

Frame 468

June 23, 1941.

Liquid phase catalysts.

According to rotating autoclave tests, char-iron catalysts were definitely improved by the addition of FeF_3 , and it might be advisable to test it in 10-liter converters.

Char-iron catalysts were found so satisfactory in 10-liter tests of pitch hydrogenation that no difficulties are to be expected on a larger scale. Any possible changes might be increased amount of letdown and a slight rise in gasification. The yield might be reduced by about 1% through higher gasification and by 1-3% by a larger amount of letdown. The results might be improved by the addition of chlorine, which is not to be recommended with the present materials and without neutralization. The use of the fluorine-containing catalysts might raise the results to the molybdenum catalysts values.

/s/ Donath

Frame 469

June 23, 1941.

Liquid phase catalysts.

Chlorine is the only molybdenum-free catalyst which does not increase the amount of ash, and since the addition of chlorine to a number of intermediate catalysts permits expecting good results, chlorine should be again tested as catalysts or catalyst component.

/s/ Peters

Frame 470

June 21, 1941.

Remarks on liquid phase catalysts.

Frame 471

Liquid phase catalysts for tar hydrogenation. June 23, 1941.

No difficulties were encountered in kilning letdown from hydrogenation of lignite tar with iron catalysts, and asphalts were adequately reduced.

Greater difficulties arise, however, in hydrogenating bituminous coal tar or pitch by insufficient reduction of asphalts at high pressure. The difficulties manifested themselves during kilning by formation of coke which could no longer be ground because of its hardness and led to the plugging up of the ball kilns. Such letdowns could, however, be topped and used to replace briquetting pitch.

Poelitz found that difficulties arose in the condensation installation behind the ball kilns when processing letdowns from mixtures of pitch with sufficient solids and asphalt hydrogenation

(when using molybdenum), because the Raschig rings were plugged up by deposition of solid aromatics (coronene, pyrene, etc.).

Kilning proceeded smoothly upon addition of coal to letdowns from mixtures of pitches (iron catalysts).

Frames 472-476

Rotating kiln tests of hydrogenation of Puertollano coal. March 29, 1943.

Summary

Puertollano coal is a young bituminous coal with 26.5% ash and is similar to Upper Silesian coal. Deashing of the coal in the laboratory was less successful (about 32% yield against 50% with Upper Silesian coal).

Hydrogenation tests of the deashed coal were more successful in utilization and gasification than with Silesian coal.

The un-deashed coal was extracted and hydrogenated (600 atm., middle oil pasting, high space utilization, 435° C.) because of the unpromising results of deashing. The high ash content of the coal caused more splitting, gasification, and greater reduction in asphalts than with Silesian coals with only 5% ash. The products filter well in the beginning, but rapidly become poorer than with Silesian coal, probably caused by the higher solids content.

/s/ Grassl

Frame 477

Table of hydrogenation results of Upper Silesian coal to 1:1 gasoline and middle oil, gasoline + 25% fuel oil and 75% middle oil, and to gasoline + 50% fuel oil + 50% middle oil.

Frames 478-483

Hydrogenation of Bruex tar in rotating autoclaves. April 3, 1943.

Summary

Phenol reduction differed little with the different catalysts. Information was obtained on better reduction of phenols with acid catalysts than with alkaline or neutral. Char-molybdenum offers but slight advantages over acid-reacting char-iron catalysts, but is superior to the iron catalysts in asphalt reduction in gasification.

An increase in hydrogen pressure acts favorably upon Bruex tar by raising the asphalt reduction and solids utilization. Phenol reduction is also definitely better at 600 atm. than at 300 atm. Asphalt reduction is much poorer at pressures below 200 atm., new solids are formed, and

splitting is also reduced. Splitting increases hardly at all between 300 and 600 atms. The throughput could be more than four times greater at 600 atms. than at 150 atms., and nearly double that at 300 atm.

Frames 484-505

Hydrogenation of bituminous coal coking tar with addition of bituminous coal.
May 10, 1943.

Summary

Short tests were run on the addition of bituminous coal during the hydrogenation of tar and pitch.

