Nitrocellulose and paste manufacture at Krupp and Bohlitz works

September 1945

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Plants for the manufacture of Nitrocellulose (N.C.) and Paste (the mixed N.C. and explosive oils such as Diethylene Glycol Dinitrate etc.) were investigated at Krummel near Hamburg on July 15, 1945 and at Bomlitz near Bremen on July 17, 1945. The N.C. was made from Woodpulp in conventional nitrating equipment but the stabilization process and equipment were quite different from those used in the U.S. The N.C. properties also were different.

The Paste was prepared for use either in Cannon or Rocket Powders. It was mixed in a water slurry, with some novel equipment at one plant. The information obtained will be of value in development work on solventless Rocket Powder in the U.S.
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</tbody>
</table>
1. Introduction.

German Cannon Powder and Rocket Powder was nearly all made from the basic intermediates, nitrocellulose (NC) and Diethylene glycol dinitrate (DEGN). These were mixed in a water slurry and filtered to about 35% water content to obtain the raw material termed Paste which was then transferred to the Powder or Rocket manufacturing areas for addition of the other ingredients, such as stabilizers, and for the further processing.

(a) Krummel

Krummel Works of the Dynamit A.G., was located about 3 miles from Dunoberg Works of the same firm. Dunoberg was the largest Cannon and Rocket Powder factory in Germany, with a total capacity of 3,000 metric tons (6,600,000 lbs.) of Powder per month. Krummel supplied the Paste for almost all of this production.

(b) Bomlitz

At Bomlitz, the Fibia, GmbH subsidiary of Wolff & Co., had built a large factory with facilities for both Paste and Powder for Cannon and Rockets. The total Paste capacity was equivalent to 2300 metric tons of Powder (5,060,000 lbs.) per month. This report covers manufacture of NC and Paste and reference should be made to the following separate Technical Reports:

1. Nitroglycerin and DEGN - No. 257 - 45
2. Cannon Powder - No. 259 - 45
3. Rocket Powder - No. 260 - 45

General information obtained concerning plant employment, auxiliary operations, etc., for Krummel and Bomlitz, has been included in this report in the Appendices.
Nitrocellulose Plant

The information in this section was obtained by translation of a report written by the Krummel Staff. (Translation by Lt. C. W. Reuss, USNR).

The Nitrocellulose plant at Krummel consisted of 2 units: the plants Heide and Abhang. The manufacturing process was the same in both. The difference was in capacity and, to a lesser degree, in the mechanical installations. The output of Heide amounted to ca. 400 tons per month and that of Abhang to ca. 900 tons per month.

(a) Raw Materials

The raw materials for the production of nitrocellulose were: bleached cellulose delivered in the form of crepe paper; the products of factories at Temming, Földmühle and Aschaffenburg were used, also the acids:

- HS-acid (88% HNO₃, 10% H₂SO₄)
- Conc. HNO₃ (99%)
- Oleum (20% SO₃ or 25% SO₃)

The acids were delivered in tank cars and pumped from the pumping station (buildings 257 and 705) into acid storage tanks of 60 cbm and 30 cbm respectively, in buildings 251, 622 and 723. The tanks were of iron except those for pure nitric acid which were aluminum.

<table>
<thead>
<tr>
<th>Storage room Heide</th>
<th>900 to</th>
<th>600</th>
</tr>
</thead>
<tbody>
<tr>
<td>HNO₃ or HS-Acid, resp.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oleum</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Storage room Abhang</th>
<th>800 to</th>
<th>600</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitric acid, or HS-acid, resp.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oleum</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Cellulose Storage

Cellulose was stored in buildings 245 and 703, each with storage capacity of 470 tons. Building 245 had only a rail-connection while building 703 had facilities for unloading from ships on the Elbe river.
2. Krummel (Cont'd)

Preparation of cellulose

Cellulose to be nitrated was torn to shreds by rotating rollers and then blown by exhausters through air conveyors (which could be heated) to the nitration house into large cellulose storage chambers. The heated conveyors dried the cellulose for nitration. The moisture content was in this way reduced from 6-7% to ca. 1-2%. In the plant Heide drying compartments were installed at the end of the conveyor on account of the short distance. The cellulose was conducted on belts through these drying compartments. Recently the drying was discontinued.

Manufacture of nitrating acid

The nitrating acid was manufactured by regeneration of waste acid, i.e., the waste acid after analysis was butted with concentrated nitric acid and oleum to the required mixed acid concentration. This was done in iron mixing tanks with built-in stirring apparatus.

In building 249 there were 6 such tanks of 60 cbm each, while in Abhang (buildings 724 and 725) there were 16 tanks of 30 cbm each.

