

## XX FERTILISERS AND NITRIC ACID

This Division covers the manufacture of Ammonium Sulphate, Nitric Acid, Calcium Nitrate (Kalksalpeter), Ammonium Nitrate and Calcium Carbonate mixture (Kalkammonsalpeter, equivalent to Nitro Chalk), Leuna Salpeter, and Phosphate Fertilisers. Dr. Ernst Willfroth was interrogated on May 12th; he was the Manager of the Nitrogen Division. There appeared to have been no major developments in what were old-established processes.

### Ammonium Sulphate

This was made by the reaction of Anhydrite with  $\text{NH}_3$  and  $\text{CO}_2$ . The capacity of the plant up to 1935 was 500 T/D  $\text{NH}_3$ ; in 1935 a part of the building was taken over to produce Leuna Salpeter, so that the capacity of Sulphate dropped to 300 T/D N.

Carbonated liquor was manufactured by the Ammonia Division by scrubbing out  $\text{CO}_2$  from catalysed gas at 1 at. This liquor contained 14% as  $(\text{NH}_4)_2 \text{CO}_3$ . These scrubbers were followed by a Sulphuric Acid wash to recover Ammonia.

The reaction with Anhydrite was carried out in the presence of excess  $\text{NH}_3$ , and after filtering the liquor could either be decomposed by heating to recover  $\text{NH}_3$  or else it could be neutralised with 50%  $\text{H}_2\text{SO}_4$  (Sulphuric Acid was made on site, the source of Sulphur being  $\text{H}_2\text{S}$  in water gas recovered in an Alkacid Plant and converted to Sulphur in a Claus Plant).

The filtered mud contained 0.5% Nitrogen ( $\approx$  2% Ammonium Sulphate) on a dry basis. Rotary filters were installed but Dr. Willfroth preferred the old wooden plate type, which was still in use; these had plates suspended inside large wooden troughs.

The evaporation of the liquor was done in double-effect evaporators. The first effect was merely a concentrator, without salting; the steam pressure was 7 lbs. g., the temperature of the solution  $106^\circ\text{C}$  and the internal pressure 100-200 mm.Hg. The second evaporator ran with steam vapours at 0.2 ats. abs. with a steam temperature of  $56-62^\circ\text{C}$  and a liquid temperature of  $46-52^\circ\text{C}$ , the final vapour pressure being 65 mm. Hg. Each evaporator had a heating surface of 124  $\text{m}^2$ , and the combined output was 170 T/D Ammonium Sulphate. Steam consumption was about 0.9 T/T Sulphate.

The solution prior to evaporation at density of 1.24, contained 530-540 g/l Ammonium Sulphate and about 0.2 g/l  $\text{H}_2\text{SO}_4$ , with a pH of about 5.

The evaporators were lead covered throughout, but it was not stated whether any alloying metal was included. The evaporator tubes were 50 mm. I.D. and 78 mm. O.D.

The anhydrite used came from a quarry in the Harz about 130 km. away. Fuller mills were originally used, and some of these were still there, but others had been replaced by Lösscher mills, which occupied far less space but had the same energy consumption. These mills worked on the principle of one roll working on a vertical axis, with two inclined rolls running on the top, the ground material being removed by hot air.

The plant itself was very old and there had been no advances made in recent years; the double-effect evaporation has been in use for many years. The plant has been shut down since May of 1944, owing to non-availability of  $\text{NH}_3$ . Direct air raid damage was not very great, and the plant personnel estimated that they could make 100 T/D N after 4-6 weeks and 300 T/D N after 10 months.

#### Nitric Acid

The plant was built in 1927 and was said to be the only plant in Germany working at 5 ats. The original capacity was 150 T/D N, but it had been enlarged (during the war?) to 260 T/D. Most, if not all the other, I.G. Plants erected elsewhere operated at 1 at. The main advantage of using a pressure process was stated to be the saving of space for the absorption system, and the process was economic if energy was not too expensive.

Power was recovered from the hot let down gases by passing them through a turbine working on the same axis as the air compressor.

The catalyst favoured was platinum with 5% rhodium. This catalyst was much better than 1% rhodium, but in a pressure process 10% rhodium was stated to give no better results.  $\text{NH}_3$  conversion efficiency was stated to be 96% on the gauze and 92-94% after absorption. Final scrubbing of the gases was done with a lime solution. The final acid contained 48%  $\text{HNO}_3$  and was all used for making Nitrate of Lime and Ammonium Nitrate mixtures.

It had been found that if welding took place in the neighbourhood of the air compressors, poisoning of the catalyst resulted, and this was put down to the presence of poisons such as  $\text{PH}_3$  in the acetylene.

The plant was not inspected closely but from a distance it appeared to be relatively undamaged.

