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Analysis of paraffins and benzines produced at the pressure pilot plant.

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Paraffins produced in Reactor IV on April 21/22, 1939, in Reactor VIII on June 23/24, 1939, and IIIa on July 17/18, 1939, have been analyzed. The primary benzine produced in the reactors VIII and III has been likewise analyzed. Table I records the operational data of the three reactors. Reactor III operated by the normal method at 7 atm. and has been examined, because it operated with complete working up. In Reactor VIII the gas flows from the bottom upwards; Reactor IIIa has been operated with water gas at single pass.

Table 2-4 record the analytical data of the paraffins. The survey that follows gives the most important results.

	Reactor IV	Reactor VIII	Reactor IIIa
Paraffin output (above 320°)	30.7%	18.7%	40.6%
Paraffin output (320-460)	19.2%	17.6%	19.7%
Paraffin output (320-460) on the basis of total paraffin of more than 320°.	62%	92%	48.5%

The values obtained at Reactor IV and Reactor VIII agree with prior results, as was to be expected. That means, about 2/3 of the paraffin obtained in normal operation may be used for the fatty acid synthesis. If we operate by a bottom-to-top method, practically all of the paraffin is in the fatty-acid boiling range; but the yield of paraffin obtained is so much lower, that practically the same percentage (on the basis of liquid products) can be oxidized. The result obtained in the water-gas Reactor IIIa is somewhat divergent; for, only about one half of the total paraffin output can be used for the fatty-acid synthesis. Substantially higher percentages of high-molecular paraffin have been here evolved. With Reactors IV and IIIa the pour points lie within the previously found range. But the data for Reactor VIII were divergent and we could not yet find a satisfactory explanation for them, since the solidification points of the Reactor VIII products had been quite normal in the analysis made in January 39. It could not be determined whether we are dealing with errors in analysis. If such an experiment with the bottom-to-top mode of operation should be repeated, it would be suitable to carry out another careful analysis of the products. It is particularly noteworthy that the solidification points of products of the water gas reactor IIIa are the usual ones; for, their iodine numbers are very high, in contrast to those of other paraffins. On the basis of the iodine values we may calculate that they contain about 10-13 mol % of olefins as compared to 2% found in the normal paraffins produced in the pressure synthesis. This result means that whenever deviations from the normal solidification points do occur, there must be differences in constitution.

Tables 5 and 6 record the analytical data of the benzine-boiling range products obtained in Reactors VIII and IIIa; the corresponding primary products have also been recorded. Figure 1 shows the octane number as a function of the characteristic boiling-point number.

With Reactor VIII, the primary octane number for the total benzines is the normal 30, for a 30% olefin content. But with Reactor IIIa the octane number is extraordinarily low (ca. 39) for a benzine with 41% of olefins (characteristic boiling number: 115). But we may assume that the octane number will go up after a chemical treatment. On the basis of prior experiments, we may assume it to be about:

with Reactor IIIa for a benzine with characteristic boiling number of 115	60-63
" " VIII " " " " " " " " "	40-45

Table 1

	Reactor IV	Reactor VIII	Reactor IIIa
Date of Sampling	April 21/22, 39 (balance April 20-23) 0.8-1.5 mm * Eirich granule kg-Th	June 23/24, 39 (balance June 22-25) 1-2 mm kg The catalyst on purified kieselguhr	July 17/18, 39 2-3 mm kg Th catalyst on purified kieselguhr
Composition of gas	Synthesis gas	Synthesis gas	Synthesis gas
CO:H2	1:1.91	1:1.92	1:1.25
Mode of Operation	from top to bottom	from bottom to top	from top to bottom
Hrs. of "	780	1310	476
Temperature			
atm. gauge press.	13.0	11.0	10.65
°C	194.1	197.4	185.7
Pressure	7.0 atm. g.p.	7.25	7.45
Charge:			
m^3/hr.	34.0	79.0	98
m^3/kg Co/hr.	0.98	1.23	1.16
m^3/nom. vol./hr.	1.12	0.98	0.88
CO-conversion (utilizable)	90.6%	46%	78.3%
Rate of liquefication			
Analysis	81%	87%	87%
Product	81%	53%	86.5%
Output			
g/m^3 Sy-gas	124.5	41.5	90.3
g/m^3 I-gas	146.0	51.5	124.5
g/m^3 effective gas ([illegible])	144.0	50.8	104
Composition of the products			
AK benzine	12.5	33.1	14.1
condensate oil	35.2	44.1	31.1
Paraffin	52.3	22.8	54.8

