

Using 700 atm. in the Vapor Phase  
at Blechhammer Extension II  
(Discussion at Welheim, 29 January 1941)

By Dr. Walter Krönig, Schlesien-Benzin, 29 January 1941

Dr. Krönig pointed out that benzinization in Blechhammer I had been definitely set @ 300 atm, and that this could not be changed, but that the question of 300 or 700 atm in the vapor phase was still open for Blechhammer II. This question should be settled not later than April 1, 1941.

For extension II were available 120000 t liquid phase gasoline to 325° C, of which an estimated 48000 t to 200° C.

Dr. Winkler of Ruhröl declared that this quantity was about right for 2 double aromatization converters, though the high proportion of liquid phase gasoline in the injection was a disadvantage.

To this Dr. Krönig remarked that it appeared fundamentally possible to make an exchange between extensions I and II in such a way that extension II would supply liquid phase gasoline to extension I and extension I supply extension II with a corresponding quantity of middle oil. The limits for such an exchange are given by the following considerations:

- 1) In extension I the quantity of liquid phase gasoline must not increase so much and the quantity of middle oil drop so much that the two double 6434 stalls will be underloaded.
- 2) The shifting between gasoline and middle oil in extension I must not go so far as to reduced the anti-knock value of the gasoline from extension I to any extent.
- 3) The exchange must not go so far as to reduce the fraction of heavy gasoline available in extension I for DHD too much.

It may be assumed, without making detailed investigations, that the liquid phase gasoline fraction to 125° C from extension II, which amounts to about 60% of the liquid phase gasoline to 200° C, 29000 t/ann. more or less, can be exchanged for middle oil from extension I. From the standpoint of distillation also, cutting out the fraction to 125° C could be done. Dr. Krönig promised Dr. Winkler to notify him, if an exchange to such a degree were possible in I.G.'s opinion. Dr. Winkler would then discontinue his small experiments along this line. Dr. Winkler will also investigate one 10 Kg sample each of liquid phase gasoline and liquid phase middle oil obtained from running Upper Silesian coal to fuel oil at Ludwigshafen, particularly for their phenol content, since phenols in higher concentration, especially carboic acid, damage the catalyst, in order to establish a basis for his opinions. On the basis of Dr. Krönig's data and the results of the then discontinued experiments, Dr. Winkler will prepare a flow sheet for extension II at Schlesien-Benzin, which will include all important details for such a decision.

Dr. Winkler submits the following data on VT-706-b, end point 165° C, from pitch liquid phase middle oil @ 700 atm and stabilized in Scholven. (The gasoline is refined with 0.5% of 96% H<sub>2</sub>SO<sub>4</sub> and then washed with a 10% solution of caustic soda. It is then redistilled to 3% residue, which is returned to the vapor phase):

Sp. Grav./15°C		0.800
Turbidity point		-4°C
Beginning of dry crystallization		-60°C
Vapor pressure		0.40 atm
Initial boil		42°C
	10%	75.5°
	20%	83.5°
	30%	91.0°
	40%	97.0°
	50%	103°
	60%	113°
	70%	125°
	80%	137°
	90%	153.5°
	95%	164.5°
Boiling and 98%	@	170°
Residue		0.9%
Reaction of residue is neutral.		
100° point		45%/vol.
Kennziffer (Mean boiling point-?)		114.1
Aniline point		-4.5°
" " (dearomatized)		+47.5°
Corrosion test		negative
Doctor test		"
Glass dish test		0.02 mg
Bomb test		0.12%
Resin not absorbed		18.4 g/100 cc
" directly " (permissible)		8.6 mg/100 cc
	(limit is 10 mg)	
Lead sludge		12 mg/200 cc
Iodine number		2.4
Aromatics + Olefins		51%/vol.
Naphthene		37%/vol.
Paraffins		12%/vol.
Elementary Analysis:		
	C	88.38%
	H	11.61%
	S	0.0072%
Octane number , M.M. without lead		81
	with 0.05% "	87.5
	" 0.12% "	91.0

Dr. Winkler recommends that Schlesien-Benzin get in touch with Dr. Dehmlow of the ministry of aviation in Berlin regarding the evaluation of the Welheim gasoline, as well as CV<sub>2</sub>b or DHD gasoline. As far as Dr. Winkler knows, the CV<sub>2</sub>b from Scholven could not meet the specifications of the air force with respect to iodine number, resin test and light stability. DHD gasoline could not meet the lead sludge test. This group of high test fuels must basically have a minimum aromatics content of 50%. Welheim gasoline is

added to the 87 octane gasoline to the extent of 70%, so that the aromatics content of the mixture is 40%.