The strong acids required for regeneration were measured in measuring vessels (vertical cylindrical containers). The required amount was obtained by means of floats in sight glasses.

The composition of the nitration acid for a nitro-cellulose-nitrogen of 11.25 - 11.50% consisted of 16-18% water, 20% HNO₃ and 64-62% H₂SO₄, and for a nitrogen content of 13.2-13.3% 9-10% water, 22.5% HNO₃ and 68.5-67.5% sulfuric acid.

The regenerated waste acid (nitration acid) was, before its use, tested again by chemical analysis.

Acid Fume Recovery

All acid storage tanks and measuring vessels were inter-connected by pipe lines for the purpose of drawing off acid vapours.
2. Krummel (Cont'd)

By means of fans these vapours together with the vapours emanating from the nitration house were blown into absorption towers, to which water was added to produce 50% nitric acid. The absorption towers in Abhang were made of V2A metal and were erected very close to the nitration houses, while in Heide the old ceramic towers were still in operation. In the latter the fans were after the towers so that the vapours were not blown into the towers but drawn through them. By means of centrifugal pumps the water was recirculated over the towers until the desired concentration of 50% had been obtained. Then this weak acid was pumped into storage tanks through V2A pipes or Mipolam pipes and either used up with the nitration acid or any excess was sent to the acid area for denitration.

Storage capacity for absorption tower acid

<table>
<thead>
<tr>
<th>Location</th>
<th>Capacity (tons)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heide</td>
<td>40 (building 251)</td>
</tr>
<tr>
<td>Abhang</td>
<td>240 (building 731, 732)</td>
</tr>
</tbody>
</table>

(b) Process and Equipment

(1) Nitration and Temperature Control

The stredded cellulose was weighed, loaded in cars and placed in nitration pots while at the same time the nitration acid was run into these pots. 25 kg. cellulose with ca. 6% moisture content were nitrated per charge. In the plant Heide the nitration charges were somewhat smaller on account of the smaller sized pots. The charge size was only 13 Kg. in the case of PE-wool and 11 Kg. in the case of S-wool. The nitration pots were provided with mechanical agitation. (See Photo No. 1 of Appendix).

The acid necessary for nitration was first brought to the required temperature in vertical cylindrical tanks. These tempering vessels contained cooling or heating coils, respectively and a good stirring apparatus. Volume of the containers was 6 cbm. The temperature was adjusted to 30 deg. C. For nitration a cellulose acid relation of 1 to 45 was maintained. The nitration time was 30 minutes.

Plant Heide: 24 nitration pots, each 0.5 cbm.
Plant Abhang: 32 nitration pots, each 0.7 cbm.
At the end of the nitration time the pots were drained into centrifuges and the waste acid was separated. This required 6 minutes at ca. 900 revolutions per minute. In the older plant Heide, 12 iron "standing" centrifuges with bottom drives were in use. Contents were removed at the top by means of large tongs. In the plant Abhang the modern V2A suspended drive centrifuges with bottom discharge were used. (See Photo No. 2 Appendix). The arrangement here was such that there was one centrifuge each for 4 pots while in Heide there was one centrifuge each for 2 pots. The N.C. slurry was run from the centrifuge in a V2A line with no trap in it.

The waste acid separated was returned to the acid storage for regeneration having first passed through rotating filter drums in order to remove any N.C., which was carried off with the waste acid. This N.C. was dropped from the slightly conical drums into water containers and was then added to the normally nitrated N.C. from the centrifuges.

Storage capacity for waste acid:

Heide: 9 tanks each 60 cbm.
Abhang: 12 tanks each 60 cbm.

All apparatus of the nitration house and all waste acid containers were provided with fume lines which were also connected by way of suction fans with the acid absorption system.

(2) Pre-Stabilization:

The nitro cellulose from the centrifuges was dropped into a stream of flowing water and then run to the next house into the so-called pre-washer; this was a container with an acid resisting lining and with mechanical agitation. Here the material was diluted, the larger lumps broken up, and finally pumped into stabilizers. These were tanks which in Heide consisted of wood and in Abhang of V2A metal. They had a double bottom of which the inner one had sieve-like holes. At the finish of the cooking the water could be discharged through a discharge pipe from the space between the two bottoms. Each cooker had 4 built-in shafts which
opened into the space between the two bottoms and into which the heating steam entered. The purpose of these shafts was to bring about circulation of the water in this way. The steam pulled the water up, threw it over the top of the charge and then sucked it in again below the sieve-bottom. The boiling was conducted with steam of 3 atm at atmospheric pressure and lasted 3 hours in the case of PE-wool and 6-8 hours in the case of S-wool.