The coal was saturated with 1.0-1.2% iron sulfate.

By adding 20% coal to a relatively low-solids and asphalt mixture of bituminous coal residues and tar oil before the addition of 2% alkalinized iron-char catalysts, the space-time yield was the same when operating in a single pass. Somewhat higher gasification and some additional asphalt in the letdown must be attributed to coal addition without any corresponding increase in solids. Kilning results were, however, somewhat improved, which points to somewhat better reduction of tar asphalts caused by the addition of coal, and to the better kilning behavior of coal asphalts than of the tar asphalts.

A mixture of tar and coal could also be smoothly hydrogenated to gasoline and middle oil.

A bituminous coal-tar pitch with 24% benzene-insolubles and 36% asphalt which could not be hydrogenated with either the iron catalysts nor even with molybdenum-char, could be hydrogenated upon the addition of 20-25% coal to gasoline and middle oil, with the still acceptable space-time yield of 0.25. Kilning was good in this case also.

Coal addition had no important effect upon the properties of the oil produced.

Frames 506-536

Hydrogenation of Silesian coal of the Castellengo-Abwehr to heavy oil excess at 600 atm. in 10-1. converters. July 8, 1941.

1:1 mixture of nut coal of the mines Castellengo (25/10 mm.) and Abwehr (20/10 mm.) in Silesia, deashed with CaCl_2 .

Summary

The suitability of this coal to hydrogenation to gasoline and middle oil was tested previously. In the report of that investigation, this coal was stated to behave much less favorable to hydrogenation to 100% gasoline + middle oil than the Gelsenberg coal, and much more unfavorably than might have been expected from its analyses.

The coal was to be tested for hydrogenation to excess heavy oil (fuel oil), in the hopes that the coal will then behave relatively more favorable than in the complete splitting of the hydrogenation products to gasoline and middle oil, which always proceeds in Silesian coals with more gasification than with Gelsenberg coal of the same C content.

Such is actually the case, as shown in the accompanying tables. Hydrogenation to heavy oil excess is compared in columns 1 and 2, and to gasoline + middle oil in columns 3 and 4 for the two kinds of coal.

Hydrogenated to:	Heavy oil excess		Gasoline + m.o.	
	Upper Silesia 478	Gelsenberg 477	Upper Silesia 470	Gelsenberg 468
Coal				
Temperature, °C.				
Throughput (kg. m.a.f. coal/l., hr.)	0.76	0.81	0.47	0.44
Conversion, %	95.4	96.4	95.4	96.1
Space-time yield (Oil production)	0.54	0.55	0.26	0.27
Gasification, %	20.8	20.5	28.9	24.7
Percent asphalt/m.a.f. coal	17.3	11.5	10.0	7.4

The conversion of the Silesian coal by both methods is uniformly somewhat poorer than of the Gelsenberg coal. Gasification of the Upper Silesian coal in conversion to gasoline and middle oil is appreciably higher for the same space-time yield, while in the process for heavy oil excess, gasification and space time yields are the same.

Only asphalt reduction is appreciably better with Gelsenberg coal in the heavy oil excess process than in the conversion of the two coals to gasoline and middle oil. More careful processing was required in kilning in ball kilns of the Silesian coal because of the poorer asphalt reduction (reduction of throughput). The asphalt decomposition was thereby improved at the cost of reduced space-time yield and slight increase in gasification.

Even with this limitation, expectations are justified that this Silesian coal is preferably hydrogenated to excess of heavy oil than to gasoline and middle oil.

Frame 537

Preliminary results of hydrogenation of different feeds with elementary sulfur.
August 12, 1943.

Summary

Hydrogenation with elementary sulfur completely dissolved at 100° C. in the corresponding oil gave the following results with different feeds:

1. Splitting of Rumanian crudes with only 0.5% asphalt was slightly improved upon the addition of 5% elementary sulfur over hydrogenation with 1.5% char iron catalysts. The products obtained with

char catalysts were somewhat better (lighter in color).

