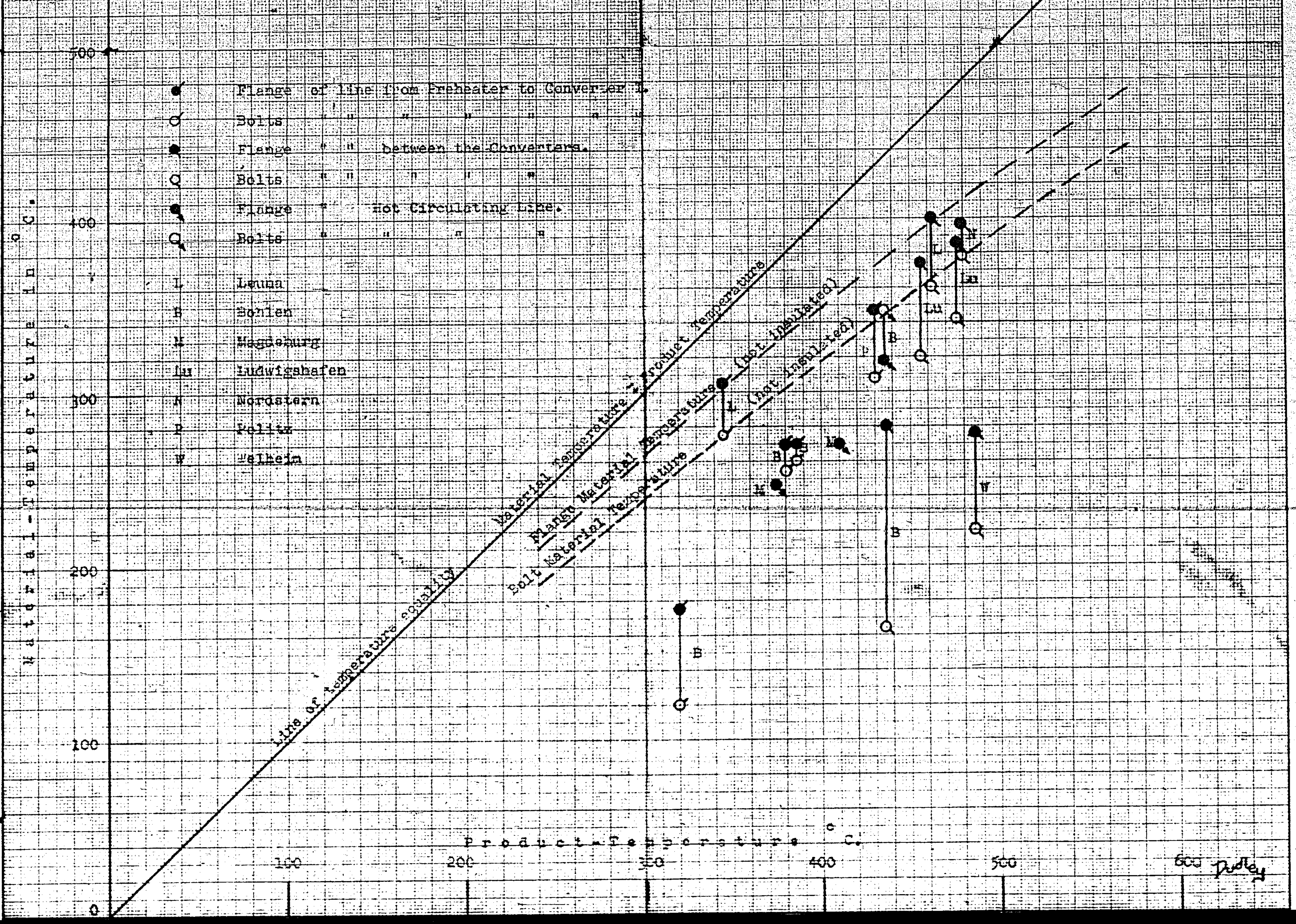
Temperatures of Flange Connections in Relation to Product Temperatures.



C.H. Equipment

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

T-230
199 KCBraun
2-5-47

INNER INSULATION OF HIGH PRESSURE CONVERTERS.

Ludwigshafen, 27 Jan. 1941

(Letter from I.G.F. to A.N.I.C. Milan, Italy.)

In reply to your letter of the 24 December, 1940, we wish to inform you that until recently we have used two different methods of insulating the inside of high pressure converters:

1). The tamping method, in which little cement and water but much asbestos is used, and

2). The casting method, in which much cement and water but little asbestos is used.

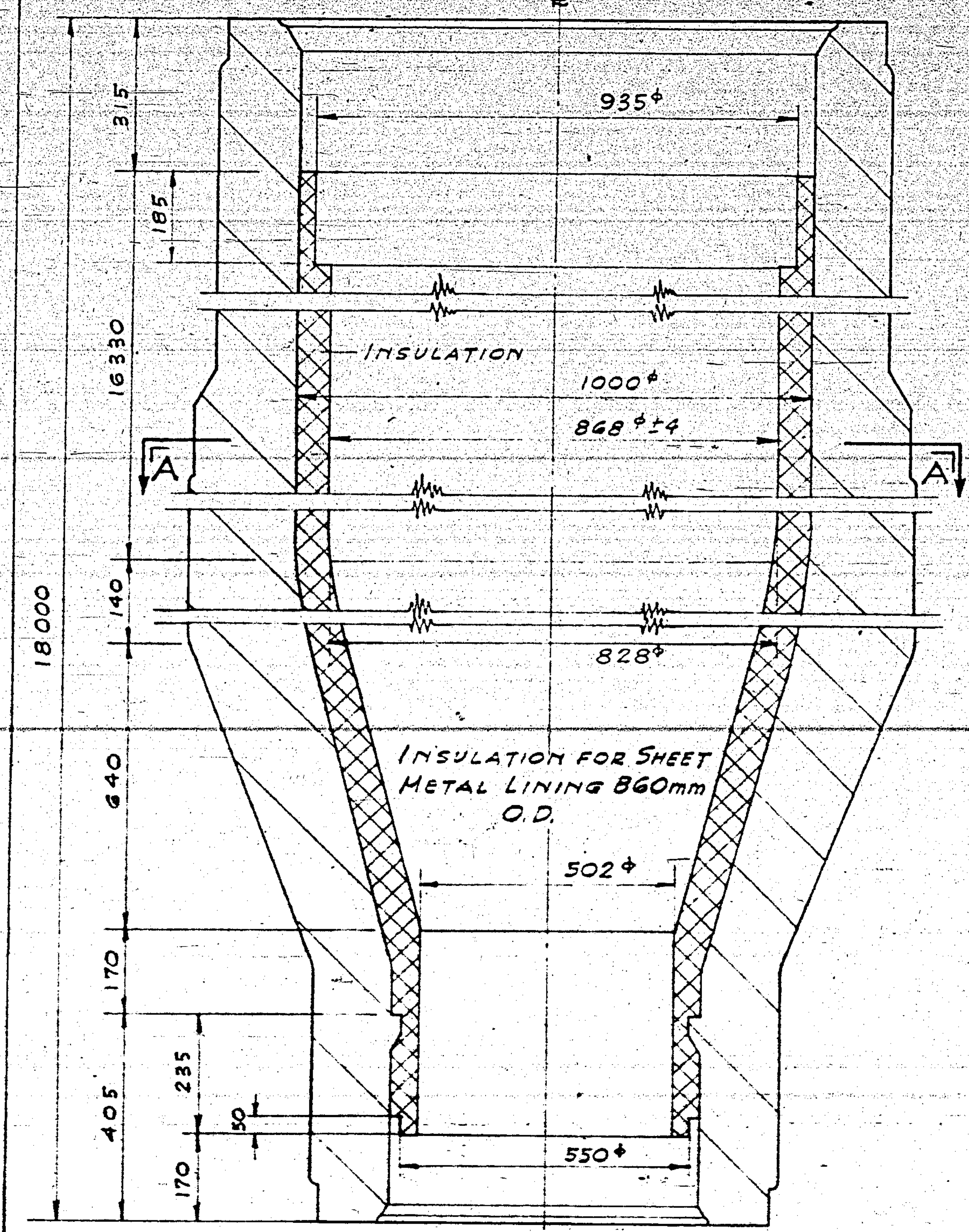
We preferred the tamping method for the insulation of vapor phase converters and the cheaper casting method for the liquid phase converters. The reason for this was that in the tamping method possible cavities and holes in the insulation could, in general, be more easily avoided. However, since there is a possibility that possible holes in the insulation may be filled in by the product in liquid phase converters, the casting method was first used for insulating the less sensitive liquid phase converters. Later, we also used the casting method for insulating the vapor phase converters, without any noticeable detriment.

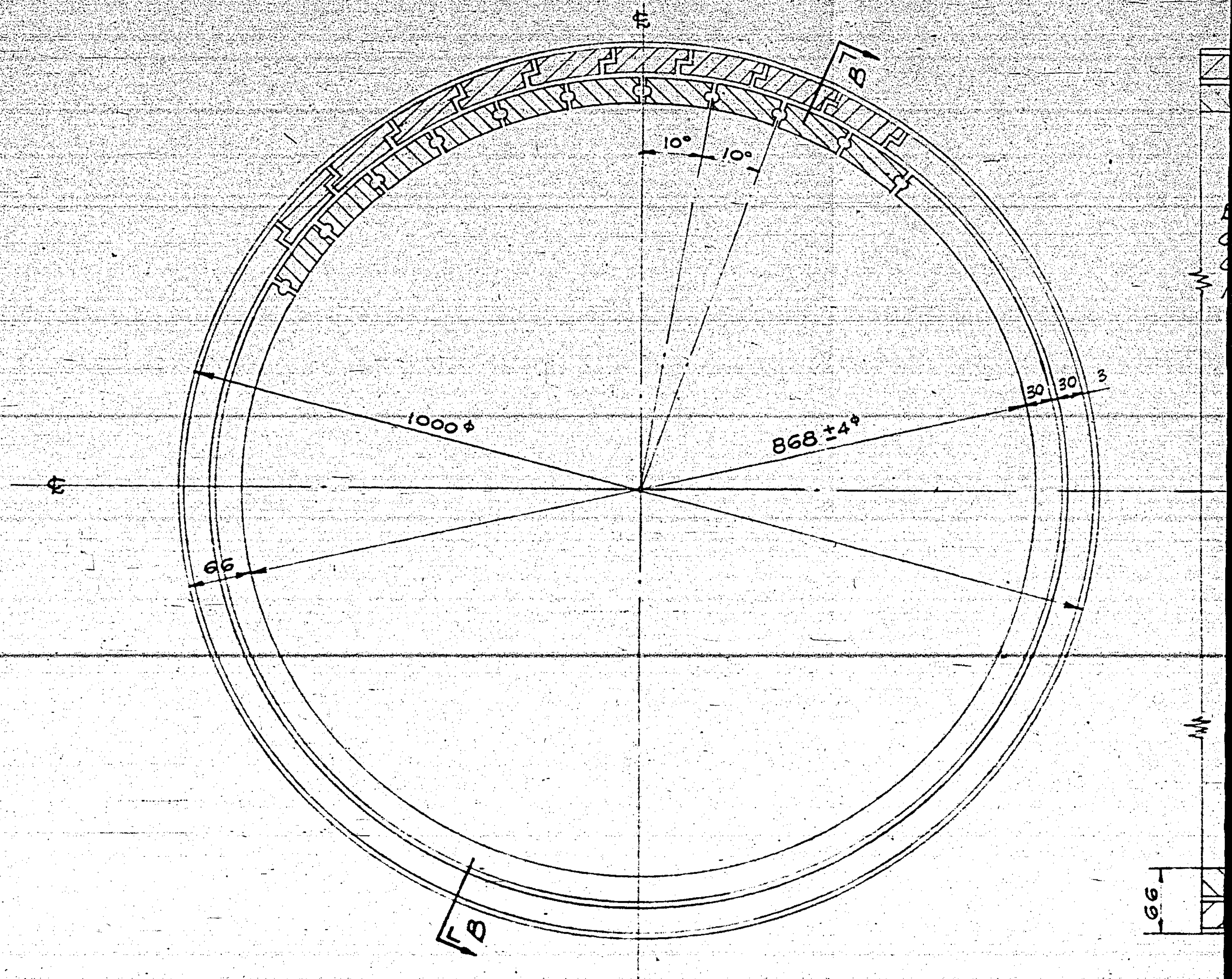
In consideration of present difficulties of procuring suitable asbestos, we have recently given up the cement-asbestos insulation and, after exhaustive operating experiments, now use an inner insulation of built-up insulating bricks. We have found that a light refractory brick made by Firma Annawerk, Schamotte und Tonwarenfabrik A.G. Oeslau/Koburg, is especially suited. This brick has the brand designation L O 9/26/2.

The attached drawings show the construction of the insulation. Drawing N 9135-2 shows the insulation of a vapor phase converter, while drawings N 9232-2 and N 3777-4 show the insulation of a liquid phase converter. The mortar consists of alumina cement and refractory grit (ground brick) in the proportion of 1:2.

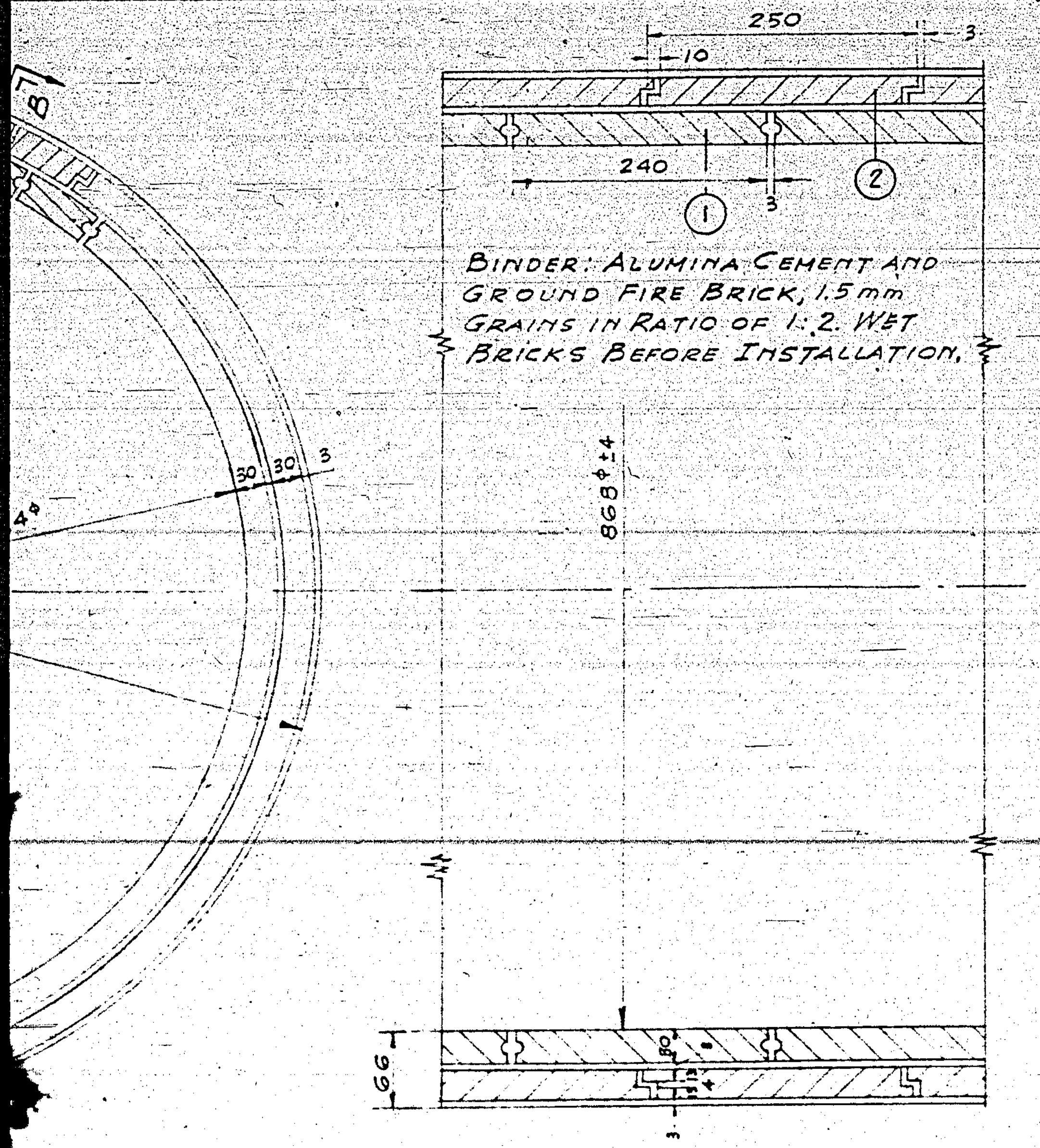
The coefficient of thermal conductivity of this brick was determined in the laboratory at 325 atm. hydrogen pressure to be about 0.675 kcal/h/m/°C.

In your letter, referred to above, you mention experiments with an insulation of a cement containing alum with diatomite and slag wool. We shall appreciate hearing of the results of these tests.


℄
935ϕ
315
185
16330
INSULATION
1000ϕ
868ϕ±4
A
A
140
18000
828ϕ
INSULATION FOR SHEET
METAL LINING 860mm
O.D.
640
502ϕ
170
235
405
30
170
550ϕ

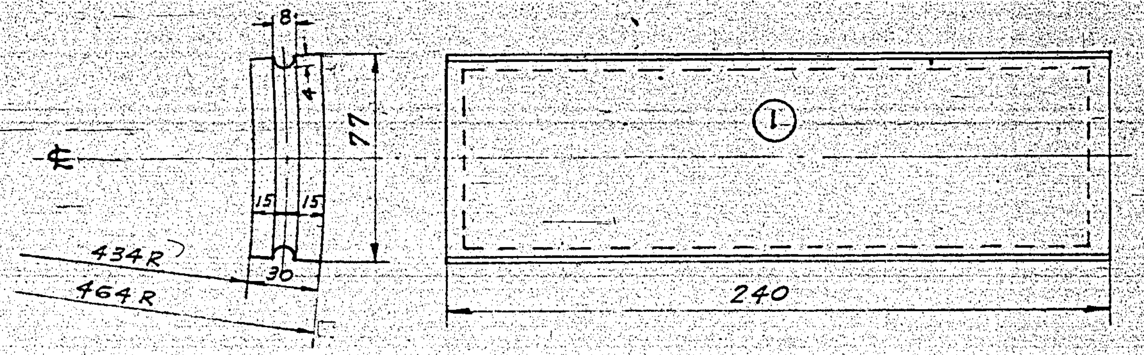

℄
℄
B
B
10°
10°
1000ϕ
868±4ϕ
30
30
3
66
66

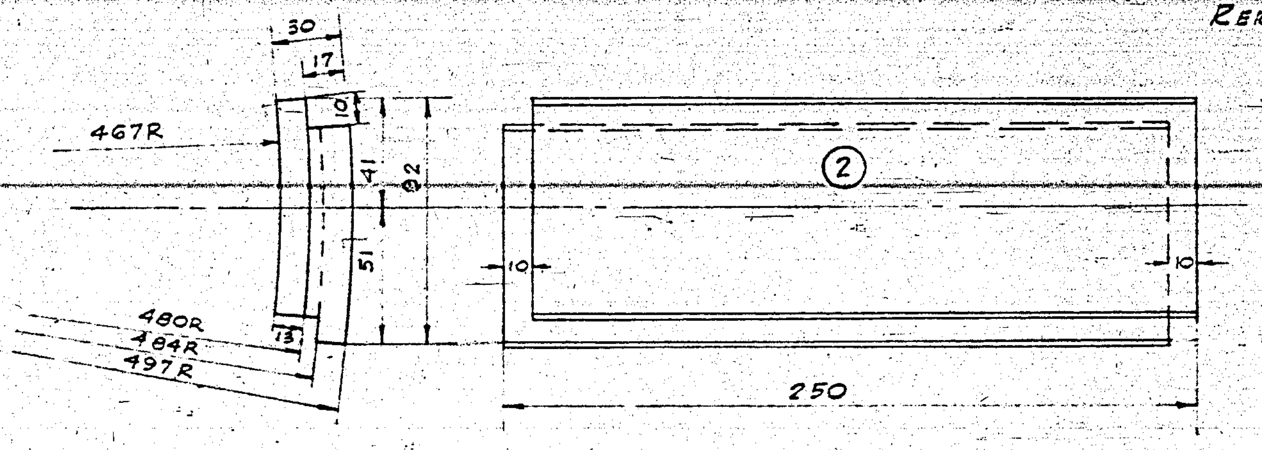
SECTION "A-A"



BINDER: ALUMINA CEMENT AND GROUND FIRE BRICK, 1.5 mm GRAINS IN RATIO OF 1:2. WET BRICKS BEFORE INSTALLATION.

SECTION "B-B"



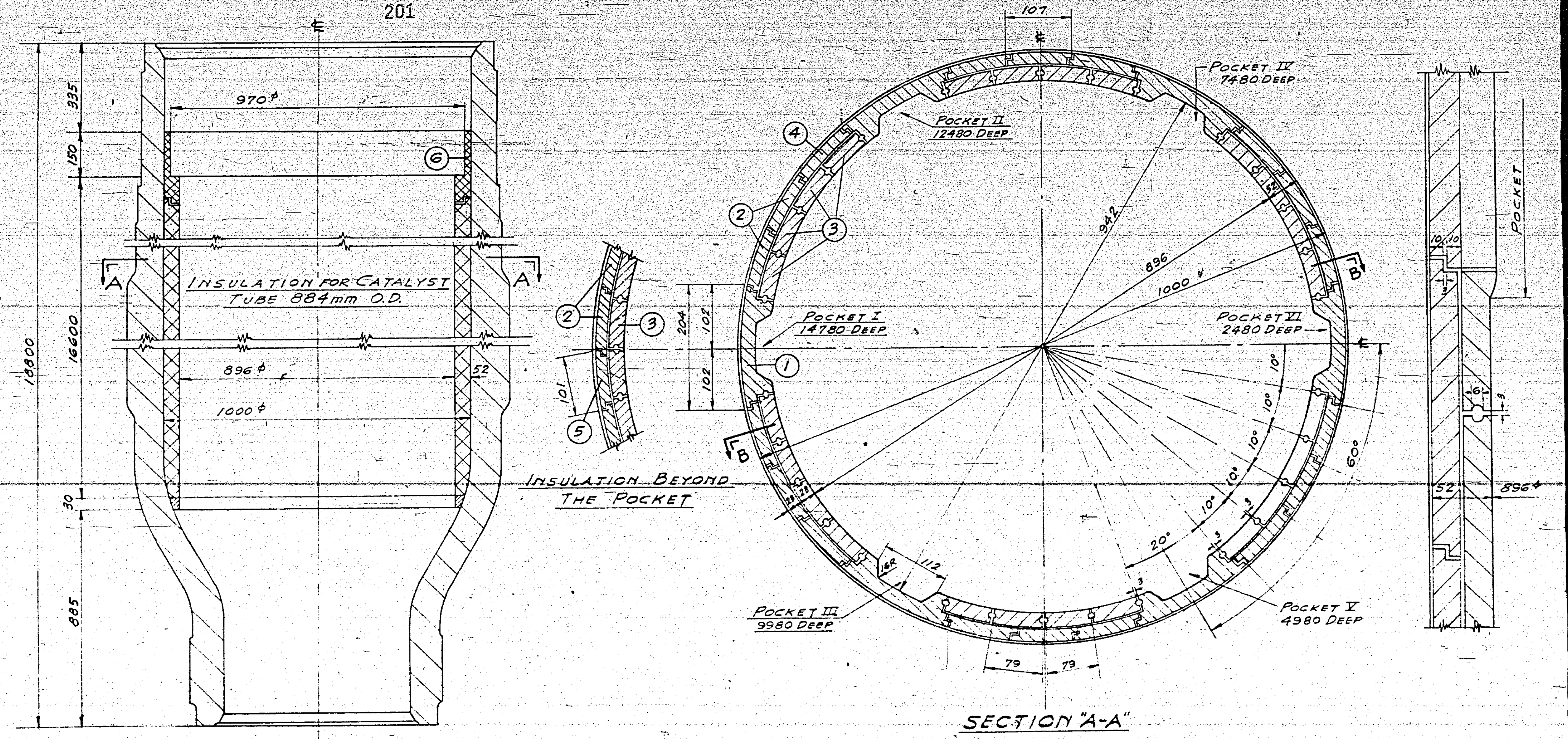


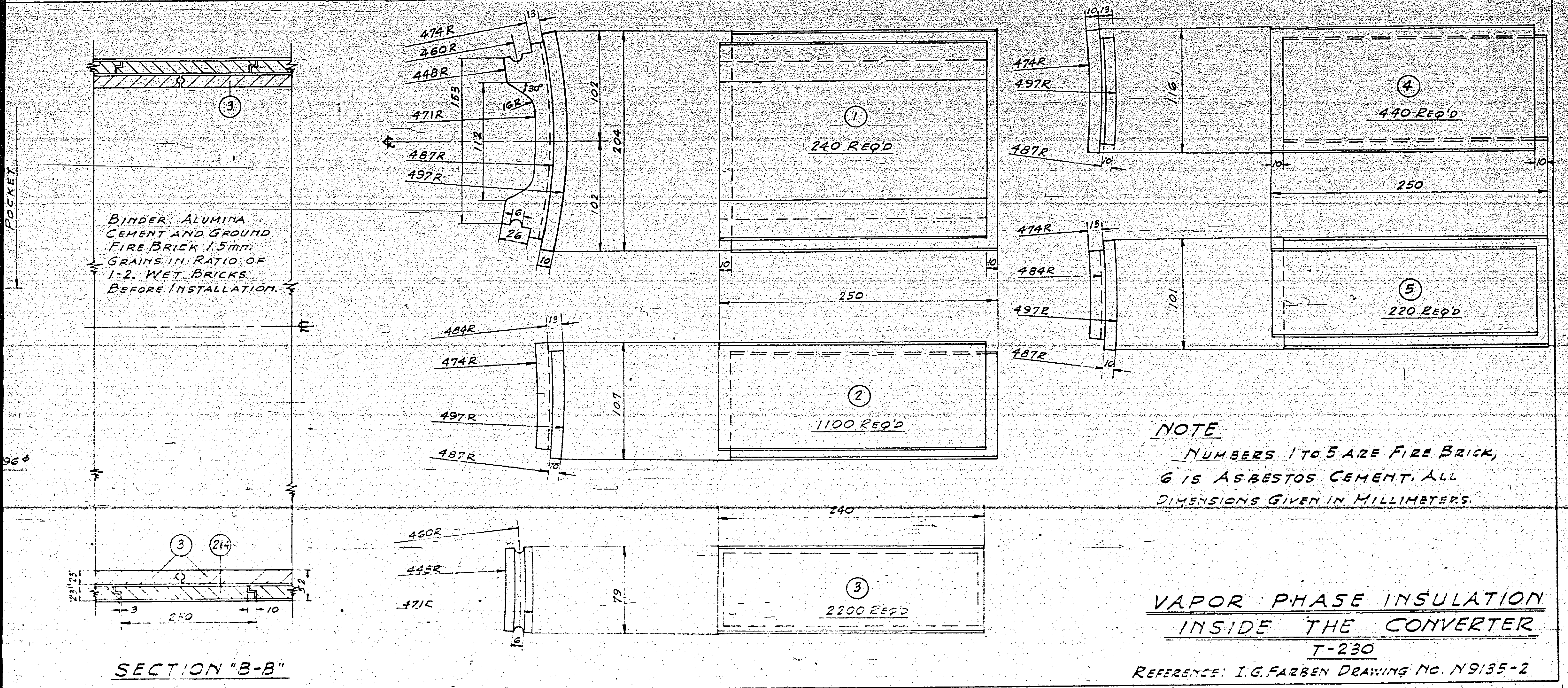
NOTE:
2600 NUMBER 1 BRICKS PER CONVERTER
2600 " 2 " " "

ALL DIMENSIONS GIVEN IN MILLIMETERS

REFERENCE: I.G. FARBEN DRAWING NO. N-9232-2

LIQUID PHASE INSULATION INSIDE THE CONVERTER
T-230


INSULATION FOR CATALYST TUBE 884mm O.D.
970 ϕ
896 ϕ
1000 ϕ
52
335
150
16600
16800
30
885
A
A
6
INSULATION BEYOND THE POCKET
2
3
5
101
204
102
102
107
POCKET IV 7480 DEEP
POCKET II 12480 DEEP
POCKET I 14780 DEEP
POCKET VI 2480 DEEP
POCKET III 9980 DEEP
POCKET V 4980 DEEP
942
896
1000
4
2
3
1
B
B
10°
20°
60°
3
16R
112
28
28
79
79
SECTION "A-A"
POCKET
10
10
6
3
52
896ϕ



BINDER: ALUMINA CEMENT AND GROUND FIRE BRICK 1.5mm GRAINS IN RATIO OF 1-2. WET BRICKS BEFORE INSTALLATION.

SECTION "B-B"

NOTE

NUMBERS 1 TO 5 ARE FIRE BRICK, 6 IS ASBESTOS CEMENT. ALL DIMENSIONS GIVEN IN MILLIMETERS.

VAPOR PHASE INSULATION
INSIDE THE CONVERTER
T-230

REFERENCE: I.G. FARBEN DRAWING NO. N9135-2

Fischer - Tropsch

TOM Reel 176
Frames 0043-
0050

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

202 T-231

Oppau, Jan. 6, 1941

PRINCIPLES OF THE OPPAU PRESSURE GAS CONVERTER

The different constantly recurring inquiries about the Oppau pressure conversion indicate the uncertainties existing in the understanding of this converter as well as the possibilities of gas converter operating under pressure. We have therefore attempted to give below a condensed summary of the basic facts to be considered here.

The conversion of carbon monoxide with hydrogen may be expressed by the equation

$$CO + H_2O = CO_2 + H_2 + 10{,}550 \text{ kcal/mol at } 0^{\circ}$$

$$(\text{or} \quad 9{,}850 \text{ " " " } 400^{\circ})$$

The equilibrium constant of this reaction is given by

$$\frac{\frac{p_{CO_2}}{p} \times \frac{p_{H_2}}{p}}{\frac{p_{CO}}{p} \times \frac{p_{H_2O}}{p}} = \frac{p_{CO_2} \times p_{H_2}}{p_{CO} \times p_{H_2O}} = \frac{(CO_2) \times (H_2)}{(CO) \times (H_2O)}$$

The total pressure $p = p_{CO_2} + p_{H_2} + p_{CO} + p_{H_2O} + p_{N_2}$

therefore disappears from this equation and the percent of the components by volume may be substituted for their partial pressures $\left[\frac{p_{CO_2}}{p}\right]$. This means, however, that pressure will not cause any change in the equilibrium constant, and the conversion will thereby be in no ways affected.

The equilibrium constant is however extraordinarily strongly affected by temperature changes (according to Maurer and Bischof):

at 500° it is around 5.1
at 400° it is around 12.7
at 300° it is around 43.5

In practice we are interested in the range of up to about 400°, because the reaction is in most cases finished between 400° and 420° C. If 101 m^3 of steam be added to 100 m^3 of raw gas composed of 5% CO_2, 38.0% H_2, 38.6% CO and 10.4% N_2, we will get, for the moist gas

$$\frac{CO_2 \times H_2}{CO \times H_2O} = \frac{2.5 \times 18.9}{19.2 \times 50.2}$$ and by a rearrangement based

on the fact that the same volume of $CO_2 + H_2$ is formed as the volume of $CO + H_2O$ which disappear,

$$\frac{CO_2 \times H_2}{CO \times H_2O} = \frac{20 \times 36.4}{1.7 \times 32.7} = 13.1$$

which corresponds roughly to the equilibrium at 400°. In a conversion up to

$$\frac{1.7 \times 100}{100 - 32.7} = 2.5\%$$ CO in dry finished gas, there will remain $\frac{32.7 \times 100}{50.2} = 65\%$ undecomposed steam, and the amount of steam to be supplied for the conversion is about 2.9 times the theoretical amount.

Were it possible to end the reaction at 300°, much steam could be saved. For the same gas mixture as in the example above, 41 kg, or abt. 55 m³, would be sufficient for 100 m³ of steam, and from the expression for moist gas, $\frac{CO_2 \times H_2}{CO \times H_2O} = \frac{3.2 \times 24.5}{24.9 \times 35.5}$ we would get for equilibrium conditions at 300°;

$$\frac{CO_2 \times H_2}{CO \times H_2O} = \frac{25.9 \times 47.2}{2.2 \times 12.8} = 43.5,$$

or again $\frac{2.2 \times 100}{100 - 12.8} = 2.5\%$ CO in the dry synthesis gas. The undecomposed steam would be $\frac{12.8 \times 100}{35.1} = 36\%$, or reduced to 1.6 times the amount required theoretically.

Unfortunately, this chance to save steam does not exist, because the available catalysts permit operations only down to 400° C. One must, therefore be resigned to use a large excess of steam, and try to overcome this disadvantage by a maximum utilization of the great heat of the reaction, where the heat of the undecomposed steam is used. It may be mentioned in this connection, that in view of the large amounts of gas to be converted, a small improvement in steam economy will result in considerable savings.

The undersigned has succeeded in 1929 in introducing a new water dispersing nozzle and in improving the distribution of water in the hot gases of the low pressure conversion, and in this way reduce the steam consumption to 110 g per m³ of the original gas, using the excess reaction heat for vaporization, which resulted in a yearly saving of RM 200,000 with a total gas consumption of 35,000 m^3/hr. The optimum steam consumption of the low pressure conversion was in this way reached, and no further improvements have been found possible, in spite of the several structural changes introduced. The reason for it was the great amount of heat lost in the central evaporation and cooling of the earlier gas producers, because of the long pipe lines and large surface of the equipment, with such a large resulting radiation loss, that the recovery of the excess heat of vaporization of steam could not be done sufficiently perfect. (v.fig.1).

These facts have not been sufficiently considered in the past, and a cure was sought exclusively in an increase of pressure (Fauser, et al). The claim has been put forward, that pressure gasification would pay for itself simply by savings on compression. True, the compression costs are lowered by the fact that the carbon dioxide formed in the reaction (one volume of CO will produce 1 vol. H_2 and 1 vol. CO_2) will be reduced in the above example by $(25.9 - 3.2) \times \frac{38.6}{24.9} = 35.2\%$ which will have to be compressed to the pressure required to scrub out the CO_2; but the steam used, received at 2.5 atm., must be compressed to 30 atm. If the steam is energetically correctly evaluated, only 20 - 30% of the saving of compression will be realized, and out of this saving will come the higher installation costs.

Pressure gasification can only then result in real saving, when, in agreement with the above reasoning, the total excess of the reaction could to a greater extent be used to reduce the steam consumption.

For this reason the undersigned has brought out in 1934 a new pressure gasification system, and further developed it subsequently. Here the central vaporizer and cooler were omitted, and each converter supplied with its own original vaporizer, which also acted as a cooler, while the conversion was carried out in a tubular catalyst converter under an increased pressure (of about 30 atm.). The increased pressure permits the use of smaller equipment with increased capacity, smaller surfaces, and therefore smaller radiation losses, while the tubular catalyst converters transmitted the heat of the reaction directly to the gas mixture inside the converter, which resulted in a most loss-free utilization of the heat of the reaction, while maintaining the most favorable temperature of the reaction. The gas mixture from the vaporizer (foam cooler, discussed below) enters from below the space inside the tubes filled with the catalyst, rises, and descends outside the catalyst tubes, nearly at the reaction temperature (v. fig. 2).

Eng. Markert has subsequently suggested to place a catalyst layer converter back of these tubular converters, directly flanged to the latter, forming in this way a closed system. This combination permits the operation of the tubular converter at a very high load, without paying much attention to the final analysis of the product, because the reaction rate far from equilibrium conditions is very high, while the remaining conversion to the desired CO concentration (1-2%) prodeeds in the catalyst layer converters at a low reaction rate at a very low velocity of the gas mixture. An opportunity is here presented to equalize the temperatures between the two converters by an injection of water which has however so far been found unnecessary. This combination converter system results in a very high gas throughput (25,000 m^3 per hour through the system), or a small installation cost, while the conversion is good and the heat of reaction is utilized to a maximum. This however justified only one side of compression gasification, and there still remained to find a solution to the far more important and difficult problem of a more perfect utilization of the residual heat of the steam in moistening the gaseous mixture.

The moistening of the gas mixture was done in the past by direct spraying of an excess of cooling water into the raw gas in a cooling tower, removing the residual heat of steam by cooling; the gas was sprayed in a vaporizer with the hot circulation water from the cooler, and so moistened. (fig. 1). Such a process can be directly introduced into the Oppau system, but, as mentioned above, permits no adequate utilization of the excess heat of the catalytic process.

The need of two heat exchangers for the saturation of the raw gas results in a two-fold temperature difference (once between the finished gas and circulation cooling water, and then between the raw gas and the circulation water), which is further magnified by the cooling losses connected with the water circuit.

There is the additional disadvantage from the use of the circulation cooling water in that, as soon as the temperature of the converted gas at the inlet to the cooler exceeds the saturation temperature of the catalyst, the circulation water of the converted gas will evaporate the steam, carried upwards, and interfere with the heat exchange. The sensible heat of the converted gas above the saturation heat can be but poorly utilized.

This disadvantage could be avoided by substituting a tubular cooler for the scrubber-cooler, but because of the indirect cooling this will require a greater temperature difference between the converted gas and the circulation water, which could be equalized only by quite a disproportionately large cooling surface.

This was the reason why the undersigned has devised an entirely new "foam evaporation cooling principle" (patent applied for in Germany), which solves the problem of saturating the incoming gas in an entirely new way. The water-containing converted gas is cooled indirectly in a tubular cooler, but not only with water, but simultaneously with water and gas. One is normally anxious not to let the presence of gas interfere with heat transfer of liquids, but here gas is deliberately added to the cooling water to improve the heat transfer. This is done by introducing gas into the cooling water by means of nozzles, or lately with perforated plates, at such a high velocity that solid foam is formed inside the whole cooling tube, from top to bottom. This foam is in violent motion. Conditions on the cooling side are similar to those in a distillation column which is boiling over.

At every place outside the individual cooling tubes there is just foam consisting of tightly packed thin walled water bubbles, occluding the introduced raw gas, while the cooling water introduced forms a very small coherent sheet under the gas inlet.

The heat transfer therefore proceeds from the cooling tubes to the skin of the bubbles in immediate contact with the tubes, and from there on from one bubble to the next, while the water skin of the bubbles evaporates into the gas they enclose. This not merely increases extraordinarily the surface of evaporation, but produces simultaneously in every part of the cooling tube the opportunity to immediately convey to the outside any heat liberated by the evaporation of the water from the skin of the bubbles into the gas. The bubbles increase in volume as a result of evaporation and expansion resulting from the raised temperature, and rise, while the skin of the bubbles becomes ever thinner, until it finally becomes completely absorbed into the raw gas and vaporized. The high gas velocity causes the bubbles between the tubes to boil and to effervesce, producing an intense mixing action, and thus also a uniform heat exchange over the whole cooler. The bubble skins are very thin, the surface of the bubbles and the volume of the occluded gas are very large, the rate of evaporation is very rapid, and the heat transfer is two to three times greater than when water is used without gas. One can in this way operate with no excess of water, i.e. one may use only as much water as must be vaporized. The recirculation of the cooling water, with all its disadvantages is therefore omitted, because cooling and evaporation are carried out simultaneously in the same apparatus, and there is the additional saving of a cooling tower and of the heat losses connected with it, and the whole installation correspondingly simplified. With the same temperature of the finished gas the outlet temperature of the raw gas is about 5° higher with this foam arrangement than with the usual cooler-evaporator principle, and about 8° than higher with the indirect cooler-vaporizer, because the heat losses are appreciably lower, there is but a single heat transfer from the finished gas to the raw gas mixture, and a much more intimate contact between water and gas.

In the temperature range used, a rise of 5° of the raw gas means an additional water absorption of 100 g/m^3, and the saturation of the raw gas is much more favorable than in the other saturation processes, in addition to the best utilization of the heat of the finished gas above the saturation temperature.

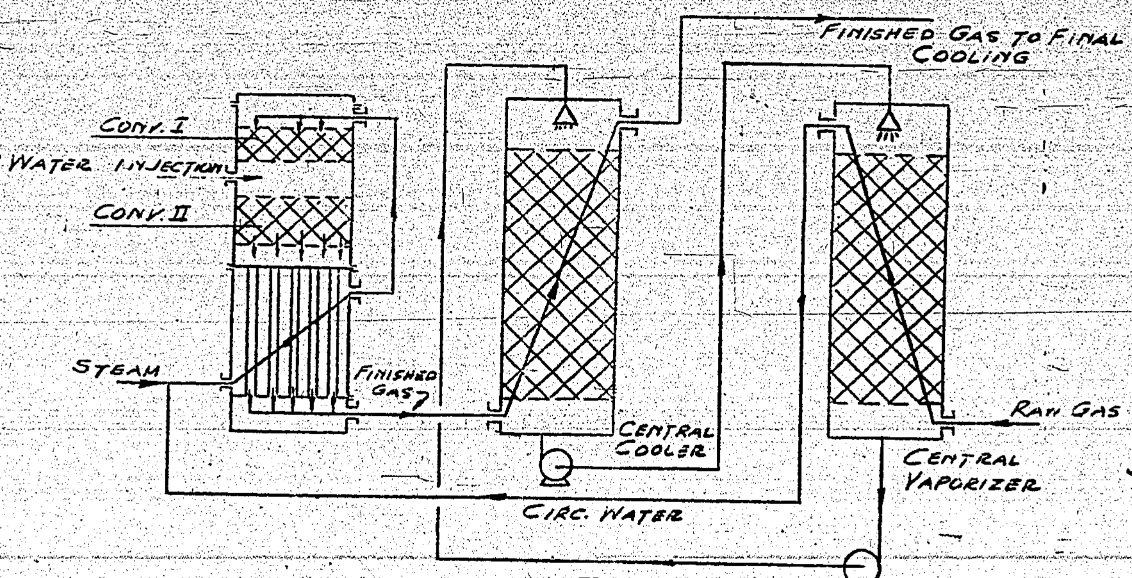
The action of this new foam cooler has been confirmed in numerous tests and also definitely proved by Mr. Markert's computations, and every foam cooler may be calculated in advance for any requirements. Tests have shown, that the heat exchange is so favorable with this cooler, that the heat supply can be lowered even below the amount required for the decomposition. Tests have also shown, that a change of the height of the foam layer affects very readily the operations of the converter, while this may be but little, or not at all done with the circulation cooler and vaporizer system, because this system produces the best results at some perfectly definite amount of circulating water, and moreover, with the large amount of cooling water, any changes will have but little effect and then only after a longer time.

This new foam cooler solves also a second problem, the utilization of the residual heat of the steam in the finished gas, and produces, coupled with the converter design described above, a pressure converter gas system, which results in considerable savings in operation and construction costs over the other systems operating at either low or higher pressures.

