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TECHNICAL REPORT NO. 257-45

NITROGLYCERIN, DIETHYLENE GLYCOL DINITRATE AND SIMILAR EXPLOSIVE OILS - MANUFACTURE AND DEVELOPMENT IN GERMANY.

SUMMARY

This report describes the manufacture and development work on explosive oils at three representative German plants. Two types of continuous units and the batch process equipment were observed.

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NITROGLYCERIN, DIETHYLENE GLYCOL DINITRATE AND SIMILAR EXPLOSIVE OILS - MANUFACTURE AND DEVELOPMENT IN GERMANY.

1. Introduction.

Plants for the manufacture of the explosive oils were investigated by this team at Schlebusch on 28 June, at Krummel on 15 July, and at Bomlitz on July 17th and 21st.

The Dynamit AG plant at Schlebusch had three units, the old batch process, a modified Schmidt-Meissner continuous plant and the Biazzi unit, the most modern. This plant made only Nitroglycerin (NG) and Ethylene Glycol Dinitrate (EGN) for use in commercial dynamites made there.

The DAG plant at Krummel had four batch process units, two for the dynamite oils and two for the Diethylene Glycol Dinitrate (DEGN) and similar oils used for cannon and rocket powders.

The Eiba plant of Wolff and Co. at Bomlitz had six complete Schmidt- Meissner continuous units, producing only DEGN for powders. There were five small units and one large one.

German cannon and rocket powder compositions were developed primarily taking advantage of the desirable properties and the availability of Diethylene Glycol Dinitrate (DEGN). The manufacturing process and equipment for this material were based on the experience gained in the plants making Nitroglycerin (NG) and Ethylene Glycol Dinitrate (EGN) for dynamite.

On the three plants visited, information was obtained on three types of nitration equipment which cover the most important differences in modern technique.

Until about 1925, all dynamite plants used the batch process for nitration of NG and EGN. At about that time Schmidt developed a continuous nitration and neutralizing unit at Krummel works of Dynamit AG, the first dynamite factory of Alfred Nobel, the inventor. A number of these units were built at other plants but there are none now at Krummel. Serious accidents occurred at several of the installations.

Introduction (Cont'd.)

Because of the inherent hazard of the Schmidt type of NG waste acid separator, the Biazzi Co. of Italy developed a continuous unit, one of which was observed on operation at the Schlebusch Works of DAG. After its successful operation was demonstrated, the Schmidt unit at that plant was changed by replacement of the separator with a Biazzi type separator.

The DEGN units observed at Bomlitz were the standard Schmidt type as manufactured by the Meissner Co. The staff at that plant readily agreed that the Biazzi separator was superior and preferable to the Schmidt separator. They preferred, however, the Schmidt washing system to the Biazzi.

This report covers only the information on explosive oils and reference to their further processing and use is contained in the following technical reports:

Nitrocellulose and Paste, No. 258-45.
Cannon Powder, No. 259-45.
Rocket Powder, No. 260-45.

2. Schlebusch.

The NG nitrator units at Schlebusch were built in bunkers with a horizontal cylindrical concrete roof covered with earth. One end of the room was made of frame and glass windows to serve as a blow-out wall in case of explosion. In all three units, nitration, separation, water washing, and soda neutralization were carried out in the one room.

For nitration of Glycerin or Ethylene Glycoldinitrate and mixtures of these, the Schlebusch dynamite plant had three units. The first and oldest was the Batch process. The second was a Schmidt-Meissner, modified by use of a Biazzi separator. The third was the most modern, the Biazzi unit.

(a) Batch Process.

The equipment used for the batch process and the sequence of operations is shown in the attached diagram.

Schlebusch. (a)(Cont'd.)

The nitrator charge was 200 kg of Glycerin and 990 kg of mixed acid containing 51-52% HNO_3 and 51-52% H_2SO_4.

The nitrator was made of V2A alloy, the separator was lead, and the wash tanks were made of V2A.

The time cycle for nitration was one hour. As the equipment was so small and had no interesting features, other details were not recorded. Full details on the DAG equipment and process for batch nitration are described in the Krummel part of this report.

(b) Schmidt-Meissner Process.

The details of the Schmidt-Meissner unit at Schlebusch have not been recorded because complete description of this equipment is given in Part IV - Bomlitz. The original separator and washer towers on this unit had been replaced by Biazzi type separators and washers because of the difficulty of cleaning and the inherent hazards. In most aspects the Schmidt unit was then quite similar to the Biazzi.

(c) Biazzi Process.

