FILM STUDY GROUP

SUBJECT INDEX AND REPORT

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Prepared by

PHILLIPS PETROLEUM CO.

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PHILLIPS PETROLEUM COMPANY

RESEARCH DEPARTMENT

BARTLESVILLE, OKLAHOMA

REPORT ON

UNITED STATES TECHNICAL OIL

MISSION MICROFILM REEL 76

Compiled and Edited

by

Technical Information Section

PHILLIPS PETROLEUM COMPANY - RESEARCH DEPARTMENT - REPORT 584-46R

UNITED STATES TECHNICAL OIL
MISSION MICROFILM REEL 76

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Compiled and Edited

by

Technical Information Section

SUMMARY

This report on United States Technical Oil Mission Microfilm Reel No. 76 is a part of the American Petroleum Institute - Technical Oil Mission program devoted to the indexing, abstracting, and translation of the German literature collected by the Technical Oil Mission. Its distribution outside Phillips Petroleum Company includes all companies participating in the program and the Library of Congress. From the Library of Congress, photostat or microfilm duplication is available to anyone making a request.

The general content of this reel is concerned with the Pölitz Hydrogenation Works. Specific items of interest include operating data on the DHD process and the CV2b process, thermal cracking in carbonization ovens and data on gasification.

JAN 2 2 1947

Albert E. Miller, Chairman
API-TOM Study Group

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POLITZ HYDROGENATION WORKS

Technical Oil Mission Microfilm Reel No. 76

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Report 584-46R

AN ABSTRACT OF A DISCUSSION OF HIGH PRESSURE VALVES AND FITTINGS

Frames 13-28

Ludwigshafen, Dec. 10 & 11, 1942

Participants in the discussion:

Chairman: Raeithel

Werk Böhlen:	Graichen, Hartmann
" Brüx	Kutter
" Gelsenberg:	Beutner, Dr. Rohnalter
" Heydebreck:	Weber
" Hüls:	Meyer
" Linz:	Drechsler
" Leuna:	Cron, Maier, Richter, von Rossum
" Ludwigshafen:	Bahlinger, Bolbach, Berger, Bernhard, Dr. Class, Dr. Dinkler, Dr. Generlich, Hahn, Dr. Kümpfe, Dr. Kautz, Koch, Mayer, Muller, Rader, Raichle, Rick, Römer, Schanzenbächer, Schappert, Schaurer, Schmidt, Wagner, Weid
" Magdeburg:	Bielek
" Pölitz:	Dr. Benke, Zimmermann
" Schkopau:	Riedl
" Scholven:	Eisenbraun, Gehb, Koetz
" Wesseling:	Dr. Erdmann, Dr. Neubauer
" Zeitz:	Nerge

NOTE: Much of this report is very difficult, and some of it impossible, to read.

Materials of Construction.

Raeithel: The high pressure materials may be subdivided into 4 main groups:

a) Materials subject to stress only, that is, which do not come in contact with the operating product. These are S1 and the K-materials. Basic color: white.

b) Materials coming in contact with the cold product and which must be leak-proof. These are S2 and S3. Basic color: green.

c) Materials coming in contact with the hot product and which must be leak-proof. These are the N-materials. Basic color: blue.

d) Materials, high temperature and corrosion-proof. These are the R-materials. Basic color: yellow.

The individual materials within these main groups again differentiate between operating pressure, temperature and dimensions. The necessity for saving insufficiently available alloyed constituents requires that we introduce new materials of construction. In W. Norm-Beiblatt 10-00 the new materials are compared with the old. The old materials are marked with simple longitudinal stripes on the basic color, the new with double stripes. The exact separation of the individual materials is extremely important.

(The author here discusses the marking and stamping of the various materials for identification in the warehouse.)

Bahlinger: All of the high pressure fitting parts of special materials, such as S3, as well as the K- and N- materials, are heat treated after forging or after delivery, if not already heat treated. The temperatures for the heating up and hardening as well as the desired degree of hardness are specified in specifications for heat treating. Changes in the composition of the steels must also be taken into consideration.

(The author here discusses heat treating temperatures and the control of heat treated materials in the workshops and the difficulties in obtaining suitable materials, government restrictions, etc. of no immediate interest to us).

Dr. Class: We try to control the use of materials in the simplest possible manner. Of particular importance is the assurance, for example, that a non-hydrogen proof steel is not used where a hydrogen-proof steel is essential. Since our hydrogen-proof steels are based on their chromium content and the chromium content can easily be checked by the spot test, the spot test for chromium must be considered the most important, and which can also be made without difficulty at the building site. Besides chromium content from about 1% on, molybdenum contents can also be recognized by the spot test. Vanadium contents however cannot be checked by a simple spot test.

The spot test, together with the hardness test, lends itself well for the individual testing of a large number of pieces. Ludwigshafen has largely followed this procedure.

……

All parts of the chromium alloyed K- and N- steels are spot tested for chromium when they are not individually analyzed. Parts of the molybdenum alloyed N9 material are also spot tested for molybdenum content.

……

Dr. Kämpfe discusses machine shop practices and high pressure testing, for example;

Pressure tests are made according to the following specifications:

	At 325 Atms.	At 700 Atms.
For all fittings pressure-tested with middle oil:		
a) Cold parts	430 atms. test pressure	910 atms. test pressure
b) Hot parts	500 atms. test pressure	910 atms. test pressure
For all assembled fittings such as valves, gates, liquid level gauges, also seal test of the entire housing as well as the shut-off parts, with nitrogen	325 atms. test pressure	700 atms. test pressure

In the nitrogen test, all parts are totally immersed in middle oil and held under pressure for a prescribed period of time, whereby any rising bubbles and a drop in the manometer pressure may be observed. In spite of this, it may happen that leaks may appear later in operation, because pores in the material often show themselves only after many hours.

(Raeithel here discusses identification of parts and the necessity for repeated tests.)

Valve Construction:

Raeithel: The former valves with inside thread have almost all been replaced by valves with outside thread. These have been repeatedly changed and improved in the course of years. The introduction of the 700 atm. stage forced us to adopt a new construction in order to avoid too heavy valves, very difficult to operate. This new construction has proved itself very successfully in operation. It was therefore advisable to reconstruct the 325 atm. valves in the same manner. The advantages of the new construction are the smaller space required, the lower weight and considerably smaller torque required to operate the valve stems. The changes consist above all in the decrease of the stem diameter, the changed location of the connection between valve seat and stem to the outside, the shortening of the stuffing boxes and the valve hubs, the drifting -(?) (Eindornen) of valve seats, and an increased hardness of the valve seat and disk. Of particular importance also is the saving in alloyed materials by welding on the valve stems and seats which were formerly made of solid alloyed materials.

Dr. Kautz: The welded on valve stems and seats must fulfill the following requirements:

1) The sealing surfaces on valve seat and disk must be corrosion-proof.

2) The hardness of the disk must be as high as possible.

3) The valve seat should be somewhat softer than the disk. The welded on layer must also be able to endure the deformation caused by the drifting (Eindornen). (Eindornen seems to imply a pressure fitting, possibly with a revolving plug (Dorn)).

(Parts of report impossible to read but seems to treat of welding procedures.)

The former valve seats of V2A were held in place by threaded rings. Later the smaller valve seats were drifted in (Eindornen), the larger were rolled in. Since 1939 valve seats to and including the 90 mm size are drifted in (Eindornen). Valve seats for the 120 mm size are rolled in, now as before. At the present time experiments are under way to weld in the valve seats. Extensive tests have shown that a better hold may be obtained if the valve seats to be drifted in (Eindornen) have protruding teeth on their outer jacket instead of the grooves formerly turned into the housing. Exact specifications for the drifting (Eindornen) exist, indicating drifting tool size (Dornzahl), drifting tool sequence (Dornstufung), and press pressures. The inspector will check the specified widening of the valve bore.

Packing of Stuffing Boxes: Precise specifications for this operation are available. At the present time only Burgmann-cord is used for packing. The rings for the individual valve sizes are cut to predetermined length, inserted into the box with a tap and pressed in on a press with predetermined pressure. The inner side of the rings is greased before insertion with a mixture of hot-steam cylinder oil and flaked graphite.

The subject of stuffing box packing was discussed at length, inasmuch as no more Burgmann-cord was available and had to be replaced by a substitute. In reports on experiences with metal packing of hard lead (for oxygen), aluminum, hydronalium and carbonyl-iron. Very good results have been obtained with aluminum packing in over-lapping (possibly V shaped) form. Valves packed in this manner were sent to a number of hydrogenation works for trial. It should be pointed out here that such stuffing boxes may be only lightly drawn. Sizes deemed suitable for tests were 24 mm for 700 atms. and 30 mm for 325 atms. Such packings of synthetic materials are unsuitable because of their inability to withstand high temperatures. Such packings of fiber have the disadvantage of shrinking when dry (this portion of the report is impossible to read).

In the assembly operation of screw parts on the large valves a spring scale with predetermined turning moment is used to regulate the applied torque.

Special Valves:

Pressure Relief Valves:

Berger: The normal type of Pressure Relief valve (needle type - patronen ventil), that is with spring held cone-disc and tungsten-carbide nozzle, screen, eccentrically arranged sealing ring, which was adopted from 200 atms. for 700 atms., showed barely tolerable wear in Gelsenberg and very strong wear in Pölitz, particularly at the nozzle outlet. Protection of the wearing surfaces by Granitherm showed very little improvement.

Dr. Rohnalter: Because of crushing (Auskolken) behind the nozzle , the nozzle soon breaks out. As a rule nozzles break out, rather than wear out. In Gelsenberg the nozzles are no longer soldered or shrunk in, but fitted in after copper plating. Copper plating does not eliminate any unevenness. Gelsenberg expects much from a short nozzle with contiguent widening, which was in operation for 1,037 hours instead of the usual 400 to 500 operating hours. Nozzles which are subdivided into individual rings will not do much good.

Zimmermann: Pölitz has achieved 1,400 operating hours with an elongated nozzle built up of 2 parts and widened at the outlet. In Pölitz also, the nozzles broke out after the surface material was gone.

Check Valves:

Berger: The spring check valves provided with mushroom disks have repeatedly stuck. This is due to the naturally low power of the springs, which weaken, particularly at higher temperatures, and to the closing pressure caused by the liquid stream, which loses effectiveness on the upper disk surface because of its characteristic construction. It is therefore proposed to build a springless mushroom disk check valve on which the closing pressure can freely act.

Regulating Valves:

Berger: While we use the present construction for the smaller sizes, we use the construction of the normal shutoff valve for sizes from 24 mm on up, in which a throttling pin or needle is attached to the disk. The throttling pin may be changed to suit operating requirements (gas, liquid, fine, or coarse regulation). In larger sizes, remote control by means of electric drive is possible. The seating surface is so formed that it is not a throttling surface, that is, not subject to wear.

Weid: Regulating valves should be so installed that the flow of the fluid is in the direction of the stem, that is, the pressure is against the under side of the disk. If the flow is in the opposite direction, the regulating cone serves as nozzle, which makes regulation difficult.

Motor Operated Valves:

(Scherer discusses the advantages of motor driven valves built according to "Bauart 1940". This discussion discloses nothing new and is of no particular interest to us.)

Gates:

Raeithel: Gates are now so constructed that the inner pressure aids the opening. With the same direction of rotation of the hand wheel, the gate movement for closing is therefor opposite to that in the valves.

Beutner reports that leaks in gates are probably due to corrosion. Hahn points out that gates should only be used where no dirt or scale is in the line. Maier reports that the nitride case has been crushed on individual gate disks.

Liquid Gauges:

Dr. Kämpfe: The sealing of large surfaces and the sensitivity of glass require a very careful construction. Many experiments have been made in the course of years to improve liquid level indicators. Experiments were particularly made on the means of producing initial tension. The initial tension must be high enough to produce an adequate seal, but must not be so high as to break the glass. The springing due to initial tension must be large enough to overcome unavoidable deviation in the glass thickness and the assembly dimensions. The following values indicate the recommended spring necessary to produce the required initial tension:

	Initial Tension:	Back Spring:
For Buna Frames:	About 1,000 kg.	About 2 mm
? For "Wellfedern"	About 8,300 kg.	About 1.6 mm
For Aluminum Wire: Compression		
From 4 to 2.5 mm	15,000 kg. 35,000 kg.	About 0.2 - 0.4 mm

Based on experiments, an initial tension of 5,000 to 6,000 kg. is sufficient. With the high initial tension produced by aluminum wire many glass-breaks occur. With Buna the frame is self-sealing, because it is put under higher tension only under operating pressure, but Buna cannot be used for 700 atms. and for various products. Therefor, most liquid gauges have, for a long time, been made with aluminum wire and aluminum foil for sealing. Since the glass thickness may vary as much as 0.1 mm, very accurate assembly is necessary. For special purposes, for which no aluminum can be used, liquid gauges of Buna frames with fiber cushions may also be used.

Fittings, Sealing Rings:

Raeithel: In the elbows the cutting edges on the inside are rounded off, in Ts they are broken. Bends are now made up to 200 mm sizes. The bend sizes 160 mm and 200 mm are somewhat longer than the elbows. Fittings with copper lining or chrome plated fittings are also available.

Sealing rings for hot lines will in the future be made only of N5A with a Brinell hardness of 180 to 220. Somewhat softer rings are acceptable for special purposes. Bellows sealing rings are the same in material and dimensions for 325 and 700 atms. Blind sealing rings are alike in material and dimensions for both pressures. Cold blind sealing rings of the larger sizes differ in material and dimensions, hot rings only in dimensions. Material and pressure are indicated on the flap.

Manometer disks are furnished only with 1 mm bore.

It is desired that fittings, particularly bends, be provided in the welding as well as the screwed type. Welding bends for 325 and 700 atms. will in the future be kept in stock. It is also desired to provide limiting dimensions for the sealing rings in order to facilitate their fitting. These rings should also be made extra large in order to provide a greater fitting range

Threads: Our threads are still cut to an accuracy corresponding to a fine fit. A medium fit should also be satisfactory just as in other industries. In order to avoid the freezing of threads they should be greased with hot-steam cylinder oil and colloidal graphite.

(Report from here on is very difficult to read but seems to deal largely with increase in stresses demanded by government authority, selection of materials, delivery details, drawings, stock lists, etc., all of which are not important for our purpose.)

Abstract submitted by:

Dr. L. L. Hirst, U. S. Bureau of Mines

AN ABSTRACT OF A REPORT ADDRESSED TO HYDRIERWERKE POLITZ DATED LUDWIGSHAFEN/RHEIN, 17 FEBRUARY 1943, ON THE STRENGTH OF TUBE STEEL IN 700 ATMOSPHERE PREHEATERS.

Frames 29-31

The relationship between circulating gas temperature, tube wall temperature, and fatigue strength, as well as the dependence of tube wall temperature on the incrustation of the tubes in operation, has always been of the utmost importance in the construction of 700 at. gas preheaters.

While in 325 at. preheaters the circulating gas temperature could, in general, be limited to the temperature corresponding to the fatigue strength of the tube wall when fully incrusted, the conditions of the 700 at. stalls have forced us to set the maximum circulating gas temperature higher than the temperature corresponding to the fatigue strength of the incrusted tube wall because of the missing, or only partially possible, paste heat exchange, as well as the high thruput. In other words, to utilize to the utmost the fatigue strength values, even with a not fully incrusted preheater.

It has been generally agreed that the layout of the preheaters should be based on a maximum crust thickness of 10 mm. allowing a certain safety factor based on the fatigue strength. The maximum wall temperature of such an incrusted tube has been calculated to be 550°C. Later observation and investigation in materials testing have shown that the materials cannot properly be evaluated on the fatigue strength alone. Under the existing mechanical and chemical influences, at the high material temperature, the materials show a time dependence on the elastic strength of the materials.

It may be remarked here that this relationship has not been fully cleared up. In particular, the influence of the crust, or the lack of a protecting oil film because of the presence of this crust, is still to be investigated. Such laboratory investigations are now being made. Investigations are also being made to determine the factors responsible for the formation of this crust, with the object in view of minimizing the crust formation. In any case, latest developments seem to indicate that increasing attention must be paid the crust thickness.

Tests so far made have shown that the maximum permissible inner wall temperature, to which N10 may be subjected to for a longer period at the full pressure of 700 ats., is for the time being limited to 520°C.

However, additional investigations must be made to determine if this temperature may be increased by proper heat treatment. It may already be said, however, that this temperature limit is strongly dependent on the mechanical stress of the tube wall, that is, on the operating pressure. If, for example, a stall is run at only 600 at. conditions become considerably more favorable. According to the latest tests, harder N10 tubes had a longer time-elastic-strength in hydrogen than softer tubes. If formerly soft N10 tubes were recommended, this was perfectly correct from general mechanical point of view. Soft N10 tubes have a greater toughness with still ample fatigue strength. As already mentioned, latest investigations have shown that the life duration of the tubes is not determined by the fatigue strength but by the time-elastic-strength and that this time-elastic-strength is insufficient for soft N10 tubes. Experience has further shown that no value need be placed on the greater toughness of the softer N10 tubes. It follows, therefore, that the tubes should be hardened to a greater degree by heat treatment. Since the wall temperature in the hot passes has exceeded 520°C, at least in some of our works, it is proper that all hair-pin tubes, which have been in service in the hot passes for about 10,000 hours, should be hardened by heat treatment. If such tubes are kept in service for a considerably longer period, lossening of the texture may be initiated, which we may not be able to correct by further heat treatment.

If in the future a wall temperature of 520°C on the inner surface is not exceeded in new installations, an after-treatment of the hair-pin tubes after 10,000 hours should not be necessary. We believe that we can attain a similar condition in all old hair-pin tubes by means of the first after-treatment.

As already mentioned, a lowering of the circulating gas temperature is not possible without lowering the thru-put because an enlargement of the preheater would be necessary.

The little available data seems to indicate that the tube wall temperature of coal preheaters has risen to 520° after about a year. Based on calculations, this corresponds to a crust thickness of 5 mm. The preheater would, therefore, have to be shut down after about a year and the tubes cleaned of the crust. The operating time is naturally largely dependent on the product, on its tendency to crust formation, and on the operating method.

In summary, the following rules for the increase in life duration of hair-pin tubes should be followed:

1) Hair pin tubes with the greatest possible Brinnell hardness should be installed at the hottest points.

2) All hair-pin tubes, which have been in service for about 10,000 hours in the hot pass at 700 ats., should receive an after-heat-treatment.

3) If an inner tube-wall temperature of 520° is not exceeded, a further after-treatment should not be necessary on new tubes and tubes which have received one after-treatment.

4) It is highly desirable that formed incrustations be removed at the latest after one year's operating time.

5) At lower pressure, for example, 650 or even 550 ats., used by some works at the present time, observations so far made seem to indicate that an after-heat-treatment after 10,000 hours of operating time is not required on normally hard hair-pin tubes.

I. G. FARBEN INDUSTRIE A. G.

Signed - Pier

Translator - L. L. Hirst,
Bureau of Mines

KCBraun/pkl

REPORT ON A ROUND TABLE DISCUSSION OF EXPERIENCES IN PREHEATER OPERATIONS, HELD AT LUDWIGSHAFEN, MARCH 22-23, 1943

Frames 32-36

Stettin-Pölitz, Apr. 1, 1943

The following points were discussed:

1. Measures taken to decrease the load on the preheaters particularly on the preheaters of the coal stalls in Blechhammer. These are smaller and therefore must be run at higher temperatures than, for instance, Pölitz. They also have a larger bore and consequently a thinner wall thickness. These effective measures for preheater load reduction were determined:

1.) To run a higher concentrated thin paste, since this will require less thick paste (Pölitz runs a 40% concentration).

2.) To run a higher concentrated thick paste (Pölitz runs a 53% concentration)

3.) Gas heat-exchange.

4.) Injection of cold paste into Converter II. A volume of approximately 6 m^3 of cold paste reduces the preheater temperature by about 20°C. By preheater temperature is meant the hairpin tube temperature and not the product temperature. Gelsenberg at the present time runs 4 tons of cold paste, making it necessary to measure the volume.

5.) The installation of a 5th converter as a pre-converter. The preheater is loaded only so that element 144 is held to 19 - 19.5 mv. Converter I is then put under draft. This measure reduces the preheater temperature by 50° C.

For comparison Leuna provides 80 to 90% of the necessary heat by heat exchange, Wesseling 65 to 70%, Welheim same, Gelsenberg 35%, Pölitz 55%, and Blechhammer tentatively 52%. Gelsenberg already runs a quadruple stall with the first converter as a preheat-converter, element 144 equals 19.5 mv and reaches reaction temperature only at the end of the first converter.

We were also informed that Scholven uses thinning oil in the hot catchpot. It is said that this has proved very successful in the centrifuge operation because the asphalts are said to be less harmful. They are more easily centrifuged and distilled. It is assumed that at high temperatures the oil aids the dissolution of the asphalts. This oil injection is properly made in the connecting line as provided in our #12 stall. In Pölitz they propose to start with catchpot heavy oil using about 2 m^3/h.

2. Crust Formation. Dr. Rank reported on his investigation of crusts at Pölitz and Gelsenberg. He told us that chromium, molybdenum and vanadium were found in the samples at Gelsenberg. Pölitz could not confirm these findings. They even found more in the vapor stage than in the coal stage. Therefore Gelsenberg seems to have run at higher temperatures than Pölitz. Dr. Rank has investigated the relationship between iron and titanium and concluded that in Gelsenberg the following relations exist:

Ash	to 1 part titanium	51 parts of iron
Bayer-mass	" " " "	8.1 " " "
Iron sulfate	" " " "	90.0 " " ", and on the
investigated crusts on the tube	to 1 part titanium	66.0 parts of iron

His conclusion that, because of similar relations of iron and titanium in the crust and in the ash the crust formation is related to the coal ash, did not find general approval. On the contrary, it is believed that the preheater attack is not attributable so much to the ash as to the catalysts which we add ahead of the preheater. Dr. Pier and Dr. Klinkhardt were especially of the opinion that besides sodium sulfide (for the prevention of chlorine corrosion) the catalysts should not be added directly to the coal, but that the Bayer-mass and the iron sulfate be added in the first converter.

3. Preheater Tube Materials. Chief Engineer Koch reported on the latest developments in high pressure materials. Experience has shown that the ability to deform decreases with time, particularly of the N10 tubes, that is, the material becomes brittle in the course of a longer operating period. The factors bringing this about are time, hydrogen, temperature, pressure, eventual crust formation and nitrogen (by nitration). Which of these components is the most important has not been determined. In any case, however, the presence of hydrogen is required. The most important of these factors are temperature and pressure. According to investigations to this date the materials investigated possess an operating life of 2 years. After that it is necessary to heat-treat the tubes in order to put them again into a normal condition. It has been shown that a greater hardness is better, because the harder material is less readily influenced. In order to give a picture of the nature of these influences he states, that a temperature reduction of 10° and a pressure reduction of 10% would mean a doubling of its life. He indicates a limiting temperature of 520°C for the inner wall and of 530 to 540°C for the outer wall. There are various measures now under way to better the materials. For example, it is proposed to decrease the carbon content and increase the vanadium content. However there are certain dangers connected with this because the fatigue strength is reduced thereby. On the other hand, the hydrogen attack is reduced. The reduction of temperature and pressure also favorably affects cold hairpin tubes, according to Mr. Koch. The foregoing therefore points to the necessity of adopting the measures outlined in paragraph 1 preceding, particularly of reducing the temperature of the preheater by means of thin paste, cold paste, etc. At Blechhammer the wall thickness of the preheater tubes is to be increased.

4. <u>Paste Press Check Valves.</u> It was determined that pressure and suction valves leaked, as a general rule, when flushed with oil. It is proposed to install a check valve behind each manometer. With the exception of Welheim no other plant has valves on the presses.

5. <u>The cutting of middle oil for prehydrogenation.</u> This point was particularly discussed in connection with the deterioration of 5058 converters in Pölitz.

Scholven has 2 prehydrogenation stalls with the following data:

1. Triple-prehydrogenation stall:

   Converter 1, Catalyst 5058, Temperature 19-21 mv. Catalyst 345 days old
   Converter 2, Catalyst 5058, Temperature 21 mv. Catalysts 127 days old
   Converter 3, Catalst 7846, Temperature 21.5 mv. Catalyst 100 days old

This chamber runs with a thru-put of 16 tons/h., 35,000 m^3 inlet gas and 55,000 m^3 (?) outlet gas. The aniline point of the middle oil rises from -12°C in the injection product to 45-46°C in the catchpot.

2. Triple-prehydrogenation chamber:

   Converter 1, Catalst 5058, Temperature 19-21 mv. Catalyst 100 days old(?)
   Converter 2, Catalyst 5058, Temperature 21-21.2 mv, Catalyst 356 days old
   Converter 3, Catalyst 7846, Temperature 22.2 mv, Catalyst 356 days old

Both stalls run with an injection product of 350°C end point. The 95% point could not be determined. No deterioration of the catalysts has been shown in either of these stalls with this injection product. The 6434 stalls also operate at low temperatures and give good results. Scholven believes, however, that the basic octane number has deteriorated since the introduction of catalyst 7846. By using lead, the gasoline may, of course, be brought to the same octane number as before, but it has a poorer overload curve.