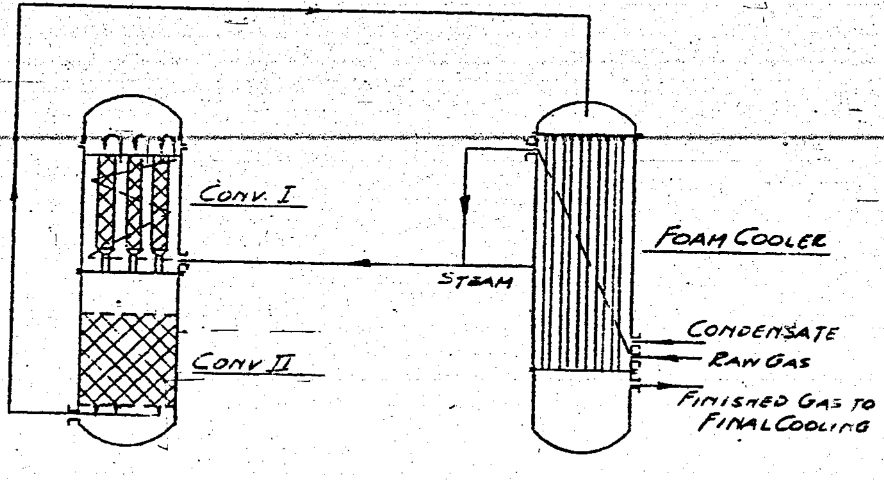
The Oppau experience has shown, that the costs of a new gas conversion pressure installation pays for itself in savings over low pressure installations in the course of 1 1/2 years.

/s/ undecipherable.

W. M. Sternberg.



LOW PRESSURE CONVERSION
FIG. 1



OPPAU PRESSURE CONVERSION
FIG. 2

THREE PAPERS ON THE HARDENING OF PLUNGERS

(From M. Pier's private files)

1. Discussion of Experience with Hardened Plungers and Piston Rods for High Pressure Machines.

I.G. Farbenindustrie A.G.
"High Pressure Experiments"
Ludwigshafen (Rhein)
Bau 558

March 3, 1942

It has been intended to use at our plant principally machine parts hardened by the Mannesmann method, as well as some nitride hardened plungers and rods. To confirm this decision, the hydrogenation works in Pölitz repeated a test with a thus treated plunger, which proved, however, to be a failure.

After this, we contacted the Gelsenberg-Benzin A.G. and our engineers Thieme and Wöhner have discussed the problem with their men. We have learned the following at this occasion:

1). Nitride hardened plungers and rods have proven with very few exceptions entirely satisfactory in Gelsenberg, but with their deliveries from one single manufacturer, the times of delivery are so unpermissibly long as to endanger continued operations.

2). Mannesmann hardened plungers and rods have not been found satisfactory in Gelsenberg; the inside cooled plungers in particular have cracked and broken after a certain operating time. A few examples of this kind were cited. The not-hollowed rods were already markedly warped before installation (piston rods of pressure release machines). After some operating period such rods assumed a curvature of up to 4 mm.

3). Autogenous hardened rods and plungers have been introduced in Scholven operations for some time, and have been found satisfactory, except for plungers of pumps for the copper solutions.

On the strength of this experience, Gelsenberg has largely turned to autogenous hardened piston rods and plungers.

Prior to making a similar decision for our plant we wish to request you for a discussion of the subject with all hydrogenation works to permit us to select the best modern method.

....

/s/ Upper Silesian Hydrogenation Works A.G
by W. Krönig; O.I. Fusshüller

2. Gelsenberg Benzin A.G.
Gelsenkirchen, 15

High Pressure Experiments
P/Leuna 558

April 4, 1942

In general, piston rods and plungers surface hardened on the stuffing box bearing surface are used for gas and liquids in high pressure machines at 300 and 700 atm. The operating experience with such rods and pistons differs very much depending on the material used. The Upper Silesian Hydrogenation Works which are about to order or to put in operation a large machine installation have requested us to collect opinions of hydrogenating works on this point.

We request you therefore to inform us your experience with:

1). Nitrided rods,

2). autogenous hardened rods, which are frequently used today,

3). case hardened rods, indicating the depth of hardening,

4). rods from the firm A. Mannesmann, Remscheid, which for some years no longer uses the so-called "Verbundhärtung" process, but the usual case-hardening methods,

5). rods from other materials (hardchromed, depth-chromed, etc), are not hardened.

These parts are to be used in compressors, gas circulation pumps, piston pumps, coal paste and catalyst pumps, hot circulation pumps, pressure release machines, etc, and we request that you make suggestions about the best methods for surface hardening. Your communication should also contain information on the length and number of strokes, also the rod velocity, pressure, principal dimensions, nature of the feed, lubrication or flushing of the stuffing boxe, operational conditions, etc, whatever is necessary to judge results. As far as possible prices, procurement possibilities or times of delivery and names of manufacturers are to be given.

....

I.G. Farbenindustrie A.G.

/s/ Holdermann, I.V. Schönjahn

1) Gelsenberg A.G.
2) Hydrogenation Works Scholven A.G.

To Messrs. Hering, Schönjahn

5/21/43.

Mannesmann Plungers for Paste Presses at 700 Atm.

Mannesmann Verbundstahl is always recommended for the bearing surfaces of plungers in the paste pumps, because it has been found by the I.G. Farbenindustrie A.G. to be best suited for this purpose, because the depth of the hardened coat permits repeated grinding if necessary. For this reason, Silesia Benzin also specified Mannesmann plungers for the 700 atm. gas compressor, although they have been used in the past for paste pumps, and not with machines with a six times greater r.p.m. For the purpose of a test, a Mannesmann plunger was installed in a 700 atm. booster compressor at the Pölitz Hydrogenation Works, where it thus was operated under conditions similar to those in the Silesia-Benzin. The hardeness of the bearing surface was entirely satisfactory, but the plunger never-the-less, after 1000 hours of operation had a damaged spot, resulting from a fault of the original material, as established by the manufacturer's test, and from that spot a crack developed towards the cooling boring, which worked its way to the outside.

An autogenous hardened experimental plunger was then installed for a test (the originally received plungers were nitride hardened), but this one also became damaged after practically the same operating time. Studies of the causes of the damage have not as yet been concluded.

The disadvantage of the nitriding process is known to consist in the thinness of the hardened layer, so that with the least damage to the rod or the plunger they must be returned to the manufacturer for re-nitriding. The question of the best hardness for 700 atm. compressors still remains open. Some nitrided piston rods and plungers have achieved a long operating time, but repetition of the nitriding has been necessary in our case as well. The experience with the autogenous hardening developed only within the last few years does not extend over sufficient time. On the other hand the suitability of the Verbandstahl may be judged from several years of successful operation of the 700 atm. paste pumps.

We request you therefore to inform us from your operating records the longest operating time of the Mannesmann plungers, by how much the diameter had to be reduced in the succeeding regrinding, and whether there were breakages. The operating time until the necessary regrinding is much affected by the paste pump feed; it can therefore not be used for booster compressors, which operate only with pure gas.

....

/s/ Mineralöl--Baugesellschaft N.H.N

W. M. Sternberg

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-233

KCBraun
2/17/47

WEAR-RESISTANT RETURN BENDS

By D.I. Schappert, Ludwigshafen,
7 March, 1942.

The attached sketch illustrates the development of the wear resistant return bends. After the first failure of the Scholven coal stall, the lower bend, which was subjected to the greatest wear, was replaced by a thick-walled return bend with right angle bases but without inserts, at first. The wear was little affected thereby. Return bends with wear-resistant inserts were then adopted.

FIG. 1 - A wear-resistant insert of Granitherm, by Ruhrstahl, is inserted into the horizontal leg. The bushings in the inlet and outlet tube connections are also of Granitherm. The horizontal insert in the bend is locked in by a screwed plug, welded in. The return bend proper is heat treated and then welded to the 2 shanks of the hairpin coil. Because the 2 inlet and outlet nipples are not at right angles to the bend piece, but at 8° from the vertical, the junction point with the unprotected connecting tubes would still show appreciable wear, see sketch. In any case, however, the life of a hairpin coil was increased from about a month to about 1/2 year by this construction.

FIG. 2 - To eliminate this weak point, vertical Granitherm bushings were inserted in the ends of the connecting hairpin tubes. These return bends are still used in Scholven today, though replacements are made according to Fig. 4. However, this coil is still not entirely protected against wear, because the junction point between the upper end of the vertical bushings and the unprotected tube still wears. However, the life of a hairpin coil according to Fig. 2 is about 2 years.

FIG. 3 - This construction was, therefore, adopted for Nordstern, Pölitz and Upper Silesia, now at 700 atm. The inclined bushings in the inlet and outlet shanks were replaced by vertical bushings.

FIG. 4 - Instead of the inlet and outlet curves with large vertical components, cubical parabolic curves were used, similar

to the transition curves used in railroad track work.

After almost 3 years of operation at Nordstern, no wear has been observed there. This was determined on an unprotected return bend in the middle pass, in which even the turning grooves (tool marks) in the material could still be observed after over 1 years' operation. Nordstern, therefore, expects to get away from special return bends entirely. Upper Silesia has ordered return bends, but does not intend to use them. Gladbeck also has ordered hairpin coils with normal return bends.

Return Bends for Gas Preheater Hairpin Coils for 325 and 700 Atm.

Leuna-Werke, 14 May 1942.

These return bends were designed by Chief Engineers Schön and Schiwy, Scholven. Because very serious damages were once caused by normal 180° return bends, special return bends seemed the only solution. The design drawings were made at Ludwigshafen and the hairpin coils with return bends were made in Leuna.

Although the design was unusual, particularly with respect to its construction in the shop, it had to be made here. The difficulties of this problem could not be foreseen, because of its novelty. The first pieces made were for 325 atm. preheaters. These were made without complaints or call backs.

The situation changed entirely when 700 atm. return bends were demanded, which were subject to much greater maximum stresses, in spite of greater wall thickness, and which were much more difficult to make in the shop, due to the increased wall thickness, because of increased heat strains in welding.

At first, the return bends had a plug on only one side. This plug should be easy to remove in case it became necessary to replace the Granitherm inserts. An N5-ring was therefore inserted to facilitate the cutting out and rewelding of the screwed-in plug. The other end of the return bend was not bored out.

Now, a break occurred at this point in a 700 atm. return bend on applying test pressure in the shop. This break could be traced to a crack, most probably caused by heat stressed in welding on the adjacent tube nipples. (Nipples too short).

It became necessary to bore thru the bottom and to provide a plug on the other end also, and to lengthen the welded-on tube nipples. This construction was applied to all return bends already made.

The seal welding of the plugs proved particularly difficult. The plug was originally screwed in to a considerable length and then seal-welded in a V-groove. Because the danger of cracks in the V-groove, due to the high pressure, was anticipated, it was decided to use the U-groove. At the same time, experiments to determine the stresses were made on models in the materials testing laboratory. These indicated very high maximum stresses in the weld, which we tried to reduce by an elastic shaped edge of the plug. Return bends with that type of plug were used in construction. The return bends were preheated for welding to prevent hardness cracks. Special furnaces were developed for this.

Return bends of this type produced unexpected failures thru leaks in the plugs. In one case the plug was blown out, causing a big fire in the stall and severe operating disturbances at Gelsenberg.

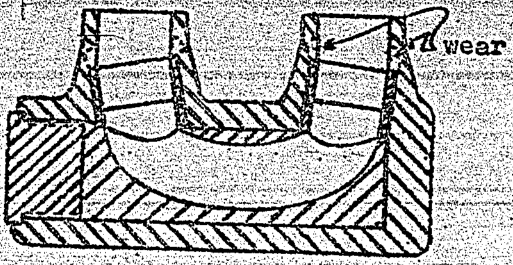
Extensive tests indicated that the creep strength in the vicinity of the plug was too low. This was not caused by the original annealing or the method of operating. Rather, it was determined that the return bends were heated above the intended preheat temperatures for welding in the heating furnace built for this purpose. An improved annealing furnace was, therefore, developed and built.

However, many plugs leaked, subsequently, even on such hairpin coils located at cooler points, and the weld at the plug was redesigned. The length of thread was reduced and the length of weld increased correspondingly. Later, the threads were eliminated entirely and the plug was welded to the return bend for its entire length. No damages have been observed since the plugs have been carefully welded in this fashion.

The costs to Leuna chargeable against this work amounts to about RM 500000.

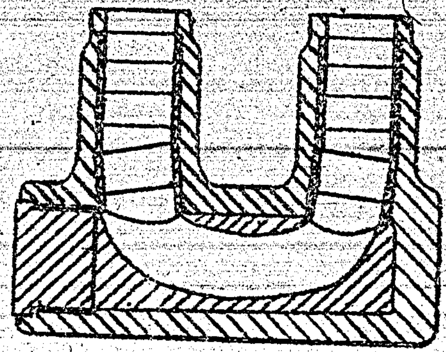
Development of wear resistant return bends

Fig. 1



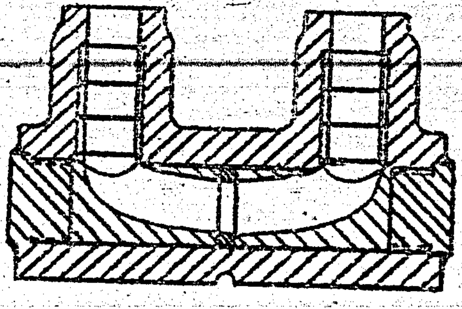
Scholven 1937

Fig. 2



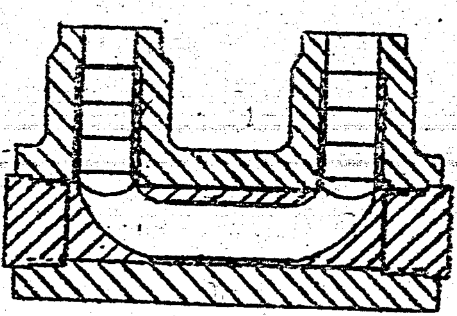
Scholven 1938

Fig. 3



Nordstern 1938
Politz 1939

Fig. 4



Scholven
Nordstern 1940
Politz
Upper Silesia

From M. Pier's private files. T-234

U. S. Bureau of Mines
Hydro. Demon. Plant Div.

DIAGRAM OF THE TEMPERATURE AND TIME OF CONTACT RELATIONSHIP IN THE LIQUID PHASE STALLS OF NORDSTERN AND PÖLITZ.

May 5, 1942

At Dr. v. Hartmann's suggestion, the time of contact in the liquid phase stalls at Nordstern and Pölitz were studied in relation to the temperatures of the coal passing through the stall.

All the data required for the computation are summarized in app. 1 and 2. The curve in app. 3 (*) was drawn from them, and the relationship between the volume of the apparatus and the feed temperatures is indicated there. A comparison of the principal parts of the two installations, i.e., the converters, was made possible by using the start of converter I as the origin. For comparisons of any kind, the volumes of the preheaters and the heat exchangers must be added.

An important assumption on the degree of filling must be made to permit one to show the temperature curve in relation to the time of contact: measurements of pressure differences in Nordstern as well as in Pölitz indicated a 50% filling. The degree of filling is however affected by the amount of gas, and a corresponding relation must be assumed to permit one to compare the gas distribution in the two stalls. The relation between the degree of filling and the gas load is shown in app. 4 (*) (fig. on the right). This curve permits the construction of the app. 4, and again, as in fig. in app. 3, the start in the converter was selected as the zero point of the time of contact.

We observe the following two characteristic differences in the time of contact of the two installations at Nordstern and Pölitz:

1. The converter temperatures in Stettin are 4° higher than in Nordstern.

2. The time of contact in the Nordstern converters is found to be 40 sec., or 2.9% greater than in Stettin.

3. Because of the paste heat exchange practised in Stettin, there is a division of paste into two streams; in Stettin as well as in Nordstern part of the paste requires around 150 sec. to be heated to 275° (in Nordstern this represents all of the paste), this time of contact is definite, because the paste and the gas travel without relative

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(*) App. 3 and 4 not available (transl.).

velocity (with respect to each other), and one may safely assume that in the forward pass through the heat exchanger the gas does possess some relative velocity with respect to the paste. This lengthens the time of contact in the heat exchanger, and the heat exchanger, like the converter, possesses a "degree of filling". Efforts will be made to determine this degree of filling. As long as it is not as yet known, the minimum time was introduced into the curve obtained for the case when gas and paste flow together.

Another important difference in the process with paste heat exchange consists in the mixing of the thick paste at 275° C with the paste heated to 430 C, which results in a cooling to about 360° C. This cooling may not be helpful for the subsequent course of the reaction. Should this be found to be true, two ways are possible to cool less the heat exchange components (to only about 385° C):

(a) By increasing the proportion of the paste fraction at the cost of the mixture, i.e., by heating the thick paste to 310° instead of the present 275° C;

(b) By increasing the thin paste component by increasing its solids concentration which will become possible when the already suggested changes in the coal grinding installation are carried out.

The products leaving the converter at the two plants have about the same time of contact in the heat exchangers and coolers. The cooling in Nordstern is done in a gas heat exchanger, air cooler, and water cooler, while in Stettin in three heat exchangers and water cooler.

<u>Summary</u>:

An expression is given for the time of contact in the installations at Nordstern and Pölitz in relation to the temperature in the liquid phase, with a special consideration of the gas load. The difference found for the two installations should not be large.

/s/ Wilde, Schappert

1) Gas volume 2.2 m³
2) 1/3 full
3) Cold gas.

App. I

Coal Stall at Nordstern

	Thruput		Conditions at Inlet			Conditions at at Outlet			Size of Equipment			Degree of filling of converters.	Velocity	Time of Contact
	t/h	m³/h	°C	Vol. m³/h		°C	Vol. m³/h		Cross Section	Length	Volume	%	m/sec.	sec.
Preheater, paste part	36	3 000	120	32 8	40	270	36 10	46	0.0064	246	1.55		1.9	130
Preheater, mixture part.	36	36 000	330	37 131	168	425	40 152	192	0.0064	422	2.7		7.8	55
Converter I	36	36 000 +8 000 3)	450	42 187	229	474	42 195	237		18	9.5	50		407
Converter II	36	44 000 +10000 3)	474	42 157	237	444	42 240	282		18	9.5	47		383
Converter III	36	54 000 +6 000 3)	474	42 239	281	474	42 265	307		18	9.5	43.5		355
Converter IV	36	60 000 +6 000 3)	466	42 272	314	466	42 289	331		15	7.75	40		266
Hot Catchpot	36	66 000 +5 000 3)	455	42 286	328	430	42 298	340			3.26 1)			32
Heat exchanger, return pass	18	62 000	430	25 258	283	320	22 224	246	0.03		0.51			7
Air Cooler	18	62 000	320	22 224	246	260	21 205	226	0.0077		0.65			10
Water Cooler	18	62 000	260	21 205	226	60	18 144	162	0.0159		3.05			59
Cold Catchpot	18	62 000	55	18 144	162	55	18 144	162	—		4.75 2)			31

1) Gas volume 2.2 m^3
2) 1/3 full
3) Cold gas

220

Appendix 2

Coal Stall, Stettin

	Thruput		Conditions at Inlet			Conditions at Outlet		Size of Equipment			Degree of filling of converters.	Velocity	Time of Contact
	t/h	m³/h	°C	Vol.	m³/h	°C	Vol. m³/h	Cross Section m²	Length m	Volume m³	%	m/sec	sec
H. E. (charging pass)	21	26 000	120	19} 79}	89	430	24} 108} 132	0.0068	3 x 17	3.47		0.36	120
Preheater, mixture part	21	4 000	110	18.5} 10.5}	29	260	21} 13} 34	0.0064	215	1.38		1.3	170
Preheater, mixture part	42	30 000	360	45} 115}	160	425	47} 125} 172		390	2.5		7.3	54
Converter I	42	30 000 +5 500$^{3)}$	445	48} 129}	177	480	49} 158} 207		18	9.5	59		412
Converter II	42	35 500 -8 500$^{3)}$	476	49} 155}	204	480	49} 195} 244		18	9.5	53.5		374
Converter III	42	44 000 +7 000$^{3)}$	476	49} 196}	245	480	49} 228} 277		18	8.5	48.0		335
Converter IV	42	51 000 +7 000$^{3)}$	476	49} 226}	275	473	49} 256} 305		15	7.75	44		250
Hot Catchpot	42	58 000	458	49} 250}	299	440	49} 245} 294		—	3.26$^{1)}$			32
Heat Exchanger, return pass	21	50 000	440	29} 211}	240	250	25} 162} 187		3 x 17	1.53		2.0	26
Water Cooler	21	50 000	250	25} 162}	187	58	21} 115} 136		144	3.05		2.1	70
Cold Catchpot	21	50 000	55	21} 114}	135	55	21} 114} 135		6	4.75$^{2)}$			27

Works		Press. Atm.	No. of Convert.	Capacity of Conv. m^3	Load		Temperature of Outlet from Preheater			Max. Temper. in Converters °C	Temp. in last converter, or hot catchpot °C
					t/h	m^3/h	Measured	Correction	Corrected Temperature.		
Scholven	Coal Stall	300	4	52	24	28 000	394	+2	396	472	435
			3	39	24	28 000	402	+0	402	472	435
			3	39	24	28 000	399	+3	402	472	437
Welheim	Tar Stall	550	4	37.8	23	17 000	461	-3	458	483 (Con. I)	456
			4	16.2	13	14 000	471	±0	471	483 (Con. I)	459
Gelsenberg	Coal Stall	700	3	26.75	21	23 000	432	±0	432	476 (Con. I & II)	465
			3	25.75	25.5	23 300	416	+0	416	476 (Con. I)	466
			4	36.25	28	27 200	423	±0	423	478 (Con. II)	464
			4	36.25	32	30 500	406	+3	409	476 (Con. II)	465
Stettin	Coal Stall	700	4	39	40+9.6	30 000	413	+7	420	476 (Con. III)	430
	Tar Stall		4	39	34.2	22 000	430	+18	448	484 (Con. III)	435
			4	39	36.4	22 000	448	+3	451	484 (Con. III)	435
Böhlen	Tar Stall	300	3	27.3	27.5	15 000	432	+16	448	488	442
"	"	300	3	26.3	37.6	18 000	442	+16	458	488	452
Magdeburg	"	286	3	30	38.0	18 000	446	+11	457	482	440
Welheim	Vapor phase	550	4	16.9	11.5	30 000	451	+4	455	500 (Con. II-IV)	500
Scholven	6434	300	2+1		18+4	30 000	399	+2	401	419 (Con. II)	392
	6434		2		12	35 000	368	+0	368	416	409
	7019		2+1		16	30 000	481	+13	494	506 (Con. II)	502
Stettin	6434	300	4		20.5	35 000	380	+14	394	398 (Con. III)	392
	7019		3		25	37 000	482	+7	489	513 (Con II)	501
Leuna	Coal Stall	200	4	27.4	26.0	28 000	403	-5	398	468 (Con. II)	447
		200	4	27.4	26.0	28 000	401	-9	392	462 (Con. II&III)	440
		200	4	27.4	26.0	28 000	384	-15	369	472 (Con. II)	449

U.S. BUREAU OF MINES 222 T-235
HYDRO. DEMON. PLANT DIV.

KCBraun
2/7/47

SAFETY MEASURES IN THE CONSTRUCTION OF HYDROGENATION STALLS, BASED ON EXPLOSIONS AT PÖLITZ, 1941

(See also T - 78)

From: - Dr. H. Sauer, Director of the Leuna Werke, I.G. Farbenindustrie, A.G.

To: - Dr. Freytag, Technical Consultant to the Association of Chemical Manufacturers, Berlin - Wilmersdorf.

Leuna Werke, 21 Jan. 1942

To your inquiry directed to Dir. Jähne of Höchst of the 17th ultimo regarding the measures proposed on the above subject matter, we take the following position:

I should like to point out at the start, the special nature of the process used at Pölits and other hydrogenation works, in order to prevent the formation of unjustifiable opinions and regulations inapplicable to other high pressure processes, which appear similar to coal hydrogenation, but whose operating conditions are considerably different, such as the ammonia and methanol syntheses, as well as other high pressure processes often called hydrogenation because of the absorption of hydrogen. My remarks herein are, therefore, solely intended for hydrogenation stalls for coal, tar and similar materials, simply called hydrogenation stalls in the following.

There is no particular danger that entire apparatus, such as converter jackets, might burst due to internal pressure, because the pressures and temperatures of these internally insulated jackets can technically be fully controlled and large scale failure of construction materials can be discounted, because of very careful inspection and acceptance tests. If we tried to provide material protection against all possibilities, the dimensions of the stall walls would become unwieldy. Rather, we must figure on the escape of gases leading to explosions caused by the following:

1.) Leaky cover closures due to imperfect sealing surfaces (the closing power of the cover bolts can always be controlled).

2.) Leaky flange connections on pipe lines due to the same cause as in 1.) or to severe fluctuations in temperature.

3.) Breaks in pipes due to

   a) failure of the material, or

b) Excessive temperatures due to runaway of the reaction (these are absorbed in the converters by the inner insulation).

The leaks caused by breaks in pipes cause the greatest damage, because the entire cross-section is released, while in 1.) and 2.) only comparatively restricted leaks occur.

Accordingly, "explosions in a hydrogenation stall", mainly due to breaks in high pressure connecting lines, are primarily concerned with the violent escape of combustible, gaseous or liquid contents of the converter. These escaping hot gases or liquids ignite either immediately or they form mixtures with the air, which then explode more or less violently, depending upon the formation of the mixture and the incontrollable period of ignition. Such pipe breaks in the stalls may, therefore, have the effect of a simple leak, fire, or violent room explosion of a cloud of mixture extending above and beyond the stall. The large number of people hurt in the explosion at Polits is due to the splinter effect of the panes of glass broken by the pressure of the explosion in the surrounding plant structures.

In the first hydrogenation works at Leuna in 1926 we still had to figure on all three of the causes mentioned above, because the development of H_2-resistant steels for the hot lines was still in its infancy. The converters were, therefore, located in a stall completely surrounded by 30 cm thick reinforced concrete walls and open at the top.

The following reasons led, in the course of development, to an almost completely open and also lighter construction of the hydrogenation stalls, which were reinforced only against leaks in cover and flange connections, see 1.) and 2.) above.

First, the quality of the steels in H_2-resistance and strength was so perfected that there was no longer any danger of the bursting of entire pipes, originally feared and actually experienced during the first years of hydrogenation.

Second, the process was developed in the meantime to such an extent that dangerous, excessive temperatures need rarely be expected any more.

Third, an explosion in the completely enclosed stalls at Leuna showed that the formation of dangerous mixtures is promoted by the completely enclosing reinforced concrete walls, and that they even had an accumulating effect, which in one case actually caused the stall walls to break.

Fourth, experience had shown us that breaks in tubes on the unprotected gas preheaters, tubes of which are severely strained due to external heating, never had any serious effect at a distance.

The two successive stall fires at Pölitz were due to special conditions in the gas circulating system in 700 atm. plants and to special circumstances in the starting up of the stalls at Pölitz. In the meantime, the conditions previously not fully known, particularly the formation of hydrates of hydrocarbons, have been clarified and proper measures taken to prevent such an accident from the same cause, where humanly possible. In spite of this, such stall fires may not be considered impossible in the future and I, too, share your opinion that certain rules ought to be formulated for new installations.

First, we should fundamentally stick to the idea to keep the stalls as open as possible in order to prevent the formation of dangerous mixtures and the effect of accumulations in explosions. However, the sides towards the operators and essential apparatus should be protected by reinforced concrete walls strong enough and high enough to prevent the spread of possible fires to work places in adjacent stalls. Reinforced concrete walls about 30 cm thick with about 150 kg/m^3 reinforcing should be ample. These dimensions have proved satisfactory at Leuna. In a skeleton structure a thickness of 15 cm with corresponding reinforcement should suffice. The reinforcement should be such, and should be tied into the adjacent walls or the framework, that extraordinary mixture explosions will cause only a bulging of the walls, but no breaks, permitting resumption of operations.

However, we should not think of a "blowout system", in order to avoid confusion with terms used in the explosives industry. The stall walls, fully closed to the top, are not so much intended to protect neighboring buildings or work places in their upper portion, but are primarily intended to prevent the spread of possible large fires to the work places of adjacent stalls. The supply track with the crane runway must be located on the open side of the stalls, from which the stall equipment is mounted and serviced. These work places are protected by a protecting wall about 4 m high. The stalls are generally located along a plant road and, at times, it cannot be avoided that railroad tracks run along them. It must also be possible to erect auxiliary manufacturing buildings at a distance of about 30 m of the open wall. However, care must be taken in planning that the traffic on these plant roads be kept to a minimum, that no trains stop there during operations, and that not many people are employed in the opposite buildings, such as in work shops.

I suggest that your proposed five clauses embody the principles discussed herein and read as follows:

1. In the pressure hydrogenation of coal, tar and similar raw materials, exothermal reactions take place in the converters. In the course of the development of the operations and processes the safety has been so perfected that a sudden rise in the converter temperature has become a rarity.

2. In spite of this, special attention must be paid to effective and rapid counter measures to an undesirable rise in temperature in the converters.

3. In the present state of development of high pressure hydrogenation of coal, tar and similar raw materials, fires and explosions in the stalls will probably continue to occur in the future.

While, fundamentally, the stalls shall be built with closed walls, they shall be open on at least one side. Within a distance of 30 m of the open side, no work places, much used roads, apparatus with inflammable contents, etc, not directly connected with the operation of the stall, shall be located. The stall walls shall be developed as resistance walls. They shall be of reinforced concrete and so well reinforced and anchored that they will stand up under practically any gas explosion in the stall. The walls shall be so wide and high that they will protect any work places in the vicinity from jet flames. In particular the protection of work places and service walks along the stall track requires a sufficiently high protective wall on the open side of the stall.

4. Work places in the vicinity of the stall, including platforms, for example, shall have ample escape possibilities.

5. Consideration shall be given a suitable alarm device, which will warn those working near the stalls in case of threatening danger.

These rules, based on the present state of development, include all possible measures for the protection of personnel.

They allow the plants sufficient freedom of action to meet their special conditions on their own responsibility. These apply primarily to new plants to be erected, and in my opinion, a fundamental change of all existing stalls would be impossible under present conditions and could neither be expected, based on what has happened to date.

U.S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-236

KCBraun
2/25/47

THE PREPARATION OF TUNGSTEN SULFIDE CATALYST

By V. Füner, Ludwigshafen, 22. May, 1943

Gmelin's handbook shows 10 methods for the preparation of WS_2.

1.) W + S + heat = WS_2.

2.) WO_3 or W + S or H_2S or CS_2 + heat (glowing) = WS_2.

3.) 1 part WO_3 + 6 parts HgS, cover with coal and raise to white heat for 1/2 hr. = WS_2.

4.) $(NH_4)_2WS_4$ raised to glowing heat in stream of CO = WS_2 pseudomorphous.

5.) S + K_2WO_4, melt 1/2 hr. in clay crucible = WS_2.

6.) WO_3 + K_2CO_3 + S, melt 1/2 hr. in clay crucible = WS_2.

7.) $Na_2S_2O_3$ + WO_3, crushed, well mixed and heated in crucible = WS_2.

8.) H_2S is passed over WCl_6 in enclosed vessel at 375°C.

9.) WS_3 calcined = WS_2.

~~10.) WS_3 calcined with KCN and the melt leached with water.~~

If these methods are checked for their suitability for the preparation of active WS_2 with a large surface, only a few processes remain for serious consideration. Almost all of these processes are based on the preparation of WS_2 at high temperature, because the oxide or metal will not convert to WS_2 at low temperature. In addition, alkali is present in 5.), 6.), 7.) and 10.).

But we already know from our experiments on ammonia catalysts that alkali, even in very small quantities, is catalytically harmful to Mo and W, which has also been confirmed with WS_6. After the sulfuring of WO_3 with H_2S at 600°C, as well as the preparation of WS_2 from WCl_2 + H_2S, produced no active catalyst, we tried the preparation from the sulfosalt by process 4.), as well as by precipitation of WS_2 and other pressureless reduction to WS_2. The results of these experiments were also negative, the latter probably because WS_3 is extraordinarily oxidizable and, therefore, not present in the pure state, the former because of too high a temperature. We, therefore, tried other ways to produce WS_2 at low temperature. In the preparation of catalysts emphasis is placed on a well developed surface.

This objective is usually gained in the preparation of metallic catalysts by the reduction of the oxides,provided the reduction temperature is not too high for the oxide in question and the loose metal produced in consequence does not start to sinter immediately, as e.g. Fe from Fe_3O_4, with NH_3 catalyst, and Co from $Co(OH)_2$ with the Fischer catalyst. If this fine distribution is not enough for the desired catalytic reaction, still more complicated compounds, such as nickel-formate, -acetate or -oxalate, which may possibly be distributed on carriers, are subjected to reduction at especially low temperature, below 250°C. If we want to prepare an active oxide or other metallic compound, such as a sulfide, we usually take the intermediate step of preparing a hydroxide or a precipitated sulfide, which is converted to the corresponding oxide by careful drying and careful further extraction of the water constituent. It is important for the catalytic effectiveness of an oxide or oxide-hydroxide catalyst, that the hydroxide or oxide-hydrate be present in a certain distinct form or that it be converted into this form under certain conditions, such as active alumina or active silicagel. But for some catalytic reactions it is sufficient, if an oxide of a higher stage is easily converted into a lower stage (Mn, Cr-catalyst). The preparation of active carbon is also based on a very complicated high molecular carbon compound, producing an exceptionally loose carbon structure by careful decomposition.

For the preparation of WS_2, Dr. Jacob, in a departure from process 4.), decomposed ammonium-sulfowolframate with H_2 at temperatures up to 400°C, and thus produced the exceptionally active 5058 catalyst.

An equally active WS_2 could later be obtained by sulfuring of WO_3 or ammonium-wolframate with H_2S under pressures of 5 to 10 atm at rising temperatures up to 410-430°C, for periods of 36-10 hours. By applying H_2S under pressure it was possible to conduct the sulfuring at low temperature in a relatively short time, which, at normal H_2S pressure, is possible only at high temperatures and even then with great difficulty. H_2S pressure has not been applied industrially in the preparation of WS_2, but rather the sulfosalt process has been further developed.

Gelsenberg - Benzin A.G.

To I.G.Farbenindustrie A.G.
Section High Pressure Experiments
Ludwigshafen a.Rh.
Hydr. Dr. Kl. 8263 2/22/43

Re: Discussion of leaky valves on paste pumps
(6 papers from Dr. M. Pier's private files)

Enclosed is a report on trouble in the coal stall caused by leaky valves on a pump.

In the process described there is an extraordinarily great danger for the installation because the entire stall is protected by one single check valve, and would become discharged through the pump should this check valve fail, which is known to happen frequently. We must be grateful for the functioning of this valve when a catastrophe is averted.

We therefore inform you of this case with a request to ascertain by means of a questionnaire to the other hydrogenation works what insurance is provided against such incidents.

No such case has yet occurred in Scholven, in spite of the long operation period, and works operating at 700 atm. should be primarily concerned in it.

We request you to emphasize the question on the nature of protective measures to prevent a simultaneous leaking of the pressure and suction valves and for the prevention of a coal stall having its pressure discharged in the reverse direction.

Gelsenberg-Benzin A.G.

2 Signatures

Gelsenberg-Benzin A.G.

Sect. Hydrogenation/Dr.Kl. Gelsenkirchen-Horst, 2/22/43

Report on coal stall troubles resulting from leaky paste pump valves.

The connections of the paste pumps in the Gelsenberg coal phase is as follows:

Every nine adjoining pumps operate for two coal stalls with a common collector main line. The collector line is divided into two halves by means of two valves each between pumps 4 and 5, and between 5 and 6. The outlet from each half is to one of the two stalls. Three pumps operate therefore per stall, with 10 te.paste, and one pump with at least 2 te.pasting oil.

Normally, pump 5 is kept in reserve, and in case of repairs on any of the pumps is connected as required to either of the two stalls. Each pump has two shut-off valves on the pressure side, operated by hand. There are check valves only in the collector line immediately before the stall inlet, but none after the individual pumps.

The following incident occurred in the early morning at 6:20 on 2/22/43. A pressure valve sprung a leak on one of the pumps in operation. The pump was therefore first connected for flushing with pasting oil prior to turning it over to repairs. The temperature in the preheater stall suddenly rose and the preheater was extinguished and the temperature quickly lowered with cold gas. The reason for this was found not to be a leak in the collector main, as at first suspected, but in that the suction valve as well on the defective pump was cut through so that the remaining three pumps forced the coal paste through that one pump back into the pasting oil tank. The leak was so strong that the pressure on the converter dropped and no paste therefore was reaching the converter. The check valve at the converter inlet remained however sufficiently tight so that the converter contents were not discharged backwards before the shut-off valves of the leaky pump were turned off. The converter could then again be started with paste and had apparently suffered no damage. A similar case, when a suction and a pressure valve on one pump began to leak at the same time, had already occurred once before but not as strongly as that, so that the difficulty was not great.

According to information from Scholven, such cases have not occurred there. There are no check valves in the pressure lines of the individual pumps, but only in the collector main to the stall.

Questionnaire To: Leuna, Scholven, Gelsenberg, Pölitz,
Welheim, Brabag, Rheinbraun, Lützkendorf,
Oberschlesien, Brüx.

High Pressure Experiments, Leuna

March 8, 1943

You will find enclosed a letter and a report of the Gelsenberg-Benzin A.G. about troubles with a paste pump.

Please inform us whether you have had any similar experience, and in particular state what measures have been taken when the paste pump was shut down because of a defective pump valve discovered on the pump, and how this pump was flushed.

We wish that you would tell us in addition whether your check valves are regularly inspected, and how this is done.

I.G.Farbenindustrie A.G.

/s/

(Not readable) Schönjahu

Gelsenberg Benzin A.G.

To: I.G.Farbenindustrie A.G.

High Pressure Experiments

Ludwigshafen a.Rh.

Gelsenkirchen, 4/27/43

We acknowledge the receipt of the information of measures used in Leuna, Lutzkendorf and Wesseling transmitted by your Mr. Schappert at the occasion of his last visit. Please notify if further information is required.

We are transmitting to you in the appendix a sketch from 4/9/43 with a suggestion to change the valve group of a paste pump by the installation of drop shaped check valve. The present vertical hand operated valve, i.d. 58 mm, will have to be installed horizontally. However, the horizontal arrangement does not interfere, and tends rather to facilitate operations and permits using the theoretically correct principle of the correct location of the valve underneath the ball, against the outside pressure, a principle which has been formerly disregarded in the vertical arrangement. The new arrangement fits readily into the present arrangement of the group of valves.

Signatures illegible.

Hydrogenation Works Pölitz A.G.

531.534 Dr.Kiehn 552.553.554.578 (2)

To: I.G.Farbenindustrie A.G.

Ludwigshafen a/Rh

High Pressure Experiments 3/8/43 TA/Ho-Mo/373 Stettin-
P/Leuna 558 Scha/Eb Pölitz
4/27/43 Eb/H

Re: Leaky paste pump valves.

We have learned from a report of the Gelsenberg-Benzin A.G. of 2/22/43 of difficulties caused by leaky paste pump valves.

Suction and pressure valves have once in our experience started leaking together. The trouble has however been discovered so early that a disturbance in the stall operations could be avoided.

One must generally count, and in particular today, on shortcomings of the service personnel and on troubles resulting from leaky valves, and we have decided to install check valves in the pressure side of each paste pump, and in addition a testing valve as shown in the accompanying sketch.

To permit occasional testing of the check valve for tightness without dismantling, the test valve mounted in a special vertical pipe was provided and during the operations of the press it is disconnected by a slide valve.

When ready to do the testing, after the pumps are shut down, pressure is applied to the oil-filled pipeline used to supply flushing oil to the manometers between the check valve II and the closed shut-off valves. The previously opened test valve permits observing any leaks in the check valve II.

In accordance with your request, we will list the steps we take when shutting down a pump:

1. Setting the pressure line to the second shut-off valve under pasting oil, and then under flushing oil.

2. Shutting off the pump.

3. Closing the shut-off valve on the side of the stall, and testing its tightness by opening the two pressure release check valves.

(Continued)

4. Closing the shut-off valve on the side of the machine, and testing it for tighness by opening and then closing the shut-off valve on the side of the stall.

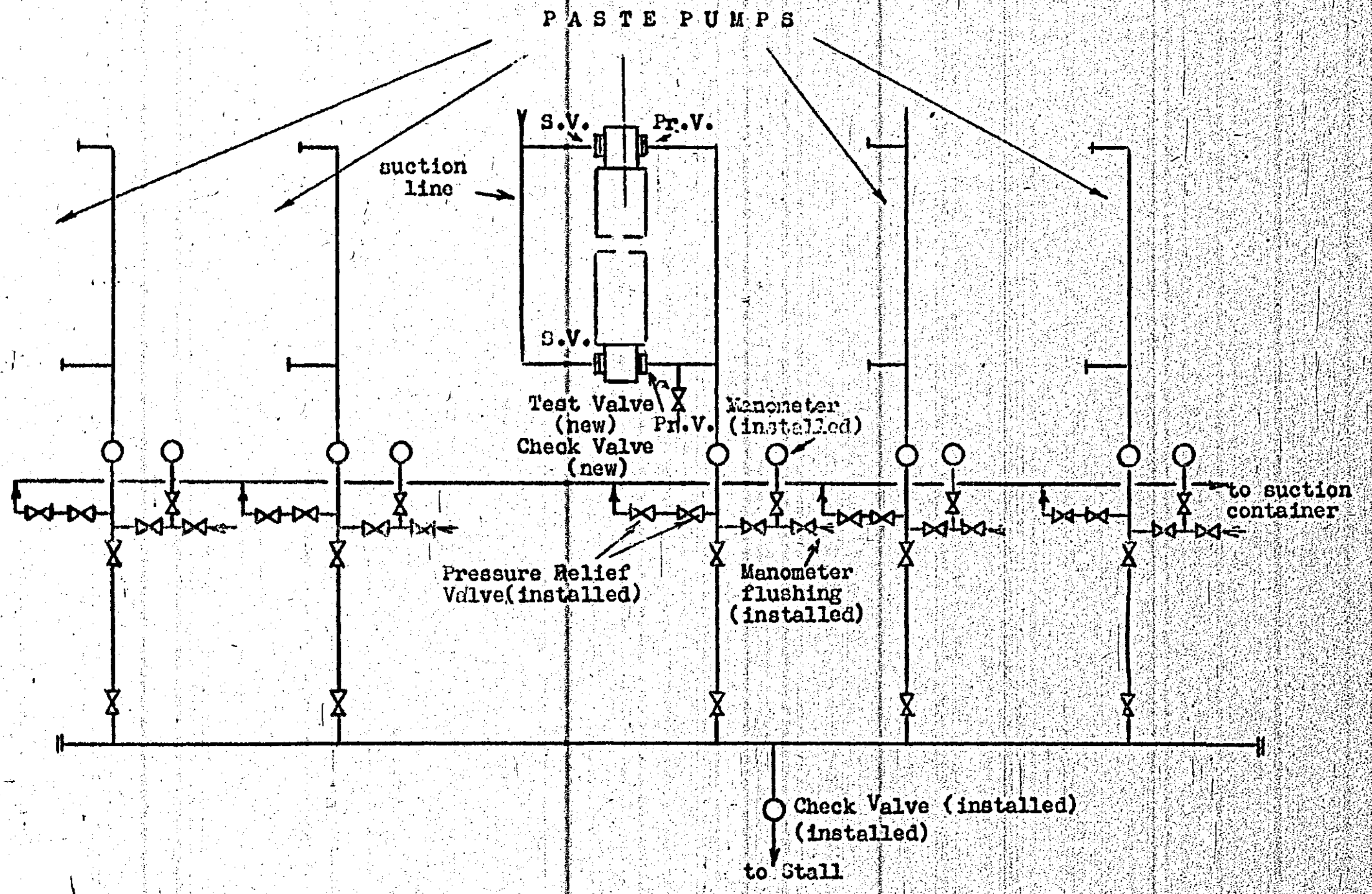
5. Short operation of the pump with flushing oil for flushing the pressure release line of the check valve.

