

Stock
Mr. Wiley

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U. S. BUREAU OF MINES
COAL TO OIL DEMONSTRATION BRANCH
Louisiana, Missouri

U.O.M. Reel 57,
Frames 00682 to 00771.

T-445
K.C. BRAUN
February 1948

ABRIDGED OPERATING JOURNAL
WESSELLING HYDROGENATION WORKS.

FROM
March 1942
to
July 1944

March 18, 1942.

Stall 3 shut down after 32 operating days.

Judging from the behavior of the thermo-elements, converters 1 to 3 appear coked up. The hot catchpot, too, seems to be coked.

During the night of the 17th-18th, occasional increased stall differential pressure was observed. The stall could have been run for some time further, except for the danger that no normal shutdown might have been possible with further indications of increased differential pressure.

March 20.

Stall 3, shut down on the 18th, is completely coked up. Converters 1 and 2 are almost entirely and converter 3 two-thirds filled with coke. Converter 4 contains coke deposits.

March 21-22.

Severe pressure indications in and boiling over of the A-column. The product squirted out of the safety valves as far as the lys scrubbers.

The feed probably contained water, because it was observed that mainly the storage vessel boiled over. At the same time, product from tank 1 was pumped over into tank 6 (liquid phase cold catchpot product, which had a tendency towards emulsifying and was therefore stored separately).

March 26.

Cold oil instead of cold gas is injected into liquid phase stall 4. The cold heat exchanger of stall 3 was dismantled after only a short period of operation. It is badly fouled on the outside of the tubes. The dirt consists largely of inorganic substances, perhaps sand or catalyst. It will be analyzed.

March 27.

The cold oil injection into converters 3 and 4 of stall 4 is proving satisfactory.

March 28.

Repeated boiling over of the A-column. Water from the liquid phase cold catchpot again got into the A-column.

March 29.

All 500 and 600 volt machines stopped. Line trouble in the power plant?

Liquid phase stall 4 ran 5 minutes without a circulating pump. No rise in temperature occurred, but a drop in pressure from 650 to 400 atm. Due to the pressure drop, the converter contents began to boil and boiled over. The consumption of heat of vaporization prevented the rise in temperature in the converters. The stall was restarted after the trouble.

April 1.

Bombing alarm during the night of the 1st to the 2nd. A hit in gas holder 6. Partial shutdown of the plant.

The anti-aircraft fired a shell into the gas holder, which tore a hole about 3' x 4' and exploded in the interior of the gas holder. The escaping gas ignited at the hole. However, the fire could be extinguished within 1/2 hour by lowering the bell. The gas plant was shut down during the fire. In the attempt to restart it the controls of the generators did not function. No pressure could be applied to the oil line. After an hour's search, it was found that a control valve had been shut off in such a position that the oil was cut off at that point. In consequence of shutting down the gas plant, the following had to be shut down:

1. Conversion.
2. Vapor phase stall.
3. Distillation, I.S.C., and the stall 3 preheater, which was just being heated up. Liquid phase stall 4 had to be changed over to parting off, but was again changed over to coal paste after a short time. Gas holders 2 and 15 had to be lowered to their lowest possible position. All departments resumed production during April 2.

April 2.

Shutdown of several paste presses, due to low level in coal paste feed tanks, caused by poor transmittal of information between departments. After this trouble, which required a reduction in the coal paste injection into the stall, the gas in the cold catchpot of stall 4 escaped into the letdown line. At the central expansion point the valves were fully opened to expand into the large gas line as well as the emergency expansion tower. In spite of this the pressure in the hydrogen gas line to the inlet to gas holder 37 rose dangerously. The rise in pressure was transmitted backwards to the breathing line connected to the cold catchpot product and raw gasoline tanks in tank area 66. In consequence all 4 tanks burst. Fortunately, all but the dewatering tank for the cold catchpot product burst only near the top so that no product was spilled into the tank yards. The dewatering tank was emptied by filling with water and letting it overflow into the suction tower for B-distillation. Fortunately, no fire occurred when the tanks burst.

April 6.

During the night of the 6th and 7th, the A-still again boiled over, due to poor separation of the liquid phase cold catchpot product and water in tank 6.

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April 9.

Liquid phase stalls 3 and 4 run with cold oil injection are running unsteadily. Stall 4 had several temperature rises above 25.5 mV. but no further coking occurred.

April 10.

Shut down stall 3 because of plugging of the hairpin coils by injected oil in the connecting line between preheater and converter I. On dismantling it was found that the hairpin itself was plugged in the middle but that the line to it was free. Accordingly, 1100 atm. pressure was brought upon the hairpin itself after the investigation.

April 11.

Fire in stall 3 at the emergency expansion group in the stall wall, caused by a sudden leak in a line. Possibly a workman stepped on a gas line. The stall was just being pressurized up and had reached 500 atm. It was immediately expanded. The fire lasted about 1/2 hour before it was extinguished by the fire department.

The neighboring stall was switched over to pasting oil and expanded from 600 to 500 atm. It was subsequently again brought up to pressure and temperature and back to coal paste injection.

April 12.

Failure of substation U because of a break in the line, possibly by anti-aircraft shell splinters. The 2 circulating pumps of the 700 atm circuit stopped, although one of them was presumably connected to the stall. Stall 4 had to be switched over to pasting oil.