2. Worms crude oil is rich in asphalt and sulfur, similar to Albanian crudes, and was used for lack of Albanian oil. Somewhat less favorable results were obtained in splitting and asphalt reduction upon addition of 2-5% elementary sulfur, than with the char catalysts. With 5% sulfur, splitting was increased by 2% and asphalt was somewhat lower, but the specific gravity of the residues of the products above 325° was higher, pointing to polymerizing action of sulfur.

3. Splitting was also somewhat improved by addition of S to Bruex tar, which is also rich in asphalt and sulfur, but the asphalt reduction was not improved. The color with the char-iron catalyst was better.

4. Bituminous coal l.t.car. tar. Results can so far be evaluated only from appearance; products with 2 and 5% sulfur were similar as with char-iron catalysts.

5. The splitting of bituminous coal-tar pitch with 5% sulfur was somewhat lower, but the asphalt reduction better than with char catalysts. The specific gravity of the residues above 325° were appreciably higher than with char catalysts.

6. Products of hydrogenation of Upper Silesian bituminous coal with 4.9% ash with 2 or 5% sulfur were apparently of as good quality as could be expected with the usual iron catalysts.

/s/ Grassl

Frames 538-539

H.O.L.D. filtration tests with addition of acetic acid ester, neosolvan and their mixtures with furfural. March 28, 1942.

Acetic acid ester, and particularly its mixture with furfural (1:1) was found to be exceptionally suitable for treating the H.O.L.D.: The filtration times were much shortened, the oil yield amounted to over 90% and the residue was dry and finely grained to powdery.

Acetic acid ester cannot be considered for industrial purposes because of its cost, and a cheap commercial mixture, consisting chiefly of ethyl, propyl, and butyl acetates (neosolvan), was used in H.O.L.D. filtration tests. Results were almost as favorable as pure acetic acid ester. The filtration rate was good, but did not exceed one minute for 33 gm. H.O.L.D.; the oil yield in the filtrate did not sink below 80% and the asphalt content of the filtrate was not below 80%; the cake was also powdery and dry. These results were obtained with 1:1 mixtures. Greater dilutions were naturally more favorable by increasing the filtration rate, but the analytical results were not improved in the same proportion. Acetic acid ester, acetic acid ester + furfural, neosolvan, neosolvan + furfural (1:1) and neosolvan + middle oil mixtures were tested. Mixtures were permitted to act upon the H.O.L.D. for one hour at 80°, and then filtered in the usual way. The analytical results

obtained with middle oil mixture were good, but the filtration rate was too small for industrial applications. The following H.O.L.D.-neosolvan mixtures were found to be particularly good in every respect.

Filtration times

1. H.O.L.D.-neosolvan, 1:1	42 seconds
2. H.O.L.D.-neosolvan-furfurol, 1:1:1	45 "
3. H.O.L.D.-neosolvan-furfurol, 1:0.5:0.5	60 "

The oil yields in the filtrate, practically solid-free were:

102.6% for No. 1
90.2% for No. 2
82.2% for No. 3

The so-called benzene solubles of H.O.L.D., to which all values are referred, amount to 78%. Neosolvan dissolves 83% from H.O.L.D., while 1:1 mixture of neosolvan-furfurol dissolves only 60%. For this reason, the oil yields under 2 and 3 are particularly good. Exact balancing will necessarily demand the use of data valid for all solvents. In the following report this was omitted to make the data more comparable.

The asphalt values were:

77.1% for No. 1
102.7% for No. 2
101.3% for No. 3

The following values were obtained for the solvent-free residue.

Solids:	1	55.6
	2	73.7
	3	78.6
Oil:	1	11.3
	2	14.6
	3	6.8
Asphalt:	1	7.7
	2	15.4
	3	4.5

It is worth pointing out that the oil and asphalt values were lower in No. 3 with the higher H.O.L.D. concentration than with the lower concentration in No. 2.

These solvents should therefore be tested on a larger scale.

Frames 540-553

Kipp filter tests with Zeitz low temperature hydrogenation catchpot bottoms (T.T.H.).
March 15, 1943.

