

The crude product is worked up to give 25% by weight of gasoline with an octane number of only 55-60 motor method, 50% of diesel oil with 50-55% cetane number, 10-15% refined wax and 10-15% lubricating oil which had a good viscosity index but a low absolute viscosity. The refined was obtained by this process is particularly suitable because of low sulphur content, for oxidation to carboxylic acids or for cracking to olefines for synthetic lubricating oil manufacture. Dr. Pier stated that latterly most of the wax product had gone to Stettin for lubricating oil manufacture.

Zeitz also operated a modified version of the above process, known as M.T.H. process. The conditions are substantially the same as for T.T.H., except that throughput is lower and the temperature higher (390°). The crude product consists of 35% gasoline, 60% diesel oil and 5% heavy bottoms which were probably sent to Bohlen for liquid phase hydrogenation. The hydrocarbon gas make is roughly 6% and the hydrogen absorption 650-700 m³ per ton of tar.

d) Bituminous Coal Tar Hydrogenation

Bituminous coal tar is hydrogenated at Pölitz and at Lutzkendorf. Bituminous coal tar pitch is used as raw material at Welheim. When treating crude tar, it is usually distilled to give middle oil which goes direct to the vapour phase stages and a light pitch which is hydrogenated at 700 ats. using the same catalyst conditions as are employed for hydrogenation of heavy fractions of brown coal-tar. The attached flowsheet, Fig. XIV, was prepared by Dr. Pier's staff and shows the yields, etc, which they feel should be obtainable under steady operating conditions in a plant working entirely on bituminous coal tar.

e) Petroleum Oil Residues.

Residues from the distillation of crude petroleum and heavy residue from cracking operations are hydrogenated in the liquid phase at Pölitz. A pressure of 700 ats is employed. At Lutzkendorf, asphaltic residues obtained from the lubricating oil plant are also hydrogenated at 700 ats. The catalyst is the normal liquid phase catalyst, iron on activated coke.

The following flowsheet, Fig. XV, is typical of operation on heavy oil residue obtained by straight distillation of German crude petroleum. The figures for gas make and gasoline yield are unlikely to vary greatly with different types of crude oil, but it should be borne in mind that the hydrogen absorption will vary appreciably according to the nature of the crude.

f) Normal 300 ats. Vapour Phase Hydrogenation

Vapour phase hydrogenation of the gasoline plus middle oil product obtained from the liquid phase operations described above or from straight distillation of tars or crude petroleum is usually carried out in two stages. The first stage consists of saturation hydrogenation, the main object of which is the removal of nitrogen compounds which reduce the reactivity of the oil in the second vapour phase splitting hydrogenation step. It is usual to fractionate the crude saturation product from the first stage into gasoline and middle oil, only the latter going forward to splitting hydrogenation. The main reason for this is to segregate the comparatively poor quality

