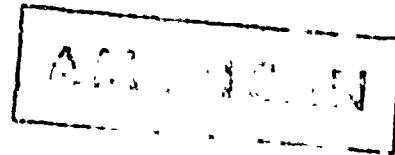


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TECHNICAL REPORT NO. 357-45

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

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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.

SEPTEMBER 1945

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TABLE OF CONTENTS

|                                                 | <u>Page</u> |
|-------------------------------------------------|-------------|
| 1. Introduction.                                | 3,4         |
| 2. Schlebusch.                                  | 4-10        |
| (a) Batch Process                               | 4,5         |
| (b) Schmidt-Meissner Equipment                  | 5           |
| (c) Biazzi Equipment                            | 5-10        |
| 3. Krummel.                                     | 10-20       |
| (a) Denitration of Explosive Oil-<br>Waste Acid | 17-20       |
| 4. Bomlitz.                                     | 20-25       |

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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.

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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 ESON 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.

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Schlebusch. (a)(Cont'd.)

The nitrator charge was 200 kg of Glycerin and 990 kg of mixed acid containing 51-52%  $\text{HNO}_3$  and 51-52%  $\text{H}_2\text{SO}_4$ .

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.

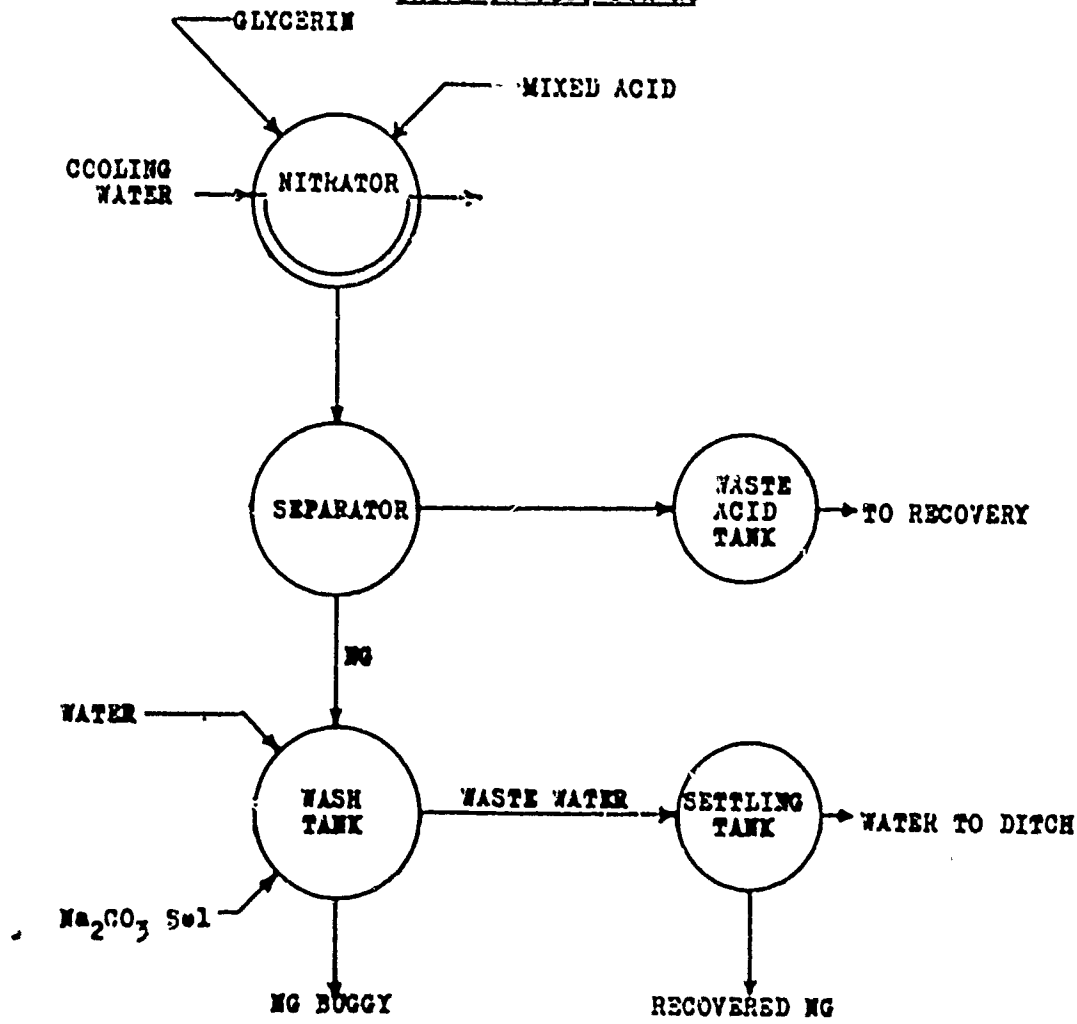
The equipment sequence and sizes for this process are shown in the attached diagram. A picture of the nitrator is also shown. All of it was located in the one house except the final N.G. separator and the wash water setting tanks.