Kipp filtration tests for the elucidation of the role of filter aids in the dewaxing of the TTH catchpot bottoms.

Summary

Testing of Zeitz residual waxes on several dates between October 16, 1942, and January 11, 1943, showed a surprising increase of density in the course of time regardless of the length of the cut of the bottoms (d_{70} 0.7895 to 0.8300), and a rise in the melting point (60 to 70° C.). The bottoms evidently become enriched with high molecular weight substances. To prevent their entering the distillate paraffin, efforts were made to withdraw suitable amounts from the process, and to return them to hydrogenation.

The residual wax obtained on October 30, 1942, was by far the worst. The residual wax of January 11, 1943, was obtained as a 10% distillation residue and was therefore naturally less active than shorter cuts, and 3-5% of it had the same effect (d_{70} = 0.780) as 0.5% filter aid A (Paraflow). The residual waxes on other dates formed 4.2, 4.6 and 5.9% of the sample and had 38%, 47% and 35% respectively lower activity than calculated on the assumption of a wax yield of 44% and 0.5% of filter aid in the TTH catchpot bottoms.

It was further found that the already used filter aid A was not harmed when the paraffin was distilled off, if distillation was conducted in a vacuum (10 mm. Hg) with steam at 300° C.; that some harm was caused by distilling the wax in vacuum (10 mm. Hg) at 360°, that the damage was much greater when distilling in a lower vacuum (80-100 mm. Hg) at 410°, and that the filter aid A becomes practically inactive after the wax was distilled to 410°, and reused three times.

A mild distillation of the wax will therefore protect the filter aid A.

Further comparative tests with present day filter aids and filter aids of earlier origin showed them to possess practically equal filtration properties. The Oppau aluminum chloride residue was found to be practically inactive. On the other hand, 2% Luetzkendorf and Ludwigs-hafen asphalt from Nienhagen oil behaved about equal to 0.5% filter aid A as far as the wax quality was concerned; the oil still contains small amounts of asphalt.

Frames 554-559

Kipp Filter Tests in Dewaxing of Crude Wax from Luetzkendorf Samples of Laboratory Scale Dehydrogenation and from Zeitz TTH Catchpot Bottoms.
April 21, 1943.

Frames 560-571

Rotating Filter Tests with Zeitz Low Temperature Hydrogenation (T.T.H.) Bottoms.
April 26, 1943.

Summary

Tests with Zeitz T.T.H. catchpot bottoms were run in the Ludwigshafen rotating filter test unit. Tests were run in batches, using filter aid A (Paraflo) and propane wax from Nienhagen oil.

The production of 170 liters/m.², hr. in the first stage, using filter aid A, can be readily reached with a dilution of 3 to 3½ and is not increased by using asphalt. The production in the second stage is surprisingly high with both filter aids. With the filter aid A, the filtration rate reached 133 l./m.², hr. or 160% of the expected production, and with the asphalt-over 200 l./m.², hr. or 190%.

The specific gravity of the feed was 0.7835 and became 0.7825 with filter aid A and 0.788 in the asphalt tests, or 0.782 asphalt-free, which means that the purity in the asphalt tests was as high as obtained in tests with filter aid A. 80-82% of distillation wax has a specific gravity of 0.777-0.778.

Tests of the T.T.H. wax from the second stage showed it to have a specific gravity of 0.777, while the specific gravity of 80-90% distillate was 0.775.

The Oppau specifies distillation wax of specific gravity 0.774, which cannot be met in two-stage dewaxing of the T.T.H. catchpot bottoms, even with complete de-oiling of the wax.

Wax could not be completely de-oiled even at 2 atm. pressure in the second stage, i.e. at high temperatures (-200). The 3-5.8% wax distillation fraction had a specific gravity of 0.7775, but somewhat higher melting point (53.5). Its oil content was, therefore, somewhat less than of the normal wax distillation fraction, specific gravity 0.777-0.778, m.p. 51° C. The filter rate tests were more successful by reaching 300 to 500 liters/m.², hr.

Frame 572

10-l. converter tests on hydrogenation of older coals at 300 atm. with tin oxide and chlorine. June 10, 1941.