**Boiling-tub room**

Heide: 16 pre-cookers, each 10 cbm (bldgs. 230)
Abhang: 24 " " 14 cbm (Bldgs. 708, 709)

The boiling tubes were emptied in Heide by hand by means of forks. In this process it was necessary that the N.C., after being boiled be washed at least two times with cold water. In Abhang the tubes were emptied through a bottom valve by means of a hose and the N.C. was thus washed into the pressure-cooking building.

**3. Pressure Cooking**

The pressure cooking served a two-fold purpose. First, it acted as a stabilization and then it served to bring about the correct viscosity. It was conducted in V2A autoclaves, partly with direct and partly with indirect steam, (heating steam 12 atm). In the interior of each cooker there was a stirring apparatus. The volume was 5 cbm, with a charge of ca. 450 kg. nitro cellulose. Up to a temperature of 100 degrees only indirect steam was used, over 100 degrees direct steam was introduced. The N.C. was heated to a temperature of 142 - 145 degrees C and then this temperature was maintained for 6 minutes. Thereupon the steam was cut off and the contents of the cooker were pumped through the bottom valve. Which maximum temperature should be used for a certain cooking process depended on the desired viscosity which could only be determined empirically. It depended on the mechanical peculiarity of the pressure cooking installation and was quite different in the various installations. Plant Heide had 3, plant Abhang 6, pressure cookers (bldgs. 236, 710, 711).

The viscosity, by the Hoppler method (Falling ball, 20°C) was determined in a 3% aceton solution. It was specified for each order.
2. Krummel (Cont'd)

(4) The Hollanders and Rotating Drum Filters

From the pressure cooker the material was pumped first into a vessel with a sieve-plate bottom where the pressure cooker water was removed. After washing with cold water in the same vessels the N.C. was transferred to the Hollanders. In Heide small cars were used for the transport, while in Abhang the de-watering vessels were placed so that the N.C. could be dropped directly into the Hollanders. Plant Heide had 6 Hollanders of an older construction (Voith-Hollander with 7.4 cbm content) and one modern Banning-Seybold Hollander of 14.5 cbm. Plant Abhang had 7 Banning-Hollenders, each 14.5 cbm.

1000 Kg. nitro cellulose (Voith-Hollander)
2400 Kg. " (Banning-Hollander)

At the start of charging 3-4 Kg. soda was added to the Hollander stock in order to neutralize the acid which would be freed from the nitrocellulose in the cutting process and thus protect the knives against corrosion. The pH-value of the Hollander liquor should be between 7 and 9. If need be, the soda addition was increased. The cutting time was ca. 6 hours. Thereupon the N.C. was drained off into a tank from which it was pumped to the rotating drum filters. This apparatus separated the perfectly cut fibres from the imperfectly cut ones. The separation took place in a vertically rotating sieve drum into which the N.C. was run, being diluted with enough water to permit the fine particles to go easily through the 0.4 mm holes of the sieve, while the coarse particles were removed from the sieve by built-in strippers. This coarse material was again conveyed into the Hollander and cut. Inasmuch as the separation takes place without difficulties only with a very great dilution of the stock, it was necessary to de-water the strongly diluted fine stock in rotating sieve drums (de-watering drums) i.e., to thicken it. The thickened N.C. now was ready for after-stabilizing while the dilution water was again conveyed back to the circulation.

Plant Heide: 2 Rotary drum filters.
Plant Abhang: 4 Rotary drum filters.
2. Krummel (Cont'd).

(5) After stabilization

The after stabilization was carried out in simple cylindrical tanks with mechanical agitation. Direct steam at atmospheric pressure was used for cooking. The cookers were either of iron (Abhang) or else they were tanks built of concrete and acid resisting brick (Heide). Volume of cookers in Abhang 14 cbm, in Heide 10 cbm.

Cooking room Heide, 14 cookers (bldg. 714)
Cooking room Abhang, 14 cookers (bldg. 714)

Nitrocellulose of low nitrogen content at the after-stabilization was heated only to cooking temperature, decanted and washed once with water. It was then, as a rule, stable according to the test by Bergmann-Junk (2 ccm NO/gr. N). Nitrocellulose of high nitrogen content on the other hand had to be cooked until stability was obtained. For this purpose samples were taken and tested at various time intervals (after 2, 3, 5, 8 hours and then after every 3 hours). In addition to the stability tests every after-cooking charge was tested for the exact nitrogen content. Depending on these tests the process was continued or stopped.