April 13.

Stall 4 is shut down because of a short circuit in the hot preheater, possibly caused by the 2 shut downs in connection with the shutdown of stall 3 and the shutdown of the 2 circulating pumps the night before.

April 14.

Coal stall 3 is restarted.

April 15.

Stall 3, operating on cold oil instead of cold gas, is running very unsteadily because it is difficult to hold the cold oil pressure. The descending line of converter I seems to be constricted, little product but mostly gas is let down.

April 16.

Temporary shutdown of the gas plant due to current failure. However, it was not necessary to reduce production in hydromanation.

April 17.

On dismantling stall 4 it was found that the connecting line between preheater and converter I was only slightly clogged, although no oil was injected.

April 19.

Water enters almost all tanks in the heavy oil tank area, possibly through the common breather line, although this phenomenon is hard to explain.

Stall 3 had to be switched to cold gas for 20 minutes, because the supply of cold oil was temporarily interrupted.

April 21.

A circulating gas scrubber and a pressure oil pump were operated below capacity, with noticeable influence on the circulating gas density.

April 22.

sudden leak in the high pressure flushing oil line on the back wall of stall 3. Oil leaked out first and later gas after closing all valves. The stall was switched from cold oil to cold gas for a short time. By securely tightening all valves in the line in question, it was possible to stop the leak and install a shut-off valve in the oil line, after which the leak on a flange connection could be repaired.

April 23.

Leak in a measuring line on the hot catchpot in stall 3. An operator went into the stall to tighten the leaky joint. It was not necessary to shut down the stall.

April 25.

Stall 3 runs away and the emergency expanded. In changing 2 coal paste presses the temperature rose in converters I and II, which was apparently not noticed for a few minutes. In spite of the injection of large quantities of cold oil (20-25 m³), and cold gas, and switching the stall to pasting oil, converters I, II and III ran away to above 45 mV. Oddly enough all lines withstood this temperature, including the emergency expansion line.

April 27.

Stall 3 is restarted after repairing some leaks which occurred in the shutdown, and after repacking the stuffing boxes on two heat exchangers.

April 29.

The circulating gas scrubber produces a better cleaning value when injecting 10 m³/h diesel oil, together with 8 m³ cold oil injection into stall 3, than 4500 m³ expanded gas from the circuit. When using scrubbing oil, the density is about 0.260 compared to 0.300 when expanding 4500 m³ circulating gas.

The liquid phase cold catchpot product boils over into the liquid phase rich gas line. It was found that the cold catchpot product repeatedly found in the rich gas line got there by the overflow of the "stork's nest," the surge tank for the cold catchpot product. Even the H₂S scrubbing tower behind the stork's nest was filled. It was found that the flow meter (Richardmessenor) behind the stork's nest was stuck. The by-pass around the meter could, therefore, not operate, because it is only insulated but not heated and the catchpot product consequently solidified in it.

April 30.

Coal stall 4 put on stream. The device for injecting oil into the stall had, in the meantime, been perfected by the installation of a surge tank and a larger header, 70 mm dia.

May 1.

Operation of stall 4 is delayed because of a leak in the letdown cooler. The letdown cooler of stall 5 is, therefore, connected to it. The stall is finally started during the night of the first and second.

May 3.

Fire in Bldg. 28, which is transmitted to Bldg. 35. In the welding of a nipple to the raw phenol water tank, the tank exploded, bursting at the bottom and the top. Its entire contents, including several hundred m³ of oil floating on top or settled as residue at the bottom escaped. Gas holders 37 and 38 were not affected, though within the reach of the flames. The production of the liquid and vapor phase stalls was interrupted for about 6 hours.

May 4 and 5.

Large accumulation of oversize screenings on the shaking screens, about 1-2 tons per shift with 30 m³ coal paste/h. At the same time, coal preparation has found that the dry coal received from the mine contains many coarse lignitic particles.

May 6 to 10.

During the night of the 8th to 9th both vertical rollers for dry coal got stuck because of poor maintenance. This caused a 5 hour shutdown of coal paste preparation. The coal stalls have to be switched to pasting oil for a few hours.

May 11.

The production of letdown gas is considerably greater than calculated by I.C., i.e. 4000 m³ with 25 t/h letdown. This amounts to 160 m³ gas/t letdown, compared to the precalculated 76 m³/t. It is probable that circulating gas is expanded with the letdown or the descending product of converter 1.

Continuous trouble with the high pressure centrifugal pumps for the A-still. During the night of the 11th to 12th only 1 pump out of 4 was available.

May 20.

Trouble with the centrifuges. Many failures by baskets getting stuck. On disassembly, the centrifuge walls were found to be covered with hard crusts containing about 80% solids. This was probably due to the accumulation of fine solids with a tendency towards deposition by continuous solids circulation with the centrifuge oil and by residue recycle. A distinct improvement was observed in the next 2 days by reducing the residue recycle from 15 to 8% and adding the centrifuge flushing oil to the L.T.C. mixture instead of to the centrifuge oil, as formerly.

May 25.

