

CATALYST FACTORY

The following information was obtained mainly from Dr. Joswig on 11 May 1945.

The particular feature of this plant was that they claimed a very high recovery of cobalt in the catalyst regeneration. The departures from the normal process which led to this result were due to the restrictions on emitting effluent from the factory, which necessitated re-using water and evaporating the final effluent.

The spent catalyst, from which the wax had been extracted, was ground with wash water from the following stage. This wash water contained about 5 gm. per liter of Co. It was then extracted with boiling nitric acid (50%) in vessels of about 30 m³ capacity. It was then filtered on a filter press and washed. The first wash water was mixed with the filtrate. The second wash water was used for grinding the incoming spent catalyst, as mentioned above. The third wash water was used for the first wash of the next batch. It was claimed that the loss of Co in the filter pressing operation was less than 0.1%. Joswig claimed that they had achieved an overall loss in the factory of only 0.3% compared with 2% at Ruhland and 4-5% at Holten.

The Co solution was then treated with soda to precipitate Fe, Al and Th. The sludge obtained by filtration (Thoriumschlamm) was sent to Ruhland. Sodium fluoride was then added to remove calcium. To ensure complete removal of calcium, an excess of sodium fluoride was added which precipitated part of the magnesium. The liquor was then filtered using kieselguhr as a filter aid. The filter cake was washed twice, the first washings being added to the filtrate and the second washings being used for the first wash of the next batch. The Co concentration was finally 40-45 gm. per liter. This was sometimes strengthened with the fresh Co added as make-up. When fresh Co was used, it was dissolved in nitric acid and treated with a small amount of soda and then filtered before mixing with the recovered Co solution.

The Mg and Th were then added to solution. After precipitation, kieselguhr was added. The filtrate from the precipitated catalyst contained about 30 gm. per liter of sodium nitrate. This figure was achieved by reducing wash waters. The filtrate was then treated with soda and settled to recover any Co which had passed through the filters. After settling, the solution was evaporated in a triple effect evaporator to recover sodium nitrate and produce distilled water for the catalyst manufacture. In the second evaporation stage, some caustic soda was added to ensure complete precipitation of any residual Co which was filtered out before the third

stage. Joswig stated that an appreciable amount of Co may remain in solution as bicarbonate.

The catalyst sludge from the presses was mixed with water and with the dust screened out from the dried catalyst. The mixture was then filtered on a rotary vacuum filter. The filter cake was put through an extruder with 6 mm. holes and then passed to a Buttner turbo-drier. The dried catalyst was screened to give a product of 1-3 mm. The dust was mixed with the new precipitate, as mentioned above. The over-size was returned to the screens and gradually broken down to the required size.

The composition of the catalyst was given as:-

100	parts	Co
10	"	MgO
5	"	ThO ₂

The catalyst was made up to give 800-900 kg. Co per chamber, using an appropriate amount of kieselguhr depending on its density. From 1.7 to 2.3 tons of kieselguhr were used per ton of Co.

The capacity of the plant was rated at 100 catalyst chamber fillings per month, but the production had been only 60 owing to limited demand. The plant started up in September 1941 but only came into operation in April 1942. Lutzkendorf made catalyst for the adjoining F.-T. plant and also for the one at Odertal (Deschowitz).

The reason for producing unreduced catalyst at Lutzkendorf and reducing it at the F.-T. plants was not very clear. The division of responsibility between the two stages seems undesirable. One reason that was given for adopting this procedure was that the F.-T. plants had pure hydrogen available as they require it for the hydrogenation in the catalyst chambers. It is possible that freshly reduced catalyst may be better than reduced catalyst which has spent some time in storage or in transit. It may be conjectured that the procedure was at least partly dictated by military considerations. If it were desired to maintain a substantial storage of manufactured catalyst against possible destruction of the catalyst manufacture, storage of the unreduced catalyst would be a much simpler problem than storage in substantial quantity of the reduced catalyst.

HYDROGENATION PLANT

The following staff were seen on 11 May 1945 and 17 May 1945 in connection with the hydrogenation section:-

Dr. Schnesberger	-	Managing Director.
Dr. Wielland,	-	Hydrogenation Manager.

The following information was obtained from interrogation and also from the notes of members of the C.A.F. team.

GENERAL.

The hydrogenation plant was built to hydrogenate the residues from the lubricating oil plant in conjunction with Bituminous Coal Tar. The planned output was 50,000 Tnes/year, but the best achieved was 42,000 Tnes/year in 1944.

The plant consisted of one liquid phase stall and one vapour phase stall, both of which operated at 700 ats. The catalyst in the vapour phase stall was 6434.