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 flow-meter. The mixed acid was added through similar equipment. The analysis of the mixed acid was 51-52%  $\text{HNO}_3$  and 51-52%  $\text{H}_2\text{SO}_4$ . 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

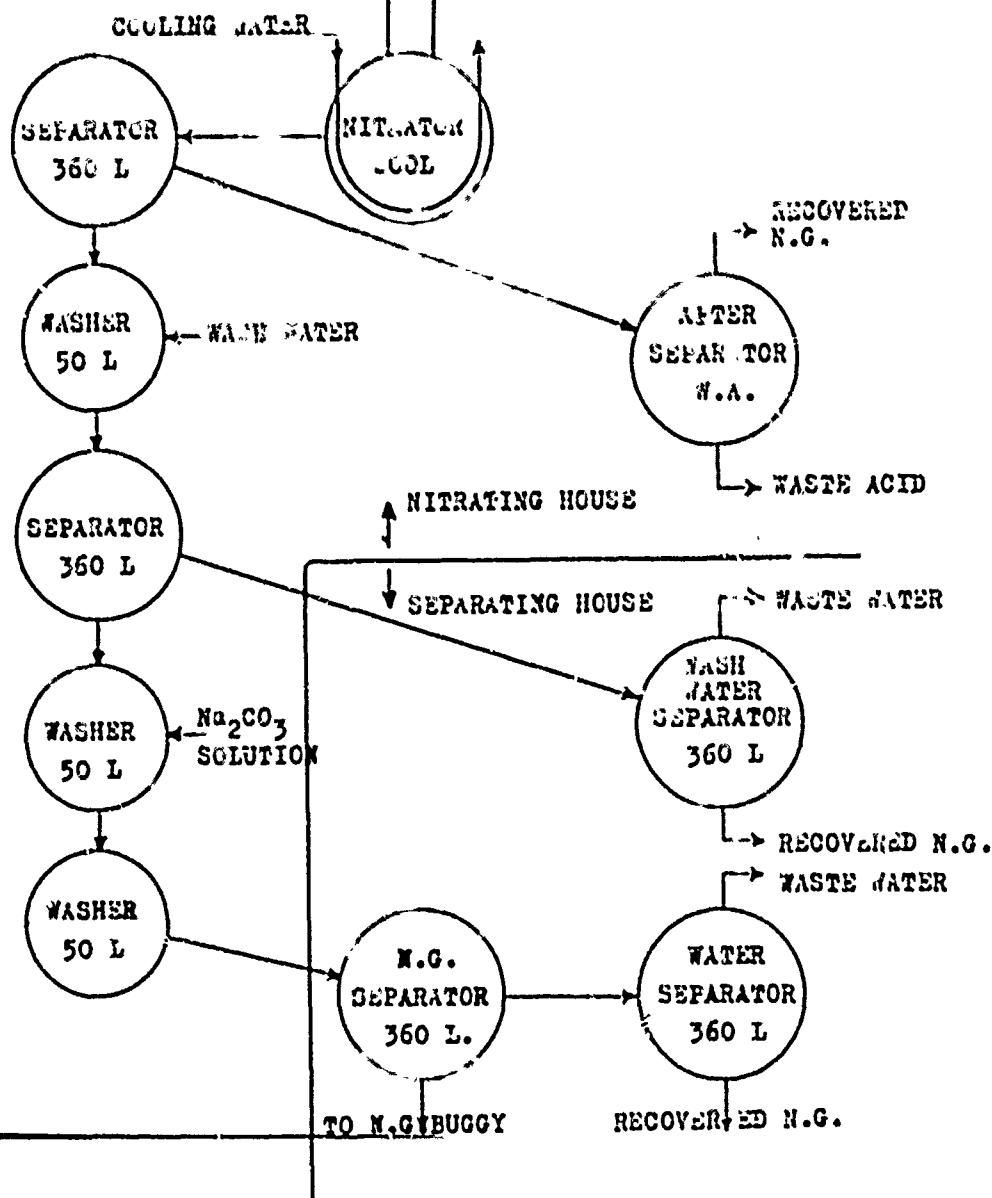
SCHLESBUSH  
BATCH PROCESS FOR NG



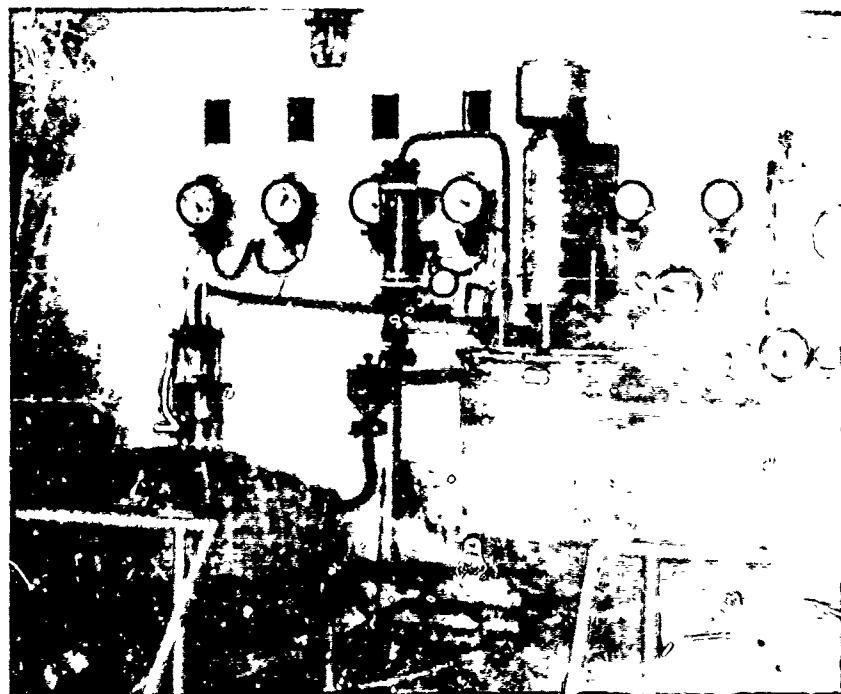
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BLAZZI PATENT  
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GLYCOLIN → H<sub>2</sub>O →



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PHOTOGRAPH OF SCHLEBUSCH-HIG-BIAZZEL  
NITRATOR

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Schlobusch (c)(Cont'd.)

per hour. With a volume of 200 liters in the nitrator, it was stated that there was about 80 kg of  $\text{H}_2\text{O}$  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.

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Schlebusch (a)(Cont'd.)

The separated oil was run out the trap and through a line to a third building where it was collected in a buggy, or a tank on wheels, for transfer to storage tanks.

The soda wash water was run to another separator and then to the ditch. Any oil recovered here was returned to the system.

The standard operating procedure required daily draining of the whole system. The nitrator and separator were then flushed out thoroughly with a synthetic waste acid (mixed acid of the same composition but no dissolved NG) to remove all NG. Due to the simple unobstructed passages and piping in the system, very good cleaning was obtained. The remainder of the equipment was washed by running water through it. At shut down, the synthetic waste acid remained in the first two vessels and the rest of the equipment was filled with water.