The K-values of the heat exchangers of the coal stalls are steadily getting worse, they drop to 50-100, while the resistance of the stalls rises to 35 atm, divided as follows:

14 atm in heat exchangers,
12 atm in preheater,
~~the rest in the converters to the stall outlet.~~

~~Reason: Rapidly increasing fouling of tubes.~~

NOTE: No report available between
this point and January 1943.

January 3, 1943.

The shaking screens for coal paste are running over, due to the high viscosity of the paste. The paste contained 43% solids and 4% water.

January 10.

At 0⁵⁵ fire underneath the pipe bridge at Bldg. 21, booster compressor. Due to carelessness in draining the oil separator behind booster compressor 4, the escaping gas caught fire. Apparently the plugged drain line was suddenly cleared. A severe fire followed, damaging the pipe bridge and Bldg. 21. The booster compressors were shut down and the 300 and 700 atm. make-up gas lines were blocked off. Hydrogenation was without make-up gas for 10 hours. The gas plant and the gas cleaning department to the CO scrubber were kept in operation because of the cold of -6°C. The gas produced was used partially for fuel, partly expanded behind Bldg. 22.

The vapor phase stalls were kept hot without injection. The coal stalls were also kept hot by injecting 1 m³ starting oil into each stall. The pressure dropped to about 200 atm in the liquid and the vapor phase. The without losses in the liquid phase were covered by the addition of 1000 m³ H₂/h.

Low pressure and distillation were put on recycle, with particular attention to keeping pasting oil, coal paste, centrifuge and L.T.C. mixtures in circulation.

Alkacid scrubbing and L.P.G. plants were shut down, keeping the apparatus under light pressure.

The final boiling point of the A-middle oil, which had been temporarily reduced to 250° because of the lack of pasting oil was gradually increased to 320°.

January 11.

Temporary shutdown of one of 4 boilers in the power house for about 1-1/2 hours. One furnace had to be shut down in the splitting plant. The CO content of the pure H₂ rose temporarily to 9%.

March 2.

The 4th coal stall put on stream. At the same time stall 3 was shut down because of a leak in the descending line on the converter cover.

March 2.

Due to running over of water from the water scrubber ahead of the propane column, water got into the precooler and the propane column, causing plugging of the precooler and the column. The precooler will be flushed with acetone.

Due to a drop in the U-circuit voltage, 6 compressors had to be shut down for a short time.

March 4.

Shutdown of coal stalls 4 and 5 due to a mistake in operating a gate valve in a coal paste line. The outlet gate from stall 4 was closed; stall 4 received no coal paste; stall 5 temporarily, 30 m³. Stall 4 had to be emergency released; stall 3 was temporarily switched to pasting oil.

Splitting furnace 3 shut down because the fuel gas heat exchanger was plugged. On opening it was found that the upper pipe band of the heat exchanger was completely plugged up by a mixture of tar and coal dust.

March 5.

Coal stall 3 again operating.

March 7.

Light ends from the A-distillation gas column will henceforth be recycled, instead of 5050-gasoline, for the separation of water from the liquid phase cold catchpot product. Recycled quantity = 12-15% based on catchpot product.

March 8.

Coal stall 6 had to be shut down for about 12 hours, due to a leak in the descending line.

The L.P.G. plant is being shut down because of a leak in the propane column reflux cooler. Considerable quantities of hy-gas are found in the cooling ammonia.

March 9.

Shutdown of coal stall 5. The hot catchpot boiled over, so that a large amount of hotdown got into the cold catchpot. It is possible that all converters have been filled with caviar, due to the long extended poor condition of converter 2 decanting. This caviar may finally have spilled over into the hot catchpot.

March 10.

A 5 mm mesh screen has been installed above the pulverizers, instead of the old 10 mm, in order to screen off all wood particles from the coal. This screen has been found too fine. In 1/2 hour over 500 kg coal was screened off into the waste boiler, compared to less than one kg/a. A 7-8 mm mesh screen is to be tried out shortly.

March 14.

Signs is added to fuel gas in the splitting plant. The heat value of the fuel gas is thereby increased from 1450 to 1800 kcal.

March 15-16.

Considerable quantities of liquid phase cold catchpot product got into the river with the plant waste water, due to the boiling over of catchpot product from the central hotdown vessels through the lean gas line and the spherical gas holder into the oily waste water.

The L.P.G. plant is again operating. After the shutdown due to the leaky propane column reflux cooler.

March 17-18.

In spite of the elimination of the boiling over of liquid phase cold catchpot product into the fuel gas holder and from there into the oily waste water, more oil is getting into the river.

The hot asphalt values reported the last few days have been shown to be wrong. The normal gasoline used for asphalt precipitation contained aromatic. The true asphalt value of the coal stalls are between 25 and 30%, instead of the 10% mistakenly reported.

March 19.

In stall 5, the stall with the 500 mm dia converter 1, the thermo element tied onto the last hairpin coil indicates 550°C. The temperature had not been observed the last few days. The stall injection is immediately

reduced by 2 m³ and the temperature reduced to 530°. The stall is shut down since coal stall 5 is restarted the same day.

The diesel oil tank in tank area 66 ran over during the night.

March 20-23.

Stall 5, shut down on the 19th, is badly coked. Converter I has large coke deposits; converter II contains caviar. Thus, the volume of converter I is too small to prevent caviar formation in converter II. Experiments with a smaller diameter first converter will be continued.