The Bituminous Coal Tar came from Mährisch Ostrau and was distilled with the product from the liquid phase stall to cut into two fractions, the cutting point being 350° C. (330° C. ?). The lighter fraction was passed over the vapour phase catalyst under straight through conditions when making petrol and diesel oil and under recycle conditions when making petrol only.

HYDROGEN SUPPLY.

The hydrogen from the hydrogenation plant came from the Wintershall generators. The raw water-gas was passed through an 8 ats CO conversion plant and the CO₂ washed out at the same pressure. The main compressors therefore had a suction pressure of 8 ats and delivered to 700 ats direct. The CO removal plant worked at 200 ats. after the third stage of the compressors. Another stage sufficed to raise the pressure to 700 ats.

The gas fed to the hydrogenation plant had a hydrogen purity of only 91-92%, which is very poor by normal standards. They had only achieved this purity by the installation of the CO conversion plant, before which they had worked with a make-up gas of about 86% purity.

For 1943 over a period of ten months, they had records of the following costs:-

Gas production	681,319 RM including
	155,000 Rm for labour.
Gas boosting	24,306 RM.
H ₂ O removal	61,252 "
-- ---	6,023 "
Oil Scrubbing	26,143 "
Org. Sulph. Rem.	88,946 "
1/5 of Gen. Costs	19,000 "
	906,989 RM which amounts to 2.70
	pf/m ³ Raw Water gas.

This is the price of raw water gas as supplied both to the Hydrogenation and Fischer-Tropsch plants. For the hydrogenation plant, the gas was further purified and the cost build-up for this section for the month of October 1943 is:-

Raw Water-Gas at 2.70 pf/m ³	222,220 RM
CO conversion and CO ₂ rem.	75,480 "
Compression	50,000 "
CO removal	17,536 "
1/5th Gen. Costs	21,338 "
	386,582 RM which,

for 5,295,700 m³ of 92% hydrogen, is 7.30 of pf/m³.

CIRCULATION AND INJECTION PLANTS.

There were four circulators with an output of 70,000 m³/hr. and one of 100,000 m³/hr., all of them being electric driven.

They had two hot recycle pumps for the liquid phase stall of 10 m³/hr. capacity, with four injectors having the same capacity for the injection of feed to the stalls. They also had four catalyst injection pumps which were steam driven and had a capacity of 0.25 m³/hr.

LIQUID PHASE STALL.

This stall was originally built with two interchangers, two convertors, a hot catchpot, a gas-fired preheater, a cooler and a cold catchpot, with arrangements for the recycle of hot oil from the base of the hot catchpot to the inlet preheater.

There was also an emergency blow-down tank for the burden of the stall in case of emergency. Owing to poor make-up gas quality, bad operation by inexperienced staff and change of composition of the feed to the liquid phase stall from that from which the plant was designed, they had not achieved the rate and feed of reaction they had expected. They were therefore short of heating capacity in the preheater and could not maintain the temperatures in the convertors as easily as they would have liked. To overcome these running difficulties, they had installed a third interchanger, enlarged the fan capacity on the preheater, and had even applied a 1" thickness of lagging to the outside of the convertors in order to increase the heat input to the inlet reactants and to reduce the heat losses.

The feed to the stall was 8-12 m³/hr. of oil with 100-150 liters/hour of catalyst paste made up as a paste of 40% solids from I.G. catalyst 10927 and vacuum residue from the Natural Oil section of the works. The running temperature was 470° C. and the reaction volume 10 m³ (from dimensions) or 16 m³ (interrogation) - more probably the former. The hot recycle rate was 6-10 m³/hour. The catalyst was adjusted so that the H.O.L.D. from the hot catchpot was a clear colour and not darkened by the presence of Asphalts. The let-down from the hot catchpot in order to purge the solids entering with the catalyst paste was about twice the paste injection. The purge contained about 1.5-1.8% hard asphalts.

The H.O.L.D. was fuggalled to give a residue which they put to the tip and a relatively clear oil of 0.2% solids which they sent to the liquid phase still. There was no gas washing plant to remove the gases made during the hydrogenation.

In the product from the cold catchpot they got 50% heavy oil which was recycled back to the stall. The product was distilled with the imported tars and the heavy oil portion fed to the liquid phase stall.

VAPOUR PHASE STALL.

The stall consisted of 3 convertors with 2 interchangers and a gas-fired preheater, a cooler and a cold catchpot.

The catalyst was the I.G. 6434 and the reaction volume would be about 13 m. When running on straight through operation they made a cold catchpot product containing 40% petrol and the remainder diesel oil. The petrol had a 74 O.N. with an FBP of 180° C. and the diesel oil a Cetane No. of 40-45 with a setting point of -40° C.