(6) Blending House

Corresponding to the nitrogen content of the individual after-cooker charges these were pumped together in large mixingvessels (built of masonry) of 100 cbm content and combined into nitro cellulose mixtures of a certain nitrogen content. About 7-9 cooker charges were combined in one blend. The homogenizing of the material was obtained in the mixers by means of strong agitation, i.e., either by means of fast moving propellor type agitators (plant Abhang) or by means of the slowly moving horizontal agitators (plant Heide.)

Blending Room:

Heide: 2 mixers, each 72 cbm.
Abhang: 4 mixers, each 105 cbm. (bldg. 716)

(7) De-Sanding

The blend in the mixer was then thinned by addition of a good deal of water, and conducted through the sand catchers where sandy impurities were separated. The sand catchers were round vessels.
2. Krummel (Cont'd)

conical at the bottom, into which the material entered at the bottom and because of the reduced velocity in the upper, wider area, heavier particles than the N.C. fibres were removed. The sand collected at the bottom of the vessel and could be removed through an opening there. Every de-sanding installation consisted of 3 individual sand catchers which were arranged in series. After de-sanding and before the N.C. reached the centrifuges for further de-watering, the material was thickened again in rotating de-watering drums, just as in the previous stage.

The plant Heide had one de-sanding unit and the plant Abhang had 3.

(3) De-Watering

For final de-watering of the nitro cellulose in the centrifuges there were so-called feed tanks from which the individual centrifuges were fed. These vessels were simply vats with agitators in which the thickened pulp was maintained in constant movement. From here the N.C. was run by gravity into centrifuges placed beneath where it was de-watered to a moisture content of 35-40%.

The plant Heide had 8 de-watering centrifuges which had belt drive with transmission from below 1000 revolutions per minute. There was also a shell centrifuge for continuous operation. It was emptied automatically by means of a shell knife. The filling and emptying took place periodically. The centrifuge drum was horizontal and had a direct motor drive.

In the plant Abhang the de-watering centrifuges were, like the acid centrifuges, suspended type centrifuges with bottom discharge. There were 8 of them and also a thrust centrifuge suitable for continuous operation. The latter had a moving drum, placed on a horizontal axle in which the sliding bottom was moved periodically to and from by means of oil regulation and pushed forward the dried material.

The nitrocellulose de-watered in the centrifuges was shovelled by hand into galvanized iron barrels and rammed down. The barrels were then weighed, marked and taken to the shipping place.
9. **Purification plant for waste water**

The waste water from the nitrocellulose operation had to be freed from the nitrocellulose in it prior to discharge into the Elbee. For this purpose it was conducted into ditches with a cross-sectional area large enough to permit the material carried in the water to settle.

In the plant Hoide all the purification ditches were built of masonry lined with plates, while in the plant Abhang there were in addition to such ditches, so-called "fume catchers" which were conical-shaped vessels with the tip pointing down. In these the water moved up, and, because of the enlargement of the cross-sectional area, caused such a large reduction of the flow that a very extensive purification of the waste water took place.

The plant Hoide had 3 purification ditches built of masonry and Abhang had 4 such ditches and 4 fume catchers.

The yield of NC, was stated as 160 to 165 lbs. of 13.37\% N\textsubscript{2} grade and 140 - 145 lbs. of 11.5\% N\textsubscript{2} grade from 100 lbs. of Cellulose. The pulp used was a mixture of about 85\% Beechwood and 15\% Pine. (End of translation).

(a) **The Manufacture of Paste - Krummel**

(Translation of Report by Krummel Staff)

The raw materials for Paste were nitrocellulose, and explosive oil. The manufacturing process depended on a good mixture of these two products.

The manufacture of explosive oil has been extensively described elsewhere in the report referred to above. The oil necessary for each mixture was weighed in the explosive oil storage in amounts required and stored in lead lined wooden boxes until needed by the paste mixing houses. It was then jetted through an injector as an oil-water-emulsion and mixed in the initial mixture vessels with nitrocellulose in a slurry.

The nitrocellulose (NC) delivered by the nitrocellulose factories in tightly closed iron barrels had to be sieved before it was used in the manufacture of pastes. In this way it was loosened and a good sample
could be taken to determine its moisture content. The moisture content of NC fluctuated as a rule between 35-40%. The determination of the moisture content was made in the laboratory by drying over night at 45 degrees.

The NC blends employed consisted of 2 components: gun cotton with a nitrogen content of 13.15 - 13.25% and one of low percentage with a nitrogen content of 11.30 - 11.45%. From these two types all necessary mixtures could be made. The main NC mixtures possessed the following nitrogen content: 11.50%, 11.90%, 12.20%, 12.50%, 12.75%, 13%. According to the amount of nitrogen content either the high or low percentage portion predominated.

The individual mixture had an average dry weight of ca. 10-12 tons. Each mixture was separately stored in boxes and after its moisture content had been determined, released for manufacture.