March 24.

Dismantling of stall 5 showed that the last preheater tubes show signs of wear near the joints. The maximum wear on the wall thickness is about 6 mm.

Both A-distillation tanks suddenly contain water instead of coal catchment product. This causes a sudden rise in pressure and, in consequence, severe leaks at both flanges of A1 column. However, it is possible to stop the leaks by shutting off the column and applying asbestos soaked in water-glass, held in place by clamps, to both flanges.

March 26-29.

Further findings on dismantling stall 5.

Converter I: - filled 2/3 with caviar, baked together in the usual manner, though deposited on the wall.

Converter II: - also shows encrusted caviar baked to the wall for its full length. This caviar seems to have originated in this converter.

Converter III & IV: - findings not yet available. The first attempt to prevent the formation of caviar by using a smaller diameter converter I has thus been a failure. However, since stall 5 has shown occasional rises in temperature, due to lack of cold gas, experiments with a smaller first converter will be continued.

April 3-6.

Experiments to perfect the desanding device. Continuous desanding with direct connection of the desanding line from converter I to the letdown line @ 500 atm.

The first attempt to produce briquetting pitch in L.T.C. kiln I is interrupted by plugged apparatus due to too rapid heating.

April 7-9.

Shutdown of vapor phase stall 7 to replace the 2 heat exchangers. The condensate injection line is extensively clogged by a deposit containing oil and lime.

Discussion with Messrs. Coesterlin and Wechsasser regarding the replacement of saturated water (Sättigerwasser) in Bldg. 5 with CO_2 , which would save 5 tons of steam. To start with, one generator is to be charged with CO_2 experimentally.

April 10-13.

Shutdown of stall 4, the second experiment with a reduced dia. converter I, 300 and 500 mm. The preheater shows severe salt crust formation and wear on the bonas.

Sudden stop of almost all rotating and screw kilns in L.T.C., apparently caused by the addition of lumps to the L.T.C. mixture.

With $15 \text{ m}^3/\text{h}$ injection in the stabilizer, we have succeeded in producing an overhead gas free of pentane and a gasoline free of butane. The ratio of overhead gas to reflux is 1:10. The overhead gas is about 1.5% of the feed.

April 15-17.

The burner in plant 3 does not permit sufficient gas flow because the fine gas slots are coked up. The fuel gas pressure is raised from 350 to about 700 mm W.G. by direct introduction of circulating gas.

There is such a congestion in the L.T.C. plant, apparently caused by the addition of residues dissolved in pasting oil, that all kilns must be shut down temporarily.

April 21.

A sudden drop in gas pressure at noon. All stalls must be reduced by 20 m^3 each.

April 22-24.

The alkacid recovery column is clogged on the inside. The lye return to the sump, a funnel-shaped pipe with a 5 mm orifice, is apparently plugged.

April 28-29.

Due to boiling over of the liquid phase cold catchpot central expansion vessels, about 6 m^3 of the catchpot product gets into the alkacid scrubber through the spherical gas holder 125. The alkacid liquor scrubber is not affected thereby, though the sight glasses appear covered by the

catchpot product solidifying at about 25°C. An attempt is made to separate the alkaloid liquor from the catchpot product by adding light gasoline.

Sand in the cooling water has again been observed lately.

May 1-3.

Deformation of the centrifuge oil tank. Water entered the tank with the precooler oil from Bldg. 82, which evaporated on entering the tank, held at 130°C.

Trouble with the Rollers in Bldg. 43, caused by scrap iron in the dry coal. Coal paste injection into stalls must be reduced.

The attempt to descend @ 600 atm. into the letdown line has proved successful. Stall 3 is to be descended in a similar manner, i. e. in such a way that the letdown valve is opened before the descending valve.

The alkaloid liquor in the liquid phase cold catchpot product can be separated by heating the mixture.

May 4.

Preparation for starting the first 643% stall. The operating method is as follows:

5058 and 643% catchpot products are mixed in such a proportion that the 5058 middle oil required for making aviation gasoline is added to the mixture. The aviation gasoline is therefore a mixture of 5058 and 643% gasoline. The 5058/643% middle oil is charged with H_2S , H_2S content about 0.5%, and injected into the 643% stall. The available H_2S will contain at least 90% H_2S , the rest CO_2 . The A-middle oil for the 5058 stall will also be sulfurated.

May 5.

Shutdown of coal stall 5 after 29 operating days because of a leak in a demulsing line. Wear of about 14 mm is visible in the preheater, i. e. with an injection of 45 m³ and 27 m³ inlet gas the wear in 30 days is 1 mm, which checks with the wear found in other stalls.

May 8-12.

There is a deficiency of H_2S , due to shutting down the 2 coal stalls and too high an iron content of the coal paste. The addition of catalyst is reduced from 6 to 5%, the addition of sulfur increased from 1% to 2%.

In a discussion of experiences with Polatz, the following points were brought up:

The sulfur content of the by-gas for the splitting catalyst. S-content to be 7 mg/m³. Iron-oxide and zinc-oxide are suitable conversion catalysts for organic sulfur.

The catalyst of a splitting furnace has been regenerated by injecting a mixture of steam and air.

May 15.

The first 6:34 stall was started with distillation residue saturated with 0.68% H₂S. Start adding 0.1% soda to the coal paste.

