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THE GERMAN SYNTHETIC AMMONIA INDUSTRY IN THE WESTERN ZONES.

THE GERMAN SYNTHETIC AMMONIA INDUSTRY

IN THE WESTERN ZONES

Reported by

Dr. F.M. Irvine
Mr. W.K. Hall.

Mr. A. Gillies
Dr. J. Manning
Mr. P.P. Moulton
Dr. W. Smith

(BIOS Trip No. 2336)

B. I. O. S. Target Nos:- See Table of Contents.

16th August-19th September 1946.

BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE

32, Bryanston Square, London, W.1.

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[&]quot;A copy of U.S. CWS Intelligence Division report No: 4058, which is referred to on pages, 6, 39, 50, 51, and 83. will be lodged with the, Board of Trade, Documents Unit, Lansdowne House, Berkeley Sq. London. W.T. Telephone. GROSvenor 4060. Ext. 2923. when received from America.

Enquirers should contact the unit by letter or telephone to ensure that the documents will be available before calling to inspect it.

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Personnel of Team - all Board of Trade:

BIOS Team	Plants visited						
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Dr. F.M. Irvine (Team Leader)	x	x	×	×	x	man oliad sull ins	x
Mr. W.K. Hall (Deputy Leader)	x	x	x	x	x	es verils jagsdenbai aneda awa	almonius almonius
Mr. A. Gillies	x	x	x	x	x	ofseimed ablished	x
Dr. J. Manning	Lie	x	x	x	10.63	X	od sapp ead no.
Mr. P.P. Moulton	x	x	x	x	olios Form	X ADAM	x
Dr. W. Smith	x	x	x	x	end Treat	x	erebras of all on villed
Dates of visit	17-19/9/46	26-27 and 30/8/46	16, 19 and 21/8/46	28-29/8/46	30/8/46	30/8/46	9/9/46

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INTRODUCTION

Members of BIOS Team No. 2336, as shown in the table above, visited all the synthetic ammonia plants in the Western Zones of Germany between 16th August and 19th September 1946. In addition the Linde company was visited, as it had supplied most of the plant for separation of gases by liquefaction which was used by the synthetic ammonia works.

The team also investigated the plants where the synthetic ammonia is worked up, and has described these in two reports supplementary to the present report:

BIOS Report No.

"The German Nitric Acid Industry in the Western Zones"

1442

"The German Nitrogenous Fertilizer Industry (excluding cyanamide) in the Western Zones"

1443

The synthetic ammonia process. The history of the development, first in Germany and later in almost all industrialized countries in the world, of the synthetic ammonia industry is well-known; and technical details of the various processes, both for the synthesis itself and for the manufacture of the necessary hydrogen, have been widely published in the literature. This report therefore does not describe each process in detail, but concentrates on the capacities of the plant installed, and any variations from the usual practice. It may however by stated summarily here, that the investigation of the synthetic ammonia plants in the three Western Zones of Germany revealed no notable development in technique during the war years. The five plants in the Western Zones are all at least 16 years old, and any radically new improvement might have been expected rather at the latest plant erected by the Germans at Linz in Austria. Though the Linz plant has not been examined in great detail, there has so far been no indication to suggest that it includes striking innovations.

FIAT Final Report No. 481, for example, shows that the process is very similar to that at Oppau; for example, a C.O.G.-oxygen-air mixture is cracked, and thereafter CO conversion, CO2 and CO removal and the ammonia synthesis is on usual Haber-Bosch lines, so that only the cracking process is particularly novel. The ammonia oxidation and lime ammonium nitrate processes at Linz also are of normal I.G. type.