To this might be added, that while 7846 gasoline has fewer aromatics, it has 1 or 2 more naphthenes. But because less gasoline is made with catalyst 7846 than in the prehydrogenation with catalyst 5058, a possible octane deterioration would have to be equalized by a greater proportion of 6434 gasoline.

Scholven also informs us, that, in order to improve the doctor test, 1/6 of the last 6434 converter is filled with 7846 catalyst in order to avoid difficulties with the gasoline.

Gelsenberg has 3 prehydrogenation stalls, all of which are combination stalls.

Stall 303 has the following data:

Converter 1, Catalyst 5058, Av.Temperature 20.8 mv, Catalyst 394 days old
(Inlet Temperature) 19.5
Converter 2, Catalyst 7846, Av.Temperature 22.2 mv, Catalyst 149 days old
Converter 3, Catalyst 7846, Av.Temperature 22.4 mv, Catalyst 149 days old

Operations were conducted under the following conditions:

Thru-put 0.7, total gas 3,500 m^3/ton injection, inlet gas 30,000 m^3/h, aniline point-13°C. The injection product has the following data: 90% - 315°C; end point 335°C.

It has been decided that from now on the 95% point shall be uniformly introduced.

There are no doubts about the distillation, 1941 specifications are available. These specify that the 325° point is determined by the Engler-volume. By the way it was not generally known that there is a difference between Engler and ASTM.

Stall 306 has the following data:

Converter 1, Catalyst 5058, Av.Temperature 20.8 mv, Catalyst 420 days old
Converter 2, Catalyst 5058, Av.Temperature 21.5 mv, Catalyst 420 days old
Converter 3, Catalyst 7846, Av.Temperature 22.0 mv, Catalyst 114 days old

"B"-middle oil aniline point = 47 - 48°C, gasoline concentration in the catchpot at 115°C = 36%.

Chamber 302 has the following data:

Converter 1, Catalyst 5058, Av.Temperature 21.1 mv, Catalyst 792 days old
Converter 2, Catalyst 5058, Av.Temperature 21.8 mv, Catalyst 792 days old
Converter 3, Catalyst 7846, Av.Temperature 21.9 mv, Catalyst 40 days old

Aniline point of "B"-middle oil = 48 - 49°C, gasoline concentration in the catchpot at 155°C = 34%.

The injection product for the benzination over catalyst 6434 has a specific gravity of 0.848 at 20°, aniline point of 48.70, 0.03% phenol, and an end point of 305°C.

There seems to be no satisfactory explanation of the damages to the catalysts in the 5058 converters in Pölitz. The end point is high, but not so high that the catalyst should necessarily deteriorate because of it. It is thought that the damages are related to the distillation of the Brüx tars, possibly because phenols which have bypassed the liquid-phase, are introduced into the vapor-phase by the "A"-middle oil. Besides, the phenol content of 25% is very high. In this connection we are advised that Scholven will put an acid refining unit into operation in about 4 weeks, in which the 5058 injection product will be washed with 25% acid.

6. Sulfurization of prehydrogenation and benzination injection products. In Pölitz the sulfurization of prehydrogenation products is no longer done with hydrogen sulfide, but with granulated sulfur, because it was found that separations occur in the former method. The ash content of the injection product is, however, normal and equals about 2 - 4 mg/liter. The extraordinarily rapid drop of the K-values of the heat exchangers in the sulfurization with H_2S is appreciably slowed-up in the sulfurization with granulated sulfur.

The question arises here, if oxygen was contained in the hydrogen sulfide? In the opinion of Ludwigshafen this would be harmful.

The method of sulfurization in Gelsenberg is at the present as follows:

5058 and 6434 are sulfurized with granulated sulfur, a sulfurization with hydrogen sulfide is being installed for both phases.

Scholven also sulfurizes the prehydrogenation injection product with granulated sulfur. It was intended to sulfurize with hydrogen sulfide but this method was dropped because of the experiences at Pölitz. No data are available on the sulfurization of 6434 injection products. In both plants the injection products are filtered after the sulfurization.

7. Operating times of coal stalls. In connection with the information on the operating times of the coal stalls at Pölitz, Dr. Urban informed us that Scholven has an average of 79%, equal to 289 production days, 5% for warming up and shutting down, and 16% for repairs.

8. Increased efficiency of kiln operations (Schwelerei). The effects determined by Dr. Horn and Dr. Hinz in our report on the tests for increasing the efficiency in the kiln operations were generally confirmed. The effects were reached but not in the same degree. Dr. Pross states that an increased addition of steam is more effective than an addition of sulfur.

9. The use of heavy oil on solid catalysts. Ludwigshafen used Zistersdorf petroleum and various other oils and found that such an operation would not do, if ash was present in the oil, because the ash would be deposited on the catalysts. If 0.001% ash was present no deposit was noticed, while at 0.1% ash a deposit was already noticed after 10 days. For TTH, 0.01% solids is considered permissible. An experiment to run crude oil without a catalyst resulted in coking up at Ludwigshafen.

In connection with the above experiments it may be said that it is necessary to put the oil first through a pressure distillation. This requires that a considerable proportion of the high boiling portion be split (weak cracking). These experiments were also partly made with the addition of crude oil. In Pölitz, however, it will be necessary to run the crude oil as before. (In spite of the negative results achieved so far in the mixing of pitch and crude oil, further mixing tests will be made.)

/S/ Wissel

Translation received from Bureau of Mines

CONTINUOUS PROCESS FOR THE PRESSURE HYDROGENATION OF COALS, TARS AND MINERAL OILS IN LIQUID PHASE

Frames 37-46

Ludwigshafen, May 25, 1943

In the continuous pressure hydrogenation of coals, tars and mineral oils in liquid phase as, for example, of coal or oil pastes the liquid, together with the hydrogen required for the reaction, is at present preheated under pressure in a special preheater and brought to the reaction temperature. At this temperature the mixture then enters the reaction vessel. Here, due to the absorption of hydrogen by the hydrogenation feed, so much heat is generated, apart from the heat less through the vessel walls, which is only a small part of the generated heat, that in practical operations cooling means must be provided as, for example, through built-in special cooling surfaces, through the introduction of cold gas or through the injection of cold oil or of coal paste.

These measures bring many disadvantages with them. In particular the introduction of cold substances brings about a decrease in the space available for the reaction and a change in the proportion between gas and reaction mass so that the course of the reaction is often influenced undesirably.

In accordance with the invention submitted herewith, these disadvantages are avoided and considerable advantages obtained. In this process the hydrogenation feed, together with hydrogen, enters at the bottom of one section of a reaction vessel divided by separating walls into two vertical sections, which are connected with each other at the top and bottom, so that the hydrogenation feed is given a circulating motion between the two sections of the vessel, whereby the greatest part of the hydrogen, together with the vaporous and as a rule also with a part of the liquid, mass is drawn off at the top.

The separating wall can advantageously consist of a centrally arranged tube in the reaction vessel, a so-called guide tube, so that the inner space of the tube forms one section and the outer space the other section of the reaction vessel.

The stream of hydrogenation feed and hydrogen gas entering at the bottom rises, for example, in the guide tube. At the upper end of the guide tube the main mass of the liquid portion, generally free of gas, then again flows downward outside of the tube, is again united at the bottom with the fresh incoming mixture and again passes upward in the guide tube. At the upper outlet of the reaction vessel a mass of gas and liquid (as such or as vapor) approximately equal to that entering at the bottom is carried off during operations.

While in the present process the gas bubbles rise to the top comparatively slowly and without order because they must overcome the viscosity of the liquid column above them and because they themselves retard the return flow of the liquid. This new process generates a lively circulating motion in the longitudinal direction of the guide tube, which can be considerably faster than the original rising velocity of the bubbles in the liquid.

Because of this an intermixture of the whole contents of the vessel is developed as well as a breaking up of the large gas bubbles into a foam of the smallest bubblets with a large surface area. A rapid intermixture of the contents of the vessel with the fresh incoming stream of the hydrogenation feed makes it possible to reduce the inlet temperature of the vapor-liquid mixture by an amount equal to the heat of reaction in the reaction vessel. This latter is directly used for further heating of the fresh incoming hydrogenation feed. This eliminates the necessity of adding cold gas in an amount at present necessary, if the inlet temperature is correspondingly low. The volume of these cooling substances may be so much reduced that the reaction may be readily controlled.

The circulating stream receives its start from the difference in uplift between the upward and downward flowing sections and from the stream energy of the mixture entering the vessel. The velocity of the stream is limited only by the resistance to the stream in its circulating path. Means for aiding this stream flow and increasing its velocity, such as a nozzle-shaped entrance opening with injector effect, rounding off the corners of the reaction vessel and the guide tube, and the largest possible diameter of reaction vessel and guide tube should properly be employed. Also the proportion of the stream cross sections should be such that the resistances in the upward and downward flow sections are as closely as possible equal to each other. Moving devices in the reaction vessel, such as power-driven mixers, etc., are to be avoided by all means.

The passage time of the gas bubblets through the guide tube, and consequently their presence in the reaction vessel, is considerably shorter than in the present process because the gas volume in the reaction vessel is reduced with increased velocity of the circulating stream and because the gas is largely contained only in the rising stream, while only smallest bubblets, which are not separated from the stream at its change of direction at the top, are carried in the downward flowing stream.

Through this decrease in the gas volume present in the reaction vessel a corresponding space is freed for the liquid hydrogenation feed so that the feed remains in the vessel considerably longer, assuming equal volumes of reaction vessels, or that a greater volume of hydrogenation feed is processed, assuming equal time periods of its presence in the vessel.

The separation of the hydrogen and other gases in the upper part of the reaction vessels is aided considerably if the rising liquid-vapor stream is given a strong twist by properly arranged entrance nozzles or by the arrangement of guiding surfaces within the guide tube. Under the influence of this twist the liquid is easily separated at the return point at the top from the gas collecting in the center so that only little gas is carried downward. Small openings in the upper part of the guide tube will have the same effect because they reduce the velocity of the liquid at this point.

The guide tube can readily be constructed with a double wall and used as a very effective cooling or heating surface.

A particular advantage of the proposed means is, as already explained, the lower preheat temperature required. The manufacture and procurement of preheater tubes for high reaction temperatures offers many difficulties since the strength of alloyed steels for this purpose drops rapidly with rising temperature so that an increase of 10°C more or less in the wall temperature of the preheater tubes is of great significance. In addition, it is possible to attain the reaction temperature only by using considerably longer tubes, if the permissible temperature difference between the heating gases and the preheater wall temperature becomes small.

If, on the contrary, it is possible to introduce the feed into the reaction vessel at a temperature lower by about 50 or 100°C than at present, that is, within the reaction temperature, the preheater can be operated at lower wall temperature, and the previously mentioned difficulties disappear entirely. In addition, the preheater resistance and required pump energy drop considerably because of the shortening of the preheater tubes. Very often the preheater may even be entirely dispensed with in operation, if the now possible temperature difference between the outgoing and incoming stream is utilized in the heat exchanger so that the preheater is used only at the start of operations.

This provides a very considerable saving of fuel and energy and a great lessening of the danger to the reaction stalls caused by the accumulated heat in the preheater walls in a sudden shutdown.

Besides the saving in fuel a considerable saving in expensive alloyed metals is possible because of a lowered preheater temperature and a small preheater.

In connection with the lowering of the preheater temperature it is further possible to protect the reaction substance, prevent it from overheating and from coking on the preheater walls.

A further advantage is gained by the previously mentioned lowering of the cold gas volume required in the present process. The use of much cold gas requires a higher pressure in the circulating pumps and therefore considerably greater energy. Furthermore, in the present process the cold gas has to be added at various points in the reaction chamber to avoid local overheating. If it is desired to add cold gas at all in the new process, this may be done at one point. Since the entire content of the high reaction vessels is completely circulated within a fraction of a minute equal temperatures (are) obtain(ed) in all parts of it. Furthermore, due to the decrease in cold gas volume it is possible to keep the proportion of gas to liquid volume in a chamber with several reaction vessels the same, so that the desired partial pressure can more easily be achieved.

A further advantage is gained in the decrease of the volume of the feed passing through the reaction chamber so that the connecting pipes and heat exchangers can be reduced in size or be used for larger capacities.

The fine distribution of the hydrogenation gases possible in this new process enables us to reach an equilibrium in the reaction, an effect, which otherwise is possible only through an increase in pressure, a longer time period required for the feed to remain in the reaction vessel, or the use of valuable catalysts. For the same reason it is possible to lower the average reaction temperature with the same capacity and hydrogenation effect.

The process is further clarified in the attached drawing.

In figure I, (1) is the inner wall of the reaction space of a hydrogenation converter, (3) is the inlet and (4) the outlet tube for the gas-liquid mixture (for example tar), (2) is the central guide tube which is arranged in converter I in such a manner that an unhindered circulating motion through the guide tube in a longitudinal direction of the converter is made possible. The mouth of tube (3) is nozzle-shaped in order to introduce the liquid mixture with increased velocity.

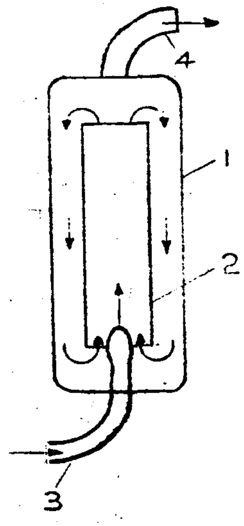


FIG I

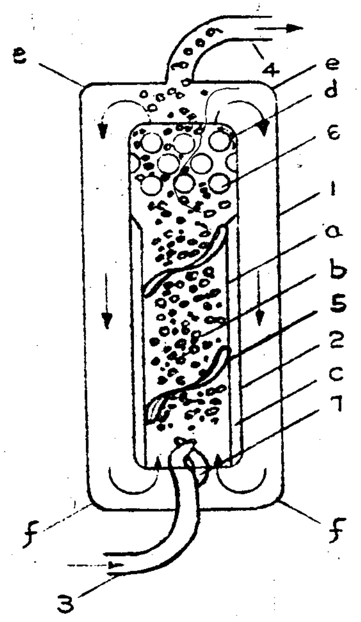


FIG · IX

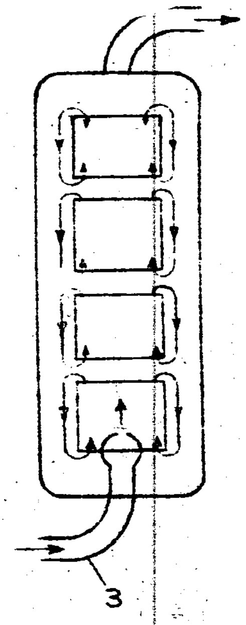


FIG VI

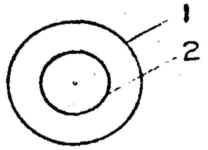


FIG II

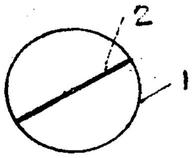


FIG III

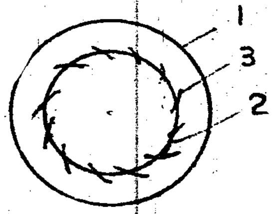


FIG V

Figure II shows a cross section through converter 1 with guide tube (2). It is immaterial whether the flowing mass rises on the inside or the outside.

Figure III shows a cross section through another construction in which the separating wall is a simple cross wall (2) instead of a guide tube.

Figure IV shows a double walled guide tube to receive heating or cooling fluid or cooling gas or provide room for electrical heating. In order to decrease the stream resistances the guide tube is rounded off at (c) and (d), the converter at (e) and (f).

In order to achieve a better separation between vapor (foam) and liquid an additional turning motion of the circulating stream is superimposed. Helical shaped guide surfaces (5), tangentially arranged inlet nozzles (7) and guide surfaces (8) on the upper and lower end of guide tube (2) are provided, as shown in figure V. Under the influence of the twist produced in this manner the liquid moves preferably along the wall of tube (2) at (a) (figure IV) while the lighter foam flows towards the top mainly in the center. In this manner the gas volume in the converter is considerably decreased. In addition, openings (6) can be arranged at the upper end of the guide tube in order to decrease the stream velocity at the return point at the top and to make possible a better separation of liquid and gas.

Figure VI shows the guide tube (2) subdivided into several sections, so that several short circulating streams besides the main circulating stream, may be formed. These small circulating streams can also be completely separated from each other through cross walls so that so-called multiple converters are formed, which then may be further developed according to figures I to V.

PATENT CLAIMS

#1.-Continuous process for pressure hydrogenation of coals, tars or mineral oils in liquid phase, producing a circulation of the hydrogenation feed in the reaction vessel, so that; the hydrogenation mass, together with hydrogen, will enter one section of the reaction vessel (which is divided into two vertical sections, connected with each other at the top and bottom) at the bottom in such a manner that the hydrogenation mass is caused to circulate between the two sections, whereby the greatest part of the hydrogen entering at the bottom is taken off at the top, together with vaporous and fluid products of reaction.

#2.-Process according to Patent Claim 1,-characterized by a tube centrally arranged within the reaction vessel and possibly provided with side openings at the top, forms the dividing wall between the two sections of the reaction vessel, so that the space within the tube forms one of the sections and the space outside of the tube forms the other section.

#3.-Process according to Patent Claim 2,-characterized by the central tube, consisting of several pieces of a tube separated from each other by intervening spaces.

#4.-Process according to Patent Claims 1 to 3,-characterized by hydrogenation feed entering tangentially, so that it will flow towards the top in a spiral stream in one of the sections.

#5.-Process according to Patent Claims 1 to 4,-characterized by the section, in which the hydrogenation feed flows towards the top, provided with guiding surfaces giving the upward flowing mass a twisting motion.

#6.-Process according to Patent Claims 1 to 5,-characterized by the hydrogenation feed (possibly after being heated by heat exchange) entering the reaction vessel at a temperature considerably below the reaction temperature.

I. G. Farben Industrie, A.G.

/S/ Kleber

Translation received from Bureau of Mines

Report 584-46R

RESEARCH ON RIBBED TUBES
(REPORT ON THE TECHNICAL TESTS AT OPPAU)

Frames 48-67

Abstract

Report 584-46R

RESEARCH ON RIBBED TUBES
(REPORT ON THE TECHNICAL TESTS AT OPPAU)

Measurement of the Heat Transmission on a Piece of Hairpin Tubing after Two Years Operation Time -- Heat of Conductance Number of the Crust -- Influence of the Ribbing on Wall Temperature Measurement.

This report is a summary of the technical tests carried out at Oppau on ribbed tubes. The report is divided into three sections. A short introduction gives the nature and purpose of the experiments. This is followed by a detailed description of the experimental installations and equipment. Part of this is illustrated in two blueprints included in the report. The third part, entitled "Measurements and Results" is a detailed description of the methods used in the measurement or determination and calculation of the following: (1) the outer heat transmission number, (2) wall temperature measurements, and (3) heat of conductance numbers of the crust.

A piece of ribbed tube was cut out of Hairpin G-72 type tube, which had been developed from a gas preheater in Gelsenberg after 15,040 hours operation time, heated, and then subjected to a technical investigation. The inside, firmly applied crust of 8 mm thickness retarded the heat transmission about half. The portion of the heat resistance of the crust at the same heat passage had become so large that the ribbing of the hairpin had only a slight influence on the heat output. The temperature in the wall of the high-pressure tubes was measured with displacable thermo-elements, and the temperature change in the pieces of tubes without ribbing determined. The temperature measurement error in dependence on the number of faulty ribs is represented by a curve included in the report. The heat conductance number of the crust was determined according to other methods to λ = 0.9 "kcal"/mh °C.

Included in the data are two blueprints and six graphs. The blueprints represent the installation of the ribbed tube in the channel. The set of graphs include the following: (1) outer heat transmission number in relation to the outer tube surface, (2) temperature course along the tube wall, (3) temperature course along the tube wall with three faulty ribs, (4) temperature course along the tube wall with six faulty ribs, (5) temperature course along the tube wall with nine faulty ribs, and (6) per cent of temperature variance in the middle of an unribbed piece of tubing in contrast to a ribbed piece of tubing.

Report 584-46R

DHD PROCESS

TABULATIONS OF PRODUCT TESTS
AND OPERATING DATA
WITH DIFFERENT CHARGE STOCKS

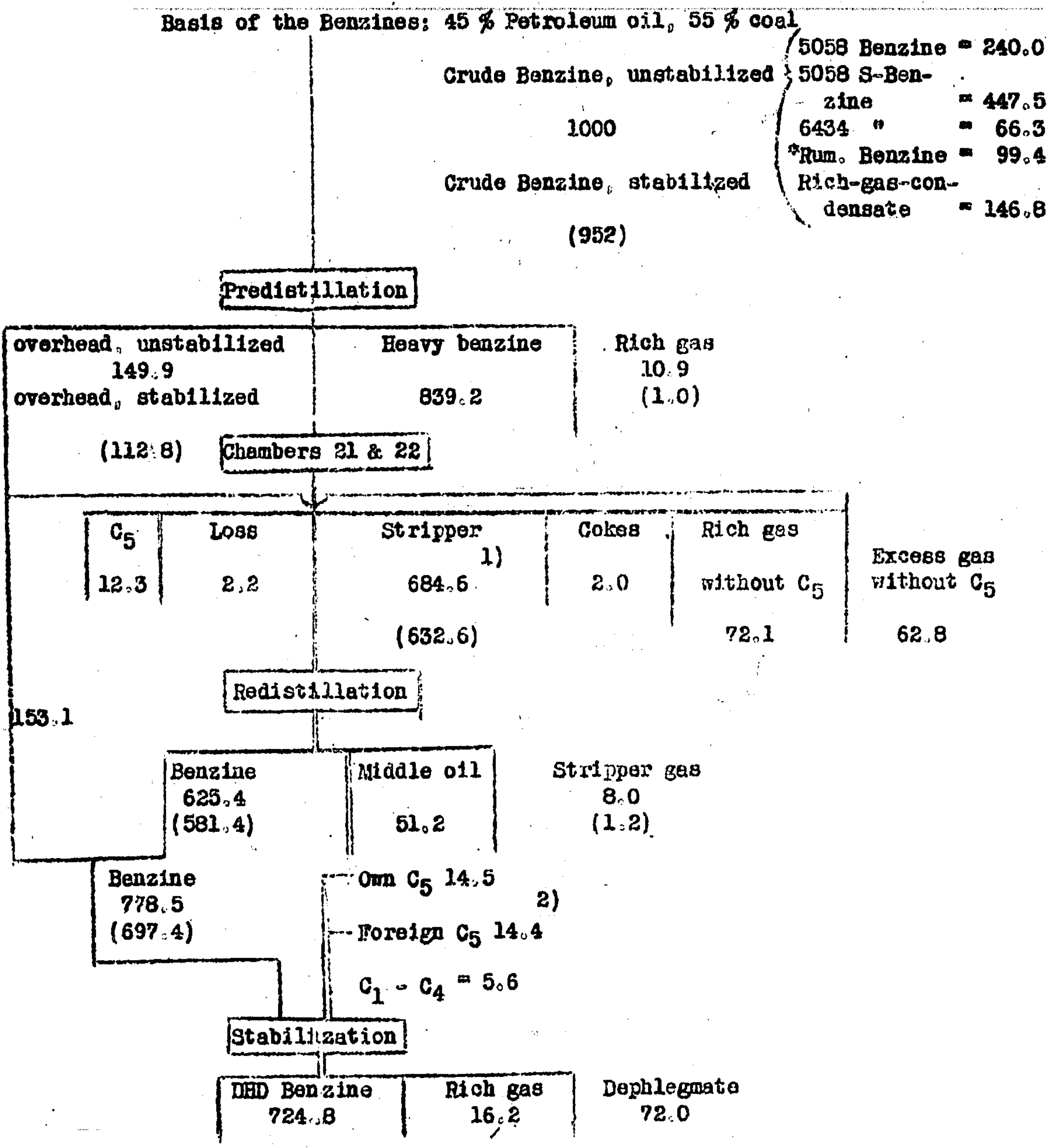
Frames 247-277

Abstract

A typical complete set of data (Frame 251) is reproduced here. Tables like the above are given for each of about 30 different charge stocks on frames 247 to 277. The charge stocks are described in tabular form beginning with frame 248.