6. Closing the two pressure release valves.

No regular testing of the check valve on the stall side can be made because it is only accessible when the stall is shut down. It is however dismantled at each shutting down of the stall and subjected to exhaustive tests.

Hydrierwerke Pölitz A.G.

/s/ Rumpf Zimmermann


P A S T E P U M P S
suction line
S.V.
Pr.V.
S.V.
Test Valve (new)
Pr.V.
Check Valve (new)
Manometer (installed)
to suction container
Pressure Relief Valve(installed)
Manometer flushing (installed)
Check Valve (installed) (installed)
to Stall

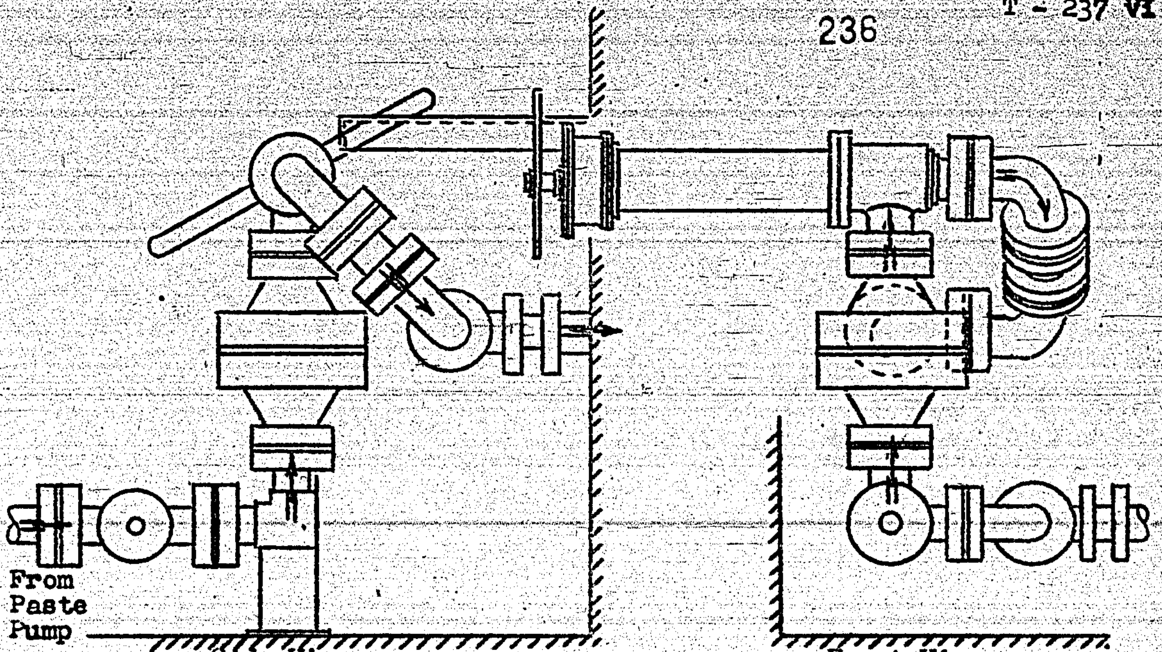
Ludwigshafen May 29, 1943

Discussions by Leuna, Scholven, Gelsenberg, Pölitz, Welheim, Brabag, Rheinbenzin, Lützkendorf, Oberschlesien, Brüx representatives.

The paper is a summary of replies obtained to a questionnaire sent to the different hydrogenation works. The safety measures adapted by the different works are too different at the different works, depending on local conditions, to permit giving any general directions. However, one observation appears universally in all the replies, that in the many years of operation of paste pumps, not a single serious damage to stall operations has been the result of leaky valves. Leaky valves were invariably discovered in time either in the operation of the pumps or of the converters. It must be considered generally true, that when any irregularities are discovered in the operation of the valves the paste pumps must first be shut down on the pressure side, and only then flush. A regular inspection of the check valves of the converters can be recommended. When considering locating check valves immediately behind every pump, one must bear in mind that the pressure valves of the pumps operate more accurately if the full pressure of the stall acts directly upon the valves when reversing the pumps. Moreover, trouble with the pump valves would be more readily recognized in the absence of check valves behind the presses. We reserve, however, the right fordeach hydrogenation works to make use of such recommendations as are most suited to their operations.

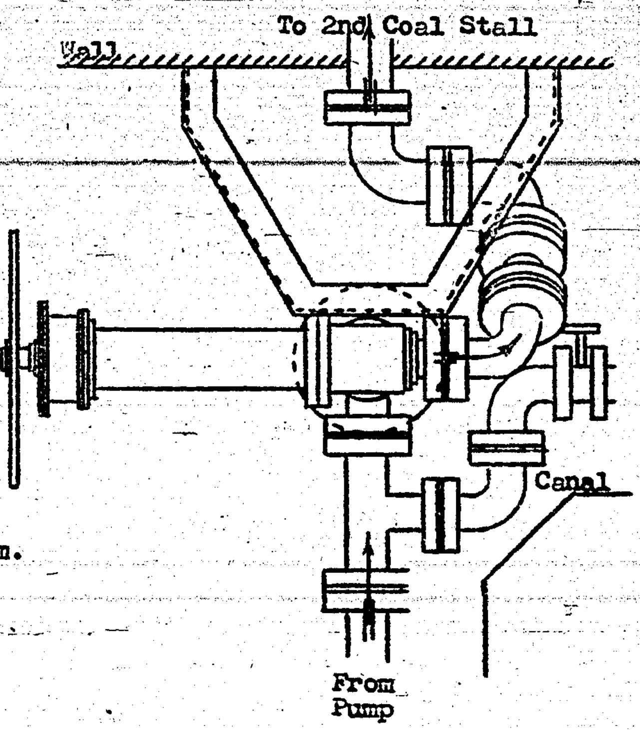
/s/ Pier Schönjahn

Various appendices (not available, Transl.)



Side View

Front View



Sketch for the installation of the check valve 58mm.D.,700 atm. pressure, in the Coal Paste Pipe Line 58 mm. i.d. from the Paste Pumps to the Collector Pipe Line.

TEMPERATURE DIAGRAM OF YOUR COAL STALLS

From Dr. M. Pier's private paper.

Hydrogenation Works Scholven, A.G.
Gelsenkirchen - Buer

High Pressure Experiments
P/Leuna, May 26, 1941

We are transmitting to you in our appendix the pyrometric evaluation of your coal stalls 1,2,3 and 5.

App. I is a summary of the K values of the individual passes, subdivided for the two methods into
a) without gas supply to the paste,
b) with gas supply to the paste.

We confirm the observation already made by you:

1) The K value of the paste part is about 1/3 lower when operating with no paste gas.

2) While the amount of gases in the mixed part (intermediate + hot path) is always the same, there is never-the-less a lowering of the K values of about 10% for both paths.

App. Ia shows an example of a computation: The measured feed temperatures, the inlet and outlet temperatures of the circulating gases are here retained; the circulating gas intermediate temperatures were computed from the heat balance. The other method, to keep all the measured circulating gas temperature values, gives the same qualitative results.

Results on the efficacy of the deflectors, installed in the hairpins to increase the velocity of the circulating gas have been calculated in App. II from the above results; a sketch of such deflectors is shown in App. III.

This installation permits raising the K value by 15% on the average. Deflectors can, however, not be built in the hot pass because of the tube wall temperature.

The improvement in the K value is of course a result of increasing the resistance to the circulation gase and the resistance of a hairpin is raised by the deflectors from 3 to about 8.5 mm water column, i.e. the installation of deflectors in a pass increases the resistance of this pass, and therefore of the whole preheater by about 30 mm water column. The circulation gas blowers are laid out for 300 mm water column, and 22 hairpins of one preheater offer at most 70 mm of that resistance, and economic considerations permitted an increase in the useful resistance.

We suggest therefore to equip at present only the cold pass of the preheater. These deflectors bring you the advantage of one additional hairpin, because they permit an increase of 1 te/hour thruput or a 7° lowering of the circulation gas temperature.

I.G. Farbenindustrie A.G.

by /s/ undecipherable

Appendix I.

Stall #	Date	Paste gas hairpin C m/sec	Paste gas hairpin Ka	Paste gas hairpin Ki	Paste part C m/sec	Paste part Ka	Paste part Ki	Mixed part Interm. pass C m/sec	Mixed part Interm. pass Ka	Mixed part Interm. pass Ki	Mixed part hot pass C m/sec	Mixed part hot pass Ka	Mixed part hot pass Ki	Hairpin C m/sec	Hairpin Ka	Hairpin Ki
a)	With no paste gas															
1	5/7/41				9.4	2.35	47	13.7	3.8	70	15.1	7.3	121	15.2	5.4	102
2	4/14/40				12.6	4.05	81	13.9	5.4	99	14.9	6.2	100			
2	3/15/40				11.8	4.8	96	13.3	5.7	105	14.0	4.3	70			
2	3/1/40				11.5	3.65	73	13.0	6.3	116	13.8	4.05	65			
2	4/1/40				12.4	4.6	92	13.7	5.4	99	14.7	6.2	100			
	Average values					3.9	78		5.3	98		5.6	91		5.4	102
b)	With paste gas															
3	9/17/40	9.5	7.7	154	10.3	7.0	140	11.1	4.4	80	12.1	9.3	141	12.2	9.1	139
3	3/1/40	11.3	8.4	168	12.0	5.7	114	4.2	6.2	112	14.3	8.9	134			
3	1/11/40	11.3	6.5	130	12.1	6.6	132	13.2	3.5	64	14.3	9.5	142	14.4	8.1	115
5	5/7/41	8.9	7.1	142	9.3	4.55	91	13.8	5.1	95	14.7	4.6	81	14.8	7.0	133
	Average values		7.4	148		6.0	120		4.8	88		8.1	124		8.1	129

Remarks: C — velocity of circulating gas in the passes
Ka — Heat transfer number, referred to the outer hairpin surface
Ki — Heat transfer number, referred to the inner hairpin surface

Temperature distribution of the Coal Stall 5, Scholven, 5/7/41

Thruput, paste + pasting oil	te/hr	23 - 1
Gas over heat exchanger	m^3/hr	28,000
Catchpot	te/hr	11.6
Outlet gas	m^3/hr	30,000
Paste gas	m^3/hr	2,000
Heat values: heat exch. forward	Kcal/° C	9,260
" return	" "	18,040
preheater, paste gas	" "	660
" " Section	" "	13,620
" mixt. Section	" "	22,880
Heat Exchange Temperature Distr.	°C	273 ← 437 54 → 382
Temper. difference	°C	111
Surface	m^2	228
K values	Kcal/°C, hr, m^2	119
Heat output,	10^3, kcal/hr	3,040

Preheater		Paste gas	Paste Sect.	Countercur. Mixt. sect.	Concurrent Mixt. sect.	Gas hairp
Temperature distribution	°C	420 ← 429° 54 → 360	429° ← 463° 154 → 210	563 ← 523° 292° → 352	523° ← 575° 402 ← 352	575° ← 586 410 ← 382
Temp. diff., circul. gas feed	°C	178	263	171	166	184
Number of hairpins		1	4	8	8	1
Outer surf. of hairpins	m^2	160	640	1570	1410	200
Ka value	kcal/°C, h, m^2	7.1	4.55	5.1	4.6	7.0
Ki value	"	142	91	95	81	133
Heat output, eff.	10^3 kcal/hr	202	764	1373	1144	259
Circulation gas stream	m^3/hr			73,000		
Free cross section	m^2	5.6	5.6	4.08	4.08	4.08
Circulation gas velocity	m/sec	8.9	9.3	13.8	14.7	14.8

Fuel gas	m^3/hr		1.290
Heat supplied	kcal/hr	1290 x 4700 x 0.92 =	5,580,000
Heat effectively transferred	" "		3,742,000
Efficiency of preheaters	%		67

°Calculated temperature

App. 2.

Improving the heat transfer number by the Installation of Deflectors (Coal Stall Scholven).

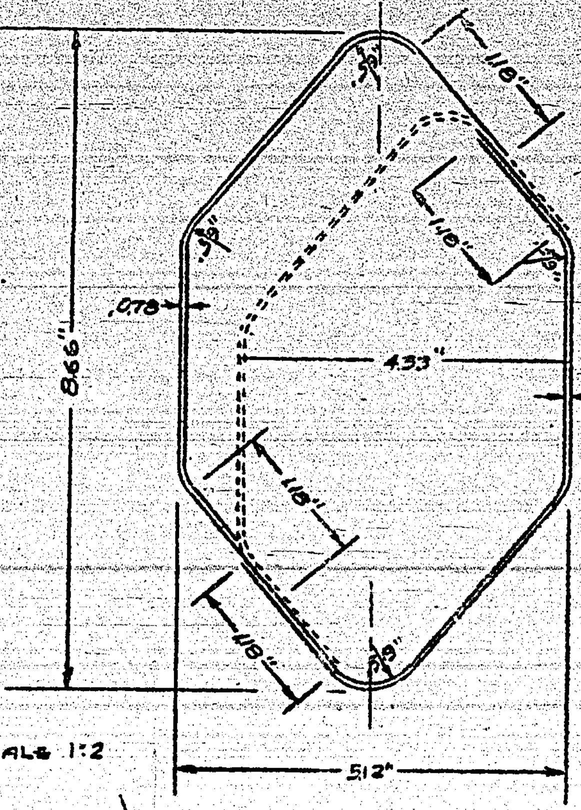
		Cold Pass	Inter. pass	Hot pass
Clear width of hairpins, mm		90/127	120/171	120/171
Ka values	kcal/°C, h, m²	6.0	4.8	8.1
Ki values	"	120	88	124
(?) with no deflectors	"		abt. 200	
$\frac{1}{Ki}$		0.00833	0.01112	0.00806
(?)		0.00052	0.00071	0.00071
$\frac{ri}{ra} \times \frac{1}{(?)}$		0.00354	0.00350	0.00350
(?)		0.00427	0.00691	0.00385
(?)		234	145	260
Percent increase of circulating gas velocity through installation of deflectors			about 65	
(?) with deflectors, kcal/°C, h, m²			(?) = 290	
$\frac{ri}{ra} \times \frac{1}{\alpha_a}$		0.00244	0.00241	0.00241
$\frac{1}{Ki}$		0.00723	0.01003	0.00697
Ki value, kcal/°C,h,m²		138	99	143
Ka value		6.9	5.4	9.2
Increase in K value through installation of deflectors, %		15	12.5	15.3

Velocity in the passes, with no deflect. abt. 14 m/sec
with deflectors " 23 "

Resistance of 1 hairpin, no deflect., mm water " 3
" " " " with " " " " 8.5

Translator's note. The ditto sheet of the original contains errors and poorly printed areas. Efforts to explain them have proven unsuccessful, and they have been indiated by (?).

W. M. Sternberg

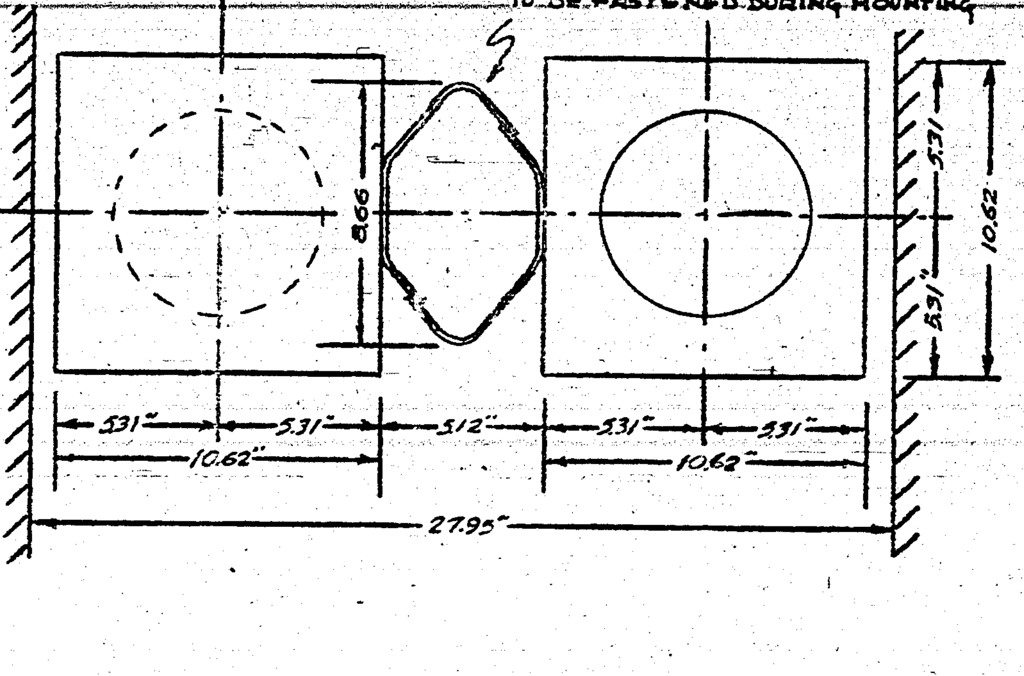


LENGTH OF DEFLECTOR
APPROX. 1000 mm.

SCALE 1:2

SCALE 1:5

TO BE FASTENED DURING MOUNTING



KCBraun
2/11/47

EXPERIENCES WITH RUNAWAY CONVERTERS, ETC.
(Discussion held at Politz)

Ludwigshafen, 17 Aug. 1942.

BEHAVIOR OF COAL STALLS AT EXCESSIVE TEMPERATURES.

MEASURES TAKEN IN LUDWIGSHAFEN: - 700 ATM.

Normal maximum temperature in converters = 25.2 mV (40°C).

A. Assumption: - Gas flow normal.

When the temperature rises excessively, cold gas is first injected to a max. temperature of 25.5 mV, at the same time cutting down the gas preheater. If the temperature rises above 25.5 mV, cold pasting oil is injected ahead of the converter in question and the presses are switched over to pasting oil at the same time. The cold oil is conveyed by a paste press, which conveys about 2 m^3 (volume of stall = 1.6 m^3). An estimated 100 liters of pasting oil is enough to save the converter. (Operating time of pump is from 3 to 5 minutes, according to the converter temperature). If the converter temperature reaches 26 or more, emergency let-down must be resorted to. This is done in the following way:

1.) The gas preheater burners are turned off, the circulating blower keeps on running.

2.) The stall is disconnected from the circulating system (electric valves, circulating system inlet and outlet).

3.) The converters are emptied in the order 1-2 (hand operated valves), irrespective of whether converters 1 or 2 ran away. The catch pot is emptied thru the nozzle in the normal manner until gas appears. The higher the converter temperature the quicker the converters are emptied.

4.) Pasting oil is continued to run thru the heat exchangers and the preheater to prevent coking up.

5.) In Ludwigshafen the letdown valves usually leak after an emergency expansion due to the runaway of a converter and the stall can not be restarted immediately. Therefore, after the converters are emptied, the stall is shut down in such a way that nitrogen is injected into the heat exchangers and the preheater after the pasting oil pumps have been shut down and the heat exchangers are desludged at the same time. As soon as the heat exchangers are empty the valves are again closed and the stall is further flushed with nitrogen, the flushing is controlled by gas analyses.

6.) Change converter letdown valve.

7.) Again flush with nitrogen, then fill with 100 atm hydrogen for starting up anew; let about 2000 m^3 gas flow thru.

8.) After checking the gas meters for correct functioning pasting oil injection is immediately started.

9.) When the catch pot level has been reached and the level gage functions properly the pressure is brought up to reaction pressure (hourly increase 50 atm) and the converter is again brought up to the injection temperature in the usual manner.

B.) Assumption: - No gas flow.

1.) For example, due to clogging of heat exchangers or preheater: Burner is turned off. Converter is cut off the circulating system and emergency letdown is made just as in A.) Using the cold oil pump, pasting oil is injected in the inlet between the preheater and Converter I and the converters are run cold to the catchpot at atmospheric pressure. According to where the preheat system is clogged, pasting oil is run thru the free portion of the passage.

2.) For example, due to clogging of air cooler, product cooler or heat exchanger return passage: Shut off burners, switch to pasting oil, cut converters off the circulating system and make an emergency letdown just as in A). The converters are run cold with pasting oil thru heat exchangers and preheater to the catch pot.

3.) For example, due to clogging of the gas inlet: Turn off burner and switch to pasting oil, put the converters off the circulating system and make emergency letdown as in A). When the inlet gas fails but the cold gas continues to function proceed likewise.

C.) Assumption: Break in Preheater.

Turn off burners, shut off gas circulating system. Disconnect the stall from the circulating system and try to empty the converters. Flush with nitrogen, as quickly as possible insert a blind disk between preheater and converters and run the converter system cold with the cold oil pump to the catch pot.

Measures taken in Leuna: 200 atm.

Normal maximum temperature of the converters is 25.9 mV (40°C), depending upon paste thruput.

A.) Assumption: Normal gas flow.

At 26.1 mV switch to pasting oil. From 1 to 3 m^3 pasting oil to 30 m^3 coal paste will bring down the temperature in all converters in 6 to 7 minutes (20% full (Füllungsgrad) at 15 mV). Letdown at high temperature cannot be done at Leuna because of the S2 material in the expansion line.

B.) Assumption: Break in Preheater.

Stall is taken off the circulating system and decompressed.

C.) Assumption: Leaks in the system.

The stall is taken off the circulating system, switched to pasting oil and decompressed.

Letdown (Entschlammung) of the converters is possible only at low temperature and slowly thru hand operated valves.

Measures taken at Pölitz: 700 atm.

Normal maximum temperature 25.2 mV (40°C).

Assumption: Normal gas flow.

The temperature rises to 25.5 mV quite often. It has been repeatedly possible to bring down the temperature with cold gas from the temperature range of up to 26 mV. Make emergency letdown at 26.5 mV in the following manner:

Close electric valves on pressure and suction side. Open converter I, angle valve, tower valve, converters 2 to 4 and catch pot in the normal manner thru nozzle. It takes 20-30 seconds to operate the electric valves. If the stall doesn't burn (break open), try to stop the letdown between 150 and 100 atm and to continue the letdown normally. The heat exchangers are emptied at the same time.

In order to keep the preheater and heat exchangers open, pasting oil is run while these measures are being taken. During the emergency letdown, provided the stall does not leak due to a break, all inlet-cold gas, paste gas, cooling gas, etc, valves are closed and then the electric valve on the pressure side of the circulating system is again opened and the individual gas passages are flushed by sound one after another by a quick opening and closing. The preheater is run cold, just as in Ludwigshafen. Depending upon conditions, the letdown may be broken off at intermediate pressures and the stall restarted (hochgefahren). During the refilling, the individual emergency letdown lines are flushed with oil.

Starting up a Coal Stall

Ludwigshafen: The compressed and thoroughly checked stall is first flushed with nitrogen in the normal direction of flow to the expansion valve just ahead of the suction side. (Flushing and refilling of the converter is also done thru the cool gas lines). A gas analysis is taken. When no oxygen is present, the stall is filled with make-up hydrogen (240 atm), with the stall by-pass open. Filling must be done very slowly, due to overloading the H_2-washer (time 5 to 8 hrs, converter volume 1.6 m^3). The circulating system is filled at the same time. When the circulating system and the stall have been compressed to 240 atm (the Oppau plant pressure), the stall by-pass is closed. The circulating pump is then started and the stall is given gas at 240 atm (2000 m^3). When everything is in order, gas meters, monometers, etc, the preheater is fired. The blind disks in the fuel gas and illuminating gas lines are pulled out for this purpose; they are inserted every time the stall is shut down. Before this, the gas preheater is flushed out with air by starting the circulating blower, producing a negative pressure in the preheater, then sucking fresh air thru the pilot flame holes and exhausting to atmosphere on the pressure side. The shutoff dampers to the burners are closed. After an hour the pilot flames are ignited. Then the middle burner of each burner group (2 groups of 3 burners each) is ignited and the converter temperature is increased 1 mV/hr to 12 mV. From 12 mV on the increase is 0.5-0.7 mV/hr. With a new converter middle oil or catch pot heavy oil injection is started at 6 mV (1000-1200 Ltr). With an old converter injection is started immediately after igniting the preheater, in order to prevent drying out and subsequent scaling and accumulation of scale in the lines. As soon as the level indicator in the catch pot registers and functions decompression is started over the letdown group. When everything is in order here, too, the pressure is slowly increased to 700 atm at the rate of 50 atm/hr. At 14-12 mV the feed is switched to pasting oil. After 700 atm. is reached pasting oil is injected at the rate later intended. At 21.5 mV the feed is switched to coal paste, after first checking the cold gases. The switch-over is made practically simultaneously to thin and thick paste. First coal paste and pasting oil is run in the proportion of 1:1 for 2 hours. (They are mixed in the suction vessel). Then, by injecting pasting oil into the thin paste line in the heat exchanger, the desired concentration of solids is slowly obtained and the switchover to full thick paste concentration is made by means of 75:25 thick paste plus pasting oil for 2 hrs. The converters slowly reach about 23.5 mV.

At this temperature the converter is left to rest and the correct gas quantity is adjusted. The quantity of gas is uniformly increased or decreased at the rate of 50 m^3/h. The converter is pulled up to reaction temperature (25.1 - 25.2 mV), at a rate depending upon the activity of the converters. Between 23.5 and 24.2 mV the temperature is increased 1/10 mV every 2 houss and the converter then left to rest a little every time. From 24.2 mV upwards, as the case may be, the operation may proceed even more carefully.

Make-up Gas.

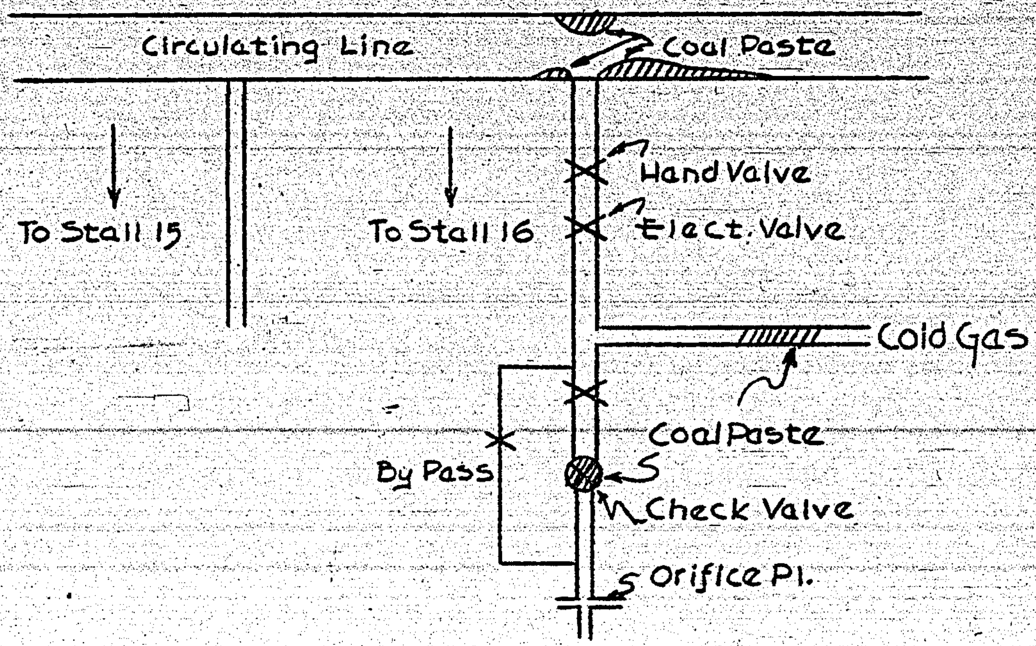
Ludwigshafen: The make-up gas line Oppau-Ludwigshafen (Lu 35) is insulated and provided with a heating tracer, but has not been heated for years. Formerly, trouble was encountered due to water (freezing). But since drainage was installed at Oppau no more trouble was had. At the point of entry at Ludwigshafen, 2 water separators have been installed in the line, in series, which are drained every week. The line is insulated and heated to about 100 m behind these separators. From there on to the inlet to the stall it is neither insulated nor heated. No difficulties have, as yet occurred due to NH_3. Abnormal quantities of CO have been indicated by remote recorder.

Leuna: Make-up gas line heated to inlet to circulating line. Because of catalyst 6434, the make-up gas for the gasoline circulating system is washed with water to 0.1 mg NH_3/m^3. To assure a satisfactory NH_3 analysis the sample is taken thru a well heated sampling tube (3 mm), extending into the middle of the line.

Pölitz: The make-up gas line was formerly heated only to the distributing group (manifold ?). Difficulties due to NH_3 were encountered, particularly in Stall Ia (T-52 operated with make-up gas). There are comparatively large lines here with small flow. Clogging also occurred in the make-up gas lines of the operating control system (flushing gas for level indicators, etc). All these lines were heated. A water wash was also installed near Stall Ia, which reduced the ammonia content of 5-8 mg in the make-up gas to about 25% thereof.

The Accident on the 26 June, 1942.

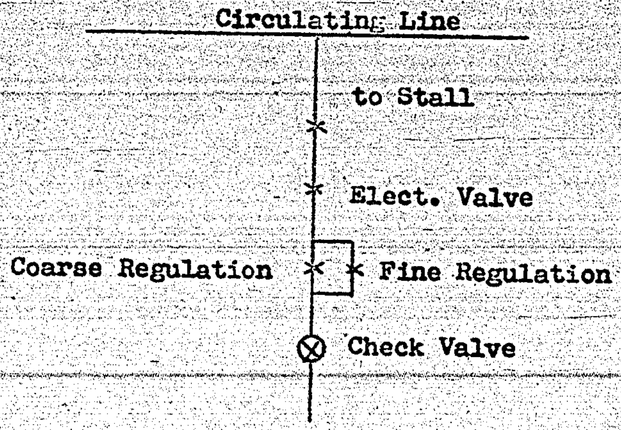
On opening the circulating line it was found that the check valve to Stall 16 was smeared with coal paste. Coal paste was also found at the branch line connection to Stall 16 in the circulating line, see Sketch I; analysis: 61% solids and 8-10% ash.



The coal paste must have found its way behind the check valve during the power failure of the 27 May, 1942. It is not known if the by-pass around the check valve was open at that time. At the time of the accident on the 26 June, 1942, the stall was operated thru the partially open by-pass and regulated by the by-pass valve. After the accident fresh thin paste was found in the by-pass line.

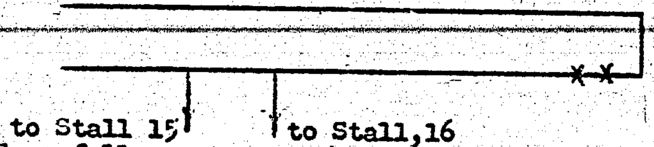
Leuna has no check valves on the gas inlet to the stalls. No disturbances due to a back flow of coal paste to the circulating line have so far occurred.

Ludwigshafen has check valves without by-pass. See Sketch II.

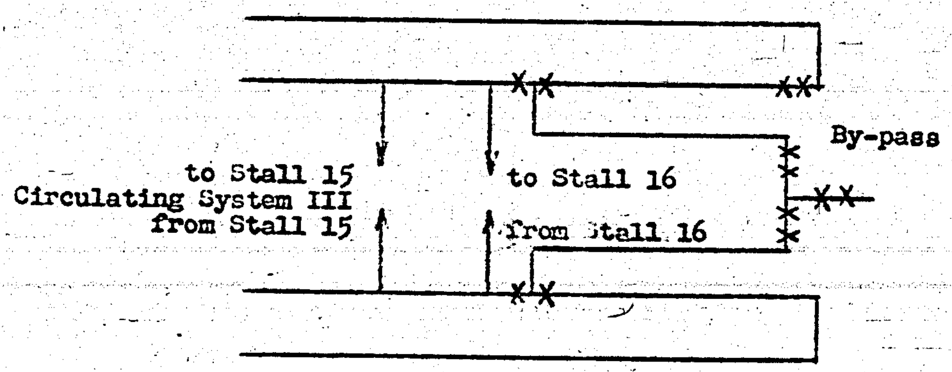


Pölitz has changed the original arrangement, Sketch I, after the accident. The new arrangement corresponds to Sketch II.

Changes in circulating system at Pölitz : The long pocket in Recirculating System III, pressure side.



was changed as follows:



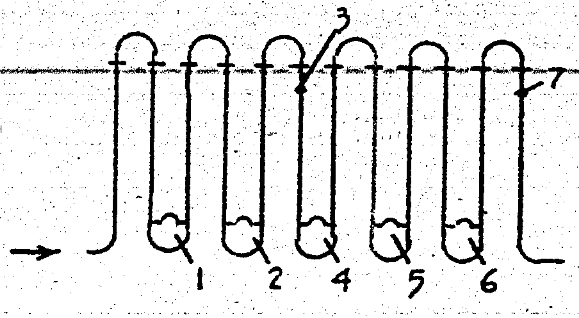
Due to this change, Circulating System III can be blocked off immediately behind the junction to Stall 16. The new by-pass ahead of the junction to Stall 16 also permits a flow in those parts not yet in operation at the time by careful opening. After the circulating system has been warmed up, connection is made by opening the last valves against Circulating System II and closing the by-pass.

Circulating System XI is disconnected at present (lowering the circulating cooler). The by-pass described has also been installed in this circulating system.

5 mm steel springs are used in the check valves, because the 8 mm springs often broke (springs are too stiff).

Electrical Preheater in the Prehydrogenation Stalls at Pölitz.

In the past, the elements have been mounted on the lower hairpin bends outside of the power supply lines, with the exception of 2 elements. Stall 8, e.g.:



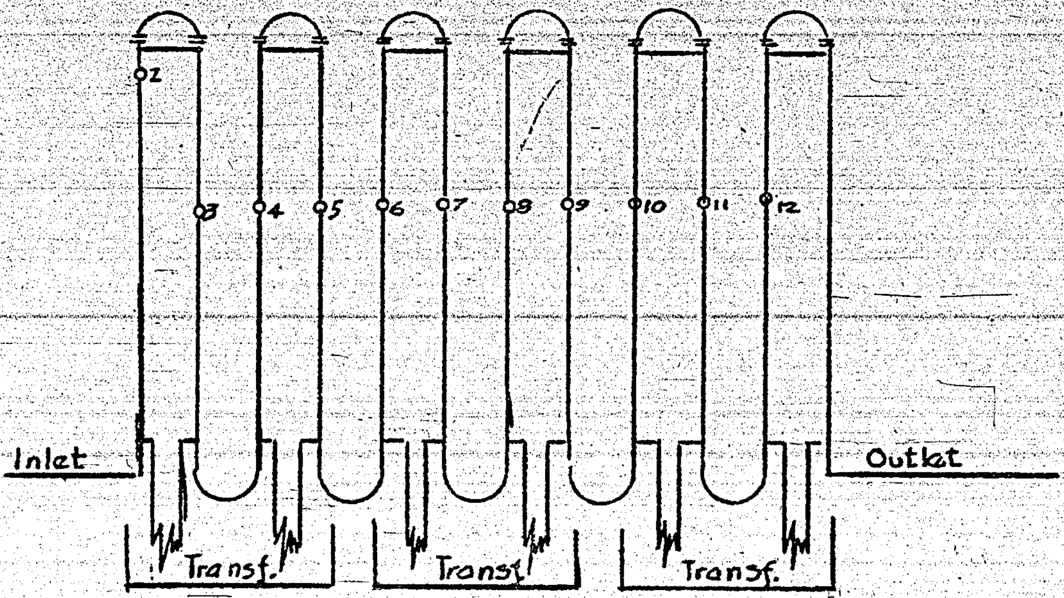
Temperatures of Stall 8 on the 25. July, 1942.

Power load = 1560 KW.

Element	1	2	3	4	5	6	7
mV	19.5	21.0	21.5	21.5	21.0	21.7	23.9

Measurements on a heated hairpin showed that the maximum temperatures occurred about in the middle of its length.

Even before this was known, a number of additional elements were mounted in the middle of the hairpins in Stall 6.



Temperatures of Stall 6 on the 26. Aug. 1942.

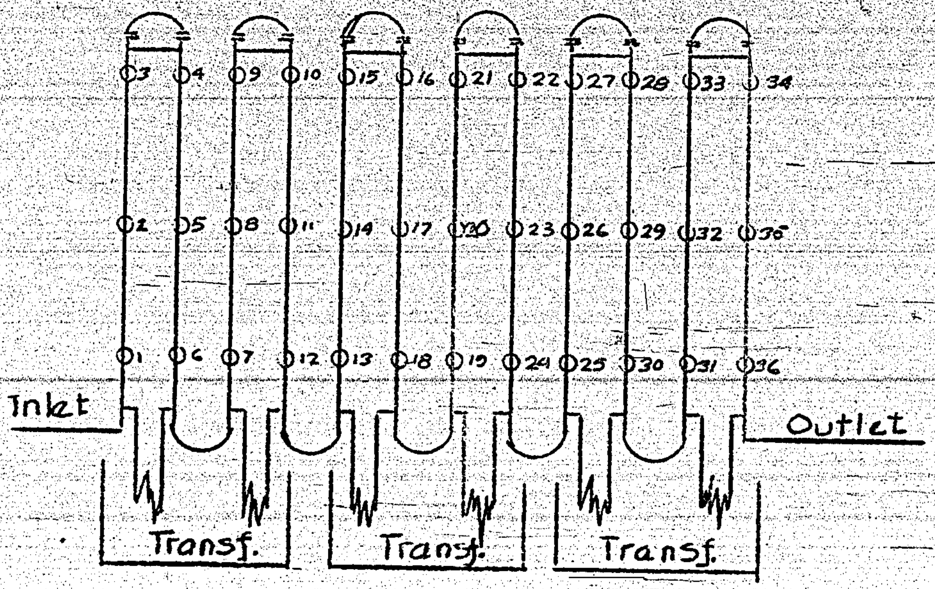
Power load = 520 + 220 + 320 = 1060 KW

Element	1	2	3	4	5	6	7
mV	9.6	20.6	21.4	21.7	22.4	19.6	26.0

Element	8	9	11	12	13
mV	24.0	20.4	20.2	25.8	21.1

In this preheater the hairpins are insulated with brick for greater efficiency.

When the Stall 7 preheater was rebuilt, due to the bursting of a tube, the elements were arranged as follows:



Temperatures of Stall 7 on the 26. Aug. 1942.

Element	mV	Element	mV
1	18.8	19	19.0
2	19.0	20	19.0
3	19.0	21	19.1
4	19.2	22	19.0
5	19.2	23	19.2
6	19.2	24	18.8
7	19.0	25	19.0
8	19.2	26	19.1
9	19.2	27	19.2
10	19.0	28	19.1
11	19.1	29	19.2
12	19.0	30	19.2
13	19.0	31	19.0
14	19.1	32	19.0
15	19.1	33	19.3
16	19.1	34	19.3
17	19.0	35	19.2
18	19.0	36	19.5

Arrangement in Electric Preheaters at Ludwigshafen.

All elements are mounted within the zone of current flow. Reserve elements have been mounted at several points, which elements can be connected to the switch box in case of failure of the normal elements.

Flushing out Stalls after Shut-Down or Failure.

Leuna: The preheater is flushed at about 6 mV. Flushing may also be done thru the cold gas lines. To control the flushing the elements are watched (rise in temperature), so that the current flow can be determined, which is of particular importance in heat exchanger short circuits.

Converter Letdown Lines.

Leuna: The converter letdown lines are worn by the sand in the letdown. For this reason the lines are inspected from time to time by taking out bends. Each stall has 2 letdown lines. Particular care must be taken that material, which has already been used in letdown lines is not used anywhere else.

Pölitz: Because several stalls are connected to a letdown line to the tower, such an inspection can be made only when all these stalls are shut down. Wherever this occurs an inspection is to be made.

Up to date wear has only been noticed in the housings of the tower valves. The letdown line is constantly under a pressure of 700 atm. The pressure against the tower is reduced to about 300 atm by 4 throttling orifice plates in the line.

Connecting Cold Gas Valves.

Leuna: There is no cold gas supply between the converters. Most of the cold gas is injected as far as possible into the converter. This cold gas quantity is kept as constant as possible. Small quantities of cold gas in the upper part of the converter are used for regulation. The converter is run so that the converter inlet (preheater outlet) is kept constant and the converter outlet is watched. There have been times when Leuna operated with the outlet elements alone on failure of the inner elements. As long as no increased temperature is observed at the outlet, there is no danger on the inside of the converter.

Pölitz and Ludwigshafen: With bituminous coal the converter can not be operated without constant observation of the inner elements.

Water Injection into Vapor Phase Stalls.

Leuna: Water is injected on the top of the cold heat exchanger and before the product cooler. Formerly the water was injected at the bottom of the rising line, which caused severe corrosion at these points (formation of hydrochloric acid).

Pölitz: Water is injected at the bottom of the rising line before the cold heat exchanger and before the product cooler. No corrosion has been observed.

Ludwigshafen: Water injection as in Leuna.

Power Failure.

Leuna: The circulating pumps are steam driven. Four gas driven pumps are also on hand. At steam failure (severe leaks) the circulating pumps continue to operate with reduced capacity.

Pölitz: Only electrically operated circulating pumps are on hand, at present, but 4 additional steam pumps are to be installed. Two power circuits are avilable, each circuit with 2 additional subdivisions, so that only half the circulating pumps drop out, if one circuit fails.

At present, only part of the circulating pumps can be switched onto the other circuit, just as in Gelsenberg, but arrangements are being made to permit switching of all pumps automatically onto the other circuit. When the power supply is sufficient, compressors are first disconnected so that the circulating pumps may receive more current. A decrease in the quantity of gas and a drop in pressure must, of course, be figured on. However, the liquid phase is not reduced at all, because the vapor phase is cut back first of all.

Ludwigshafen: The circulating pumps are electrically driven. Two circuits are available, which, however, can not be switched over automatically.

Switchover or Connection of Circulating Lines.

Leuna: The procedure varies, according to whether long or short portions are to be connected. It also depends upon the temperature of the portions to be connected. There are no expansion bends in Leuna and one must, therefore, proceed very carefully in order to prevent leaks. It has happened in Leuna that water condensed in cold portions of the circulating line (pressure side). The switchover is primarily made only in day time, and then in the fore-noon. Opening the last gate valve takes 1 to 2 hours.

Politz: Liquid Phase: New Procedures:

1.) At switch overs in the circulating line the thruput of the coal stalls is reduced (perhaps 20 to 40%) and the temperature is reduced by 0.5 to 0.7 mV, to provide greater leeway in case of a runaway due to gas failure.

2.) In circulating system III the 50 m pocket was eliminated by moving the gate valves close to the branch line to Stall 16.

3.) 30 mm by-pass lines were installed on the shutoffs between the pressure and the suction side (takeoffs between the 2 shutoff valves.