May 16-17.

Fire on the flow meter platform, Bldg. 43. The fire started in an idle "Konzentra" mill. In order to prevent this in the future, the coal feed hopper to the Konzentra mills is to be filled with catalyst before shutting down the mill, to prevent the flow of too much air through the mill.

June 16-17.

Current failure in circuit U. Temporarily there is only one 700 atm circulating pump in operation. The coal stalls are switched to pasting oil. Only one machine is operating in the compressor building. In consequence, the expansion engines in Bldg. 22 are getting gas. The line from the expansion engines to low pressure breaks at a flange connection. Copper liquor escapes; one man poisoned by CO.

The splitting plant is temporarily shut down, because of failure of the air blower. Only one is in operation. The shutdown and consequent cooling off causes leaks and a fire in the H₂-gas heat exchanger at splitting furnace 4.

In dismantling stall 3 the brake on the stall crane fails to hold while a 15 m converter is being lifted. The converter slips off, the motor bursts. Fortunately, the converter drops to the ground beside stalls 5 and 6 in such a manner that nothing serious happens.

June 18.

Shut down stall 4 because of a leak in converter I.

June 22.

Desanding of stall 5 seems to be out of order, low solids content, less than 10%. This trouble, as well as the shutdown of stall 4, is due to the current failure of June 16-17.

June 23-25.

Desanding of stall 5 shown to be actually defective by repeated desanding analyses.

Renewed trouble on the large stall crane. A coal converter, presumably not hooked in straight, slips out of the traverse while being tipped

over and falls on a liquid phase converter, which breaks like glass at the bottom, but, fortunately, does not touch the crane leg itself.

In the splitting plant a steam pipe bursts on an Oschatz boiler. At the same time there is a short circuit in the fuel gas/splitting gas exchanger on furnace 3.

In the coal pasting plant the accumulations on the shaking screens in Bldg. 45 seem to be related to the load on the mills in Bldg. 43. With a mill load of 15T of dry coal the screen tailings = 0-500 kg/shift, with 25T dry coal the tailings = 1000-1500 kg/shift.

The addition of soda, 0.1% based on dry coal, is discontinued, because the wear in the preheater coils in stall 3, shut down after 25 days of operation, was just as severe as elsewhere without soda.

June 28-29

Air attack. About 50 fire bombs fell into the plant. Small fire near distillation, which was quickly put out. Idling. Restarting without incidents. Loss in production about 500%.

July 3-4

Air attack. About 100 fire bombs fell inside the plant. The roof of the gas holder is pierced by a fire bomb, a small gas flame. Idling with expansion. Restarting without trouble. Vapor phase stall 7 (5058) shows increased resistance after restarting.

July 8-9

Air attack. Idling. CO₂ and CO scrubbing are expanded to about 10 atm. Liquid phase high pressure system is inadvertently expanded to 80 atm through the expansion lines at the booster compressors because of negligence in idling to shut the 700 atm make-up gas valves. Failure of the U power circuit. Restarted plant on the 9th.

July 24-30

Stall 10 (6434) put on stream the 24th. The stall has 3 converters. On the 30th the injection was 45 m³/h. This was increased shortly afterwards to 48 m³/h = 40 T/h, corresponding to a catalyst load of 1 T/m³ reaction space.

The difference in density between feed and catchpot product is 75 to 80. The gasoline constituent of the catchpot product, to 155°C, is 52%. The feed contains 0.3 to 0.4% E₂S.

The inlet gas of the 6434 stall is washed to 0.2 mg H₂/m³, compared to 1 mg/m³, by the installation of a cooler for the waste waters of the Ni₂ scrubber.

August 1-10.

Collapse of brickwork in the fire box of B3-distillation (6434) preheater. It was found that only one side wall was affected.

To relieve B3-distillation, 6434 catchpot product (48 m³/h) is switched to B2-distillation and the 5058 catchpot product (35 m³/h), formerly processed by B2-distillation, switched to B3-distillation.

Gradual reduction of the injection of all coal stalls due to the deterioration of the K-values of the heat exchangers, i.e. for stall 3 from 45 to 40 m³, for stall 4 from 42 to 32 m³, and for stall 5 from 36 to 36 m³.

CO₂ instead of steam is added to the injected air in the weak gas generators. The weak gas contains a little more CO and correspondingly less H₂, while the heat value remains the same @ 1450 kcal.

August 10-11.

Temporary shutdown of 6434 stall because of a leak in the cooler. A similar shutdown had been necessary a week before.

Coal stall 6 is started with a different heat exchanger connection. In order to prevent the deposition of heavier particles from the coal paste in exchanger 1, the paste travels from top to bottom.

August 13.

Shutdown of plant because of complete power failure. In the splitting plant, the catalyst in a splitting furnace sintered because of a rise in temperature. The first converters of all 3 coal stalls in operation coked up. When restarting the plant, we found CO in the make-up gas, because of poor conversion due to lack of steam.

CO scrubbing got a raw gas with about 15% CO, which was washed out to a CO-content of 4%. Due to this CO-content the temperature in the 5058 stall rose to 30 mV, but since the stalls were still running without injection, the converter operators could get them under control.

August 20.

