

Bag 2743, Target 30/4.09
Scholven - Item 5 (A)

Comparison of Hydrogenation Products of Gelsenberg and Scholven

June 12, 1940

In the following report are compared the analyses carried out at Scholven on the products of the Gelsenberg-Benzin A.G. from February and May 1940, and those of our plant from March and May 1940. For the purpose of eliminating accidental results as much as possible, two series of analyses were made in each case. There are differences even in a comparison of analyses from the same plant, particularly the Scholven gas-phase products. This can be attributed to the improved method of operation which has been introduced in the meantime.

Moreover, there are also found individual differences between the plants which are due to the various methods of operation. The latter is manifested in the sump phase in that Gelsenberg has a more extensive decomposition, and that the iron concentration in the ash is so high (Table 2) that a recovery of the latter can perhaps be considered. This more extensive decomposition, however, also brings about a more extensive destruction of the aromatic structure, which is manifested in the A-Benzines (Table 5) as well as in a lower C content of the Gelsenberg A-Middle Oil (Table 4). The fact that the aromatic content of the Scholven A-Benzine is nearly twice as high makes the difference even more pronounced (Table 5). The weaker sump-phase hydrogenation is even manifested in the high content of hydroaromatics in the finished benzine. It is probably necessary to take into consideration the fact that the content of pyridine bases in the Gelsenberg A-Benzine is considerably higher than that of the Scholven A-Benzine (last line of Table 5).

In the injection product of the 5058 chamber (Table 6) the great differences are caused by the fact that at Gelsenberg, contrary to what is done at Scholven, the A-Benzine is injected at the same time. For the same reason a comparison of the catch-pot products of these chambers (Table 7) is possible only with reservations. It is, however, interesting to note that the quantity of benzine is approximately the same in the two cases, from which it may be concluded that the injection of the A-Benzine considerably decreases the new formation of benzine. The hydrogenating action is more intensive at Scholven, which is particularly shown by the higher aniline point, and the ultimate analyses. This is also true in hydrogenation with 6434-catalyst at Scholven, as indicated by the aniline point, content of aromatic hydrocarbons, and ultimate analyses of the 6434 catch-pot product (Table 9). True, the Scholven benzine does not have a particularly good light test, which is due to the deficient light stability of the A-benzine. This is however only a defect in beauty since the benzine never comes in contact with light, and for this reason this test is not specified by the purchasers. By way of trial, the A-Benzine has recently been refined over 5058 catalyst, despite the losses connected therewith, whereby the light test was good.

It is, of course, possible to establish still further differences. If these are slight they must be considered particularly critically in order to avoid the danger of establishing variations in the process which are not actually present but are solely due to accidental differences in the taking

of samples or in the analyses.

The Scholven gas-phase method of operation has been improved in the last few weeks. By the addition of all the fresh hydrogen of the gas phase in the 6434 chamber there is obtained a better activity of the contact, which enables the reaction temperature, despite the increased injection of oil, to be reduced from the previous 23 mv to about 22 - 22.5 mv (30° Basis). Moreover, it is now possible for the undesired intensive hydrogenation action of the preliminary refining 5058 to be reduced by lowering the temperature from the previous 22 - 22.5 mv. to 21.5 - 22.0 mv. The octane number of our benzine is 73 and higher since these expedients were adopted.

Table 1Investigation of Coal Paste

	<u>Gelsenberg Coal Paste</u>		<u>Scholven Coal Paste</u>	
	<u>Feb. '40</u>	<u>May '40</u>	<u>March '40</u>	<u>May '40</u>
Solids in coal paste	58.2%	54.5%	54.2%	54.6%
Ash in solid material	9.9%	10.4%	7.5%	8.1%
Asphalt in oil	6.9%	10.8%	10.5%	11.5%
Water	1.2%	1.0%	0.7%	0.6%

Table 2

Investigation of "Abschlamm"
(Mixture of oil and ash and non-convertible constituents)

	Gelsenberg		Scholven	
	Feb. '40	May '40	March '40	May '40
d_{100}	1.200	1.206	1.250	1.250
Softening point	10.8°C	10.5°C	38° C	38° C
Solid material	24.1 %	24.2%	28.5%	28.5%
Ash in solid material	55.2%	58.5%	27.4%	27.4%
Asphalt in oil	12.1%	12.0%	22.5%	22.5%
% by wt. up to 325°	8.6%	7.6%	3.0%	3.0%
Soda in "Abschlamm"	0.0%	0.08%	0.0%	0.08%

Gelsenberg "Abschlamm"
reacts neutrally to
phenolphthalein.
Its methyl orange
alkalinity amounts to
12 cc N/10 H₂SO₄/100 g
"Abschlamm"

Vacuum distillation curve:

	124° C	123° C	122° C	129° C
18 mm Hg				
First Drop:				
Up to 150° C	3% Vol.	2% Vol.	1% Vol.	1% Vol.
200° C	14%	13%	8%	8%
225° C	27%	27%	21%	20%
250° C	39%	40%	27%	32%
300° C	56%	56%	47%	45%
350° C	66%	67%	58%	56%
	362°/72%	364°/72%	365°/62%	369°/61%
Decomposition:	362°	364°	365°	369°

Composition of Ash:

Soluble in water:

Na ₂ SO ₄	7.6%	6.9% and Cl
CaSO ₄	2.6%	
MgSO ₄	1.2%	

Insoluble in water:

SiO ₂	20.9%	32.2%
Fe ₂ O ₃	34.2%	12.8%
Al ₂ O ₃	23.5%	25.1%
CaO	0.9%	2.9%
MgO	2.2%	6.2%
Na ₂ O + K ₂ O	4.3%	4.9%
P ₂ O ₅	0.3%	0.9%
SO ₃	2.1%	7.7%

Table 3

Investigation of Liquid Product from Hydrogenating Converter

	<u>Gelsenberg</u>		<u>Scholven</u>	
	Feb. '40	May '40	March '40	May '40
d_{15}	1.004	1.003	1.022	1.019
Solid material mg/l.	38	40	46	60
Engler distillation curve				
First drop:	68°C	73°C	69°C	67°C
Up to 155° C	3.8% (wt.)	4.2% (wt.)	4.3% (wt.)	4.2% (wt.)
170°	5.4	5.3	5.4	5.6
210°	10.7	11.6	12.4	11.5
250°	21.7	20.6	24.0	22.4
275°	29.4	28.0	29.6	27.8
300°	36.9	37.4	39.2	36.5
325°	50.7	48.7	49.6	48.6
345°	63.0	61.5	61.8	59.6
Residue at 345°	36.6	38.2	37.6	40.1
Loss	0.4	0.3	0.6	0.3
d_{15} -325° C:	0.947	0.946	0.948	0.946

Table 4

A-Middle Oil

	<u>Gelsenberg</u> <u>May '40</u>	<u>Scholven</u> <u>May '40</u>
d_{15}	0.957	0.955
Aniline point of phenol-containing prod.	-21° C	-19° C
Aniline point of dephenolized product	-19° C	-19° C
Phenol	131 g/l	130 g/l
Sulfur	0.03%	0.07%
Unsaturated hydrocarbons	26.0%	24.0%
Distillation Curve:		
First drop:	136° C	140° C
Up to 155° C	3% Vol.	4% Vol.
175°	7%	9%
185°	11%	15%
195°	16%	20%
225°	32%	40%
250°	48%	56%
275°	63%	71%
300°	78%	88%
325°	93%	97%
	340°/98%	332° C/98%
Residue at:	340° 1%	332° 1%
Loss	1%	1%
Ultimate Analysis:		
% C	85.82%	86.73%
% H	9.57	9.51
% N	0.70	0.35
% S	0.03	0.07
% Cl	0.004	0.004

Table 5

A-Bensine

	<u>Gelsenberg Product</u> <u>Refined and Distilled</u>			<u>Scholven Product</u> <u>Refined and Distilled</u>	
	<u>Feb. '40</u>	<u>From A-Product</u> <u>To 155°</u>	<u>May '40</u>	<u>Mar. '40</u>	<u>May '40</u>
d_{15}	0.7322	0.7526	0.7226	0.7480	0.7351
Cu+Al strip	very good	very good	very good	very good	very good
Aniline pt.	+52°	+35°	+41.5°	+34.5°	+38°
Doctor test	neg.	neg.	neg.	neg.	neg.
Acid content in distillation residue	0.0	0.0	0.0	0.0	0.0
Vapor pressure 37.6°C.	0.67	0.26	0.65	0.40	0.56
Octane No.	68.6	64.7	70.2	65.1	70.4
Glass dish test mg/100 cc.	0.0	0.0	0.0	0.8	0.0
ASTM Distillation Curve:					
First Drop	33°	69°	41°	52°	45°
Up to 40°C	2% Vol.	-	-	-	-
50° C	-	-	3% Vol.	-	1.5% Vol.
60° C	11%	-	11%	5%	5%
70° C	22%	-	27%	11%	14%
80° C	34%	7.5%	55%	20%	32%
90° C	64%	25.0%	84%	39%	64%
100° C	85%	47.0%	95%	60%	88%
110° C	94%	67.0%	105°/97%	74%	94%
120°	118°/97%	80.0%	-	84%	112°/95%
130°	-	85.0%	-	91%	-
140°	-	93.0%	-	96%	-
150°	-	95.5%	-	98.5%	-
		155°/96.5%			
Olefins	-	-	10%	6%	7%
Aromatics	-	-	5%	12%	12%
Naphthenes	-	-	57%	53.3%	58.1%
Paraffins	-	-	28%	28.7%	22.9%
<u>Ultimate Analysis</u>					
% C	84.77	-	85.13	85.56	85.73
% H	14.15	-	14.09	13.67	13.76
% N	0.62	-	0.11	0.41	0.16
% S	0.004	-	0.02	0.003	0.02
% Cl	0.05	-	0.005	0.03	0.008
Pyridine Bases	0.056	-	0.025	0.007	0.009