251

1000 M. ton diagram of the dehydrogenation. March, '44 (Values of the operating controls)



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1) $C_1 - C_4$ = 52 ton
2) C_4 = 1.5 ton
* Rumanian

(continued)

PRODUCT TESTING. VALUES FOR PRE-AND RE-DISTILLATION

Datum tested	Crude Benzine	Overhead	Injection	Stripper	Benzine 60% Ar.	Middle Oil	Ad-Fertig Bi TK 124_3
Yield Gravity							
d_{20}	.751	.639	.769	.784	.789	.973	.767
Boiling point °C	35	20	102	32	37	194	40
5 vol % °C	74	25	114	60	58	201	53
10 "	103	28	118	72	68	204	59
30 "	136	39	129	105	100	209	80
50 "	145	52	139	130	121	216	104
70 "	155	68	148	149	138	228	129
90 "	171	-	159	-	159	300	152
95 "	-	-	166	-	169	350	163
End point °C	177/92	94/84	173/98	200/88.5	178/97	350/95	173/98
Loss %	7	15		9.5	2		-
Vol % 70°C	4.5				11		21
" 100°C	9.5				30		47
Aniline Point I			50		-11.2		5.2
Aniline Point II			58.6		52.2		61.2
Aromatics Vol. %			10		59.0		49.0
Naphthenes "			35		16.0		14.0
Paraffins "			55		25.0		37.0
Octane #I					83.0		80.4
Octane #II					-		92.2
Iodine Number					0.9		1.7
Octane #I of the remaining benzine					--		--

Raw products	d_{20}	b p	10%	50%	90%	95%	e p	Aromatics	Naphthenes	Paraffins	API °C	S_2 wt. %
5058 Heavy Benzine	.779	134	142	154	170	178	214	9.0	32	59	50.1	-
5058 Benzine	.758	49	85	127	173	180	183	8.5	52.5	39	47.5	-
Petroleum oil	.750	84	104	126	154	167	-	14.5	20	65.5	49.4	0.02
Condensate	.656	34	39	48	82	100	108	-	-	-	60.6	-

(continued)

Gas Analyses

Name	Rich gas	Excess gas	Stripper gas g/kg
H_2 Vol %	3.7	55.3	
CO "			
CO_2 "			
N_2 "			
C_1 "	15.8	29.7	3.5
C_2 "	33.4	10.3	
C_3 "	30.6	3.2	21.8
i C_4 Vol %	12.3	0.5	17.7
n C_4 "			34.7
C_5 + higher Vol	140		
C_5 g/m³		8.0	
A^3/to Injection	57	204	
Gravity kg/m³	1.6	.43	

	Chamber	Chamber + Pre + redistillation	Total, inclusive foreign C_5	Total, inclusive foreign C_5
Injection	1000			
Crude benzine	--	1000(1)	1000(1)	1000(2)
Foreign gas-forming	--	--	14.4	14.4
Benzine	711	--	--	--
DHD + benzine	--	745	757.9	724.8
Middle oil	61	54	54	51.2
Gas + Loss	226	199	200.5	236.4
Cokes	2	2	2	2.0

1) Stabilized
2) Unstabilized

B K Values:

Sa	Bi	Mi	Propelled gas	Lean gas	Rich gas	Cokes + Loss
1014.4	724.8	51.2	118.0	62.8	53.4	4.2

Product factor: 1.307 to/to Bi + Mi
Waste gas : 23.18 % on basis of Bi + Mi + Waste gas

Frame	Description			DHD-Benzine
248	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized	Approx. Benzine = 862.92 *Rum. Benzine = 127.14 Petroleum Stripper Benzine = 9.94		724.9
249	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized	Approz. Primary Benzine = 799.62 *Rum. Benzine = 200.38		741.96
250	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 959.62	556.7 to 5058 heavy (benzine) 131.3 to 6434 heavy (benzine) 158.8 to petroleum stripper benzine 163.2 to condensate	Basis 40 to 50% petrol. oil, rest coal	708.77
251	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized (952)	5058 Benzine = 240.0 5058 Heavy Benzine = 447.5 6434 Heavy Benzine = 66.3 *Rum. Benzine = 99.4 Rich gas condensate = 146.8	Basis = 45% petrol. oil 55% coal	724.8
252	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized (949.1)	5058 Benzine 500.0 5058 Heavy Benzine 105.8 6434 Heavy Benzine 97.2 Petroleum oil Benzine 122.0 Rich gas condensate 175.0 1000.0	Basis 50% petrol. oil 50% coal	724.8
253	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized (980.1)	5058 Benzine 13.0% 5058 Heavy Benzine 41.5% Petrol. oil Benzine 34.0% 6434 Heavy Benzine 3.5% Rich gas condensate 8.0%	Basis = 40-50% petrol oil	730.7

*Rumanian

Frame		Description		DHD-Benzine
254	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 978	820.2 to 5058 Benzine 65.0 to 5058 Heavy Benzine 62.4 to 6434 Heavy Benzine 52.4 to Rich gas condensate		766.5
255	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 982.4	772 Benzine 5058 117 Heavy Benzine 5058 80 Heavy Benzine 6434 31 Rich gas condensate	Basis 25-45% petrol. oil, rest-pitch + coal	766.1
256	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 986 to	580.0 Benzine 5058 248.0 Heavy Benzine 5058 173.2 Heavy Benzine 6434 34.8 Rich gas condensate	Basis 45% petrol. oil rest-pitch + coal	785.6
257	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 988	898.8 Benzine 5058, of which ca 230 to heavy (benzine) 101.2 Heavy Benzine 6434	Basis 45% petrol. oil rest-pitch + coal	766.5
258	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 983.3 to	688 5058 Benzine (of which 250 Heavy Benzine) 140 6434 Benzine (of which 100 Heavy Benzine) 172 petrol. oil Benzine	Mixing strongly fluctuating Basis 30% petrol. oil, rest-pitch + coal	739.2
259	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized (984)	51.8% 5058 Benzine } Mainly 17.8% 6434 Benzine } heavy benzines 20.4% petrol. oil 120-180°C	Basis 45% petrol. oil rest-pitch + coal	753 to 10.2%

Frame	Description			DHD-Benzine
260	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 995.0	5058 Benzine = 619 6434 Benzine = 132 Petrol. oil Benzine = 249	Basis 40% petrol. oil rest-pitch + coal	755.8
261	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 998.2	406.8 5058 Benzine 128.3 6434 Benzine 372.1**Ung. Benzine 92.8 *Rum. Benzine		782.7
262	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 968.2	60% 5058/7846 Benzine 20% 6434 Benzine 16% Hungarian petrol. oil Benzine 4% Rumanian petrol. oil Benzine 5058/7846 { 25% petrol. oil 20% coal tar 55% coal products		757.6
263	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 957 to	52 wt. % 5058/7846 Benzine 48 " 6434 Benzine	Basis 40% coal; 60% Brüxdr. tar	94.1% 770
264	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 980	85.3% 5058/7846 Benzine 12.6% 6434 Benzine 2.1% petrol. oil, crude Benzine	Basis 30% coal; 70% Brüxdr. tar	828.3

*Rumanian
**Hungarian

Frame	Description			DHD-Benzine
265	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 974	5058/7846 Benzine 85% 6434 Benzine 10% Petrol. oil 5%	Basis 30% coal 70% Brüxdr. tar	99.4% 805
266	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 960	5058/7846 Benzine 50% 6434 Benzine 50%	Basis {15% petrol. oil 30% Brüxdr. tar rest: coal	93.5% 740
267	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 986	5058/7846 Benzine 75% Crude Benzine from petrol. oil 25%	Basis {40% petrol. oil 30% Brüxdr. tar rest:coal	98.4% 789
268	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 980	5058/7846 93% Crude benzine 7%	Basis {40% petrol. oil 25% Brüxdr. tar rest:coal	97% 782
269	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 928	5058/7846 Benzine 90% Crude benzine 10%	Basis {40% petrol. oil 24% Brüxdr. tar rest:coal	726
270	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized 945	5058/7846 Benzine	Basis {55% petrol. oil 12% Brüxdr. tar rest: coal	92.5% 715

Frame	Description			DHD-Benzine
271	Crude benzine, unstabilized 1000 ton Crude benzine, stabilized	5058 Benzine 90% 6434 Benzine 10%	Basis 30-60% petrol. oil rest: coal	104.6% 798
272	Crude benzine, unstabilized 1000 ton 1310 m^3	5058 benzine	Basis 30% petrol. oil rest: coal	855 to 1084 m^3
273	Crude benzine, unstabilized 1000 ton 1310 m^3	5058 benzine	Basis 30% petrol. oil rest: coal	852 to 1110 m^3
274	Crude benzine, unstabilized 1000 ton 1350 m^3	6434/5058 benzine (34 vol % 6434)	Basis 40% petrol. oil 60% coal	735 to 945 m^3
275	Crude benzine, unstabilized 1000 ton 1350 m^3	6434/5058 benzine (35 vol % 6434)	Basis 40% petrol. oil 60% coal	711 to 930 m^3
276	Crude benzine, unstabilized 1000 ton	6434/5058 benzine (33 vol % 6434)	Basis 40% petrol. oil 60% coal	740 to 50% aromatics
277	Crude benzine, unstabilized 1000 ton	6434/5058 benzine (55% 6434)	Basis 35% petrol. oil 65% coal	90.7% 707.1

DHD PROCESS
PRODUCT INSPECTION

Frames 278-285

Abstract

RESIDUE BENZINE - INVESTIGATION APRIL 1944

Tank Sample Number	1244	1247	1250
d_{20}	.689.5	.684.5	.685.5
Octane Number I	63.7	63.0	62.6
Octane Number II	88.5	88.1	87.5
Initial b. p. °C	41	40.5	39
50 vol % °C	75	74.5	75
95 vol % °C	150	152.0	147
Endpoint °C	157	159.0	159
Aromatics vol %	5.0	5.0	4.0
Naphthenes vol %	23.0	18.5	16.0
Paraffins vol %	72.0	76.5	77.0
Aromatic Breakdown			
Benzene vol %	7.2	7.7	5.2
Toluene vol %	27.9	25.8	23.1
Xylene vol %	35.0	34.9	35.9
Higher Aromatics	26.8	27.8	33.1
Loss	1.5	0.6	0.9

DHD - PRODUCT BENZINE - INVESTIGATION APRIL 1944

Date	4/2	4/6	4/8	4/11	4/18	4/25	4/28
Tank Sample Number	1244	1245	1246	1247	1248	1249	1250
d_{20}	.767	.768	.769	.769	.769	.774^7	.774
Initial b. p.	40	39.5	40	40.5	40	40	40
10 vol % °C	61	61	60	60	60	61	61
50 vol % °C	106	109	111	111	112^5	116	115
95 vol % °C	165	163	165	165	165	165	165
Endpoint °C	175	173	174	174	170	173	171
-70 vol %	20.0	18.5	19.5	20.0	20.0	18.0	18.0
-100 vol %	45.5	45.5	42.5	42.5	42.0	39.5	40.0
Vapor pressure	.46	.45	.45	.46	.44	.45	.46
Aniline point I	+6.4	+6.1	+4.6	+4.8	+4.7	+1.5	+2.1
Aniline point II	61.4	61.8	62.5	62.8	62.8	62.5	62.5
Aromatics vol %	47.5	49.5	50.0	50.5	50.5	53.0	52.0
Naphthenes vol %	15.0	13.5	12.5	12.0	12.0	11.5	12.0
Iodine Number vol %	1.8	1.65	1.8	2.05	2.7	2.4	3.0
Oxidation test after aging		2.2					
Octane Number I	79.6	80.3	80.2	80.3	80.4	80.5	80.5
Octane Number II	92.8	91.9	92.5	92.4	92.6	93.0	92.6
Overload curve avg.							
Effective pressure							
values, kg cm^2 over CV$_2$b							
$\lambda^* = 0.8$	1.4	0.1	0.0	0.0	0.2	1.3	0.5
$\lambda^* = 1.1$	0.5	0.7	0.7	0.8	0.8	0.5	1.0

---

* λ = excess air number

DHD - PRODUCT BENZINE - INVESTIGATION MAY 1944

Date	5/1	5/5	5/11	5/14	5/23	5/25
Tank Sample Number	1251	1252	1253	1254	1255	1256
d_{20}	.759^{4}	.770	.772	.771^{8}	.773	.773^{6}
Initial b. p.	40	40	40	40	41	40
10 vol % °C	61	59	62	60^{5}	60^{5}	60
50 vol % °C	112^{5}	114^{5}	118	115^{5}	115^{5}	117
95 vol % °C	165	164	165	163^{5}	164	165
Endpoint °C	171	171	173	171	172	174
-70 vol %	19	20	17	19	19^{5}	19
-100 vol %	42	42	38	40	39^{5}	41
Vapor pressure	.44	.44	.47	.44	.44	.45
Aniline point I	+5.2	+5.4	+3.2	+3.2	+2.5	+2.2
Aniline point II	62.7	62.6	63.1	63.3	63.0	63.2
Aromatics vol %	50.5	50.5	52.0	52.5	52.5	53.0
Naphthenes vol %	12.0	12.0	11.0	10.5	11.0	10.5
Iodine Number vol %	2.6^{4}	2.7	2.4^{5}	2.3	2.6	2.6
Oxidation test after aging						
Octane Number I	79.7	80.6	81.0	81.1	81.4	81.5
Octane Number II		92.8	92.4	92.6	--	92.5
Overload curve avg.						
Effective pressure values, kg cm^{2} over CV$_{2}$b						
$\lambda^{*} = 0.8$	0.1	0.2	0.0	0.0	0.0	0.2
$\lambda^{*} = 1.1$	0.7	0.5	0.1	1.3	1.0	1.0

---

* λ = excess air number

DHD - PRODUCT BENZINE - INVESTIGATION AUGUST 1944

Date	8/9	8/13	8/19	8/23	9/8
Tank Sample Number	1263	1264	1265	1266	P. IX/487
d_{20}	.771	.771	.774	.775	$.742^8$
Initial b. p. °C	39^5	40	45^5	42	40
10 vol % °C	62	61	63	65	63^5
50 vol % °C	103	104	107	113	108
95 vol % °C	165	168	169^5	169	170
Endpoint °C	188	190	182	182	179
-70 vol %	19	19	18^5	14^5	15
-100 vol %	47	46^5	42	39	42^5
Vapor pressure	$.42^5$	.44	.43	.43	42
Aniline point I	+0.5	+1.8	+0.6	+0.8	36.1
Aniline point II	61.6	62.0	61.7	62.4	60.3
Aromatics vol %	52.0	52.0	52.5	52.5	24.0
Naphthenes vol %	13.5	12.5	13.0	12.0	24.5
Iodine Number vol %	3.4^5	3.4^5	3.2	2.2	1.5
Octane Number I	81.4	81.3	80.2	80.4	71.4
Octane Number II	92.3	92.5	92.3	92.3	86.6
Overload curve avg.					
Effective pressure values, kg cm^2 over CV$_2$b					
$\lambda^* = 0.8$	0.6	0.7	0.9	0.5	-0.8
$\lambda^* = 1.1$	1.9	2.1	1.5	1.6	+0.2

---

* λ = excess air number

DHD - PRODUCT BENZINE - INVESTIGATION SEPTEMBER 1944

Date	9/23	9/26	9/28	9/30
Tank Sample Number	1267+	1268	1269	1270
d_{20}	.776	.779	.778	$.777^7$
Initial b. p. °C	42	42	40	39^5
10 vol % °C	67	66	63	63
50 vol % °C	115	113^5	113	112
95 vol % °C	173	172	174	174
Endpoint °C	188	187	190	189
-70 vol %	12.5	14.0	14.5	16.5
-100 vol %	37.0	38.0	39.0	39.5
Vapor pressure	$.42^5$	.37	$.37^5$	.42
Aniline point I	+4.5	+0.4	+1.0	+1.4
Aniline point II	63.0	63.3	63.5	63.2
Aromatics vol %	51.5	54.5	54.0	53.5
Naphthenes vol %	11.0	10.0	10.0	10.5
Iodine Number vol %	5.6^5	8.0	4.0	3.3^5
Octane Number I	79.0	79.4	80.2	81.1
Octane Number II	89.1	91.2	90.6	91.3
Overload curve avg.				
Effective pressure values, kg cm^2 over CV_2b				
$\lambda^* = 0.8$	-0.4	0.1	0.0	0.5
$\lambda^* = 1.1$	0.2	1.1	+0.6	1.3

+ Still contains VT Benzine
(enthält noch VT Benzine)

---

* λ = excess air number

DHD - PRODUCT BENZINE - INVESTIGATION OCTOBER 1944

Date	10/2	10/4	10/6	10/12	10/19	10/21
Tank Sample Number	1271	1272	1273	1274	1275	1276
d_{20}	$.775^4$	.772	$.769^5$	.773	$.777^6$	.776
Initial b. p. °C	40	40	41	40	41	40
10 vol % °C	60^5	60	61	61	63	62^5
50 vol % °C	112^5	108	108	109	109	110
95 vol % °C	174	174^5	175	173	176	172
Endpoint °C	190	190	190	190	190	189^5
-70 vol %	18	19	20^5	20	19	18
-100 vol %	39^5	42	44^5	43	43	42
Vapor pressure	$.42^5$	$.42^5$	.45	.42	.40	.40
Aniline point I	+0.7	+3.2	+5.0	+1.8	+0.2	+1.3
Aniline point II	63.6	62.8	63.3	63.4	63.6	63.1
Aromatics vol %	53.5	52.5	50.5	53.5	54.0	53.0
Naphthenes vol %	10.0	11.5	11.0	10.0	9.5	10.5
Iodine Number vol %	2.8^5	2.3	2.3	--	2.6	2.7
Octane Number I	80.9	80.8	80.9	--	82.2	82.7
Octane Number II	91.5	--	91.4	--	92.6	91.2
Overload curve avg.						
Effective pressure values, kg cm^2 over CV$_2$b						
$\lambda^* = 0.8$	0.3	0.2	-0.6	0.3	+1.0	1.1
$\lambda^* = 1.1$	1.2	1.2	0.4	1.8	+1.6	0.6

---

* λ = excess air number

OCTOBER 1944

Date	10/23	10/25	10/27	10/29	10/31
Tank Sample Number	1277	1278	1279	1280	1281
d_{20}	.776	.774	.772^9	.773	.772
Initial b. p. °C	42^5	40^5	41	41	43
10 vol % °C	62	62	61^5	61	62
50 vol % °C	108	108^5	106	109	107
95 vol % °C	169	170	170	171^5	173^5
Endpoint °C	190	190	197^5	190	189^5
-70 vol %	18	18	19^5	19^5	20
-100 vol %	43^5	43	46^5	43^5	44^5
Vapor pressure	.41	.40	.40^5	.43	.42
Aniline point I	+0.1	+2.3	+2.3	+2.6	+2.6
Aniline point II	63.0	62.5	62.9	63.0	63.2
Aromatics vol %	53.5	52.0	52.0	52.5	52.5
Naphthenes vol %	10.5	11.5	11.0	11.0	10.5
Iodine Number vol %	2.1	2.3	1.9^5	2.4	2.5^5
Octane Number I	81.2	81.5	81.7	80.6	81.8
Octane Number II	92.5	92.5	92.0	92.1	92.5
Overload curve avg.					
Effective pressure values, kg cm^2 over CV$_2$b					
$\lambda^* = 0.8$	1.6	0.8	0.7	+0.7	+0.8
$\lambda^* = 1.1$	1.6	1.8	1.0	+1.3	1.4

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* λ = excess air number

Report 584-46R

DHD PROCESS

MONTHLY OPERATING REPORTS

Frames 331-754

Abstract

These monthly operating reports begin in January, 1942. The following is an abstract of the May, '42 report.

DEHYDROGENATION OPERATION
MONTHLY REPORT FOR MAY, 1942

Operation: DHD Chambers 1, 21 and 22,
Dehydrogenation of 5058 Benzine, Fraction 80-170 °C

SUMMARY

Only 5058 benzine, based on 30 per cent petroleum oil, was used in the process. The injection product had an aniline point of 36 °C and only 17.6 wt. per cent of it was vaporized in the chambers. About 9150 M. tons of Benzine with 60 vol. per cent aromatics were produced, sufficient for 11,000 M. tons DHD Benzine. Including the May stock, 11,562 M tons of specification DHD benzine was produced. The loading curve was usually 1.5 to 3.5 pme higher than the value for the comparison fuel, CV_2b. The improvement in quality was attributed to a pentane recycling in the stabilization.

After six months of operation the DHD Reforming Chamber was shut down on May 6. The catalyst showed no disintegration, the chamber was in good condition.

On May 16, chamber 22 was put into operation. The vaporization is at present somewhat higher than in chamber 21. This is due to a 3 atm higher pressure in the DHD furnaces.

Evaluation

1) The DHD of 5058 benzine proceeded very much like earlier experience. Using a catalyst loading of 0.6 kg/ltr/hr. production-times of 200 hours with regeneration-times of 17 hours were attained. The vaporization was between 17 and 18 wt. per cent.

2) Newest gas analyses indicate that the butane fraction of the rich gas contains 50 vol per cent isobutane, and that of the stripper gas contains 40 wt. per cent isobutane.

A) Yields

Injection -	11806 M. ton	100 wt. %
Stripper	10141 M. ton	85.5 wt. %
Benzine + C_5	9150 M. ton	77.5 %
Middle oil	570 M. ton	4.8 %
Vaporization	2,074 M. ton	17.6 %
Cokes	12 M. ton	0.1 %

B) Repairs

The DHD reforming chamber was inspected after 14 months operation. No corrosion was found.

Various minor changes and repairs are described.

C) By Products

D) Operation Details

Chamber 21, Period 17

A heavy benzine from the 5058 stripper boiling from 80 to 170 °C and having an aniline point of 36-37 °C was dehydrogenated.

At a pressure of 35 atm after furnace IV, 19 m^3 per hour with a gas volume of 14000 m^3 (Gas: product as 1:1) was injected. The gas density rose in the 120 hours from 0.32 to 0.42.

Periods 18, 19, and 20 we described as similar with some detailed differences in pressures, temperatures, etc. given.

Chamber 22, Period 1.

5058 Benzine was used, boiling between 80 and 170 °C, and with an aniline point of 36 °C. The chamber was operated at a lower rate, namely 10 m^3/hr. The density of the circulating gas was 0.36 and remained constant during the 68 operating hours.

Period 2 was similar except for a greater throughput of 13 m^3/hr at a stripper pressure of 30 atm. Across the chamber was an excessive pressure drop of 15 atm compared to 11 atm in chamber 21.

Period 3 was similar.

E) Operation details during Regeneration

In chamber 21, 18 hours were used for regeneration of which 8 hours were with air. In chamber 22 corresponding times were 17 and 6. Coke deposits on the catalysts were as follows in chamber 21:

Furnace	I	II	III	IV
Coking	.47 M. ton	.32 M. ton	.39 M. ton	.15 M. ton

F) Isobutane Gain during DHD of 5058 Benzine

Analyses of the rich gas and stripper gas are as follows:

	Rich gas	Stripper gas	
H_2	8.2 vol %	--	--
CH_4	14.0 vol %	--	--
C_2H_6	34.8 vol %	3.3	2.4 g/kg
C_3H_8	26.8 vol %	18.9	15.3 g/kg
Iso C_4H_{10}	5.6 vol %	14.4	11.8 g/kg
nC_4H_{10}	5.5 vol %	24.0	16.4 g/kg
C_5	4.2 vol %	22.4	--

And the butane fractions contained 50 vol per cent isobutane for the rich gas and 37 to 42 wt. per cent for the stripper gas.

G) Finished Product

In May, Tanks 1033 to 1041 were filled with specification benzines. The benzines contained 50-53 vol per cent aromatics and 16 to 20 per cent of product boiling below 70 °C.

Up to tank 1037 the overloading curve was about 1.5 pme higher than that of the comparison fuel CV_2b. In the later tanks this value rose to 2.5 to 3.5 pme. This was due to a difference in the stabilization. In the later ones some of the low boiling pentanes were removed.

Frame 450 is a table giving analyses of product benzine and residue.

AD - PRODUCT BENZINE INVESTIGATION MAY 1942

PRODUCT BENZINE - INVESTIGATION

Date	5/1	5/4	5/9	5/13	5/18	5/21	5/23	5/28	5/31
Tank Sample Number	P 1033	1034	1035	1036	1037	1038++	1039	1040	1041
d_{20}	.767	.768	.770	.772	.769	.770	.773	.768	.770
Initial b. p. °C	48	44	44	47	47	45	47	46	44
10 vol % °C	65	64	63	64	62	62	65	64	65
50 vol % °C	101	103	100	100	99	97	100	97	99
95 vol % °C	154	157	155	156	157	156	157	155	157
Endpoint °C	160	164	160	162	163	162	162	163	162
-70 vol %	16	16	18	18	20	20	17	19	17
-100 vol %	48	47	50	50	51	52	50	55	51
Vapor pressure	.40	.40	.40	.40	.39	.40	.39	.40	.39
Aniline point I	0.2	0.9	-0.7	-1.1	0.5	2.4	-1.6	5.3	1.4
Aniline point II	62.1	62.0	61.9	61.4	61.4	61.2	60.8	61.2	60.9
Aromatics vol %	51.0	52.5	52.0	52.0	50.5	53.0	52.0	51.0	50.0
Naphthenes vol %	13.0	12.5	13.0	13.5	14.0	14.0	14.5	14.5	15.0
Iodine Number vol %	1.3	1.2	1.3	1.3	1.5	1.5	1.1	1.5+	1.2
Oxidation test after aging	1.4	1.4	1.9	Water 1 mg	1.2	1.8	1.2	2.0	2.0
Octane Number I	80.9	81.5	81.7	81.7	81.0	81.5	80.9	80.4	81.5
Octane Number II	92.4	92.9	93.4	93.0	93.0	94.4	94.8	99.0	93.1
Overload curve avg. Effective pressure values, kg cm^2 over CV$_2$b									
λ* = 0.8	0.5	0.2	1.7	2.0	1.0	1.1	1.0	0.9	0.9
λ* = 1.1	1.6	1.8	1.7	1.4	1.4	1.5	2.5	2.4	2.7

---

* λ = excess air number

+Fugitive endpoint

++Beginning with tank P 1030 a part of the pentane is taken off for stabilization with the addition of fresh pentanes so that a greater part of the higher boiling pentanes has been concentrated in the product benzine. The end boiling point of the residue decreased. The benzine from tank 1038 contains 0.02 wt. % inhibitor.