Table 2

Reactor IV April 21/22, 1939
on basis of more than 320° on basis of total reaction

320-340°	12.45%	3.82%	
360°	22.22%	6.80%	Pour Point
380°	29.88%	9.15%	+31.5% (sic!)
400	40.30%	12.35%	[illegible]
420°	47.04%	14.68%	+44.5%
440	56.48%	17.57%	+50.0%
460°	61.68%	19.16%	+55.0%
480°	68.99%	21.40%	+59.8%
500°	72.01%	22.32%	+64.5%
520°	77.21%	23.92%	+68.5%
540°	80.73%	24.99%	+72.3%
560°	83.80%	25.93%	+79.5%
580°	84.74%	26.23%	+83.0%
600°	87.80%	27.15%	+86.0%
620°	89.36%	27.63%	+89.0%

Residue 3.32%
Loss 0.53%

Table 3

For Reactor VIII June 23/24, 1939
on basis of more than 320° on basis of total production

320-340°	13.60% by weight	[illegible] by weight	[illegible]
380°	59.19% by weight	11.14 "	23.8%
400°	73.39% by weight	14.?7 "	30.2%
420°	83.79% by weight	16.01 "	36.0%
440°	92.39% by weight	17.20 "	40.0%
460°	94.19 by weight	17.42 "	45.0%
480°	95.19% by weight	17.[illegible] "	51.2%
500°	97.15% by weight	18.[illegible] "	[illegible]
Residue	2.85% by weight	0.5[illegible] "	[illegible]
Loss	%		

Table 4

For Reactor IIIa July 17/18, 1939
on basis of more than 320° On basis of total production [illegible] pour points [illegible] thermometer

320-340°	6.22% by wt.	2.53% by weight	14.3	
360°	12.96 "	5.27 "		26.2%
380°	21.21 "	8.63 "	6.1	32.8%
400°	28.83 "	11.73 "		39.0%
420°	36.96 "	14.63 "	5.1	45.2%
440°	43.26 "	17.60 "		50.0%
460°	48.40 "	19.69 "	5.3	54.0%
480°	53.50 "	21.77 "		58.9%
500°	60.21 "	24.50 "	6.0	62.8%
520°	63.77 "	25.95 "		68.0%
540°	69.32 "	28.20 "	5.2	[illegible]
560°	73.04 "	29.73 "		75.2%
580°	76.68 "	31.20 "	4.6	79.6%
600°	[illegible] "	[illegible] "		[illegible]
620°	88.32 "	35.91 "	6.8	87.4%
Residue:	11.68 "	4.74		

Table 5

Reactor VIII [illegible]

Condensate + A.K. Fraction [illegible] by weight
Products - 200°C

Condensate = 16.68 kg = 38.3% by vol.
Benzine = [illegible] kg = [illegible] by vol.
41.32 kg = 54.2% " of total yields

Fraction - 120°C = 53% by vol., 52.0% by weight of the total benzine
28.4% by weight of the total yields
Fraction - 160°C = 69.7% by vol., 68.3% by weight of the total benzine