Failure of all 4 boilers. The low pressure steam drops to 1/2 atm; the high pressure, to about 3 atm.

The coal stalls are switched to pasting oil. The vapor phase stalls are sharply reduced. The splitting furnaces get their own 18 atm steam. All steam pumps are down, i.e. all coal paste, letdown, L.T.C. mixture and centrifuge mixture pumps. The letdown is switched to emergency letdown.

In Bldg. 182, the last preheater for L.T.C. mixture still in operation, coked up. The L.T.C. kilns process 2.5 tons without preheater and 3.0 tons/h with preheater.

August 27.

Coal stall 3 has to be shut down. It was materially effected by the current failure of August 12.

August 28.

Coal stall 5 has to be shut down because there is suddenly no gas flow. Converter 1 in particular is badly coked. The closest converters of these 2 stalls are to be installed in stall 3. The repairs will probably take 10 days. The large stall crane is out of commission for 2-1/2 days.

August 30.

Discussion with the sulfur company about the possible saving of sulfur. It was pointed out that we require sulfur for asphalt decomposition and the prevention of suit cracks in the liquid phase, as well as for sulfurizing the aviation gasoline stall feed with H_2S .

We have already tried to get along with a sulfur addition of 1.2% based on dry coal and to increase the yield in H_2S recovery by gassing the wash waters of the liquid phase rich gas scrubber with CO_2 , but due to the increase in asphalt the sulfur addition has had to be increased again to 1.5%.

The sulfur company has offered us gas cleaning media (Gastreinigungs-masse) charged with sulfur for use as catalyst. However, we already know that such gas cleaning media have a poorer catalytic effect than Pyrogasol.

September 1-2.

Trouble in L.F.O. because of too high asphalt content of the lotdown. To abate this difficulty the sulfur addition to the dry coal is increased from 1.2% to 1.5%.

September 8-15.

After restarting coal stall 3, the injection into coal stall 6 is reduced to 38 m^3 because the preheater was overloaded. Stalls 5 and 4 are getting an injection of 42 m^3 ; 45 m^3 produces too high asphalt values in the lotdown.

September 19-22.

Vapor phase stall 7 has to be shut down again because of excessive stall resistance. On dismantling, it was found that the lowest catalyst screen-tray in converter 5 was damaged. In consequence, catalyst got into the connecting line between converter 5 and the hot heat exchanger. Converter 5 had to be emptied for repairs.

In order to shift the rich gas/poor gas ratio towards the rich gas side, the pressure in the central lotdown vessels is increased in the vapor

phase, the liquid phase cold washes and the liquid phase wash oil. At the same time the quantity of lathum gas is lowered by reducing the heavy oil injection into the hot catchpots from 8 to 4 m³/h, thereby increasing the lathum concentration from 15 to 22% solids.

Discussion about the cleaning catalyst in the distilling plant. Dr. Neist will send us some other cleaning catalyst, to be used in the Fischer plant, to experiment with. The Fischer catalyst is improved with 2% alkali. Dr. Neist recommends a test @ 200°C.

September 25-26.

Further reduction in road tar delivery. For equalization the boiling end of the middle oil is reduced to 315°C, with consequent lower M-oil production.

October 1.

The asphalt decomposition of the coal stalle is considerably improved by the reduced injection and the temporary cessation of the road tar addition to the pasting oil. The asphalt content of the lathum oil drops from 25-30% to 15-20%.

October 7-14.

With the return to the former road tar addition of 9% to the pasting oil the asphalt content of the lathum is increased from 15-20% to 25-30%, and causes trouble in I.T.C. The change of the coal stalle is therefore somewhat reduced.

By adding CO₂ instead of steam to the air in the weak gas generators, an increase in production from 50000 to 70000 m³/h of weak gas is obtained.

October 15-18.

The mixture of thick and thin tar in the ratio of 1:1 is difficult to centrifuge. For this reason several parts of lathum condensate are added to the mixture, with good results.

Temporary pressure drop in the vapor phase by about 70 atm, with consequent greater gasification, particularly in the 6th stall. During this time I.P.C. produces 8 m³/h butane instead of 5 m³/h.

During the night of the 15th-16th, the coal paste concentration rises temporarily to 45%. The shaking screens in Bldg. 45 are running over. The coal stalle must be temporarily switched to pasting oil.

Experiments to reduce the CO₂ content of the water gas are to be made in the gas plant. The aim is to obtain a higher CO and H₂ content in the water gas, equivalent to an increase in production.

An attempt is also to be made to add as much CO_2 to the circulating gas injected into the generator shafts as is given off by the gas produced. The purpose is to increase production and save steam at the same time. It is assumed that steam conversion can operate more efficiently than the gas plant.

October 20.

Dr. Glessen submits an interesting report on the purity of n-butylene. It contains about 0.5% butadiene, which causes a lot of trouble in subsequent alkylation by increasing the H_2SO_4 consumption by 100-200%.

It was further reported that sulfur in the n-butene will cause trouble in dehydrogenation as well as in alkylation. It is recommended to scrub our liquid butene with NaOH before hydrogenation.

The 700 atm circulating gas scrubbers were unpacked and the heavily fouled filler cleaned. The load on the scrubber after the cleaning was 75000 m^3/h gas and 40 m^3/h oil, compared to 30000 m^3/h and 40 m^3/h , respectively, before.