(continued)

AD - PRODUCT BENZINE INVESTIGATION MAY 1942

RESIDUE BENZINE - INVESTIGATION

Date	5/1	5/4	5/9	5/13	5/18	5/21	5/23	5/28
Wt. of the Benzine	45.4	41.9	42.2	42.3	46.5	44.3	45.8	
d_{20}	.683	.681	.681	.681	.689	.680	.630	.690
Octane Number I	65	64.9	66.0	65.4	68.5	65.8	67.6	69.0
Octane Number II	85	83.2	87.2	84.5	82.8	88.0	87.6	90.1
Initial b. p. °C	38	38	39	38	39	38	38	39.5
50 vol % °C	73	73	72	73	69	70	70	70
95 vol % °C	146	147	147	147	148	141	142	142
Endpoint °C	152	155	150	151	152	148	150	146
Aromatics vol %	5.5	5.5	4	5.5	6	4	3.5	4.5
Naphthenes vol %	24.5	24	25	24.5	27	27	28	29
Paraffins vol %	70	70.5	71	70	67	69	68.5	64.5
Aromatic breakdown vol %								
Benzene	11.0	10.2	11.0	12.6	14.4	13.5	13.9	
Toluene	34.8	32.6	33.3	35.8	35.3	36.2	36.2	
Xylene	32.6	31.4	31.9	31.4	30.2	30.8	30.4	
Higher Aromatics	18.8	21.4	18.3	17.6	17.2	17.3	17.1	

The yields expected by the pilot plant can be attained in the future. Uniform operation of the chambers and sharp fractionation in the pre- and redistillation will be necessary.

Crude Benzine:

The DHD benzine used as raw product sometimes contained up to 24 per cent crude benzine from petroleum oil and up to 50 per cent of 6434 benzine. These benzines originated 15 to 60 per cent from petroleum oil and the rest from coal tar and coal. The benzine produced in all cases met specifications and contained 40 to 50 per cent aromatics.

Benzine Yield:

No correlation is evident between the amount of gas formed and the kind of crude benzine used. This is due to two reasons. Since the crude benzine is unstabilized, it contains varying amounts of C_2 to C_4 hydrocarbons. Then also, varying amounts of crude pentanes were added which also contain volatile fractions. When corrections are made for these effects, the amount of gas formed, and the yields of benzine and middle oil show a linear dependence on the aniline point of the crude benzine.

The following are the average yields for 1942

DHD Finished Benzine	76.1 wt. %
Middle Oil	4.6
Refinery Gas	6.8
Excess Gas	7.5
Rich Gas	3.7
Cokes and Loss	1.3
Gas-forming, cokes and loss, total	19.3

These yields are on the basis of a total of 119,663 ton finished benzine produced from April to December, averaging 13,300 ton per month.

Comparison to the Ludwigshafen Pilot Plant Yields

The Pölitz yields for 1942 were slightly lower than the pilot plant. For instance, in one case the pilot plant produced 78.3 wt. per cent DHD Benzine and the plant from a similar crude produced 76.7 wt. per cent.

The following summarizing graphs and charts are given:

Table A. A chart showing the monthly compositions (i.e. percentages of petrol. oil, 6434 Benzine, etc.) of the raw materials used.

Table B. A chart showing the monthly fractions of the finished product (percentage of gas formed, product benzine, middle oil, refinery gas, etc.)

Table C. A graph showing the aniline points of the manufactured benzines in terms of the compositions of both raw materials and products.

DHD RAW PRODUCTS AND YIELDS

Monthly Values 1942 at Pölitz Plant

Month	April	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Average
Source of Benzines										
Prehydrogenation	100	100	88	94	96	91	92	73	50	
6434	-	-	12	6	4	-	1	3	50	
Petroleum oil	-	-	-	-	-	8	7	24		
Basis of the Crude Product Wt %										
Petroleum oil	30	30	45	60	50	50	40	40	15	
Coal	70	70	55	15	30	30	40	30	55	
Tar (Brük)	-	-	-	25	20	20	20	30	30	
Properties of the Injection Product										
B. P. °C	80-180	80-175	90-175	80-175	100-180	95-180	95-180	95-180	95-180	
Paraffins vol %	36	35	45	51	49	47	46	42	46	
Naphthenes "	46	45	45	39	41	43	43	42	43	
Aromatics "	18	20	10	10	10	10	11	16	11	
Aniline point °C	38	36	45	49	49	46	44	41	46	
Yield of Finished Benzine after addition of foreign pentanes, basis of unstabilized crude										
Benzine wt %	73.6	73.0	72.3	72.3	71.1	72.5	78.1	78.9	74.0	74.0
Middle oil "	7.7	4.8	4.7	4.9	5.6	3.5	3.7	3.7	4.7	4.8
Gas, cokes, + loss "	18.7	22.3	23.0	22.8	24.3	24.0	19.2	17.4	21.3	21.2
Yields of the Chambers, on the Basis of the Injection										
Benzine, wt %	77.4	77.8	72.0	71.0	70.3	73.5	73.4	75.0	72.6	
Middle oil "	5.5	5.1	5.2	5.5	6.9	4.4	4.5	4.5	5.9	
Gas, cokes, + loss "	17.1	17.1	22.0	23.5	22.8	22.1	22.1	20.5	21.5	
Yield of Finished benzine after addition of foreign pentanes on the basis of C_4 free crude benzine										
Benzine wt %	77.6	78.4	73.5	73.6	73.9	76.6	76.7	76.2	76.7	76.1
Middle oil "	5.1	4.7	4.8	5.0	5.8	3.8	3.8	3.8	4.9	4.6
Gas, cokes, + loss "	17.2	16.9	21.7	21.5	20.3	19.6	19.9	18.9	18.4	19.3
Addition of foreign C_5 in wt % of crude benzine	8.3	13.0	7.0	5.6	1.3	1.5	2.2	2.2	0.3	
Gas in Crude Benzine wt %	1.8	1.5	1.5	1.5	5.5	7.2	2.0	1.4	4.0	

CONTACT VOLUMES AND THROUGHPUT OF THE DHD-CHAMBERS, PÖLITZ

Name	Reforming Chamber	Chamber 21 or 22	Chamber 19 or 20	Unit Chamber
Production of DHD-Benzine ton per year	36,000	78,000	100,000	100,000
Number of the furnaces	3 + 1	4 + 1	5 + 1	5 + 1
Catalyst Volumes m^3	22 + 8	33 + 8	42 + 9.6	42 + 9.6
Throughput:				
Coal-benzine ton/hr.	7.5-8.4	12.4 (16 m^3)	16.0 (21 m^3)	17.3 (22.5 m^3)
Catalyst performance kg/ltr/hr.		0.38	0.38	0.41
Petrol. oil benzine, ton/hr.	0.34-.38	13.8 (18 m^3)	17.6 (23 m^3)	18.4 (24 m^3)
Catalyst performance kg/ltr/hr.		0.42	0.42	0.44
DHD hours/year		6,500	6,500	6,000
Input:				
Petrol. oil crude benzine ton/year		105,000	134,000	134,000
Coal benzine, ton/year		97,000	125,000	125,000

Total Yield Pölitz Chamber 19 - 22 ---356,000 ton/year.

Report 584-46R

YEARLY REPORT FOR 1943 ON DHD AT POLITZ

SUMMARY

The raw material varied considerably in composition during 1943. Besides the dehydro benzines there were added up to 50 per cent of crude benzines from Rumanian and Hungarian petroleum oils. The paraffin hydrocarbon content of the injection varied between 30 and 65 per cent, and the average for the year was 46.5 vol %.

The average monthly yield finished product benzine was 13,500 ton. One thousand ton unstabilized crude benzine with the addition of 17 ton foreign pentanes produced on the average:

773	ton	finished benzine
47	"	Middle oil
197	"	gas, including cokes and loss

On the basis of C_4 free crude benzine without the addition of foreign pentanes the yields consisted of:

76.5	wt. %	finished benzine
4.8	"	middle oil
18.7	"	gas, including cokes and loss

The vaporization curve was about 1 per cent lower than in 1942. This was due to the fact that both chambers operated as 5-fold chambers in 1943.

The DHD chambers were used as follows:

Dehydrogenation	85 % of the time
Regeneration	15 % of the time
Repairing	5 % of the time

Depending on the kind of raw product, operating periods of 100 to 400 hours were used.

A) Raw Materials

Mixtures of the following raw materials were used during 1943:

1) Benzines like 6434, which consisted of up to 50 per cent petrol. oil and the rest of extractions from coal tars, pitch, and coal.

2) Heavy benzines of these, boiling from 130 to 180°C.

3) Crude benzines from Rumanian and Hungarian petrol. oils.

ANALYSES OF THE RAW MATERIAL BENZINES

	Benzine 5058	Benzine 6434	Heavy Benzine 5058	Heavy Benzine 6434	Rumanian Petrol. oil	Hungarian Petrol. oil
d_{20}	.747	.728	.784	.784	.749	.745
Initial b.p.	42	40	123	121	90	58
5 Vol % °C	69	57	134	132	99	-
10 "	79	63	138	134	104	79
30 "	101	84	143	143	114	101
50 "	118	104	151	151	124	115
70 "	137	127	157	159	132	130
90 "	163	160	171	172	147	153
95 "	171	170	177	179	-	-
Endpoint °C	174	174	198	211	159	181
Aniline point I °C	48.4	53.2	49.1	51.2	49.7	44.2
Aniline point II °C	55.0	59.0	57.8	59.3	63	60.4
Aromatics Vol %	8.0	7.0	8.0	9.0	16	17
Naphthenes "	45	34	37	32	19	26
Paraffins "	47	59	55	59	65	57
N_2 wt %	-	-	-	-	0.02	0.007
S_2 wt %	-	-	-	-	0.05	0.002
Octane # I	-	-	-	-	52	57

B) Yields and Gas Formation.

The injection product contained 31 to 65 vol. per cent paraffin hydrocarbons, and the year's average was 46.5. This was 2.5 per cent higher than the previous year. The wt. per cent of volatile hydrocarbons for the crude benzines varied from 0.2 to 4.3, and was highest for 6434 benzine.

The highest yield was 828 ton benzine in February, and the lowest was 730 ton in August.

C) Behavior of the Chambers

The down time was unusually low during 1943.

DHD - PRODUCT INSERTION AND YIELD

Monthly Values 1943. DHD - Plant Poelitz

Month	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	(Illegible
						Source of the benzines wt %							
5058 Benzine	90	15	52	60	40	--	22	44	70	54	77.2	62	
5058 Heavy Benzine	--	--	--	--	--	62	40	25	20	25	11.7	6.5	
6434 Benzine	10	13	43	20	--	--	--	4	--	--	--	--	
6434 Heavy Benzine	--	--	--	--	13	13	18	10	10	12	6.0	6.2	
Petrol. oil Benzine	--	2	--	20	47	25	20	17	--	--	--	--	
Gas Condensate	--	--	--	--	--	--	--	--	--	5	3.1	5.3	
						Basis of Hydrobenzene Vol %							
Petrol. Oil	5	--	--	25	35	35	45	30	45	45	25-45	25-30	
Pitch	--	--	--	--	65	86	55	70	56	50	Rust	Rust	
Coal	25	30	40	55									
Tar	70	70	80	20	--	--	--	--	--	--	--	--	
						Properties of the Injection Material							
Boiling Rnage °C	98/173	97/179	92/180	92/183	108-190	95-188		99-177	96-162	102-180	95-174	98-173	
Paraffins Vol %	36	34	40	46	52.5	51.5	56	54	47	50	44	42	
Naphthenes " "	52.5	57	43	48.5	34.0	40.0		37	45	41	45	51	
Aromatics " "	11.5	9	11	11.5	13.5	8.5							
Aniline point I °C	41.0	43	44	46.3	45.1	50.1	51.5	49	46	49.7	49	46.3	

(continued)

Month	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	(Illegible)
Yields of finished benzine after addition of foreign C_5 on the basis of unstabilized benzine													
Benzine wt. %	80.4	62.6	77.0	75.8	78.3	75.6	75.3	73.9	76.7	79.6	76.5	76.7	77.3
Middle oil wt. %	5.3	2.7	2.9	4.5	4.4	6.4	5.5	5.0	5.3	5.1	4.5	5.0	4.7
Gas coke + loss wt. %	18.0	17.2	20.1	21.5	20.7	19.0	20.9	22.2	19.2	19.3	19.6	18.3	19.7
Foreign C_5	3.2	2.7	0.0	1.8	1.4	1.0	1.7	1.1	1.2	3.0	0.9	0.0	
? ? (Illegible)	2.6	2.0	4.3	3.2	0.2	0.5	1.6	1.7	1.2	1.2	1.4	1.8	
Yields of the Chambers on the basis of injection													
Benzine wt %	75.7	79.9	76.7	63.3	72.0	72.2	71.7	71.0	72.2	71.4	73.1	74.4	
Middle oil wt %	6.4	3.0	3.6	4.5	4.7	6.0	5.9	5.5	5.0	5.8	5.3	6.0	
Gas coke + loss wt %	17.9	17.1	19.7	22.2	23.3	21.0	22.4	23.5	21.8	22.8	21.6	13.6	
Yield of finished benzine after subtraction of foreign C_5 on the basis of C_4 free crude benzine													
Benzine wt %	78.8	82.1	79.7	77.1	75.2	73.9	73.0	72.9	75.6	76.0	75.8	78.4	76.5
Middle oil wt %	5.4	2.7	1.0	4.6	4.4	6.4	5.6	5.1	5.4	5.1	4.6	5.2	4.0
Gas coke + loss wt %	15.8	15.2	17.3	18.3	20.4	79.7	21.4	22.0	19.0	18.9	19.6	16.4	16.7
Density of the cycle gas	.395	.310	.375	.445	.443	.42	.46	.447	.43	.45	.396	.34	

(continued)

Month	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	(Illegible)
						Finished Benzine							
Aromatics Vol %	48	53	50	49	53	52	52	52	46	46	45	47	
Vapor pressure	.40	.39	.40	.39	.40	.40	.40	.43	.44	.44	--	--	
Vol % - 100°C	47	42	47	47	39	36	38	39	46	46	50	46	
Overcharge curve	.3/.4	.3/.4	.7/.6	.1/.5	.3/.5	.4/.5	1.5/.9	.6/1.1	.2/.9	.5/.9	1./.8	1.1/1.0	

D. Operating Details

An operating period lasted 175 hours and regeneration 20 hours on the average.

Maximum throughput was 20 m^3/hr. The pressure after furnace IV was between 35 and 40 atm.

The aromatic content of the stripper was held at 60 vol per cent. The percentage of hydrocarbons boiling below 100°C varied between 30 and 45 vol per cent. This percentage depended on the boiling behavior of the injection material, whereas the operating conditions seemed to have no appreciable effect.

E. Finished Product

The product practically always came up to specifications.

The finished benzine contained 45 to 55 vol per cent of aromatics and 35 to 50 vol per cent hydrocarbons boiling below 100°C. The octane #I of the product benzine varied from 79-82 and the octane #II between 91 and 94.

The following summarizing graphs and charts are given:

Table A.	A chart showing the monthly compositions of the raw materials used.
Table B.	A table showing analyses of the raw materials and of the products is reproduced here. (See Frame 679)
Table C.	A graph showing the distillation temperature of the injection materials for each month.
Table D.	A graph showing the degree of gas formation and the density of the cycle gas.
Table E.	Graphs showing the length of operating periods and the catalyst activities at the end of periods both as functions of the vol per cent paraffin hydrocarbons present in the injection material.
Table F.	Shows monthly values of yields, paraffin hydrocarbon content of the injection, gas formation, etc.

DHD PROCESS COSTS

Frames 763-776

SUMMARY

The second half of 1942 was considered normal operating and the costs are given on the basis of these 6 months. The cost of the finished benzine during this period was as follows:

Raw products	471.97	Reich	marks	per	metric	ton
Operating costs	34.79	"	"	"	"	"
Process costs	27.97	"	"	"	"	"
Price of finished benzine	534.73	"	"	"	"	"

The price is mainly dependent on the cost of raw material. After subtracting the refinery gas gain, the price becomes 516.49 Reich marks per ton.

A) The working costs during the last half of 1942.

1) Use of the chambers. They were on production 80 per cent of the time. Design calculations were based on production during only 68 per cent of the time.

2) The raw materials used and products were as follows:

Raw Material	
Unstabilized benzine from the prehydrogenation	82.16 wt %
Unstabilized 6434 benzine	9.13 wt %
Petrol. oil benzine	6.50 wt %
Crude pentane	1.91 wt %

Products	
DHD finished benzine	72.68 wt %
Rich gas condensate	1.26 wt %
DHD Middle oil	4.22 wt %
Between Products	1.11 wt %
Refinery gas	8.60 wt %
Rich gas	3.65 wt %
Excess gas	7.05 wt %
Cokes + loss	1.43 wt %

3) Working costs in the second half of 1942.

a) Raw material costs

The hydro-benzines costs 350 Reich marks per m. ton, the petrol. oils between 300 and 360, so that the total was 370 Reich marks per m. ton. By-products sold at 40.7 Reich marks per ton of DHD benzine.

b) Operating costs were 34.787 Reich marks per m. ton. Of this the chambers used 23.36, the pre-and redistillation used 7.19.

c) Process costs

The plant is to be amortized in 5 years. Twenty per cent is annually written off. Depreciation costs are based on estimates.

d) So the normal total working costs came to 524.297 Reich marks per m. ton.

DHD PROCESS REPAIRS

Frames 777-813

SUMMARY

Chamber 21

After 2 years of operation the catalyst was changed, and a complete overhaul undertaken. The catalyst was well regenerated and will be tested for activity in LU. The installation of contact 6108 in the refinery furnace was given up. A rotor in a centrifugal blower was repaired. Severe corrosion was found before and after the stripper, but not in the cooler nor in the cold regenerator. Furnace V was replaced by an 8 m DHD furnace, and the high pressure regenerator was replaced by a DHD-regenerator.

1) Catalyst Change

The 7360 catalyst cubes were quite satisfactory. They were well regenerated. Fifteen samples were sent to LU. for activity measurements. The dust formed was 10 to 15 per cent of original catalyst. The 6108 catalyst was not satisfactory, showing a black nucleus.

The refill of 32 m. ton consisted of 21.3 ton 7360 cubes, and 7.3 ton of a used catalyst. The refining catalyst which had been used 2 years was again put in.

Chamber 22

1) Catalysts

The catalyst 7935 (7360 L) was poorly regenerated, the coke increasing toward the furnace exit. This is due to too low a porosity. It should be produced in another form which is more porous.

The 7360 catalyst was quite satisfactory showing no appreciable dusting and good regeneration.

The 6108 catalyst is used as a polymerization catalyst in the refining furnace. These were coked in the nuclei of the pellets. The regeneration was effective only near the surface. It is concluded that the density of pellets is too high.

2) Furnaces

Various repairs and changes were made in the furnace insulations and arrangements.

Chamber 21 April 12 to 17, 1943

The DHD pelleted catalyst in Furnace II rapidly lost activity, and the gas formation increased 2 per cent. Inspection showed that the grates which had been installed had improved the regeneration. But due to non-uniform permeability of the grates "coke nests" had been formed. Unregenerated pellets had been baked together.

The catalyst was replaced by 7360 cubes. The grates in chamber 22 were redesigned.

Chamber 21 December 7 to 12, 1942

Chamber 21 was dismatled to check on SO_2 corrosion and to inspect catalyst 7360 L for coking. Before the stripper no corrosion was found, but in the restricted release passages after the stripper corrosion was evident. In the future lye will be added when the stream contains sulphur. The lower part of Furnace IV was poorly regenerated. To improve the gas circulation two grates were built into the lower part of the furnace.

Various minor repairs are described.

Change of Chamber 22 to a 5 Furnace Chamber

1) A fifth furnace was added in order to raise the production of 13.5 m. ton per hour without increasing the contact load of 0.4 kg/ltr./hour. A new connection was chosen for the preheater.

2) The condition of the walls was good. No appreciable corrosion was found.

3) The catalyst was less completely regenerated than in the reforming chamber. This is probably due to the greater cross section of the furnaces and to the fact that the catalyst is in only 1 chamber.

4) The "cold gas pipe" entering furnace IV from below was found to contain "coke nests", thus indicating that part of the stream is shunted through this pipe. The "cold gas pipe" was removed.

5) A lye cycle was added in order to handle benzines containing up to 0.05 wt per cent sulphur.

The DHD Reforming Chambers. June 4, 1942

These were shut down after 14 months operation. The production cycles had averaged 96 hours and the regeneration 29 hours. They had been operated at a stripper pressure of 35-50 atm. at a rate of 6-8 m. tons per hour. The furnace entrance temperatures had been 515 to 530°C.

In the two upper parts the catalyst 7360 was black. The lower parts contained bright brown catalyst (uniform throughout the pellet). From the 3rd part on the catalyst was well regenerated. The catalyst had a dust layer on top at several places about 3 mm thick. This was mainly iron oxide.

DHD PROCESS

DHD CHAMBER OPERATION

Frames 815-816

New Operations for the DHD Chambers. February 15, 1945

1) Chamber 21 is to be used for refining cracked benzines. The heavy

fraction is to be treated normally. The light fraction (B. P. below 80°) is to be injected directly into the fifth furnace. No difficulties other than rapid catalyst deterioration is expected.

2) Cracking of petroleum oil residue in Chamber 22.

Residue with less than 1 per cent asphalt and with about 20 per cent boiling below 350° are to be cracked. Pilot plant studies suggest adding a stripper from which 50 per cent boils below 530°.

Difficulties are expected due to stoppages by the high ash content, and due to corrosion by the sulphur content.

DHD PROCESS

CHAMBER TEMPERATURES IN THE DHD PROCESS

Frames 817-829

1) Regeneration

The heat capacity (? or power) of the regeneration is very large. In the cold regenerator K-values of 320 Cal/°C hour m^2 are reached and in the hot regenerator the values of 200 are reached. These values are checked by a heat balance calculation. To keep the refining furnace, between Reg. I and II at constant temperature, part of the stream (5-10 per cent) is by-passed around the hot regenerator.

2) Preheater and Blowers.

The total capacity of the 2 blowers is 70-75000 m^3/hour. At a temperature of 480°C at the rotor this volume requires 358 Kw of power. An equal volume produced by 1 blower requires only 264 Kw. So it is best to operate with 1 blower.

3) Efficiency of the Preheater.

A 3,000,000 Cal/hour heater at 500°C has a theoretical efficiency of 69 per cent, but here only 40 per cent was obtained. Further checks are being made to account for this difference.

4) Heat Loss of the Furnace.

The insulation is calculated to have a heat conductivity value of 5.4 Cal/°C. hour. m^2. Furnaces I, II and IV are calculated to lose about 66,000 Cal/hr and the Refining Furnace about 37,000 Cal/hr.

5) Heat of Reaction

From the heat loss etc. the heats of reaction can be calculated:

Furnace I-------------------50 Cal/kg
" II-------------------30 Cal/kg
" IV------------------- Negligible
Total heat of reaction 80 Cal/kg

6) The measured tube wall temperatures agree with the calculated values except in the case of the intermediate preheater.

7) The maximum throughput that can be heated adequately in the preheater is 15 m ton/hr.