Connecting the circulating lines is done so that when the shutoff gates and the by-pass are closed the pressure and the suction side are filled by temporary 6 mm lines from the pressure or suction side respectively. The differential pressure valve of the part to be connected is closed meanwhile. The filling takes 24 hours. Then the valves at the junction are slowly opened and the gas is allowed to flow carefully thru the by-pass for 12 hours (abt. 20000 m^3), until the new portion of the circulating line is thoroughly warmed up. The junction is considered to be that end of the new circulating line, from which the filling was done, i.e. towards the pumps on the pressure side and towards the scrubber on the suction side. Before opening the by-pass, the operator at the differential pressure valve on the other circulating line and the operators at the gas inlets and the converters of the stalls on this circulating line are notified. Then we proceed to open the gate to the stall side of the circulating line. It takes about an hour to open this gate fully. Then the by-pass is slowly closed. No change is made in the stall until 3 hours after connecting a new circulating line.

The pressure side of the circulating lines is not insulated. The temperature (heat of compression) of the pipes is about 60-70° C. The suction side is entirely heated and insulated and has a temperature of 85°C. The temperatures are measured by round tied-on thermometers and are regularly checked and registered.

Disconnecting a Portion of a Circulating Line.

After closing the valve maintaining the pressure differential, the line to be disconnected is first cut off on the pressure side by slowly closing the shutoff valve on the stall side (duration one hour). If no disturbance is indicated, the part of the line to be disconnected is also blocked off on the side of the pumps.

Analogous procedure on the suction side.

THERMOCOUPLE MEASUREMENTS IN HIGH PRESSURE CONVERTERS.

From Dr. Pier's Private Files.

High pressure experiments
P/Leuna 558

Ludwigshafen a.Rh.
April 9, 1942.

Methods have been proposed permitting an early recognition of the "passing through" the converter or to prevent its undesirable effect upon the operation of the stall. During the discussion reference was made to the observation first made in Pölitz of compression a thermocouple sleeve in an immersion well of the cover insulation.

You will find in the appendix a report of the Gelsenberg Benzin A.G. of investigation of such a compressed thermocouple socket, especially in connection to the effects on temperature measurements introduced by it.

2 signatures, illegible.

Appendix.

Gelsenkirchen Benzin A.G.
Control Division.

Gelsenkirchen-Horst,
1/23/42.

Report of Tests No. 5

BEHAVIOR OF THE THERMOCOUPLES DURING A LOCAL "PASSING THROUGH" OF CONVERTERS.

Operations.

On the 11/5/41 at 17:30 o'clock the bottom thermocouple 101 of the coal stall 706 indicated a rise from 23.5 to 26.5 mv and the stall was changed over to oil operations. At 18:30 o'clock the thermocouple 101 returned to its original reading of 23.5 mv. Attempts were then made to resume coal injection. The thermocouple reading again rose (to 24.5 mv) at 1:50 o'clock and the thermocouple 105 also ready 25.2 mv, and operations were changed over to pasting oil. The thermocouples again came down to normal and at 3:30 o'clock a new attempt was made to inject coal paste. At 5:40 all the thermocouples in one socket rose to 30 - 40 mv, and the stall operations had to be changed over immediately. The couples came again back to normal, but again when operations with paste were attempted the temperatures rose. These experiences in operation have led us to assume that the converter was becoming slowly coked up from below, without offering any explanations on why this should have happened in converter I.

However, dismantling the converter showed it to our surprise to be completely unobstructed and that the thermocouple sleeve was merely squeezed together over a length of 280 mm inside the immersion well, indicating thus the existence of some local reaction inside the well. Dismantling showed also that the originally observed rise of the bottom thermocouple did not represent a real rise in temperature in the converter but only in the thermocouple well. Fig. 1 again represents the squeezed place on a greatly increased scale. The compression of the well is seen to be so great, that all the vacant space is completely filled with the ceramic insulation and the wires, and the wires themselves are greatly elongated. It is evident, that with such a compression of the well over a length of 280 mm the ceramic material was crushed and the wires formed a more or less perfect contact with each other.

It was now tested what temperatures would be indicated by such bundles of the elements.

1. Behavior of a single new thermocouple by heating to different temperatures in two standarizing furnaces.

A new thermocouple was first carried through two standardizing furnaces.

The junction was located in furnace No. I, the leads were insulated and passed through the furnace No. II. In this case the thermocouple indicates correctly the temperature of the junction, regardless of the temperature of the second furnace, provided the changes in resistance were taken into consideration. Should however a contact between the wires be formed inside the second furnace, the reading will represent the temperature of the second furnace. The EMF of the first furnace is shortcircuited and disappears entirely. A short circuit in the second furnace transfers the temperature measurement from furnace I to furnace II.

2. The behavior of the dismantled and squeezed thermal well when heated in two furnaces.

The squeezed piece of the dismantled thermocouple was cut out, the elements separated, connected to a new thermocouple, and the same test as described under 1. was repeated. Fundamentally the same results are obtained. The different thermocouples are interconnected, so that the same constantan wire can be used for the measurement of the thermoelectric power of every iron wire. The temperature of both furnaces was at first kept the same. This temperature was indicated by every constantan wire against every iron wire, with very few exceptions. There was no connection in series of the different couples.

The standardizing furnace I. i.e. the rear furnace, was then kept at 17 mv, while the front furnace with the squeezed part was raised to 24.1 mv. Almost all the constantan wires showed with the iron wires the temperature of the furnace II. A few of the couples gave more or less lower readings, to be attributed to loose shorts in the 280 mm of the squeezed section.

Were we to apply these results to the operational observations described above, we will see that the local immersion well reaction had first shorted the elements, and indicated therefore the temperatures in these particular points. As the squeezing progressed the elements became shorted all over, and all the thermocouples gave readings of the squeezed section. In case of a local contraction of the thermal well the junctions are to all intents transferred to the contracted section, giving an impression as if the temperature of the whole converter was rising, while one actually reads only the temperature of the compressed section.

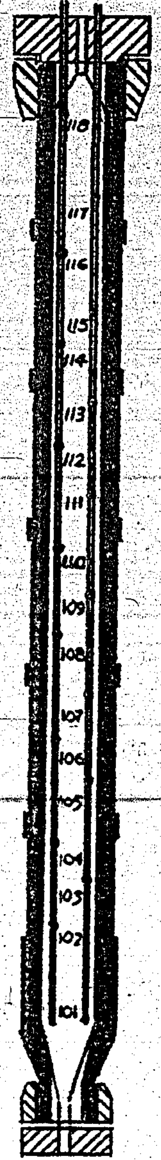
Summary.

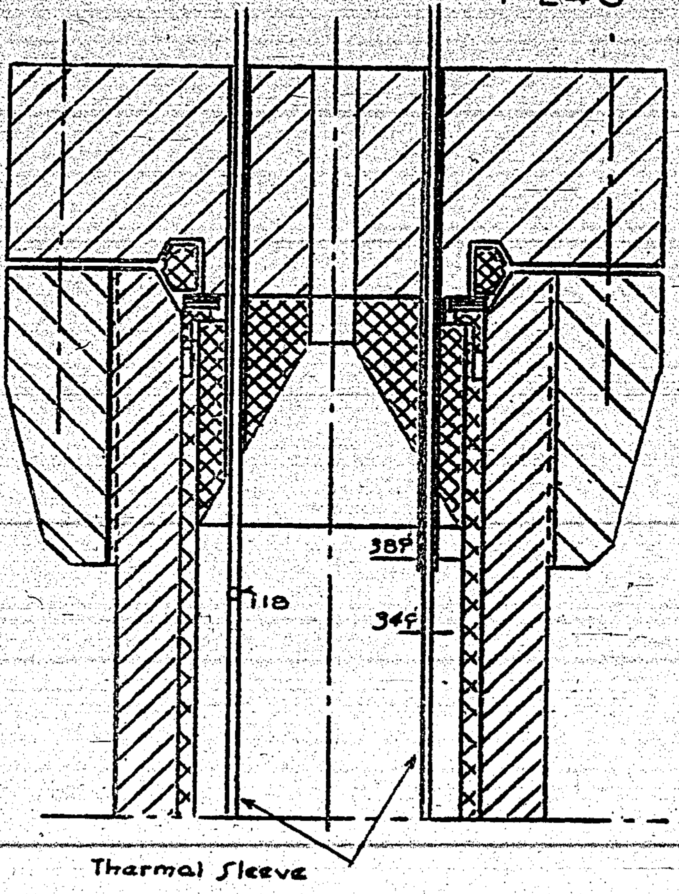
In stall 706 the readings of the bottom thermocouples rose first, then of all the thermocouples to 40 mv. This has led to the suspicion the converter was coked from the bottom up. Dismantling showed however a compression of the thermocouple tubes inside the thermal wells. It has been proven, that the junction of the elements is shifted by the squeezing together of the wells, and that the temperature readings are made in a different spot. The thermocouples have not been observed becoming connected in series, but too low readings were observed on some of the thermocouples, which were attributed to shorts inside the squeezed section.

Signed-- The Underwriters.

W.M. Sternberg.

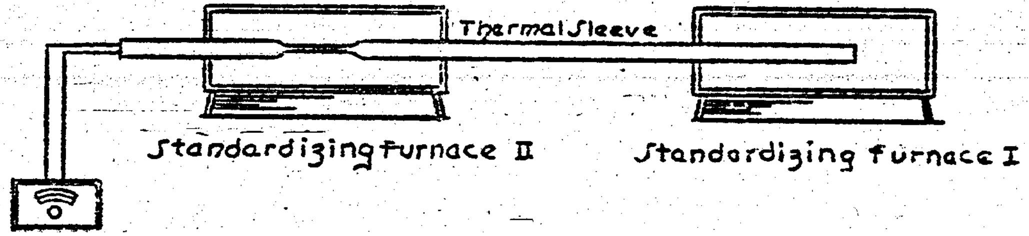
Constantan-Wire	against	Fe-Wire	Test I	Test II
No. 1		No. 1	17.2 mV	24.0 mV
" 1		" 2	17.2 "	22.5 "
" 1		" 3	16.7 "	22.0 "
" 1		" 4	17.2 "	23.0 "
" 1		" 5	17.2 "	22.5 "
" 1		" 6	17.2 "	22.7 "
" 2		" 1	17.0 "	17.0 "
" 2		" 2	17.0 "	18.0 "
" 2		" 3	17.2 "	21.5 "
" 2		" 4	17.2 "	18.3 "
" 2		" 5	17.0 "	16.8 "
" 2		" 6	17.0 "	18.5 "
" 3		" 1	17.0 "	23.3 "
" 3		" 2	17.2 "	22.0 "
" 3		" 3	17.0 "	23.3 "
" 3		" 4	17.0 "	23.2 "
" 3		" 5	17.0 "	24.8 "
" 3		" 6	17.2 "	22.5 "
" 4		" 1	17.2 "	22.5 "
" 4		" 2	17.0 "	23.8 "
" 4		" 3	17.2 "	22.2 "
" 4		" 4	17.2 "	25.2 "
" 4		" 5	17.0 "	23.6 "
" 4		" 6	16.2 "	22.6 "
" 5		" 1	17.2 "	14.3 "
" 5		" 2	17.2 "	12.4 "
" 5		" 3	17.2 "	12.7 "
" 5		" 4	17.2 "	12.4 "
" 5		" 5	17.2 "	10.7 "
" 5		" 6	17.2 "	20.0 "
" 6		" 1	17.2 "	20.0 "
" 6		" 2	17.2 "	20.0 "
" 6		" 3	17.2 "	19.6 "
" 6		" 4	17.2 "	19.0 "
" 6		" 5	17.2 "	21.5 "
" 6		" 6	17.2 "	18.3 "
" 7		" 1	17.2 "	19.7 "
" 7		" 2	17.2 "	22.3 "
" 7		" 3	17.2 "	23.5 "
" 7		" 4	17.2 "	23.7 "
" 7		" 5	17.2 "	22.2 "
" 7		" 6	17.2 "	23.2 "
" 8		" 1	17.2 "	13.3 "
" 8		" 2	17.2 "	16.0 "
" 8		" 3	17.2	17.2 "
" 8		" 4	17.2 "	17.0 "
" 8		" 5	17.2 "	15.6 "
" 8		6	17.2 "	17.0 "





COAL STALL 706, CONVERTER I
REACTION IN THE IMMERSION WELL.
11/5/1941

Sleeve compressed 280 mm.



U. S. Bureau of Mines

KCBraun
2-14-47

THE CENTRIFUGING OF TARS AND LIQUID PHASE SLUDGES (Let-Downs) OF TARS

Ludwigshafen, 7 June, 1944

The data on centrifuging of brown coal crude tars and liquid phase sludges (let-downs) of such tars supplied us by the hydrogenation plants are summarized and compared in the following tables.

Table I contains data on centrifuging of crude tars. Various types of centrifuges are used, Haubold, Escher-Wyss, and Heine. Centrifuging is done partly continuously, partly in batches. Zeitz, Leuna and Brüx use high speed Haubold high capacity centrifuges, Code No. 1200. It is clearly apparent that the results of centrifuging are very much dependent upon the quality of the tar. For this reason, plants processing tars of different origin together use suitable mixtures with tars of good centrifuging properties. For example, Böhlen mixes its own tar with foreign tars and Leuna mixes gas producer tars with Wölfersheimer low temperature carbonization tar (Schwelteer), plus small quantities of sulfuric acid. The Brüxer thick tar (high asphalt content), which is less suited to centrifuging, stands out clearly in its smaller centrifuge thruputs. Because of the high requirements of purity in the processing of TTH at Zeitz, centrifuging is followed by filtration and water washing.

Table II deals with the centrifuging of liquid phase letdown from brown coal tar hydrogenation. The Böhlen letdown can be filtered in filter presses under normal conditions, in which process the solids content is reduced to 0.3%. The filtered letdown is mixed with the crude tar and centrifuged. If the letdown is of poor quality or there is too much of it (not enough filtrate), an attempt is made to remove the solids by repeated centrifuging.

It was shown in Magdeburg that the addition of their letdown to the crude tar in centrifuging caused an unbearable deterioration in the quality of the pure tar in respect to solids content. Because the existing slow speed Escher-Wyss centrifuges could not successfully process the original letdown, high speed Heine centrifuges were installed (capacity 200 kg, hand operated, widened chute).

The high speed Haubold centrifuges used at Brüx must be batch operated, 25 minutes per batch, in order to reduce the solids content of the centrifuge oil to 6% when centrifuging Brüx letdown. The centrifuging capacity is, therefore, very low. Centrifuging the letdown mixed with fresh tar was also unsatisfactory, just as in Magdeburg. The accumulation of the finest solids in the centrifuge oil, which is returned to hydrogenation, is very pronounced. Leuna has experimented in centrifuging some tank cars full of letdown centrifuge oil from Brüx mixed with their usual tar mixture (Wölfersheimer tar and producer tar). When 10% of the Brüx centrifuge oil was added, the centrifuging time had to be increased from the normal 3 minutes to 6 minutes in order to obtain the same purity in the centrifuge oil as with tar alone.

Just as the crude tars themselves, the letdowns from the various tars showed wide variations in their ability to be centrifuged or filtered. The letdown from Brüx tar will probably have to be processed mixed with foreign tars easily centrifuged, or part of the letdown produced may have to be topped or carbonized (L.T.C.)

Table I. Centrifuging Tars

PLANT	BÖHLEN	MAGDEBURG	ZEITZ	LEUNA		BRÜX
1. Centrifuge Feed.	Fresh Tars: ASW Böhlen Foreign Tars { " Hirschfelde " Espenhain Kulkwitz	Fresh Tars: Offleben Riebeck Nachterstedt Salzdethfurt	Fresh Tars: ASW Böhlen " Espenhain Riebeck Deuben	Crude Tars with over 1% water or dust. Wölfersheimer L.T.C. Tar, Kapfenberg producer tar Langelsheim " " Allendorf " " Riebeck L.T.C. " Producer tars must be mixed with L.T.C. tars. Centrifuge at 100°C, add 40-100 L conc. H_2SO_4/40 m^3 tars.		Thick Tar from Centrifuge
2. Kind & Type of Centrifuge.	Hambold Chemnitz	Escher-Wyss	Hambold Chemnitz	Heine	Hambold	Hambold Chemnitz Ho.C.St. 1500
R.P.M.	600	600	1200	580	1200	1200
Dia.-mm	2000	2000	1500	2000	1500	1500
Code Number	400	400	1200	375	1200	1200
Other special Characteristics	Batch operated	Filling, scraping & cutting out is oil pressure controlled.		Belt drive	Motor & gear drive. Scraping & cut-out devices changed by Leuna.	
				Closed off gas-tight & blanketed with nitrogen.		
3. Operating Conditions: Feed Temp.	75-80°	70-80°	80°	90 - 100°		104°
Circulating Line Press.	3-5 atm.	2 atm.	4-6 atm.	2 atm.		2-3 atm.
Orifice dia.	--		Inlet nozzle 6mm	--	35 mm	Inlet nozzle 6mm
Other	--	Filling valve CO_2 Blanket		Filling time 45min.	Filling Time 2 min. +	Continuous Operation
				Centrifuge Time -3 min.		

Table I. Continued

PLANT	BOHLEN	MAGDEBURG	ZEITZ	LEUNA		BRUX
4. Operating Results:				(Heine)	(Hambold)	
Feed/h/Contr.	4.5-5.0 t	5.08 t	4.6 t	4	2	1.18 t
Contr. Oil	4.3-4.5 t (92.8%)	—	3.74 t (81.3%)	abt. 94.6%		1.16 t (98.3%)
Contr. Residue	0.18-0.39 t (6.0%)	0.0235 t (0.5%)	0.49 t (10.7%)	" 3.6%		0.02 t (1.7%)
Loss	0.009-0.012 t (0.2%)	—	0.37 t (8.0%) Loss partly recovered	" 1.8%		—
5. Total number of Contrif.	11	7	26	5 9	4	9
No. in operation	63-98%	85.5%	65.5%	6		80%
" " repair	0.7 – 11%	3.5%	13.4%	2		10%
" ready for operation	1.9-26%	11.0%	21.1%	1 Centrif. operated only daytime (240 h/month)		10%
6. Is flushing done?	Yes	No	No	Residue is cutout after 5-10 fillings.		No
Average Operating Period	1 year	1-2 years	—	6 months	3 months	Operating 2 years.

TABLE I Continued

PLANT	BÖHLEN			MAGDEBURG			ZEITZ			LEUNA					BRÜX		
7. Analyses:	Inj. Prod.	Cent. Oil	Cent. Resid.	Inj. Prod.	Cent. Oil	Cent. Resid.	Inj. Prod.	Cent. Oil	Cent. Resid.	Wölfsh. Tar	Pred. Tar	Cent. Mix.	Cent. Oil.	Cent. Resid.	Inj. Prod.	Cent. Oil	Cent. Resid.
Spec. Grav.	0.949 60°	0.940 60°	1.078 25°	0.920 0.950 50°	0.990 1.000 50°	1.150 1.200 80°	0.948 50°	—	—	0.940 50°	1.050 50°	0.976 50°	0.930 80°	—	1.075 80°	1.053 80°	—
% Solids (benzol)	0.36	0.08	43.2	0.2- 0.8	—	—	0.49	0.06	45.0	1.2	0.8	0.8	0.1	45.0	—	—	—
% " (Xylol)	—	—	—	—	—	—	—	—	—	—	—	—	—	—	2.7	2.0	49.3
% Water	—	—	—	—	—	—	—	—	—	1.2	13.0	3.0	0.07	8.0	—	—	—
% Ash in solids	—	—	22	30	—	—	10	—	—	—	31.1	17.3	—	—	—	—	—
% Asphalt in oil	—	3.2	—	2.5	—	—	2.8	—	—	0.8	10.3	4.2	4.0	—	19.5	19.5	—
% -325° " "	—	28.7	—	30	—	—	33	—	—	25-35	25-35	25-35	25-35	25-35	7.3	7.3	—
% -325° " vacuum	—	65.8	—	—	—	—	90	—	—	—	—	—	—	—	—	—	—
Organic Solids:																	
% C							91.5										
% H							8.5										
Oil —— % C		83.48						83.6								82.50	
% H		10.38						10.6								8.60	
% O		4.81						4.0								7.91	
% N		0.22														0.54	
% S		1.11						1.8								0.45	
Ash in % SiO							13.0										
% Fe_2O_3							8.9										
% Al_2O_3							12.3										
% CaO							21.2										
% MgO							4.5										
% Na_2O							1.0										
% SO_3							33.1										
% Cl							Trace										
Particle Size	<2u			—			—										

Table I - Continued

PLANT	BÖHLEN	MAGDEBURG	ZEITZ	LEUNA	BRUX
8. Centrifuge Time	Böhlen Tar-3 min, with foreign tar addition up to 7 minutes		The high speed centrifuges showed no increased centrifuge effect with increased centrifuge time for similar tars, therefore, continuous operation (double thruput compared to batch operation).		Better results were obtained with continuous operation (6 mm inj. nozzle, 1.2 t/h thruput) than with batch operation, 5 min.
9. Further processing of the centrifuged tars and the residue.	Pure tar with light oil to liq. ph. distillation. Residue partly processed in extraction filter presses, partly to dump.	Pure tar to hydrogenation. Residue briquetted with producer ash.	After filtration in presses, wash with H_2O and centrifuge off the H_2O. Together with light oil to hydrogenation. Residue processed in other plants.	Pure tar together with coal letdown to distillation. Residue to dump, later proposed L.T.C. (H_2O content)	Pure tar to liquid phase. Residue mixed with abt. 50% pulv. coal for shipment.

TABLE II Centrifuging Liquid Phase Tar Sludges.

PLANT	BOHLEN	MAGDEBURG	BRÜX
1. Centrifuge Feed	a) Filtered Liq. Ph. Letdown, 0.3% Solids Content, mixed with crude tar. b) If solids content of filtrate exceeds 0.3%, mix with waste oil. c) If more letdown is produced than filter presses can take, centrifuge repeatedly alone and with addition of waste oil and crude tar.	Liquid Phase Letdown.	Liquid Phase Letdown.
2. Kind & Type of Centrifuge:	Hanbold	Heine	Hanbold
R.P.M.	600	1500	1200
Dia.-mm	2000	1200	1500
Code Number	400	1500	1200
Other Special characteristics	Batch Operation	Hand operated scraping knife, cutting out is done with motor shut off, widened chute, poking is necessary.	Batch operation
3. Operating Conditions:			
Feed temp.	Compare Table I	80-100°	96°
Circ. Line Press.		0.5 atm.	2-3 atm.
Orifice Dia.			Adjustable Plate Valve
Other		(CO_2 blanket. scraping knife must be replaced after 400-500 fillings.	

TABLE II Continued

PLANT	BÖHLEN			MAGDEBURG			BRUX		
4. Operating Results: Feed/h/Centr. Centrif. Oil Cent. Residue Loss	Compare Table I			Capacity 200 Kg. 60-65% 40-35% —			0.52 t 0.41 t (79%) 0.11 t (21%)		
5. Total Number of Centrifuges No. in operation " " reserve " ready for operation	Compare Table I.			2 90% 10%			11 90% 5% 5%		
6. Is flushing done? average operating period	Compare Table I.			No 1-2 years					
7. Analyses:	Inject. Prod.	Centrif Oil	Centrif. Residue	Inject. Prod.	Centrif. Oil	Centrif Residue	Inject. Prod.	Centrif. Oil	Centrif. Residue
Spec. Grav.							1.147/80°	1.058/80°	
% Solids (Benzol)	>0.3	0.12-0		25	1-2	50			
% " (Xylol)							15.1	5.8	50.9
% Water							—	—	
% ash in solids							49.6	—	
% asphalt in oil							1.1	1.1	
%-325° " "							9.4	9.4	
%-325° " vacuum							10.5	10	
Oil — % C								84	
% H								7.1	
% O								6.7	
% N								0.42	
% S								0.22	
Particle Size							60% <1 u		
8. Centrifuge time	Up to 20 min (3-7 min for tars)			15 min.			25 min.		
9. Miscellaneous	Letdown filtrate in filter presses. 5m³/24h/press (@5-6 atm pump pressure).								

From Dr. Pier's private files.
U. S. Bureau of Mines
Hydro. Demon. Plant Div.
T-242

Discussions between Leuna, Scholven,
Gelsenberg, Pölitz, Welheim, Brabag
Rheinbraun, Lützkendorf, Oberschlesien,
and Brüx

High Pressure Experiments
P/Leuna 558

Ludwigshafen a. Rh
April 1, 1941.

TEMPERATURE MEASUREMENTS AT THE PREHEATER OUTLET.

I. In general, measurements of temperatures are important for the calculation of the heat efficiency and of the K value of a gas heated preheater. The most important temperature is, however, the outlet preheater temperature (in most cases designated as El.44). This measurement is difficult, because pressure-resistant thermocouples have for several reasons been discarded and we are in most cases forced to use tied on, welded on or even thermocouples stuck into bore holes for measurements. Their temperature indications are in most cases 5 to 15° C too low, depending on the way they are connected, and on the insolation. In this temperature range, 10° already make a large difference in the dimensions of a preheater.

The errors of these locations of measurement can however be rather closely established by comparing the readings of this thermocouple (El.44) with those of the first converter thermocouple, e.g. with the average of threeecouples (such a comparison with the average of three equivalent thermocouples takes into concideration, the error of determination of El. 44), and namely immediately before injection, i.e. at a relatively high temperature, e.g. at 17 mv. No heat of the reaction is here as yet present, and the difference in reading of the preheater outlet couple (El 44) against the reading of the first converter couple indicates the error in measurement to be considered when evaluating the readings of El 44.

In order to permit us to judge and evaluate this important information of the preheater outlet temperature in the construction of new preheaters we ask you to fill out the adjoining table and to return it to us.

A special column is provided in the table to furnish us at this opportunity the highest converter temperature, the knowledge of which is essential in the selection of the material for the riser pipe line.

signatures (illegible)

Temperature Measurement of the Preheater Outlet.

Example:

Stall	Liquid phase stall
No.	2
Pressure, at.	700
number and sizes of converters	3S 18 m. long 1S 15 m. long all 1000 mm diameter
Total volume of converters	39 m³
Nature and method of installation of El. 44	welded, insolated with mats

Before injection:

Preheater load	m³/h	20,000
	t/h	20 pasting oil
Average of thermocouples 1, 2 and 3 of the converter I		348°C
Temper., El 44		335°C
Correct., El 44		+13°C

After injection, permanent.

Preheater load	m³/h	35,000
	t/h	35
Temper. El. 44		411°C
Correct.temp. el.44		424°C

Maximum converter temp. 475°C in converter III

Outlet temperature from last convert. or hot catchpot. 459°C

II.

High pressure Experiments
Leuna 558

April 17, 1942. Scha/Le

Table at the end gives a summary of replies to our questionaire.

Brabag wishes to have their replies considered as confidential, and their reply has not been made accessible to all the works, regardless of the interest their results offer for general consideration.

1). Corrections varied between -16°(Leuna) and -18°(Stettin).

2). The introduction of temperature corrections produces uniform intake temperatures for the individual stalls of the works,

e.g., Stettin Tar Stall,	uncorrected	430 - 448
	corrected	448 - 451

and moreover, the temperatures of stalls of the same kind at the different works become alike, e.g.

Tar stall, Stettin, avr. temp.	450°
Tar stall, Brabag, avr. temp.	454°

Gelsenberg measurements form an exception, in that the true intake temperatures vary very much after a correction of 10°, namely, 409 - 432°. The high intake temperature represents a very large load of the preheater, as may be seen from the actual thruputs:

3 converter stall, intake temperature	432, thruput	21 t.
3 converter stall, intake temperature	416, thruput	25 t.
4 " " " "	423 "	28 "
4 " " " "	409 "	32 "

An explanation of the differences may possibly be found in the return flow, with a large return flow in the 4 converter stalls.

3). There is a return flow in converter I, which affects the preheater outlet temperature (El.44), in lowering this temperature when the return flow is increased, i.e. the load on the preheater is reduced. Accordingly, the larger amount of return flow in Scholven is thus indicated, and this might be connected with the catalyst (chlorine).

The height of the intake temperature (El. 44) is indicative of the maximum permissible amount of cold paste (or tar) injection between converters I and II.

4) Analogous values for the vapor phase have no similar significance, and are important only for the preheater proper. Moreover, the intake temperature of the converters mostly rises with the age of the catalyst.

Summary.

The results of the evaluation are interesting, but are of value only for general considerations.

It is recommended to treat each works separately when replying.

The variations in the behavire of the Leuna liquid phase stall will be discussed in Leuna at the first opportunity.

W. M. Sternberg

From Dr. M. Pier's Private Files.

High Pressure Experiments Ludwigshafen a Rh.
P/Leuna 558 Feb. 20, 1942, Scha/Eb

Summarized experience of Leuna, Scholven, Gelsenberg, Pölitz, Welheim, Brabag, Rheinbraun, Lützkendorf, Oberschlesien, Brüx

on

THE HOT TIGHTENING UP OF STALLS.

We are transmitting to you the results of a questionaire suggested by Gelsenberg regarding warm tightening up of stalls.

As you may see from the table, the warm tightening up of the stalls is done very differently at the different works.

The works at Leuna, Welheim and Brabag - and in Böhlen except for the liquid phase omit warm tightening up.

On the other hand, in the newer works of Scholven, Gelsenberg and Pölitz the liquid as well as the vapor stalls are tightened up after warming them up. It is left at the discretion of these works to try gradually getting away from hot tightening up.

2 signatures (Illegible)

W.M. Sternberg.

REMARKS:

* Characteristic are not only the temperature, but also the order of the connections with reference to adjusting the flange distance.

** Hot tightening up after stall repairs only in the newly added parts.

*** Only after major repairs.

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WARM TIGHTENING UP OF THE STALLS.

(Remarks on following page)

Hydrogenation Works		Leuna			Brabag Böhlen	Brabag Magdeburg		Brabag Zeitz	Welheim		Scholven		Pölitz		Lützkendorf		Gelsenber
Pressure	At	Liq.Ph. 230	V.Ph 230	Liq.Ph 300	v.Ph	Liq.Ph 300	V.Ph. 300	TTH Ph. 300	Liq.Ph. 620	V.Ph. 620	Liq.Ph 300	V.Ph. 300	Liq.Ph. 700	Vap.Ph. 325	Liq.Ph. 700	V.Ph. 700	Liq.Ph 700	V. 3
Maximum Operat. Temp. at Outlet of Preheater	°C	490	400	458		465		394	475	560	425	410-500	400	410-500	423	392	ab.440	410
in the Converter	°C	490	500	478		485		434	485	500	475	445-510	ab.460	445-510	421-426	410-415	ab.460	44
i.d. Vertical Pipe Line of Converter	mm	160	120	90		90		120	90,120	90	160	120	120	120	120	120	120	
of heat Exchangers	"	120	120	90		90		90		70,90	120	120	90	90	90	90	90	
i.d. of Preheater Hairpins	"	120	120	120		120		90	70,90	70	90- 120	90	90	90	90	990	90	
Are bellows gaskets installed?		no	yes	yes		no		yes all gask. 90,120 i.d.	yes	yes	no only HOLD pipe line	no	yes	yes	yes	yes	yes	
Starting at Which Temperature?	°C		ab. 330	a few°					200	200			200	200	hot pipe lines 50-120 mm i.d.			
Is Warm Tightening up Done?		no	no	yes	no	rarely at 5 °C		no	no	no	yes	yes	yes	yes	no	no	yes	
At What Occasion?											after each repair		only when starting hot after rep.				after major pair	
At What Pressure?	at			5		5					5	5	0	0				
At What Convert. Temper.?	°C			250		250-300					300	300	380	380				
In How Much time is the Temp. Reached?	h			20 after start of stall		10 after start of stall.					8 after press. release to 5 atm.	8	30	25				
What Parts are Hot Tightened?				all** connect. of hot pipe lines		All newly made connections					All loosened threads in stall & Preh.		All the threads in the stall					
How Long After Pressure is Reached do the Employees Enter the Stall?	h			50 Atm; beginning of tight. 5 atm.		50 atm; begin tighten. at 5 atm.					at once		0	0				
How Much Time is Required for Hot Tightening Up?	h			8		6-8					8-10	6-8	4	3				
Loss of Production Caused by Warm Tightening up.	h			18		20-24					24	24	36	32				

U.S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

KCBraun
2/14/47

CHECK VALVES WITH DROP-SHAPED DISK.

Ludwigshafen, 19 March 1943.

The desire to improve the drop check valves by an interchangeable seat has been repeatedly expressed by hydrogenation plants.

Various changes, dictated by experience, have already been made on these drop-shape check valves, the most important of which are:

1.) Lightening the drop by hollowing it out.

2.) Reducing the lift to reduce the impact.

3.) Increasing the clearance in the guide to minimize the danger of it getting stuck.

4.) Drilling a safety hole, which would indicate excessive wear of the housing by a leak.

At the suggestion of one of the plants we have tried out other possibilities for improvement, with the following results:

To 1.) - A further lightening of the drop is possible by milling out the halves with a suitably shaped milling cutter, as well as by welding them together and thus eliminating the threaded portion (NB 306-2). Still further lightening of the drop would be possible by having it open on the bottom (NB 236-4), although this would somewhat increase the resistance to flow.

To 2.) - A further reduction in lift is not possible in most sizes. However, the seating surface should be widened as much as possible to reduce the impact effect by a cushion of oil or paste.

To 3). - Check valves of N8 or N8A have stuck, due to ferrous-sulfide deposits. It is possible to eliminate this danger by an RM1 bushing in the housing. The bushing could be rolled from a plate and welded, and would require little material. The guiding surfaces on the wings could likewise be made of welded-on RM1 or RM2. The drops themselves are no longer made entirely of RM-steel but have RM-steel welded on at essential points.

To 4). - The safety hole was adopted years ago at the suggestion of one of the plants, because the drop of a check valve had pounded itself thru to the neck of the housing, unobserved, which caused a fire in the stall. On the contrary, some of the plants request the elimination of this safety hole because they consider it dangerous. We are of the opinion that check valves must be serviced like any other part of a machine, i.e. every plant must interchange them at regular intervals as their own experience would indicate.

5.) - The seat plate is no longer made of solid RM-steel, but only the wearing surface is of welded-on RM-metal.

EXPERIENCES WITH BELLOWS LENSES

Ludwigshafen, 14 May, 1942.

The attached table summarizes the evaluation of the bellows lense by the various plants, in reply to a circular inquiry of 2 February, 1942. Accordingly, the bellows lense is equal to the most severe demands in hydrogenation works.

Chief Engineer Zimmermann of Pölitz referred to a quenching test made in 1936 on both normal and bellows lenses. A 120 mm line was quenched with hydrogen at 15°C. While all normal lens connections leaked, due to the rapid cooling, the bellows lenses remained tight.

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BELLOWS LENSES

Plant	Leuna		Böhlen		Zeitz	Welheim		Scholven		Gelsenberg		Pölitz		Lützkendorf		Wesseling	
	L.Ph.	V.Ph.	L.Ph.	V.Ph.	TTH-Ph.	L.Ph.	V.Ph.	L.Ph.	V.Ph.	L.Ph.	V.Ph.	L.Ph.	V.Ph.	L.Ph.	V.Ph.	L.Ph.	V.Ph.
Pressure-atm.	230	230	300	300	300	700	700	300	300	700	325	700	325	700	700	700	325
Since when used?	Mid-1936		No	1937	1939	1938	1939	1937	Propos.	1938		1940		1940		1941	
Reason	Avoid leaks			Trial	Recom. by Leuna	Greater elasticity		Avoid	leaks	Specified by I.G.		Specified by I.G.		Greater Elasticity		Specified by I.G.	
Temperature °C	410			380 400	430	Max. 480	Max. 480	300 480		420 480	400 520	420 460	390 420	Max. 465	Max. 450	200	
Temp. Fluctuations	No			No	100		20 30	20 30 350		50	500	30 460	30 420	50 in 4h	50 in 4h	40	
Which lines have bellows lenses?	In preh. line	All hot lines		Risers Betw. Conv. & to H.E.I	All lines fr. H.E.II Inlet to H.E.II Outlet	Lines fr. H.E, conv, preheat, & Letdown.		Riser betw. conv. & Energ. Exp.		In all hot lines from 45 mm up.		In all hot lines from 45 mm. up		In all hot lines from 58 mm up		All N8 & N10 lines of stall from-mm 45 — 90	
Are they used only in Straight lines or also in fittings?	In lines & fittings			Only in risers	In lines & fittings	In lines & fittings		In Lines & fittgs.		In lines & fittings.		In lines & fittings.		In lines & fittings.		In lines & fittings	
Line sizes? (mm)	120	70 120		120	90-120	45-120	45-90	45-160		45-120	90 120	45-120	90 120	58 - 150		45-120	90 120
Material of lines and fittings.	N6 N8	N6, N7, N8		N8	N8	N8 & N10		N8		N8 N10	N8	N8, N10	N8	N8 & 10 N8V & N9		N8, N8V N9, N10	N8 N10
Bell. Lense Matl.	N5	N5		N5, N5	N5	N8		N5		N8	N5, N8	N8, N5A	N8, N5A	N8 N5A		N8, N5A	N8, N5A
Are flanges especially stressed?	Bending			Bending	Bending	Bending		Bending		Bending		Bending		Tension & Bending		Bending	
Any special rules for tightening flgs?	Tighten cold			No	No	No		No		No		No		Tighten cold & check		No	
What trouble with B.L's, if any?	No			No	No	No		No		No		No		No		No	

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

KCBraun
2/17/47

WASHING GASOLINE, HY-GAS & L.F.G. (LIQUIFIED FUEL GASES), AND FLUSHING IN THE LIQUID PHASE.

Discussion in Scholven, 28 Jan. 1941.
By Dr. W. Krönig, Schlesien-Benzin.

Washing Gasoline

As long as Scholven was running auto gasoline, gasoline was washed twice with lye, which caused an accumulation of 200 g phenols/l in the spent lye. After conversion to aviation gasoline, the gasoline was washed with lye only once, because it seemed more profitable to use up the spent lye completely elsewhere.

Dr. Urban proposes to use the lye used in the gasoline wash subsequently in the hy-gas wash. He would then inject CO_2 into the completely spent lye and dephenolize it.

Washing Hy-Gas and L.F.G. (Treibgas).

Even if the hy-gas is washed with caustic soda, washing the L.F.G. with caustic soda can not be dispensed with, because we must always figure on the possibility of the H_2S not being completely removed in the hy-gas wash. The caustic soda used in the L.F.G. wash is used up completely in the hy-gas wash.

Flushing in the Liquid Phase.

With an injection of 22-23 t/h of coal paste per stall, 500-1500 kg/h of pasting oil is added. Flushing is done with let-down heavy oil at 1000 kg/h, about one half to the stuffing boxes of the 2 paste presses and the other half for flushing the manometer, let-down and catalyst lines of the converters.

U.S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

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KCBraun
2/17/47

REMARKS ON THE VAPOR PHASE STALL IN THE HYDRO. - DEMON. PLANT AT LOUISIANA, MO.

Based on Drg. 1) - 1081
By Dr. Frese, 2/17/47

According to the above drawing, the vapor phase converter has a catalyst space 25-3/8" dia. x 34'-9½" long. It has 6 mixing baffles, each 15" long. About 20% of the free catalyst space in the converter is taken up by the baffles.

The proposed injection is about 3 t/h of A + B product, which corresponds approximately to a load of 1 t/m^3 of catalyst volume/h. If we figure 2500 m^3 of inlet + cold gas per ton of injection, at which a satisfactory operating efficiency could be expected, we arrive at a total gas volume of 7500 m^3/h. With this quantity we get a velocity of 2.9 cm/sec. of the gases flowing thru the free catalyst space, which will be increased to 6-7 cm/sec. with the catalyst in place. The retention time of the gases between 2 cold gas inlets is therefore very long. This velocity is very low and about 1/2.5 below the limit at which the vapor phase converter at Ruhröl could still be operated with adequate safety. The reason why the subject of gas velocity in vapor phase stalls has been so neglected in the available literature on hydrogenation is that the velocities of abt. 35-40 cm in the filled catalyst space of the present 300 atm. stalls was far above the critical limit. In general, the reaction gases had a very long way to go thru the 3 or 4 converters arranged in series, where each converter usually had 6 cold gas inlets.

In 1941 and 1942 Ruhröl, in consideration of the influence on aromatization of the proportion of gas supply to injection, made a number of large scale experiments. These showed that if the gas velocity in the catalyst space (catalyst in place) was reduced below 12-15 cm ~~in~~ the converter could no longer be controlled. The velocity indicates the time required by the gases to travel from one cold gas inlet to the next. The reaction between the two cold gas inlets can not be influenced from the outside. This retention time was about 15 seconds in Welheim with 3 converters in series and a gas velocity of 15 cm, while in Louisiana it is 36 seconds with a velocity of 6 cm/sec. With 300 atm. stalls the retention time between 2 cold gas rings, of course, is much less than in Welheim at 700 atm. This is necessary because of the lower density of the gas due to the lower pressure.

At Louisiana the following second disadvantage must also be considered. In Germany each converter had 2 thermo-wells, each containing 6 thermometers. Consequently, each converter had 12 temperature measuring points. Thus, the operator could recognize a starting reaction in one half the retention time between 2 cold gas inlets and act caccordingly. This is not provided for in Louisiana, with 6 measuring points. For a period of 36 seconds, it is not possible to control the product in its passage thru the converter and recognize a starting reaction. When, however, a reaction has once started, it takes only a few moments to raise the temperature to 800-1000° C.

Based on these considerations, the following may be said about the temperature. With an injection of 3000 kg/h and a gas supply of 7500 m^3/h, 3000 x 130 = 390000 kcal/h are liberated. If we assume that 90000 kcal are lost in radiation by the converter, there remain 300000 kcal/h, which must be carried off by the cold gas. In 36 seconds, or the retention time between 2 cold gas rings, 300000 x 36/3600 = 3000 kcal must be carried off. At first, the lower 3 or 4 cold gas rings will have to take this load, decreasingly in the order of their arrangement. The gas intake in 36 seconds = 7500 x 36/3600 = 75 m^3 and the product injection = 3000 x 36/3600 = 30 kg (mean mol. wt = 150). This is equal to about 80 m^3 with a specific heat of about 0.4/m^3. To raise these 80 m^3 by 1° requires 80 x 0.4 = 32 kcal. With 3000 kcal to be carried off these gases will rise by 3000/32 = 94° C. Assuming that this rise in temperature occurs between the first 3 cold gas rings, the temperature between 2 adjacent cold gas rings will rise by 94/3 = 31° C in normal operation. Very probably, however, the first cold gas rings will have to take away more heat than that, so that a temperature difference of 40 - 50° C may exist between them. In practical operations that means that, with such low velocities, temperature differences of 50° C between 2 cold gas rings must be figured on. This is a dangerous operating method, if, with a correct temperature of 500° C, the temperature is permitted to fluctuate between 475 and 525° C. On the other hand if we operate with a temperature range of 450-500° C, the capacity is greatly reduced.

The Louisiana disadvantage is due to the fact that, while the catalyst charge for a comparatively large diameter converter was correctly chosen, the velocity, i.e. the retention time between 2 cold gas inlets, was not given sufficient weight. In the present plants, this is characterized by a 3 or 4 converter system arranged in series. In Louisiana, the converters are practically arranged in parallel, but wherein each converter receives only 1/4 of the available cold gas supply. Both, velocity and charge, are important factors in hydrogenation, which must absolutely be considered together. Apart from the dangerous operating method, such a plant is not well suited to operations in which results of experiments must be determined. In such a converter with 6 thermo-elements the temperature may be measured so that either the coldest points, i.e. just beyond the cold gas inlets, or the hottest points, just this side of the cold gas inlets are measured. Between these 2 points the above mentioned great temperature difference may exist, and the indicated course of the temperature may give an entirely wrong picture, even though no very great temperature differences are indicated if all the elements are always located at the same point.