The size of an initial mixture in the paste-mixing houses amounted throughout to 400 kg, dry weight. For a 30% paste there was thus the following initial mixture:

\[
\begin{align*}
&280 \, \text{kg}, \quad \text{NC dry} \\
&120 \, \text{kg}, \quad \text{Explosive oil} \\
&400 \, \text{kg}, \quad \text{paste dry}
\end{align*}
\]

The NC, weighed in rubber lined sacks, was transported from the NC storage to the mixing houses by means of electrical trucks. It was then transferred into 3cbm mixing vessels into which water had been placed. For about 10 minutes it was stirred and then explosive oil was added, as already mentioned, as an emulsion. The mixture was again stirred for 10 minutes. The initial mixture was now ready. It was drained off into centrifuges where the paste was reduced to ca. 35% moisture. The centrifuges had a bottom outlet and thus the contents could be taken off into sacks lined with rubber by merely holding the sacks under the centrifuges. These rubber lined sacks were inserted into linen sacks, and transported to the paste storage by means of electrical trucks, and then shipped.

The water from the slurry was collected in purification vats and re-used for other initial mixtures. The initial mixture ratio was roughly 1:4 with respect to the dry weight, of the paste initial mixture. The output of a paste mixing house was stated as 48 tons per day.
2. **Krummel (Cont'd)**

Communication between the mixing houses and the explosive oil storage from which the weighed explosive oil was taken was by means of light signals. That which has been described above was equally true in the manufacture of paste using nitroglycerine, diglycol dinitrate, triglycol dinitrate, and methylo dinitrate. The control test of the finished product was done by extraction of the explosive oil by means of ether and by re-weighing of the dry nitro-cellulose.

The composition of the paste was specified by the Duneberg plant orders. A representative list of the various types required is given in the following table:

<table>
<thead>
<tr>
<th>Block-Nr.</th>
<th>Paste</th>
<th>N2</th>
<th>Beech</th>
<th>Pine</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>11 - 999</td>
<td></td>
<td>40.0% Ngl.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1001 - 1999</td>
<td></td>
<td>28.0% Ngl.</td>
<td>12.75</td>
<td>-</td>
<td>100</td>
</tr>
<tr>
<td>2501 - 2999</td>
<td></td>
<td>31.7% Ngl.</td>
<td>11.45</td>
<td>-</td>
<td>100</td>
</tr>
<tr>
<td>3001 - 3999</td>
<td></td>
<td>30.0% Digl.</td>
<td>13.45</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>4001 - 4999</td>
<td></td>
<td>45.0% Ngl.</td>
<td>12.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>5001 - 5999</td>
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<td>35.0% Digl.</td>
<td>13.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>6001 - 6999</td>
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<td>36.0% Digl.</td>
<td>12.90</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>7001 - 7999</td>
<td></td>
<td>18.0% Methr.</td>
<td>13.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>8001 - 8999</td>
<td></td>
<td>17.0% Trigl.</td>
<td>12.65</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>9001 - 999</td>
<td></td>
<td>26.7% Digl.</td>
<td>12.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>1001 - 10999</td>
<td></td>
<td>37.2% Digl.</td>
<td>13.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>11001 - 11999</td>
<td></td>
<td>18.0% Methr.</td>
<td>11.75</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>12001 - 12999</td>
<td></td>
<td>17.0% Trigl.</td>
<td>12.45</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>13001 - 13999</td>
<td></td>
<td>26.7% Digl.</td>
<td>12.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>14001 - 14999</td>
<td></td>
<td>50.0% Digl.</td>
<td>13.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>15001 - 15999</td>
<td></td>
<td>20.0% Digl.</td>
<td>13.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>16001 - 16999</td>
<td></td>
<td>35.6% Digl.</td>
<td>12.25</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>17001 - 17999</td>
<td></td>
<td>45.0% Digl.</td>
<td>13.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>18001 - 18999</td>
<td></td>
<td>26.7% Digl.</td>
<td>12.00</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>19001 - 19999</td>
<td></td>
<td>45.0% Digl.</td>
<td>12.80</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>20001 - 20999</td>
<td></td>
<td>26.7% Digl.</td>
<td>11.45</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>21001 - 21999</td>
<td></td>
<td>18.0% Methr.</td>
<td>12.65</td>
<td>85</td>
<td>15</td>
</tr>
</tbody>
</table>
The N,C, operation at Bomlitz was carried on in two identical lines, each consisting of the following buildings:

1. Pulp Shredder and Dryer,
2. Nitrator House,
3. Boiling or Pre-Stabilization House,
4. Hollander House for Comminuting,
5. Pressure Cooking and Final Stabilization,

(b) N,C, Process and Equipment

The raw material, cellulose, was creped paper made from a mixture of beech and fir or pine wood pulp.