THERMAL CONDITIONS FOR THE 6TH PERIOD, 20TH OPERATING HOUR

Pressure, initial, atm	44				
Pressure, exit, atm	31				
Injection, m. ton/hr	12.9				
Entrance gas, m^3/hr	15,000				
Stripper, m. ton/hr	11.25				
Exit gas, m^3/hr	17,300				
Heat values					
Reg. way there Cal/°C	14,470 (Spec. heat 0.75)				
Reg. way back "	14,150 (" " 0.77)				
Preheater "	15,100 (" " 0.80)				
Regeneration					
Temp building °C	81 ← 298 36 → 239	304 ← 494 239 → 413			
Temp. Diff. °C	51	72			
Heat power 10^3 Cal/hr.	2940	2520			
Heat loss "	90	200			
Conductivity Cal/°C, hr, m^2	306	186			
Area m^2	188	188			
Through by-pass and Reg. I come back	5				
Preheater	Main	Preheater	Intermediate	Preheater	
Blast gas m^3/hr	73,000	24,000	21,000	28,000	
Temp. building °C	478 ← 516 410 → 460	520 ← 603 460 → 502	510 ← 642 449 → 509	525 ← 589 468 → 505	
Temp. Diff. °C	61	78	90	69	
Heat Power 10^3 Cal/hr	755	633	905	560	2853
Heat Loss "	190	44	35	47	316
Number of hair pins (?)	10	6	8	6	30
Conductivity Cal/°C, hr, m^2	6.2	6.8	6.3	6.8	6.5
Heating gas, m^3/hr	4125	6.6 x 10^6 Cal/hr			
Lower heating value, Cal/m^3	1600				
Efficiency of the Preheater, %	43.5				
Heat of Reaction	Furnace I	II	IV	Refin. Furnace	
Temperature, °C	502 → 452	509 → 471	503 → 496	304 → 304	
Heat added, 10^3 Cal/hr	755	574	106	---	
Heat loss "	68	68	68	38	
Heat added to product 10^3Cal/hr	687	506	38	-38	
Heat of reaction Cal/kg	-53.2	-39.2	-2.95	+2.95	

$$\underbrace{-53.2 \quad -39.2 \quad -2.95 \quad +2.95}_{-91.4}$$

Aniline Point	
Injection	+38.3
Stripper	- 3.7
Aromatic Content	
After Furnace I	37
" " II	50
" " IV	62
Refining Furnace	71

Period		5	5	6	6
Hours Operated		20	70	20	100
Pressure					
Entrance	atm	51	51	44	45
Exit	atm	35	36	31	30
Injection	ton/hour	12.5	12.3	12.9	12.8
Entrance gas	m^3/hr	18,000	18,000	15,000	15,000
Stripper	ton/hour	13.8	10.35	11.25	(9.9)
Exit gas	m^3/hr	21,400	20,500	17,300	17,500
Regeneration					
Temp. rise on	°C	430	430	413	427
Conductivity values for: Hot Reg.	Cal/°C, hr, m^2	221	220	186	216
Cold Reg.	"	371	300	306	316
Preheater					
Blast stream	m^3/hr	71,000	70,000	73,000	69,000
Through Intermediate Preheater I	"	21,000	20,000	21,000	20,000
Through Intermediate Preheater II	"	29,000	28,000	28,000	29,000
Through Main Preheater	"	21,000	22,000	24,000	20,000
Blast gas temp.					
Entrance to Inter. Preheater I	°C	641	642	642	642
Entrance to " " II	"	589	594	589	603
Entrance to Main "	"	594	594	603	620
Exit	"	485	487	478	487
Conductivity value	Cal/°C, hr, m^2	6.7	6.1	6.5	6.55
Efficiency of Preheater	%	41.3	38.8	43.5	39.1
Heat of Reaction					
Furnace I	Cal/kg	-50.4	-50.5	-53.5	-52.2
" II	"	-27.5	-30.0	-39.2	-28.8
		-69.6	-72.1	-92.4	-79.8
" IV	"	+ 5.4	+ 5.4	- 2.95	- 1.7
Refining furnace	"	+ 2.9	+ 3.0	+ 2.95	+ 2.9

Specifications for the two fuels VT708 and VT348 and some changes in specifications are listed in frames 825-827.

Report 584-46R

Aug. 11, 1943

COVERING THE DHD-CATALYST BY MEANS OF RASCHIG RINGS-PATENT APPLICATION OF I.G.

Frames 830-831

Translation

It was frequently noticed during operation of the DHD reforming chambers in 1941, that even after only a few operating periods the pressure drop across the refining furnace would rise sharply. (Reports 7-3-41 and 8-11-41). Investigation showed that the surface of the catalyst contained a funnel shaped crust of powdered catalyst. It was supposed that the product-saturated catalyst is not very resistant and is pulverized by the entering gas stream. Since during operation, fluid products are encountered only in the refining furnace, whose temperature is 15 mv, this effect did not occur in the other furnaces. (Report 9-12-41).

To prevent the above difficulty a part of the catalyst at the head of the refining furnace was removed during the shut down 9-9 to 9-11-41 and replaced by Raschig rings of burned clay of 25 mm ∅. By means of this covering the gas stream should be slowed down and the crust formation be prevented.

This device has proved to be successful. The pressure drop across the refining furnace has remained constant since then. The covering of the refining catalyst with Raschig rings was applied with success, also in chambers 21 and 22

Later, Ludwigshafen began to recommend covering DHD catalysts with Raschig rings in all DHD furnaces. New installations all use this method.

The above operating improvement was recently submitted as a patent application by I.G. Lu. Since the improvement was suggested and first tried by us, the dates of operation can be used as official dates. We, at that time, did not recognize the necessity of patent protection but now beg to submit the application.

Dehydrogenation Operations

Sch.

To prevent powdering and crust formation, we know that since years ago catalyst fillings of gas-phase furnaces have been covered by a layer of cokes about 20 cm thick. When our operation was begun (middle 1940) it was recognized that the strength of existing cokes was not sufficient, but would yield considerable dust during filling. To cover the catalyst fillings Steinzeug-Raschig rings (20 mm rings) have been used (except for a few trial runs) practically from the beginning (i.e. from August 1940 until today).

To substantiate this we point out that the necessary Raschig rings from the high pressure operation have been held in storage (building 329) and are now ordered from F chs, Lotschet and Son, Baumbach.

The fact that covering with Raschig rings would be suitable, also for the DHD-refining furnaces was told to Dr. Pier in the Fall of 1941, and it was also mentioned that under certain conditions covering of each catalyst field by Raschig rings might be useful. (In reference to a condensation by cold gas in the diaphrams).

OPERATING RESULTS SHOWING THE APPLICABILITY OF DIFFERENT CRUDE GASOLINES FOR THE PRODUCTION OF HIGH PRESSURE DEHYDROGENATION (DHD) GASOLINE, ASSUMING THAT ALL LIGHT GASOLINE ACCUMULATING IN THE FINISHED PRODUCT CAN BE CRACKED

Anonymous

Stettin-Pölitz

March 24, 1942

Frames 833-856

Abstract

This discussion relates to the utilization of the light gasoline fractions originating in the sump and gas phase hydrogenation chambers by a combination of cracking and blending with other gasoline fractions to product aviation and motor fuels. These light gasoline fractions contain 10-35 per cent by volume of C_4's, 30-[illegible] per cent by volume of C_5's, and varying amounts of higher hydrocarbons, depending upon the conditions of separation. Difficulties have been encountered with current operation of the hydrogenation plants because such a relatively large proportion of the charge to the vapor phase hydrogenation was comprised of petroleum and other oil fractions instead of the usual sump phase coal hydrogenation oil product, that the higher boiling gasoline fractions did not have sufficiently high octane number to permit blending with the light gasoline fractions to give fuel of adequate octane rating. As a result, the problem of utilizing these light gasoline fractions arose. Considerable attention is given to the proportions in which the 6434 and 5058 gasolines (the numbers indicate code designations of hydrogenation catalysts used in their preparation) must be produced and treated in the high pressure dehydrogenation step in order to utilize as much of the light gasoline fractions as possible without cracking thereof in blending with the dehydrogenation products to produce a maximum amount of aviation and motor gasoline of suitable quality. A considerable amount of data is presented in tabular form relating to material balances for operation of the hydrogenation and high pressure dehydrogenation plants, as well as to blending of the products for the production of aviation and motor fuels.

DEHYDROGENATION OF SULFUR-CONTAINING BENZINE

Frames 867-8

Abstract

Poelitz: July 30, 1942

This report concerns the manufacture of straight run benzine out of Rumanian petroleum in chambers 21 and 22. The sulfur compounds of the injection pro-

duct yielded SO_2 in the regeneration phase and together with the water from the catalyst deflagration led to SO_2-corrosion in the cold parts (under 200°C) of the apparatus. In Ludwigshaven a 3 per cent soda solution was introduced during the regeneration into the cooler, whereby 10 per cent of the maximum SO_2 accumulation was neutralized, not enough to stop the corrosion. In Poelitz in order to neutralize 1/3 of the maximum accumulation of SO_2, 41.5 m^3 of 3 per cent soda solution was needed each regeneration. On this basis the dehydrogenation of sulfur-containing benzine is disregarded in Poelitz, until the development work in Ludwigshaven is conclusive.

Accompanying the report is a balance sheet concerning the SO_2 accumulation and Na_2CO_3 neutralization.

INVESTIGATION OF A DHD-BENZINE BEFORE THE CONNECTED REFINING FURNACES.

Frames 869-71

Abstract

Poelitz: August 21, 1942

Dr. Wissel and Dr. Schmitt

This report concerns the differences between DHD-benzine before the refining furnace and DHD-benzine after the refining furnace. Some testing points mentioned are stability in light, oxygen-aging test, and the resin test on leaded benzine. Some mention is also made of the stripper products.

Accompanying the report are two tables comparing the DHD-benzine before and after the refining furnaces as to specific weight, aniline point, aromatic substances, naphthenes, paraffins, vapor pressure, iodine number, light test, copper test, doctor test, and boiling curves.

INVESTIGATION OF CONDENSATES FROM GASOLINE REFINING, FROM PREHYDROGENATION AND FROM GASOLINE PRODUCTION UNIT

Frames 872-874

Abstract

This report compares the condensates from gasoline refining, from prehydrogenation, and from the production unit. It is seen from data presented that the 5058 + 7846-W gasoline has a maximum fractionation at 125 to 150°C, practically the same as 5058, prehydrogenation gasoline. The 6434 gasoline has a lower maximum in the range of 100 to 125°C.

After raising the boiling point of the various condensation products, the following is true:

5058 + 7846-W Gasoline:

- Aromatic content, small, remaining practically the same (2 to 3 per cent).
- Naphthenic content decreases considerably (57→25 per cent).
- Paraffinic content increases considerably (40→74 per cent).

5058 Gasoline:

- Large aromatic content, with apparently the maximum in toluene (10.5→19→15.5 per cent).
- Naphthenic content decreases moderately (55→30 per cent).
- Paraffinic content rises moderately (34→58 per cent).

6434 Gasoline:

- Aromatic content rises moderately (6→13.5 per cent).
- Naphthenic content decreases moderately (37→17 per cent).
- Large paraffinic content; increases a little (55→71 per cent).

Since the naphthenic content in all the condensation products was found to be highest in the lower fractions, it is to be expected that the disillation curves will be better for the dehydrogenation of the outlet gasoline in the lower layer.

Report 584-46R

PREHYDROGENATION

Frames 875-877

Abstract

Dehydrogenation of the gasoline at the various plants is reviewed. The peculiarities of the procedures followed, the yield in each case, the upkeep and maintenance of equipment, the difficulties encountered in the production of DHD-manufactured gasoline, prehydrogenation gasoline and sulfur-containing gasoline is discussed. A method is given for keeping down the gas formation (vaporization) during dehydrogenation. The method of neutralizing the SO_2 that accumulates during the manufacture of sulfur-containing gasoline is given in detail. It is mentioned that the excess gas (mostly pentane) formed during the process is washed, causing a better yield of refined gas. The difficulty of supplying chemicals, especially $P_2 O_5$ and I_2, is discussed, the fact being mentioned that some of the tests had to be omitted or others substituted for them.

Dehydrogenation Operations Manager

DHD-GASOLINE

Frames 878-880

Abstract

This is a short discussion of the effect of inadmissable amounts of normal-heptane on the sinking of the over load curve in the fatty area. The normal-heptane seems to be present as a result of a high distillation end point of the first-run DHD-gasoline. Specifications are included for standard DHD-gasoline. There is a detailed statement of the operation periods during which a sinking under the theoretical value occurred in the "overload" curve and also the periods during which time there was an increase in the distillation end point. A graph is included showing the DHD-Gasoline over load curve and the distillation conduct of the charge.

Dehydrogenation Operations Manager
Hydrogenation Plant, Pölitz

DEHYDROGENATION

Frames 881-882

Abstract

In the manufacture of petroleum-distillate gasoline a vast decrease in catalyst activity was observed, which caused a shortening of the effective operation period and a reduction in conductivity of the equipment. The scientists at Pölitz, after performing experiments along this line, were of the opinion that the rapid decrease in catalyst activity in petroleum-distillate gasoline could be traced back to the presence of nitrogen compounds. Experiments were being made to determine a suitable method for the removal of nitrogen compounds prior to dehydrogenation. It is requested in the letter to I. G. Farbinindustrie that the scientists at Ludwigshafen communicate with the hydrogenation plant at Pölitz concerning any experiments they had made in this direction. Data are included on operations during the period.

Hydrogenation Plant, Pölitz

HIGH CASE TEMPERATURES in DHD FURNACES

Frames 883-884

Abstract

This is a discussion of the findings of Dr. Hemann and Dr. Appel of the hydrogenation plant at Pölitz on the most appropriate materials for use in DHD furnaces that must endure high case temperatures, the type of construction for most efficient operation, and the maintenance of this equipment. It gives the names of the types of cement and other materials preferred, including chemical analyses of such.

Technical Division on High Pressure
Zimmermann

DEHYDROGENATION

Frames 885-886

Abstract

A short review of the operations at the Pölitz Hydrogenation Plant ll during 1943 is contained herein. The operation time of the DHD chamber was as follows: dehydrogenation, 85 per cent; regeneration, 10 per cent; and repairs, 5 per cent. It is mentioned that the repair time during this period was very slight. The vaporization curve is reported as 1 wt. per cent lower than in the previous year. It is asked that I. G. Farbinindustrie send their vaporization curves for comparison.

Report 584-46R

REPAIRING OF DHD-CHAMBER

Frames 887-890

Abstract

After 196 operation days without time out for repairs, the chamber was overhauled for damages caused by the high temperatures of the ovens. An outline is given of the damages found and photographs included which illustrate each type.

THE LEUNA DHD PLANT

Frames 891-899

Abstract

This report, which was written in November, 1943, gives a resumé of the effectiveness of the DHD plant in Leuna that had been in operation since August, 1943. This chamber manufactured hydrogenation gasoline from lignite and produced a qualitatively good DHD-gasoline. Gas formation was about two weight per cent higher than had been expected, probably because of the quality of Leuna catalyst used in the process. Of technical interest is the discussion of the construction of the preheater and chamber in a parallel arrangement, the method of saving cable and piping, the simple ventilators used, and the highly automatic efficiency of the machinery.

A detailed description is given of the equipment and machinery including a discussion of the following: chamber, preheater, cooler, stripper, and ventilator groups, service station, machine house, recycle and staple receptacles, and the system of central supervision.

A complete discussion is made of the operations including the various stages in the manufacturing process. The following are discussed: the raw product, the dehydrogenation process, gas formation, catalyst conditions, regeneration, and a description of the qualities, data, physical and chemical properties of the finished product.

Dehydrogenation OperationS
Manager

INFLUENCE OF OPERATING METHODS ON PROPERTIES OF GASOLINE PRODUCED

Frames 900-907

Abstract

Experiments were run at Pölitz to determine the influence of variation in the operating methods on the finished product, the gasoline produced. The details are included for the experiments that were run. The following conclusions were made from the results: (1) the operation methods of the DHD-chambers upon materials with high and low aromatic content have a decided influence on the course of the "overload" curve, especially in the excess fuel range; (2) the extract from a gasoline having a high aromatic content, and the gasoline itself, has a high mixed value; (3) the octane numbers and analysis of the residual gasoline do not give exact results; therefore, an investigation of the "(überlademassige)" was necessary; and (4) the analysis of the extract could prove to have great significance in answer to most of the questions (concerning the effect of operation methods on the product) not answered by these investigations.

* "uberlademassige" - probably "overload mass"

DHD AROMATIC FORMATION

Frames 908-909

Abstract

In this letter to Dr. Wissel from the Hydrogenation Plant at Pölitz, data are given showing the production of the DHD-reconstruction chamber from March 13, 1941 to September 30, 1941. This includes the aromatic formation during this period. The average new aromatic formation is seen to be 0.128 kg/Ltr. of catalyst.

DEHYDROGENATION OF HEAVY GASOLINES

Frames 922-924

Translation

Type of Gasoline	5058/6434 Gasoline from Liquidfied Coal	Russian Gasoline	Technical Shale Oil Gasoline
Outlet Gasoline			
Distilling Range	110°-180°	95°-156°	84-202°
Spec. Grav./15°	0.786	0.744	0.780
Aniline Point 1	+40°	+52.2°	+42°
Aniline Point 11	+52.5°	-	-
Beginning Dist.	110°	95°	84°
%, 90°	-	-	-
%, 100°	-	4.5	3.2
%, 110°	-	-	-
%, 120°	-	75	-
%, 130°	-	-	-
%, 140°	-	91.5	-
%, 150°	73.8	-	54
%, 160°	-	-	-
%, 170°	-	-	-
%, 180°	-	-	81.5
Endpoint	180°	156°/97.8	202°/98.5
Residue	-	1.5	-
Loss	-	0.7	-
Summary:			
Unsat., Wt. %	-	1.5	1.0
Aromatic	-	3.0	15.5
Naphthenic	-	-	32.5
Paraffinic	-	-	51.0
Knock Value,			
Residual Meth.	64.5	67.2	
Motor Meth.	60.0	68	
Motor Meth. + 0.12% Pb	84.5	84	

HD-Gasoline	Coal Gasoline	Russian Gasoline		Shale Oil Gasoline		
(°C run to)	165°	163°	162°	243°	256°	220°
Total Gasoline						
Spec. Grav./15°C	0.786	0.784	0.796	0.808	0.817	0.833
Knock Value,	94.3	92	96	-	-	-
Residual Method						
Res. Meth. + 0.12% Pb	105.0	-	-	-	-	-
Motor Method	80.5	79.5	86	-	-	-
Motor Meth. + 0.12% Pb	91.5	91	-	-	-	-
Aniline Point 1, °C	-2.5	+2°	-7.5°	+2.8°	-10° "(ber)"	-20.0°
Aniline Point 11, °C	+55.8°	+59.5°	+63.0°	+60.0°	+60.5°	+61.5°
Distillation Curve						
Beginning	52°C	43°C	36°C	42°C	40°C	48°C
%-50°C	-	-	-	1.2	1.5	-
60°C	2.0	2.0	5.5	3.0	3.2	2.0
70°C	7.5	3.5	11.0	5.0	6.0	4.0
80°C	16.8	8.0	17.0	8.0	9.5	8.0
90°C	28.0	16.0	24.0	13.0	18.0	14.0
100°C	41.0	26.0	32.0	22.0	22.0	21.0
110°C	53.0	43.0	43.5	32.5	30.0	30.0
120°C	64.5	60.5	59.5	41.5	38.0	40.0
130°C	74.0	75.5	75.0	51.0	46.5	48.0
140°C	83.0	86.0	87.0	59.0	55.0	57.0
150°C	90.0	92.5	93.5	68.0	64.0	67.0
160°C	94.0	95.0	-	74.0	70.0	75.0
Endpoint/%	165°/96.5	163°/96.0	162°/97.0	243°/98.5	256°/97.5	220°/98.0
Residue	1.0	1.0	0.5	1.0	1.0	1.0
Loss	2.0	3.0	2.5	0.5	1.5	0.8
Unsat., Wt. %	3.0	5.5	5.0	1.5	4.5	4.0
Aromatic	55.5	55.0	67.0	54.5	67.0	77.5
Naphthenic	19.0	13.5	6.5	14.5	8.5	5.0
Paraffinic	22.5	26.0	21.5	29.5	20.0	13.5
I_2 Number	-	-	-	-	12.6	-

	Coal Gasoline	Russian Gasoline		Shale Oil Gasoline		
Residue Gasoline (Aromatic free)						
Yield, Wt. %	43.5	46.0	31.0	45.3	34.5	25.0
Spec. Grav./15°	0.703	0.711	0.677	0.726	0.707	0.713
Knock Value Residual Method	75.0	-	-	-	-	-
Res. Meth. +0.12% Pb	90.5	-	-	-	-	-
Motor Meth.	76.5	-	-	-	-	-
Motor Meth. +0.12% Pb	90.0	-	-	-	-	-
Aniline Point 1	55.4	-	62.3	57.0	56.5	54.0
Aniline Point 11	56.4	-	-	60.5	61.0	61.5
Distillation Curve						
Beginning	42°	42°	35°	32°	37°	34°
%-50°C	2.5	-	-	1.0	2.0	3.0
60°	12.5	5.0	23.0	3.0	6.0	5.0
70°	31.0	10.0	40.0	5.5	13.5	11.2
80°	48.0	21.0	53.0	11.2	26.0	24.0
90°	67.0	35.0	72.0	23.0	42.8	40.0
100°	80.0	53.0	80.0	37.0	57.0	53.0
110°	86.2	74.0	88.0	49.0	66.0	62.5
120°	90.0	87.0	91.0	60.0	73.0	71.0
130°	93.0	94.0	94.0	69.0	70.5	76.0
140°	95.0	-	-	74.5	83.0	81.0
150°	-	-	-	80.0	87.0	87.0
160°	-	-	-	86.0	90.0	88.5
Endpoint/%	147°/97.5	138°/97.5	137°/97.0	194°/96.0	188°/96.0	162°/93.0
Residue	1.0	1.5	1.0	1.0	1.0	1.2
Loss	1.5	1.0	2.0	3.0?	3.0?	5.3?
Unsat., Wt. %	2.0	-	-	1.9	2.5	1.5
Aromatic %	1.0	-	-	3.0	4.0	7.0
Naphthenic %	45.0	-	-	30.0	28.0	25.5
Paraffinic %	52.0	-	-	66.0	65.5	66.0
I_2 Number	-	-	-	-	13.0	-

Aromatic Extract	Coal Gasoline	Russian Gasoline		Shale Oil Gasoline		
Yield, Wt. %	56.5	54.0	59.0	64.7	65.5	75.0
Spec. Grav./15°C	0.870	0.8685	0.862	0.896	0.877	0.889
Aniline Point [illegible]	56.2°	-50.4	-61.0°	-64.0°	-54.5	-56.5
Benzol, Vol. %	7.3	-		7.8	10.0	8.3
Toluol, Vol. %	46.0		-	18.2	18.1	25.2
Xylene, Vol. %	32.1	-		25.7	26.4	27.6
High Arom. Vol. %	12.7	-	-	7.0	11.9	37.5
Loss	0.3			1.3	1.7	1.3
% C	87.69					
% H	12.02					
[illegible] H/100C	13.74					

Main Laboratory, Pölitz

THE FURNACE INSULATION

Frames 9[illegible]-978

Abstract

This is a report from the Hydrogenation Plant (Pölitz) on the difficulties experienced in their DHD-plant as a result of high temperatures occurring in the ovens of the DHD-chambers. A detailed description is made of the operation history of each oven, the portion of each oven in which these temperatures occurred, the time lost because of these high temperatures, etc. All possible reasons for the increase in temperature within the ovens is discussed, with a description of the investigations made up to that time of the causes. After an investigation of the material used in building these ovens, the following facts were noted: (1) the quality of the stone had changed considerably in the two years of operation (in room weight and resistance); (2) the stability of the insulation stone against hydrocarbons could not be guaranteed; (3) it had not been proven whether or not the stone acted as a catalyst, thus causing the extraordinarily high temperatures; and, (4) from investigations, it seemed to be a great disadvantage that the higher percentage of the cement in the stone did not adhere to the mantle (casing) of the oven. It was advised that "[illegible]" be used in the ovens at the Leuna DHD plant.

Report 584-46R

DHD FURNACE

Frames 973-974

Abstract

A group of telegrams interchanged between Dr. Zimmermann of the Leuna DHD-plant and Dr. Berger of the Pölitz plant discusses the suitability of various types of material for the construction of DHD-furnaces. The results both favorable and unfavorable, are given for "Rolandhütte" cement, a stone with a high percentage of cement, and alumina-cement for the outside layer of insulation. These telegrams compare the various types of insulating materials for DHD-furnaces, but do not seem to indicate which one the Germans preferred.

Report 584-46R

INVESTIGATION OF THE AROMATIZATION ACTION OF THE 7019-CHAMBER 3 DURING THE STARTING PERIOD.

Firront

Poelitz: March 5, 1941

Frames 978-95

Abstract

For the purpose of finding out the aromatization action of chamber 3, the same aniline point determinations were carried through on daily cross-section samples of the injection products on the one hand and of the stripper product on the other hand, as well as on certain fractions. The preliminary results are plotted graphically on diagram sheet I, of the graphs and tables, which accompanied this report. From this the following determinations are to be inferred:

1. The aniline point of the total stripper lies in all cases higher than that of the injection products.

2. While the injection product in the whole as in the part shows no noticeable change in the aniline point, the aniline point of the stripper decreases progressively with the time, and this decrease is especially found in the lower fractions.

3. From the aniline points of the lower, that is, the benzine fraction, it is evident that the aromatic formation in the first descent is extended to the fraction 110-140°C. The boiling curve of benzine, prepared in diagrams II and III shows that this fraction is replaced by about 20%.

4. The aniline points of the stripper middle-oil fractions show, in contrast to the corresponding injection-product fraction, a distinct hydrogenation.

For ascertaining the mode of action of the refining furnace, further stripper samplings were taken out after the third furnace, and likewise examined for aniline points. The data obtained from this are compared in diagram IV to the corresponding investigation results of normal stripper, that is, stripper which accumulates after the refining furnace. Moreover, the benzine investigations before and after the refining furnace were carried out, the results of which are to be found in the tables following the diagrams.