Table 6
 Injected Material for 5058-Chamber

	<u>Gelsenberg Product</u>		<u>Scholven Product</u>	
	<u>Feb. '40</u>	<u>May '40</u>	<u>Mar. '40</u>	<u>May '40</u>
d_{15}	0.931	0.943	0.967	0.960
Aniline point of phenol-containing product	-	-18° C	-	-20° C
Aniline point of dephenolized product		-18° C		-20° C
Phenol g/l	121	107	157	141
<u>Distillation Curve:</u>				
First drop	62°	64°	163°	162°
Up to 75° C	2% Vol.	2% Vol.		
100°	5%	4%		
125°	10%	7%		
155°	15%	11%		
170°	18%	15%	0.5% Vol.	2.5% Vol.
175°	20%	17%	1.0%	3.0%
185°	23%	19%	2.0%	5.0%
195°	25%	22%	6.0%	9.0%
225°	40%	38%	34.0%	33.0%
250°	55%	53%	49.0%	50.0%
275°	70%	66%	67.0%	67.0%
300°	85%	80%	86.0%	86.0%
310°	92%	86%	91.0%	91.0%
320°	96%	92%	95.0%	94.0%
325°	97%	94%	96.0%	96.0%
	334°/99%	342°/98.5%	339°/98.0%	339°/98.5%
<u>Ultimate Analysis</u>				
% C	86.41	86.78	86.93	87.40
% H	10.42	10.03	9.30	9.75
% N	0.93	0.72	0.84	0.58
% S	0.41	0.32	0.12	0.09
% Cl	0.02	0.005	0.007	0.007

Table 7

Catch-Pot Product from 5058-Chamber

	Gelsenberg		Scholven	
	Feb. '40	May '40	March '40	May '40
Specific gravity at 15°	0.820	0.810	0.811	0.815
Aniline point	+41°	+40°	+46°	+42.5°
Phenol - g/l	0.50	0.76	0.41	0.63
<u>Column Distillation:</u>				
Calorgas	1.4% wt.	1.8% wt.	2.7% wt.	1.8% wt.
Bensine - 155°	36.5% wt.	35.4% wt.	37.0% wt.	34.8% wt.
Sp. gr. at 15°	0.760	0.756	0.763	0.715
Aniline point	39°	38°	39.5°	37.8°
Phenol g/l	0.63	1.12	0.50	0.95
Middle oil	61.6% wt.	62.4% wt.	60.0% wt.	63.0% wt.
Sp. gr. at 15°	0.863	0.863	0.846	0.852
Aniline point	44°	43°	51°	48°
Phenol g/l	0.45	0.63	0.36	0.68
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Engler Distillation Curve (% by volume)				
First drop	56°	44°	57°	42°
Up to 50° C				1.0%
75°	3%	3.5%	3%	3%
100°	10%	11%	10%	9%
125°	22%	23%	22%	19%
155°	36%	38%	37%	36%
175°	45%	47%	46%	46%
195°	54%	56%	62%	57%
225°	67%	73%	77%	75%
250°	80%	81%	87%	86%
275°	90%	89%	95%	94%
300°	96%	95%	285°/96%	289°/97.5%
	307°/97%	304°/96%		
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Benzine - 155°				
Olefins	2.0%	3.0%	1.5%	2.0%
Aromatics	11.0%	9.0%	10.5%	12.0%
Naphthenes	59.4%	54.2%	57.6%	57.2%
Paraffins	27.6%	33.8%	30.8%	28.8%
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Ultimate Analysis:				
% C	85.17	86.24	86.08	86.54
% H	12.89	13.36	13.74	13.25
% N	0.51	0.25	0.0	0.03
% S	0.02	0.0	0.02	0.0
% Cl	0.003	0.007	0.003	0.004

Table 8

Injected material for 6434-Chamber

	<u>Gelsenberg Product</u>		<u>Scholven Product</u>	
	<u>Feb. '40</u>	<u>May '40</u>	<u>March '40</u>	<u>May '40</u>
d_{15}	0.858	0.852	0.838	0.849
Aniline point	+43°	+44.5°	+51°	+47°
Phenol g/l	0.32	0.45	0.50	0.50
Distillation curve:				
First drop	160°	153°	157°	158°
Up to 170°C	1.5% Vol.	5.5% Vol.	5% Vol.	3.5% Vol.
175°	3.0%	9.0%	15%	5%
185°	10%	23%	23%	16%
195°	23%	41%	40%	30%
225°	62%	70%	73%	68%
250°	78%	82%	95%	86%
275°	90%	92%	97%	95%
	288°/95%	297°/98.5%	276°/98%	292°/98.5%