The gases made in the hydrogenation section were not treated to remove the liquifiable gases and the lean gases were sent to the fuel gas system and the rich gas to the Wintershall generators.

Overall hydrogen requirements are estimated at about 1,300 m³/Tns of feed when making petrol and diesel oil.

HYDROGENATION RUNNING COSTS 1943.

Liquid Phase Injection & Circulation	3.85 RM/Tne of feed.
Vapour " " "	10.84 " "
Liquid Phase Stall	17.17 " of Cold Catchpot Product.
Vapour Phase Stall	7.87 RM/Tne of Cold Catchpot Product
Total Distillation	45.96 RM/Tne feed.

In 10 months of 1943 they made 29,523 tonnes of Vapour Phase Cold Catchpot product at a cost of 7,448,456 RM., i.e. 255 RM/Tne. This is the running cost only and does not include any amortization or interest, etc.

LABOUR REQUIREMENTS.

Average figures for the early part of 1944 show the following:

Gas Production	-	500
Fischer Synthesis	-	245
Hydrogenation	-	280
O ₂ Steam Elec.	-	50
Nat. Oil Section	-	200
Final Refining	-	110
Power Plant	-	270
Workshops	-	275
Construction etc.	-	200
Watchmen etc.	-	580

In addition to these, they had other workers whose exact location is not available from the field notes.

The total strength was made up of about 4,000 workers and 380 staff, but of the 4,000, about 1,000 were engaged on alteration and extension work, and not on production.

CAPITAL COSTS.

The split of capital costs were given as:-

Gas Plant	-	22,610,614
Fischer Plant	-	18,182,250
Hydrogenation	-	18,606,851
Lube Oil	-	9,488,452
Final Dist.	-	6,092,751
Auxiliary	-	5,033,343
Railways	-	2,955,458
Water Supply	-	7,000,994
Power Plant	-	18,297,489
General	-	9,138,176
		<u>117,405,389</u>

The engineer estimated that for every tonne of steel in the finished plant, they had had to spend about 1,660 RM.

LUBRICATING OIL

Specifications on gasolines and diesel oils manufactured at Lützkendorf.

The personnel interviewed at the plant on 11 May 1945 included Dr. Schneeberger, manager of the entire plant, Dr. Neumann, in charge of lubricating oil manufacture, and Dr. Billig, in charge of the chemical and engine-testing laboratory.

The plant is of conventional design and includes installations for atmospheric crude distillation, vacuum distillation of the atmospheric residue, propane deasphalting and deresining, phenol extraction and propane dewaxing, as well as conventional acid treating and clay contacting. The crude throughput was about 60,000 bbls/month.

A mixture of Hanover and Austrian crude was processed mostly, but more recently Austrian crude only was used. Typical data on two Austrian crudes are shown in Table I. The atmospheric distillation gave the following yields for overhead, three side-streams and bottoms, respectively:-

	Percent by volume of crude	Bbls/month
Gasoline.....	10.6	6,350
Kerosine.....	20.0	12,000
Spindle Oil.....	16.7	10,000
Light Neutral dist. (about 130 SSU/100° F.)....	3.0	1,800
Residue.....	43.6	26,200
Loss.....	6.1	3,650

Vacuum distillation of the atmospheric residue yielded the following distillates:-

Product	Percent by volume of crude	Bbls/month
Spindle Oil (60 SSU/100° F.).....	7.0	4,180
Heavy Neutral dist. (300 SSU/100° F. Min.)..	10.7	6,420
Residue.....	26.0	15,600

The gasoline was not used for automotive purposes, but was utilized for special purposes (precipitation naphtha, petroleum ether, etc.). Kerosine was caustic washed and used for illumination; part of the kerosine was blended with spindle oil from the atmospheric distillation and the blend was used as diesel fuel. The remainder of the spindle oil was blended with the corresponding fraction from the vacuum distillation. Average inspection data on the distillation products are given in Table 2.

Finished neutral oils were made by dewaxing and acid and clay treatment of the 60 vis spindle oil and the light and heavy neutrals. Treating losses are given as follows:-

Charge stock	Percent by weight paraffin removed	Percent by weight acid sludge removed
Spindle oil 60 Vis.	20	4-5
Light neutral distillate..	20	4-5
Heavy neutral distillate..	20-25	4-5