The valuation of the existing investigation material --from a technical basis only a limited number of separate tests could be taken after the third furnace -- shows that the aromatic content of the total stripper as well as of the fractions before and after the refining furnace in general show no noticeable difference. The stronger deviations of the aniline points of the fractions 110-140° from 21.2 could depend upon the fact that in the one case it concerns a momentary sampling and in the other it concerns a daily cross-section sampling. In the same way can the drop of the aromatic + olefin content from 40 per cent in benzine behind furnace 3 to 38 per cent in benzine behind furnace 4 be explained by the decline of the olefin constituent, since a simultaneous decrease of the iodine number from 3.06 to 1.41 was determined. On the other hand on February 25 there was observed in the benzine behind furnace 4 an increase of the aromatic-olefin content, whereby there also must have occurred here a drop of the olefin components corresponding to the iodine decrease from 2.45 to 1.46,

—Report 584-46R

CATALYST 7019 (SCHOLVEN)

URBAN

Scholven: January 15, 1941

Frames 996-8

Abstract

In semi-annual large-scale operations in the double-furnace system = 16 m^3 catalyst volume, the catalyst 7019 at 250 atm H_2-pressure proved industrially all right. The method of operation is relatively simple. The catalyst is very sensitive at low temperatures toward CO-fluctuations in the gas, and in the case of sudden CO-breaking down, it is inclined to transition. In the case of treatment of coal-liquefaction middle-oil, there is obtained, with refluxing of suitable B-oils, a 165° aviation gasoline, stabilized to 0.4 vapor pressure, with a yield between 0.20-0.25 in 25-30 per cent benzine concentration and a total yield of 75-80 per cent by wt. of the inserted middle oil. The benzine contains about 50 per cent aromatics with a specific weight around 0.80/15°C. For the present, the too-high iodine number in benzine above 4 g/100 g and the too-high oxidation test above 10 mg/100 cm^3 after leading permit only a limited storage of the leaded-benzine.

By means of the connection of a refining contact between the regenerators of the 7019 system, the iodine number and the oxidation tendency of the 7019-benzine should be diminished.

However, the large-scale operations at Scholven has confirmed most extensively the values of the short and semi-technical experiments of the I. G. Farben division of the high-pressure experiment

GASOLINE FROM CV2b AND DHD PROCESSES
DR. HIISCHBERGER AND DR. SCHMITT

Translation
Frames 1011-1018

April 1, 1941

(1) Conditions for suitable overloading.

(a) Aromatic content (excluding unsaturated) of total benzine: 50 per cent by vol. = 55 per cent by wt.

(b) Knocking value unleaded from aromatic-free residual benzine: at least octane number of 62 (Scholven: 63-65)

(c) Boiling curve: at least 45 per cent at 100°C

The necessary aromatic content is easily attainable, and the residual benzine is better in the case of use of tar-sump middle oil than in the case of liquefaction middle oil. Scholven uses in the fresh product of the CV2b-chamber less than 1/3 in tar-sump middle oil.

(2) Development of the aromatic-containing benzine.

As yet the CV2b-benzine was mixed with iso-octane for the finished refined product, whereby the vapor pressure was lowered from the maximum 0.4 by the mixing. The efforts are no longer going to substitute the iso-octane mixing components by means of 6434-light benzine for a C_3-fuel. Consequently it will probably be necessary, that the CV2b-benzine as such be furnished at a lower vapor pressure (about 0.35) by the removal of the total amount of butane. The laboratory of I. G. Farben Industries experimented for a time, in a stabilization column with connected Bunsen-Schillings apparatus for continual determination of the vapor density, to make the CV2b-benzine practically (under 0.1%) butane-free.

(3) Oxygen Bomb Test.

The development of the bomb test proceeded somewhat in the following manner: A long time ago it was noticed on the front that the containers with leaded benzine (gasoline) contained a lead sludge. Hereupon the use of corresponding benzine was temporarily stopped by RLM. The RLM authorized the D VL to institute a series of experiments under cooperation of the benzine firm in relation to the proportion of leaded benzine. The results of this series of experiments were discussed in Berlin at the RLM on March 24, 1941.
Result: The bomb test of the leaded benzine in the present form is useless, since it gives a false picture. The tests not only scatter the different testing points, but also in the case of these testing points in very wide limits from under 10 up to 70 mg. Also lead sludge I was herewith quantitatively determined by means of filtration through a glass drop funnel. Moreover, it was observed, that alkaline glass showed a higher steam test, while trace of acids in the benzine yielded a lower steam test. Recently experiments were instituted also by RLM with inhibitors, as, for example, lecithin. It appeared as if the quality requirement "without foreign addition" would fall within any predetermined time and inhibitors would be admtited. Appropriately, HWP made leaded bomb tests in time intervals from deposited benzine, unleaded as well as leaded.

(4) Discussion.

I. G. Farben Industries sent, on Tuesday, April 1, 1941, Dr. Lajus as well as the laboratory assistant Brown for aromatic-extraction experiments at Poelitz. Since Thursday, Dr. Hirschberger has been in Poelitz for the rest of the week. Since at present the knocking-value determinations of the I. G. Farben Industries are not in order, Dr. Hirschberger asked to be informed concerning the sample shipment of our octane number.

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Dr. Hirschberger received from the chief laboratory the following:

CV2b-quality directions of the RLM of September, 1940; 2 curves for the aromatic determination from the aniline point;

CV2b-benzine classification of March 29, 1941; HWP-proposal for aromatic determination of March 29, 1941;

Characteristics of the required lye, classification of March 29, 1941.

CV2b-BENZINE RESEARCH

Poelitz: March 29, 1941

Product:	Quality Determinations				
Benzine type		CV2b	CV2b + DHD	CV2b	CV2b + DHD
Chamber		3 Stabilization Plant 1	3 + 1 C_2-distillation unstabilized	3 Stabilization Plant 1	3 + 1
Tank					202
Date:		Feb. 25, 1941	Mar. 16, 1941	Mar. 22, 1941	Mar. 27, 1941
Amount present:					1400 m.t.
Receiver:		I. G. Farben	I. G. Farben	Intava.	I. G. Farben, Intava.
Test Amount:		50 liters	1 liter	50 liters	50 liters
Test Completion:		Feb. 28, 1941	Mar. 17, 1941	Mar. 22, 1941	Mar. 27, 1941
Test Number:		V576	BD8	V702	V735a
Knocking value	at least octane no. 77	74.6	78.1	78.2	77.3
+0.12 % lead	" " " " 90	90.5	89.7	90.4	90.0
Specific weight/15°	0.790-0.810	0.7715	0.802	0.7915	0.7944
Boiling Curve:					
Beginning:	45-50°C	39°	45°	43°	42°
10% product	80°C	65°	91°	72°	74°
20% "		79°	101°	85°	86°
30% "	100°C	89°	106°	93°	94°
40% "		96°	113°	100°	101°
50% "		103°	120°	107°	108°
60% "		111°	122°	115°	116°
70% "		122°	135°	125°	127°
80% "		136°	145°	135°	136°
90% "		151°	153°	147°	148°
95% "	160°C	161°	161°	155°	156°
Endpoint/%	165°C	164°/97%	165°/97.5%	171°/92.5%	161°/98%
% at 70°		13	3	9.5	8.5
% at 100°		44	19	40.0	39.0
Residue % by vol.		1	1	1	1
Waste % by vol.	not above 2.0 % by vol.	2	1.5	1.5	1

(continued)

Product:	Quality Determinations				
Benzine type		CV2b	CV2b + DHD	CV2b	CV2b + DHD
Chamber		3	3 + 1	3	3 + 1
		Stabilization Plant 1	C_2- distillation unstabilized	Stabilization Plant 1	
Tank					202
Date:		Feb. 25, 1941	Mar. 16, 1941	Mar. 22, 1941	Mar. 27, 1941
Amount Present:					1400 m.t.
Receiver:		I. G. Farben	I. G. Farben	Intava.	I. G. Farben Intava.
Test Amount:		50 liters	1 liter	50 liters	50 liters
Test Completion:		Fe. 28, 1941	Mar. 17, 1941	Mar. 22, 1941	Mar. 27, 1941
Test Number		V576	BD8	V702	V735a
Aniline point I:		18.2	+3	+2	1.3
Aniline point II:		51.6	52.4	49.5	48.5
Vapor pressure at 37.8°:	not above 0.4	0.47	0.4	0.33	0.36
Evaporating test leaded:					
before aging	5 mg. unleaded			2.3	
after aging					
resin	not above 10			4.5-4.5	5.1-10.1
lead sludge				5.4-7.7	
Sulfur content	not over 0.05%	0.013			0.006
Iodine number/100 g	not over 4 g	0.82	3.8	5.3	5.8
Melting point:	under-60°C				under -60°
Corrosion:	without	neg.	neg.		neg.
Unsaturated % by vol:		2	3	1	2.0
Aromatic substances % by vol:		31.0	48.	44.5	44.5
Naphthenes % by vol:		41.0	28.5	37.5	38.0
Paraffins % by vol:		26.0	20.5	17.0	13.5

CV2b-AND DHD-BENZINE

Poelitz: March 27, 1941

Benzine type	CV2b	CV2b	CV2b +DHD	DHD
Chamber/tank	3	201	201	1
Distillation	Laboratory	Technical	Technical	Laboratory
Date	March 18, 1941	March 18, 1941	March 19, 1941	March 18, 1941
Test Number	V679	F71	F73	V679
Knocking value	78.9	74.6	77.6	82.5
+0.12% Pb	90.3	89.3	90.8	91.2
Specific wt./15°	0.792	0.776	0.787	0.804
Boiling curve				
Benginning	40°	45°	44°	53°
10% Product	71°	70°	75°	91°
20% "	84°	81°	90°	105°
30% "	91°	89°	99°	113°
40% "	97°	96°	105°	121°
50% "	103°	103°	113°	126°
60% "	109°	111°	121°	132°
70% "	118°	120°	130°	137°
80% "	131°	132°	139°	143°
90% "	153°	145°	148°	150°
95% "	170°	154°	157°	158°
End Point/%	175°/97	160°/98	162°/98	168°/98
Residue % by vol.	1	1	1	1
Waste % by vol	2	1	1	1
% at 70° " "	9	10	7	1
% at 100° " "	47	47	31	17
Aniline Point I	+6	+18.3	+8.1	-3
Aniline Point II	+48	+50.7	+51.9	+59.5
Vapor pressure/38°C	0.44	0.36	0.39	0.25
Evapn. test leaded				
before aging	0.7-1.6	2.0-2.9	0.7-1.7	0.4-2.0
after aging				
Resin +Pb II				
/Resin	4.1-9.3/3.5-8.5	6.2-10.1/6.0-8.8	3.0-4.7/3.5-4.7	7.5-17.1/2.0-7.2
Iodine number	2.2	1.6	2.3	3.5
Corrosion	without	without	without	without
Doctor test	negative	negative	negative	negative
Unsaturated %				
by vol	2.5	1	1.5	2.5
Aromatic substances%				
by vol	42	32	38.5	55
Naphthenes% by vol	40.5	43	36	14.5
Paraffins % by vol	15	24	24	28

CV2b - AND DHD-STRIPPER: BRIEF LABORATORY INVESTIGATION

Poelitz: March 27, 1941

Product	CV2b-Stripper	DHD-Stripper
Chamber	3	1
Date	March 16, 1941	March 16, 1941
Tank number	BD7	BD6
Amount	1 drum	1 drum
Total product		
Specific weight/15°	0.859	0.824
Aniline point	-0.7°	- 14°
Boiling curve		
Beginning	55°	55°
-70°	1	1
-100°	4.5	5
-120°	7.5	13.5
-140°	12	35
-160°	18.5	65
-180°	30	87
-200°	50	95
-225°	75	-
-250°	85.5	-
-275°	93	-
-300°	96	-
Endpoint/%	308°/97.5	219°/97.5
Residue	1	1.5
Waste	1.5	1
Benzine at 165°C		
% by vol	20	75
Aniline point I	+2	-7
Aniline point II	+48.2	+60
Unsaturated + Aromatic % by vol	40	57
Iodine number	2.5	3.7

CHARACTERISTICS OF THE TECHNICALLY NEEDED LYE USED FOR THE LYEWASH OF CV2b-OR DHD-BENZINE.CALCUALTION OF DEGREE OF SATURATION. POELITZ: MARCH 27, 1941.

Total NaOH:	109.6 gr/l
Free NaOH:	79.6 "
CO_2:	4.8 "
H_2S:	9.8 "
Phenols:	4.0 "
Degree of Saturation:	29
Saturation degree 100 =	Na_2S
Saturation degree 200 =	NaSH

The lye needs to be used only up to a saturation degree of 80-90.

Calculation of the degree of saturation:

40 (mg: NaOH)	:	34 (mg: H_2S)	=	79.6 (free NaOH)	:	x (still usable H_2S)
x	:	200	=	9.8 (used H_2S)	:	y (saturation degree)

TECHNICAL PRODUCTION CONDITIONS FOR CV2b 1018

(1) Purity: The fuel must be water-clear, free from undissolved water and acid, and may contain no solid foreign material.

(2) Classification: Hydrogenation benzine without additions.

(3) Resistance against knocking: Octane number without addition of tetraethyl lead at least 77. By addition of at most 1.2 cm^3 tetraethyl lead to 1000 cm^3 benzine must attain at least an octane number of 90.

(4) Density/15°C: Between 0.790 and 0.810 kg/l.

(5) Boiling behavior: Boiling begins at 45°-50°C.
There must distill over:
10% by vol. at 60°C
30 " " 100°C
95 " " 160°C
Boiling ceases at 165°C
Distillation loss not over 2% by vol.

(6) Acid content: The residue remaining after the distillation into the flask should not react acidic.

(7) Vapor pressure: Not over 0.4 at 37.8°C.

(8) Evaporation-residue: In the case of the evaporation of 100 cm^3 of the fuel should at most 5 mg of residue remain.

(9) Sulfur content: Not over 0.05% by wt.

(10) Iodine number: Not over 4 g/100 g.

(11) Melting point: The melting point of the fuel cooled to crystalization may not lie above -60°C.

(12) Corrosion: No gray or black spots or corrosion in the case of the copper streak method.

(13) Aging test: The evaporation residue after 4 hours in the bomb at 100° and 7 atmospheres excess pressure of oxygen with and without 0.12% by vol. of tetraethyl lead may not exceed 10 mg.

PREPARATION OF CV2b-GASOLINE
DR. HIRSCHBERGER AND DR. SCHMITT

Poelitz: February 17, 1941

Frame 1019

Abstract

This concerns the sampling and testing of CV2b-gasoline. The sampling point was between the aromatization catalyst and the hydrogenation catalyst. In one case the benzine had been steeped in lye before the stabilization and in another after the stabilization.

For shipping, the investigation samples to testing laboratories, the unleaded samples were sent in pure iron bleaching containers (black sheet and not zinc-coated), without leather packing, since leather is fat-containing.

There followed a series of investigation data on the knocking value, iodine number, aromatic content, vapor pressure/37.8°C, end point, freezing point, and turbidity point.

INVESTIGATION OF PRODUCTS
DR. SCHMITT

Poelitz: August 8, 1941

Frames 1020-32

Abstract

This consists of three sets of tables (titled Short Investigation of Chamber 3) with identical headings, but different data. These are three daily reports on the oil, gas, and water products from chamber 3. Following this is a general, summary table with complete date on all material entering and leaving chamber 3. This last section is translated below in full.

COMPLETE INVESTIGATION OF PRODUCTS FROM CHAMBER 3

Raw material: R 401/6 sm + Recycle (30:70)

Fresh product:	
Specific weight/15°	0.981
Boiling curve	
Beginning	178°C
10%	220°C
20%	231°C

(continued)

Boiling curve	
30%	237° C
40%	247° C
50%	258° C
60%	272° C
70%	287° C
80%	300° C
90%	317° C
95%	325° C
End point/%	338°/98%

Injection mixture:

Specific weight/15°	934
Aniline point	3.5°
Boiling curve	
Beginning	182°
200°C	22.5%
225°C	47.5%
250°C	66.5%
275°C	80.0%
300°C	91.0%
325°C	98.0%
Endpoint/%	329°/98.5%
Phenole	1.3

	Specific wt.	Aniline Point
180-210°	0.883	-13.8°
210-230°	0.904	-19.5°
240-270°	0.946	-26.9°
280-310°	0.992	-37.9°

Elementary analysis:	
% C	87.90
% H	10.23
% O	1.55
% N	0.23
% S	0.09
gr H/100 C	11.63
H dispersion	11.34

Stripper product:

Specific weight/15°	0.872
Aniline point	-12.5°C
Boiling curve	
Beginning	53°C
70°C	1.0%
100°C	4.0%
120°C	7.0%
140°C	12.0%

(continued)

Stripper product:

Boiling curve	
165°C	20.5%
180°C	29.0%
200°C	51.0%
225°C	74.0%
250°C	85.5%
275°C	92.5%
300°C	96.0%
Endpoint/%	321°/97%
Residue	1
Condensed gas	1.5
Waste	0.5
Elementary analysis:	
% C	87.00
% H	10.85
% O	0.12
% N	0.007
% S	0.023
gr H/100 C	12.19
H dispersion	12.12

Stripper benzine at 165°C (stabilized):

Yield	20.6% by wt.
Knocking value	79.2
" " + 0.12% Pb	90.9
Specific weight/15°	0.796
Boiling curve	
Beginning	40°C
70°C	7.0%
80°C	13.0%
90°C	22.0%
100°C	38.0%
110°C	53.0%
120°C	65.0%
130°C	75.0%
140°C	83.0%
150°C	89.0%
160°C	93.5%
Endpoint/%	174°/98%
Residue	1
Waste	1
Vapor pressure/37.8°	0.41
Aniline point I	-8.0°C
Aniline point II	+48.3°C
Unsaturated + Aromatics	53.0% by vol.

(continued)

Stripper benzine:

Naphthenes	36.5% by vol.
Paraffins	10.5% by vol.
Iodine number	about 3
% C	88.30
% H	11.60
gr H/100 C	13.14

Middle oil at 165°C:

Specific weight	0.904
Aniline point	-12°
Boiling curve	
Beginning	176°
180°C	6.0%
200°C	47.0%
225°C	72.0%
250°C	85.0%
300°C	96.5%
Endpoint/%	324°/98.5%
Residue	1.0
% C	89.00
% H	10.84
gr H/100 C	12.18

Stripper water:

Phenols	20 mg/l
NH_3	20.2 g/l
Cl	4 mg/l
Fe	19 mg/l

Gas investigations:

	Circulation gas			
Product	Entrance	Exit	Poor gas	Rich gas
Test number	637	638	635	636
CO_2	0.4	0.5	0.1	0.1
H_2S	0.0	0.0	0.0	0.0
H_2	90.5	86.6	68.5	30.8
N_2	4.5	5.6	5.5	2.6
CH_4	1.2	1.3	2.1	0.4
C_2H_6	2.3	4.3	18.4	24.9
C_2H_4	0.4	0.3	0.0	0.0
C_3H_8	0.6	0.8	3.5	22.4
C_3H_6	0.0	0.0	0.0	0.0
i-C_4H_{10}	0.1	0.5	0.5	13.9
i-C_4H_8			0.0	0.1
n-C_4H_{10}			1.3	2.9
n-C_4H_8			0.0	0.0
C_5 + higher	0.0	0.1	0.1	1.9

HYDROFORMING PLANT AT MOOSBIERBAUM

DR. STAFFEN

July 20, 1943

On July 8 and 9, 1943, the HF plant at Moosbierbaum near Vienna, which was in operation a year on July 16, 1943, was inspected.

Raw and finished products.

The plant produces 5000 mt/month of HF-benzine from Hungarian and Rumanian auto benzine (or gasoline). The latter have the following characteristics:

Raw benzine:	
Boiling progress:	50-175°C
-100°C:	25-30% by vol.
Paraffin hydrocarbons:	40-50% by vol.

It is especially required that the crude benzine possess satisfactory constituents up to 100°C.

In the predistillation a cut was made from the crude benzine of 10 per cent by vol. in the first run-boiling endpoint about 90°C. The residue of the distillation was charged to the furnace plant into a stripper with an aniline point of -13°C (directly determined). This contained 63 per cent by vol. of aromatic substances. It was stabilized at 10 atmospheres. The gas which accumulates is used as fuel gas to burners. The stabilized stripper is washed with caustic solution, treated with about 3/4 per cent by wt. concentrated H_2SO_4, and re-treated with water and caustic. Then, with caustic and ammonia addition in the redistillation, it is decomposed into benzine and residue.

About 3 per cent by wt. accumulates as residue. This is liquid, of red color, contains about 95 per cent in aromatic substances and is mixed with a diesel oil. The benzine of the redistillation is blended with the first run of the pre-distillation into the refined benzine, of the following specifications.

HF-benzine:		
-100°C	30-35% by vol.	
-165°C	95% by vol.	
Vapor pressure	0.5 atmospheres excess pressure	
Aromatic substances	55-56% by vol.	
Iodine number	about 12	1035
Color	water clear	

The overloading curve at 2 = 0.8 is sufficient, and at 2 = 1.1 it lies about 0.5 - 1.5 pme above the standard value of the C_3 II_d fuel. The overloading tests are carried out by the Iravemünde laboratory. The difficulty, to get the overloading curve into a profitable area, occurs especially, when too-few low-boiling hydrocarbons are present in the crude benzine.

HF plant.

This includes the furnace, engine room, and operating area, and the distillation units. All together 168 men are employed, of which 100 are employed in the furnace operation, 17 in the laboratory and the rest in the general process work.

Equipment:

Remarkable for the furnace plant are the six short, compact furnaces, which are isolated in a series of concrete structures. The furnaces have a pear shape and are almost as wide as they are high. Each holds 7 1/2 m^3 of catalyst, which is regulated in a compartment. Three types of catalyst were employed.

Catalyst A consisted of alumina, saturated with 5 per cent molybdic acid. It has a spherical shape and is less active. Catalyst B is based on an especially active alumina, which likewise contains 5 per cent molybdic acid. It has a cubic shape of 8-10 mm length and shows medium activity. Especially active is Catalyst C, likewise cubic-shaped, with a very active alumina content saturated with 10 per cent molybdic acid. Furnaces I and II are filled with the same amount of catalyst A, B, and C. Furnaces III and IV contain only the catalysts B and C, and furnaces V and VI only catalyst C. Each furnace is equipped with 8 automatic control valves-oil-controlled slide-valve of the firm Schäfer and Buddenberg. For the time being two control valves, connected behind one another, are located on the two inlet headers and the two outlet headers at the foot of the furnace. The furnaces I, III and V or furnaces II, IV, and VI are connected as groups either with the product circulation or with the regeneration circulation. Furthermore, remarkable are the large insulated transfer lines, of 300 mm diameter, the large automatic controlled engine, with which the plant is operated, and the two furnace housings of conventional type. The later are gas-burning tube heaters, which divide the product in two parallel streams. The tubes have a diameter of 135 mm. 1036

In the engine room are located 3 gas-circulation pumps with belt drive, whose pressure is from 15 to 25 atm. The largest gives 9500 m^3/hr and the other two 4500 m^3/hr. For the injection of the product a circulating pump of 15 m^3/hr output is used, which is operated without re-expansion, and should prove to be good. Further, the engine room contains two high-revolving bellows, which revolve 13,000 m^3 of gas each hour, but which are not tight. In addition there are two vertical two-stage compressors with a capacity of 1500 m^3/hr each, in which air is condensed as inert gas or waste gas.

Process:

In the present process, one furnace group, which is connected with the product circulation, runs for about 10 hours on dehydrogenation, while the second furnace group connected with the regeneration circulation, is operated simultaneously in regeneration, which is necessary for about 9 hours. At the end and at the beginning of the regeneration both groups run for about 1/2 hour on dehydrogenation.

Dehydrogenation:

In the case of the dehydrogenation, 9500 m^3 of circulation gas are pumped hourly with a pressure of 25 atm. into the system, which is mixed with 15 m^3 of heavy benzine, charged by a circulating pump. Product and gas are charged through 3 horizontal heat exchangers, by conventional method. The mixture then goes into the main preheater. This, conventional heater contains 270 tubes, each of 6.75 m length and 135 mm width. The tubes have a total length of 1800 m and are arranged in two parallel rows. The main preheater raises the product to a temperature of2-8 mv.

The flow through the first dehydrogenation furnace, has a temperature drop of 3 mv . The temperature of the product in the second tube heater of the same construction, which contains 60 tubes with a total length of 400 m, is brought up to 28.2 mv. In the second dehydrogenation furnace the temperature drop is about 1.5 mv. The third tube heater, likewise consisting of 60 tubes, is built with the second in one furnace chamber. In it the temperature of the product is brought up to 28.3 mv, at which point the product enters the third furnace. In this--the last dehydrogenation furnace--occur strong exothermic reactions. At the exit the temperature is about 0.5-1.5 mv higher than at the entrance. Also this temperature increase rises during the 10 hours of the aromatization period.

1038 The plant was so operated, that the entrance temperatures of the second and third furnaces increased constantly. A stripper test is taken after each furnace, which determines the aniline point. This lies after furnace I at 30°C, after III at 10°C. Leaving the last aromatization furnace, the product runs through a vapor heater, which yields about 4 mt/hr of vapor at 18 atm. This is recovered soon after by means of a high-pressure regenerator.