The unit was started the next day by addition of the Glycerin and mixed acid, first at reduced rate and then at normal rate. The agitation speeds and the cooling capacity, once established, required very little further adjustment.

It was stated that a yield of 334 lbs. of NG was obtained from 100 lbs. of glycerin -- a conventional yield.

The Biazzi unit was completed in 1942. During the first six months of operation some small alterations to piping were made but since then the Schlebusch staff has considered this process and equipment to be most satisfactory.

Soon after the start of operations, there occurred one small accident when a slight explosion ripped open a piece of the waste acid line from the first separator to the after separator. This occurred on a Sunday afternoon while the unit was shut down and damage was negligible. It was due to a small trap in the line which had not washed clean. This line was straightened and no further accidents have occurred.

The above information was obtained from Dr. Demoff, Acting Plant Manager, and Dr. Von Sommerfeld the NG Area Superintendent. The latter is considered by other German NG technicians to be one of the present leading authorities in this field. While at Schlebusch a vis-

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Schlebusch (c)(Cont'd.)

it was paid to Dr. Ph. Naoum, former head of DAG's laboratory, now retired. He was the author of the world-renowned book on Nitroglycerin and is considered to be one of the world's authorities on high explosive manufacture. Dr. Naoum has not been active since 1939 when he lost his leg in an explosion of an experimental unit.