The plants in the three Western Zones are however of some historical interest, as they include the prototypes of two of the processes (the Haber and the Mont Cenis), and in addition they illustrate a considerable range of the known processes for synthetic ammonia:-

Works	Commenced operation in	Process	ses for manu	facture of synthetic ammonia	Present capacity (tons N/ year)	(See p.)
Oppau	1913	gas (Bo c.o.g. (with	gas/producer osch); also cracking oxygen)and separation	Haber	280,000	(10)
Herne- Sodingen	1927 (now dismanthed)		separation	Mont-Cenis (Uhde)	-feig atero ha	(77)
Hibernia	1928		do.	do.	46,000	(39)
Victor	1928		do.	Claude (change- over to Haber)	63,000	(50)
Ruhr- ohemie	1929		do.	Casale	15,750	(26)
Ewald	1930	(withou	cracking it oxygen), eparation	N.E.C.	30,000	(61)

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was produced by the Liede produce. The emmonia

The US-CWS Int. Div. Dep. 4058 (14/8/45) quotes the synthetic armonia capacity in Germany before the bombing as follows:

Present Zone	Company	Plant location	Tons N/	Remarks
British	Ruhr- chemie	Oberhausen- Holten	54,000	MANO CONTRACTOR OF THE PARTY OF
	Hibernia	Wanne- Eickel	50,000	Enlarged during the war
	Victor	Castrop- Rauxel	50,000	Claude plus I.G. process
	Ewald	Oer- Erkenschwick	35,000	
French (Total,	I.G.	Oppau	270,000	Selve 4 Language
Western Zones)			459,000	Sodingen Sin
Russian	I.G.	Leuna	365,000	Enlarged during
Poland	I.G.	Heydebreck	100,000	Not completed - max. production was 3,000 tons N/month.
Total Germany			924,000	
AUSTRIA	I.G.	Linz	60,000	rext ligitable to

Thus the Western Zones had 459,000 tons N, or 56%, and the Russian Zone 365,000 tons N or 44% of the total synthetic ammonia capacity of the present area of Germany (824,000 tons N/year).

BADISCHE ANILIN- UND SODAFABRIK A.G. (formerly part of I.G. Farbenindustrie A.-G.)

Oppau bei Ludwigshafen-am-Rhein (French Zone)

Dates of visit: 17th-19th September 1946 Detaint saw of tot lot list ed!

By: Dr. F.M. Irvine
Mr. W.K. Hall
Mr. A. Gillies

Mr. P.P. Moulton

Dr. W. Smith

Personnel interviewed:

Dr. Göggel - Manager of Nitrogen Department

Dr. Gloth - Manager of Low-pressure Plant

Dr. Allsert Heinzel - Manager of High-Pressure Plant

Oppau is of particular interest as the birthplace of the synthetic ammonia industry, the first commercial production having commenced in September 1913. The remarkable story of the development of the Haber-Bosch process is now well known, and only a few salient dates are recapitulated below to illustrate the continuous developments in processes which have occurred at Oppau:

- The B.A.S.F. financed Haber in the continuation 1909 of his work on the equilibria for the system ammonia-hydrogen-nitrogen and on catalysis.
- Dr. Carl Bosch and Dr. Mittasch developed a 1910 practical synthetic ammonia process at Ludwigshafen on a small scale.
- Construction of commercial-scale plant was started at Oppau, using the Linde process for the 1911 separation of hydrogen at low temperature from water gas; and all types of high-pressure compressors were tested.
 - The process wentinto production, using pure nitrogen also produced by the Linde process. The ammonia was recovered at first as anhydrous liquid and then in
 - 1915 water scrubbing was introduced to recover the ammonia. (Later in 1926 the recovery of ammonia was changed back to recovery in anhydrous liquid form)

- 1914/15 The war demand for fixed nitrogen stimulated development and the production of the hydrogennitrogen mixture was changed over to the "Bosch" process. In this the water-gas, after removal of hydrogen sulphide in oxide boxes, was passed with steam through a catalyst to convert the carbon monoxide and the steam to hydrogen and CO₂. The bulk of the CO₂ was removed by scrubbing with water at 25 atmospheres. The residual carbon monoxide was removed by scrubbing with copper formate at 200 atü. and the residual CO₂ with an alkaline solution also at 200 atü.
- 1923/24 The desulphurising of the water-gas was changed from the oxide box to the active carbon process. The sulphur was recovered from the active carbon in a pure state and used for sulphuric acid manufacture. Electrical heating was now used in the synthesis process for the first time in the reduction of the catalyst and for starting up. Previously the mixed gas had been heated up by external gas heating.
- leader and C.O.G. separation plants were installed capable of producing 80 tons N/day.
- 1943 Oppau attained its highest production of 750 tons N/day.