To avoid these difficulties, the following suggestions apply to Louisiana:

It would be best, if 2 converters were arranged in series, each the same length as the present converter, with a converter volume of about 1½ m^3. A larger diametor thermo-well, which could hold 12 thermo-elements, one additional element between the 6 cold gas inlets, would be desirable. If it should be impossible to provide 2 such converters, we should try to reduce the volume of the present converter to about 2-2.5 m^3 by inserts, even though the capacity would thereby be somewhat reduced. The thermo-well should also be enlarged to accommodate 12 elements. It is not recommended to reduce the volume by an increase in the thickness of the insulation, because this would mean additional cold gas consumption. From a pure operating standpoint, we could operate with more inlet gas with the same product injection. If need be, the proportion of product to gas could be changed to 1:4000 or 1:5000. To be sure, this would affect the yield and the octane number of the gasoline, but it would, nevertheless, greatly improve the temperature condition and provide greater safety. This would also put a greater load on the gas circulating pumps.

The following considerations may help to decide whether baffles for better mixing of the cold gas with the main gas stream should be installed. These baffles were first introduced in the middle '30's after experiments at Leuna. They provided a thorough mixture of the cold gas with the main gas stream and were intended to prevent the formation of reaction nests. The converter volume, which really determines the converter capacity, is reduced by the baffles. In a converter with baffles the temperature is such that after the mixing, the lowest temperature is always in the baffle and the highest temperature just this side of the baffle. There is a steady rise and fall in the temperature of this system, which, as a rule, is so much greater the greater the retention time of the gases in the catalyst space between the baffles. At the same time, there is a certain factor of safety in these baffles; they enable even a poor operator to run the converter. A converter without baffles has a more irregular course of temperature, a less distinct temperature chart. A good operator will produce a good, a poor operator a less favorable, temperature chart, on which the yield is based. A converter without baffles is, therefore, more difficult to operate and more dangerous. The experiments with and without baffles conducted at that time under equal reaction conditions provided an adequate picture of the effect of the baffles. Some spoke in favor of baffles, others against them. All in all, based on free converter volume, it is safer to operate with baffles, while a greater yield may be obtained without them. In all later German plants the converters were equipped with baffles for reasons of safety. Fundamentally, the following should be given consideration:

Converters with baffles should be used for catalysts with high heats of reaction or for low velocities of flow, which favor the formation of reaction nests or pockets, while for catalysts with low heats of reaction or for very high velocities of flow a greater yield may be obtained without baffles, because of the enlarged catalyst space.

For conditions at Louisiana, in which new catalysts and new products are to be experimented with, baffles are satisfactory, for the time being. But further thought should be given to simplification of construction (smaller volume) and greater safety. The installation of the baffles in the converter is particularly difficult for Louisiana, because, due to the small diameter, a man cannot work inside of the converter. The greatest care must be taken to provide a perfect seal between the baffles and the thermo-well and the cold gas inlet pipe. If short circuits should occur at these points, the converter will run away without a doubt. If this problem can not be solved satisfactorily, a converter without baffles but with perforated cold gas rings is the safest way.

The converter temperature is properly always measured at the hottest points, i.e. just this side of the baffles. As already mentioned, a second temperature measurement should always be taken at about half ways between baffles, at least in the lower half of the converter. This point would give a good average temperature.

U.S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

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KCBRAUN
2/19/47

ESTIMATE OF COST OF PLANT FOR PRODUCING 500,000 BARRELS GASOLINE PER DAY (60,000 TONS PER DAY OR 22,000,000 TONS PER ANNUM).

Dr. Erich Frese - 2/19/47

This estimate is based on very large, ideal future plants in which the heat and power are largely recovered as outlined in the report "The Thermal Efficiency of Coal Hydrogenation--Present and Future" prepared by Skinner, Dressler, Chaffee, Miller, and L.L. Hirst.

The liquid and vapor phases are interconnected without pressure expansion.

The residue processing plant is rather small, because only the solids from the last liquid phase converter are processed here.

The total production is distributed among ten (10) plants - capacity of each 50,000 barrels per day. Each plant has twenty (20) interconnected liquid and vapor phase stalls of 60 + 35 m^3 converter volume each. The gas circulation to these twenty (20) stalls is properly subdivided into three systems each connected to about one-third of the stalls.

The total production is summarized as follows: -

Number of plants	:	Ten (10)
Total cost of plants	:	$ 3,187,000,000
Iron requirement	:	14,000,000 tons - 75% equipment - 25% buildings
Personnel requirement	:	30,000 men (three shifts) (700 tons gasoline per man per annum)
Repair costs	:	$128,000,000 per annum (4% of invested capital)
Iron required for repairs:		150,000 tons per annum

The plant costs are allocated as follows to the individual plants.

1.	H_2 production	$ 910,000,000
2.	Hydrogenation	1,221,000,000
3.	Hy-Gas Tanks	10,000,000
4.	Power Plant	400,000,000
5.	Power Distribution	148,000,000
6.	Auxiliary Buildings	315,000,000
7.	Miscellaneous	183,000,000
	Total -	$ 3,187,000,000

Detailed costs for one plant of 50,000 barrels of gasoline per day (6100 tons per day or 260 tons per hour).

PRODUCTION DATA

H_2 consumption : 510,000 m^3 per hour
(305,000 m^3 from Koppers generators
and 205,000 m^3 from hy-gas splitting)

Power - Electrical : 300,000 KWH per hour
Steam : @ 32 ats. - 117 tons per hour
" : @ 11 " - 60 " " "
" : @ 3-1/2" - 826 " " "

Coal for production : Raw - 520 tons per hour
Water and ash free -(445) " " "

" " H_2 " : Raw 170 " " "

" " Current & Steam : Raw 250 " " "
Total - Raw 940 " " "
Total - Water & ash free 445

For one ton of gasoline: 3.6 tons raw coal
1.7 " water & ash free coal

Water consumption : 88,000 m^3 per hour

I. H_2 Production - 510,000 m^3 per hour

Water gas generators	(60%)	$23,700,000
Hy-gas splitting	(40%)	4,650,000
Oxygen plant		18,200,000
H_2S cleaning		5,550,000
Conversion		9,100,000
Gas tanks		1,370,000
CO_2 cleaning		6,330,000
Compressors to 700 ats.		18,800,000
Pipe lines		3,300,000
Total -		$91,000,000

2. Hydrogenation ($122,100,000)

(a) Liquid Phase		
Coal crushing & drying	$10,800,000	
Paste presses (32 @ 25 tons per hour)	10,500,000	
Gas circulating pumps (together with vapor phase)	4,000,000	
Coal hydrogenation stalls (20 stalls @ 60 m^3 converter volume) 6 converters @ 10 m^3 or 4 " @ 15 m^3	49,000,000	
Gas wash	5,500,000	
Residue processing	3,000,000	
Tanks	200,000	
High and low pressure lines Gas circulating line	6,000,000	
(a) Total -	89,000,000	$89,000,000
(b) Vapor Phase (700 ats.) Connected to Liquid phase		
Vapor phase stalls (20 stalls connected to liquid phase Ea. 35 m^3 converter volume	$16,500,000	
Tanks	2,500,000	
Distillation	3,400,000	
High & low pressure lines	3,600,000	
(b) Total - -	$26,000,000	26,000,000
(c) General		
Filling & distbr. stations	4,100,000	
Insulation	700,000	
Lines	2,300,000	
(c) Total -	$ 7,100,000	7,100,000
	Total	$122,100,000

3. Hy-Gas ($1,000,000)

Gas tanks	$900,000
Miscellaneous	100,000
	$1,000,000

4.	Power plant (300,000 KWH per hr.)	40,000,000	
5.	Power, water & steam distribution	14,800,000	(Steam lines, water lines, electrical lines, including transformers, misc.)

6. Auxiliary Buildings ($30,500,000)

Machine shop, instrument repair shop, electrical shop, warehouse	$ 5,500,000
Laboratories	4,200,000
Wash rooms	1,200,000
Railroad tracks and plant roads	4,500,000
Sewers	2,400,000
Phenol water cleaning	1,000,000
Electrical & Water installation in buildings	700,000
Engineering office	300,000
Railroad cars	4,200,000
Trucks, (gas. & elect.) automobiles, etc.	300,000
Fencing	200,000
Preparation of site	3,700,000
Fire protection	1,500,000
Miscellaneous	1,800,000
Total -	$31,500,000

7. <u>Miscellaneous ($18,300,000)</u>

Catalyst plant	$1,500,000
Design	3,500,000
Construction and supervision	6,000,000
Homes for workers	7,000,000
Miscellaneous	300,000
Total -	$18,300,000

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

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KCBraun
2/20/47

CONSUMPTION DATA FOR 100000 T/ANN AUTO GASOLINE

By v. Hochstetter, Ludwigshafen/Rh,
28 June, 1944.

The figures on power consumption given in the attached table are based on data submitted by the various plants.

The personnel includes production workers only. It does not include workers for auxiliary purposes, such as plant protection, fire protection, apprentices, personnel division, sales division, accounting, kitchen, stores, air protection, transportation, cleaning, etc. About 40% must be added for these purposes. The rest is based on ideal figures, which, however, are closely met at present by the various plants.

The proportion of iron requirements for machines to that for buildings was figured as 2:1 everywhere. Iron required for repairs is based on estimates from plant reports. The Demag process was figured for H_2 from coke; the Winkler process for H_2 from coal and lignite or carbonization coke (Grude).

Table I

Consumption Figures for 100000 T/Ann. Auto Gasoline.
(Calculated from 200000 T/Ann. Plants)

		Vapor Phase			Petroleum (mixed base)		Brown Coal Tar (Middle German)		Brown Coal (Middle German)		Bitum. Coal		TTH 100000 T/Ann. Product 77% Gasol. + Gas oil 8% Lubric. oil 15% Paraffin	D HD 100000 T/ann. DHD-Gasol.
		1. Stage Gas Oil	2 Stages		Total	Vapor Phase Alone	Total	Liquid Phase Alone	Total	Liquid Phase Alone	Total	Liquid Phase Alone		
			Mi-Oil from Brown Coal	Mi-Oil from Bitum. Coal										
		A.	B.	C.	D.	E.	F.	G.	H.	I.	J.	K.	L.	M.
Consumption Figures:														
Raw Matl. (Dry)	t/h.	14.0	13.6	13.5	14.25	14.1	15.5	15.5	28.5	28.5	21.5	21.5	13.75	15.8
Current	KW	2750	6000	7100	9500	4600	11500	3500	21250	15000	35000	27000	10000	2400
H.P. Steam	t/h	1,25	2.5	2.5	8.75	4,25	11,25	6.75	55	44	18.5	13.5	7.5	3-3.5
L.P. Steam	t/h.	7.5	15	17	22	10.5	23.7	7	28	20.5	67.5	50	45	3.5-4.0
Water	$m^3/h.$	800	1700	1900	2375	850	3125	1000	4000	3125	5750	4000	1800	340
Fuelgas	10^6 Kcal	6	5	8.5	16	8.75	13	7	18	15	33.5	25	17	14
Hygas-Credit	10^6 Kcal	8.5	10.5	13.5	21	10.5	29	13.5	55	44	74	50.5	6.5	36
Difference		2.5	5.5	5	5	1.75	16	6.5	37	29	40.5	35.5	-10.5	22
Hydrogen	$m^3/h.$	5000	8000	10000	7200	3200	10400	3500	29750	21750	35750	25500	6250	—
Coke req'd for H_2 (for watergas)	t/h.	4.5	7.1	9	6.5	3	9.5	3.15	26.7	19.5	34	23	5.5	—
Rest-Watergas credit.	10^6 Kcal	(3.1)	(5.0)	(6.3)	(4.5	(2.0)	(6.6)	(2.2)	(18.7)	(13.7)	(22.5)	(16.0)	(4.0)	—
or expressed in t Coke		(0.4)	(0.7)	(0.8)	(0.6)	(0.3)	(0,9)	(0.3)	(2.5)	(1.8)	(3.0)	(2.2)	(0.5)	—
or Coal (f.Winkler)	t/h.	4.0	6.0	8	5.75	2.5	$7.75^{3)}$	$2.65^{3)}$	$22.5^{3)}$	$17^{3)}$	29	20.5	5	—
or Hygas (Incl. fuel gas for splitting plant)	10^6Kcal/h	17	27	34	24.5	11	35.5	12	100	75	123	87.5	21.5	—

Table I - Continued

Personnel Req'd:		A.	B.	C.	D.	E.	F.	G.	H.	I.	J.	K.	L.	M.
Operators	Men	130	200	240	280	125	340	155	500	360	628	445	200	65
Millwrights	Men	95	150	180	210	95	255	115	370 7	270	470	350	135	60
Iron Req'd,	t	12500	20000	20000	30000	16000	37500	16000	72500	55000	80000	60000	?	?
(for equipment)	t	8500	13500	13500	20000	10500	25000	10500	50000	37500	55000	40000	?	?
(for bldgs.)	t	4000	6500	6500	10000	5500	12500	5500	22500	17500	25000	20000	?	?
(for repairs)	t/Ann.	150	270	270	500	280	550	280	1050	800	1200	900	900	350
Raw Matl. Req'd/Ann.														
(for Hydrogenation)		110000	109000	108000	114000	114000	125000	125000	227500	225000	172000	172000	110000	126000
(for H_2	t Coke	35000	57000	72000	52000	24000	76000	25000	215000	156000	272500	185000	44000	—
or	t Coal	32000	48000	64000	46000	20000	62000$^{3)}$	21000$^{3)}$	(180000$^{3)}$	132000$^{3)}$	227500	165000	40000	—
t Coal, current & steam together		39000$^{1)}$	34000$^{1)}$	40000$^{1)}$	57000$^{1)}$	27500$^{1)}$	83000$^{2)}$	29500$^{2)}$	182500$^{2)}$	135000$^{2)}$	192000	147500	92500	—

1) 6300 Kcal/kg 2) 5000 Kcal/kg 3) Crude (L.T. Coke)

C.H. - Economics

From Dr. Pier's files

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U.S. BUREAU OF MINES T-250
HYDRO. DEMON. PLANT DIV.

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2/20/47

COST OF AVIATION GASOLINE FROM BITUMINOUS COAL

Ludwigshafen/Rh, 6 September 1941

Prices.

The prices for hydrogenation coal and catalyst are taken from the Pölitz basic cost calculations of July, 1940, those for coke, power coal and coal for fuel gas production from their basic cost calculations of June, 1941. Fuel gas and H_2 prices were calculated from them.

Liquid Fuel Gas and H_2.

According to a statement from Pölitz, the liquid fuel gas is recovered only from the rich gases. Hygas calculations for Gelsenberg and Upper Silesia showed that of the total $C_1 + C_4$ an average of 88.5% is contained in the rich gas. It was assumed that 90% of the $C_1 + C_4$ could be recovered from the 95% utilizable rich gases. 58% of the H_2 required can be recovered from the residual hygas. The fuel gas required for hygas splitting is produced in generators. Generator gas is also used for fuel gas for hydrogenation insofar as the fuel gas supply for this purpose is not covered by the residual water gas from the H_2 production from coke.

Power.

The outside power supply is 200 x 10^6 Kw/ann. (Pölitz calculations of July, 1940).

Since no special power calculation was made, the steam prices were derived from a comparison of ordinary with basic cost calculations. Prices for current and water are taken from the Pölitz calculations.

Repairs.

To determine the number of men required for repairs, the average percent rate of repairs on the invested capital was first determined from repairs in individual plant divisions, in Hydrogenation, H_2 production and the power plant. The sum derived in this manner gave us the number of millwrights, based on RM 3.40 per working hour.

Balance.

The balance for Upper Silesia of the 16 November 1940 was taken as a basis. However, as a result of large scale experiments in Ludwigshafen, the conversion was increased from 94 to 95%, and the gasification decreased from 27 to 24%. The yield per unit of converter volume (Leistung) is unchanged at 0.26.

Power Consumption

Newly determined were:

Plant Costs:

a.)-Hydrogenation: Existing plant estimates were reestimated for the Pölitz capacity.

b.)-Hydrogen: Reestimated from the Saar plant estimate (50% from hygas, 50% from coke).

c.)-Power Plant: Average of estimate for the Saar plant and Upper Silesia, taking into consideration the outside power supply.

Power Coal Quantity.

A heating value of 6300 Kcal is figured, based on the price of power coal.

Fuel Gas Coal.

The fuel gas yield is figured as 5×10^6 kcal/ton of coal.

The balance of the 16 November 1940 for aviation gasoline from Upper Silesian coal, Beuthen-Heinitz, was used as a base at 95% yield, hygases of a heating value of 230×10^6 kcal/h are produced. In Pölitz, the L.F.G. is recovered only from the rich gases. According to hygas calculations for Gelsenberg, 89% of the total propane and butane of the hygases is contained in the rich gases; 88% according to calculations for Upper Silesia. We, therefore, figure a mean of 88.5% for Pölitz. With column decomposition the yield in $C_3 + C_4$ is assumed to be 90%. Accordingly, 8.9 t/h of propane and butane are produced.

The lower heat value (Hu) of 1 kg L.F.G. = 11000 kcal, or $8.9 \times 11 \times 10^6 = 98 \times 10^6$ kcal/h. Since the fuel gas required by hydrogenation and hygas splitting is supplied by the residual water gas from H_2 production from coke and by generator fuel gas, the amount available for H_2 production from hygas is 230-98 = 132 x 10^6 kcal/h. From this may be recovered (132 x 10^6)/2400 = 55000 m^3 H_2/h = 57.8% of total H_2. Total H_2 requirement of 95200-55000 = 40200 m^3 H_2 must, accordingly, be produced from coke, corresponding to 33.4 tons of coke.

Fuel gas req'd by hydrogenation = 86 x 10^6 kcal/h
Fuel gas req'd by hygas splitting = 55 x 10^6 kcal/h

Total 141 x 10^6 kcal/h

Residual water gas produced = 25 x 10^6 kcal/h
Fuel gas to be prod. in generator = 116 x 10^6 kcal/h

One generator produces 6 x 10^6 kcal/h, or 20 - 3 = 23 required.

One ton raw coal supplies 5 x 10^6 kcal, or 116/5 = 23.2 tons/h fuel gas coal required.

Cost of fuel gas plant = RM 9,500,000.

ESTIMATE OF COST OF PLANT

Producing 244000 t/ann. Aviation Gasoline from Upper Silesian Bituminous Coal.

Liquid Phase 700 Atm.

Coal crushing, pulverizing, drying, pasting, 66.5 5/h	RM. 6,700,000.-
Gas Circulating Pumping Unit, 230000 m^3/h	1,500,000.-
Paste Press Unit, 143 t/h	4,700,000.-
Vapor Phase Stalls, 4 x 4	21,200,000.-
Circulating Gas Wash Unit, 173 m^3/h	4,000,000.-
Residue Centrifuging Unit, 85 t/h	2,300,000.-
Residue Carbonization Unit (L.T.C.), 18.5 t/h	3,300,000.-
Heavy Oil Tanks	2,000,000.-
Catch-Pot Product Distillation, 89 t/h	2,300,000.-
High & Low Pressure Lines	2,500,000.-
Compressor Unit, 68600 m^3/h	2,000,000.-
Total	RM. 52,500,000.-

Common Installations:
(only liquid phase portion)

Sales storage tanks, filling station.	
L.F.G. & debenzination units, storage, filling station, 4.5 t/h	4,300,000.-
Pressure test pit, insulation scaffolding, emergency expansion, foamite fire extinguishing installation.	

Summary:

Liquid Phase	RM.	52,500,000.-
Common Install. (Liq. ph.)		4,300,000.-
	RM	56,800,000.-

Aviation Gasoline and Coal.

Details of Vapor Phase Installation Costs.	(Million RM.)
Injection pumps & gas circulation 1.5 + 1.7	3.2
4 gasol. stalls, 3 (000) + 1 (00), 4.8 + 4.9	9.7
Light Oil Storage tanks	1.8
Distillation	1.9
Stabilization & wash	0.75
High & low press. lines	1.4
	18.75

Common Installations:

Sales storage tanks & filling station	0.6
Debenzination & L.F.G. units, 4.4 t/h	2.5
Pressure test equipment, insul. scaffolding, foamite installation, pipe lines	0.15
	3.25
	18.75
	22.00

Plant Costs for Aviation Gasoline from Upper Silesian Bituminous Coal

Hydrogenation:	Liquid Phase	RM 56,800,000.-
	Vapor Phase	22,000,000.-
H_2 Production, 95200 m^3/h		39,000,000.-
Power Production, (Steam, elec, water)		44,000,000.-
Fuel Gas Production		9,000,000.-
		170,800,000.-
Auxiliary Units, 25%		42,700,000.-
		213,500,000.-
Miscell. Capital Req'd, 15%		32,000,000.-
	Total	RM 245,500,000.-

Power Requirement for Aviat. Gasol. from Upp. Sil. Bit. Coal

	H.P.	L.P.	Water	Current	Men/Day
Fuel Gas			500	800	
Hydrogenation	24	82	6250	31500	909
H_2 Prod. fr. coke	(27)	56	3600	21500	
" " " Hygas		46	5000	33000	
Water works		1		4500	550
Auxil. Units		16	1400	4100	
	(3)	201	16750	95500	1790

Calculation of Power Coal Quantity:

The lower heat value (Hu)/kg of coal = 6300 kcal
(Assumed at freight prepaid price of RM. 12.50/t)
Eff. = 0.8

Heat value of H.P. steam = 800 kcal/kg.
" " " feed water = 200 "
Heat to be supplied = 600 kcal/kg.

201 t. L.P. - steam = 292 t H.P. steam = 41000 KW (7.1 kg/KW)
95500 - 25000 outside current = 70500
-41000
29500 x 4.2 =

124.0 t
292.0 t
416.0 t steam
-3.0 t
413.0 t steam

$$\frac{413000 \times 600}{6300 \times 0.8} = 49 \text{ t power coal.}$$

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POWER TABLE FOR AV. GASOL. FR. UPP. SIL. BIT. COAL.

	Quantity	H.P. Steam	L.P. Steam	Gas	Water	High Tension	Low Tension	Men/ Shift
Liq. Ph.-700 Atm.								
Crushing	66.5	—	0.67	—	—	443	110	5
Drying	67.0	—	0.44	8900	—	570	75	5
Coal Pulver.	137.0	2.4	3.15	—	5	2500	250	5
Paste Presses	143.0	—	5.7	—	—	9350	99	18
Water & Flush. Oil Pumps	9.0	—	—	—	—	360	1	3
Gas Circul.	230000	—	—	—	—	1370	23	6
Compression	68600	—	—	—	206	4800	—	7
Stalls	146.0	1.46	24.8	31400	1650	1830	410	20
Oil Wash	173	—	17.3	—	58	1120	190	9
Centrifuging	85	2.98	1.54	—	2	—	240	12
L.T.C. (Schwel.)	18.5	1.12	0.86	8700	240	740	278	36
C.P. Distill.	89.0	—	5.75	19600	1220	—	276	12
Vap. Ph. 300 Atm.								
5058 Inj. Pumps	36.7	—	—	—	—	168	8	4
Wat. Pumps	3.7	0.57	—	—	4	—	77	2
Gas Circ.	147000	—	0.53	—	—	735	12	4
Stalls	36.7	—	0.70	—	400	565	42	10
C.P. Dist.	35.0	—	1.77	6950	500	—	140	5
6434 Inj. Pumps	35.8	—	—	—	—	455	6	4
Wat. Pumps	3.6	0.6	—	—	1	—	72	2
Gas Circ.	72000	—	0.25	—	—	360	5	3
Stalls	35.8	—	0.63	3580	386	550	41	10
C.P. Dist.	31.8	—	1.54	6050	452	—	127	5
Debenz. & L.F.G. Recov.	11.4	5.7	1.9	—	620	2120	—	4
Gasol. Stabl.	30.6	4.95	0.87	—	263	—	61	2
Gasol. Wash	30.6	—	0.59	—	9	—	23	2
Int'md. Tanks	30.6	3.4	6.8	—	—	—	430	5
Sales Tanks Hygas Tanks Office & Laborat.	30.6	0.92	6.1	306	218	31	250	3
	—	24.10	81.94	85486	6234	28367	3246	202
						31 613		+ 50% 303

Aviat. Gasol. fr. Upp. Sil. Bit. Coal
Basic Cost Calculation

Production 244,000 t/ann. Av. Gas in 4(0000) = 141 m^3 (18 : 15 m conv. = 3:2)

Input 532,000 " raw coal with 5% ash & 10% water

A. Operating Costs: — RM/t Av. Gas.

Coke: 267,000 t/ann @ 30.-/t = 8,000,000.- 32.80
Power Coal 389,000 " @ 13.80 = 5,370,000.-- 22.00
Fuel Gas Coal 185,000 " @ 18.80 = 3,480,000.-- 14.25
Outs. Current 200 X 10^6 KW/ann @2 Pf = 4,000,000.-- 16.40

Catalyst & Chemicals:

Liq. Phase Cat. I 5850 t @ 16.50 = 96,500.--
" II 1470 t @ 300.- = 440,000.--
" III 7700 t @ 18.10 = 139,400.--
675,900.-- = 2.77

Vap. Phase 5058,46 m^3 @ 12500.- = 575,000.-- = 2.36
6434,42 " @ 4150.- = 174,000.-- = -.71

Splitting Cat. I 20.5 t @ 3000.- = 61,500.--
" II 18.7 t @ 2000.- = 37,400.--
" III 60.5 t @ 800.- = 48,500.--
147,000.-- = -.61

Conversion 93.5 t @ 1650.- 154,000.-- = -.63
Chemicals 1,220 t NaOH @ 150.- = 183,000.-- = -.75 7.83

Wages: 1,790 · 8 · 365 @ 1.- = 5,230,000.-- = 21.40

Salaries: 20% of wages = 1,050,000.-- = 4.28

Repairs: 1075 · 8 · 365 @ 3.40 = 10,700,000.-- = 43.90

Operating: 15% of wages 785,000.-- = 3.21

Welfare: 20% of wages & salaries 1,255,000.-- = 5.14 77.93

Operating Costs RM/t Av. Gas 171.21

B. Raw material:		RM/t Av. Gas.
Brought Forward		171.21
Hydrog. Coal: 532,000 t/ann @ 22.50 = 11,950,000.- =		49.--
Preparation Costs RM/t Av. Gas.		220.21
General: 2.5% of Prepar. Costs, + Amortization & Fire Protect. + Taxes (= RM/t 327.71)		8.20
License:		5.--
		233.41
C. Capital Burden:		
Amortization: 10% of 213.5 Mill.M. = 21,350,000.-- =	87.40	
Interest: 3% of 213.5 Mill.M. = 6,400,000.-- =	26.20	
5% of 32.0 Mill.M. = 1,610,000.-- =	6.60	
Fire Protect. - Taxes: 2% of 213.5 Mill = 4,270,000.-- =	17.50	137.70
		371.11
Credits: 15,000 t/ann. L.T.C. Tar fr. Fuel Gas @ 90.- = 1,350,000.-- =		5.53
71,000 t/ann. L.F.G. @ 150.- 10,650,000.--		43.70
RM/t Av. Gas.		321.88

Av. Gas fr. Upp. Sil. Coal.

Cost Estimate
(Bal. 16. Nov. 40 fig. in)

Basis:

L-Ph. 700 atm; 96% Decomp. 0.25 yield on available oil recovery.

24% Gasification on available oil recov. + gasif.
4 m³ gas/kg raw coal.

V-Phase; 5058; 300 atm;Burd. 0.8; 3.5% gasif.; 33% conc.
4 m³ Gas/kg. inject.

6434; 300 atm; Y 0, 45; 20% gasif.; 60% concent.
2 m³ Gas/kg inject.

Raw Coal Consumption: 532,000 t/ann. dry coal with 5% ash = 455,000 t/ann. raw coal

Av. Gas-Production: 244,000 t/ann at 4 (0000) L-Phase-Capacity.

		RM/t/Av.Gas.	
Raw Material:			
2.18 t Raw Coal	@ 22.50	49.-	
3113 m³ H_2	@ 5.15	160.-	
4310 m³ Hygas - Credit	@ 0.56	(24.10)	
290 kg L.F.G.	@ 15 Pf.	(43.50	142.40
Cat. & Chemicals:			
24.0 kg $FeSO_4 \cdot 7 H_2O$	@ 1.65 Pf.	-.40	
31.6 " Bayermass	@ 1.8 Pf.	-.57	
6.0 " Sulfigran	@ 30.0 Pf.	1.80	
0.189 Lit. 5058	@ RM. 12.50	2.36	
0.172 " 6434	@ RM. 4.15	-.71	
5 kg NaOH	@ 15.0 Pf.	-.75	6.59
Power:			
0.79 t H.P. Steam	@ 2.50	1.98	
3.2 t L.P. Steam	@ 2.20	7.05	
2790 m³ Fuel Gas	@ 56 Pf. (1 m³ = 1000 Kcal)	15.50	
250 m³ Water	@ 1 Pf.	2.50	
1165 KW Current	@ 2 Pf.	23.30	50.43
Wages:			
a) 303 Men/Shift = 11.0 Work. Hr. @ 1.-		11.00	
b) Salaries + Addit. 30%		3.30	
c) Oper. Matls. + Lab. Costs 20%		2.20	
d) Social Burden 20% of a) to c)		3.20	19.80
			219.42

	Brought Forw.	219.42
Repairs etc.		
6.5% of 98.5 Mill	26.20	
2% " " " Fire Protect. + Taxes	8.08	
10% " " " Amortization	40.35	74.63
	Product.Costs	294.05
General: 2.5% of Product. Costs	7.35	
Interest: 3% of 98.5 Mill.	12.30	
5% " 14.8 "	3.03	
License:	5.-	27.48
	RM/t/Av. Gas	321.53

Fuel Gas in Pölitz.

	RM/10^6 Kcal
0.2 t Coal @ 18.80	3.76
4 m^3 water @ @ 1 Pf.	-.04
7 KW Current @ 2 Pf.	-.14
a) 0.3 Work Hr.@ 1.-	-.33
b) 50% Addit.	-.17
20% of a) + b)	-.10
Repairs 5% of 9.0 Mill.	-.46
Fire Prot. & Tax 2% of 11.2 Mill.	-.24
Amortization of " "	1.20
	6.46
General 2.5% of Prod. Costs	-.16
Interest 3% of 11.2 Mill.	-.36
5% " 1.7 "	-.09
	7.07
Credits: 16 kg Tar @ 9 Pf.	(1.44)
RM	5.65

H_2 in Pölitz.

			RM/1000 m^3 H_2	
0.35 t Coke @ 30.000	10.50	18.36		
1390 m^3 Hygas @ 0.56	7.80			
0.027 kg @ 3.-			-.08	
0.025 " " 2.-			-.05	-.39
0.08 " " -.80			-.06	
0.122 " " 1.65			-.20	
1.08 t L.P. Steam @ 2.30		2.38		
90 m^3 Water @ 1 Pf.		-.90	17.92	
570 KW Current @ 2 Pf.		11.40		
578 m^3 Fuel Gas @ 0.56		3.04		
a) 1.28 Work Hrs. @ 1.-		1.28		
b) 50 % Addit.		-.64	2.30	
20 % of a)& b)		-.38		
6.5 Rep. of 39.0		3.32		
2 % Fire Prot. & Tax of 48.7		1.28		
10 % Amortization		6.40	11.00	
General: 2.5% of Prod. Costs			49.97	
		1.24		
Interest: 3% of 48.7 Mill.		1.92	3.64	
5% " 7.3 "		-.48		
		53.51		
Credits: 0.28 t H.P. Steam @ 2.50		(0.70)		
0.245 · 10^6 Kcal = 5.68		(1.38)		
		51.53		

Av. Gas. fr. Bit. Coal
(Calculation 2/Sept/41)

	Raw Matl.	Power	Wages	Repairs	Fire Prot. & Tax	Amortization	Interest	General & License	Total RM/t Av. Gas
Raw Coal, (Dry) 2.18 t @ 22.50	49.-							5.78	148.18
H_2 3113 m^3 @ 5.15 Pf.	161.-								
Hygas 4.31 x 10^6 Kcal 0.56 Pf./1000 Kcal	(24.10)								
L.F.G. 290 kg @ 15 Pf.	(43.50)								
Cat. & Chem.	6.59							-.27	6.86
Crush. & Dry		2.48	-.90	2.12	-.65	3.26	1.23	-.40	11.04
Pasting		2.26	-.47	-.12	-.04	-.18	-.06	-.13	3.26
H_2-Compression 300-700 Atm.		3.21	-.65	-.67	-.20	1.02	-.39	-.24	6.38
Gas Circ. 700 Atm.		-.90	-.58	-.50	-.15	-.77	-.29	-.12	3.31
Paste Presses, Pasting Oil. Flush. Oil & Wat. Pumps.		6.83	1.98	1.57	45	2.41	.10	56	14.73
Oil Wash 700 Atm. Circ. gas		2.18	-.90	1.33	-.41	2.05	-.77	-.29	7.93
Stalls 700 Atm. L-Phase		12.49	1.94	7.06	2.17	10.85	4.08	1.46	40.05
Centrifuging		-.51	1.16	-.77	-.24	1.18	-.44	-.17	4.47
L.T.C.		2.51	3.50	1.10	-.34	1.69	-.64	-.39	10.17
Distillation L. ÷ V-Phase		7.70	2.16	1.40	-.43	2.15	-.81	-.58	15.23
Inj. Pumps		-.80	1.18	-.50	-.15	-.77	-.29	-.15	3.84
300 Atm. V-Pg.		-.78	-.72	-.57	-.17	-.87	-.33	-.13	3.57
Gas Circ. 300 Atm.		3.84	1.96	3.22	-.99	4.95	1.86	-.65	17.48
Stalls		-.59	-.20	-.19	-.06	-.30	-.11	-.07	1.52
Stabilization		-.07	-.20	-.06	-.02	-.09	-.04	-.02	-.50
Gasol. Wash		2.19	-.38	1.56	-.51	2.56	-.96	-.32	8.58
Debenz. & L.F.G. Units		1.84	-.78	1.97	-.61	3.03	1.14	-.36	9.73
Storage Tanks H.& L. Press. Lines Press. Test Pit, Insul. Scaffold, Emerg. Expans. Foamite Install.				1.45	-.45	2.23	-.84	-.18	5.15
Total RM/t Av. Gas	143.99	51.78	19.67	26.26	8.07	40.36	15.18	12.28	321.98

Av. Gas. fr. Bit. Coal Tar - Hard Pitch (Bal. 28. Dec. 39)

L.F.G. Recovery 1) 4.4 t/h

Total Heat Value of Hygas fr. L + V - Phase 100.5×10^6 Kcal/h
Less Heat Value of L.F.G. (14.4 t) 48.4×10^6 " "

For H_2-Production are available 152×10^6 Kcal/h

Recoverable therefrom $\frac{52.1}{2400}$ = 21,600 m³ H_2 = 57 % of Total H_2

H_2-Reqmt. 37,800 m³/h;
To be produced from coke

$$\begin{array}{r} 37{,}800 \\ -21{,}600 \\ \hline 16{,}200 \text{ m}^3\ H_2 \end{array}$$

For which coke req'd = 13.5 t

Resid. wat. gas yield 16.2 x 0.63 = 10.2×10^6 Kcal/h

Fuel gas req'd for hydrogenation 28.8×10^6 Kcal/h

" " " " splitting 21.6×10^6 " "

$$\begin{array}{r} 50.4 \times 10^6 \text{ Kcal/h} \\ -\ 10.2 \times 10^6 \text{ Kcal/h} \\ \hline \end{array}$$

Fuel gas to be produced in generator 40.2×10^6 Kcal/h

For which fuel gas coal required = 8.0 t.

	H.P. Steam	L.P. Steam	Water	Current	Men/Day	
Fuel gas production	--	--	240	380		
Hydrogenation	8.0	30.0	2430	11400	420	
H_2-Product fr. Coke	(11.3)	22.7	1450	8660	150	
" " " Hygas	---	15.8	1785	11200		
Waterworks	--	0.3	--	1700	255	Power Water + -Fuel Gas
Auxil. Install.	--	6.0	570	1600		
	(3.3)	74.8	6235	34560	825	

---

1) For Calcul. see "Av. Gas. fr. Bit. Coal".

Power Coal Calculation for Av. Gas. fr. Hard Pitch.

75 t L.P. Steam ⟶ 109 t Live steam = 15,300 KW

34,560
-15,300
19,260 KW x 4.2 = 81 t Steam
109
190 t Steam
- 3
187 t Steam

$$\frac{187,000 \times 600}{6300 \times 0.8} = \frac{187 \times 600}{5.05} = 37.0 \times 600 = 22.8 \text{ t Power Coal}$$

Plant Costs:

Hydrogenation:	L-Phase	21.5 Mill.	(Estimate 31.140)
	V- "	13.6 "	(" ")
	Common Installations	3.4 "	(" " + L.F.G. Plant)
H_2-Production	37,800 m^3	16.0 "	(Est. 15. July 40)
Power Production (Steam Current Water)		20.0 "	" "
Fuel Gas Production		3.0 "	
		77.5 Mill.	
Auxil. Install.		19.4 "	
		96.9 Mill.	
Other Capital Expendit. 15%		14.6 "	
		111.4 Mill.	

Av. Gas. fr. Bit. Coal Tar - Hard Pitch E.P. 66.5°
Basic Cost Calculation

Production: 102,500 t/ann. Av. Gas. in 2(0000) = 70.5 m³ (18:15 m Conv. = 3:2)

Input: 165,000 t/ann Pitch

A. **Operating Costs:**

				RM/t Av.Gas.	
Coke:	107,500 t/ann	@ 30.-/t	= 3,230,000.-	31.50	
Power Coal:	178,000 t/ann	@ 13.80	= 2,460,000.-	24.--	
Fuel Gas Coal:	64,000 "	@ 18.80	= 1,200,000.-	11.70	
Catalyst + Chemicals:					
L.-Ph.	3,300 t/ann Coke	@ 12,500.-	= 1,055,000.-	= 10.30	
V.-Ph.	5058: 22 m³	@ 12,500.-	= 278,000.-	= 2.71	
" "	6434: 23 "	@ 4,150.-	= 95,500.-	= -.93	
Splitting: Cat.	I 8.05 t	@ 3,000.-	= 24,200.-	= -.24	
"	II 7.33 t	@ 2,000.-	= 14,700.-	= -.14	
"	III 23.8 t	@ 800.-	= 19,000.-	= -.19	
Conversion:	37.6 t	@ 1,650.-	= 62,000.-	= -.61	
Chemicals:	512 t NaOH	@ 150.-	= 76,900.-	= -.75	15.87
Wages:	825 x 8 x 365	@ 1.-	= 2,410,000.	= 23.50	
Salaries:	20 % of Wages		= 480,000.-	= 4.68	
Repairs:	485 x 8 x 365 (4.95% of 96.9 Mill. Plant + Aux. Pl. Costs)	@ 3.40	= 4,820,000.-	47.-	
Operat. Matl!	15 % of Wages		= 362,000.-	= 3.53	
Welfare:	20% of Wages & Salaries		= 579,000.-	= 5.64	84.35

Operating Costs RM/t Av. Gas. 167.42

B. **Raw Material**

Pitch: 165,000 t/ann @ 35.-	-= 5,780,000.- =	55.40
Production Costs RM/t Av. Gas.		223.82
General: 2.5% of Prod. Costs, Amortization, Fire Protect. & Taxes (RM/t 344.02)		8.62
License:		5.--
		237.44

	Brought Forward		237.44
C. **Capital Burden:**			
Amortization:	10% of 96.9 Mill. = 9,690,000.-	= 94.40	
Interest:	3% " " " = 2,000,000.-	= 28.30	
	5% " 14.5 " = 725,000.-	= 7.00	
Fire Protect. & Tax:	2% of 96.9 Mill = 1,950,000.--	= 18.90	148.66
			386.10
Credits:	5,100 t/ann L.T.C. Tar fr. Fuel Gas prod. @ 90.- 460,000.-	=	(4.50)
	35,000 t/ann. L.F.G. @ 150.- = 5,250,000.-	=	(51.20)
	RM/t Av. Gas		330.40

Av. Gas. fr. Hard Pitch
Estimate of Cost

(Bal. 28 Dec. 39).

Basis:

Pitch Quality: E.P. 66.5°, 10% Solids; 1% Ash in Solids, 85% Solids Decomp, 91.7% C: 4.5 available H.

L-Phase, 700 atm; 24% Gasif.; 0.22 Yield - 325°; Conc. 50%; 2 m^3 Gas/kg Inj.

V-Phase, 300 atm; 5058; Burden 0.7; 3% Gasif.; 15% conc.; 4 m^3 Gas/kg Inj.

V-Phase, 300 atm; 6434; Yield 0.45; 20% Gasif.; 60% Conc., 2 m^3 Gas/kg Inj.

Pitch Input: 165,000 t/ann

Av. Gas Production: 102,500 @ 2(0000) L-Phase-Quality.

	RM/t Av. Gas	
Raw Material:		
1.61 t Pitch @ 35.- 1)	56.30	
2940 m^3 H_2 @ 5.15 Pf.	151.50	
4070 m^3 Hygas-Credit @ 1000 Kcal 0.56 Pf./m^3 1)	(22.80)	
344 kg L.F.G. @ 15 Pf.	(51.60)	133.40
Cat. & Chemicals:		
32 kg Coke-Iron @ 32 Pf.	10.30	
0.214 Lit. 5058 @ 12.50	2.68	
0.224 " 5434 @ 4.15	-.93	
5 kg NaOH @ 15 Pf.	-.75	14.66
Power:		
0.625 t H.P. Steam @ 2.50	1.56	
2.34 t L.P. Steam @ 2.20	6.25	
234 m^3 water @ 1 Pf.	2.34	
1020 KW Current @ 2 Pf.	20.40	
2250 m^3 Fuel Gas @ 0.56 Pf.	12.00	42.55
Wages:		
a) 140 Men/Shift = 12.1 Work Hrs.	12.10	
b) Salar. + Addit. 30%	3.64	
c) Oper.-Mat. + Lab.-Costs 20%	2.42	
Burden 20% of a) to c)	3.63	21.79
Repairs etc.:		
6.5 % of 48.1 Mill. Rep.	30.50	
2 % " " " Fire Prot. & Tax	9.40	
10 % " " " Amortization	46.90	86.80
Prod. Costs		299.20

	Brought Forward		299.20
General:	2.5% of Prod. Costs	7.50	
Interest:	3 % of 48.1 Mill.	14.10	
	5 % " 7.2 "	3.51	
License		5.--	30.11
	RM/t Av. Gas		329.31

L) For Calcul. of H_2 & Fuel Gas see " Av. Gas. fr. Bit. Coal".

Balance for Pitch Mixture to Av. Gas. (10 Lit. Conv.)

1,000 kg Pitch mixture — 88.3 % C; 6.05 H avail.
755 kg Recycle heavy oil — 11.2 % Solids; 4.4 % Ash in Solids
20 kg Cat. Coke — 15 % Asphalt

1,775 kg Inj.
500 kg Letdown

2,275 kg Total Inj.