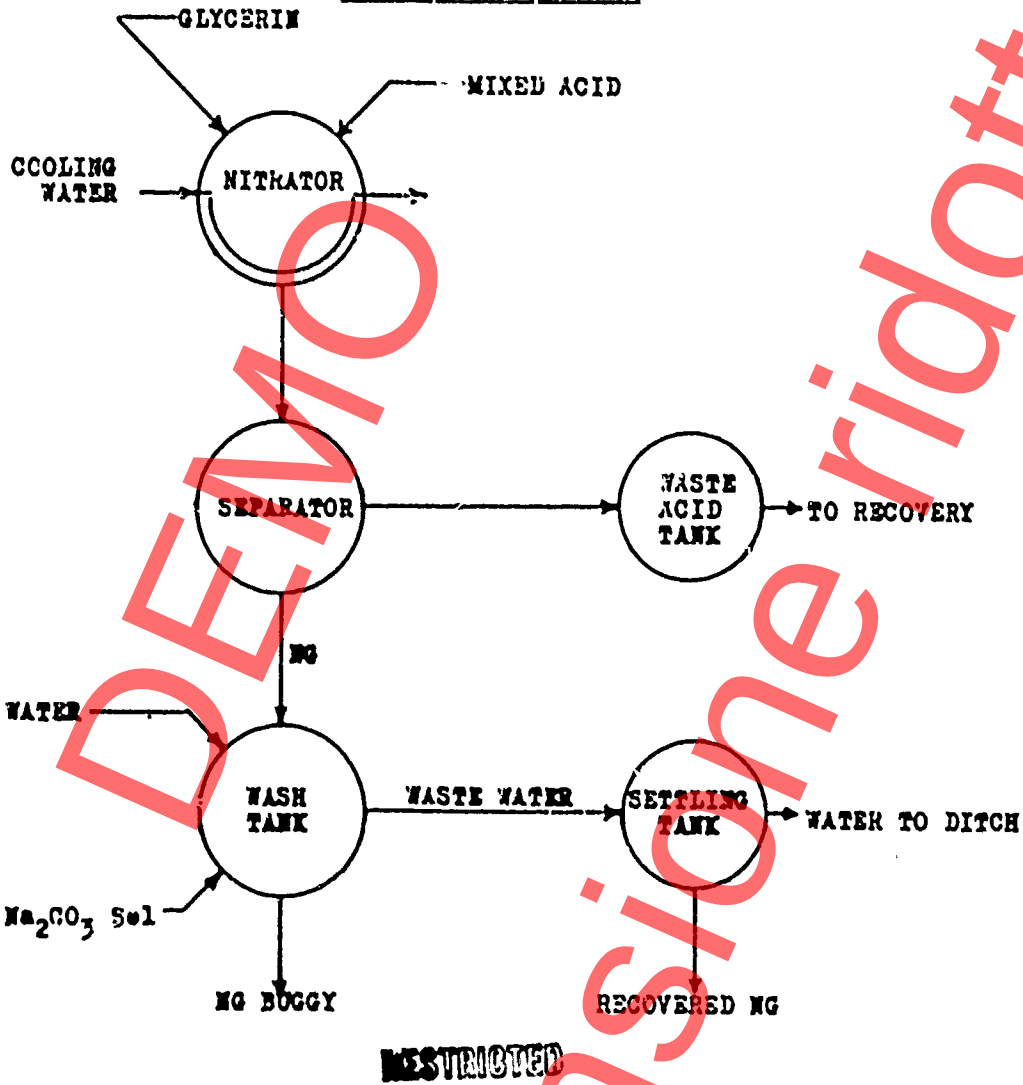
The equipment sequence and sizes for this process are shown in the attached diagram. A picture of the nitrator is also shown. All of it was located in the one house except the final N.G. separator and the wash water setting tanks.

In the Biazzi process, small vessels (50 liters) with intensive mechanical agitation were used for washing the NG. These mixtures were then separated continuously in larger vessels (360 liters) which were quite similar to a conventional cyclone separator. All of the equipment was made of V2A alloy. The welding of this equipment appeared to be very well done.

The Glycerine or Ethylene Glycol mixture with Glycerin was fed continuously from a constant level tank through a Rotameter type flowmeter. The mixed acid was added through similar equipment. The analysis of the mixed acid was 51-52% HNO_3 and 51-52% H_2SO_4. The mixed acid to Glycerin ratio was 4.9 parts to 1 part.