From the beginning developments have continued in all phases of the process, in the design of compressors, high-pressure vessels, joints, methods of purification, etc. and the production of suitable catalysts.

Experience at Oppau was used in the design of Leuna in 1917. and of Linz (Austria) in 1938.

also arriaged to the thits process. The amount were reported to former as serverous like and and

The gas-separation plants supplied by Linde, according to Linde's own statements, (p. 80) were as follows:

Year Gas treated	Capacity (m3/hour)	Gas	Remarks
1910 Water-gas	40	H ₂	
1911 " "	200	H ₂	
1912 " "	2x2300	H ₂	
1912 Air	4x350	N ₂	Possibly for Leuna?
1917 "	2x2500	N ₂	
1920 Mixed gas	80	H ₂	For ammonia?
1921 Water-gas	1250	H ₂	
1934 Crude gas	600	H ₂	
1937 C.O.G.	3x9350	H ₂	
1938 Air	3x1000	0 ₂	efiling edf
	3x3500	N ₂	alcomma ofter

The Ludwigshafen-Oppau complex, which includes chemical plants of all kinds, was very heavily damaged by bombing and military operations, and interruptions to supply through delays in discovering the damage done to cables and pipe-lines are still occurring.

In 1925 the B.A.S.F. was the most important of the founder firms of the I.G. Farbenindustrie A.-G. With the dissolution of the I.G. Farben by order of the Allied Control Council, the Ludwigshafen-Oppau works in the French Zone have returned to their original name of Badische Anilin- und Sodafabrik A.-G.

Summary of process. Most of the hydrogen-nitrogen "mixed gas" is made by the Bosch process. Water-gas from the periods of the production cycle before and after the main "make" period (i.e. in effect water-gas plus some producergas, with ca. 14% of N) is scrubbed and HgS and organic sulphur compounds removed by catalytic oxidation over active carbon, sulphur being recovered. Steam is added and the catalysed "shift" reaction (at either ordinary pressure or in one high-pressure converter at 25 atu.) converts the CO + H2O to CO2 + H2. Scrubbing under pressure removes most of the CO2 with water, the CO with ammoniacal cuprous carbonate solution and the remainder of the CO2 with ammonia solution. The carbon dioxide is recovered and used for the manufacture of dry ice and in making ures and ammonium bicarbonate, whilst the less pure CO2 was formerly used for ammonium sulphate from gypsum; and the carbon monoxide is also recovered and used for carbonyls or organic syntheses.

Smaller amounts of hydrogen (equivalent to 80 tons N/day each) are obtained (i) by cracking coke-oven gas with oxygen-enriched air and (ii) by the ordinary partial liquefaction of coke-oven gas, scrubbing with liquid nitrogen. There are no by-products (e.g. carbon black) from (i), but from (ii) the usual methane fraction and ethylene fractions are recovered, and a carbon monoxidenitrogen mixture.

The <u>nitrogen</u> (capacity ca. 10,500 Nm³/hour) for the gas-separation process is supplied by a Linde airseparation plant, which also produces pure <u>oxygen</u> (capacity ca. 3,000 Nm³/hour) used in the C.O.G. cracking process and for other purposes.

The purified "mixed gas" $(3H_2 + N_2)$ is converted to synthetic ammonia by the Haber-Bosch process at 250 atū. The capacity is 770-800 tons N/day.

Raw materials and energy. Coke, coal and coke-oven gas are supplied from the Saar.