Nearly all details of operation and equipment for N,C, at Bomlitz are identical to those already described for Krummel and to eliminate repetition, only the points where differences were observed are reported here.

There were 24 nitrator dipping pots and six centrifuges in each house. The charge size was 23 Kg. of cellulose which was nitrated at 20 deg. C for 30 minutes. The charge size was set by the maximum capacity of centrifuges. This equipment had no bottom outlet.
3. **Domlitz (Cont'd)**

and it was necessary to lift the acid N.C. out with tongs and then drop it down a chute for drowning it in a stream of water. This slurry ran by gravity to the boiling tub house.

Either high Nitrogen (13.2 - 13.3% N₂) Schiesswolle or low Nitrogen (11.4 - 11.7% N₂) P.E. was made. The Schiesswolle was boiled for 8 to 10 hours in the TZA lined tubs, then sent to the Hollander house for reduction to the proper particle size. The P.E. was boiled for a shorter time. The final N.C. was tested by the Bergman-Junk test.

The Hollander had vertical beater wheels, one rotor and one stator.

After the Hollander, the N.C. was sent to the pressure cooker and further stabilized under 37 lbs. steam pressure - the 13.3% N₂ for 1½ to 2 hours and the 11.5% N₂ N.C. for ½ to 1 hour.

The material was then blown to the final stabilizing or poaching tanks. Depending on the quality, it was boiled for periods varying from 4 to 12 hours at 90 - 100 degrees C.

The two grades of N.C. were then mixed in the proper proportion to obtain the desired 12.2% N₂ in a blender with a capacity of 15,000 Kg. of N.C.

The seven blends of N.C. Prepared at Domlitz for use in manufacturing solventless powders were as follows: (Quoted from U.S. Army Ordnance interrogation)

<table>
<thead>
<tr>
<th>TYPE</th>
<th>COMPOSITION</th>
<th>NITROGEN CONTENT</th>
<th>VISCOSITY</th>
</tr>
</thead>
<tbody>
<tr>
<td>D</td>
<td>70%</td>
<td>13.15 - 13.25%</td>
<td>30 - 45 seconds</td>
</tr>
<tr>
<td></td>
<td>30%</td>
<td>11.0 - 11.5</td>
<td>10 - 15 seconds</td>
</tr>
<tr>
<td></td>
<td></td>
<td>12.6 - 12.7</td>
<td>20 - 35</td>
</tr>
<tr>
<td>D</td>
<td>50%</td>
<td>13.2 - 13.3</td>
<td>10 - 15 seconds</td>
</tr>
<tr>
<td></td>
<td>50%</td>
<td>11.0 - 11.5</td>
<td>10 - 15 seconds</td>
</tr>
<tr>
<td></td>
<td></td>
<td>12.2 - 12.3</td>
<td>10 - 15</td>
</tr>
<tr>
<td>DO-7R</td>
<td>50%</td>
<td>13.2 - 13.3%</td>
<td>20 - 30 seconds</td>
</tr>
<tr>
<td></td>
<td>50%</td>
<td>11.0 - 11.5</td>
<td>10 - 20 seconds</td>
</tr>
<tr>
<td></td>
<td></td>
<td>12.2 - 12.3</td>
<td>15 - 25</td>
</tr>
</tbody>
</table>

-16-
3. Domlitz (Cont'd)

<table>
<thead>
<tr>
<th>TYPE</th>
<th>COMPOSITION</th>
<th>NITROGEN CONTENT</th>
<th>VIScosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-50</td>
<td>30 - 35%</td>
<td>13.2 - 13.3%</td>
<td>40 - 60 sec</td>
</tr>
<tr>
<td></td>
<td>65 - 70%</td>
<td>11.0 - 11.5%</td>
<td>10 - 15 sec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>11.8 - 11.9%</td>
<td>15 - 25 sec</td>
</tr>
<tr>
<td>W-1</td>
<td>90%</td>
<td>13.2 - 13.3%</td>
<td>20 - 30 sec</td>
</tr>
<tr>
<td></td>
<td>10%</td>
<td>11.0 - 11.5%</td>
<td>10 - 15 sec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>13.1</td>
<td>15 - 20 sec</td>
</tr>
<tr>
<td>R-1</td>
<td>55%</td>
<td>13.1 - 13.3%</td>
<td>about 30 sec</td>
</tr>
<tr>
<td></td>
<td>45%</td>
<td>11.0 - 11.5%</td>
<td>about 20 sec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>12.4</td>
<td>about 25 sec</td>
</tr>
<tr>
<td>D-60</td>
<td>Same as W-1 but with viscosity of 40 - 60 sec</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

There were two systems at Domlitz for transferring the N.C. to the Paste mixers. With the older one, the N.C. was centrifuged to a water content of 30 -35%, loaded into bags, and transferred to the Paste Area.