L-Ph. 700 Atm; 21% Gasif./Gas. + H_2 + Gasif; Yield 0.28
Recovery 85%; Conc. 50%; 2.5 m^3 Gas/kg Fresh Oil

623 kg Letdown → 500 kg Recycle
623 kg Letdown → 123 kg to L.T.C.
123 kg to L.T.C. → 69 kg L.T.C. Oil
123 kg to L.T.C. → 51 kg Solids + Coke
123 kg to L.T.C. → 3 kg Gas + Loss

175 kg Gasif. mean C 1.8; 2% Co
69 kg L.T.C. Oil
1,446 kg C.P. Product

1,515 kg T Distill.

5 kg Loss

755 kg Gasol. - Mi-Oil 86.3 C; 10 H avail.
755 kg C.P. Heavy Oil

H_2-Consumption

Chem.bd. 828 m^3 H_2/t Pitch Mixture
Free 190 " " " "
Loss 25 " " " "
1043 m^3 H_2/t Pitch Mixture

5058; 300 Atm; Burden 0.8; Gasif. 3%; 4 m^3 Gas/Kg. Inj.
25 % Conc.

20 kg C Gasif. mean C 2.4

743 kg C.P.
Distillation
3 kg Loss

H_2-Consumption

Chem. bd. 352 m^3 H_2/t Pitch Mixture
Free 15 " " " "
Loss. 15 " " " "
382 m^3 H_2/t Pitch Mixture

185 kg Gasol. 86.3 %; 16.1 H Avail.
555 kg Mi-Oil 87.5 %; 14.4 H Avail.
297 kg "B" Mi-Oil
852 kg

6434, 300 atm; Yield 0.45; Conc. 60%; Gasif. 20%; 2 m^3 Gas/kg Inj.

H_2-Consumption

97 kg C Gasif, mean C 3.7
756 kg C.P. Product
4 kg Loss

Chem. bd. 226 m^3 H_2/t Pitch Mixture
Free 32 " " " "
Loss 10 " " " "
268 m^3 H_2/t Pitch Mixture

455 kg Gasol. 85.5% C; 17.0 H Avail.
297 kg "B" Mi-Oil

640 kg Av. Gas.

Hygas Yield

172 kg C @ 14500 = 2,500,000.-
3 kg C @ 5700 = 17,000.-
237 m^3 H_2 @ 2360 = 560,000.-
20 kg C @ 13800 = 276,000.-
97 kg C @ 13300 = 1,290,000.-
4,643,000.- Gasol.

1,693 m^3 H_2/t Pitch Mixture
= 2,650 " H_2/t Av. Gas.

= 4.40 x 10^6 Kcal/t Pitch Mixture.

Av. Gas. fr. Pitch Mixture

L.F.G. Recovery[1] 4.8 t/h

Total Heat Value of Hygas fr. L + V - Phase:

(95 % Yield) 115 x 10^6 Kcal/h

Less Heat Value of L.F.G. 52.8 x 10^6 Kcal/h

For H_2-Prod. are available 62.2 x 10^6 Kcal/h

From which may be recovered $\frac{62.2}{2400}$ = 25,900 m^3 H_2 = 58.5 % of Total H_2

H_2-Reqmt. 44,200 m^3/h

To be produced from coke 44,200 - 25,900 = 18,300 m^3/h

For which 15.2 t coke req'd.

Fuelgas req'd for Hydrogenation 32.9 x 10^6 Kcal/h

" " " Splitting 25.9 x 10^6 Kcal/h

58.8 x 10^6 Kcal/h

Resid. watergas -11.5 x " "

Fuelgas to be produced in generator 47.3 x 10^6 Kcal/h

For which 9.5 t fuel gas coke req'd

	H.P. Steam	L.P. Steam	Water	Current	MAN/. DAY
Fuel Gas Prod.	--	--	280	450	36
Hydrogenation	10.1	33.0	2845	12865	405
H_2-Prod. fr. coke	(12.8)	25.6	1640	9800	162
" " " Hygas	--	18.9	2140	13400	
Waterworks	--	0.35	--	2000	226
Aux. Install.	--	7.8	740	2080	
	(2.7)	85.65	7645	44595	829

(Power Prod)

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[1] For Calc. see "Av. Gas. fr. Bit. Coal".

Power Consumption for Av. Gas. fr. Pitch Mixture

	Water	H.P. Steam	L.P. Steam	Gas	Water	High Tension	Low Tension	Men/Shift
Pitch Melting	26.1	—	1.4	—	—	—	—	3
L-Ph. 700 Atm.								
Pasting	59.3	0.93	1.23	—	2	965	97	4
Paste Presses	59.3	—	2.4	—	—	3800	42	3
Water Pumps, Flush. Oil Pp.	6.0	—	—	—	—	240	1	3
Gas Circ.	65000	—	—	—	—	390	7	2
Compressors	27300	—	—	—	82	2450	—	2
Oil Wash	55	—	5.5	—	17	335	60	3
Stalls	59.3	0.59	10.1	12800	670	740	165	10
L.T.C.	3.2	0.21	0.15	1520	42	129	49	6
C.P. Distill.	39.6	—	2.5	8800	550	—	123	6
V-Phase. 300 Atm.								
5058 Inj. Pumps	19.7	—	—	—	—	250	4	1
Wat. Pumps	2.0	0.32	—	—	1	—	40	1
Gas. Circ.	79000	—	0.28	—	—	390	7	2
Stalls	19.7	—	0.4	—	214	300	23	10
6434 Inj. Pumps	22.3	—	—	—	—	283	5	1
Wat. Pumps	2.2	0.32	—	—	2	—	45	1
Gas Circ.	44600	—	0.16	—	—	194	2	1
Stalls	22.3	—	0.42	2230	251	342	25	10
C.P. Distill.	39.1	—	1.89	7400	555	—	157	6
Debenz & L.F.G.	6.1	2.2	0.7	—	200	780	—	4
Gasol. Wash & Stab.	16.7	2.9	0.78	—	144	—	45	4
Int. Tanks	16.7	2.1	3.7	—	—	—	230	3
Sales Tanks Hygas Tank Offices, Labs.	16.7	0.3	3.3	154	115	17	132	2
	—	10.07	32.91	23914	2845	11605	1260	90 + 50% = 135
						12 865		

Power Coal Calculation for Av. Gas fr. Pitch Mixture

85.65 t L.P. Steam - 124 t Live Steam = 17,500 KW

$$\begin{array}{r} 44{,}595 \\ -\ 17{,}500 \\ \hline 29{,}095 \end{array} \text{ KW} \times 4.2 = \begin{array}{r} 114 \text{ t Steam} \\ 124 \text{ '' ''} \\ \hline 238 \text{ t Steam} \\ -\ 3 \phantom{\text{ t Steam}} \\ \hline 235 \text{ t Steam} \end{array}$$

$$\frac{235{,}000 \times 600}{6300 \times 0.8} = \frac{225 \times 600}{5.05} = 465{,}600 = 28.0 \text{ t Power Coal}$$

Plant Costs:

Hydrogenation L-Phase	22.0	Mill.	(Est. 3/Jan./40, reestim.
	14.9	"	(" " "
	3.8	"	(" " " + L.F.G.)
H_2 Production: 44200 m^3	18.7	"	(Est. 15/July/40 Frankenthal)
Power Production:	25.2	"	
Fuel Gas Production:	3.5	"	
	88.1	Mill.	
Auxil. Install. 25%	22.0	"	
	110.1	Mill.	
Other Capital Reqmts.	16.5	"	
	126.6	Mill.	

Av. Gas. fr. Pitch Mixture

Bit. Coal Tar Pitch : Producer tar : Bit. Coal Tar Distill. Oil
-350° (1:1:1)

Basic Cost Calculation.

Production: 133,500 t/ann Av.Gas in 2(0000) = 70.5 m3 (18:15 m Conv.=3:2)
Input: 209,000 " Pitch Mixture

A. Operating Costs:	RM/t Av. Gas
Coke: 121,500 t/ann @ 30.-/t = 3,650,000.-	27.30
Power Coal- 224,000 " @ 13.80/t= 3,000,000.-	22.90
Fuel Gas Coal 76,000 " @ 18.80/t= 1,430,000.-	10.70
Catalyst + Chemicals: L-Ph. 4170 t/ann coke @ 320.- = 1,340,000.- = 10.--	
V-Ph. 24.6 m^3 @ 12500.-(5058) = 308,000.-=2.30	
" " 26.4 m3 @ 4150.-(6434) = 109,500.-= .82	
Splitting: Cat. I 9.65 t @ 3000.= 29,000.- = .22	
" II 8.8 t @ 2000.- = 17,600. = .13	
" III 28.6 t @ 800.- = 22,850. = .17	
Conversion: 44 t @ 1650.- = 72,500. = .54	
Chemicals: 670 t NaOH @ 150.- = 100,000. = .75	14.93
Wages: 829 x 8 x 365 @ 1.- = 2,420,000.- = 18.12	
Salaries: 20% of wages = 484,000.- = 3.62	
Repairs: 540 x 8 x 365 @ 3.40 = 5,335,000.- = 40.-- (4.85% of Plant & Aux. Pl. Costs)	
Operating Matl. 15% of wages = 363,000.-= 2.72	
Wellfare: 20% of wages & salaries 580,000.- = 4.35	68.31
Operating Costs RM/tAv. Gas.	144.14
B. Raw Material:	
Pitch Mixture: 209,000 t/ann @ 60.- = 12,550,000.-	94.--
Production Costs RM/t Av.Gas.	238.14
General: 2.5 % of Prod. Costs, Amortization, Fire Prot. & Tax (RM/t 337.12)	8.40
License	5.--
Carried Forward	251.54

Brought Forward 251.54

C. Capital Burden:

Amortization: 10% of 110.1 Mill. = 11,010,000.= 82.50

Interest: 3% " " " = 3,300,000.= 24.70

5 % " 16.5 " = 825,000.= 6.18

Fire Prot. & Tax: 2% of 110.1 Mill.= 2,200,000.=16.48 129.86

381.40

Credits: 6100 t/ann L.T.C. Tar fr. Fuel Gas Prod. @ 90.- = 549,000.-- (4.11)

38,400 t/ann L.F.G. @ 150.- = 5,760,000.- (43.10)

334.19

Av. Gas. fr. Pitch Mixture
(Bal. 21. Aug. 41)
Estimate of Costs.

Basis:

Pitch Mixture: Bit. Coal Tar Pitch + Producer Tar + Bit. Coal Distill. Oil >350°

Properties: 11.2% Solids; 4.4 % Ash in Solids; 88.3 % C; 6.05 H avail.

L-Phase, 700 atm; 21% Gasif./Gasol + Mi-Oil + Gasif.; Yield 0.28; Solids Decomp. 85%; Conc. 50%; 2.5 m³ Gas/kg Fresh Oil.

V-Phase, 300 atm; 5058; Burden 0.8; Gasif. 3%; 4 m³ Gas/Kg Inj.

25 % Concentration

6434; Yield 0.45; Gasif. 20% Conc. 60%; 2 m³ Gas/kg Inj.

Pitch Mixture Input: 204,000 t/ann.
Av. Gas. Production: 133,500 " @ 2(0000) L-Phase-Capacity

		RM/t Av. Gas.	
Raw Material:			
1.56 t Pitch Mix	@ 60.-	97.00	
2650 m³ H2	@ 5.15 Pf.	136.50	
3720 m³ Hygas-Credit	@ 0.56 Pf.	(20.80)	
287 kg L.P.G. @	@ 15 Pf.	(43.--)	166.70
Cat. & Chem.			
31 kg Coke-Iron	@ 32 Pf.	10.00	
0.184 Lit. 5058	@ 12.50	2.30	
0.198 " 6434	@ 4.15	-.82	
5 kg NaOH	@ 15 Pf.	-.75	13.87
Power:			
0.6 t H.P. Steam	@ 2.50	1.50	
2.44 t L.P. Steam	@ 2.20	5.37	
1988 m³ Fuel Gas	@ 0.56 Pf.	11.10	
214 m³ Water	@ 1 Pf.	2.14	
895 KW Current	@ 2 Pf.	17.90	38.01
Wages:			
a) 135 Men/Shift = 8.93 Work Hrs.	@ 1.-	8.93	
b) Salaries + Addit. 30%		2.68	
c) Oper.-Mat. + Lab. Cost 20%		1.79	
Social Burden 20 % of a) to c)		2.68	16.08
Repairs etc.:			
6.5 % of 51.0 Mill. Rep.		24.80	
2 % " " Fire Prot. & Tax		7.62	
10 % " " Amortization		38.20	70.62
	Production Cost		305.38

	Brought Forward		305.38
General:	2.5% of Prod. Costs	7.65	
Interest:	3 % of 51 Mill.	11.45	
"	5 % " 7.65 Mill.	2.86	
License		5.--	26.96
	RM/t Av. Gas		332.34

TOM Reel 7 U. S. BUREAU OF MINES T-251
HYDRO. DEMON. PLANT DIV.
KCBraun
2-21-47

INFLUENCE OF THE PROPORTION OF FEED TO GAS IN 600 ATM. VAPOR PHASE

Main Laboratory, Ruhröl, 4 June, 1943.

In our work in 600 atm. vapor phase, the results of which are recorded in the report on "The single stage splitting hydrogenation in the vapor phase at 600 atm.," we have always worked with the constant feed: gas proportion of 1:3000.

To supplement the work done to date, we, therefore, made other experiments (G70/1-5) to determine the influence of various quantities of inlet gas at constant feed charge.

The operating conditions of the vapor phase stall were:

Catalyst volume:	5.4 l
Catalyst:	K534
Injection Feed:	P 101/102/302
Temperature:	25.5 mV
Pressure:	550 atm.
Gas:	Make-up (Frischgas)
Load:	1.0 kg/l/h.
Feed: gas proportion=	1:2000 1:3000 1:6000

The results of these experiments are given in the attached table.

They show that yield, gasification and analyses of the products of reaction do not differ very much and, therefore, are largely independent of the inlet gas, within the limits of the conditions of the experiments.

But the experiments clearly showed, what is not apparent in the table, that with the low feed: gas proportion of 1:2000, and, therefore, a low oil partial pressure, the effectiveness of the catalyst deteriorates much faster than with a proportion of 1:3000 and above.

The lower retention time, due to the large gas flow, such as 1:6000, is characterized by a lowering of the phenol and base reduction, a lower aniline point of the gasoline, and a decrease in hydrogenation of the B-middle oil. This means, as we could prove in experiments G 70/4 and 5, a decrease in yield in the return of the B-middle oil compared to an operating method with lower gas input.

The increase in gasification with rising gas input, seen in the table, does not necessarily represent an actual increase in the formation of gaseous hydrocarbons, since this could, at best, be decreased with a decrease in the retention time.

The higher figures are due to the fact that, with the greater gas input, more lower boiling constituents of the catch pot product overflow with the gas, which then cause a sharp increase in the C-number in the gas analysis.

In large scale production, where the gas is always recycled, gasification probably is independent of the gas thruput, within the limits of the conditions of these experiments.

In summary, our experiments have shown that, in the single stage hydrogenation of middle oil in the 600 atm. vapor phase, too low a gas input, i.e. a feed: gas proportion of 1:2000 and below, damages the catalyst, while too high an input (feed: gas proportion of 1:6000 and above) reduces the yield and effects a poor reduction of phenols and bases. The most favorable is the feed: product proportion of about 1:3000, used in our experiments and large scale production. Limiting values may be considered to be 1:2500 and 1:4000.

	Proportion of Product to Inlet Gas 1:2000	Proportion of Product to Inlet Gas 1:3000	Proportion of Product to Inlet Gas 1:6000
Yield: Gasol-165°/1/h	0.48	0.50	0.50
Hydrocarbon Gasification / Gasol.-165° + Gasification	19%	21%	22%
C.P. Product Analysis:			
D_4^{15}	0.832	0.828	0.830
A.P.	-7.5°	-5.5°	-8.0°
-165° (Column)	60%/vol.	62%/vol.	62%/vol.
Phenol	0.03	0.02	0.04
Bases	0.003	0.002	0.004
Gasoline Analysis:			
D_4^{15}	0.785	0.783	0.787
A.P.	+8.5°	+9.5°	+7.5°
-100° (A.S.T.M.)	49%/vol.	51%/vol.	50%/vol.
Aromatics (A.P.-Meth.)	43%/wt.	42%/wt.	44%/wt.
Middle Oil Analysis:			
D_4^{15}	0.909	0.910	0.918
A.P.	<-25°	<-25°	<-25°

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T-252

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2-26-47

LIQUID PHASE OPERATIONS AT PÖLITZ 1942

Ludwigshafen, 10 Feb. 1942/Wch.$_4$

Attached Table 1 shows a comparison between the operating method in the coal phase at Pölitz of October 19-20th with that of January 11-20th, 1942. Great differences are indicated in decomposition, thruput, asphalt, solids in the letdown and pasting oil, circulating gas quantity, etc, at equal maximum converter temperature.

To the yield of 0.29 during all of October may be remarked that, from the actual yield of gasoline plus middle oil from coal in October, a yield of only 0.18 is calculated by extrapolation to 8000 working hours per annum based on the monthly reports from Pölitz. The same yield is also obtained for September, taking into consideration that the stalls only started running on the 9th, and converting to the full month.

Table I. Pölitz Liquid Phase.

	Ideal Calculation 1)	Oct. 11-20,-41 Averages From K13 & K14	Jan. 11-20,-42 Averages From K13 & K15
Conv. Temp. max. mV		25.0-25.1	25.1
C.P. Temperature max. mV		22.0-23.0	22.5-23.1
Gas Input incl. Cold Gas/paste	1.5-1.8 4)	1.7 m^3	2.1 m^3
Letdown/Paste	41.0% 2)	41.2%	58.4%))105.61
C.P. Product/Paste	50.0%	41.5%	47.2%
Letdown % Solids	25%	30%	19.3%
Asphalt in Oil	14%	8.5%	14.8%
Pasting Oil % Solids	9%	14.3%	5.4%
C.P. Product %-325°	47%	51.4%	47.5%
Centrifuge Residue % Solids	38%	38.2%	40.5%
L.T.C. Yield	80%	88.0%	80.7%
Yield $t/m^3/h$	0.26	0.3 5)	0.22
Decomposition	96%	93.3%	84.7%
Paste Thruput/m^3 3)	1.07 t	1.18 t	0.82 5

1) 5.7% Ash in dry coal
45.9% Solids in Paste,
no letdown recycle.

2) At 23% Solids Content
54% at 19% solids content.

3) Thick & thin paste

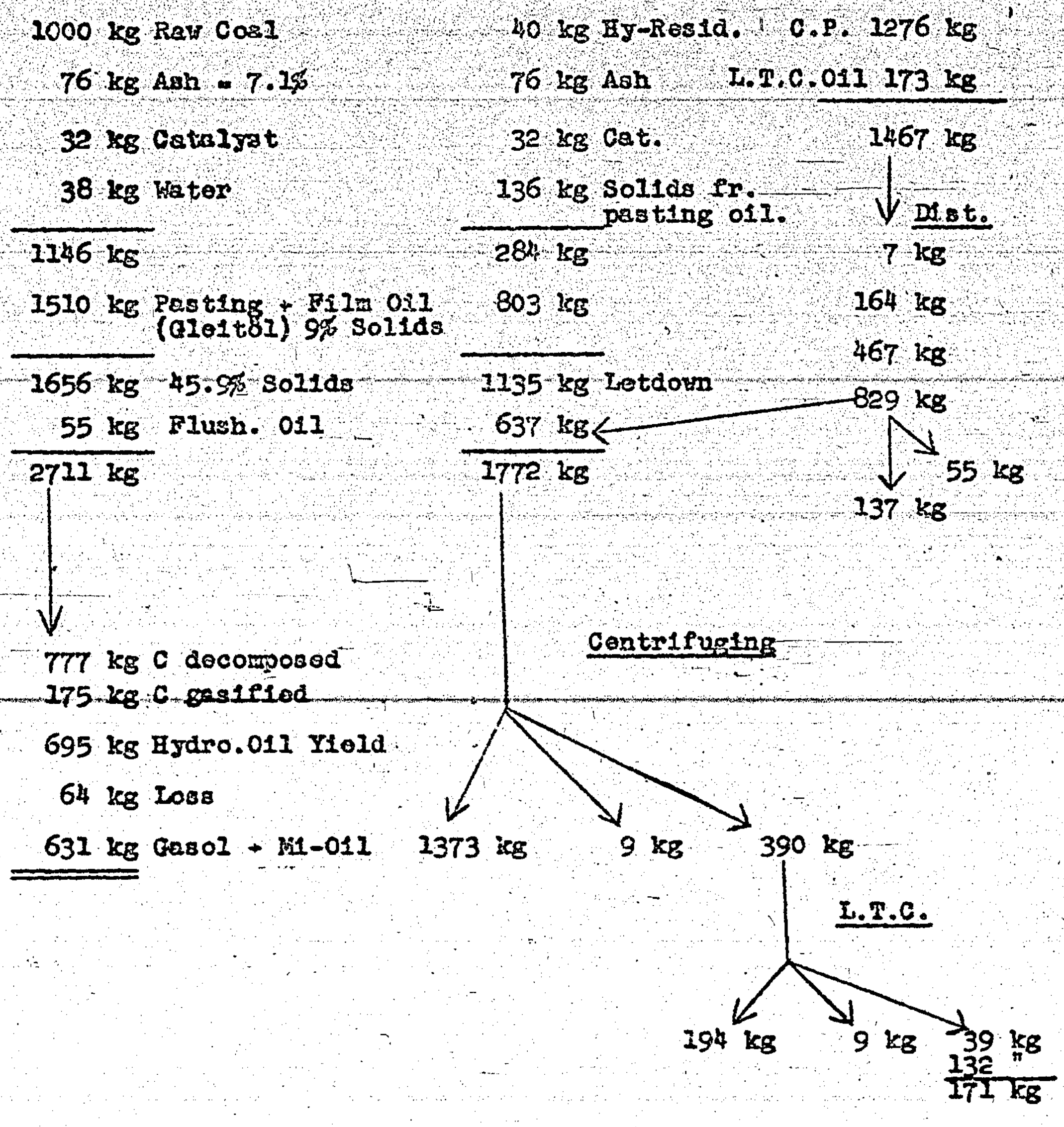
4) 4-4.85 m^3 Gas/t Raw Coal

5) From 1.-31.Oct. Yield 0.29

Table 2 shows the Ludwigshafen ideal balance for coal to gasoline and middle oil converted to the Pölitz operating method with paste heat exchange and consequent lower solids content of the paste, omitting the letdown cold recycle and figuring 7.1% ash in the dry coal and 3.4 water in the raw coal, which corresponds to the coal input in September and October.

Table 3 shows a comparison between thruput or consumption figures for the oil recovery available per ton so calculated and the average Pölitz production schedule for September-October, after converting to 1 ton available oil recovery, consisting of 98.4% gasoline plus middle oil and 1.6% heavy oil. It indicates that the gasification and consequent H_2 consumption and product factor are still far above the ideal. The input into the L.T.C. (Schwelerei) is disproportionally large, and this unit also has very heavy losses, partly in the form of gas, partly not specifically designated. On the contrary, the oil yield is normal

Table II. Lu Ideal Balance for Politz Operating Method.



1000 kg Raw Coal
76 kg Ash = 7.1%
32 kg Catalyst
38 kg Water
1146 kg
1510 kg Pasting + Film Oil (Gleitöl) 9% Solids
1656 kg 45.9% Solids
55 kg Flush. Oil
2711 kg

777 kg C decomposed
175 kg C gasified
695 kg Hydro.Oil Yield
64 kg Loss
631 kg Gasol + Mi-Oil

40 kg Hy-Resid.
76 kg Ash
32 kg Cat.
136 kg Solids fr. pasting oil.
284 kg
803 kg
1135 kg Letdown
637 kg
1772 kg

C.P. 1276 kg
L.T.C.Oil 173 kg
1467 kg
Dist.
7 kg
164 kg
467 kg
829 kg
55 kg
137 kg

Centrifuging
1373 kg
9 kg
390 kg

L.T.C.
194 kg
9 kg
39 kg
132 "
171 kg

Change compared to Balance of 4. Oct.41

7.1% Ash in dry coal.
45.9% Solids in Paste
No Letdown cold recycle.

Table III

1000 kg Available Oil Recovery

In Lu, Gasol. - Mi-Oil = 100%
In Pö, Gasol. + Mi-Oil = 98.4% + 1.6% Heavy oil.

	Lu Ideal Balance Pölitz Oper. Method Dry Coal 7.1% Ash	Monthly Average Sept. Oct. Pölitz	% of Lu
Make-up gas	186 kg H_2	226 kg Make-upgas	121
Coal (Raw)	1585 kg	2260 kg	143
Pasting + Film Oil (Gleitöl)	2390 kg	4180 kg	175
Flushing Oil	87 kg	360 kg	413
Stall Intake (liquid)	4300 kg	7060 kg	164
Letdown	1800 kg	3340 kg	186
Centrifuging	2802 kg	5010 kg	179
L.T.C. (Schwelerei)	618 kg	1136 kg	184
L.T.C. Oil	307 kg	550 kg	180
Loss + Gas	14 kg	46 kg	327
Residue	270 kg	540 kg	200
C.P. Product	2020 kg	2690 kg	133
Gasification	384 kg = 24% C. Gasif. (or 368 kg hydrocarbon + CO$_2$) Expenses & Raw Material 306.--	730 kg (37% C-Gasif) Expenses & Raw Material 465.	186

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THE RECOVERY OF PHENOLS IN HYDROGENATION

By Dr. Becker, Ludwigshafen, 16 Feb. 1942.

Taking out the lower phenols (to Xylenol) from bituminous coal liquefaction middle oil has the effect of deteriorating the quality of the aviation gasoline, which however, can be compensated for by the proper operating method.

We are at present working on these problems. According to results of experiments to date, removing carbol, cresol and xylenol has the following effect:

1.) The 87 octane aviation gasoline from bituminous coal liquefaction middle oil deteriorates by 1/2 to 1 octane number.

2.) The same thing is true for rhenish brown coal.

3.) Experiments with Brüx tar are still in progress. The deterioration of the octane number on removal of the phenols appears to be somewhat greater here.

4.) Aromatization with catalyst 7019 is practically impossible after dephenolizing because of too low a yield.

5.) For the DHD processes, the removal of phenols has the effect of lowering the yield of DHD-gasoline by 0.15% for each percent phenol.

6.) The autothermal operation of the prehydrogenation converters is made more difficult by the removal of phenols.

In the final analysis, all these influences are economic questions, except for aromatization, because lowering the octane number of the aviation gasoline can be compensated for by lower cutting off (abschneiden) of the gasoline.

The phenol oil requirement is 150,000 t/ann, of which 100,000 tons are required for plastics and 50,000 tons for fiber board for the aviation industry.

The following production had been planned up to the present time:

Merseburg Phenol Oil,	10,000	t/ann phenol and cresol
Brüx I	12,000	" " " "
DEA Rositz	2,500	" " " "
ASW	12,000	" " " "
Brüx II	12,000	" " " "
Rütgerswerke	8,000	" " " "
From bit. coal tar distill.	23,300	" " " "
I.G. Leverkusen	4,500	" " " "
Synthesis	9,200	" " " "
Blechhammer	5,100	" " " "
From bit. coal L.T.C.	15,000 1)	" " " "
Total	113,600	t/ann

1.) Old figures. It is proposed to increase this to 50,000 t/ann.

To cover the deficiency of about 50,000 tons, recourse may be had to hydrogenation (Gelsenberg, Scholven, Pölitz, Rheinbraun, Brabag, enlarged Blechhammer) as well as to brown coal tar distillation (additional production of 30,000 t/ann. possible), or also to bituminous coal low temperature carbonization.

Recovery of Phenols in Scholven & Gelsenberg.

By Dr. Donath, Ludwigshafen, 11 February 1942.

No direct laboratory experiments with dephenolized bituminous coal oils have been made. From experiments in prehydrogenation of bit. coal middle oils, with and without liquid phase gasoline, the following values were obtained for gasolines with an end point of 150°C:

Bit. coal liq. ph. gasoline (Prehydr.)	O.N.	60	-100°	abt 50%
Prehydr. gasol. fr bit. coal middle oil,	O.N.	69	-100°	10%
6434 gasoline,	O.N.	76	-100°	60%

By taking out the phenols, equivalent to omitting the prehydrogenation gasolines produced from them, the gasoline production per 100 parts of the removed phenol, cresol or xylenol is reduced by 90, 91 or 92 parts, respectively, plus the loss in process of dephenolizing. The octane number of the mixed gasoline is practically unchanged, though it could deteriorate if, of the phenols, only phenol itself and/or cresol were removed. The % of-100°C is increased abt. 2% by the removal of 5% phenols, if benzination is not run differently. The naphthene and aromatics content of the gasoline is reduced 2.5% by the removal of each 5% phenols.

In the DHD process the quality is not affected or only very little, but the lower naphthene content of the gasoline might reduce the yield by abt. 0.15%/1% phenol removed.

The removal of phenols reduces the heat of reaction in prehydrogenation, since bit. coal middle oil generates about 350 kcal/kg injection, while phenols generate about 550 kcal/kg in the reduction to naphthenes. By the removal of 10% phenols the heat of reaction is, therefore, reduced from 350 to 330 Kcal. While this reduction is small, the phenol reduction is very fast and is, therefore, important to the incubation (anspringen) of the converter, so that autothermal operation of the prehydrogenation converters is thereby impeded.

CHANGE IN QUANTITY AND QUALITY OF AVIATION GASOLINE FROM BITUMINOUS COAL LIQUEFACTION MIDDLE OIL BY THE REMOVAL OF PHENOLS.

By Dr. Peters, Ludwigshafen, 10 February, 1942.

If the phenols are removed from the bit. coal liquefaction middle oil, the following changes take place in prehydrogenation with catalyst 7846W and succeeding benzination with catalyst 6434:

1.) Instead of 1000 kg liquefaction middle oil, only 800 kg dephenolized middle oil are available for prehydrogenation.

2.) The quantity of prehydrogenation gasoline is decreased from 125 kg to 45 kg, or 65% (Gasoline to 150°C).

3.) Instead of 668 kg, only 597 kg. of prehydrogenation B-middle oil, or 90%, are available for the 6434-stage, for each 1000 kg. liquefaction middle oil.

4.) The yield in the 6434-stage is unchanged. However, the total gasification in the prehydrogenation plus the benzination stage rises from 18.3% to 19.8%, because the gasoline constituent from the phenols is produced with very little gasification.

5.) The quality of the prehydrogenation and the 6434-gasoline is practically unchanged, as far as can be foreseen. However, the quality of the mixed gasoline changes with the correspondingly changed mixture proportion. The product containing phenols consists of 84 parts 6434-gasoline and 16 parts prehydrogenation gasoline, the dephenolized product of 93 parts 6434-gasoline and 7 parts prehydrogenation gasoline.

6.) Due to the reduced quantity of prehydrogenation gasoline, the aviation gasoline from the dephenolized product contains a little less naphthene and a little more (low-boiling) paraffin. The anti-knock quality of the gasoline is, therefore, improved somewhat.

7.) With 5058/6434 all indicated changes should be less pronounced than with 7646 W/6434.

PHENOL RECOVERY FROM HYDROGENATION, PARTICULARLY BIT. COAL HYDROGENATION

By Drs. Becker, Donath, et al, Ludw. 11 Feb. 1942.

I. Quantities:

According to experimental data, the quantities produced in bituminous coal hydrogenation are:

a) When running middle oil:

7-8% phenols (carbolic acid to and incl. xylenols) based on practical oil yield.

b) When running fuel oil:

3.5-4% phenols (carbolic acid to and incl. xylenols) based on practical oil yield.

They consist of:
15-20% carbolic acid
35-40% cresols
40-50% xylenols.

If we assume that these phenols can be recovered with a yield of 80%, we then obtain:

Instead of 100,000 t av. gas, O.N. 87,
93-94,000 t av. gas, O.N. 87
+ 7-8,000 t phenols, or:

Instead of 100,000 t av. gas + fuel oil
(47,500 t - 52,500 t),
44,500 t aviation gasoline
+ 3,200 t phenols
+42,500 t fuel oil, or:

Instead of 100,000 t DHD-gasoline,
91,500-92,500 t DHD gasoline
+ 8,750-10,000 t phenols

	Bit. Coal Liquefaction Middle Oil >150°			→ →	200 kg Phenols 800 kg Dephen. Mi-Oil.		
Spec. Grav.	0.960				0.954		
Anil. Point	- 18				- 18		
Phenols	20 %				< 1 %		
Boil. Range	180 / 325				180 / 325		
	Prehydrogenation 7846 W 250						
	10 kg Gasification				8 kg Gasification		
	30 kg Water						
	125 kg Prehy. Gasol. to 150°				45 kg Prehy. Gasol. to 150°		
	835 kg Mi-Oil >150°				747 kg Mi-Oil >150°		
	Benzination 6434						
	167 kg Gasification				150 kg Gasification		
	668 kg 6434-Gasol. to 150°				597 kg 6434-Gasol. to 150°		
	177 kg Total Gasification				158 kg Total Gasification		
	793 kg Total Gasoline				642 kg Total Gasoline		
Gasif/Gas + Gasif.	18.3 %				19.8 %		
	Prehy. Gas	6434-Gas	Mix.Gas		Prehy. Gas.	6434-Gas.	Mix. Gas.
kg	125	668	793		45	597	642
Parts	16	84	100		7	93	100
Spec. Grav.	0.759	0.731	0.739		—	—	0.735
A.P. I	38	52	48		—	—	49
A.P. II	41	54	51		—	—	52
Init. Boil.	85	57	53		—	—	60
% - 70	—	7	5		—	—	6
% - 100	34	54	50		—	—	52
End Point	155	152	152		—	—	152
Paraffins	3	43	34		—	—	39
Naphthenes	92	52	61		—	—	56
Aromatics	4	4	4		—	—	4
Unsaturated	1	1	1		—	—	1
O.N. MM	71	74	73.5		—	—	74
O.N. with 0.12% lead		91.5	90		—	—	91

Since valuable cyclic compounds are removed with the removal of phenols for the DHD process, the removal of the phenols is accompanied by a loss in yield greater than the quantity of phenols recovered and which equals abt. 0.15% DHD gasoline for each percent phenols.

II. Qualities:

a). Influence of the Removal of Phenols on the Quality of Aviation Gasoline, Octane No. 87.

If all "sour oils" are removed from the gasoline and middle oil from bit. coal liquefaction, practically no change in octane number of the 87-gasoline takes place with the usual prehydrogenation with 5058 or 7846 W. If, as proposed, hydrogenation is weaker, a deterioration up to 1 octane number could occur.

If only phenols, cresols and xylenols are removed, a deterioration in the octane number of the 87-gasoline of ½ to 1 point will take place, when using the present day prehydrogenation catalysts.

What applies to bituminous coal also applies to Rhenish brown coal, since the phenol content to and including the xylenols, based on gasoline, is about the same as for bituminous coal.

Again, what applies to bituminous coal also tentatively applies to the removal of phenols to and including xylenols from the A-middle oil from Brüx tar, according to available data. Removing the phenols from A- and liq. phase-middle oil will probably lower the octane number somewhat more than with bituminous coal.

b). Influence of the Removal of Phenols on the Quality of High Performance Fuels.

The removal of phenols greatly influences aromatization with catalyst 7019. Contrary to former small scale experiments, in which no sharp difference between the processing of middle oils containing phenols and those from which the phenols had been removed had been observed, a new small scale experiment with dephenolized bituminous coal liquefaction middle oil indicated that the yield dropped to about 2/3, gasification increased considerably and that the aromatics content in the aromatization gasoline dropped by some percent at the same time. Judging from results of experiments to date, dephenolizing middle oil for 7019 aromatization is out of the question. With 700 atm aromatization, dephenolizing may not have such a strong effect.

Dephenolizing the feed in the production of DHD-gasoline also has an unfavorable effect, even though not nearly as much as in aromatization. If we want a DHD gasoline with equal aromatics content, its quality will be essentially the same with bituminous coal, but somewhat inferior with brown coal, than if the phenols were not previously removed. However, the yield in DHD-gasoline is reduced by 0.15% for each percent of phenols removed. There is also danger that too much low boiling constituents are formed because the feed gasoline contains less naphthenes.

III. Heat of Reaction.

In the prehydrogenation of bit. coal liquefaction middle oil, the heat of reaction drops from about 350 to about 330 kcal/kg injection when the phenol is removed, which makes the auto-thermal operation of the prehydrogenation converters more difficult.

UTILIZABLE PHENOLS (CARBOLIC ACID+CRESOLS) IN THE CATCH POT PRODUCT. (LARGE SCALE EXPERIMENT WITH BIT. COAL IN STALL 804).

By Löcker, Ludw. 2 February, 1942.

SUMMARY.

With the 35% fuel oil operating method (Heizölfahrweise), the utilizable phenols in the catch pot product was calculated to be 2.5%. When running 50% fuel oil the phenol content was 2.86% by analysis.

For 136 t/h catch pot product in Upper Silesia (at 8000 working hours per annum), 27,400 t/ann. utilizable phenols may, therefore, be expected with the 35% method and 31,200 t/ann. with the 50% method.

CALCULATION OF THE PERCENT OF UTILIZABLE PHENOLS IN THE C.P. PRODUCT WITH THE 35% FUEL OIL OPERATING METHOD.

Mean yield in C.P.P. - L.T.C. Oil II = 1103 kg/h,
divided by decadic mean into:
119 kg gasol. @ 6.22% phenols = 7.4 kg. util. phen.
551 kg Mi-Oil @ 3.8% phenols =20.4 kg. util. phen.

433 kg L.T.C. oil 27.4 kg = 2.52%
of the catch pot prod.

136 t/h x 8000 oper. hrs. x 2.52% phenols = 27,400 t/ann.

PHENOL RECOVERY IN THE PROCESSING OF UPPER SILESIAN BIT. COAL TO FUEL OIL.

By Dr. W. Krönig, Schlesien-Benzin 13 Feb 1942

The Reichsamt has requested I.G. to indicate the possible recovery of phenols in the works operating under its license. Schlesien-Benzin has received a similar request for its Blechhammer plant. In order to prevent I.G. from submitting figures differing from ours, the question was discussed with them in Ludwigshafen, 13 February 1943.

The pressure hydrogenation of Upper Silesian coal to gasoline, middle oil and 50% heavy oil excess (fuel oil), experiments with K 1197 (Heinitz coal) in a 10 liter converter, served as a basis for discussion. While the calculation of recoverable phenols is relatively simple when processing coal to gasoline and middle oil, because the quantities of gasoline and middle oil in the catchpot product agree both quantitively and qualitively with those in the oil yield, the calculation is much more complicated when running fuel oil, since a considerable portion of the middle oil in the catch pot product, 77% in the case of Upper Silesia, is returned to the liquid phase. Only the phenol in the liquid phase gasoline can, therefore, be considered recoverable without question, since this is not returned to the liquid phase. By comparison, the recoverable phenol in the middle oil can not be accurately determined. It may hardly be assumed that all the phenols in the middle oil of the catchpot product can be recovered, since dephenolized middle oil would then be returned to the liquid stall, which would produce a different phenol proportion (Phenolspiegel). To transpose the phenol content, as found in the catch pot product, to middle oils in the oil yield is only conditionally possible, because, according to analyses available today, it is impossible to say separetly, how much phenol is contained in the gasoline and how much in the middle oil. These considerations lead to different methods of calculation.

METHOD I.

The fraction to about 300°C is cut out of the unstabilized catch pot product of the coal stall and the phenols isolated from it. The phenols are then redistilled to the fraction 150-250°C. This phenol fraction is decomposed in the Podbielniak column into carbolic acid, o-cresol, m + p cresols, xylenols to 325°C, and higher phenols 225-250°C.

Available analyses indicate the following results:

Phenols 150-250°C = 9% (mean) of the catch pot product, 75% of the phenols 150-250° C boil between 150 and 225°C (socalled utilizable phenols) = 6.75% of the catch pot product.

The utilizable phenols 150-225°C consist of the following:

			% of C.P. Product		% of Oil Yield	
Carbolic Acid,	30%		2.02		4.26	
o-Cresol,	8%	40%	0.55	2.71	1.12	5.62
m + p-Cresols,	32%		2.16		4.50	
Xylenols,	30%		2.02		4.26	
		Total	6.75	Total	14.14	

We must assume that these figures are probably too high, because their validity would have to be based on the assumption that phenols C_6 - C_8 are always completely converted into gasoline in the distillation of the catchpot product, i.e., are not recycled with the middle oil. This assumption may still be valid for carbolic acid but its validity for phenols above C_6 is highly questionable.

METHOD II.

In the liquid phase product test the stabilized catchpot product, including light ends (Gasbenzin), is split into gasoline to 200°C and middle oil 200-325°C. The total phenol content of the two fractions is found to be 20% for gasoline and 16% for middle oil.

The gasoline equals 10% and the middle oil 55% of the catch pot product, giving a total of 10.8% phenols in the catchpot product. The proportion of phenols 150-250°C in the total phenol is unfortunately not known. We must, therefore, resort to an estimate. It was assumed that the phenols to 250°C in the constituents to 325°C equal the total oil to 250°C, namely 50%. Most probably, however, the phenol proportion to 250°C is much higher, because experience has shown that there are hardly any more phenols in the oils betw. 250-325°C. The following data then result:

50% of 10.8% = 5.4% of the catch pot product = phenols to 250°C. 75% of this = 4.05% of the catchpot products = utilizable phenols to 225°C, which are divided as follows:

	% of C.P. Prod.		% of Oil Yield	
Carbolic Acid	1.21		2.52	
o-Cresol,	0.33	1.63	0.69	3.40
m + p-Cresols,	1.30		2.71	
Xylenols,	1.21		2.52	
Total	4.05	Total	8.44	

METHOD III

This method is a combination of I & II. According to I, there are 6.75% utilizable phenols in the catch pot product, according to II, there are 2% (based on catch pot product) phenols in the gasoline to 200°C, so that 4.75% (based on c.p. prod.) remain for the middle oil, i.e., 54% of the phenols in the middle oil (200-325°) are, therefore, utilizable. The oil yield distribution for Blechhammer is:

Gasoline to 200° = 20.9%
Mi-Oil 200-325° = 28.1%
Heavy Oil over 325° = 51.0%

Accordingly:

20.9% gasoline x 20% phenols phenols in the gasoline,	=	4.2%
28.1% Mi-Oil x 16% total phenols x 54% utilizable phenols in the Mi-Oil	=	2.43%
Total		6.63%

Now, if the 6.63% utilizable phenols are normally divided, we get:

	% of Oil Yield	
Carbolic Acid	2.00	
o-Cresol,	0.52	2.63
m - p-Cresols	2.11	
Xylenols,	2.00	
Total	6.63	

METHOD IV.

The distribution of phenols to the various kinds according to III is based on the assumption, which cannot be proved, that the phenols in the oil yield are the same as those in the catchpot product. However, one can also assume that they are different. For example: The phenols in the gasoline consist entirely of carbolic acid, the phenols in the middle oil consist of the rest of the carbolic acid, the cresols and xylenols, according to the normal distribution formula. With this method of calculation, therefore, carbolic acid is strongly favored.