The inspection data on the finished neutral oils are given in Table 3. The heavy neutral distillate was also used in the manufacture of automotive and aviation oils and for this purpose was subjected to phenol extraction instead of acid treatment. The yield of automotive oil was 70-75% by weight of the distillate, whereas the yield of aviation oil was 55-60% by weight. Inspection data of the finished oils are given in Table 4. The vacuum residue was propane de-asphalted and de-resined (yield of resin and asphalt free Oil: 50-60% by weight of vacuum residue charged). For the manufacture of automotive bright stock, the propane-treated vacuum residue was treated with 150 vols. of phenol yielding about 41% by weight of raffinate (calculated on vacuum residue charged). The raffinate was propane dewaxed and clay treated with an ultimate yield of automotive bright stock of about 28% by weight of the vacuum residue. In case that aviation bright stock was required, the raffinate from the phenol extraction was freed from phenol and again phenol-treated (total volume of phenol used in both stages: 500) yielding a raffinate which represented about 28% of the vacuum residue. The final yield of aviation bright stock after propane de-waxing and clay-treating was approximately 18.5% by weight of the vacuum residues. The inspection data for the two grades of bright stock are given in Table 5.

Propane deasphalting was carried out in a two-stage unit comprising 4 horizontal contactors and precipitated asphalts and resins were removed separately. Both of these products were destructively hydrogenated. The yield of deasphalted oil averaged 50% on charge.

Phenol extraction was carried out in a Kellogg unit built in 1938. It has a conventional tower type unit with perforated trays and water injection was employed to improve the selectivity of the solvent.

When automotive bright stocks were processed, the normal solvent ratio was 150%, the tower top temperature 90° C. and the temperature gradient 10° C. Aviation bright stocks, on the other hand, are solvent extracted twice, first under the above conditions, and then re-extracted with 500% of phenol at 110-120° C.

Dewaxing was carried out in a Dorr rotary pressure filter using discontinuous cooling. The charge stock was cooled from 86° F. to -40° F. in one hour. The design filtration rate was 7.5 gals. per cubic-foot per hour, but actually a rate of 19 gals. per cubic-foot per hour was obtained.

The working pressure was 150-200 mm. Hg. gauge. The double dilution technique was not employed and there was no re-pulping of the wax. The oil content of the finished wax averages 20%

LUBRICATING OIL SPECIFICATIONS:

Motor Oils

Three grades were produced having viscosities at 50° C. of 8, 10, and 12° E. and a maximum pole height of 2.24.

Aero Oils

The following specification was adhered to:-

S.G. @ 20° C.	< 0.90
Conradson Carbon Residue	max. 0.3
Viscosity @ 50° C.	17° E.
" 100° C.	2.5° E.
Pole Height	max. 2.0
Pour Point	-17° F.
(No oxidation test)	

It was stated that when production started at this refinery, a 100 ton batch of aero oil to this specification was prepared and, after engine builders had carried out acceptance tests with satisfactory results, regular production began and continued without interruption.

OTHER PRODUCTS

Gasolines. Typical data - March 1942

	<u>Hydro</u>	<u>Fischer-Tropsch</u>
Sp. gr. @ 20° C.	0.749	0.700
I.B.P. °C.	41	40
5%	60	58
20%	79	74
50%	98	99
70%	113	127
90%	138	165
95%	150	183
F.B.P., °C.	154	192
Octane No. clear	73.5	56.5
V.P. atm.	0.66	0.32

Hydro gasoline was formerly of 180° C. end point, but this was reduced to 150-155° C. when the Hydro Gas Oil pour point specification was limited to -40° C. max. Fischer-Tropsch gasoline end point was later reduced for a similar reason, in this case the diesel oil cut being 160-320° C. of -12° C. pour point. Fischer-Tropsch Gasoline of 160° C. end point had a clear Octane Number of ca.60.

No aviation gasoline was produced at Lutzkendorf.

DIESEL OILS

" Fischer-Tropsch and hydro gas oils were despatched separately from Lutzkendorf to various WIFO blending stations; while a blend of petroleum kerosine and atmospheric spindle oil was used internally in tractors and by local transport organizations.

	<u>Hydro gas oil</u>	<u>Fischer-Tropsch gas oil</u>
Sp. gr. @ 20° C.	0.865	0.743
I.B.P., °C.	179	173
20%	200	180
50%	224	195
70%	254	205
90%	328	254
Cloud point, °C.	-25	-38
Pour point, °C.	-41	-40
Filtration rate, secs.	38.4	2.6
	@-28° C.	@-39° C.
Cetane number	35.6	68.2

Hydro diesel oil specification called for a maximum pour point of -40° C. and a Cetane Number of 40-45.

ENGINE TESTING

In the engine testing laboratory, the following engines were installed:-

I. G. Prüfmotor
C.F.R. Motor Method Engine
and Deutz Diesel Engine.

These were employed for routine Octane Number and Cetane Number determinations.

MERSOL MANUFACTURE

It was stated that a Fischer-Tropsch fraction b.p. 320 to 340° C. was despatched for Mersol manufacture, and that material boiling above 340° C. containing 10% wax, was sent to Witten for fatty acid manufacture.