In the new equipment the blend was made with water pumped back from the Paste filter. The mix of N.C., with about 10% N.C. in the slurry, was pumped to the new Paste mixer.

(c) Paste - Process and Equipment

In line with each of the 5 small Schmidt DEGN nitrators there was one Paste mixing house. For the large Schmidt nitrator there was a new very large mixing house.

Each small mixing house had two rooms on the upper level where four, 1000 litre V2A mixers were located. On the lower level there was one centrifuge for de-watering the slurry from each set of 4 mixers.

The mixers were first charged with water, then the proper number of bags of N.C. were dumped and then the DEGN was run in to the slurry. The temperature of the charge was maintained at 25-30 deg. C, by 80 deg. C water in the jacket of the mixer. A slurry containing 20% solids was made in this way with a charge of 100 kg., dry weight, of Paste.

It required 20 minutes per mixer cycle. The mixed slurry passed over a magnetic separator as it was run to the centrifuge which held just one
3. Domlitz (Cont'd)

mixture charge. The Paste was forked out the bottom opening of the centrifuge, fell through a rotating drum for breaking the cakes, and blending, into a hopper. From this the Paste, containing 35% water was loaded into bags for transfer to the Powder Area.

Nearly all Paste made at Domlitz was either the 20% DEGN grade or the 35% DEGN grade. Details of composition are described in the report of Powder Manufacture. (Technical Report 239-45).

In the new Domlitz Paste mixing house, there were two large steel mixer tanks each with a capacity of 10 m³ tons (22000 lbs) dry weight of Paste. These had a volume of 100 cubic meters and were 4 meters in diameter. The agitation was provided by a 60 kW motor. The vertical shaft was supported in a water cooled foot bearing.

The N.C. was first charged as a 10% slurry pumped from the N.C. Area. Then the DEGN emulsified in water was added over a period of 2 hours. The final slurry contained 10% solids. The charge was agitated then for a further 2 hours. The outlet valve in the conical bottom of the mixer was opened and the whole charge was pumped 500 meters to the Filter House. A centrifugal cast steel pump with a water lantern stuffing box was used for this purpose. In the line there was a booster pump of the same type half way to the filter house.

At the Filter House there were two vertical steel tanks for storage of Paste slurry. The slurry as received contained 10% solids. This was continuously mixed with filtrate water to make a 2% slurry for feed to the filter. The filter was a rotary vacuum drum. It had a 4–500 mm vacuum for cake pick up and a 200 mm vacuum for de-watering. The cake was covered by a continuous blanket for assistance in de-watering. A cake 2 cm in thickness was obtained with the water varying from 35 to 37% at times. The system had been in use for one year.

From the filter the paste was transferred by a screw conveyor, to break the lumps, to the charging hopper where the bags were filled for transfer to the Powder Area.
Krummel Works was built as the first dynamite plant in the world in 1865 by Alfred Nobel. His company later became the Dynamit A.G., the present owners. The following operations are carried on at this plant:

1. Sulfuric Acid Concentration - Pauling process.
2. Nitric Acid Concentration - Pauling Process and Pre Concentration.
3. PETN - 150 - 180 M. tons/mo.
4. Oleum Manufacture - 800 M tons/mo. from Pyrites.
5. Nitro-Cellulose - 1350 M. tons/mo.
6. Hexogen - RDX - W - Salt Process - Plant destroyed, 80-100 M tons/mo.
8. TNT - From Toluene - 3000 M tons/mo.
11. Nitroglycerin and DEGW Nitrators (4 lines) 2000 M tons/mo.
12. Paste Plant - 3000 M. tons/mo.
13. RDX dry House Line.

The Nitric Acid used at Krummel was shipped from other plants, principally Embsen. It was received as 99% Acid and as a Mixed Acid containing 90% Nitric and 10% Sulfuric.

Although Schmidt made his invention of the continuous N.G. nitrator at Krummel, there are none of these units in operation there. Only the bath nitration equipment is used.

The only development work being done at this plant was in the field of Shell loading. Any research in connection with Powder was conducted at Dumeberg where all Paste made at Krummel was shipped.
The maximum employment at Krummel was 9500 workers, of which from 40 to 50% were foreigners.