The product enters into three heat exchangers followed by two horizontal water coolers, and into the vertical stripper. The pressure in the stripper is 15 atmospheres excess pressure. From here the liquid product is pressured directly to the stabilization column, and the circulation gas is fed to the circulation pump. A portion of the gas, the excess gas, of which about 3000 m^3/hr accumulates, is sent either to the 3000 m^3 excess gas tank or to the same sized fuel gas tank. From the latter, the gas is taken directly to the heating of the furnace chamber. Up to now the plant has run through 525 production periods.

Regeneration:

After the dehydrogenation ends, the product is charged to the second furnace group. The polymer-charged catalysts of the furnaces are washed with circulation gas for one hour, in order to dry the catalyst. The hereby accumulating benzine is collected in a separate stripper, and amounts to about 1/2 m^3. Then the circulation gas is displaced by regeneration gas, which is taken out from the regeneration tank by means of the condenser and brought to pressure. If it has reached a density of 1.2, it is charged by means of two high-speed blasts over the regeneration circulation through the furnaces. The regeneration circulation contains 6 vertical heat exchangers and gas-fired coiled heater. The gas stream then enters into the first furnace; air is added before the heater. After the first furnace the gas flows through a vapor heater. More air is added, and the gas is sent into the second furnace. There is likewise a vapor heater connected to the outlet side. The gas amounts produced in both are extremely slight. Then the gas, which is mixed again with air, enters into the third furnace. Leaving this, the gas flows through the heat exchanger and enters at a temperature of 100°C into a water cooling tower, to which again, because strong corrosion occurs, caustic is added. Over a stripper the gas is led to the blasts. The blasts are strongly porous and lose about 500 m^3 of gas hourly. The combustion in the furnaces takes place at temperatures from 22-29 mv. First of all the third furnace is ignited, for which about 6 hours of combustion are necessary. Shortly after, the second furnace is regenerated, requiring 4 hours. At the end, air is added to the third furnace. This needs about 2 hours of regeneration. The osygen-waste during the whole regeneration amounts to 0.2 per cent. After the combustion period, there is additional regeneration for an hour with an oxygen-excess of 5 per cent. From the regeneration gas, so long as this contains only 0.2 per cent O_2, a portion is stored in a 3000 m^3 gasometer. 1039

The inert gas is displaced after the regeneration from the apparatus by means of the excess gas, which takes the corresponding gasometer and by means of the two compressors up to pressure. If the density of the circulation gas amounts to about 0.55, the regeneration furnace group is connected to the dehydrogenation.

Gasification:

The plant runs with a catalyst charge of 0.5 kg/ltr/hr. For a Rumanian crude benzine with 50 per cent by vol. in paraffin hydrocarbons, which gives an injection with about 46 per cent by vol. in paraffin hydrocarbons, the gas formation should amount to 20 per cent by weight, related to C_4-free crude benzine. The accumulation of coke constitute 0.4 by per cent wt. of the injection. Therefore, a crude benzine, which contains 1 per cent by wt. in gaseous hydrocarbons, shows the following yields:

HF-benzine	750 mt
Polymer liquid-products	30 mt
Coke	4 mt
Waste	212 mt

In the DHD-plant at Poelitz a gas formation of 18.5 per cent by wt. is to be expected for such a benzine, so that the gasification of the HF-plant is about 1 1/2 per cent by wt. higher than in the DHD-plant.

Developing work:

The developing work tends especially to lessen the gasification of the HF-plant and to lengthen the period duration. The circulation gas should be washed during the aromatization, in order to increase the hydrogen partial pressure and to reduce the coke accumulation. An extention of the operation periods is thereby hoped for.

Instead of three furnaces operating in a group, in the second HF-plant, which is in construction at the time, only one furnace with a catalyst volume of 7 1/2 m^3 is installed. In this, the so-called tube furnace, the temperature decrease should be avoided through direct heating during the reaction. By means of the tube furnace, a lowering of the gas formation and an increase of the production period by about 18 hours is expected. 1040

In addition one has given up the regeneration circulation in the second HF-plant. Each tube furnace is connected with only one circulation, as with the DHD-plant.

Valuation:

1. The HF-plant manufactures distilled benzine from petroleum and is not connected with a hydrogenation plant. In its construction it has consciously approached petroleum refining. Instead of a revolving gas preheater as exhibited in our DHD-chamber with 30 hairpins with a collective length of 540 m, the HF-plant uses two furnace chambers with tubes of a collective length of 2600 m. This results in slight variations in maintaining the reaction temperatures, and demands that the quality of the injection product be kept as constant as possible.

2. In order to hold down the gasification the HF-plant is operated at a pressure of 15-25 atm. The production period lasts only 10 hours. The short production time, which makes extraordinarily difficult an optimum operation of the plant, diminishes the production per m^3 of catalyst.

If the HF-plant requires 45 m^3 of catalyst for 5000 mt/month of benzine and the DHD-plant at Poelitz requires 64 m^3 of catalyst to produce 13,000 mt/month of benzine, then on these bases production of 1000 mt/month of benzine requires respectively 9 or 4.8 m^3 of catalyst. Since this is only a standard for the combined efficiency of the total plant, and since the aromatization is an important part of the operation cost, the operation cost of the HF-process is higher than that of the DHD-process.

In spite of the lowered pressure, the volume of gas generated in the HF-plant is larger than in the DHD-plant, (whereby the higher coke accumulation is clear).

3. The removal of the easily polymerized products from the aromatization benzine is not undertaken as in the case of the DHD-process through hydrogenation, but through sulfuric acid refining. This requires an additional consumption in sulfuric acid and caustic and increased operating cost.

4. In order to produce a product having a deviable boiling curve, the HF-plant places increased restrictions on the quality of the crude benzine. The crude benzine must possess a sufficient portion in low-boiling hydrocarbons, in order that the boiling curve is attained in a profitable region. The HF-benzine contains 55-56 per cent by vol. in aromatic substances and has a vapor pressure of 0.5 atm.

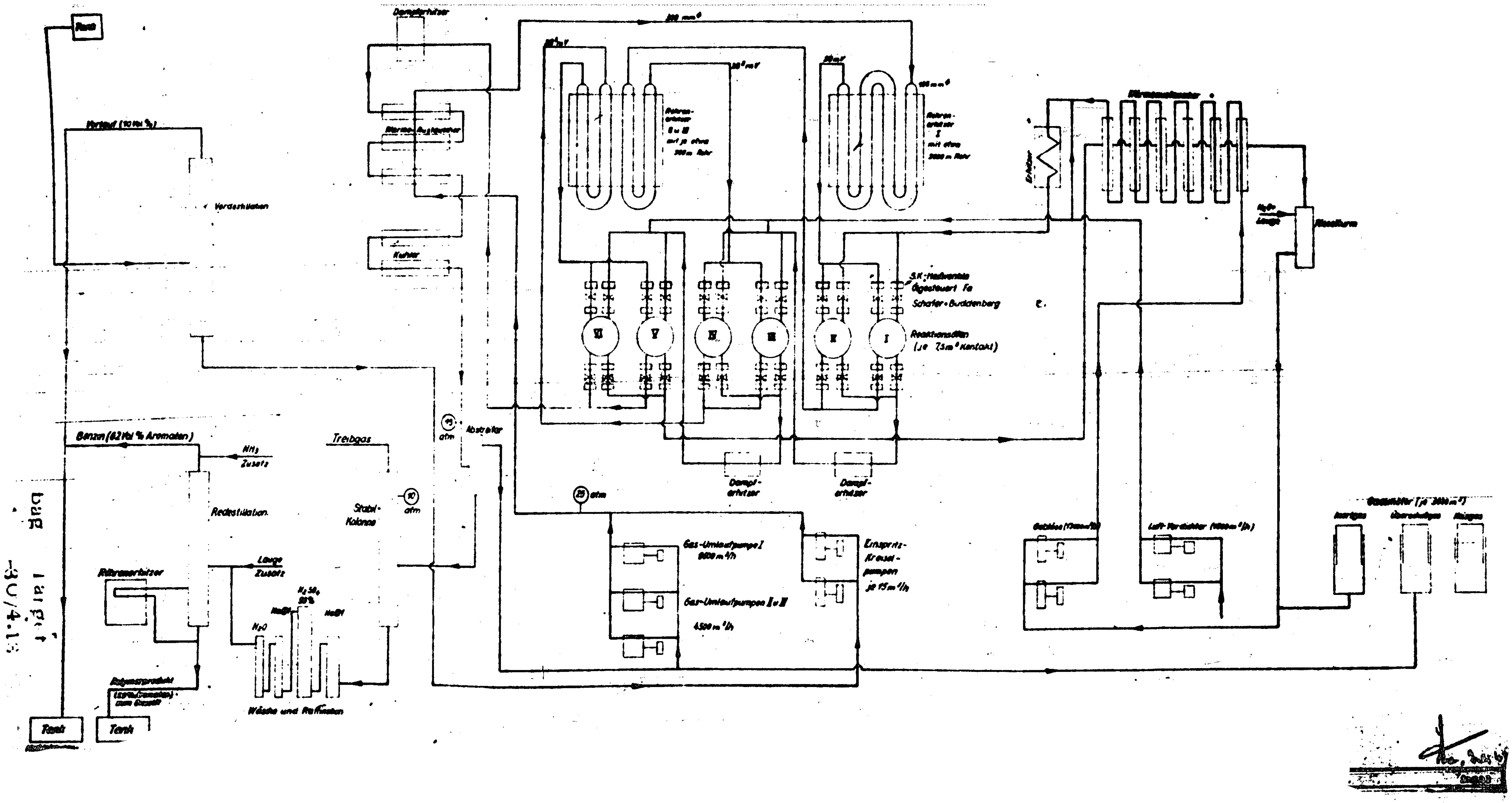
SCHEME OF THE HF-PLANT AT MOOSBIERBAUM, JULY 1943.

1041

Throughput: 12 mt/hr crude benzine from petroleum.
Production: 5000 mt/month of HF-benzine.

Schema der HF-Anlage Moos-Bierbaum. Juli 1943.

Durchsatz 12 Stuto Rohbenzin aus Erdölen. Erzeugung 5000 moto HF-Benzin.



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HEAVY OIL CRACKING
STEFFEN

Poelitz: June 8, 1943

Frames 1045-7

Abstract

The cracking of stripper heavy-oil, obtained from petroleum products, is practicable. On the other hand, knowledge is nonexistant on the cracking of stripper heavy-oil obtained from coal. There does exist, however, the probability that this too can be cracked into middle oil. Concerning this, some short experiments should clarify the situation.

Stripper heavy-oil, a product of coal or sump-phase hydrogenation, forms in the distillation of the 700 atm. sump-phase stripper. After separation of the benzine and middle oils, it is obtained as residue. The first two go to pre-hydrogenation and benzination and are by means of hydrogenation and cracking transformed into benzine. On the other hand the heavy oil is sent back to the sump-phase chamber, in order to be hydrogenated and cracked to middle oil by means of a further prolonged stay at 700 atm.

In the case of a 700 atm. coal chamber, which is operated on benzine and middle oil, the accumulation in heavy oil amounts to about 1.5 times the accumulation in benzine and middle oil, and by increased charge attains a multiple of this value. In the 700 atm. sump-phase chamber the hydrogenation is good, the cracking is slight. This is worked out separately in the case of the manufacture of petroleum products, which in themselves are already hydrogen-rich.

If this heavy oil should not be sent back to the chamber, for the purpose of transforming the heavy oils into middle oil and benzine the following two methods are offered: the first exists in the hydrogenation and the connecting cracking. This can take place in the same chamber through refluxing or in a second 700 atm. chamber over a compactly arranged contact. The second possibility is first to crack the heavy oil in a hydrogen-poor middle oil and then to hydrogenate and benzinate in the gas phase.

Therefore, the question arises, can heavy oil of the sump-phase be transformed, by cracking, into middle oil, whereby coke formation should be eliminated as much as possible.

SPRAY GRAIN AND COAL-GRINDING
STEFFEN

Poelitz: April 14, 1944

Frames 1048-53

Abstract

In March the spray grain accumulation of 36 mt/day was important. Its reason lay in a poor coal grinding. The fineness of grinding also of the portion

of coal under 1 mm size is spoiled with an increasing amount of spray grain, and the hydrogenation capacity of the coal is reduced. A reduction, as much as possible an elimination, of the spray grain accumulation must be effected.

The poor coal grinding is conditioned by the over-loading of the grinding aggregate in buildings 270 and 271. Of the two systems in building 270-each consisting of a hammer-and centrifuge-mill--only one is operated at a throughput of 1600 mt/day. In the case of this ratio, about 400 mt/day in coarse grain are caught in the swinging sieves of building 271 and are sent over the rolling cradle. Overloading of the rolling cradle results that the product, which is not sieved, contains up to 40 per cent coarse grain. If a part of it is crushed in the concentration mill, the rest, however, which is considerable, furnishes the spray grain.

For the purpose of better grinding, is is necessary that in the case of a throughput of more than 1200 mt/day two systems be operated in building 270. A reserve is again available, as soon as two newly-ordered hammer mills come into operation.

For the removal of the spray grain, instead of the present wet filtration, which makes oil waste unavoidable and gives a product, for which no use exists, a dry filtration has been installed in building 271. On all occasion must the coal be screened after the rolling cradle. To that purpose, the products are to be sent collectively after the rolling cradle over one of the two swinging sieves, which are newly erected for the fifth furnace. The coarse grain captured by this is removed from the system and amounts to about 2 per cent of the coal.

CRACKING OF OILS IN A SPHERICAL FURNACE

DR. STEFFEN

Poelitz: November 24, 1944

Translation Frame 1054

Dr. Pier:

I refer to my letter of October 21, 1944, with which I reported to you concerning the intention to crack crude oil residue in a spherical furnace. Meantime a spherical furnace has been running all right for about three weeks, so that it has proved, that the coke formed in the cracking is crushed by the grinding substances and is discharged from the furnace in the form of powder. I think, therefore, that a basis is given for a patent announcement, and we should make an outline of our idea.

I ask for your criticism and aid in the patent presentation.

With friendly greetings.

POELITZ CRACKING PROCESS-PATENT SPECIFICATIONS

DR. STEFFEN

Poelitz: December 2, 1944

Frames 1055-8

Abstract

The report is a duplicate of a report abstracted under the title Cracking Process at Poelitz (Reel 76, Frames 1071-6).

CRACKING WITH APPARATUS PRESENT IN THE HYDROGENATION PLANT

DR. STEFFEN

Poelitz: November 27, 1944

Frames 1059-65

Abstract

After the bombing attacks of 1944, a new problem appeared at the hydrogenation plant at Poelitz. The plant for the production, compression and circulation of the hydrogen has been the most susceptible and required the longest repair time of any in the whole unit. Therefore, production must be attempted, without hydrogen consumption, by returning to petroleum products and tar as raw material. The cracking process is used, the petroleum products being cracked partly under pressure in the liquid phase, and without pressure in the gas phase. It is important, then, to examine the plant apparatus and if necessary reconstruct and install a portion for the cracking of petroleum products and tars.

For cracking under pressure, in addition to separators, coolers, heat exchangers and fractioning columns, a tube furnace is also necessary. The tube diameters are 100 mm, and the furnace must be able to withstand a temperature of 500°C at 70 atm. One coal chamber was prepared for the cracking and a second for the distillation of the cracked products which accumulate. The pre-heater of the

cracking chamber included in both the cold and medium channels 7 capillary pins of 90 mm diameter and in the hot part 10 pins of 45 mm diameter. In the distillation chamber, a high-pressure furnace filled with Rashig's rings was experimentally installed as a distillation column, in order to obtain a heavy oil, in addition to benzine and middle oil.

As explained by the flow sheets which are attached to the report, the following operation is planned. The petroleum product, charged to 50 atm., is led over a regenerator and the cold part of the preheater to the heat exchanger of the cracking chamber. There the high-boiling tars and inorganic constituents are separated and given up over a cooler. The main products of the heat separator, in which the cracked products are centered, are fed over regenerators and coolers to the stripper container and the distillation chamber. After decomposing in the latter into benzine and middle oil, these products are sent to a separator, where benzine, middle oil and residue gases are drawn off, and the heavy oil, which accumulated, passes through a cooler into a reflux tank. This heavy oil is charged to the cracking chamber, passing through two regenerators, the preheater, reaction furnace and heat exchanger. By 500°C heating, and expansion from 50 to 7 atm., it is guaranteed that only the distillate is through the cracking zone of the preheater, eliminating any coking which might otherwise occur.

For cracking at atmospheric pressure, a carbonization furnace was reconverted, pictures of which were shown at the end of the report. The following method of operation is used. The petroleum residue is charged to the preheater, attaining a temperature of 350°C and a pressure of 4-8 atm., and after the addition of more vapor, it is injected into the furnace. The latter is a rotating drum furnace, containing a grinding substance, and here catalytic conversion takes place. The raw materials which are gaseous at furnace temperature leave the furnace and enter a quiet zone, where their velocity diminishes, allowing the coke dust which was carried over to settle. The materials then pass through the tar separator into the pre-cooler. By spraying with accumulating pre-cooler oil, the high-boiling hydrocarbons are precipitated here. The gaseous products enter the after-cooler and are sprayed with the accumulating after-cooler oil, a mixture of 30 per cent benzine and 70 per cent hydrocarbons boiling under 350°C. The residue gases are charged over a collector main, in which further benzine accumulates, and, by means of a blower, into the open. The coke forming in the drum is pulverized by the grinding substance. The rotating of the drum is temporarily stepped up, and the coke is removed--dry, powdery, and ash-free. The after-cooler oil is yielded in the distillation, while the pre-cooler oil is stored, as recycle stock.

PREPARATION OF MOTOR FUEL BY CRACKING IN A SPHERICAL FURNACE

DR. STEFFEN

Poelitz: December 4, 1944

Frames 1066-9

Abstract

The spherical furnace of the hydrogenation plant permits the products from petroleum and tars to be heated to 550°C. The experiments in Poelitz have shown that at this temperature in the spherical furnace, motor fuel is obtained by continuous

cracking of petroleum residue and coke. The cracking process evolved in Poelitz permitted the manufacture of asphalt-free residue oils, of low hydrogen content, and should be called upon for cracking of tars. The spherical furnace is described below, as it was used for carbonization and as it was used after reconstruction for cracking.

As used for carbonization, the furnace consisted chiefly of the preheater, the carbonization drum and the condensation plant. The method of operation was the following: Catalyst is added to the centrifuge residue, to produce better carbonization reaction, and the mixture is circulated to the carbonization furnace. After passing through the preheaters, it enters, with a temperature of 350°C, the carbonization drum, a hollow rotating cylinder containing grinding substances; and maintaining a wall-temperature of 530°C. The residue turns to coke, which is in turn pulverized. Low-pressure steam is passed over it, and carries the oil vapors formed into the pre-cooler and then the after-cooler. In these they are condensed by spraying with the accumulating cooled oils. The carbonization gas goes out the roof, and the coke is sent back to the hydrogenation.

In the case of cracking, there are some minor changes in the spherical furnace, the principal change being that the furnace is covered, allowing space only for an injection tube with vapor connections. The cracking operation is as follows: The petroleum residue is heated to 300°C in the pre-heater. Under steam pressure, it enters the drum, which maintains a wall-temperature of 550-570°C. The oil vapors are decomposed, forming low-boiling products, coke and gas. The coke (much less than in the carbonization) is crushed by a grinding substance in the drum, and carried out over a conveyor belt. The gaseous reaction products pass out of a drum through a coil and are precipitated on the coke dust. Passing into the tar separator, they accumulate as tar and more coke, at which point they are transfered to the condensation plant, which is operated as in the carbonization process. Here pre-cooler and after-cooler oils accumulate. The former is sent with the raw material back into the process, while the latter is yielded in the distillation. It consists of about one-third benzine and the rest gas oil, boiling under 350°C.

Report 584-46R

CRACKING PROCESS AT POELITZ

DR. STEFFEN

Poelitz; December 2, 1944

Frames 1071-6

Abstract

The cracking process has found use in the production of low-boiling hydrocarbons from high-boiling ones. Under the influence of high temperatures, at atmospheric or higher pressure, benzines are obtained by the cracking of gas oil or other still-higher boiling products. Since, in the case of cracking, together with low-boiling fractions, high-boiling tars accumulate in slight amounts, and since these easily incline to coking, in order to reach a long operation time, it was necessary to draw off these tars from the process and to send only distillates through the cracking zone, otherwise coke formation would necessitate cleaning of the apparatus. Most of the cracking methods used in industry cracked the distillates and avoided the accumulation of coke. They are therefore confined to asphalt-free products, with as low a hydrogen content as possible.

It was, however, the intent to develop a process, which produces rich, asphalt-containing residue oils and tars, and permitted a cracking to coke. The coke, which accumulates in this case, is ash-free and finds a further field of application, for the production of elctrodes. The processes used up till now for coke cracking are discontinuous types throughout. The coke remains in the apparatus and must be cleared after the interruption of the process. This type of operation has found only limited application in industry.

A process is described, which has proved in practice to crack residue oils to coke, whereby the coke accumulates in a form which is continuously drawn off. For this purpose, the pre-heated product is charged into a heated, rotating cracking-furnace, which is filled with grinding substances. These latter pulverize the coke. The coke dust is led to a coil, which continuously brings it out. This operation method is carried through, first of all, for cracking at atmospheric or a slightly higher pressure. It can, however, by corresponding changes of the rotating furnace and exit, be changed to cracking under pressure. Furthermore, the method permits the addition of catalysts, which influence the cracking or the condition of the coke.

A sample process, mentioned in the original report, has already been described in the abstract of <u>Cracking with Apparatus Present in the Hydrogenation Furnace</u> (Reel 76, Frames 1059-65). Some specific production data, however, might be mentioned. In the case of a throughput of 2 m^3/hr of petroleum, 25 m^3 of pre-cooler oil and 19 m^3 of after-cooler oil is obtained in 24 hours. In addition, about 50 m^3/hr of residue gas, a slight amount of tar and 0.8 mt/day of coke is also obtained.

Report 584-46R

CRACKING EXPERIMENTS IN A CARBONIZATION FURNACE

DR. STEFFEN

Poelitz: November 14, 1944

Frames 1078-94

Abstract

Carbonization furnace IX was installed after technical alterations for experiments on cracking from petroleum residues to coke. On November 2, the first cracked benzine was produced, and the furnace has been in operation since, running with a charge of 2 m^3/hr of raw material at 550°C. The results up to now promise to be successful. Some general experimental data is given in the following. Into the carbonization furnace, which is operated at a pressure of 40 mm of water, is charged petroleum residue at a temperature of 550°C, forming cracked gas, benzine, gas oil and coke. The coke accumulates chiefly in the powder form and is carried out as such. Alterations are being worked out for the continuous removal of the coke. The present condensation plant, which was erected for high-boiling oils, is not sufficient to completely condense the accumulating benzine, and alterations are needed. On the basis of results up to now, carbonization furnace X was reconstructed for cracking and should go into operation on November 22.

On the proposal of Dr. Wissel, the experiments were begun in order to produce, in one of the carbonization furnaces, benzine and gas oil from petroleum residues. For this purpose, carbonization furnace IX, a spherical furnace, was rebuilt from October 24 to 30. It was necessary to lengthen the time of stay of the vaporous hydrocarbons in the drum, in order to obtain in the case of pressureless cracking at 550°C the effective reaction time. Therefore, the washing head was removed from the entrance of the carbonization furnace. In the peep holes at the side of the coil in the exit of the furnace, there was connected a siphon pipe for the products which were removed. This was connected over a separator pot of about 200 l. capacity with the pre-cooler column. This arrangement forced the hydrocarbon vapor to stream through the whole furnace. A sketch accompanying the report showed the normal carbonization furnace and one rebuilt for cracking.

On October 30, the pre-heater was heated to 350°C, and petroleum residue was sent through. In the pre-heater a pressure of 4 atm. was maintained for 24 hours, and a pressure of 8 atm. was held for another 24 hours. Under these conditions, as investigations showed, no cracking occurred in the 100 m. long pre-heater tube with a charge of 2 m^3/hr.

In the 1st experiment, the drum temperature was brought up to 530°C and a charge of 1.7 m^3/hr. was injected, in which case a cracking was detected. There were obtained a pre-cooler product, which had a boiling ratio like the crude product and an after-cooler oil, which contained about 10 per cent by vol. boiling up to 200° and 58 per cent by vol. boiling up to 350°C. Therefore, benzine and gas oil were formed, plus an hourly accumulation of 50 m^3 of gas.

The 2nd experiment, conducted analogously to the 1st, confirmed the results of the latter. In the products formed there was 4.5 per cent by vol. in benzine and 7 per cent by vol. in gas oil boiling from 200 to 350°C. On account of the high temperatures of the after-cooler, which lay at 50-65°C, a portion of the benzine could not be condensed, so that the daily yield lay above this value.