Energy is supplied by Oppau's own power station, operated by producer-gas engines using brown coal briquettes, and supplied at 6000 and 220 v.

For requirements of fuels at almost full capacity (750 tons N/day) with the associated manufacture also of methanol and butyl, see below, p. 23.

At full pre-war output, Oppau required 600-700,000 m³/day of Rhine water, mostly for cooling.

Processes.

A simple flow-sheet of the whole process, from coke and C.O.G. to the ammonia synthesis, is given in Diagram No. 1, but the main processes are described separately below under

Air separation
Mixed gas from coke
C.O.G. cracking
C.O.G. separation
Ammonia synthesis

A brief description of the processes at Oppau as they were in about the year 1928, with excellent photographs, was given in an 'I.G. brochure entitled "Die Stickstoff-düngemittel der I.G. Farbenindustrie A.-G. und ihre Erzeugungstätten Oppau und Leuna", 1928 (70 pp.) (see BIOS/DOCS/2336/1233/18). This

shows for example: 0.0 and of believes at asyon	Page
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Pipe-bridges in the gas plant (a characteristic and impressive feature)	18
Gas compressors Pressure water pumps	22-23
H.P. gas purification pumps CO-removal tower Ammonia synthesis converters	21 24

AIR SEPARATION

The Bosch process (and later modifications) for preparing the "mixed gas" (3H₂ + N₂) does not depend for its nitrogen on the liquefaction of air, as do all the Ruhr plants based on hydrogen from C.O.G. which are described later - Ruhrchemie, Hibernia, Victor and Ewald. As mentioned above, Oppau installed 4 small Linde air-separation units for a total of 1,400 m³/hour of N₂, in 1912. This was to supply nitrogen for mixing with the 4,600 m³/hour of H₂ produced by partial liquefaction of water-gas. This process was however abandoned for the Bosch process in about 1915.

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There are now three Linde air-separation units, installed in 1938, each with a capacity of:

CONTRACTOR DESCRIPTION	m3/hour		
Tout ded led an maximite of	N2 OL Deams	02	
(according to BASF)	3,500 (99.95% pure)	1,100 (99.5% pure)	
(according to Linde)	3,500	1,000	

They are mixed-pressure type of standard Linde design with the usual CO₂ removal, pre-cooling, ammonia cooling followed by expansion of the H.P. air through an expansion valve, and rectification of the liquid air. The "life" of successive runs is 3-4 months.

The nitrogen is supplied to the C.O.G. separation plant (see below), for correcting the ratio of H2: N2 in the ammonia synthesis gas and for purging parts of plant before opening up for overhaul, etc. The oxygen is used in the conversion process to maintain the temperature of the catalyst; in the cracking plant for/residual gas from the methanol synthesis; for welding purposes; and for catalyst making.

MIXED GAS FROM COKE.

A much-simplified flow-sheet of this process and of its connections with the recovery of hydrogen from C.O.G. (with or without previous cracking) is shown in Diagram No. 1. The process was originally worked out by Bosch.

Water Gas Generation. There are 30 generators each of 6-7,000 m³/hour capacity. Some are automatic, some hand-operated and some with revolving grates. The total gas output is 180,000 m³/hour, 60% of which is used in the synthesis of ammonia. The gas-making period of the cycle of operations is divided into three parts; the first and third parts (i.e. including part of the "blow" period, or, in effect, producer gas) yield the gas for ammonia production, while the middle part (i.e. the "pure" water gas "make") gives a gas with only 2% N which is taken separately for (i) production of pure hydrogen (97% H₂, 3% N₂) and (ii) for methanol synthesis.

The flow-sheets for these processes (i) and (ii) are similar to that for "mixed gas" for ammonia synthesis, as shown in parallel lines on Diagram No. 1, but apart from their inter-connection with "mixed gas" manufacture are not dealt with here.