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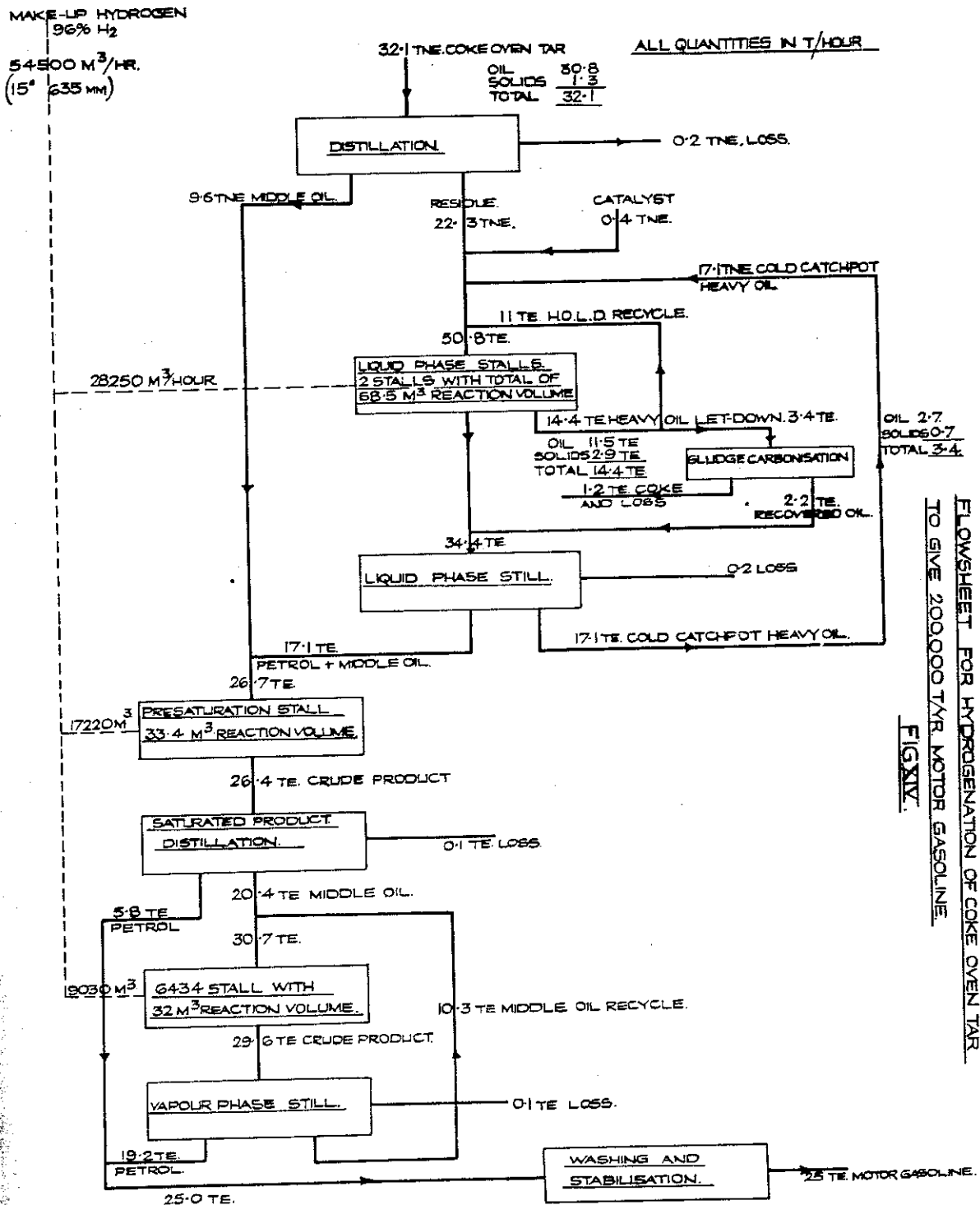
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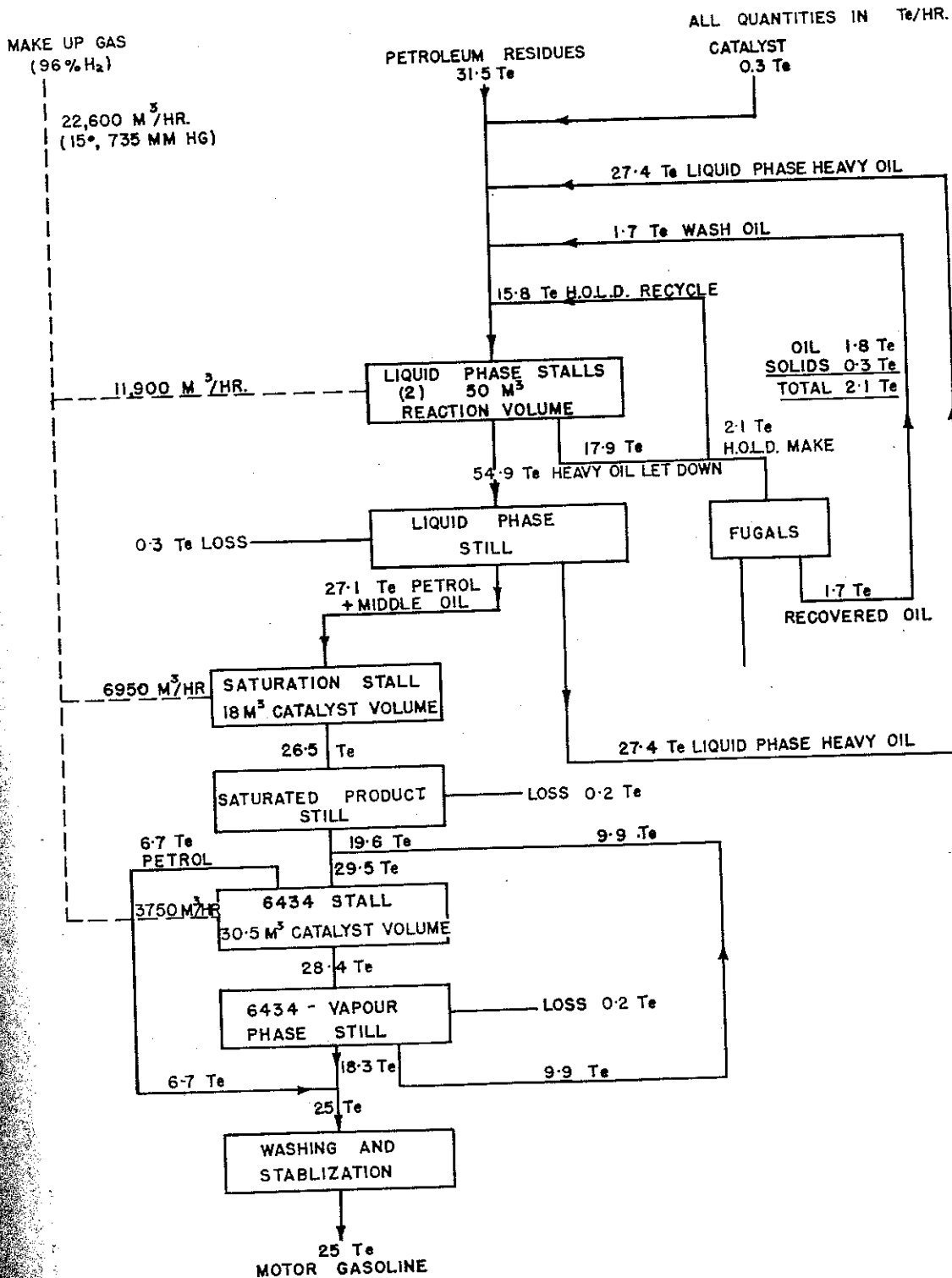
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FLWSHEET FOR THE HYDROGENATION OF PETROLEUM RESIDUES TO 200,000 T_e/YR. MOTOR GASOLINE