37.3% by weight of the total yields

	6-23/24-39 Condensate Reactor VIII	6-24-39 A.K. Benzine Reactor VIII	9-23/24 Condensate + Benzine Reactor VIII Fraction -120°	6-23/24 Condensate + Benzine Reactor VIII Fraction -160°	6-23/24 Condensate + Benzine Reactor VIII Fraction -200°
Start	114	33	39	34	36
-40°		2.0	Traces	1.0	0.5
-60°		23.5	21.5	14.0	13.0
-80°		[illegible]	[illegible]	[illegible]	[illegible]
-100°		65.0	79.5	56.0	39.0
-120°	Traces	[illegible]	[illegible]	[illegible]	52.0
-140°	1.5%	91.0	96.0	90.5	62.0
-160°	5.5%	94.5		96.0	79.0
-180°	[illegible]				89.5
-200°	28.0%				95.0
-220°	40.0%				
-240°	53.0%				
-260°	60.0%				

-280°	69.5%				
-300°	80.0%				
-320°	88.0%				
-340°	93.5%				
Termination of boiling	343/96.0	164/95	140.96	170.98	204/95.5
Last Runnings	1.0	0.5	0.5	-	0.5
Residue	2.5	1.5			
Dist. Loss	0.5	3.0			
Charact. No.	242.9	90.2			
Spec. Wt.	0.759/15°	0.672/15°	0.670/15°	0.650/15°	0.694/15°
Olefine		29.0%	30%	26.5%	25.0%
Aniline Point		[illegible]	[illegible]	[illegible]	[illegible]
Vapor pr Press.		0.90 kg/cm2	0.72 kg	0.74 kg/cm2	0.74 kg/cm2
Vapor pressure of stab. A.H		0.78 kg/cm2			
Octane No. of Stab. Benz.		54.6			
Vapor Press. Original		0.82 kg/cm2			
O. No.		52.2	47.8	C.P.R.40	I.G.30

Table 6

Reactor IIIa [illegible]

Condensate -200°C = 59.9% by vol. 57.8% by weight

Products -200°C: A.H. Benzine 3.59 kg.
, Condensate 40.40 kg.
71.99 kg = 32.1% by weight of total yield

The following fractions of the products -200° have been prepared.

Fraction -125°C = 47.0% by vol., 45.3% by weight of the total benzine
14.85% by weight of the total output

Fraction - 150°C = 64.2% by vol. 62.8% by weight of the total benzine
20.2% by weight of the total output

Fraction - 175 = 82.2% by vol., 81.2% by weight of the total benzine
26.1% by weight of the total output

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Table I (Continued)

Reactor III – [illegible] 1939

[illegible]	[illegible] Oil	Total Synthetic Reactor III	A.K. Benzine Stable	Primary Benzine Fraction – 125°
[illegible]	[illegible]	[illegible]	30	39
[illegible]			Traces	
[illegible]		[illegible]	23.5	16.5
[illegible]	Traces	[illegible]	[illegible]	[illegible]
[illegible]	1.0	10.0	77.0	73.5
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	17.0	19.0	[illegible]	
[illegible]	29.0	21.5	97.0	
[illegible]	44.0	28.0		
[illegible]	57.0	33.0		
[illegible]	[illegible]	[illegible]		
[illegible]	[illegible]	[illegible]		
[illegible]	33.0	46.0		
[illegible]	38.0	50.0		
[illegible]	[illegible]	54.0		
[illegible]		[illegible]		
[illegible]		62.0		
[illegible]		66.5		
[illegible]	320/96.5	300/66.5	160/9[illegible].0	132/9[illegible].5
[illegible]	[illegible]	[illegible]	0.5	1.5
[illegible]	[illegible]	1.0	2.0	2.0
[illegible]		31.0	1.5	2.0
[illegible]	[illegible]/15°	0.875/15° weighed in the 100 measuring cylinder	0.677/15°	0.676/15°
[illegible]			45.0	45.0
[illegible] Point			52.0°	49.0°
Vapor Pressure			0.75 kg/cm²	0.70 kg/cm²
[illegible] No.			54 C.F.R.	53.6 C.F.R.
[illegible] Pressure, Orig.			0.99 kg	