### Concentrated Nitric Acid

This was not made by working up 48%  $\text{HNO}_3$ , but was made directly from the nitric acid oxidation gases. Gases after the waste heat boiler were further cooled and then oxidation of  $\text{NO}$  to  $\text{NO}_2$  allowed to take place. The gases were then indirectly cooled with water to remove  $\text{H}_2\text{O}$  as weak  $\text{HNO}_3$ . The gases were then further cooled to  $-15^\circ\text{C}$  to give liquid  $\text{NO}_2$ . This  $\text{NO}_2$  was then treated in an autoclave at 80-100 atms. and  $70^\circ\text{C}$  with dilute Nitric Acid and Oxygen, yielding pure  $\text{HNO}_3$  plus  $\text{N}_2\text{O}_4$ . On distillation this mixture evolved  $\text{NO}_2$ , which was returned to the process and gave 99%  $\text{HNO}_3$  at the bottom of the still.

### Ammonium Nitrate

Only a small quantity of the pure material was made; the bulk was made into Kalkammonsalpeter. This latter plant was not investigated but the process was said to be very similar to that used in making Nitro-Chalk. The spray tower was at the end of the building.

### Calcium Nitrate (Kalksalpeter)

This plant was very heavily damaged but the material could be produced in the Nitro-Chalk plant, although not at the same time as Nitro-Chalk.

### Phosphate Fertilizers

This was contained in the same building as Ammonium Sulphate, but none had been made during the war, owing to shortage of phosphates.

### Leuna Salpeter (Amn. Sulphate & Nitrate)

This plant also formed part of the Ammonium Sulphate plant, but was not inspected in detail; it had suffered little air raid damage.

### Ammonium Chloride Fertilizers

No work had been done on developing Ammonium Chloride fertilizers, which were said to be unsuitable for German soils.

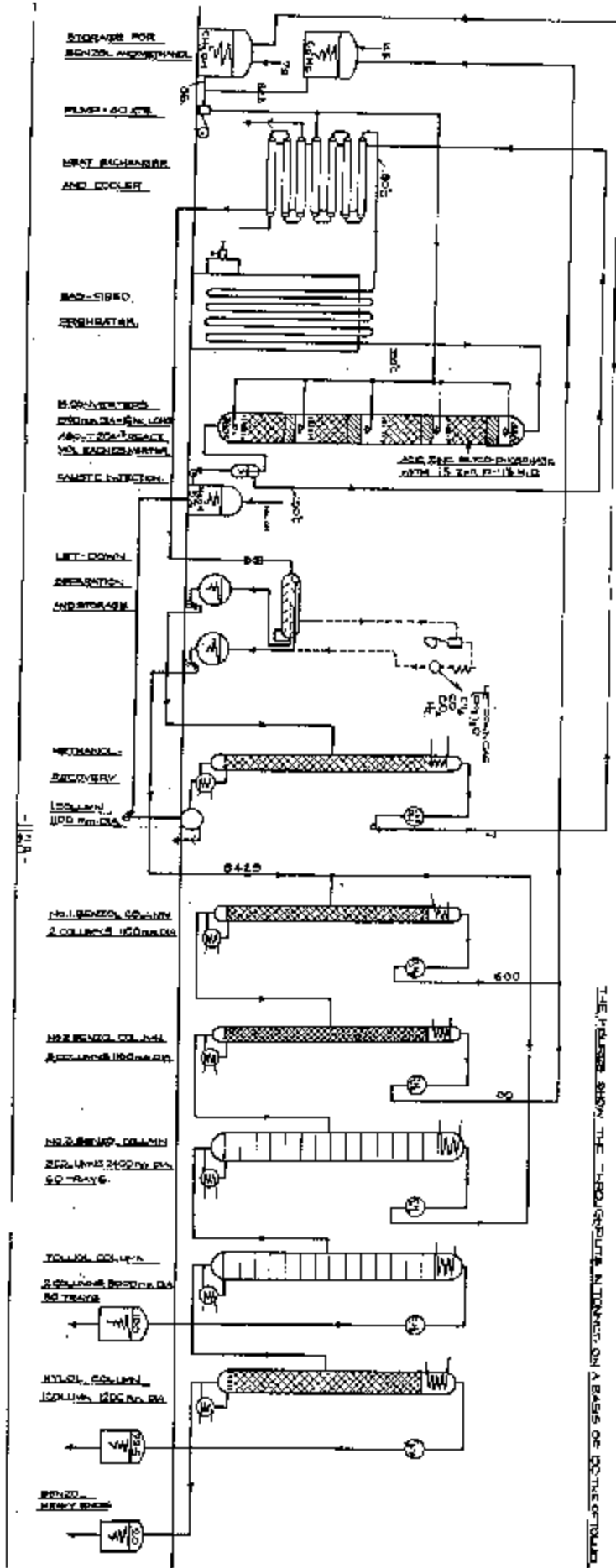


FIGURE 2000000  
 TOLUOL SYNTHESIS CAPACITY 5000 TON/MONTH. WALDENBURG. SCHLEICHEN

THE FIGURES SHOW THE THROUGHPUT MINIMUM ON A BASIS OF CONTINUOUS