The approximate composition of the gas for ammonia synthesis is as follows:-

	1	%
Ho		40
H2		36-38
CO2		10
N2		14 beach
CHA		0.2
Rare	gases	0.2
	Total	100.4-102.4
		CONTRACTOR OF THE PROPERTY OF

"Blow"/

"Blow" gas can be added to adjust the nitrogen content to the correct ratio.

The gas from the generators, after the usual water scrubbing process for the removal of dust and tars, passes to a gasholder.

Removal of H2S and organic sulphur compounds. The water-gas contains

hydrogen sulphide 2.0-2.2 organic sulphur compounds (measured as S) 0.3-0.5

These are removed by catalytic oxidation to S over active carbon. The oxygen necessary is added to the water-gas (in slight excess of the amount theoretically necessary) either as air or as gaseous oxygen; and a small amount of ammonia is also added, to accelerate the The mixture is then passed downwards through reaction. a layer, 1.2 m. thick, of active carbon, of which the upper half is of granules 1-2 mm. in diameter and the lower half of granules 2-4 mm. in diameter. The carbon, 20 m3 in volume, is contained in a vessel 4.5 m. in diameter and ca. 3 m. high. There are 30 such vessels, each capable of treating 6,000 m3/hour of water-gas. It is claimed that all the HoS is removed and about half the organic sulphur, and the residual sulphur is of the order of 0.15 g./m3.

Recovery of S. The "life" of each bed of active carbon is 20 days, after which the free S is extracted at normal temperatures with an ammonium sulphide solution containing 12% of NH3 and 7% of S. Four treatments are required to remove the S almost completely from the active carbon, after which the carbon is washed with water and finally steamed for about 10 hours until the condensate is free from S. The vessel is purged with nitrogen and the carbon is then ready for putting into operation again.

The ammonium polysulphide liquor is steam-distilled at 3 atü. to recover the free sulphur, in a column about 7 m. high and 0.75 m. in diameter, constructed in V2A steel, packed with earthenware Raschig rings. There are 4 such columns available. The feed of liquor is 2-3 m3/hour. Ammonia vapour and a little S pass from the top of the column at 130° C. to a cooler, after which dilution takes place to produce a suitable strength liquor again for S extraction. Sulphur and water are run off at the base/

base into a steam-heated, lagged separating tank. The molten sulphur is then run into a shallow tank from which, after cooling and solidification, it can be dug out by hand.

The steam consumption for this process is $l_2^{\frac{1}{2}}$ tons/hour at 14 atü. when working at 50/60,000 m³ gas/hour. The sulphur production at this load is about 3 tons/day.

CO Conversion. This process has in the past been operated at normal pressures and the bulk of the gas is still so treated. A conversion plant for 25,000 m³ gas/hour and working at 25 atü. has however been built, though it has worked for only two months so far.

The reaction is sometimes termed the water-gas "shift" reaction and the equation is:

$$CO + H_2O = CO_2 + H_2$$

The gas composition is changed roughly as follows:

	Before conversion (desulphurised water-gas)	After conversion ("catalysed gas")
	%	%
H	40	52
CÖ	36 - 38	3-5
COo	10	26
No	14	18-20
CO ₂ N ₂ Total of these	100-102	101-103

Catalyst for the conversion. This is made from waste FeSO₄ from Leverkusen by precipitating ferrous hydroxide with soda. The precipitate is filtered, mixed with chromium nitrate and dried and then pressed and heated to 450° C. to obtain pieces 5-15 mm. in size. The Cr₂O₃ content of the catalyst is 8%. The reduction of the catalyst is carried out in the converter by heating with combustion gases (water gas burning with wair) to 400° C. and then changing over to water gas. The reduction takes 3-4 hours.

The same catalyst is used for both the L.P. and the H.P. conversion processes. These two processes are described, with flow-sheets, in CIOS Report No. XXX-103 "I.G. Farbenindustrie A.-G. Works, Ludwigshafen and Oppau" by R. Holroyd, 16/8/45, on pp. 2-5 and Figs. 1-3; but this refers/

refers to the I.G. processes in general, at seven different works, e.g. the L.P. flowsheet is for Leuna and the H.P. flowsheets for Blechhammer and for Brüx. The present report however describes only Oppau.