[illegible]	Primary Benzine Fraction – 150°	Primary Benzine Fraction – 175°	Primary Benzine Fraction – 200°
Start	32	36	34
[illegible]	2.0	1.0	1.5
[illegible]	[illegible]	[illegible]	13.0
[illegible]	32.0	25.5	26.0
[illegible]	55.0	40.0	39.5
[illegible]	[illegible]	57.0	52.5
[illegible]	98.0	73.5	65.0
[illegible]		87.0	77.5
[illegible]			87.5

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[illegible] 93.5

[illegible] 175/94.5 [illegible]

[illegible] between [illegible]

[illegible] have [illegible]

[illegible]

With the experimental reactors we are obtaining a somewhat higher yield of [illegible] to be used for oxidizing purposes than in the large-scale reactors. But the overall values of 65-70% are also obtained in the large scale plant.

The total output of paraffin is rather high in the sample in question, more than 30%. However this figure fails to agree with previous results. Therefore it must be subjected to more detailed examinations.

In general, those paraffins, where the portion belonging to the boiling range up to 460° is highest, will have the termination of the boiling range at the lowest temperature, e.g. reactor VIII [illegible] the reactors III and IV contain ca. 10% of fractions boiling at more than 500°. [illegible] boiling at ca. 500° and the range ends at 520-540°. Hard paraffin contains [illegible] In [illegible], only at 460° [illegible] about 45% will go over, at a temperature of up to [illegible] ca. 70% [illegible]

In Figure 2 (x) the [illegible] have been entered, along with the solidification points of the individual distillates. The agreement between the values obtained for the solidification points and those of the melting points of the corresponding paraffins, is in some cases very satisfactory, e.g. with table paraffin and with Reactor [illegible] are [illegible] curve, in particular, in the lower ranges. These discrepancies cannot be due to [illegible] boiling analyses and for the solidification points when control analyses were [illegible] lower the melting points. Regarding Reactor II it is also possible that the olefin content of the fractions plays a role [illegible] the melting [illegible]

On the basis of the boiling behavior of the paraffin hydrocarbons, the following C-number ranges are suitable for table paraffin, hard paraffin and paraffin batch for producing fatty acids.

1. Paraffin batch for fatty acids 320-460°
   = C_{19}-C_{30}, incl. solidification-point range: 23-66.

2. Table paraffins 340-530, C_{20}-C_{39}
   solidification-point range: 35-79

3. Hard paraffins more than 530 and above C_{28}, solidification-point range starts at ca. 66°.

(x) Cannot be traced. M.H.

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Analysis of Paraffin Contained in Pressure Synthesis Products

(Experimental Plant)

Table 1

	Reactor II	Reactor III	Reactor IV	Reactor VIII
Date of Sample	Dec. 7/15, 38	Jan. 10/21, 39	Jan. 6/7, 39	Jan. 2/5, 39
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
Composition of gas	water gas	synthesis gas 1:2	synthesis gas 1:2	synthesis gas 1:2
[illegible]	[illegible]			
Mode of Operation	Top to bottom	Top to bottom	Top to bottom	Bottom to top
Hours of Operation	[illegible]	[illegible]	924	2144
[illegible]				
Liq. Benzine	68.6% by wt.	18.0% by wt.	11.4% by wt.	33.7% by wt.
Oil Condensate	28.8 "	42.[illegible] "	35.[illegible] "	[illegible] "
Paraffin	2.6 "	39.9 "	52.9 "	31.7 "
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible] of paraffin				
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]				
in 320-460° range				
paraffin of [illegible]				
[illegible]		[illegible]	[illegible]	[illegible]

Table [illegible] Paraffin, Charges 111 and 113, and Hard Paraffin "V"

	Table Paraffin Charges 111 and 113	Hard Paraffin "V"
[illegible]	47.9°	96°
[illegible]	48.6°	98°
[illegible]	47.4°	85°
[illegible]	—	92°
Solidification point [illegible] thermometer	53.5°	93.0°
Solidification point [illegible]	54.5°	93-94° (residence not clear)
[illegible]	0.01	
[illegible]	0.08	

HEr:op

[illegible] 7, 19[illegible]

cc: All Divs.
AEM (25)