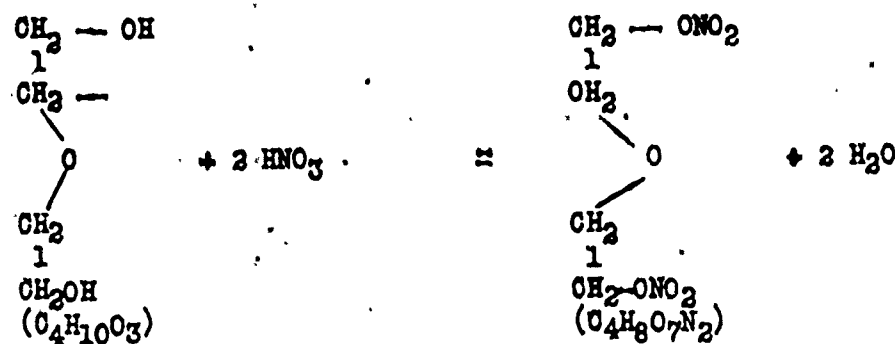
3. Krummel

A very thorough description of the Krummel NG operations was prepared by the staff at that plant. A translation of this report is as follows:

Technical and Manufacturing Processes of Diglycoldinitrate - Triglycoldinitrate and Methrioltrinitrate.

The explosive oils necessary for the production of powder paste were made in Krummel in 2 plants. In the older plant (during the last few years only Triglycoldinitrate was made here) nitration and washing was conducted in separate buildings. In the new plant the complete process from nitration to washing was carried out in one building. The new plant had two nitration houses. Here Diglycoldinitrate was made exclusively. For the Methrioltrinitrate a trial plant was installed in one nitration house. Production on a large scale such as in the case of Diglycoldinitrate had not yet been carried out.

The production of Diglycoldinitrate from diglycol was by means of esterification with nitric acid according to the following equation:



Since esterification is an equilibrium reaction, for nitration a pure nitric acid was not used but a mixture of 65%  $\text{HNO}_3$  and 35%  $\text{H}_2\text{SO}_4$ .

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Krummel (Cont'd.)

The sulfuric acid combined with the resulting water and thus shifted the balance of the reaction toward the ester side.

In Krummel only the discontinuous or batch process was employed. Charges of 420 Kg. diglycol were used. The average yield of Diglycoldinitrate was ca. 710-715 kg per charge; i.e., in round figures 170%. Theoretically 777 kg Diglycoldinitrate should be obtained from 420 kg diglycol. Part of the loss of explosive oil was in the waste acid, and also a smaller part was lost by washing.

Diglycol is a liquid clear as water and has a specific gravity of 1.126. As a rule it contained ca. 0.1% moisture and up to 1% glycol. The nitration was carried out by slow addition of diglycol to the nitration acid with intensive air stirring and good cooling with brine. The latter was a 35% sodium nitrate solution which was pumped at a temperature of  $-15^{\circ}$  through the cooling jackets.

As already mentioned, a mixed acid was used having the following composition:



The acid should be as free from nitroso as possible. It was obtained either ready mixed from the I.G. or else mixed in Krummel from 26% fuming sulfuric acid and 98-99% nitric acid or HS acid which contained 88%  $\text{HNO}_3$  and 11%  $\text{H}_2\text{SO}_4$ . The initial ratio was 1:2.9 i.e. for 420 kg diglycol 1218 kg mixed acid was used. Of that ca. 790 kg was  $\text{HNO}_3$  and 428 kg  $\text{H}_2\text{SO}_4$ . Theoretically only 497 kg  $\text{HNO}_3$  are necessary. Thus an excess of 293 kg was used. By this relatively high excess a higher stability of the resulting waste acid was obtained. Therein lies the great difference between the waste acid of diglycol dinitrate and nitroglycerin. While the NG acid lasts for days and could be "stored" in the after-separation vessels the diglycol waste acid had to be processed immediately because it had a tendency after several hours to decompose. At the same time the temperature rose rapidly and large quantities of nitrous gases escaped, emanating from the overheated ester.

The maximum temperature of nitration was  $25^{\circ} \text{C}$ . Two thermometers each with a different dip-depth made it possible to watch over the temperature in the nitration apparatus. The nitration with a cooling

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Krummel (Cont'd.)

brine of  $-14^{\circ}$  to  $-15^{\circ}$  lasted ca. 22-25 minutes. After nitration the explosive oil acid mixture was cooled down to  $-15^{\circ}$  and then drained into a separator. Here the heavier waste acid separated from the light acid oil. The separation lasted 7 minutes. The clearly separated waste acid was drained off for denitration, the acid oil run into the preliminary washer in which there were ca. 300 liter water, and a wash water consisting of ca. 34%  $\text{HNO}_3$ , 5%  $\text{H}_2\text{SO}_4$ , 2-3% ester, remainder water resulted. This washwater was also used in the denitration. The waste acid just mentioned had the following composition:

64-66%  $\text{H}_2\text{SO}_4$   
8-9 %  $\text{HNO}_3$   
4-5 % ester, rest water.