The calculation is as follows:

We start with the phenol distribution in the catchpot product as determined by Method I. From this we deduct the 2.0% phenols in the gasoline (according to Method II) as carbolic acid. For the residual phenols in the middle oil we then get a new percentage distribution, with which the phenols of the middle oil in the oil yield calculated by Method III are converted. This results in the following distribution, based on the oil yield:

	% of Oil Yield	
Carbolic Acid in Gasoline,	4.20	4.21
Carbolic Acid in Mi-Oil	0.01	
o-Cresol in Mi-Oil,	0.29	
m + p-Cresols in Mi-Oil,	1.10	
xylenols in Mi-Oil	1.03	
Total	6.63	

METHOD V.

This is closely related to Method II. The 20% phenols in the gasoline to 200°C of the oil yield are considered utilizable from the start. The utilizable phenols in the middle oil of the oil yield are calculated as follows:

Of the 16% phenols in the middle oil 4.8% are assumed to boil to 250°C, based on the boiling curve of the middle oil, of which 75% = 3.6% of the middle oil are assumed to be utilizable. We then have 20% utilizable phenols in the gasoline and 3.6% in the middle oil. By using the normal oil yield distribution we then get 5.20% utilizable phenols in the oil yield. According to the normal distribution formula, these are:

	% of Oil Yield	
Carbolic Acid,	1.56	
o-Cresol,	0.42	2.08
m + p-Cresols	1.66	
Xylenols	1.56	
Total	5.20	

METHOD VI.

This method served as a basis for our report to the Reichsamt of the 3 Feb. 1942, and was based on the compilation by Dr. v Hartmann of the 21 August 1941, according to which the liquid phase gasoline to 200°C contains 20% phenols, consisting of 50% carbolic acid and 49% cresols, and the liquid phase middle oil 200-325° contains 18% phenols, consisting of 60% xylenols and 40% higher phenols.

Using the distribution of the oil yield given in Method III, the following data are computed:

	% of Oil Yield
Carbolic Acid,	2.09
Cresols,	2.05
Xylenols,	3.04
Total	7.18

METHOD VII.

For the investigation of the phenols in Leverkusen, catch pot products from the collected quantities of gasoline, middle oil and heavy oil, from the large scale experiment in stall 804 and from a 10 liter converter experiment, were "synthetically" mixed. This "synthetic" catch pot product was then distilled in the fine fractionating column and a fraction 150-197°C cut out. The phenol content of this fraction was then determined. The phenols of this fraction equalled 2.52% of the catch pot product for stall 804 and 3.04% for the 10 liter converter. If we assume that this fraction contains all the carbolic acid and cresols, but no xylenols, we get the following quantities of carbolic acid and cresols:

Stall 804	27,400 t/ann.
10 l converter	33,200 t/ann.

SUMMARY.

The following table shows the distribution of utilizable phenols in percent of the liquid phase oil yield by the various methods and in t/ann, based on conditions at Blechhammer.

Of course, an objective evaluation of the various methods is hardly possible. But it may well be argued that Method I is certainly too high, Method V probably too low. Method III would probably come nearest the truth. Since the deviations of Method VI, on which the Schlesien-Benzin report to the Reichsamt is based, from Method III are slight, it is not necessary that Schlesien-Benzin revise the figures submitted to the Reichsamt.

It is advisable to use Method III for reports to the Reichsamt and to call their attention to the fact that the quantities have been analytically determined, from which, of course, a certain

Utilizable Phenols in % of Liq. Ph. Oil Yield and t/ann.

Method	I		II		III		IV		V		VI		VII
	%	t/ann.	%	t/ann.	%	t/ann.	%	t/ann.	%	t/ann.	%	t/ann	
Carbolic Acid	4.26	21800	2.52	13200	2.00	10500	4.21	22060	1.56	8150	2.09	10950	Carbolic Acid + Cresols abt. 30000
o-Cresol	1.12	5870	0.69	3620	0.52	2720	0.29	1530	0.42	2200	(2.05	10750	
m + p-Cresols	4.50	23600	2.71	14200	2.11	11050	1.10	5770	1.66	8680	(		
Xylenols	4.26	21800	2.52	13200	2.00	10500	1.03	5410	1.56	8150	3.04	15920	
Total	14.14	73070	8.44	44220	6.63	34770	6.63	34770	5.20	27180	7.18	37620	

loss in isolation must be deducted. In our report to the Reichsamt we deducted 20% from the computed values as reserve.

RESUME.

The discussion has shown the advisability of improving the methods of determining the phenols. In the future the phenols are to be determined in the following manner:

1). The stabilized total catch pot product will be divided into constituents to 325° and over 325°C. In the constituents to 325° the phenols will be analytically determined and isolated. The isolated phenols will be split up by distillation into phenols to 250° and phenols over 250°. The phenols to 250° will be fractionated into carbolic acid, o-cresols, m + p-cresols, xylenols and phenols 225-250°.

2). The gasoline to 200° is cut out of the same catch pot sample by double distillation in the same manner as this is done in the socalled "large test". The phenols in the gasoline will be determined and isolated. The isolated phenols will be fractionated the same as above.

The difference in phenols by 1.) and 2.) equals the phenols in the middle oil. The phenol content of the oil yield is then computed by means of these figures.

3.) To do this, we cannot use the Engler curve to compute a balancing oil yield distribution, as formerly, but it must be done by column splitting, as in 1.) and 2.) and as was also done in the large scale experiment in Stall 804. Of course, this change also applies to the distillation of the catch pot product, i.e., this must no longer be done according to the Engler curve, but by column splitting, as in 1.) and 2.).

This will guarantee that the removal of phenols from the liquid phase recycle oil will agree both quantitatively and qualitatively, with the analytical determination or the computation. The more accurately the cut at 200 °x) both quantitatively and qualitatively, in practical operations agrees with that of the column splitting in the laboratory, the easier it is to prevent such phenols being considered newly formed phenols, which in reality are only recycle products.

x) These conditions will become clearer and simpler as soon as Dr. Pier's suggestion not to take the critical cut at 200°, but at 250°, is adopted. It is impossible to compute the possibilities of phenol recovery by this method of operations, because no experimental data of any kind is available.

The Recovery of Phenols from L.T.C. Tars.
Report of Discussion Held At Ludwigshafen on the 5 February, 1943.

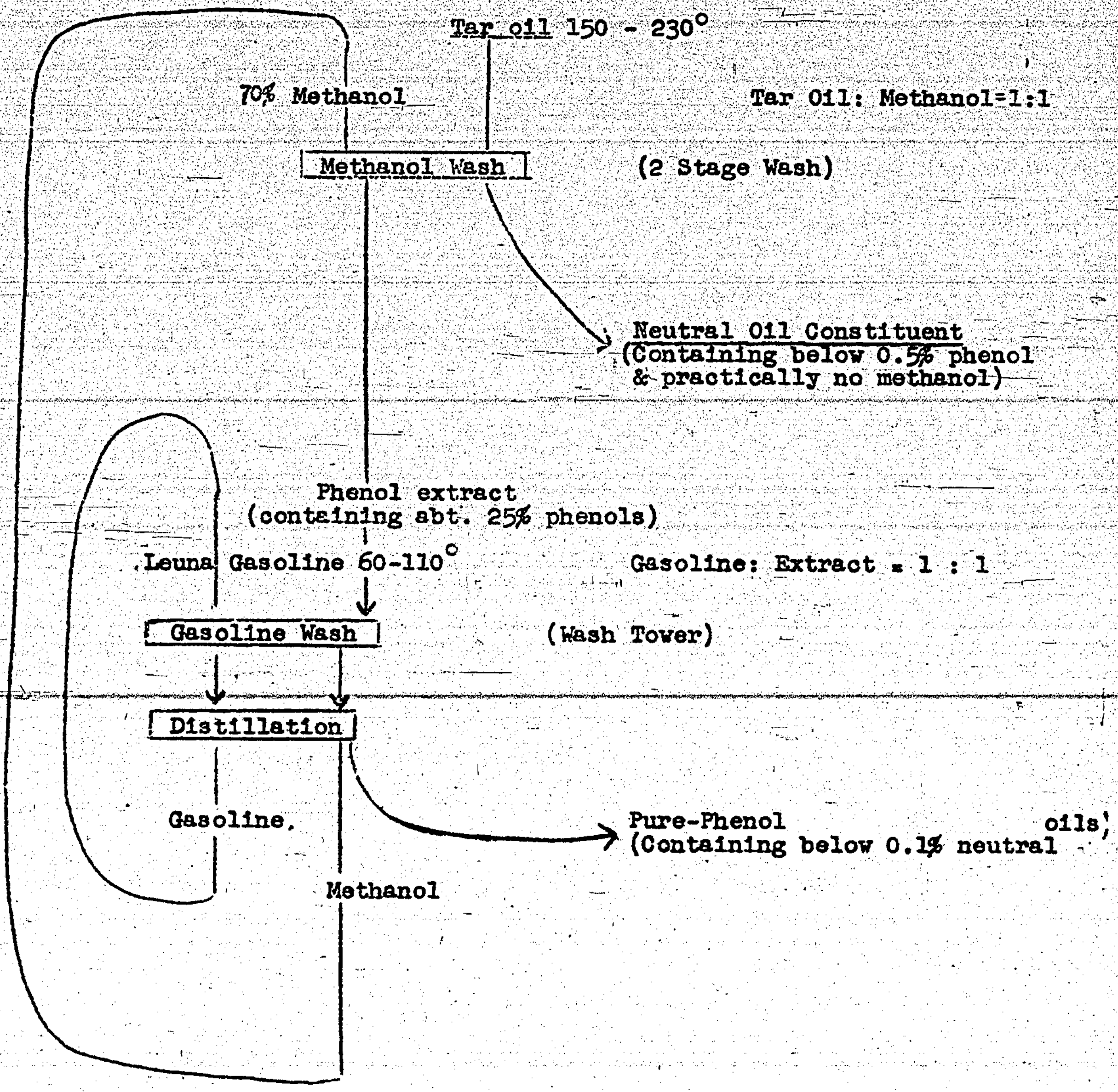
By Dr. Rank, Ludw. 8 February 1943.

Leverkusen has developed a process for the recovery of phenols from the various L.T.C. tars produced.

The first run fuel oil (boiling range 150-230°) is taken as the raw material for the recovery of phenols. The L.T.C. fuel oil cut off at about 400°C is adjusted to the proper flash point by the removal of this first run and still meets the required tests after the removal of these light constituents. The proportion of phenols in this fraction is very high at 50-60%, and the processing of this oil, therefore, offers a great incentive from the standpoint of economy of the process. The economy rises with the concentration of phenols in the feed stock. The cost of the phenol oil from L.T.C. is said to be 12 pf. (Compare bit. coal hydro. mi-oil at only abt. 26-28% phenol content abt. 18 Pf.). With the phenol oil from L.T.C. the processing costs are abt. 6-7 Pf, so that the finished phenols will cost abt. 20 Pf, which represents a favorable price position.

No lye is used for the process, only the work involved in distillation must be furnished.

Flow Diagram



10% of the oil is recycled. The process works at normal pressure and temperature, and is similar to the Riebeck spray water process. Advantages are the 2-stage wash and the combination with gasoline. (The Byk-Gulden process works with gasoline). The patent situation is not quite clear. The Leuna gasoline used is cut off at 110°, because gasoline cut off at 200° can not be separated from the methanol. With higher phenols the process does not work so well. At present only phenols-230° are further processed chemically, though it is hoped that higher phenols may also be utilized later.

Besides the phenols, a poor diesel oil is produced in the Leverkusen process. The cetane number of this diesel oil is 25 for heating surface tars and 36 for flushing gas tars. It can, therefore, be used only as a mixture component with good diesel oil.

According to Dr. Diericks, Upper Silesian tar contains 1.8% carbolic acid, 4.8% cresols and 12.5% xylenols, compared to 1.5% carbolic acid, 3.5% cresols, 2.2% xylenols and 7.2% higher phenols for bit. coal liquefaction.

In discussing the position of the L.T.C. program in general, it was brought out that the Reichsamt (Dr. Fahr) endeavored to increase the value of the L.T.C. tars by chemical processing, thereby making the L.T.C. process more economical. Because of the better quality of the tars, the heating surface processes were preferred at present, in certain cases without the combination with hydrogenation. Leverkusen was interested to find out Ludwigshafen's opinion on the development of the L.T.C. program, because the chemical work was to be based on a safe raw material. To which Dr. Pier and Dr. Bähr replied that the decisive factor in L.T.C. was and would be coke, because it was the principal product. The chemical processing of tars, because of more valuable products, could never be the determining factor or determine the choice of the process for the problem as a whole. The correct method was a combination of L.T.C. and hydrogenation, in which L.T.C. process with the greatest yield at lowest cost is to be preferred. (Flushing gas process, briquetting).

The flexibility of such a combination guarantees refined tars as well as the maximum yield in acceptable fuel oil. The recovery of chemically valuable substances in the combination of L.T.C.-hydrogenation could be effected just as well or better and simpler (asphalt-free oils) from the products of hydrogenation. The combination with anhydrated coal makes further production of L.T.C. coke suitable for blast furnace use possible. This problem is of particular importance to Lorraine and Upper Silesia. The production of vehicle coke by L.T.C. is of secondary importance.

The heating surface processes (Heizflächenverfahren) are considerably more expensive than the flushing gas processes. The difference permits the construction of a hydrogenation unit, producing a combination capable of meeting all conditions. By comparison, the heating surface processes are less flexible in respect to raw material, tar yield and quality.

Dr. Bähr offered a flow sheet of an L.T.C.-hydrogenation combination using 90 parts coal in L.T.C. and 10 in hydrogenation.

Leverkusen showed further interest in hydrogenation products, such as purest toluol, purest xylol, (o-m-p) carbazol and 6-ring naphthenes. Unsaturated would cause trouble for xylol, but not hydroaromatics. Carbazol is used as insecticide (Nirosan), in which there is a great scarcity. Anthracene residues are being used as raw material, wherein the anthracene is processed to soot. Annual carbazol requirements are 10,000 tons. (Carbazol could cost 70 Pf). About 0.1% gasoline is produced in bit. coal hydrogenation by distillation of the L.T.C. oil.'

The DHD feed gasoline from bit. coal contains about 50-60% naphthene over its entire boiling range, the 80° fraction still contains 40-50% naphthene.

In dephenolizing the waste waters at Gelsenberg by the phenol solvent process, Dr. Dierichs found more trouble with this waste water than with the waste waters at Pölitz and Leuna. The Gelsenberg water yields poor phenol (pollution with 12-14% neutral oil). The trouble must be caused by a substance (abt. 0.5 g/Ltr) which is soluble in water, can be removed with caustic soda, comes within the boiling range of phenol and possibly polymerizes in isolation (diolefin?). Ludwigshafen will try to identify this substance and explain its presence in Gelsenberg.

Gelsenberg has 4.5 g phenol in the waste water, Pölitz more, 6g. The gas generator waste water is added to it here. The economic limit of the phenol solvent process (Phenolsolvanverfahren) is at abt. 4.5g phenols per liter waste water.

Phenols in Tars and Coal Liquefaction

			Bit. Coal			Brown Coal		
			Coke Plant Tar	L.T.C. Tar	Coal Liquefaction	L.T.C. Tar Deuben	Coal Liquefaction Central German	Coal Liquefaction Rhein-land
Fr. Total Oil:	Carbolic Acid	%	0.3	0.1	1.5	0.4	1.0	1.6
	Cresols	%	0.6	3.7	3.5	1.0	2.9	3.1
	Xylenols	%	0.15	3.8	2.2	1.2	2.1	3.9
	Total	%	1.05	7.6	7.2	2.6	6.0	8.6
Fr. Coal		%	0.042	0.76	4.3	0.4	3.6	5.6

PHENOL RECOVERY IN COAL HYDROGENATION

By v. Hartmann, Ludw. 18 January, 1940.

The following table shows the phenol content of various brown and bituminous coals, based on pure coal and oil yield, as well as the distribution of the phenols.

The table shows that the gasoline and middle oil yield from each coal contains 5-8% low (utilizable) phenols irrespective of C and O content and operating method. This proportion is somewhat higher for bit. coal than for brown coal, due to the fact that the isolation of phenols for some brown coals is much more difficult than for bit. coals, because of the presence of larger quantities of ketones and aldehydes.

The still split-up of the phenols does not give exact values, so that no conclusions can be drawn from differences in composition appearing in the table. (Next page.)

PHENOLS IN HYDROGENATION WASTE WATER.

H. P. Research, Ludwigshafen, 29 May 1941

Phenols contained in 1 Ltr ~~atm~~:

Carbolic Acid,	5.0 g
o-Cresol,	0.5 g
m-Cresol,	0.5 g
p-Cresol,	0.5 g
Sym. Xylenol.	0.7 g
Total	7.2 g
Propionic Acid,	2.0 g
Sodium Sulfite,	6.0 g
Sodium Thiosulfate,	15.0 g
Ammonium Carbonate,	15.0 g

Phenol Yield in Coal Hydrogenation

Kind of Coal	Brown Coals				Bit. Coals				
No. District	K 148 East Elbe	K 149 East Elbe	K 48 Control German	K 1135 French	K 241 Ruhr	K 1013 Ruhr	K 1152 Saar	K 1090 Ruhr	K 1086 Ruhr
% C in Pure Coal	68.5	69.2	72.0	75.0	82.0	82.0	83.0	83.9	86.8
% O in Pure Coal	24.0	26.0	17.0	12.4	9.7	9.7	9.6	8.0	6.8
End Prod.	Gas. & M.Oil	Gas&M.O.	Gas&M.O.	Gas &M.O.	Gas&M.O.	Gas&M.O.	Gas&M.O.	Gas&M.O.	Gas. & Fuel Oil
H_2 Press.-atm.	250	250	250	250	250	250	600	600	600
H.P. Oil Yield-% of P.C.	63	65	67	67	69	72	71	72	71
Pract. Oil Yield. % of P.C	56	58	60	60	64	66	65	66	67
% Phenols in Gas & M. Oil based on P.C.	11.7	9.3	10.8	7.5	8.5	9.1	8.6	10.5	4.3
Compos. of Phenols									
Carbolic Acid	7.5	7.1	5.4	17.5	3	8.6	12.5	5.5	12.9
o-Cresol	4.6	5.8	6.7	4.9	34	8.0	8.1	9.7	5.1
m-u & p Cresol	8.3	15.2	9.4	18.6		8.7	21.4	19.8	16.9
Xylenols	1.6	18.4	11.8	12.6	18	15.4	14.4	13.0	15.7
Higher Phenols	75.0	53.6	66.6	46.4	45	59.3	43.6	52.0	49.4
Phenol $C_6 - C_8$ — % of Pract. Oil Yield Total	5.3	7.3	6.0	6.7	7.3	5.6	7.5	7.6	3.2
Carbolic Acid	1.6	1.1	1.0	2.2	0.4	1.2	1.7	0.9	0.8
o-Cresol	1.0	0.9	1.2	0.6	4.5	1.1	1.1	1.5	0.3
m-u & p-Cresol	1.7	2.4	1.7	2.3		1.2	2.8	3.1	1.1
Xylenols	1.0	2.9	2.1	1.6	2.4	2.1	1.9	2.1	1.0
Converter Date	451 Jan./Feb. 35	703 Mar.35	455 Jan.35	455 Feb./Mar. 38	451 May 34	703 Jan.35	411 May 39	451 Feb.38	411 May 38

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PERCENT OF COAL CONVERSION IN HYDROGENATION CONVERTERS AT SCHOLVEN

Abstract from Scholven report, 11. Nov. 1937.

By. E.E. Donath, March 3, 1947.

Ref: % Conversion in 3 consecutive coal converters at 300 atm. pressure.

The following data are available from the operation of Stall II at a time when optimum operating conditions had not been reached:

	Coal Paste 45% Fresh Coal	After Converter I (1 m Ø)	II (1.2 m Ø)	III (1.2 m Ø)	HOLD
% Total Solids	51.9	38.3	23.4	20.4	19.2
% Ash/solids	6.0	21.7	33.5	39.3	38.4
% Ash/total	3.14	8.32	7.85	8.02	7.37
Softening Point °C	9.5	52.5	20.5	13.5	29.0
% H_2O	0.6	-	-	-	-
% Na_2CO_3	0.5	0.3	0.3	0.3	0.33
% Total Alkali	1.6	5.1	3.6	3.3	3.1
Oil Sp. Grav./100°C	1.005	1.087	1.087	1.093	1.100
% Asphalts/Oil	6.4	21.2	19.8	18.7	18.5
% Coal Conversion (Approximate)	0	82	92	95.5	95

KCBraun
3/3/47

THE THEORY OF CAVIAR FORMATION.

By Schmidt, Ludw. 7. June, 1944.

In his letter of the 3. May, 1944, Dr. Moll of Rheinbraun expresses the opinion that the formation of caviar is due to the precipitation of the salts dissolved in the steam at 700 atm. and 450°C. He points to the work of Prof. Otto Fuchs and Dr. Spillner on "High Pressure Steam as Solvent" in "Chem. Fabrik", 1940, page 405, in which the solubility of various salts in steam at 70-225 atm. and 407°C was demonstrated and quantitatively determined.

In order to judge this opinion, it is necessary to clarify the magnitude and quantity relations by the following short calculation.

A coal converter of 9 m^3 becomes clogged in abt. 60 days with $CaCO_3$ deposits in the form of nodules, called caviar. This is about 8 tons of $CaCO_3$. During this time 13000 tons of coal paste with 26,000,000 m^3 H_2 (cold, at atmosph. pressure) are run thru it. The water contained in the coal paste and formed during the reaction equals a maximum of 15%.

For 13000 tons of coal paste this equals 2000 tons of water = 2,600,000 m^3 steam (cold, at atmosph. pressure). This gives a mol relation, based on H_2 of

$$\frac{2,600,000}{26,000,000 + 2,600,000} = 9 \text{ mol \% } H_2O$$

The partial pressure of water at 700 atm. total pressure in the converter = 63 atm. max. But, according to the articles referred to above, 1 kg steam @ 70 atm. and 407°C can dissolve only 8 mg NaCl; compare the following table:

Solubility of Salts in High Pressure Steam, by Spillner

Press. atm.	Temp.	mg NaCl/kg water	mg KCl/kg water
75	407°	8	
100	"	10	9
125	"	18	19
150	"	33	31
175	"	57	38
200	"	140	70
225	"	380	
250	"	570	220
275	"	1240	
280	"	(40000)	410

The measurements appear to have been made very carefully, so that there is no reason to doubt them. Fuchs and Spillner selected the salts of greatest solubility in steam for their measurements. It can, therefore, hardly be assumed that the solubility of Ca-salts lies in a higher order of magnitude.

Consequently, our 2000 tons of steam can dissolve only 16 kg of salt under the given conditions. They are, therefore, not in a position to separate 8000 kg of salts in passing thru the converter.

Dr. Moll was apparently mistaken in the magnitude of the steam pressure. The two articles mentioned also gave the values at the critical point (373°C and 227 atm), though this is outside of our conditions.

It appears much more probable that the calcium salts, such as Ca-salt of humic acid or the like, truly dissolve in the liquid oil-water mixture. (At a pressure of 70 atm some water is dissolved in oil, even above the critical conditions). By the conversion of the calcium salts to $CaCO_3$, their solubility is greatly reduced, and the $CaCO_3$ is separated about any nuclei.

This assumption gives us a way to eliminate the caviar formation, which should be tried out. Either must care be taken that the Ca enters the converter as an insoluble salt from the start or it must be combined with a strongly dissociated acid residual.

This might, for example, be done by converting the calcium salt into a sulfate or phosphate. This would eliminate a conversion combined with a radical change in solubility and no caviar would then probably be formed.

The fact that in heating coal paste with water under pressure the ash is in the watery phase in cooling, on which the patent application OZ. 14 018 by Dr. Rank and Dr. Grassl is based, does not deny that the salts can be dissolved in the liquid oil-water mixture phase. Since the second liquid phase, water with a small amount of oil, is formed only in cooling, the ash can be separated from the oil only after the formation of the second phase. The salts dissolved by heat in the watery oil phase (wasserhaltigen Ölphase) are taken over in its formation by the second oily water phase (ölhaltigen wasserphase.)

Summary

The opinion expressed by Dr. Moll that the formation of caviar is caused by the strong solubility of salt in high pressure steam is questioned. Steam has a partial-pressure of only 75 atm. maximum and the quantity of salts dissolved in it is very much smaller than the caviar formation.

It is assumed that organic calcium salts are truly dissolved in the liquid watery oil phase and that the separation is due to cabonate.

It is proposed to convert the calcium salts first into salts of strongly dissociated acids difficult to dissolve, such as $CaSO_4$ and $Ca_3\ (PO4)_2$.

THE OCCURENCE OF A WATERY PHASE IN THE COAL-LIQUID PHASE.

By Schneider, Ludw. 9. June, 1944

(A contribution to the theory of caviar formation).

It has been suspected that the solubility of inorganic salts in H_2O plays a part in the formation of caviar. However, if we compute a steam partial pressure from the molar H_2O concentration, this comes out so low that no solubility worth mentioning can be expected.

An appreciable solubility could, however, be expected if 100% steam were present at the prevailing pressure. This can actually happen in this way, that a "watery phase" is formed apart from the H_2-atmosphere. Such a watery phase has about the characteristics of pure steam at the given temperature and pressure, particularly its density and solvent property. It is in equilibrium with the H_2-atmosphere, which absorbs H_2O only to a limited extent.

An investigation by Laschakoff into the displacement of the methanol equilibrium by pressure pointed to such behavior. Laschakoff has calculated the volatility of the components of a methanol-H_2-CO mixture with the aid of a mixture rule formulated by Beattie. The figures calculated by him are remarkable, insofar as certain mixtures poor in methanol show a greater methanol-volatility than pure methanol and, conversely, mixtures poor in H_2 show a greater H_2-volatility than pure hydrogen. But this means, once we assume that the mixture rule used is at least qualitatively correct, that such mixtures are not stable, but that 2 phases are formed, a methanol phase and a permanent vapor phase, although Laschakoff did not draw this conclusion. This phenomenon occurs particularly at 250 and 300°C, but even at abt. 350°C. Thus, an example has been given in which 2 separate phases, a methanol phase and a permanent vapor phase can exist together, even at a temperature about 100°C above the critical point of the higher boiling substance.

A similar behavior may be expected with water, because water, like methanol, belongs to the abnormal substances and, even more than the latter, possesses powers of association. Investigations into the H_2-H_2O system under the conditions in question have apparently not been made. However, we may make the following appraisal:- The volatility of steam in a mixture with hydrogen probably is several times that of the partial pressure, in anology with methanol, while the volatility of 100% steam at 450° C and 400 atm is only 154 atm, calculated from the pV curve. Accordingly, in equilibrium with a steam phase, there is a phase rich in hydrogen, which contains roughly 20 mol % H_2O, or less, based on equal H_2O volatility in both phases. With an increase in pressure, the H_2O-content in the H_2-rich phase must drop considerably, so that the equilibrium concentration at 700 atm may very easily be below 9 mol %.

In the coal converter, the H_2O concentration, based on H_2, is abt. 9 mol %, so that the presence of a "watery phase" may well be taken into consideration. This "watery phase" has about the characteristics of 100% superheated steam under the given conditions. Its density, for example, at 450° C and 700 atm is around 0.3 - 0.4.

In the catch pot, therefore, the "watery phase" probably accumulates above the sludge.

Mr. Wiley

U. S. BUREAU OF MINES 355 T-256
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KCBraun
3/3/47

COMMENTS ON DRAWING R-1015

By Dr. Frese, March 3, 1947

1.) The main trouble on all high speed machines in German hydrogenation plants @ 700 atm was due to leaks in the stuffing box packings. Improvements were made by circulating oil cooling and good lubrication. After a stuffing box had been repaired it was necessary to run in the pump, starting at low pressure, before it was connected to the stall. For this purpose, a small container was located near the pump, but outside of the building, from which the pump feed was drawn and into which it was again discharged thru an expansion valve.

This procedure was also used to run in the pump for serving the high pressure stall. The starting up was so conducted that the pressure was gradually increased to operating pressure by throttling the expansion valve. As soon as the stall pressure was reached the valve to the stall was opened and the by-pass closed. This procedure avoided excess pressures in the lines between the pump and the stall valve.

The same thing applies to the water injection pumps. Because of the low compressibility of water, these caused far more trouble than the oil pumps. Many water injection pump bodies broke and many plants converted to low speed paste pumps in place of the high speed pumps.

In the plant at Louisiana high speed pumps are provided as follows: 2 light naphtha pumps, 2 wash oil pumps, 2 splitting feed injection pumps, and 5 water injection pumps. In direct comparison with conditions in Germany, it would be well to install 2 trial-run circulating systems and provide one container for all oil pumps and one for all water pumps. The fact that oils are inherently different would make little difference for this purpose. The lines could be held to small dimensions.

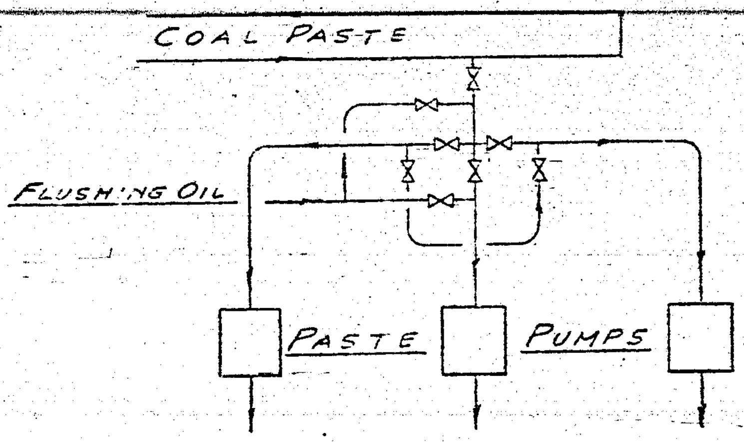
The expansion valve in the oil circulating system could be an ordinary long cone (plug type globe) valve, while that in the water circulating line would properly have to be a needle valve.

This method is also to be preferred for safety reasons. The newly packed pump gets a trial run under stall pressure conditions, wherein all defects can be ascertained beforehand. Even though each pump is protected by a check valve on the stall side, these valves can not be depended upon 100%. The possibility, therefore, exists that the stall might be decompressed into the pump room thru a leaky valve and defective stuffing box packing, which can be avoided by a trial run.

2.) The coal catch pot products in the cold catch pot often have a tendency to foam in the separation of gas and product, causing some of the product to foam over into the gas circulating line. This foam gets into the circulating gas wash and there also causes foaming of the washing liquid. The separators ahead of the gas circulating pumps usually do not separate the foam from the gas, and foam and liquid get into the gas circulating pumps and from these again to the stall, and so cause failure of the gas flow meters. A horizontal or inclined catch pot will better prevent this foam formation than a vertical one with small free liquid surface, as provided for at Louisiana.

3.) A connection for N_2 and flushing oil should be provided behind the valves in the H.O.L.D. line to the experimental letdown vessel, so that the line can be blown out and flushed after it has been shut off. This will prevent clogging the line.

4.) The supply of coal paste to the paste pumps must be arranged so that no coal can deposit in and clog up the reserve lines. The pump must always be ready to run. The flushing oil connection directly behind the shut-off valve should be arranged about as shown on the following sketch.



All branch lines off the coal paste line should be taken off the top of the line, not off the bottom. These details are not shown on the drawing.

5.) The satisfactory operation of the liquid level indicator on the hot catch pot is an important function in the hydrogenation process. The small supply of gas introduced to indicate the level hydrostatically must be held constant. Small drops of oil, such as lubricating oil from the make-up H_2 and condensed oil in the H_2 from the circulating line, often throttled the regulating valves and caused fluctuating level indications. For this reason, a small oil separator was installed just this side of the level indicator in the main supply line of all liquid phase stalls, to remove the oil in the gas. A large high pressure tube about 15" long would be good enough. To make sure that the make-up H_2 in Louisiana does not carry small drops of lubricating oil, in spite of the oil separator behind the compressor, it may be well to install this small separator, in view of the great importance of the catch pot level measurement.

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

T-257

KCBraun
3/4/47

INCUBATION TEMPERATURE

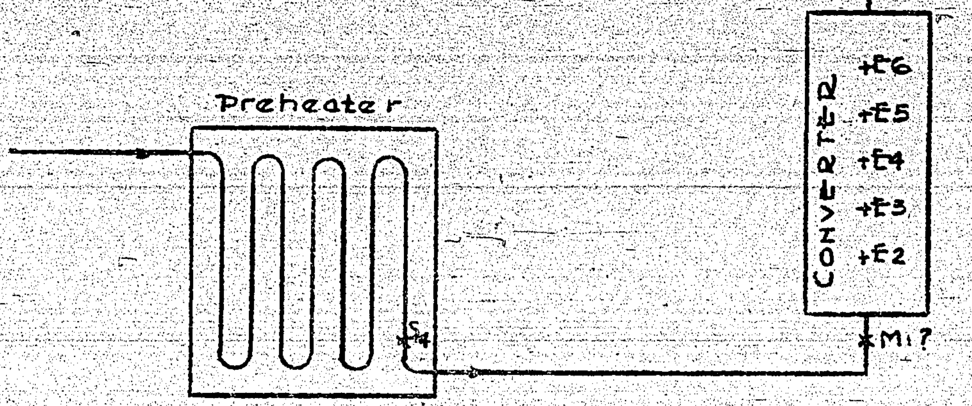
By Leonhardt & Hupfer, Ludwigshafen,
29. Dec. 1943.

Experiments in 10 liter converters in processing Heinitz coal (K 1197) to gasoline & middle oil, Heinitz coal & hot L.T.C. tar (P 1376), in the proportion of 1:0.2, to little heavy oil excess, and of P 1376 alone to heavy oil excess, indicated progressive temperatures as shown in table below and attached diagram.

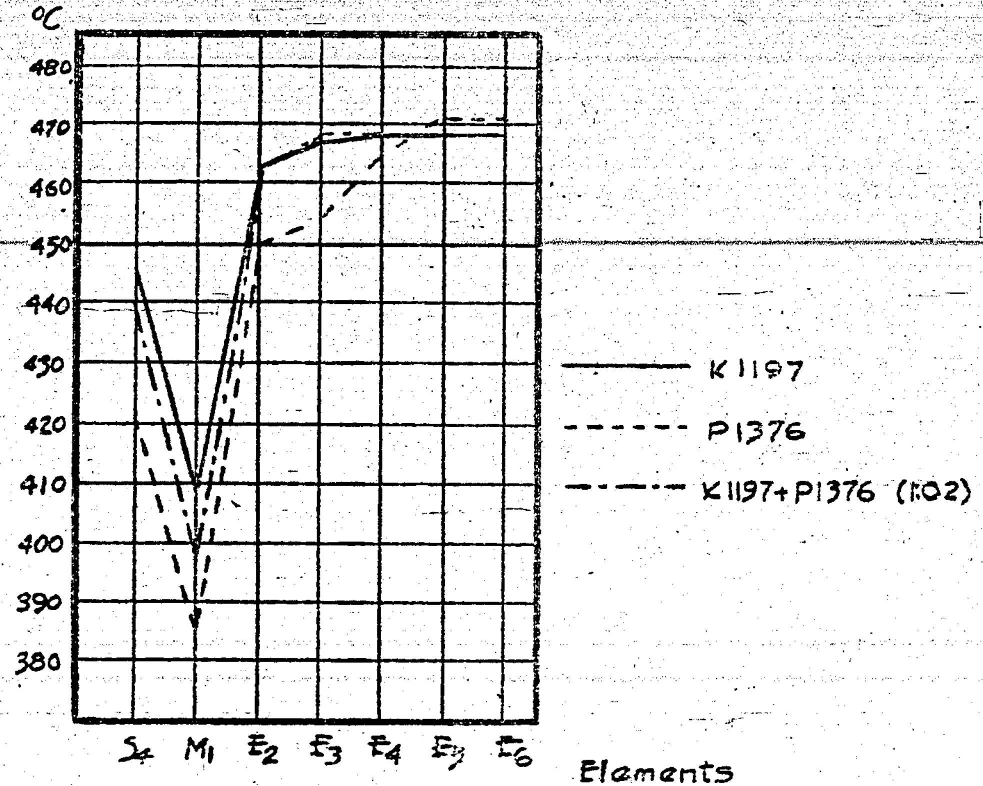
1.) In order to reach optimum working conditions in the converter, coal requires a preheater temperature 30° higher than hot L.T.C. tar. Tar is, therefore, much more reactive than coal.

2.) In the mixture of coal and tar the preheater temperature lies between the individual components, probably dependent upon the mixture proportion. Mixing a more reactive constituent with the coal will, therefore, permit lowering the preheater temperature and thereby relieve the preheater, presumably in the same degree as the mixture proportion.

		K 1197	K 1197 - P 1376	P 1376
Converter		451	451	411
Date, 1940		12 & 22. July	4. Aug.	10. & 14.July
Thruput		0.45	0.44	0.61
Element S_4	°C	447	440	420
" M_1	"	407	398	385
" E_2	"	463	463	450
" E_3	"	467	468	454
" E_4	"	468	468	465
" E_5	"	468	468	471
" E_6	"	468	468	471



INCUBATION TEMPERATURE



360 T-258

U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

K. C. BRAUN
3/7/47

HYDROGEN CONSUMPTION,
LOSS IN SOLUTION AND RECOVERY IN EXPANSION
(SEE ALSO T-260 and T-261)

H_2-Consumption at Nordstern.
By D. I. Schappert, Ludwigshafen, 17, Jan., 1942.

Nordstern records on H_2 consumption of the 700 atm. plant disclose the following interesting information:

1.) They indicate that the greater solubility of 700 atm. causes an additional requirement of 200 m^3/t of gasoline, or possibly a purely mechanical overflow. For this reason they have already provided a larger catchpot, 1000mm ø instead of 600mm ø, for the seventh coal stall. But, as shown in Table 2, the additional H_2 given off is only about 30 m^3/t gasoline, or only 1/10 of the deficiency sought.

2.) To be sure, the estimated best agree with the actual figures in washing. Nevertheless, these figures indicate the possibility of H_2 being carried over. The catchpots are connected to the 100 atm. vessel by 2 valves. If these valves leak, there is a constant short circuit, so that gas flows uncontrolled to the poor gas, which of course, is measured together with that drawn off by the expansion machines.

The fact that the estimated agrees with the actual washer off-gas, on the one hand, and the indicated gasification is lower than the estimated, on the other, leads to the conclusion that gas is carried over uncontrolled in washing. The H_2-analysis would show that.

3.) A small part of the excess, about 40 m^3/t gasoline, could also originate in the catchpot.

4.) It should also be pointed out here, that the L.T.C. (Schwelerei) has so far been disregarded. The estimate was based on a yield (Ausbeute) of 80%, which, however is not obtained in Nordstern, to my knowledge. The average yield in Scholven is 74%.

Now, an L.T.C. yield 6% below the estimated means an oil loss of 0.72 t/h with a centrifuge residue of 19.2 t/h. But to reproduce this quantity of oil, in the liquid phase only, requires 0.72 x 2000 = about 1400 m^3 H_2, or 41 m^3/t gasoline with an hourly production of 34.2 t of gasoline.

Summary:

It is reported that the solubility, including mechanical carryover, is greater by 200 m^3/t of gasoline than estimated. Of this amount, about 20-40 m^3 each may originate in the catchpot product and the hot catchpot.

Since there is a sump 2 m deep in the washer, a mechanical carry-over is hard to conceive. However, the possibility of a short circuit over a small sampling catchpot exists.

An L.T.C. yield of 6% less requires an added H_2-consumption of about 40 m^3/t of gasoline.

Table 1

H_2 CONSUMPTION IN NORDSTERN

		Triple Stall	Quadruple Stall
	Aviation Gasoline Production	34.2 t/h	
I.	Total C.P. Product	93.2 t/h	
	C.P. Product/Stall	13.3	17.7 t/h
	Feed Gas/Stall	abt. 40 000	50 000 m^3/h
	Catch Pot Ø		1 000 mm
	Catch Pot Length		6 m
	Residence time (1/3 full)	131	105 Sec.
II.	Wash Oil		225 m^3/h
	Total Gas		264 000 m^3/h
	Number of Washers		4
	Wash Oil/Washer		56 m^3/h
	Gas/Washer		66 000 m^3/h
	Washer-Ø		1 000 mm
	Washer Height		15 m
	Washer Sump		2 m
III.	Letdown		48.5 t/h
	Solids		30 %
	Oil free of solids		34 t/h
IV.	Centrifuge Residue		19.2 t/h
	Solids		38.0 %
	Oil free of solids		11.9 t/h
	Oil loss		20 %
	Oil loss		3.8 t/h

TABLE 2

			Estimated	Actual
I. Stall				
	Poor Gas	m^3/t gasoline	78	
	Rich Gas	m^3/t gasoline	50	
	Total	m^3/t gasoline	128	205
	H_2 Content	%	40	40
	H_2	m^3/t gasoline	50	80
II. Washer				
	Poor Gas	m^3/t gasoline	402	
	Rich Gas	m^3/t gasoline	170	
	Total	m^3/t gasoline	572	590
	H_2 Content	%	45	45
	H_2	m^3/t gasoline	260	270
III. Letdown (Abschlamm)				
	Total Gas	m^3/t gasoline	62	118
	H_2 Content	%	70	70
	H_2	m^3/t gasoline	42	85
IV. L.T.C. (Schwelerei)				
	Total Gas	m^3/t gasoline	34	
	H_2 Content	%	60	
	H_2	m^3/t gasoline	20	

H_2-CONSUMPTION IN BIT. COAL LIQUEFACTION TO AUTO GASOLINE AT GELSENBERG

Ludwigshafen, 10 Nov. 1941

Basis: Conversion (Abbau) 96%,
gasification 22.5%, based on oil recovery + gasification
H_2 dissolved in liquid phase 62.7% of gasified hydrocarbon,
86% C, 11 H available/100 C in oil recovery,
mean C of liquid phase gasification 1.9, 1.8% C as CO and
1.2% as CO_2.

Then the H_2-consumption/t auto gasoline=

Liquid Phase: 700 atm.		**Vapor Phase: 300 atm.**	
Chemically bound,	1420 m^3	Chemically bound	730 m^3
Dissolved,	470 m^3	Dissolved,	55 m^3
Loss,	75 m^3	Loss,	35 m^3
Total	1965 m^3	Total	820 m^3

Total

Chemically bound,	2150 m^3
Dissolved,	525 m^3
Loss,	110 m^3
Total	2785 m^3 @ 15° C & 735 mm

H_2-CONSUMPTION IN BIT. COAL LIQUEFACTION TO AUTO GASOLINE IN SCHOLVEN.

Ludwigshafen, 10 November, 1941

Liquid Phase: 300 Atm.	
Chemically bound,	1750 m^3
Dissolved,	220 m^3
Loss,	50 m^3
Total	2020 m^3

Vapor Phase: 300 atm.	
Chemically bound,	790 m^3
Dissolved,	45 m^3
Loss,	30 m^3
Total	865 m^3

TOTAL

Chemically bound,	2540 m^3
Dissolved,	265 m^3
Loss,	80 m^3
Total	2885 m^3 @ 15°C & 735 mm. or 2660 Nm^3 @ 0°C & 760 mm

In large scale experiments in Stall 804, in which Upper Silesian coal was liquified to gasoline + middle oil @ 700 atm, the following quantities of H_2 per ton of auto gasoline were consumed, based on the elementary analysis of the oil recovery and the hygases obtained, if the losses in residues processing are reduced to the same scale used in the hydrogenation works:

Liquid Phase:	
Chemically bound,	1540 m^3
Dissolved,	490 m^3
Loss, gas, (estim. 1% of circ.	100 m^3
Total	2130 m^3

Vapor Phase:		
Chemically bound,	670 m^3	(Calcul.)
Dissolved,	≈ 45 m^3	"
Loss,	30 m^3	"
Total	745 m^3	

TOTAL

Chemically bound,	2210 m^3
Dissolved,	535 m^3
Loss,	130 m^3
Total	2875 m^3 @ 15°C & 735 mm
	or 2640 Nm^3 @ 0°C & 760 mm

The larger H_2-consumption by dissolution @ 700 atm is, therefore, counter balanced by the smaller consumption of chemically sound H_2 @ 700 atm, because of lower gasification.