Ultimate Analysis:

% C	86.29	86.33	85.51	85.55
% H	12.67	12.84	13.28	13.03
% N	0.49	0.28	0.48	0.49
% S	0.45	0.34	0.43	0.24
% Cl	0.004	0.005	0.003	0.005

Table 9

Catch-Pot Product from 6L3L Chamber

	Gelsenberg Product		Scholven Product	
	Feb. '40	May '40	Mar. '40	May '40
Specific gravity at 15°	0.760	0.750	0.736	0.755
Aniline point	+48°	+47°	+53°	+49°
Phenol g/l	0.023	0.0	0.0	0.0
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<u>Column Distillation</u>				
Calorgas	12.5% Wt.	9.6% Wt.	10.9% Wt.	7.5% Wt.
Benzine - 155°	44.1% Wt.	56.6% Wt.	49.0% Wt.	51.0% Wt.
Sp. gr. 15°	0.732	0.722	0.720	0.732
Aniline point	+47°	+49°	+53°	+49°
Middle oil	42.8% Vol.	33.4% Vol.	40.0% Vol.	41.4% Vol.
Sp. gr.	0.839	0.831	0.816	0.830
Aniline point	+48°	+46°	+54.5°	
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<u>Engler Distillation Curve (% by volume)</u>				
First drop	31°	33°	32°	34°
Up to 40° C	2.5%	1.0%	1.5%	1.0%
50°	5%	4%	4%	3%
75°	17%	15%	12%	13%
100°	26%	28%	28%	29%
125°	37%	41%	40%	40%
155°	48%	55%	54%	55%
175°	57%	67%	64%	65%
195°	67%	76%	74%	76%
225°	79%	83%	82%	86%
250°	84%	86%	85%	89%
	278°/87.5%	255°/86.5%	264°/86%	253°/90%
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<u>Benzine - 155°</u>				
Olefins	3.0%	2.0%	2.0%	2.0%
Aromatics	8.0%	6.0%	6.0%	7.0%
Naphthenes	38.5%	44.4%	45.3%	47.0%
Paraffins	50.5%	47.6%	46.7%	44.0%
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<u>Ultimate Analysis</u>				
% C	85.44	85.49	84.88	85.08
% H	14.20	14.08	14.91	14.52
% N	0.30	0.17	0.0	0.05
% S	0.03	0.01	0.05	0.03
% Cl	0.002	0.005	0.004	0.003

Table 10

Benzine -- Finished Product

	Gelsenberg Benzine		Scholven Benzine	
	Apr. '40	May '40	Apr. '40	May '40
d_{15}	0.7370	0.7380	0.7342	0.7418
Octane Number without Pb	71.6	72.6	71.2	72.3
Octane Number with 0.9 Pb	-	87.7	-	87.0
Cu-Al strip test	very good	very good	very good	very good
Oxidation test:				
without Pb mg/100 cc	-	0.0	-	1.0
with 0.9 Pb "	-	1.2	-	1.8
Initial distillation	60°	60°	60°	60°
Vapor pressure at 37.8° C	-	0.40	-	0.45
Aniline point	+44.5°	+45°	+46.5°	+43.5°
Iodine number	-	2.6	-	4.5
Doctor test	-	positive	positive	positive
% Mercaptans	-	0.0011	-	0.0014
Refractive index	-	1.4090	-	1.4106
Light test				
After 7 min.	-	1	-	3
" 15 "	-	1	-	5
" 56 "	-	1	-	7
" 120 "	-	1	-	7
<u>ASTM Distillation curve:</u>				
First drop	53°	48°	45°	45°
10%	66°	64°	62°	64°
20%	72°	73°	71°	74°
30%	80°	79°	79°	83°
40%	87°	86°	86°	91°
50%	94°	94°	94°	98°
60%	102°	102°	101°	104°
70%	110°	108°	110°	113°
80%	120°	119°	121°	122°
90%	135°	135°	137°	137°
95%	148°	145°	149°	149°
End point	152°	151°	155°	156°
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Olefins	2%	1.5%	3.0%	2.0%
Aromatics	9%	7.5%	7.0%	9.0%
Naphthenes	41.5%	46.9%	48.0%	51.9%
Paraffins	47.5%	44.1%	42.0%	37.1%
Ultimate Analysis:				
% C		84.86		84.59
% H		14.75		15.02
% N		0.13		0.09
% S		0.002		0.002
% Cl		0.006		0.005