From the preliminary washer the explosive oil then was run into the final washing apparatus. There first, washing with ca. 500 liters cold water was used. Then with 150 liters ca. 5% soda solution, heated to  $60^{\circ}$ . The soda wash lasted 10 minutes with intensive stir-stirring. The final washing was made with ca. 500 liters cold water. From this washed oil a sample was taken to the laboratory. The test (reaction to potassium iodide-starch paper) must last at  $82^{\circ}$  at least 20 minutes. If the test was satisfactory, the oil was then released for the manufacture of paste.

The Diglycoldinitrate is a yellowish oil with a spec. gravity of 1.38 - 1.39 and a nitrogen content of 14.1 - 14.2%. The decomposition point is  $162^{\circ}$ . At  $300^{\circ}$  it explodes. Sensitivity to impact by a 2 kg drop hammer amounts to 150 cm. Diglycoldinitrate is in this respect, compared to nitroglycerin (with 2 kg drop hammer only 4 cm), considerably less sensitive. The freezing point is below  $-10^{\circ}$ . The water solubility is ca. 0.4%.

As to the nitration plant itself, all buildings were massive reinforced concrete structures which were surrounded by earth barricades. The front side of these buildings had a large window meant to be a blow-out wall. In the case of an explosion it would fall toward the outside and thus would make an escape route for the high pressure. Each building had two escape corridors, one at the upper exit and one at the lower exit. In the building were the nitration apparatus, separator, preliminary washer and final washer, all made from V2A. The required cocks were of clay, and partly armor-plated. The drowning

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Krummel (Cont'd.)

tank (safety vat) was of iron lined inside with rubber. It was large enough to receive the whole charge from the nitration or separation apparatus with a goodly amount of dilution with water. As the name indicates, it was installed in order to prevent the charge from exploding in case decomposition sets in by means of strong dilution. If during nitration or even afterwards an abnormal rise in temperature was noted, then the operator would immediately pull the emergency lever. In this way, the automatic safety installation was put in action. By means of the the operating air which was present in the air mains—

- (1) Bell signals were switched on by way of a relay in and on top of the building, also in the adjacent buildings,
- (2) By way of a so-called gun (kanone) the cocks of the nitration apparatus and the separator were opened towards the drowing tank,
- (3) A membrane valve was opened, permitting fresh water to flow, for the purpose of dilution, into the safety vat, and,
- (4) The air-stirring in the separator and safety vat was started. In case of disturbances in the plant air each nitrating house had a reserve air vessel which could be switched over in order to finish the charge which was in progress.

Communication between the houses which belong together was carried out by means of signal installations, e.g., between the weighing house and the nitrating house, between the nitrating house and oil storage, between nitrating house and denitration. Only after confirmation of a light signal by a return signal could operations in question be resumed.

The capacity of the entire nitration plants for regular operations was stated as 60 tons per day or 1500 per month, on the basis of continuous 3 shifts and based on 25 working days. For this 25 operators and 1 foreman were required.

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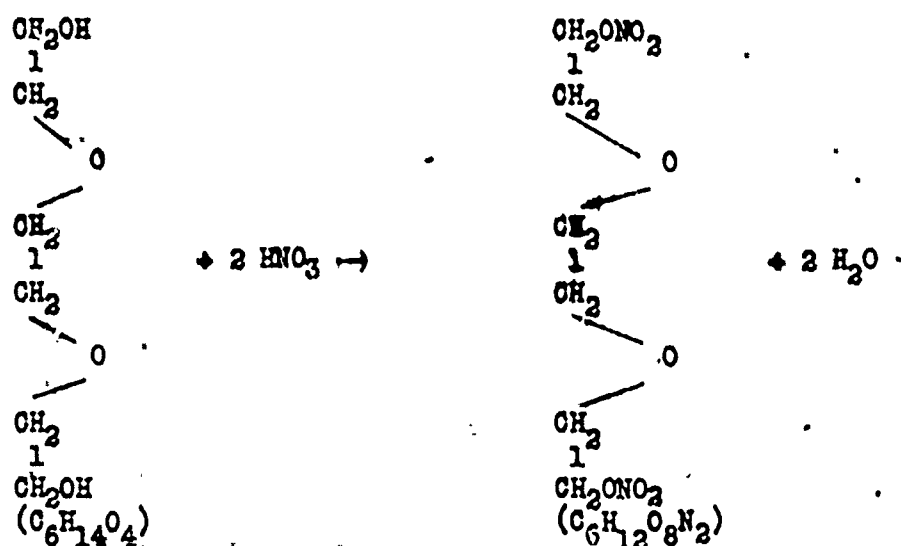
In the nitration of triglycol a mixed acid composed of 70%  $\text{HNO}_3$  and 30%  $\text{H}_2\text{SO}_4$  was used. The initial ratio was 1:2.5, i.e., for 500 kg