L.P. conversion. The desulphurised water-gas is compressed in a blower to 2 m. W.G. and passes up a tower packed with wooden grids, down which flows water at 80° C. recovered from a condensing cooler at the end of the process. The gas is thus saturated with water-vapour, and live steam is then added to increase the H₂O content to the proportion necessary for the reaction.

In the converter itself there are 3 layers of catalyst supported on grids, superimposed on a tubular heat-exchanger. There are 45 such L.P. converters.

The water-gas - steam mixture is heated in the exchanger up to 350° C. before entering the top layer of catalyst. The exothermic reaction raises the temperature to 550-600° C., and the gas is then quickly cooled by passage through a layer of Raschig rings sprayed with condensed water. This also introduces additional water-vapour into the gas, which then passes through the second layer of catalyst, followed by more water-sprayed Raschig rings to control the temperature in the third layer of catalyst to 360-380° C. The fully catalysed gas then passes through the heat exchanger where it is cooled to 150° C.

Two coolers follow, one cooling the gas to condense out hot water at 80° C. for use in the saturator mentioned above and a final cooler to bring the temperature of the gas down to normal. The converted gas then passes to a gasholder.

The steam consumption in this process is 600 g./m3 of gas and the total water for cooling between the layers of catalyst 100 g./m3.

H.P. conversion. There is only one H.P. conversion system, but by operation at 25 atü. its capacity is increased to 25,000 Nm³ of water-gas per hour, as compared with only 3,000 Nm³/hour for a L.P. converter of similar catalyst volume.

The/

[▼] CIOS Report No. XXX-103, Fig. 2 shows only 420 g./m³ at Leuna.

The water-gas saturator is similar, but the steam injected is at 40 atu. The gas-steam mixture is heated in an exchanger by the catalysed gas leaving the converter. In the upper part of the converter the catalyst is in tubes which are cooled externally by the incoming water-gas. The gas leaving the catalyst in this section is then cooled as in the L.P. converter by water addition to control the temperature of the deep layer of catalyst in the lower half of the converter.

Removal of CO₂. Catalysed gas from the gasholder of the L.P. system is compressed to 25 atü. in 3 stages before passing to the water scrubbing towers for CO₂ removal. There are 24 towers each 16 m. high (with 11 m. Raschig ring packing) and 1.4 m. in diameter, and 4 towers each 21 m. high and 2 m. in diameter. The 24 towers can deal with 200,000 m³/hour and the 4 towers with 60,000 m³/hour.

Each of the 24 towers is fed with 400 m3 water/hour and the water leaving the base of the tower at 25 atu. is passed through 4 sets of 3 Francis turbines to recover up to 40% of the power necessary to pump the water over the The pump, electric motor and Francis turbine are coupled together and mounted on one badplate in each case. Six towers are connected into a common water outlet manifold feeding three turbines so that two turbines can be left on full load and the third turbine can be controlled to maintain the level in the base of the towers. pump turbine units are such that the turbine lets down the water pressure from 25 atu. to 3 atu. and the gases released from the water at this pressure (mainly hydrogen and nitrogen) are collected and can be sent back to the process for hydrogen recovery. On letting down the pressure of water from 3 atmospheres to atmospheric pressure the gas released now consists of 99% CO, which is for the production of urea , dry ice and ammonium bicarbonate; the production of the last is 400-500 tons/ month.

The gas released from the water let down in one stage to atmospheric pressure contains 92-93% of CO2, the rest being largely hydrogen and nitrogen; it was formerly used for ammonium sulphate production by the gypsum conversion process.

The catalysed gas leaving the water scrubbing towers contains 1.5-2.0% of CO₂, which is left in the gas in order to/

^{*} See companion BIOS Report No. 1443, p. 48 (urea) and p. 21 (ammonium sulphate).