Frames 573-578

T.T.H. hydrogenation of Deuben l.t.car. tar with alumina-molybdenum catalysts 7846 and 5058 + 7846 at 250 atm. April 1, 1941.

Summary

Filtered Deuben l.t.car. tar can be hydrogenated over alumina-molybdenum catalysts 7846 by the T.T.H. process without reduction of catalyst activity and with the production of good Diesel oil, crude lubricating oil and wax.

Preliminary hydrogenation over 5058 improves somewhat the lubricating oil yield and improves the cetane number of gas oil by one or more units.

The quality of the products and the yield with 7846 are at least as good at the same temperature, as with 6719. Gas oil and lubricating oil are somewhat better refined with 7846, and the oxygen somewhat lower in the products. The reaction temperature is 400 higher than with 5058. The lubricating oil yield is somewhat higher with 5058, and so is the cetane number of the gas oil.

Frames 579-593

Hydrogenation of old Gelsenberg coal at 600 atm. to gasoline and middle oil.
June 4, 1943.

A Ruhr coal with 86.6% C was hydrogenated in older tests in 10-liter converters at 600 atm. to gasoline and middle oil. 3% sulfurized Lux mass was the catalyst used. The activity of this catalyst is somewhat exceeded by those used today (combination of iron sulfate, Lux or Bayer mass, sodium sulfide), but hydrogenation proceeded smoothly, except that the chlorine content of the effluent water was too high for lack of neutralization.

The coal was very well converted, but the space-time yield, gasification and asphalt reduction was somewhat poorer than with the younger Gelsenberg coal. The oils produced were more highly aromatic, and the octane number of the liquid phase gasoline was higher.

Asphalt reduction was appreciably improved with the same space-time yield and gasification in a short test with 1.5% Lux mass, 0.03% tin oxide, and 0.1% ammonium chloride.

Frames 594-628

Hydrogenation of German petroleums in 10-liter converters. February 5, 1943.

Topping residues of German petroleums were hydrogenated with char catalysts in 10-liter converters at 600 and 250 atm.

The copy is very nearly illegible.

Frames 629-643

Report of an inspection in Poelitz. August, 1942.

The three liquid phase stalls operate with the same throughput. The liquid phase stall catchpot bottoms are mixed with the bottoms from Bruex tar stall, and hydrogenated in the tar stall. The amount of catchpot bottoms produced is not uniform, leaving a small excess.

The thin paste is produced by mixing thick paste with pasting oil on the pressure side of the pumps (mixing distance about 50-60 m.).

The coal grinding plant does not work satisfactorily and the fineness of the grind is affected with increase in load (3-4 stalls).

Poelitz is of the opinion that the conversion is relatively poor because of the poor grinding of coal, which manifests itself immediately by failure of rocker screens. Poelitz intends to supplement its grinding aggregate the way Gelsenberg has done it.

Coal contains
Thick paste
Catalyst

Pasting oil

6.5% ash/dry coal
52-55% solids
1.2% FeSO_4
2.7% Bayer mass
0.15% sodium sulfide
8-9% solids
25% ash in solids

Screen Analysis of Coal Paste

	<u>Good grinding</u>	<u>Poor grinding</u>
>0.3 mm.	1.5%	> 3%
>0.1 mm.	14.15%	> 20%
through 250 mesh screen	over 70-75%	65%

The thin paste contains

31-35% total solids
25-26% m.a.f. coal

The m.a.f. coal throughput is about 0.37 kg./l., hr. (35.5 m.³ reaction space per stall).

Conversion 92%
Space-time efficiency 0.23
Gasification 25%

Cold catchpot bottoms
H.O.L.D.

39.5-42% -325° C.
22% solids
38% ash in solids
2.5-3% -325° in oil
18-19% asphalt in oil

Centrifuging:

H.O.L.D. diluted with 50% catchpot bottoms to 15% solids
and produces 8.9% solids in oil
and 41% solids in the cake.

Throughput 3-4 tonnes/centrifuge. Only 20 out of
40 centrifuges are in use for three stalls.