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Krummel (Cont'd.)

triglycol 1250 kg mixed acid was used. The yield amounted to roughly 650 kg with the above mentioned initial mixture. The solubility of the triglycol dinitrate in the waste acid was very high, amounting to ca. 8-9%. The denitration of this waste acid was difficult on account of the high explosive oil content. Inasmuch as the separation which lasted 30 minutes and more, was subject to certain dangers the oil-acid mixture was diluted after nitration and this acid water was then processed in the denitration. In this manner the acid losses remained within the usual limits.

The manufacture of the triglycol dinitrate proceeded according to the following equation:



Basically the manufacture of triglycol dinitrate was the same as that of diglycol dinitrate, with this difference:

Nitration time amounted to roughly 30 minutes. After 2 washings with water a soda wash with 30 kg soda per initial mixture takes place, thereupon the usual after-wash.

The triglycol dinitrate is a brownish oily liquid with a specific gravity of 1.335. The nitrogen content is 12.1-12.2%. Theoretically it should be only 11.7%. This difference is explained by the diglycol content of the triglycol amounting up to 2%. The sensitive-

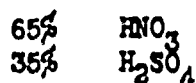
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Krummel (Cont'd.)

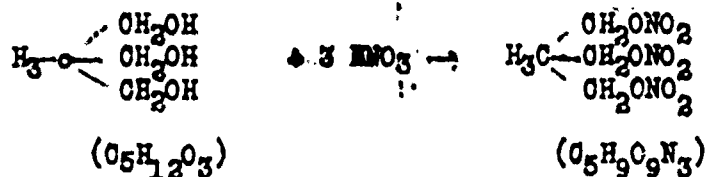
ness to impact of the triglycol dinitrate is still less than that of diglycol dinitrate. A test with the 2 kg fall hammer showed no effect. The iodide starch paper test should last 20 minutes at 82 degrees.

The capacity of the nitration plants for the manufacture of triglycol dinitrate was stated to be 44 tons per day.

In addition to diglycol dinitrate and triglycol dinitrate occasionally smaller amounts of methriol trinitrate were also manufactured. For this nitration the same mixed acid was used as for diglycol dinitrate, thus



The initial mixture ratio was 1:3.5. The formation of explosive oil takes place according to the following equation:



The average yield was ca. 200%. The nitrogen content of the explosive oil theoretically amounts to 16.47%, the actual values were between 16.32 - 16.36%.

While the methriol is a white, powdery material with a moisture content of ca. 0.5 - 1%, the ester obtained from it is an oily, slightly cloudy liquid whose density is 1.460 at 20 degrees. The decomposition of the explosive oil starts at 182°. The sensitivity to impact corresponds to that of nitroglycerin: 2 kg fall hammer 4 cm height. The oil is practically insoluble in water. The starch iodide paper test should last 20 minutes at 82°.

A small experimental apparatus was available for the nitration of methriol. For the purpose of introducing (into the apparatus) methriol which is a fine powder (previously sieved), an apportioning screw was

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Krummel (Cont'd.)

installed which permitted the material to be fed in a fine stream as evenly as possible. The formation of lumps should be avoided at all costs, for they may easily cause decomposition.

The period of nitration for 50 kg methriol was ca. 20 minutes at a maximum brine temperature of 20°. For the separation ca. 15 minutes should be required. The temperature in the pre-washer and at the soda wash should be 40° because at lower temperature methriol is considerably more viscous than diglycol dinitrate. The soda wash lasts 20 minutes, thereupon the usual after-wash with water.

The capacity of the experimental plant for methriol trinitrate was 60 tons per month.

The new plant for the manufacture of explosive oil differs from the old plant mainly in that the nitration and washing were done in one building. Thus a considerable labor saving was obtained. While the old plant required 4 men per shift, the new plant with 2 nitration houses, required only 5 men. A disadvantage, however, was that according to safety regulations the nitration of a new charge could be started only when the finished oil of the previous charge had left the building. This regulation was changed so that between 2 operations there had to be 2 empty vessels. Practically, the result was that a new nitration was started at a time when the previous one was in the soda wash.