FIGURE XX

saturation stage gasoline from the main splitting hydrogenation product. The saturation stage gasoline is frequently used as feed for the D.H.D. process which is described later.

The saturation stage consists of once-through treatment at 300 ats pressure and temperature of 400-410°C over an active catalyst. Formerly, the catalyst used was tungsten sulphide (5058) but this has now been largely substituted by a new catalyst developed by Ludwigshafen which consists of 20-25% tungsten sulphide and 3% nickel sulphide supported on activated alumina, (8376). It is claimed that this catalyst has as good an activity for the purification of oils from nitrogen, oxygen, etc. as 5058 but it causes less splitting to lighter products. This is advantageous because it reduces the amount of comparatively poor quality petrol made in the saturation stage. Actually, the impression was gained that the main reason for employment of the new catalyst was the necessity to conserve tungsten and that the new catalyst was in certain respects, e.g. life, inferior to 5058. Dr. Pier also stated that Bohlen and Magdeburg had used 6434 (see below) as a saturation catalyst. Hydrocarbon gas make in the saturation stage is small; of the order of 1% on the oil treated.

As will be seen from the flowsheets in Figs. XII, XIV and XV, the throughput through the saturation stall, expressed in kgms. per litre of catalyst per hour, varies with the nature of the feed. The determining factor is the temperature control of the converter, which is achieved by arranging the catalyst in beds with intermediate introduction of cold hydrogen. Aromatic feed stocks requiring a higher hydrogen absorption give rise to a greater heat of reaction and have to be fed at a lower rate.

In the splitting hydrogenation stage, saturated middle oil is reacted over tungsten sulphide on activated Terrane catalyst (6434) at a temperature of 410-420°C and pressure of 300 ats. The pass converter to gasoline is of the order of 60%. Hydrocarbon gas make depends on the end-point and volatility of the petrol product. When making a motor gasoline of end-point about 180, it is about 12% by weight on the middle oil hydrogenated and this rises to 18-20% when aviation gasoline of 150 end-point or 65-70% volatility at 100°C is the required product.