Welheim gasoline is produced in a stall containing 10.5 m<sup>3</sup> c atalyst. The stall consists of:

- 2 heat exchangers
  - 1 gas fired preheater, with 23 hairpin coils (fuel gas consumption = 4000 m<sup>3</sup>/h @ 5500 kcal).
  - 2 converters
    - 1 @ 900 mm Ø x 12 m long, and
    - 1 @1000 mm Ø x 15 m " .
- Both converters have standard insulation.

The converters have no trays or baffles. Nevertheless, the temperature distribution in the converter is excellent. With an injection <sup>x)</sup> of

9 t/h fresh oil : B-middle oil = 1:1 <sup>xx)</sup>  
 28000 m<sup>3</sup>/h inlet gas (72-74% H<sub>2</sub> = 450 atm H<sub>2</sub> partial pressure)  
 5000 m<sup>3</sup>/h cold gas

the converter temperatures were:

Converter I

Elements	1,	2,	3,	4,	5,	6.
Millivolt	25.1,	25.5,	25.7,	26.2,	26.2,	26.2.

Converter II

Elements	112,	111,	110,	109,	108,	107,	106,	105,	104,	103,	102,	101.
Millivolts	25.0	25.1	25.5,	25.8,	26.0,	26.2,	26.0,	26.2,	26.2,	26.0,	26.2,	26.0.

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<sup>x)</sup> Triplex beam pumps were formerly used. These were entirely unsatisfactory because they could not be satisfactorily regulated. At present, 5 m<sup>3</sup> paste presses are used, which, though more expensive, can be accurately regulated.

<sup>xx)</sup> The reflux is held around 1:1, if possible, because the converter operates most uniformly with a strong reflux, even when starting a new catalyst. This is also important because of the phenol content, which should not exceed 10% in the injection, since a higher content deteriorates the catalyst appreciably.  
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Controlling the temperature with a thruput (space time) of 0.4 offers no greater difficulties than a 5058 stall in operation for some time. Within 2 years of operation the stall temperature has risen beyond control only 3 times, in each case for mechanical reasons not connected with the process. With an injection of 10.5 t/h the inlet gas is 28000 m<sup>3</sup>/h. t he cold gas 6000m<sup>3</sup>/h. The cold catch pot product concentration is 50-55%/vol. (Abstreiferkonzentration). In the benzination of liquid phase gasoline + liquid phase middle oil from the pitch stall the high pressure losses are 12.4%, the losses in stabilization and transportation to Scholven 3.1-5.1%. Dr. Winkler will make up a C-balance, from which the accurate composition of gasification, including stable gas, can also be determined. In previous experiments the composition of the gasified C, without stable gas, was as follows:

C <sub>1</sub>	C <sub>2</sub>	C <sub>3</sub>	n-C <sub>4</sub>	i-C <sub>5</sub>
16.2	22.1	31.8	21.1	8.8

The H<sub>2</sub> consumption is 700 IG-m<sup>3</sup>/t (@ 735 mm & 15° C) of unstabilized gasoline. There is no circulating gas wash. On the contrary, the aromatization stall acts as a gas wash for the liquid phase stall, since it is one the same gas circuit.

Dr. Winkler figures on a catalyst life of at least 1/2 year, possibly over 1 year. The longest catalyst life so far obtained in large scale production has been 180 days. A check of this catalyst in a small converter showed that it was still quite active. In any case, the deterioration of the catalyst is much less than with 7019 in Scholven. The catalyst is used in pills of 10 mm. It is practically free of government controlled metals. It would be supplied by Welheim. The cost of the catalyst was roughly estimated at about RM. 1500/m<sub>3</sub>. Its bulk weight (Schüttgewicht) is 0.65. Its strength is outstanding. The latest catalyst differs from that formerly used in that it produces gasolines rich in aromatics from the very start, while the former catalyst had to be run in for some time before it would produce highly aromatic gasoline.

To the question of using 700 atm vapor phase in peace time, Dr. Winkler declared that in peace time the gasoline would cut off at 185° C. He was convinced that a premium would also be paid for its use in automobiles, compared to 70 octane gasoline, that Welheim gasoline to 185° C possesses benzene-gasoline blend quality (Aralqualität) and is a desirable blend gasoline for Fischer gasoline as well. If gasoline rich in aromatics should really find no sale in peace time, there was always the possibility of producing H<sub>2</sub>-rich gasoline with a different catalyst at a temperature of 22 mV.

#### Summary.

The discussion brought out that the use of 700 atm in the vapor phase would appear to be very advantageous for Blechhammer II, assuming that:

- 1) an exchange of liquid phase gasoline and liquid phase middle oil between extensions I and II is possible without harming the requirements of extension I.
- 2) a high test gasoline able to meet air force specifications can also be obtained from Scholven middle oil @ 700 atm.
- 3) the government (Reichsamt) is satisfied with the reduced quantity of isobutane.