October 25.

The 5 atm liquid phase rich gas compressor in the L.P.G. plant was destroyed by the breaking of the piston rod at the piston. One of the 3 - 25 atm compressors will be connected to the liquid phase rich gas.

November 2-4.

During one air raid on Cologne. On resuming production the bell outlet valve on gas holder 15 could not be opened. The compressor got hot catalyst got directly from the systems, because the catalyst system coolers were stopped with CO_2 -water too late. This delayed the plant resumption 1 hour.

November 6.

Because of carelessness of an operator the catchpot of a vapor phase still ran over, causing liquid knocks in several circulating pumps, though no serious trouble. The application of level indicators on the separators in the circulating pump suction lines is imperative.

November 20-22.

Continuous trouble with the water injection pumps on the vapor phase circulating system. These result in insufficient water sprays on the air scrubbing tower ahead of the 6 $\frac{1}{2}$ stall, 1 m^3 H_2O injection instead of 4 m^3 . Performance of the 6 $\frac{1}{2}$ stall drops considerably, due to the poor H_2 scrubbing.

In connection herewith the octane number of our aviation gasoline drops temporarily to below 70 (69.6). Consequently, the 2058 catchpot product is cut off at 155 instead of 145°C, and the 6 $\frac{1}{2}$ product at 155 instead of 165°C.

1537

November 27-29.

During the night of November 27-28th, current failure in circuits U and K. Because of improper hook-up of the motors driving the lubricating oil pumps on the 700 atm circulating pumps, all these circulating pumps are shut down. All coal piles must be emergency expended.

Fire in H₂-distillation, 300^o catchpot product, on the 29th. The column safety valve slips out of wire rope and smashes the product line between the heat exchangers and preheater. A small fire ensues, which causes little damage, though the plant is down for 20 hours.

December 15.

Shutdown of stall 5 due to a leak in the descending line.

January 24, 1954.

Three CO₂-scrubbers cleaned free of algae with chlorine water. Chlorine solution: 50 g/l of water in 6 batches, at about 30^oC. The water was circulated for 20 hours by a 50 m³/h pump.

Suggestion for a production premium pay for converter operators.

The fuel gas for the splitting plant is not to be preheated in the future. Compressed air to be injected into the splitting gas from the splitting furnace for cooling the gas. The fuel gas heat exchangers can probably be used for other purposes.

January 26.

Attempt to start stall 5, provided with a new descending device. The installation of converter I consists of a riser pipe 6 m long, a make-up gas inlet in the back channel and a 2 m dia x 9 m long direct connection between converters I and II. The connecting line was plugged. Considerable quantities of solid deposits, mainly calcium carbonate, from the preheater were found on dismantling.

The next stall equipped with such a descending device will probably also retain the old descending device, in order to be able to let down deposits of solids, with high pressure differences, as the case may be, when starting up after idling and during other difficulties.

A decrease of calcium carbonate deposits on the flange of the fine gas blower, probably caused by the entry of stuffing box water into the blower.

January 30.

The first attempt with a new descending device, in stall 3, connecting line between converter I and II, seems to prove satisfactory. However, a protecting device against salt crystals torn out of the preheater hairpin coils should be provided.

February 17.

Fire in gas holder 57. The gas holder spilled over due to improper operation. The spilled gas, rich in gasoline, caught fire. Eight men were killed and others badly hurt.

February 2.

Overflow of copper liquor in one of the washers due to the failure of a sight glass to function properly. The liquor got as far as the booster compressors. These were saved from destruction by immediate shutdown.

March 4.

On starting up after idling too little steam injected into conversion. The CO content of the catalyst gas was 25%, that of the pure H_2 was 10%. The make-up gas valves on the vapor phase apparatus were shut immediately and the injection into the 643rd stall was stopped. The temperature in the first converters of all coal stalls rose to 26.5 °C. The converters do not seem to be coked.

Difficulties in the dephenolizing plant, caused by a rise in the viscosity of the dephenolizing oil after only little use and consequent deterioration of the phenol scrubbing. According to Leuna the dephenolizing oil no longer contains any triphenylphosphate, compared to 60% formerly. In the future the dephenolizing oil is to contain about 35% triphenylphosphate.

March 30.

Fire between Bldgs. 21 and 22, caused by a sudden leak in a let-down valve. Consequent temporary shutoff of the gas supply to hydrogenation.

May 1.

The CO-content of the raw H_2 rose occasionally to 8% and could just be washed out by utilizing our entire copper liquor capacity.

May 4-5.

The splitting gas is being supplied to conversion experimentally thru separate catalyst systems. Although the splitting gas contained only 15% CO , the catalyst systems continued to operate autothermal. This method of operation will probably be retained in the future with maximum throughput.

April 22.

The first stall provided with a short descending line between converters I and II had been running 49 days without any trouble. Then the stall had to be shut down due to plugging of the short descending line. On dismantling the stall especially severe salt encrustations were found in converter I. The lower part of the converter, provided with a risertube, is badly obstructed with salt crusts, and coarse particles of catalyst and coal.

Converter II contains some badly worn miniature catalyst, apparently formed during the first few days of the operating period, in which the temperature in the lower part of converter I was held below 24 MF.