No catalyst other than 6434 has been used for large scale splitting hydrogenation at 300 ats. Other catalysts have been tried in the laboratory, particularly an iron on activated Terrane catalyst which was suggested by A.N.I.C. (Italy). None of these alternative catalysts have been found to be superior to 6434.

g) 700 ats. Vapour Phase Hydrogenation

An interesting development at the Welheim and Lutzendorf plants has been the elimination of the saturation stage and the direct splitting hydrogenation of liquid phase gasoline and middle oil at 700 ats. In the case of Welheim, a new splitting catalyst is being used, probably consisting of 3% chrome oxide and 1% molybdic oxide supported on Florida earth. The I.G. Research Department at Ludwigshafen have been working on parallel lines and have found this to be the optimum catalyst. The reaction temperature is 450°C. i.e. some 30° higher than that employed in the normal

TABLE II

Plant	Main Raw Material(s)	Type of Hydrogenation Process	D.H.D. Plant installed or working	Is-octane or Alkylate Plant working or installed	Anl. Prodn. Cap. of Finished Fuels T/YR.
SCHOLVEN	Bituminous Coal	300 ats Liquid & Vapour Phase	2 units of 100,000 T/yr. Wkg.	60,000 T/yr. Alkylate. Pit Operating	200,000
GELSENBERG		700 ats Liquid Phase. 300 ats Vapour Phase	None	None	350,000
BLECHHAMMER			2 units in course of erection.	Plant in course of erection.	400,000
POLITZ	Bituminous Coal	700 ats L.Phase	4 units working.	Alkylate. Plant operating.	600,000
WESSELING	Bituminous Coal Tar	300 ats V.Phase	None	60-70,000 T.yr.	200,000
	Petroleum Residues			Alkylate. 20,000 T/yr. Plant in course of erection	600,000
LEUNA	Brown Coal	250 ats Liquid & Vapour Phase	4 units. 1 working.	Alkylate. Plant operating. 60,000 T/yr	150,000
	WELHEIM	Bituminous Coal Tar Fractions and Pitch	None	None	250-300,000
BOHLEN		250-300 ats L.&V.Phase	2 units not ready	Alkylate. Plant in course of erection	250-300,000
MAGDEBURG			None	None	250-300,000
ZEITZ		300 ats T.T.H. process	2 units not finished	Alkylate. Plant in course of erection	400,000
BRUX		Normal 300 ats L. & V. phase	2 units not finished.	None	50,000
LUTZKENDORF	Petroleum Residues	700 ats. Liquid	None	None	?
HEYDERECH	Bituminous Coal Tar Fractions			Is-octane plant partly working	?
Auschwitz	Higher Alcohols			Is-octane plant partly working	?

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TABLE III

COST CALCULATION FOR THE PRODUCTION OF 200,000 T/YEAR

Raw Material	Bituminous Coal	RM/te Motor Gasoline
Raw Materials	1.84 te.Bit.Coal (1) at 21.50 RM/te	39.50
Hydrogen (100%)	2800 cbm at 4.5 Pfg/m ³	126.00
Catalysts and Chemicals		5.00
<u>Credits:</u>		
<u>Liquefied Gases</u>	230 kg at 220.65 RM/te (3)	<u>50.80</u>
Fuel Gases (2)	3.4 x 10 ⁶ WE at 6 RM	<u>20.40</u>
Running Costs		121.35
Production Cost		220.65
Export Cost		3.00
General Costs and Licence	5% of Production Cost	11.05
Total Cost (Loaded)		234.70

(1) Bituminous Coal with 8% Water and 4% Ash.

(2) If Ethane is recovered the credit is increased by RM 2.-/te.

(3) Valued at the production cost of Motor Gasoline.