In the 3rd experiment, the drum temperature was increased to 550°C with the same charge. In this experiment the drum had been filled with coke when the siphon pipe suddenly choked, so that the circulation also had to be stopped. The opening of the coil showed that this was completely free, whereas the siphon pipe was choked with coke, while the greatest amount of coke was found in powder form in the tar trap. The stream of the vapor escaping through the siphon tube had carried over the coke dust. After the cleaning of the tubes and the tar trap, the experiment continued for thirty-six operating hours. In the after-cooler oil, 15 per cent benzine was found, in spite of the high after-cooler temperature of 60-90°C. Therefore, as expected, the cracking was stronger at 550°C. The coke contained about 15 per cent benzene constituents. As the result of this experiment, it was determined that the drum discharges the coke mostly in the powder form. It was considered, therefore, that velocity of the escaping vapors be so reduced, that the coke would fall into the exit casing, and the vapors would escape for condensation.

In the 4th experiment, in order to operate the apparatus further, the coke was discharged discontinuously. The drum temperature was 550°C and the charge 2.4 m^3/hr. For discontinuous discharge of the coke, after 20 operation hours the drum was stopped and operated dry for 2 hrs. Then, the number of revolutions, which had amounted to about one per minute, was increased to 8 for about 2 hours. Thereby, a portion of the coke was discharged and carried away over the waste belt. After two hours, material was again injected. This coke discharge was repeated after another 20 hours, whereby the dry operation was limited to 1 hour and the operation at high rotation was likewise reduced to 1 hr. Altogether, the experiment ran over 50 hours, of which the apparatus was operated for 44 hours in production. The after-cooler temperature was held at 40-50°C. In order to reach this, the temperature in the pre-cooler had to be kept below 150°C. Thereby was obtained a greater amount of pre-cooler oil and a smaller amount of after-cooler oil. The latter, however, yielded 36 per cent by vol. in benzine boiling at 200°C and 81 per cent by vol. in benzine boiling at 350°C. In the new formation was obtained 7.5 per cent by vol. benzine and 8.6 per cent by vol. gas oil. To the coke product was added 0.5 per cent by weight soda. In addition, there was in all experiments about 18 atm. steam to pulverize the products in the injection.

The results of the above experiments prove that in order to obtain continuous coke discharge, the velocity of the vapor being carried into the coil for condensation must be so reduced, that the coke is not carried over. If, for the present, the peep holes in the coil were used, in order to connect the conduction for condensation, it was recommended to make a quiet zone. In this quiet zone the velocity of the escaping vapors is reduced to about 1/10 of the previous value. Secondly, it was considered to improve the present condensation for the precipitation of low-boiling benzines. From the carbonization furnace, the vapors arrive in the pre-cooler at about 370°C. There they are precipitated by the pre-cooler oil, which is operated counter in the circulation over two coil coolers. The temperature of this pre-cooler wash oil can be strongly varied through the cooler. If the temperature is maintained at 300°C, there accumulates in the pre-cooler column an oil, which resembles the boiling characteristics of the raw material, and contains about 20 per cent in products boiling under 350°C. If the temperature is brought to 150°C, there is obtained around 30 per cent in hydrocarbons boiling up to 350°C, therefore a great amount of the gas oil formed. From the pre-cooler head the vapors enter the after-cooler at 300° or 150°C. In this the low-boiling fractions are precipitated in an analogous method by spraying with after-cooler oil. The temperature of the after-cooler oil is determined by the output of this and the temperature of the vapors coming from the pre-cooler. The product of the pre-cooler is added as recycle to the fresh product, and returned to the furnace. The product of the after-cooler is so regulated by the temperature in the pre-cooler, that it contains as low a portion as possible in over-350°C boiling hydrocarbons (about 5 percent). In addition, through the low temperature-state of the after-cooler products of about 20°C, the benzine produced is carried off in the after-cooler oil The after-cooler oil, then, chiefly consists of benzine and gas oil. The last half of this report consisted of the drawings and data sheets already described.

CRACKING PROCESS AT POELITZ

DR. STEFFEN

Poelitz: November 23, 1944

Frames 1095-1100

Abstract

The introduction and general process described in the original report has already been presented in the abstract of Cracking Process at Poelitz (Reel 76, Frames 1071-6). The sample process, however, is not indentical with the one previously described, and will be presented here. The liquid products are brought to temperature in the heating coil under pressure, in which case no noteworthy cracking takes place. From here it goes to the rotating furnace, which is heated by gas from the outside. The furnace contains grinding substances which moves during the rotation and crushes the coke. The vaporous products run through the furnace and are cracked. They are drawn off at the other end of the furnace and conducted to the condensation plant. In this, by means of corresponding fractioning apparatus, there forms a heavy residue oil, a gas oil and a benzine. At the bottom exit of the furnace, the coke is removed. Through the rotary action of the furnace drum, the coke is raised sideways and tossed through suitable scoops into the exit coil. The exit coil then pressures the coke over a dipping into the open, and carried away by means of a conveyor outfit. Supplementary drawings are used to explain the operation. In this apparatus, residues were cracked from petroleum, of which 20 per cent boiled below 350°C. In addition to the cracked gas, benzine and gas oil, a heavy oil accumulated, which recycles into the process, while the coke is brought out continuously in a dry form.

Report 584-46R

CRACKING EXPERIMENTS IN A CARBONIZATION FURNACE

DR. STAFFAN

Poelitz: December 12, 1944

Frames 1101-10

Abstract

After installation of a new pre-heater, furnace IX was again put into production on November 13. By suitable operation of the condenser, and the after-cooler, it was possible to regulate oil to a boiling point of 350°C. At first the injection was held at 2 m^3 and the drum temperature at 550-70°C. The pre-cooler oil contained coke dust in the amounts of 0.1-1.0 per cent. On the 19th, while drawing off the tar, the hydrocarbon vapors flashed in the tar trap. The chief, however, extinguished the blaze without greater damage. As counter-precautions, the following improvements were undertaken during the course of repairs. To the tar trap was connected a 1/2 m^3 container as a sluice with a covered connection. Thereby an ignition of the tar vapors is avoided in the future. In addition, in order to reduce the velocity of the vapors escaping from the drum and thereby favor a settling of the coke dust, above the coke-discharge chute a box was installed. From this the vapors moved to the tar trap and from there to the condenser. Furthermore, an after-cooler oil container was installed, from which the product could be charged into a tank car. On account of the starting of the first coal chamber and the finishing of additional carbonization furnaces, the repair could be concluded within a week. After these alterations the furnace was put into operation again on November 27. At first with drum temperatures of 550-570°C, the furnace was operated on an after-cooler oil which contained 65 per cent benzine and middle oil. The injection was 2 m^3/hr. In this time the new formation in benzine amounted to 23.8 per cent by weight. With a drum temperature of 570°, on December 1 the injection was increased to 3 m^3 and the after-cooler oil was operated at a boiling point of 350°C. There accumulated 0.62 mt/hr or 0.75 m^3/hr in benzine and middle. The after-cooler oil contained 40 per cent benzine. The coke was brought out in intervals of 12 hours by increasing the number of revolutions of the drum to 8 1/2 revolutions per minute. There accumulated in the process 0.8-1.0 mt/day in discharged coke. This was present in the greatest amount as powder, which, however, contained single lumps. On December 4, pre-cooler oil was added in the same portions to petroleum residue. On the 5th the injection was increased to 4 m^3. On this day the furnace had to be interrupted because of an air alarm. In starting again, it was discovered that the pre-heater had a leaky flange connection. In addition, the drum was damaged near the injection. The drum had up to now 14,000 operating hours, so that the damage must be attributed to the long operating time. In order to have a furnace in operation again, furnace X was made ready. The carbonization furnace X ran 338 hours on cracking. Twice stoppages occurred in the oil gas connections before the tar trap, which, by installation of a coke cleaner in this place, should be eliminated. There were injected 0.8 m^3/hr of petroleum residue and 2.2 m3/hr of pre-cooler-oil (ratio 1:2.9). There was produced 0.6 m^3/hr or 0.5 mt/hr or 350 mt/month in after-cooler oil. In order to reach a higher production, the drum temperature should be increased to 580°C. In addition the burning jet should be increased from 13 to 16 mm. The use of round grinding substances in furnace

X has not been approved in the case of cracking. The furnace contained coke not in powder form, but in great round lumps. The old grinding substances were injected again. From 100 kg of petroleum residue with 24 per cent in under-350°boiling products were produced: 21.9 kg benzine; 50.8 kg middle oil; 18.3 kg cracked gas; 9.0 kg tar, coke and waste. The cracked gas corresponded to a vapor-phase cracked gas, and contained 13 per cent ethylene, 11 per cent propylene and 5 per cent butylene. The heat value was 11,000 cal.

CRACKING EXPERIMENTS IN A CARBONIZATION FURNACE

DR. STAFFEN

Poelitz: January 12, 1945

Frames 1111-16

Abstract

The first part of this report can be found in the information on Chamber X in the last of the abstract of Cracking Experiments in a Carbonization Furnace (Reel 76, Frames 1101-10). Carbonization furnace X was rebuilt in the same manner for cracking as furnace lX. The only difference was that furnace X was filled with round grinding substances instead of angular as the other furnace. The petroleum residue was cracked under the refluxing of the pre-cooler oil. The crude product was pumped from the tank car into the 100 m^3 tank lV and from there charged to the 100 m^3 mixing tank III as required. The mixture (soda was added) was charged to the pre-heater and heated up, at 4 atm., to 250-300°C. Then the product was injected under addition of vapor into the drum. The injection was 3 m^3/hr; the drum elements had a constant temperature of 570°. An experiment, to increase the charge to 4 m^3, caused a decrease of the temperatures at the entrance to the drum, so that the maximum charge was for the present 3.5 m^3/hr. After 100 hours the vapor addition was given up, and the number of rotations of the drum was increased, so that the coke could be removed. After an additional 50 hours of operation, in order to force the coke discharge, for two hours the drum was operated dry and then after the addition of vapor the number of rotations was increased. There came out only coke lumps. After re-starting, the furnace soon clogged up before the condenser and had to be operated cold. Examination showed that the connection before the tar trap was adulterated with coke, which was removed. Injection was started again shortly before the air raid on December 21, when the furnace had to be shut off again. After a stand still of about a week, furnace X was back into production on the 28th. Altogether, furnace X ran 338 hours on production with a charge of 3 m^3/hr. From 100 kg of petroleum residue the following yield was obtained: benzine (200°C)-20.1 kg; gas benzine-1.8 kg; after-cooler oil (over 200°C)-50.8 kg; tar-2.2 kg, cracked gas-18.3 kg; coke-5.2 kg; and waste-1.6 kg. The yield in auto benzine amounted to about 22 per cent and the diesel fuel gas about 50 per cent of the injected petroleum residue. The hourly charge lay at 3 m^3 injection or 0.8 m^3 fresh product. The after-cooler oil production amounted to 0.6 m^3/hr or 0.5 mt/hr. In order to produce 500 mt/month in after-cooler oil at each spherical furnace, the charge must be increased to 4-4.5 m^3/hr. For this purpose the burner heads, which serve for the heating of the drum, are to be enlarged. In addition the end temperature of the oils heated in the pre-heater will be brought up to 350-380°C. The drum temperature should be set up by way of experiment to 580°C.

Report 584-46R

CRACKING OF CARBONIZATION TAR FROM BROWN COAL

DR. STEFFEN

Poelitz: February 23, 1945

Frames 1117-21

Abstract

In Böhlen a DHD-double chamber is under construction and is almost completed. In the case of the temporary benzine situation, Böhlen sought another charge for this chamber and has in brief experiments cracked brown-coal-carbonization tar over a DHD-contact. The apparatus consisted of a furnace with 1.6 l. of pulverized 7360-catalyst, an electric pre-heater, injection pump and cooler. The experiments were undertaken the beginning of November. In 2 experiments over 200 and 300 hours, at a temperature of 25 mv there was injected 1.6 l/hr of brown coal tar. By addition of about 500 l/hr of hydrogen, the pressure was maintained at 50-80 atm. The gas which accumulated contained 88 per cent hydrogen. The accumulating stripper was investigated concerning its percentage content in products boiling under 330°C, which in the beginning was about 50 per cent by vol. If this portion should fall to 40 per cent, the experiment is stopped and the contact is regenerated by burning with air at atmospheric pressure. The purpose of this pressure cracking over DHD-contact is the production of J_2-fuel. The solidifying point of this should lie below-30°C. In order to carry the experiment over into a large scale, one of the DHD-chambers was rebuilt, and 2 preheater channels, with regenerator and two furnaces, were installed. It is planned to operate the 6-fold chamber and the 3-fold chamber in parallel. Thereby each part consisted of a regenerator, two furnaces and two pre-heater channels. It is especially important that the injection product be free from A-middle oil, and therefore free from products boiling under 325°C. If these are present, however, hydrogen should be used. In addition, it was observed that an increase of the reaction temperature to about 28 mv produced strong asphalt formation (7.9 per cent asphalt in stripper), while the product boiling under 325°C underwent no increase. The injection product had the following composition: carbon-87.3 per cent, hydrogen-9.89 per cent, oxygen-1.76 per cent, nitrogen-0.21 per cent and sulfur-0.84 per cent. The benzine obtained from the stripper has the following composition: carbon-86.45 per cent, hydrogen-11.98 per cent, sulfur-0.41 per cent, nitrogen-0.09 per cent, oxygen-1.31 per cent, chlorine-0.03 per cent.

Report 584-46R

DECOMPOSITION OF HIGHER-MOLECULAR-WEIGHT HYDROCARBONS WITH STEAM

DR. SCHILLER AND DR. HUTTNER

Oppau: April 7, 1930

Frames 1123-33

Abstract

(poorly legible)

In the case of the decomposition of illuminating gas using the tube process there occurred soot formation in the catalyst. The gas, however, was conducted over a molybdenum catalyst at 350° before the decomposition, and C_2N_2 and a part of the organic sulfur compounds were removed, so that the soot formation ceased. In this case, therefore, a contamination was the occasion for the soot formation. Higher molecular hydrocarbons, especially the unsaturated hydrocarbons, even in the pure form indicate soot formation. Ethane acts practically like methane and propane in forming slight amounts of soot. Ten per cent ethylene and propylene in methane (or H_2) can be decomposed over Ni-U-catalyst(1) without soot formation if the catalyst temperature is above 600° and a suitably large excess of steam is used. For gas mixtures (propane-propylene, the Ni-U-catalyst(1) or Ni-V-catalyst(2) is shown to be the best. Such a Ni-U-catalyst(1) was produced in great amounts and sent to America for experimental purposes.

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1. $Ni_2O_2 + UO_2$ + kaolin
2. $Ni_2O_2 + V_2O_2$ + kaolin

APPARATUS FOR THE DETERMINATION OF TOTAL SULFUR IN GASES AND VOLITILE SULFUR IN COAL

DR. FR. GRASSNER

Oppau: May 27, 1936

Frames 1135-43

Abstract

In an earlier paper, a method was described by us for the determination of the total sulfur in gases, by ignition with oxygen on platinum, in one of the apparatus developed from the Pregl's beaded tube. The sulfuric acid formed is determined volumetrically or micro-gravimetrically as barium sulfate. The apparatus which is used for the combustion of gases containing little or no carbon compounds has little use when the gases contain hydrocarbons of high carbon content, for example, brown coal-gas, propane, etc.; the combustion rate must be chosen so small that at most about 10 l/hr of gas can be burned, which in the case of a slight sulfur content of the gas leads to a longer combustion period. Suitably the new apparatus described as follows offers the possibility of burning carbon-rich gases quickly and nevertheless absorbing the oxide of sulfur which forms. In the new construction, the inside width of the combustion tubes is increased from 8 to 14 mm; in place of the absorption parts of the Pregl's beaded tubes there is an intensively active absorption collector main with fritted glass liner. With this apparatus, illuminating gas can be burned with a stream velocity of 50 l/hr, propane with 25 l/hr and butane with 20 l/hr. The starting of the apparatus is done in the following manner: The glass beads of the collector main are sprinkled above and below the filter plate with about 20 cm^3 of 3 per cent perhydrol solution, and the collector main is fastened on the rock crytal tube. After the suction pump has been installed, platinum gauze is brought to a glow by a bunsen burner and put in the oxygen stream. The amount of oxygen is determined by the type of gas to be burned. In this case the gas was burned with 160 l/hr of oxygen. In all cases it is to be seen that a slight vacuum pervails in the combustion.tube, since otherwise the flame would be extinguished. After the washing of the gas meter with the gas to be investigated, the gas from the capillaries is ignited and to flame adjusted at 2-3 cm. length. The capillaries are now inserted into the rock crystal tube; the gas must burn with a quiet, level flame. Suction air, oxygen, and gas are suitably regulated, as much as necessary, during the combustion. The water formed during the combustion is collected in the collector main and eventually, after short stoppage of the apparatus, blown out of the main by compressed air. After finishing the combustion, the gas stream is shut off, then the suction air and finally the oxygen stream; the collector main is removed and washed out with distilled water. The rock crystal tube is likewise washed out; the collected wash waters are concentrated to about 20 cm^3 and sulfuric acid is precipitated in the filtered solution as barium sulfate. The amount of barium sulfate is then determined by micro-analysis.

For the determination of volatile sulfur in solid fuels (hard coal, brown coal, briquet), the Pregl's tube was used by us in the original plan of Pregl; recently the absorption part of the tube was replaced by an absorption apparatus with fritted glass lining. The collector main is fed with 15 cm^3 of a 3 per cent perhydrol solution; about 100 mg of coal is burned in a slow air or oxygen stream. The temperature of the electric furnace in the case of brown coal is 600°, in the case of hard coal is 800°. After the washing of the absorption collector main and the combustion tube, the sulfuric acid precipitated as barium sulfate is determined by micro-analysis.

EXAMINATION OF SAAR GAS (SULFUR COMPOUNDS, BENZENE, CYANOGEN AND ACETYLENE)

DR. FR. GRASSNER

Oppau: April 22, 1938

Frames 1145-57

Abstract

For the manufacture of Saar gas into hydrogen, methane, etc., it is of importance to become acquainted with the secondary constituents and impurities of the gas; especially the sulfur compounds contained in the gas should be investigated closely. For the individual investigations, the gas was taken to the

analytical laboratory at Oppau, where a technical gas analysis gave the following composition: CO_2-2.6 per cent; unsaturated hydrocarbons-3.1 per cent; O_2-0.5 per cent; CO-7.2 per cent; H_2-52.3 per cent; CH_4-25.8 per cent and N_2-8.5 per cent. The determination of the total sulfur was carried out by burning about 50 to 100 l. of gas with purified air on platinum gauze; in the condensate obtained, SO_4^{--} was determined as $BaSO_4$. The sulfur value obtained in the course of 60 days varied from 105.9 mg to 17.1 mg of sulfur per m^3 of gas reduced, with an average of 128.9 mg S/m^3 gas. Inorganic sulfur compounds (H_2S) could not be detected; cadmium acetate solution, in the case of daily running through of the gas, remained completely clear and colorless. In the organic compounds of sulfur there came into observation: carbon disulfide, mercaptans (C_xH_y-SH) perhaps also thio ether (C_xH_y-S-C_x-H_y) and others, thiophenes and carbon oxysulfide. The determination of these sulfur compounds was undertaken in the following manner: The gas was passed through a dry gas meter and a calcium chloride tower and finally to a refrigeration vessel, cooled to-80°C, which contained 10 cm^3 of absolute alcohol. In this vessel in addition to benzene there were condensed thiophenes and higher-boiling mercaptans, thio ether, and carbon disulfide. The gas then streamed through two energy wash flasks (one with fritted glass), which are each charged with 400 cm^3 of a mixture of about 300 cm^3 of 1N methyl alcohol-potassium hydroxide and about 100 cm^3 of 1N aqueous potassium hydroxide; carbon oxysulfide forms with the alcoholic potassium hydroxide the compound $CO_2S(CH_3)K$. The sulfur values obtained over 39 days as mg S/m^3 gas were the following: sulfur as CS_2 varied from 30.4 to 60.0 with an average of 42.5; sulfur as COS from 67.6 to 102.0 with average of 83.6; sulfur as mercaptans, thio ether etc. from 1.4 to 8.9 with average of 4.7; thiophene always less than 1 mg.

The benzene determination took place, according to the method of Berl, by conducting the gas over activated charcoal, freeing the benzene from the charcoal by steam and measuring in a micro-burette. The cyanogen determination took place, according to Drehschmidt, by pumping about 500-800 l. of gas in about 16 hours through two bell-shaped washing flasks, each charged with a mixture of 50 cm^3 of 10 per cent fresh ferrous sulfate solution and 100 cm^3 of 25 per cent sodium hydroxide solution. After washing out the wash flasks, the liquid was acidified with HCl, the hydrogen cyanide filtered out and washed with a little cold water. The filtrate was heated in a beaker with about 10 per cent potassium hydroxide solution, and the potassium ferrocyanide which forms was filtered by suction over a glass filter. After washing out the filter, the liquid was evaporated in a platinum dish with sulfuric acid, and the residue was ignited slightly. The determination of the iron oxide was accomplished either by gravimetric, iodometric or colorimetric analysis. (1g Fe = 2.79g Cyanogen). Over 25 days the value ranged from 0.25 to 12.0 mg of cyanogen per m^3 of gas reduced, with an average of 3.7 mg. For the acetylene determination, about 2 l. gas was mixed in a calibrated gas flask with 50 cm^3 of Ilosvay's reagent; the copper salt formed was suction-filtered through a glass filter and analytically determined according to Willstätter and Maschmann. (1 cm^3 0.1N $KMnO_4$ = 1.301 mg acetylene). Over 11 days the value varied from 1.76-2.58 g acetylene per m^3 of gas reduced, with an average of 2.12 g.

CALCULATION OF THEORETICAL GAS QUANTITY IN CONVERSION BY MEANS OF NOMOGRAMS

DR. STEFFEN

Poelitz: May 7, 1942

Frames 1164-7

Abstract

A mathematical discussion of the calculation of the "theoretical gas quantity in conversion", which takes place according to the water gas equilibrium:

$$CO + H_2O = CO_2 + H_2$$

is presented together with nomograms for simple and quick calculations of the same quantity.

A sample calculation is given showing the use of the nomograms.

Abstractor - G. C. Brous

STABILIZATION OF MATERIAL FROM LIQUID SEPARATOR UNDER PRESSURE IN THE ABSENCE OF INTERMEDIATE EXPANSION AND RICH GAS COMPRESSION

DR. KURT WISSEL

Poelitz: December 1, 1944

Frames 1172-6

Abstract

After the last attack, the technical condition of the manufacturing operation was the following: the hydrogen production, including the wash and compressor, had suffered heavy damage; however, a partial return to operation was possible in a relatively short time, as was the case with most of the chambers of the high-pressure operation. On the other hand, the after-treatment of the high-pressure operation was destroyed to an extraordinary degree. The distilla-

tion and the intermediate tanks, as well as the benzine wash, stabilization and rich-gas producer were found in such a state that they could not possibly be fixed in a short time. Out of the total number of 39 intermediate tanks for gas-phase and liquid separator products, only 3 were in complete order. Of the 8 distillers present only 2 could be repaired within a month, while 4 required 6 months and the rest 9 months for repair. The rich gas system was out of operation for 2 months; and the liquid gas treating system would be about 60 per cent repaired in 5 months. Out of this preparation it was shown that with a new method of operation, a higher capacity could be reached. With this new method, the stripper is no longer expanded to atmospheric pressure, but brought directly from the 25 atm.-expansion flask into a pressure container with a capacity of 150 m^3, which is analogous to the motor fuel tank. In this fuel tank, the separation of water and product is undertaken, which removes the ammonium sulfide-ammonia water at container pressure (14-18 atm); and the water-free product is stabilized in the pressure column and charged into the steam-heated distillation of DHD II plant. From there the benzine is drawn off over the sodium hydroxide wash, water-wash and after-stabilization as a finished product. The benzine must be taken over the supplmentary sodium hydroxide wash, water wash and stabilization, since a complete stabilization, of the gas-phase liquid separator in the case of the high content of the charge could not be produced.

INSTALLATION OF SECONDARY APPARATUS FOR THE PRODUCTION OF MOTOR FUEL

DR. KURT WISSEL

Poelitz: December 1, 1944

Frames 1177-80

Abstract

This report is similar to a previous one which has been abstracted under the title, Cracking with Apparatus Present in the Hydrogenation Plant (Reel 76, Frames 1059-65).

SULFUR-CONTAINING FUEL GAS

DR. KURT WISSEL

Poelitz: December 1, 1944

Frames 1181-4

Abstract

The sulfur-containing fuel gas can be pre-heated only to about 350°, if ordinary iron is used as material for the apparatus. In the case of higher temperatures there occurs strong corrosion through the formation of ferrous sulfide The flue gases should be cooled only so far, that the wall temperature does not fall below 275°C, otherwise there occurs the deposit of acidic ferrous sulfate and corresponding strong corrosion. The temperature of 275°C is, thereby, only a stopping value, which varies with the sulfur content and the oxygen excess used in the combustion. However, in the case of the preparation of inert gas from fuel gas, the cooling of the combusted gas below 300°C causes a desulfurization of the fuel gas.