Kilns: Maximum throughput 4500 kg., yield 75%, kiln residue 0.3-0.4% oil.

Kiln gas 30 m.³/T. feed (formerly 76-80 m.³).

S + Na₂S are used as catalysts. The kiln residue is sold.

Six of the ball kilns were in use for three stalls.

Heat exchange in one of the stalls on different dates about one week apart:

K values

Heat exchanger I	Heat exchanger II	Heat exchanger III
272	440	282
259	350	265
390	294	246
276	302	252

2. Bruex tar liquid phase

Feed: Bruex tar + catchpot bottoms 70:30. The catchpot bottoms consist of about 3 parts from the coal stalls, 1 part from Bruex tar. The stall is equipped for thick paste and thin paste passes like the coal stall. It has four converters and three heat exchangers.

Composition of the feed (Bruex tar + catchpot bottoms 70:30).

Sp. gr. 1.02/20°
Solids 1.8%
I.b.p. 290°
% -325 20-15
Asphalt 9-11%

Catchpot products:

Sp. gr. 0.960/0.970/20°
Gasoline - 160° 3-4%
Middle oil 160-325 39-40%
Aniline point of m.o. +10-+12

H.O.L.D.:

% solids 18-22
Ash in solids 45-50%
Oil - 325° 12-15 !!
Asphalt 0.5-1 %
Vacuum residue over 325°/10 mm. 8-12%, color blue

8-10% of H.O.L.D. is produced referred to make-up tar. Catalysts: 1.5% Leuna catalysts + 0.5% flowers of sulfur added to 30% solid paste (catchpot bottoms used for pasting oil). The H.O.L.D. is combined with the H.O.L.D. from the petroleum stall and vacuum distilled.

Total space-time yield 0.32
Gasification 14-16%
Asphalt reduction 99-100%

3. Petroleum residues liquid phase stall

Rumanian oil residuum over 360° + recycled catchpot bottoms are used as feed in proportion 70:30. The total feed is heat exchanged. Three converters, three heat exchangers.

Feed (oil residuum + catchpot bottoms 70:30)

Sp. gr.	0.915/50°
I.b.p.	370°
Vacuum residue	above 325°/10 mm. 15%
Solids	0.3%
Ash in solids	50%
Asphalt	1%

Catchpot products:

Sp. gr.	0.890/20°
Gasoline -160° C.	4%
Middle oil, 160-370°	53.1-56%
A.p. of m.o.	50-60
Sp. gr. of m.o.	0.860/20

H.O.L.D.

Sp. gr.	0.960/100°
Solids	18-23%
Ash in solids	48-50%
% oil - 370°	20-25%
No asphalt	
Vacuum residue of oil over 325°/10 mm.	10-15%, color blue

Catalysts: 1.5% Leuna catalysts with fresh oil in H.O.L.D. pasted to 30% paste (10% catalysts). The catalyst was formerly pasted with coal catchpot bottoms, but this resulted in deposition of higher aromatics in the stall outlet.

5-6% H.O.L.D. is removed and vacuum distilled with the H.O.L.D. from the tar stall. 50% oil was distilled off and the residue used as a fuel in one steam kettle.

Petroleum residues are very readily hydrogenated at 700 atm. because of their low asphalt and vacuum distillation residues. The amount of catalyst (1.5%) is high. A slight increase in converter temperature results in disappearance of H.O.L.D. The hot catchpot must be operated lower to prevent drying out of the H.O.L.D., which results in a higher middle oil content in the H.O.L.D. and poorer utilization of the heat exchange. The latter limits the possibility of producing more H.O.L.D. by increasing the throughput, because the preheater is used to capacity.

No hot recycling or mixing gas is used in the stall.

Make-up oil throughput is 0.45-0.46 with 26.7 m.³ reaction space.

Gasification 15-17%, space-time yield 0.33.