MOTOR GASOLINE BY THE HYDROGENATION OF VARIOUS RAW MATERIALS

Bituminous Coal High Temperature Tar		Natural Oil Residuuum.....	
	RM/te Motor Gasoline		RM/te Motor Gasoline
1.29 te.H.T.Tar at 40 RM/te	51.50	1.26 te.Residuuum at 40 RM/te	50.50
2090 cbm at 4.5 Pfg/m ³	94.90	870 cmb at 4.5 Pfg/m ³	39.20
	7.20		3.00
186 kg at 193 RM/te	35.80	143 kg at 148 RM/te	21.20
2.0 x 10 ⁶ WE at 6 RM	12.00	1.44 x 10 ⁶ WE at 6 RM	8.60
	87.20		85.10
	193.00		148.00
	3.00		3.00
	9.65		7.40
	205.65		158.40
			170.00
			3.00
			8.50
			181.50
			204.60

Residues at 80 RM/te give Total Cost of

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TABLE IV.
ANALYSIS OF RUNNING COSTS PER TONNE OF MOTOR GASOLINE

	Bituminous Coal		Total
	Liquid Phase	Vapour Phase	
Capital Cost in Mill. RM			
Hydrogenation Proper.	46	17	63
Associated Bldgs. 25%(1)			16
			79
<u>Running Costs</u>			
Wages, Man-days	525	220	745
Man-hrs/Te gasoline	7.75	3.2	10.95
		RM/Te Motor Spirit	
Wages at 1.30 RM/hr.	10.---	4.15	14.15(2)
Salaries 20% of Wages	2.---	0.85	2.85
<u>Energy Costs</u>			
Water at 0.01 RM/cbm	(110m ³) 1.10	(80m ³) 0.80	1.90
H.P. Steam at 3.-/Te	(0.43Te) 1.30	(0.2Te) 0.60	1.90
L.P. Steam at 2.25/Te	(1.64Te) 3.70	(0.59Te) 1.30	5.---
Electricity Energy			
at 0.015/KWH	(800KW) 12.---	(146KW) 2.10	14.10
Fuel Gas at 6.-/10 ⁶ WE	(2x10 ⁶ WE) 12.---	(0.7) 4.20	16.20
<u>Maintenance Costs</u>			
6% of the plant	13.90	5.10	19.---
2% of the associated buildings	1.15	0.45	1.60
<u>Working Materials</u>			
10% of wages	1.00	0.40	1.40
<u>Amortization</u>			
10% of the plant	23.00	8.50	31.50
5% of the associated buildings	2.85	1.05	3.90
<u>Taxes, Fire Protection, etc.</u>			
2% of the Capl. cost	5.75	2.10	7.85
	89.75	31.60	121.35

(1) The "Associated Buildings" include:- Main Offices, Main Workshop, Gas Protection, Laboratories, Change-rooms, Construction equipment, Garages, Railway sidings, Roads, Stores, Pipe Bridges, Drains, Culverts, etc.

In this cost are generally included the costs for:- initial design work, Construction management, Equipping of stores, Spare catalyst, Personnel training, Interest on capital during construction, Working capital, Land Costs, Railway connections and housing. These vary greatly according to the location of the plant and are not included.

(2) Because of the necessity of using untrained and unqualified staff the personnel requirement is 1/3 higher than necessary.

I.C. 7375

(Plant capacity: 200,000 Tes/year Motor Gasoline)
 Bituminous Coal High Temperature Tar Natural Oil Residues

Bituminous Coal High Temperature Tar			Natural Oil Residues		
Liquid Phase	V. Phase	Total	Liquid Phase	V. Phase	Total
31.5	17	48.5	30	17	47
		12			12
		60.5			59
285	220	505	260	220	480
4.2	3.2	7.4	3.8	3.2	7.0
RM/Te Motor Spirit			RM/Te Motor Spirit		
5.45	4.15	9.60	4.95	4.15	9.10
1.10	0.85	1.95	1.--	0.85	1.85
(80 m ³)	0.80	1.60	(80 m ³)	0.80	1.60
(0.33 Te)	1.00	1.60	(0.23 Te)	0.70	1.30
(0.76 Te)	1.70	3.--	(0.69 Te)	1.55	2.85
(400 KW)	6.--	8.10	(420 KW)	6.30	8.40
(1.2x10 ⁶ WE)	7.20	11.40	(1.2x10 ⁶ WE)	7.20	11.40
	9.40	14.50		9.--	14.10
	0.80	1.20		0.80	1.20
	0.55	0.95		0.50	0.90
	15.75	24.25		15.--	23.50
	1.95	3.--		1.95	3.--
	3.95	6.05		3.80	5.90
55.65	31.55	87.20	53.55	31.55	85.10

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