Mr. James Neale, Plant Manager, was the grandson of one of the English partners of Alfred Nobel. Although he had been jailed by the Nazis, he was later returned to his position. Mr. Neale had a description of NC and Paste manufacture prepared for this team. This has been translated and included in the detailed section.

The Sulfuric Acid Concentrating equipment was the conventional Pauling Retort system used in Germany. This has been described in the report on TNT (Technical Report No. 160-45) and those details have not been repeated. The Pauling equipment for the concentration of Nitric Acid was also described in that report.

The Oleum for TNT was made from Pyrites in a conventional contact plant.

The RDX Plant at Krummel is described in the report on that subject (Technical Report No. 262-45). Also described in other reports are the remaining subjects listed above with the exception of the Plastic line and the Commercial Dynamite line which were not investigated.

APPENDIX II

GENERAL INFORMATION ON DOMLITZ WORKS

At Domlitz near Walsrode, about 50 miles east of Dremen, the firm of Wolff Co. had its headquarters and main plants. This company was about the third largest explosives manufacturer in Germany. The Wolff family had started making Black Powder in the early 1800s and later went into the NC business which led to manufacture of propellant explosives.

There were three present-owners, members of the Wolff family, Gerd and Hans, brothers, and their cousin Gunther. During peacetime, they operated a plant at Domlitz for making cellulose products such as sausage casings. In 1935 they started the construction of the
the three plants they operated for propellant powder. These facilities were built with funds advanced by the government-owned firm named Montan A.G. Wolff & Co organized what they termed a "daughter" firm named "Eibia" with title to the new plants and equipment. The three plants were located at Domlitz, Liebenau and Dorverden. Employment at Domlitz reached about 4000, at Liebenau it was 3000 and at Dorverden about 900. Domlitz was the only plant of this firm visited by this team. It had produced 2000 M tons per month of Diglycol powder and 300 M tons per month of N.C. powder. Liebenau had produced 2000 M tons/month and was installing an additional 2000 tons capacity. Dorverden made only 600 M tons/month of N.C. Powder.

From the total German propellant explosives production figures furnished, it has been estimated that the following division was made:

<table>
<thead>
<tr>
<th>Firm</th>
<th>Powder Capacity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dynamit A.G. (DAG)</td>
<td>4,500</td>
</tr>
<tr>
<td>Westfalische Anhaltische Sprengstoffe A.G. (WASAG)</td>
<td>16,600</td>
</tr>
<tr>
<td>and any other smaller firms such as Wolff &amp; Co, and Eibia</td>
<td>6,900</td>
</tr>
<tr>
<td>Total</td>
<td>28,000 M Tons/mo</td>
</tr>
</tbody>
</table>

While the above estimate agrees with other reported figures, the WASAG representatives have not been available to this team for interrogation.

The Bomlitz plant of Eibia was located in a dense coniferous forest about 2 miles from the Wolff & Co. commercial plant. The plant layout was similar to the DAG plant at Krummel but cost of building construction and roads and expenditures for camouflage appeared to have been much greater at Bomlitz. It was stated to be 15% more expensive (By DAG).
General Information on Bomlitz Works (Cont'd)

The major operations at Eibia Bomlitz were as follows:

1. Sulfuric Acid Concentration - Pauling System.
2. Nitric Acid Concentration and Waste Acid Denitrification - Pauling System.
3. DEGN Plants - 6 Schmidt Continuous Units.
4. N.C. - 2 Units.
5. Paste Mixing Units.
6. Rolling and Pressing Units for Cannon and Rocket Powders.
7. Proving Grounds for Cannon and Rocket Powder (located at Loverschen, 10 km away).

The nitric acid used at Bomlitz was shipped there from Embson, (Technical Report No. 484-45) the I.G. plant. The sulfuric acid required for nitration of Diglydol was received as 60% Oleum while the N.C. area received 27% Oleum to make up losses.

Gunther Wolff was Director (Manager) of the Eibia Bomlitz Works. The three main operating area superintendents were as follows:

Acid and DEGN - Dr. Werner Schmedding
N.C. - Dr. Sprengmann
Powder & Rockets - Dr. Stankiewicz.

These men were responsible for operations and also development work in their fields. Extensive research was not conducted by Eibia, rather they adopted the standardized compositions and procedures developed by the Duneberg staff of D.A.G in collaboration with German Army authorities.

Prepared by:
C. H. Brooks
Lt. Cdr., USNR
G. H. Neff
Lt., USNR
C. W. Reuss
Lt., USNR
T. L. Miller
Lt., USNR
W. E. Lawson, Dr.
J. B. Castmer, Technician
Photo No. 1. N.C. Nitrators - Krummel.
Photo No. 2. N.C. Centrifuges - Krummel.
Schema der PR-Herstellung