4. Scrubbing in the liquid phase operations: 5 scrubbers

		<u>Gas</u>	<u>Scrubbing oil</u>
Scrubber load:	Scrubber I	40,000 m. ³	28 m. ³
	Do. II	Do.	Do.
	Do. III	46,000 m. ³	Do.
	Do. IV	4,000 m. ³	Do.
	Do. V	45,000 m. ³	Do.
Gas density, inlet		0.269, temp. inlet 41°	
Gas density, outlet		0.248, do. outlet 42°	
Pressure, inlet		620 atm.	
Pressure, outlet		580 atm.	
Scrubbing oil consumption		0.60-0.70 kg. scrubbing oil/m. ³ gas	

5. Saturation stall

1 stall gasoline refining: 1 converter catalyst 5058, 2 converters catalyst 7846, 2 heat exchangers, reaction volume 8 + 16 m.³.

Connections: Heat exchanger → 5058 converter → gas heated preheater → 2 7846 converters.

Feed: 40% coal liquid phase gasoline + 30% Bruex tar gasoline + 30% petroleum liquid phase gasoline.

One Saturation Stall

Connections: 2 heat exchangers → one 5058 converter → gas-heated preheater → two 7846 converters. Reaction volume 8 + 16 m.³.

Feed: 55% coal + tar middle oil + 15% Bruex tar middle oil + 30% petroleum middle oil.

One Saturation Stall

Connections: 2 heat exchangers → 1 electric preheater → two 5058 converters. Reaction volume 16 m.³.

Feed: As above.

Frames 644-645

Composition of gasification in the 600 atm. process for heavy oil production operating with Silesian bituminous coal and bituminous coal coking tar. January 31, 1941.

Frames 646-648

Substitution for ammonium chloride (rotating autoclave tests at 350 atm. pressure). January 3, 1944.

Ferrous chloride or aqueous solution of chlorine-containing alumina appear the best substitutes for ammonium chloride in hydrogenating chlorine-containing materials. An amount of ferrous chloride with

the same amount of chlorine produces results about equal to those with ammonium chloride, but with a slightly higher gasification. Chlorinated alumina produces nearly as good results as ammonium chloride but requires larger amounts than the latter because of the low chlorine content, which will involve greater amounts of residues to be processed. 1.9% water-soluble alumina is distinctly inferior: Gasification is considerably increased because of the higher temperature.

Hydrated aluminum chloride ($\text{AlCl}_3 \cdot 6 \text{H}_2\text{O}$) is appreciably inferior to ammonium chloride; raising the temperature by 3/10 mv. improves results, but gasification is then greatly increased. Two samples of aluminum oxychloride which contain more chlorine than the water-soluble alumina, gave better splitting, but failed to approach the ammonium chloride results in asphalt reduction and gasification.

Frames 649-652

Fuel oil discussion in Gelsenberg.

January 5, 1942.

Frames 653-654

Scholven experience with addition of cold paste. January 5, 1942.

One four-converter stall with 48 m.³ reaction space has cold paste injected into the second converter. Formerly the cold paste was injected into the emergency pressure release line in converter 2. The tee connection was found to be corroded by chlorine. The paste is now injected laterally into the second converter. A total of 34 m.³ of coal paste is fed daily to the stall. A higher temperature is maintained than without cold paste addition. This stall was recently equipped with three gas hairpins which permits increasing the throughput from 25 m.³ to 28-30 m.³ even without cold paste injection, and the cold paste injection amounts to only 5 m.³ injection paste. 100% gasoline + middle oil are produced.

Comparison of a stall without and with cold paste injection

	No cold paste	With cold paste
Catchpot bottoms: H.O.L.D.,		
% by vol.	48:52	35:65
H.O.L.D.:coal	1.15:1	1.25:1
Solids in H.O.L.D.	26-28%	22-23%
Asphalt in H.O.L.D.	?	23%

The hot catchpot temperature in the stall with cold paste injection is too low, which causes a reduction of solids in the H.O.L.D. and affects adversely the catchpot bottoms: H.O.L.D. proportion. Should the figures be recalculated to dilution, the proper theoretical proportion will be again obtained.

The figures indicate that the stall with cold paste addition is in equilibrium like the other stalls and is not overloaded.