May 20-24.

All coal stails are provided with a make-up gas connection to the hot catchpots. This seems to reduce the heavy oil consumption of the stails, probably due to better asphalt decomposition and lower stall temperature, since now as before a letdown concentration of 20% is maintained, obviously produced by subsequent (reactions) concentration of the hot catchpot contents. Accordingly, the converters are probably running with a lower solids concentration than formerly.

May 27.

Beginning today the letdown gas, which contains 90% H₂ due to the injection of make-up gas into the hot catchpot, will be fed into the catalyst gas ahead of gas holder 15.

June 2-3.

Idling. On restarting an explosion occurred in the air lines in Bldg. 5, which delayed operations 2 hours. In the future the air lines are to be flushed with CO₂ before starting.

All coal stails are provided with automatic liquid-level controls on the hot catchpots.

June 21-22.

Large scale air attack on the plant, which largely failed of its objective because of the excellent cooperation of the various anti-aircraft units. Only 20 bombs fell into the plant, of which 15 exploded, 5 being dud or delayed action bombs doing little damage. Little damage was done to pipe bridges and tank cars, causing small fires, which were soon put out. Some bombs fell into the workers camp, killing 4 women and wounding 10. Other losses consisted of 1 watchman killed near the tank farm.

The plant was immediately switched to idling and shut down 10 minutes later. It was restarted with little trouble. The letdown line of a coal stall had become plugged due to solidification of its contents and the descending operation of coal stall 3 did not function any more after 130 operating days.

June 30.

Restarting of coal stall 6, provided with a counter-current heat exchanger. This heat exchanger works as follows:

The coal paste flows from top to bottom, the gas from bottom to top. By uniting the paste stream coming off the bottom with the gas coming off the top at mid-height of the heat exchanger, the exchanger is kept full

of liquid. The deposition of heavy solids particles is to be prevented by the downward flow of the paste and simultaneous intermixture with the upward flowing gas, with the exchanger full of liquid.

In stall 4, started June 17th, the hot catchpot was provided with baffles. These baffles are to effect a temperature equalization of outgoing product and incoming make-up gas. This device has proved satisfactory.

6434-stall 10 was shut down because of sudden plugging. The stall had shown excessive resistance for weeks previously, almost 35 atm @ 40 m³ injection and 25000 m³ inlet gas. The catalyst in all converters was changed. It had worked badly after a year's operating time and had forced only about 40% gasoline to 150°C in the catchpot product. However, the new catalyst did not work any better after restarting. The poor operating results must, therefore, be due to the quality of the feed, which contains about 20% bituminous-coal products.

July 14-16.

Shutdown of liquid phase product distillation. The catchpot product suddenly contained up to 1% water. Due to a short circuit in the paste heat exchanger of a coal stall, coal paste got into the catchpot product causing emulsion with water. This short circuit had apparently existed for 2 weeks without being noticed, because there is no coal paste in the upper part of this heat exchanger (counter current).

A Pintsch-Hilshbrand watergas generator has been operating since July 12th with the addition of 400 m³ bygas. The purpose of this operating method is to increase production and relieve the splitting plant. The gas production of the generator is 900 m³ above normal. The bygas is completely split; the methane content of the watergas is less than 1.5%.

Rise in temperature to 50 mF occurred in the 3 year old 5058/7846 stall 7. The stall was shut down and the catalyst changed. The former converter arrangement, 5058/7846/7846, was changed to 7846/7846/5058, as at Politz.

A coal paste heat exchanger was flushed with liquid phase cold catchpot residue, at the same time blowing it out with CO₂ and heating it with high pressure steam. The flushing operation seems to have been successful. It is hoped to avoid more extensive repairs on the heat exchanger by flushing in this manner.

July 18-19, 1944.

Severe air attack at night. About 1000 explosive bombs hit the plant, mostly 250 kg. The housing area was also badly hit.

This attack was favorable for the enemy planes, because there was no smoke screen and flames dropped by the planes lit up the plant. Heavy fires occurred shortly after the first bombs exploded. All communications with the outside were destroyed.

The plant was idled and shutdown conscientiously, although only 4 minutes were available. The losses in the plant were 3 dead and 25 wounded.

The following damage was done:

Largely destroyed were the gas plant, coal preparation plant, compressor building, circulating pump building, intermediate tankage and heavy oil pump house. Almost all gas holders were destroyed. Only the hy-rich gas holder and the hy-fuel gas holder can be repaired.

Almost no damage was done to the smaller tanks for H_2 and H_2S . The stalls, splitting plant, conversion, CO-cleaning, distillation, L.P.G. and dephenolizing units were less heavily damaged. Plant piping was damaged in many places, as were the water supply and sewerage systems, as well as the power and telephone cables.

We are counting on a 2-3 month shutdown, when 75% production is expected.

NOTES: This seems to be the end of the Wesseling Hydrogenation Works, since the war ended less than 9 months later and allied forces were close at hand.

The terms coal stall, coal converter, or coal catchpot are synonymous with liquid phase stall converter or catchpot.

The terms gasoline stall, gasoline converter, or gasoline catchpot are synonymous with vapor phase stall, converter or catchpot.

Where not otherwise indicated, production figures in German plants are usually given in units per hour.