HEAT OF REACTION AND H_2-CONSUMPTION

By Dr. Donath, Ludwigshafen, 13 Feb. 1943

A) Quantities per Unit

	Heat of Reaction kcal / kg		H_2-Consumption m^3/t
1) **Liquid Phase**			
Bituminous Coal	200/Paste	650/M-Oil	1 850/M-Oil
Brown Coal-Leuna	120/Paste	550/M-Oil	1 450/M-Oil
Bit. Coal Crude Tar Residue	140/Injection	400/M-Oil	1 050/M-Oil
Brown Coal Tar Residue-Leuna	50/Injection	150/M-Oil	450/M-Oil
Petrol. Residue, H-poor	(100/Injection	270/M-Oil	700/M-Oil
Petrol. Residue, H-rich	(50/Injection	150/M-Oil	480/M-Oil
2) **Vapor Phase**			
a) Prehydrogenation			
Bit. Coal Mi-Oil	350/Injection	--	650/Injection
Br. Coal Mi-Oil-Leuna	300/Injection	--	550/Injection
b) 6434 Auto-Gasol.			
Bit. Coal Prehydr. Mi-Oil	140/Injection	230/Gasoline	375/Gasoline
Br. Coal Prehydro. Mi-Oil	140/Injection	220/Gasoline	370/Gasoline
Petrol Mi-Oil, AP. 40	--	300/Gasoline	600/Gasoline
Petrol Mi-Oil, AP. 70	--	150/Gasoline	300/Gasoline

B) Quantities per Stall

	m^3 Cat.	t Endprod./h	Consumption m^3 H_2/h	Heat-Reaction kcal/h
Ammonia Stall	2.2	1.5 - 2	3500-4600	1.1-1.5 10^6
Methanol Stall	3.5	5	14 000 1)	3.5 10^6
Bit. Coal Liq. Phase	54	10	18 500	6 10^6
4 Conv. 300 atm. Yield 0.18 t/m^3/h				
Bit. Coal Prehydr.	32	22	14 500	8 10^6
4 Conv. 300 atm Input 0.7 t/m^3/h				
Bit. Coal 6434 Benzination	32	19	7 000	4.5 10^6
4 Conv. 300 atm Yield 0.6 t/m^3/h				

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(1) CO + H_2

H_2-REQUIREMENT IN GELSENBERG AND H_2-LOSS IN SOLUTION

By Dr. Donath, Ludwigshafen, 18 Jan. 1942

In order to clarify the influence of the solubility values presently found in Gelsenberg compared to those used by Ludwigshafen, as gas balance was also calculated with Gelsenberg solubility values, see Table 3. The results of the calculation and the comparison are given in the condensed flow diagram, Table 4. In short, it is as follows:

(15°-735 mm/m³)	H_2 Content Catch Pot Outlet	m³ H_2 Dissolved		Addit. H_2 pr. t aviat. gasol. by solu in liq. phase compared to 1)
		Pr. ton Pure Coal	Pr. ton Aviat. Gasol.	
1) Lu Estimate 21 May 1, 1937	78.1%	149	285	0
2) Ditto with Gelsenberg Solubilities, Mar. 8, 1940	78.1%	199	380	95
3) Ditto with H_2 solubility for Wash Oil 0.085 from Gelsenberg Report, Sept 17, 1940	78.1%	176	335	50
4) Gelsenberg actual of Mar.8,1940	81.9%	300	570	285

The additional consumption by solution in the liquid phase of 2) or 3) compared to 1) of 50-100 m³/t aviation gasoline is due to the too low H_2-solubility assumed by Ludwigshafen. The additional consumption by solution of 4) against 2) of 570-380= 190 m³/t aviation gasoline is based on:

1) higher methane-content of liquid phase gasification (equal to about 60 m^3),

2) higher H_2-content of circulating gas (81.9 against 78.1), with correspondingly higher wash oil consumption (equal to about 115 m^3).

3) lower H_2-content of make-up gas, (97.7% against 97.7%, equal to about 15 m^3).

Gelsenberg Report 6592 of the 6, Jan. 1942, page 1, by comparison, shows solution losses of 333 m^3/t aviation gasoline, based on LU estimate. The actual solution losses are 50% higher, bottom page 3. 50% of 333 m^3 is 167 m^3, compared to added consumption of 200 m^3 shown on bottom of page 3. The solution losses in the liquid phase accordingly are actually 500-533 m^3/t, and, therefore, below the Gelsenberg value of the 8, March, 1940, top page 4. A subdivision of these losses is impossible, because no details are available.

TABLE 3

GAS BALANCE BASED ON BALANCE FROM LUDW. ESTIMATE AND GELSENBERG GAS SOLUBILITIES OF 8 MARCH, 1940

	Solubility in Letdown Oil	Dissolved in 0.54 t Letdown Oil	Solubility in C.P. Prod.	Dissolved in 1.26 t C.P. Prod.	In circulating gas after catch pot			Solubility in Wash Oil	Dissolved in 2.6 m³ wash oil	Total dissolved in letdown, C.P. Prod. & Wash Oil	Total dissolved based on Lu bal.
	Ltr/kg atm.	m^3	Ltr/kg atm	m^3	m^3	atm	%	Ltr/Ltr atm	m^3	m^3	m^3
H_2	0.06	17	0.08	52	3105	516	78.1	0.097	130	199	149
$CO + N_2$	0.18	6	0.10	8	370	61	9.3	0.11	17	31 } 84	25 } 84
C_1	0.182	5	0.16	10	290	48	7.3	0.305	38	53	59
C_2	0.197	2	0.35	7	120	20	3.0	0.701	37	46	46
C_3	0.114	0	0.72	9	68	11	1.7	1.18	34	43 } 120	38 } 104
C_4	0.474	1	2.31	12	24	4	0.6	1.72	18	31	20
Total		31		98	3977	660			274	403	337
% H_2 in Hygas based on 188 m^3 inerts.										51%	44%

TABLE 4

GELSENBERG GAS DIAGRAM

Products from Converter		Lu Estimate of 21, May, 1937	Lu Estimate recalculated with Gelsenberg solubilities	Gelsenberg actual of 8, March, 1940
Letdown Gas	m^3	38	31	40
	m^3 H_2	26	17	25
	% H_2	69	55	63
Catch Pot Gas	m^3	50	98	80
	m^3 H_2	25	52	51
	% H_2	50	53	64
Washer Gas	m^3	262	274	400
	m^3 H_2	98	130	225
	% H_2	37.5	47.5	56.5
Circulating Gas	% H_2	78.1	78.1	81.9

H_2-RECOVERY BY EXPANSION FROM 700 TO 300 ATM.

By Dr. Donath, Ludwigshafen, 23 Jan. 1942

The quantity of H_2 liberated in the expansion of the wash oil from 700 to 300 atm. was calculated from the Gelsenberg solubility values and the Ludwigshafen estimate. Table 5 shows the figures. A rough calculation of the gases dissolved in the catch pot product from the Gelsenberg solubility values results in the following values pr. ton of aviation gasoline:

% H_2 in Circul. gas, catch pot outlet.	78.1%	81.9%
m^3 H_2/t aviation gas dissolved in liq. phase	350	510
m^3 gas liberated in expansion from 700 to 300 atm.	160	250
of which m^3 H_2	130	210
% H_2	82.7	83.8
% N_2 + CO	7.0	5.9
% CH_4	8.4	9.0

The quantity of H_2 liberated at 300 atm., equal to 130-210 m^3/t aviation gasoline, is considerable, though its H_2-content of 83% is low for use as make-up gas. Expansion to 50 atm. and recovery from the poor gas in the Linde process might be more advantageous.

TABLE 5

SOLUBILITY IN LIQUID PHASE WASH OIL

Solubilities by H_2-Content at catch pot outlet	Gelsenberg 8, March 1940		Lu Estimate
	78.1%	81.9%	78.1%
m^3 Wash Oil/t Pure Coal	2.6	4.3	3.1
Dissolved in 1 m^3 Wash Oil			
m^3 Gas	101.4	91.5	81.0
m^3 H_2	50.0	52.2	31.6
Liberated in expansion from 700 to 300 atm.			
m^3 Gas	24	26.8	10.4
m^3 H_2	20	22	8.2
% H_2 of total dissolved in wash oil contained in 300 atm. expansion gas.	40	43	26
300 atm. Expansion Gas % H_2	82.7	83.8	78.5
% N_2 + CO	7.0	5.9	10.4
% CH_4	8.4	9.0	9.4
Static Gas Solubility 1/1 atm			
H_2	0.12		0.1
N_2 + CO	0.18		0.16
CH_4	0.49		0.5
C_2H_6	3.03		3.2
C_3H_8	11.5		12.9
C_4H_{10}	50		50

C. H. Material Balances

375

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U. S. BUREAU OF MINES
HYDRO. DEMON. PLANT DIV.

FLOW DIAGRAM R1014 FOR LOUISIANA. REMARKS ON THE BASIC FIGURES.

By Dr. Donath February 26, 1944.

The following yields based on 100 parts m.a.f.-coal were derived from the flow diagram.

A. Liquid Phase

Kg. C	Kg net H_2	
83.0	3.50	100 Kg. maf coal
↓	↓	↓ L. Ph. hydrogenation
43.9	4.60	50.1 Kg new formed oil.[x)]
1.2	0.24	1.4 Kg pentane
13.5	1.22	15.2 Kg oil loss (HOLD-recovery)
20.5	4.67	26.7 Kg gases (4.1 Wt. % CO, 3.7 CO_2, 17.1 C_1, 24.7 C_2, 29.7 C_3 and 20.7 C_4)
3.9	0.16	4.1 Kg organ. residue
		10.8 Kg liquor
83.0	10.89	108.3
	Δ= 7.39	

This corresponds to the following specific data:

95.9 % Coal conversion
24 % C as gas/ converted C
32 % C as gas/ C in new formed oil + gas
23 % Oil loss/ new formed oil + oil loss
0.97 Kg/ 1,h m.a.f. coal paste thruput and a space time yield of total oil 0.24 and of oil to 325°C of 0.18.

x) 9.7 Kg oil are subtracted, corresponding to 435.3 #/h M.O. as diluting oil in HOLD recovery.

B. Gas Phase

Kg. C	Kg. net H	
43.9	4.60	50.1 kg L.Ph. MO, naphtha and gasoline
↓	↓	↓ G. Ph. hydrogenation
35.8	5.30	41.1 kg gasoline incl. gas phase C_5 and 5% C_4
8.1	1.90	10.0 kg gases (11 Wt. % C_1, 23 C_2, 37 C_3 and 29 C_4)
		1.7 kg liquor
43.9	7.20 Δ = 2.60	52.8 [Δ = 2.60]

Gasoline yield including L. Ph. C_5 = 42.5 kg gasoline incl. all C_5 and 5 Wt. % C_4.

This corresponds to the following specific data:

19 % C as gas/C in gasoline (incl. C_4) and gas.
31 % gasoline/gasoline+recycle oil, about 0.44 kg/l,h. gasoline space time yield based on 1.5 kg/l.h total thruput.

C. Hydrogen Consumption

	(From R 1014)			Recalculated		
Kg/100 kg maf coal	L. Ph.	G. Ph.	Total	L.Ph.	G.Ph.	Total
Absorbed H_2	8.0	2.8	10.8	7.4	2.6	10.0
Dissolved H_2	2.0	0.5	2.5	--	--	--
Total H_2	10.0	3.3	13.3	--	--	--

The total hydrogen consumption according to R1014 of 13.3 kg is 1480 cbm x)/Tonne maf coal corresponding to 3500 cbm/Tonne gasoline.

D. Discussion

The figures for coal conversion and thruput and for the gas formation based on converted coal agree with the corresponding German data and are even on the safe side. The oil loss in the sludge recovery plant without kiln is high. This is the reason for the high hydrogen consumption per ton of gasoline. It should be possible to increase the amount of solids in the concentrate from the centrifuges by steam distillation from 40 to about 60%. This would reduce the

---

x) cbm at 0°C and 760 mm Hg.

oil losses from 15.2% to 6.8% and raise the gasoline yield from 42.5% to 48% on maf coal. This corresponds to a hydrogen consumption of abt. 3250 cbm/Tonne gasoline instead of 3500 cbm.

In the gas phase the concentration of gasoline in the hydrogenated product (31%) is lower than that obtained in experimental and plant scale work in Germany.

The following is a composition of the gases compared with German average figures.

	Liquid Phase		Gas Phase	
Wt % of C in the Gases	R 1014	German data[1]	R 1014	German Data
CO	2.5	5.0		--
CO_2	1.5	0.5		--
C_1	16.6	24.5	11	13
C_2	25.8	23.0	23	23
C_3	31.6	30.0	37	31
C_4	22.0	17.0	29[2]	33[2]

Furthermore the following remarks to the flow of materials may be made: It was German practice to add the catalyst to the coal; this seems important for the $FeSO_4$ which acts to neutralize the coal ash and thus form finely divided iron oxide. Mixing of Na_2S with heavy oils at temp. of 100°C was repeatedly the reason for an increase of the asphalt content in these oils and stoppages.

In case of failure of the hot recirculation pump a cooled pipe might be advisable to provide the injection of HOLD with the paste pumps.

The specific gravity of the L. Ph. wash oil is 0.928; an oil with lower gravity (e.g. G. Ph. recycle M.O.) has better solubility.

It is improbable that the make-up gas contains 0.5% CO_2 and no CO.

The solid content of the HOLD (34%) seems high.

The solid content of the centrifuged oil (5%) seems low; that of the concentrate (40%) high in comparison to the results obtained with Alfa Laval centrifuges in Germany.

1) For bit. coal with 83% C in maf coal.
2) Considering the butane contained in the gasoline.

For paste temperatures of about 210°F there is the danger of foaming if the water content of the coal rises occasionally.

The assumed analytical data for oils and gases give a correct carbon balance and 10% H_2 absorbed on maf-coal in comparison to 10.8% in diagram R 1014. The figures for the dissolved hydrogen will be recalculated later. It may be remarked that practice of Ludwigshafen was to assume about 1/2% loss in flow diagrams as well as of the liquid and vapor-phase oils and of the recirculated gases.

Dr. Donath

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U. S. BUREAU OF MINES
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KCBraun
3-10-47

SOLUBILITIES OF GASES @ 700 ATM. (LIQUID PHASE - BIT. COAL)

By Dr. Donath, Ludwigshafen, 11 March, 1942.

The attached Table I contains solubility values of the bituminous coal liquid phase for Gelsenberg, Scholven and Ludwigshafen Stall 804.

Table II shows the values to be submitted to the Upper Silesian Hydrogenation Works. They are largely based on the Gelsenberg figures.

The figures available to date indicate that the proportion of $H_2:N_2$, or the CH_4 solubility, is more favorable with a wash oil with a low specific gravity, e.g. 0.850. The use of such wash oils should be recommended to Gelsenberg.

It would be very helpful to the accuracy and completeness of these tables, if additional solubility values were available from industrial practice.

Table II.

Solubilities of Gases for the 700 Atm. Bit. Coal Liquid Phase.

Tentative values based on figures from Gelsenberg and Ludwigshafen, large scale experiments.

Solubility expressed in	1/kg atm.		1/l atm. Wash Oil	
Product	C.P. Product Spec. Grav. 1.0	Letdown	Spec. Grav. 0.95	0.85
H_2	0.08	0.06	0.09	0.12
CO_2	(1.0)	(0.1)	(1.0)	(1.2)
CO	0.17	0.2	0.19	0.21
N_2	0.085	0.10	0.10	0.16
C_1	0.17	0.18	0.27	0.43
C_2	0.47	0.2	0.76	1.25
C_3	1.1	0.3	1.2	2.0
C_4	1.9	0.4	1.8	6.0

Table I. — Solubilities

	Gasoline + Middle Oil Operation:					Fuel Oil Operation			
						Stall 804, Ludwigshafen			
							50% Fuel Oil Operation		
	Gelsenberg 8.3.40	Scholven 7.3.40	Computed Values Upp.Sil. 4.5.40 (C.)	Stall 804, 3.8	Im 4./5.8.	35% Fuel-Oil Operation 16.8.	2.9.	6.9	18.9
	A.	B.		D.	E.	F.	G.	H.	I.
Washoil: D @ 15°C	0.96	0.832		0.95		0.95			
m^3 Washoil/t Pure Coal				4.68	4.55	3.93	3.76	3.8	3.2
Solubility m^3/m^3/atm.	700 atm.	300 atm.	700 atm.		700 atm.				
H_2	0.093	0.12	0.09	0.062	0.067	0.075	0.064	0.058	0.061
H_2S		1.24							
CO_2	0.056		1.5	0.466	0.34	1.094	0.74		0.338
CO	0.199	—	0.13	0.113	0.104	0.135	0.105	0.108	0.114
N_2	0.105	0.177	0.11	0.079	0.066	0.091	0.073	0.0725	0.083
C_1	0.292	0.5	0.3	0.258	0.246	0.226	0.198	0.153	0.185
C_2	0.674	1.62	0.8	0.921	0.83	0.672	0.589	0.505	0.497
C_3	1.13	3.08	1.2	1.385	1.195	1.06	1.06	1.04	0.702
C_4	1.65	3.05	3.5	1.9	1.47	1.34	1.13	1.17	1.08
O.P. Product: D @ 15°C				1.0	1.0	0.992	0.986	0.984	0.986
Solubility m^3/t/atm.									
H_2	0.08	0.084	0.08	0.058	0.069	0.058	0.07	0.063	0.055
H_2S		1.69		0.147					0.61
CO_2	0.23		1.0	0.828	0.215	1.66	1.71	1.55	0.527
CO	0.17	—	0.1	0.111	0.098	0.083	0.096	0.097	0.089
N_2	0.085	0.0985	0.085	0.086	0.0797	0.064	0.081	0.095	0.056
C_1	0.16	0.254	0.16	0.182	0.187	0.161	0.198	0.182	0.169
C_2	0.35	0.659	0.35	0.71	0.566	0.599	0.621	0.492	0.613
C_3	0.72	2.11	0.72	1.21	1.49	1.080	1.200	1.030	1.320
C_4	2.31	3.83	2.3	1.20	1.187	1.65	1.325	1.169	1.56

Table I - Solubilities, Continued

	A.	B.	C.	D.	E.	F.	G.	H.	I.
Lattern Oil: D-100°C		1.093		1.096		1.112	1.114	1.106	
Solubility $m^3/t/atm.$									
H_2	0.06	0.081	0.06	0.187	0.234	0.133	0.142	0.152	0.122
H_2S									
CO_2	0.134	0.65	0.05	1.61	1.74	0.392	0.652	1.618	0.274
CO	0.287	—	0.08	0.215	0.257	0.116	0.112	0.142	0.124
N_2	0.103	0.074	0.07	0.152	0.191	0.124	0.138	0.161	0.103
C_1	0.182	0.16	0.14	0.261	0.336	0.167	0.172	0.20	0.145
C_2	0.197	0.172	0.2	0.447	0.470	0.250	0.250	0.244	0.215
C_3	0.114	0.19	0.3	0.573	0.587	0.321	0.316	0.369	0.278
C_4	0.474	0.379	0.4	0.477	0.548	0.356	0.360	0.408	0.427

KCBraun
3/10/47

RECOVERY OF HYDROGEN IN THE LINDE LIQUEFACTION PROCESS.

Marl/Ruhr, 25. May, 1945.

Coke Gas Apparatus.

The given data refer to decomposition of coke gas, for one unit, in Nm^3/h. ($0^{\circ}C$ & 760 mm).

Gas decomposition (quantities of gas entering and leaving):

100 m^3 washing N_2

9200 m^3 coke gas

450 m^3 raw C_2H_4
2220 m^3 residual gas
1680 m^3 CO-fraction
4880 m^3 H_2

The analytical composition of the products entering and leaving are given below:

	Coke Gas %	Raw C_2H_4 %	Resid. Gas %	CO-Fraction %	H_2-%
CO_2	0.01	0.0	0.0	0.0	0.0
C_2H_2	0.04	0.0	0.0	0.0	0.0
Olefins	1.0	19.0	0.03	0.0	0.0
CO	4.0	2.5	3.0	17.3	0.0
H_2	53.0	1.0	4.0	3.3	97.0
Hydrocarbons	26.0	70.0	86.0	8.9	0.0
N_2	15.0	7.0	6.0	69.5	2.9
O_2	0.3	0.3	0.3	0.8	0.1
	100	100	100	100	100

Each unit requires an N_2 circulating compressor, 630 KW power req'd, and an N_2 vacuum pump, 120 KW power req'd.

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ELECTROLYTIC HYDROGEN PRODUCTION

By Dr. Michael, Ludwigshafen, 24. June, 1944.

The decentralization of a hydrogenation plant is hampered principally by the fact that the method of producing gas used in large plants is less suitable for small plants, for various reasons. The question now, is, if an electrolytic H_2 production, as used in some countries for producing ammonia is better suitable for small plants.

By pressure electrolysis we can save water gas production, its cleaning, conversion and, at least partially, its compression. The gas tank would be eliminated, which would be valuable for camouflage. Undoubtedly, the gas plant would become very compact and take up only a fraction of the space normally required. In addition, pressure electrolysis would mean a saving in power, compared to pressureless electrolysis. The oxygen produced at the same time could be used for oxygen gasification of coal or coke.

Dr. G. Pfleiderer tells us that, to his knowledge, pressure electrolysis had not been developed sufficiently for practical application, apparently for lack of economic interest, though it might possibly have been developed secretly by interested electrical manufacturers. The saving of power, compared to pressureless electrolysis, was not very great, while the solubility of the gases in the electrolyte was appreciable, which made the separation of the gas more difficult.

The power required to produce 1 m^3 H_2 in pressureless electrolysis was 5 KWh. If we assume a hydrogenation process, in which 1 m^3 H_2 is used per kg product, a daily production of 100 tons, corresponding to an annual production of 35000 tons, would currently require 20000 KW for H_2 production alone. This is considerable, if we consider that 6 such plants would be required to produce 200,000 t/ann and that the present power situation is stressed to the utmost.

Dr. Pfleiderer thought that perhaps the familiar method of producing H_2 by the reduction of steam by iron and subsequent reduction of the latter by reducing gases, e.g. fuel gas, was applicable especially to smaller plants. He could not say, if such a plant was actually in operation on an industrial scale anywhere.

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3/11/47

HEAT TREATMENT & DELIVERY SPECIFICATION FOR N-10 MATERIAL

By D.I. Obenaus, Ludwigshafen, 1. April, 1941.

Defective heat treatment, such as too great and non-uniform hardness, insufficient toughness, or inherent stresses, are an important cause of all breaks in pipes and fittings, so that specifications for heat treatment must be reformulated to suit the equipment of the steel works.

Because operating conditions in the various steel works do not guarantee a satisfactory air hardening in a moving air stream, the question of oil hardening must be further examined. This is now possible, since the temperature at which the creep strength is to be calculated has been lowered from 560 to 520°C.

According to representations by the steel works, only Mannesmann can guarantee a satisfactory tempering in an air stream. All other works plead for an oil temper to obtain assured and uniform hardness.

The following specifications were agreed upon with representatives of the various suppliers of N-10 material:

I. Heat Treating Specifications.

A). Forgings and full-wall tubes.

1). Tempering temp.: 1020-1050°C.

2). Cooling: In the interval betw. 800 & 600°C at least 25°/min.
Required hardness, abt. 360 Brinell, limits depending upon the carbon content.

3). Annealing:

a) quenching in air, 740-780°C, 4 hrs.
required hardness, 180-220 Brinell.

b) quenching in oil, 720-760°C, 4 hrs.
required hardness, 200-240 Brinell.

B). Upset tubes.

1). Quench as above.

2). Anneal: 680-700°C, 6 hrs, 220-260 Brinell.

II. Acceptance & Delivery Specifications.

1). Minimum yield point is eliminated as acceptance value. Breaking and notched bar impact tests are essentially toughness tests to determine the characteristics of the heat.

2). Pieces already heat treated are delivered with the former hardnesses, insofar as they do not exceed 260 Brinell. When measuring the ball pressure hardness, particular care must be taken to remove the soft skin. With forgings and bored tubes, grinding off 2 mm will, as a rule, suffice. Rolled tubes must be ground deeper. For the present, the ball pressure hardness is to be measured on both ends and in the middle, at several points on the circumference, until more tests results are available. With forgings, several ball pressure points should be suitably distributed.

3). With rolled tubes, the decarbonized inner layer (soft skin) must be bored out. Measurements are still being made in the individual works to determine the thickness of the layer to be removed.

4). Further agreements between I.G. and the suppliers will be formulated in writing as quickly as possible in individual discussions with their respective representatives. These discussions will be held as soon as the first results of tests based on the new delivery specifications are available. The individual works will determine the terms agreeable to them with Ludwigshafen and Leuna.

The possibility of interchanging N10 and N8V will be further discussed within I.G, so that the heat treatment of N8V may be determined in the above named discussions with the suppliers.

TOM Reel 205
Frames 802-807

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THE INFLUENCE OF GRAIN OR PILL SIZE ON THE SPLITTING ACTIVITY OF CATALYST 5058.

By Peters & Trofimow, Ludwigshafen, 27.May,1943

Equal quantities of catalyst 5058 (45 g) of one and the same batch was put into eight 25 m^3 converters (10 mm high pressure tube) in 3 different pill sizes and 5 different grain sizes from crushed and screened pills.

The converters were first run for 8 days @ 250 atm. and 11 to 18 mV with a benzol-diisobutylene mixture. The results are not yet available in full, but they appear to verify the following statements.

Following these experiments, the splitting activity of the catalysts was investigated at 20 mV = 392°C with Bruchsal gas oil, to which 0.75% CS_2 had been added, at the rate of 2 kg/lit. cat./h and 2 m^3 H_2/kg.

The first tentative results are given in the following table:

Grain Size	Proportion of Catalyst Surface, 10 mm Pills = 1	Spec. Grav. of Product	% Gasol. to 150°C	% Gasol. to 180°C	Anil.Point Mi-Oil >150°
Grains:					
0.01 mm	1000	0.810	15	26	66.5
0.1 "	100	0.790	20	33	69.5
1.0 "	10	0.756	38	56	72.5
2-3 "	7	0.746	46	64	72.5
5-6 "	2	0.756	40	56	72.0
Pills:					
3 mm	3	0.770	31	45	71
6 "	2	0.780	23	39	71
10 "	1	0.784	27	38	70

The experiments show:

1). Grains between 2 and 3 mm show a distinct maximum splitting activity. Activity decreases very rapidly with smaller grains, while with coarser grains it decreases slowly.

2). Pills are less active than grains of about the same size. Activity drops slowly with increasing pill size.

3). Hydrogenation also is lower with the finest grains than with the coarser grains, as can be seen from the aniline points of the B-middle oils. Apparently hydrogenation is also slightly lower with pills than with grains of the same size.

In explanation of the results of these experiments, it must be assumed that the smallest grains have been damaged to a certain extent by a surface reaction. Two reactions enter into consideration:

a). Oxidation of the catalyst in the preparation of the grains from the pills, which would explain the more severe damage to the fine grains, due to the larger oxidizing surface.

b). Since the catalysts were previously used with a diisobutylene-benzol mixture for 8 days, damage of the catalyst by condensation reactions is conceivable, which could be a function of the outer surface.

The smaller activity of the pills, compared with the grains, could be explained by the greater resistance offered by the especially smooth and hard surface of the pills to diffusion. The declining splitting activity of grains and pills from 3 mm ø upwards points to diffusion processes, which distinctly influence the speed of catalytic splitting reaction of tungsten sulfide.

These results confirm the results of experiments made by Krönig, Schmitt and Schappert on March 5th and April 7, 1931, with various sizes of pills and pieces of pills, up to 15 mm, of catalyst 5058, when running Rhenania gas oil in 25 cm^3 and kilo converters.

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KCBraun
3/12/47

LIQUID PHASE CATALYSTS

Stettin-Pölitz, 22 May 1943

Inquiry addressed to Ludwigshafen.

We have found that the alkalinity of our Upper Silesian hydrogenation coal has risen in the course of the past months and now is about 30 g H_2SO_4/kg. The alkalinity per 1% ash is about double that of Ruhr coal (Scholven and Gelsenberg), and its absolute value, considering the neutralization of chlorine, is even much greater.

Comparative data are given below:

Dry Coal	Scholven	Gelsenberg	Pölitz
% Ash	4.3	3.8	5.8
Alkalinity (g H_2SO_4/kg)	abt. 10	8	32
Alkalinity/ 1% ash	2.5	2.1	5.5
% Cl	0.045	0.1	0.03

The alkalinity of Upper Silesian coal is, therefore, almost as great as that of brown coal, so that, just as for brown coal, corrosion of the preheater tubes, due to the chlorine content of our coal, may hardly be expected.

The method used to determine the alkalinity was agreed upon in common at one time, it is later described herein.

We should now like to know, if you still have any doubts about adding sulfigran, used for neutralizing chlorine, in the first converter, instead of before the preheater, in order to eliminate the influence of sulfigran on the formation of crusts in the preheater.

DETERMINING THE ALKALINITY OF COAL

By Wittmann, Lu, 9 April, 1943

Five grams of pulverized coal passing a 900 mesh screen are first mixed with 5 cc n/10 Nekal solution (wetting agent) and then with 50 cc n/10 H_2SO_4, boiled for ½ hr. and filtered. The coal is washed free of acid. In the filtrate the unused acid is retitrated against methyl orange with n/10 NaOH. With very strongly alkalinic coals possibly 100 cc n/10 H_2SO_4 are used.

Alkalinity values are given in grams H_2SO_4/kg dry coal.

The water content of the coal is determined by the xylol method.

The n/10 Nekal solution is prepared from pure Nekal by dilution with distilled water and so adjusted that 1 cc of the n/10 Nekal solution uses 1 cc n/10 NaOH (about 25 g pure Nekal to 1 liter solution).

IRON SULFATE CATALYST FOR BIT. COAL.

By Hupfer, Ludwigshafen, 9 April, 1943.

The following data have been determined from experiments to use iron sulfate as catalyst for the hydrogenation of bituminous coal:

1.) Iron sulfate is the most effective iron catalyst for bituminous coal hydrogenation at the present time. (In all experiments and practical applications to date the iron sulfate was added before preheating and thereby reduced to sulfide.) The greater activity is due to the better form of distribution. However, at lower pressure its activity is too low.

2.) The greatest effect is obtained by sprinkling the salt (solution-?) on the coal.

3.) The addition of soda to iron sulfate is harmful.

4.) The activity of iron sulfate is probably increased by the addition of sodium sulfide, which is necessary to neutralize the chlorine.

5.) The addition of chlorine to the coal soaked in iron sulfate gave no satisfactory results, even when the simultaneous addition of alkalies was omitted. Experiments in revolving autoclaves indicated that with iron sulfate - zinc oxalate + chlorine the same asphalt decomposition may be obtained as with tin oxalate and chlorine, though with greater gasification. This result requires verification in continuous experiments.

6.) Iron sulfide appears to have a tendency to form crusts in the preheater, particularly in the presence of soda and sodium sulfide.

7.) Bayermass treated with sulfuric acid (21.1%) or iron sulfate on Grude-coke (3%) is apparently just as effective in hydrogenation of bituminous coal as the iron catalyst combination of 1.2% iron sulfate + 1.5% Bayermass, presently used.

DETAILS OF EXPERIMENTS

I. Activity

1.) Without Additions.

After the suitability of iron as catalyst for bituminous coal liquefaction became known, it seemed proper to give it increased activity by especially fine distribution by sprinkling it in the form of a salt soluble in water on the coal before drying. Iron sulfate, $FeSO_4 . 7 H_2O$, received first consideration because of its cheapness and availability in large quantities.

Considerably better results in asphalt decomposition and gasification were obtained in auto-claves with Gelsenberg coal @ 600 atm with 1.2% iron sulfate than with 1.2% spent Luxmass. At 350 atm a better gasification was obtained with sprinkled-on sulfate than with spent Luxmass, other things being equal. Furthermore, 1.85% sulfate + 0.2% Na_2S proved to be better in asphalt and splitting than 3% Luxmass + 0.2% Na_2S, although gasification was correspondingly lower with Luxmass. The superiority of iron sulfate over Luxmass in equal concentration was also shown with Gardannelignite (K 1065) with respect to asphalt reduction and gasification.

The greater effectiveness of 1% sulfate compared to 1% sulfured Luxmass on bituminous coal, with respect to yield, gasification and asphalt, was verified in a 1.5 liter convertor, partly with 0.3% Na_2S.

With quantities below 1.5% sulfate without Luxmass, however, the recycle oil became too viscous in the course of the experiment, so that the preheater became clogged. On the other hand, continuous operation seemed possible with 3.7% sulfate + 0.8% sulfigran.

It could be supposed that the advantage of iron sulfate over oxidic or sulfidic iron catalysts was based on the fact that the sulfate partially neutralizes the alkalinic coal ash, giving greater effectiveness to the chlorine in the coal. In the meantime, iron sulfate also proved to be better than sulfured Luxmass with practically chlorine-free Silesian coal in autoclaves. The greater effectiveness is, therefore, due to better distribution. Sprinkled-on ferric sulfate split less than ferrous sulfate.

2.) Influence of the Form of Addition.

Iron sulfate develops its greatest effectiveness when sprinkled on the coal. Adding the dry salt to the paste caused deterioration in asphalt decomposition in the revolving autoclave; in another experiment @ 350 atm the conversion dropped as well; and in still another the conversion and gasification was less favorable. With Gelsenberg coal in a 10 liter converter gasification rose from 21.7 to 23.9%.

A further possibility to deposit iron on the coal by means of iron sulfate, and thereby reduce the harmful ash constituents of the coal at the same time, exists in the intensive treatment of the coal with a sulfate solution for the purpose of exchanging bases. With Silesian coal 1115, 96.5% of the CaO and 83% of the MgO could be dissolved in this manner. At the same time the iron content rose to 4 times the original. Apparently, humic acids were also leached out by this treatment, because the C-content rose from 83.7 to 85% and the oxygen content decreased. The volatile constituents were reduced from 35.5 to 32.8%. In the revolving auto-clave, coal prepared in this manner produced more new products-325° and a better asphalt value than untreated coal, although the gasification was also greater.

3.) Addition of Luxmass.

The exclusive use of iron sulfate as catalyst for bituminous coal liquefaction may be frustrated by the large quantities required and the price. On the other hand, it has been possible to obtain satisfactory results by the addition of iron oxide or sulfide in the form of Luxmass or the similarly acting Bayermass to a quantity of sulfate within bearable cost limits. The addition of 15% Luxmass to 1.8% sulfate improved the asphalt decomposition, while gasification rose correspondingly.

4.) Addition of Soda.

Leuna proposed to use a combination of iron sulfate and soda as catalyst for bituminous coal liquefaction, which had proved satisfactory in brown coal hydrogenation. Tests conducted at Ludwigshafen clearly showed, however, that this combination was entirely unsuitable.

Sulfate and soda was compared with MoO_3 + Cl in a rotating auto-clave. The result was almost twice as much asphalt and a lower conversion, 92 against 98. A further experiment clearly showed the harmful influence of soda. An addition of 0.1% to 1.85% iron sulfate @ 600 atm (K1090) decreased the splitting considerably and increased the asphalt yield from 7 to 9.8%.

Similar results were obtained with sulfate and soda in a 1.1 liter converter, and with double the quantity of catalyst they were even worse.

In a 10 liter converter also, sulfate and soda on Hibernia coal @ 250 atm, brought results worse in every respect than tin oxide + Cl. Only the color of the sludge was alike.

Like all poor catalysts, this catalyst combination goes to extremes, with very low yield, i.e, towards the asphalts and gas, gasificating reaching the extremely high figure of 30.4%. Hydrogenation of the heavy oil was somewhat worse than with tin oxide - Cl. In the middle oil the unsaturated were also a little higher. The vacuum residue was larger in proportion to the asphalts. Increasing the temperature merely increased the yield, but could not compensate for the inferior activity of the catalyst. The poor reduction ability of this catalyst combination also manifested itself in increased phenols in the middle oil.

5.) Addition of Sodium Sulfide.

Substituting sodium sulfide for soda proved advantageous in experiments with and without Cl. In a rotating auto-clave (@ 250 atm + Cl) a slight improvement in conversion and in asphalt decomposition was obtained. Similar results were obtained without Cl.

Experiments in a 10 liter converter @ 250 atm with Hibernia coal and 6.6% iron sulfate + soda indicated a certain increase in conversion and hydrogenation of the heavy oil, when half of the soda (2.6%) was replaced by Na_2S. Taken as a whole, this combination was also unsatisfactory.

On the other hand, experiments in auto-claves showed an activating effect of sodium sulfide on iron sulfate without soda. An addition of 0.2% Na_2S to k 1090 with 1.85% sulfate improved the gasification as well as the asphalt value.

An addition of 0.3% sulfigran to bituminous coal with the iron catalyst combination of 1.2% iron sulfate + 1.5% Luxmass or Bayermass, which appeared to be necessary to neutralize the chlorine liberated from the coal as HCl, may, therefore, also be considered catalytically favorable.

6.) Addition of other Sulfides.

The addition of other metal sulfides instead of Na_2S showed less favorable results in the autoclave. Ammonium sulfide @ 250 atm acted better on iron sulfate than Na_2S.

7.) Addition of Chlorine

Iron sulfate could not replace tin oxalate in the processing of bituminous coal with chlorine.

This was shown in the 1.1 liter converter @ 250 atm. with Brassert coal, and in the autoclave about equally unfavorable results were obtained, with and without chlorine, compared to molybdenum. A further experiment in bituminous coal liquefaction with sprinkled-on sulfate (1.3% Fe) and on addition of 0.75% Cl not only showed unfavorable results in asphalt and gasification against tin oxalate + Cl, but also compared to a smaller quantity of spent gas cleaning substance (0.65% Fe) + Chlorine.

8.) Addition of Chlorine Besides Other Additions.

The effectiveness of the combination of iron sulfate + soda or sodium sulfide cannot be improved by adding chlorine, because the chlorine is rapidly absorbed by the alkaline constituents.

The ineffectiveness of chlorine additions was demonstrated in a rotating autoclave @ 250 atm. and in a 1 liter recirculating converter. Another experiment in a 100 liter converter with Brassert bituminous coal and iron sulfate, soda and chlorine, of which 50% was injected directly into the converter, gave negative results. In particiular, the asphalt content, (asphalt spiegel) rose so much that it appreciably deteriorated the centrifugal effect. Insufficient reduction also manifested itself in a rise in the asphalt content of the oil produced.

When 2/3 of the tin oxalate was replaced by 1.2% iron sulfate, leaving 0.2% tin oxalate besides the iron sulfate, the greater splitting activity of iron compared to tin was clearly manifest, but also the lower hydrogenation effect in conversion, asphalt and gasification. Iron powder, in place of the residual 0.02%

tin oxalate, produced a still lower asphalt reduction. Iron sulfide was possibly a little better than iron powder. On the other hand, the 0.02% tin oxalate could be replaced by 0.3% zinc oxalate without further deterioration. These experiments were made in autoclaves with Zweckel coal at low pressure.

At a slightly higher temperature even somewhat better asphalt values were obtained with this combination, 1.2% iron sulfate + 0.3% zinc oxalate + Cl, than with tin oxalate and chlorine. The substitution of zinc oxalate by 0.6% zinc dust produced about the same asphalt decomposition and new products as tin oxalate. Iron sulfide and antimony pentasulfide were not as good. With respect to gasification all combinations with iron sulfate were inferior to tin oxalate.

B. Behavior in Preheating.

A number of observations lead us to believe that the iron sulfate sprinkled on the coal or added to the paste acts unfavorably in the presence of alkalinically and sulfidically acting components.

In the 10 liter converter experiments with iron sulfate and soda, or soda + sodium sulfide, the preheater coal lost 34% of its volume. The crust consisted of 98.5% benzol solids containing 82% FeS and 9% C. Since only iron-free catalysts had been used before, the crust must have been largely formed in the short operating period of 2 weeks. Its continuous formation manifested itself in the temperature measurements during the experiments.

No positive conclusions about FeS encrustations in the preheater can be drawn from the short 100 liter converter experiments with iron sulfate, soda and chlorine. Compared to tin oxalate and molybdenum, the pressure difference in the preheater was much higher and the heat transmission poorer. By injecting the chlorine into the converter, instead of into the paste, the pressure difference was apparently temporarily improved, so that it appears not improbable that the combined effect of chlorine and iron in the preheater leads to polymerizations. Even without chlorine additions to the paste the pressure difference in the preheater rose so much that it had to be shut off, possibly because of the insufficient activity of the catalyst combination used.

In bituminous coal hydrogenation @ 600 atm. with the iron catalyst combination of 1.2% sprinkled-on sulfate + 1.5% Luxmass developed meanwhile and varying additions of sodium sulfides considerable partial encrustations of the coil of the 10 liter converter was observed. These crusts consisted largely of Fe and S. Similar crusts were observed regularly in all experiments to date but it was thought that the higher velocity of flow in the much larger industrial equipment would largely prevent such crust formations.

It has been repeatedly stated that the addition of sodium sulfide has an unfavorable effect on the preheater. The particularly heavy encrustation mentioned above occurred when using a very large quantity (0.8%) of Na_2S. In further experiments a considerable deterioration in heat transmission was observed when Na_2S was added. At the time this was believed at least partly due to polymerizations in the pasting oil, such as may be observed under the microscope without pressure, caused by the apparently partially oxidized Na_2S. In other experiments with K-1090 by Dr. Krönig, not yet evaluated, the addition of sodium sulfide behind the preheater seemed to check the further deterioration of the heat transmission.

C. Substitute for Iron Sulfate.

In view of the experiences in the preheater and difficulties of its supply, various substitutes for iron sulfate catalysts have been sought.

According to results in autoclaves the usual iron combination of 1.2% iron sulfate + 1.5% Bayermass can be replaced by an equivalent amount (2.1%) of Bayermass, which has been treated with an amount of 60% sulfuric acid proportionate to the iron sulfate. The use of Luxmass prepared in a similar manner resulted in a less favorable asphalt decomposition.

The deposition of iron sulfide on the preheater tube walls, could possibly be prevented by adding a carrier to the iron sulfate, which would not be changed during the heating process. An autoclave experiment with 3% Grude (Winkler gas prod. coke), containing 20% iron sulfate sprinkled on, instead of the usual amount of sulfate + Bayermass, resulted in equal asphalt decomposition and lower gasification in the hydrogenation of Gelsenberg bituminous coal. The conversion was somewhat lower. By adding titanium-dioxide a further improvement in the asphalt value was obtained, though this could not be verified in 10 liter converter experiments.