The completely washed explosive oil was then transferred to the storage. The explosive oil line was located in a tunnel in which a man could walk. At the end of the tunnel was a swinging arm from which the oil could be run into a wagon. This wagon was large enough to take a whole charge, i.e., ca. 700 kg explosive oil. The wagon was used to transport the oil into the real storage. Here, according to regulations, up to 4000 kg explosive oil could be stored. The storage was built so that it had 2 "exactly equal" halves, each for 2000 kg explosive oil. From each half 2 paste mixing houses could be supplied with explosive oil. In each half there were 3 rectangular tanks each with 700 kg content. When the explosive oil was run out of the wagon it flowed across a filter which had to be washed regularly. At the bottom of the tanks there were drainage cocks from which the oil could be drained off into a scale by way of a rigid pipeline and a swinging arm. This scale was used to weigh the explosive oil per charge for each in-

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Krummel (Cont'd.)

dividual initial mixture of paste. The explosive oil flowed from the scale into a container from which it was ejected by means of water into the paste mixing houses. For the operation of the storage one man was required for each half. At full production the weighing had to be done quickly but correctly.

(a) Denitration of the Explosive Oil:- Waste Acid.

In the denitration operation of the factory at Krummel the waste acid emerging from the separator of the nitration house and the wash acid, which was formed by drowning of the acid oil, were made usable again.

The acids were freed from the explosive oil dissolved in them by running through the so-called breakdown columns at high temperature and thus made usable again. In the case of the waste acid from diglycol normally no separation of the explosive oil took place, or else only in traces.

Due to the action of moisture the waste acid from diglycol was easily decomposed and therefore could only be stored for a few hours at the most. The decomposition started at one place and spread farther and farther at a jump-like increase of temperature. The liquid then boiled and developed large quantities of nitrous gases.

The same process occurred in the breakdown columns; however, here it was regulated and continuous. In order to increase the oxygen surplus already present in the nitric acid in the waste acid, and thus to be sure that all ester was burned, a 50% nitric acid or, in its place, wash acid was added.

Incomplete burning of the ester was shown by dark coloring of the waste sulfuric acid as a result of suspended carbon particles. Incomplete oxidation could cause a clogging of the columns by means of organic material.

The ester dissolved in the wash acid was destroyed merely by running through the breakdown column and by addition of steam at 100° C.

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Krummel (a)(Cont'd.)

Operating Rules.

At the start: Careful heating of the column.

1 hr. 75 cbm water 150 cbm  $H_2SO_4$ /10 sec.

1 hr. 125 cbm " 250 " " " "

1 hr. 250 cbm " 400 " " " "

then addition of steam up to 4 gradation marks of the scale.

1. Denitration of the Waste Acid.

1000 - 1500 cbm waste acid

80 cbm wash acid/10 sec.

2. Temperature of the Column:

at the top 120

at the bottom 160

Steam until necessary temperature is reached.

3. Working of Wash Acid:

800 - 1200 cbm wash acid/10 sec.

Steam until necessary temperature is reached.

Temperature at upper end of column 100°

Temperature at lower end of column 105°

4. Concentration of Nitric Acid.

(Was carried out only when the building was available.)

800 cbm sulfuric acid/10 sec.

500 cbm nitric acid/10 sec.

Addition of steam

Result: 96% nitric acid.

Physical data and a summary of the re-worked and manufactured acid.

Waste acid from diglycol (not yet denitrated)

One batch: 385 liter

Spec. wgt. 1.66

RESTRICTED

Krummel (n) (Cont'd.)

Waste acid from diglycol (not yet denitrated)

4-5% ester dissolved in the acid  
64-66%  $H_2SO_4$   
9%  $HNO_3$

Waste Acid. (denitrated)

Spec. wgt. 1.64  
71%  $H_2SO_4$   
0%  $N_2O_4$

Wash Acid (Prior to running through the denitrator)

One batch: 400 liter  
Spec. wgt. 1.26  
2-3% ester  
5%  $H_2SO_4$   
32-34%  $HNO_3$

Wash Acid

Tower: 45-50%  $HNO_3$   
Bleaching apparatus 38-40%  $HNO_3$   
Tank: receipt from tower and wash acid from denitrator 33%  $HNO_3$   
3.3%  $H_2SO_4$

Remarks

The denitrated waste acid was tested with  $FeSO_4$  solution during operations for the presence of  $HNO_3$ , or  $N_2O_4$ , respectively.