The capacity of the unit was 800 to 1000 kg. (2200 lbs.) of NG

SCHLEBUSH
BATCH PROCESS FOR NG



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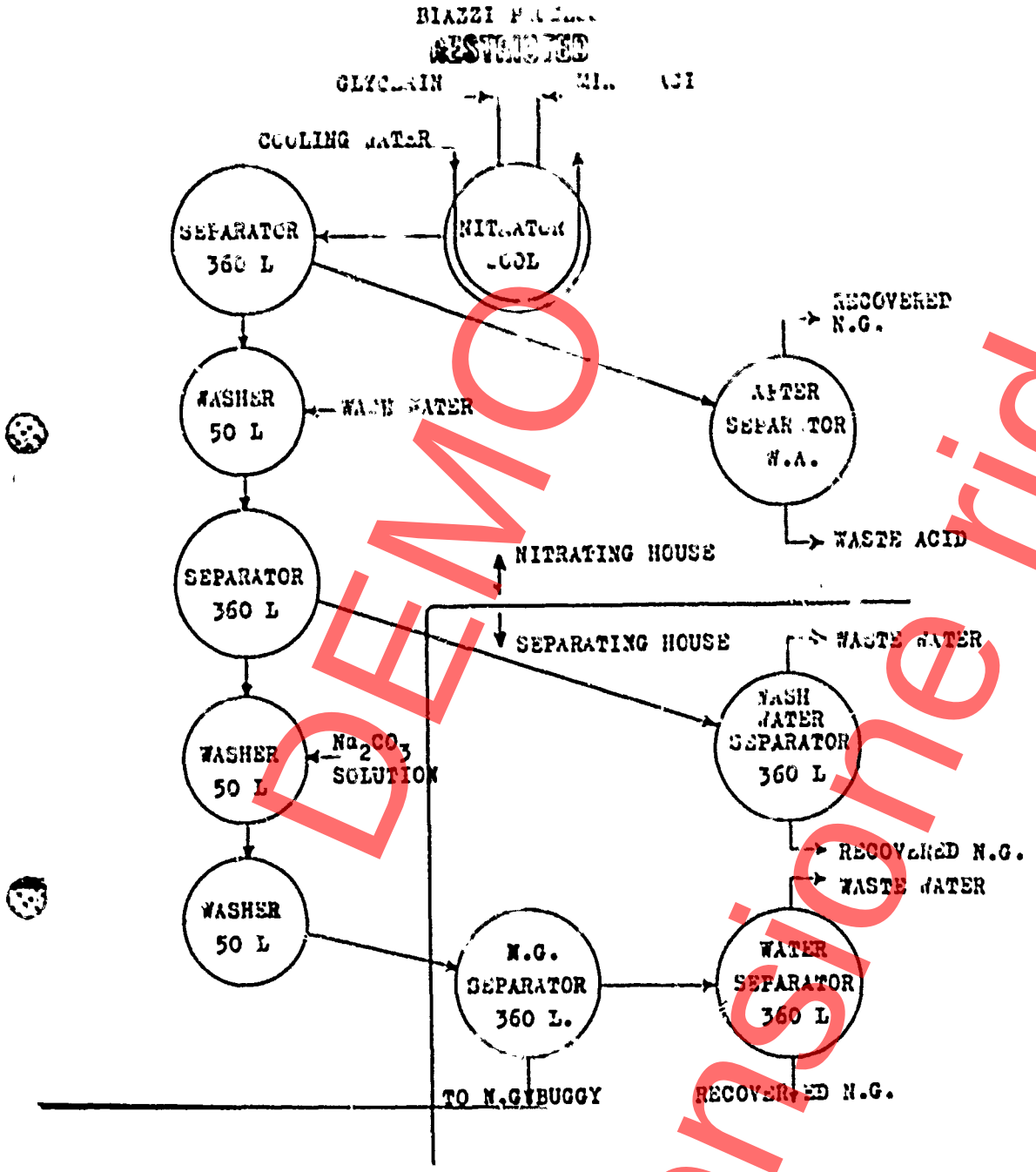

PHOTOGRAPH OF SCHLEBUSCH-NG-BIAZZI
NITRATOR

Schlebusch (c)(Cont'd.)

per hour. With a volume of 200 liters in the nitrator, it was stated that there was about 80 kg of NG retained in the vessel.

The nitrator had six cooling coils. The agitator blade turned at 400 RPM and pulled the charge downward. The degree of emulsification of the oil and acid was highly important since too much agitation hindered proper separation. The temperature of the charge was maintained at 23-25° C.

The nitration mixture was run from an outlet pipe at the top of the nitrator over to the side inlet of the separator. This inlet was placed to provide tangential flow. The separator was a cylindrical tank with a conical bottom. With the emulsified feed fed in at the middle point, the separated waste acid was run out the bottom through a trap rising up to a line to the after separator. The acid oil was run off from a top outlet of the separator and over to the first washer. The simple construction and the lack of baffles or pockets in the Biazzi separator were distinct advantages over the Schmidt separators. It was stated that separation was quite uniformly good and no agents such as guhr for improving separation were needed.

The first washer for the acid oil was a 50 liter agitated vessel where the wash-water was thoroughly mixed (420 RPM) with the oil continuously. The overflow of the mixture was led to the next separator which was built similar to the other vessel described above. From here the wash water was sent to a final separator in the next building and then either to the sewer or to the denitrating recovery tower. Any oil collected in the final water separator was returned to the system at the neutralizer washer.

The pre washed oil was next run to two agitated washers in series. These had been provided to allow the addition of sodium carbonate solution at two points. In practice, the soda solution was added only in the first. The additional agitation provided in the second mixer assisted in obtaining complete neutralization of the oil. The emulsion of oil and soda water was then run by gravity thru V2A pipe to the second building, about 50 feet away, for final separation.

This building was built as a bunker similar to the first but with the blowout wall at right angles to it. The final separator for oil and soda water was exactly the same as the others described above.