The diglycol waste acid from the nitration house flowed into a storage container and from here it ran by way of the DAG separator into the denitrator. The ester dissolved in the waste acid was burned by addition of wash acid or nitric acid and steam. The nitrous gases thus developed were drawn by a fan through the condensers and forced into the absorption tower. Back of each condenser a bleaching apparatus was connected in which the nitric acid flowing off from there was freed by means of air-suction from the nitrous acid contained therein. The nitric acid ran by way of a rotameter to the tank.

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Krummel (a) (Cont'd.)

The sulfuric acid from the lower end of the denitrator ran through a siphon into the cooling column, from there by way of a cooler into an intermediary vessel and then was pumped into the tank. The air current emanating from the cooling column still containing smoke particles pulled along from the sulfuric acid was, prior to its release into the open, conducted into a condensing column which was sprayed with water obtained from the condensers.

In the concentration of nitric acid the acid went to the bleaching apparatus by way of a pre-warmer heated with steam into the exhaust column where the nitric oxides present were removed by air induced by a fan. The acid ran through a siphon into an intermediate container and was pumped from there into the tank. The wash acid ran from the storage container by way of a small separator and a nitrocellulose filter into the denitrator. From the denitrator the acid flowed through the cooler into the intermediate container. The cooling columns were not needed here.

4. Bonlitz.

At the Bonlitz plant of Eibitz, there were six Schmidt-Meissner nitration units installed to make DEON for the paste which was used for cannon and rocket powder. The five small units each had a capacity of 300 kg per hour and the one large unit had a capacity of 1000 kg per hour.

The NG area was described by Dr. Werner Schmedding, Superintendent of the NG and Acid Area operations. He had been at the plant for six years and had supervised the start up operations in 1939. There had been no explosions or serious accidents on this area.

Dr. Schmedding was well acquainted with many other explosive oil factories in Germany and is probably the best authority on Schmidt-Meissner units.

After Schmidt developed this equipment, the manufacture and installation of plants was undertaken by the large European firm of Meissner and Co. This concern trained the first plant operators but did not provide operating manuals. Unfortunately, the plant drawings of this equipment could not be located.

It was Dr. Schmedding's opinion that the best continuous NG equip-

RESTRICTED

Bomlitz (Cont'd.)

ment would consist of Schmidt nitrators, Biaszi separators and Schmidt washers.

Each unit consisted of three bunkers or earth barricaded concrete igloos with one end made of frame. In the first, there were two rooms, one each for Diethylene Glycol (DEG) tanks and mixed acid storage tanks. The second contained the nitrator, separator, and washing systems. The third contained the  $\text{HNO}_3$  storage tanks and the vessels for emulsifying the oil and water for transfer to the Paste mixing houses.

The expenditure for construction of this plant was apparently much more than that on the DAG plants. The provisions for camouflage trees and for sinking the buildings into side hills were much more obvious.

The raw material, DEG was obtained in tank cars from the I.G. factories at Huls/Recklinghausen, near Essen and at Leuna/Merseberg near Berlin. Each car load was analysed for water content, for unsaturates and the boiling point range was determined. The maximum specification for water was 0.5% as determined by benzene distillation. The specification for unsaturates allowed none as determined by the test with silver nitrate and ammonia. The boiling point range was  $246-249^\circ \text{C}$ .

The mixed acid used had the following composition:

|     |                         |
|-----|-------------------------|
| 62% | $\text{HNO}_3$          |
| 35% | $\text{H}_2\text{SO}_4$ |
| 3%  | $\text{H}_2\text{O}$    |

A ratio of 3.1 parts mixed acid to 1 part DEG was used and a waste acid of the following composition was obtained:

|     |                         |
|-----|-------------------------|
| 10% | $\text{HNO}_3$          |
| 60% | $\text{H}_2\text{SO}_4$ |
| 8%  | DEGN                    |
| 25% | $\text{H}_2\text{O}$    |

It was also necessary to provide a synthetic waste acid with the following composition for cleaning the nitrator and separator:

|     |                         |
|-----|-------------------------|
| 10% | $\text{HNO}_3$          |
| 60% | $\text{H}_2\text{SO}_4$ |
| 30% | $\text{H}_2\text{O}$    |

RESTRICTED

Bowlitz (Cont'd.)

It was stated that a yield of 173 lbs. of DEON was obtained from 100 lbs. of DEG when using the most efficient wash water recovery system. The overall plant Nitric Acid loss was 0.75 lbs. per lb. of DEON made. The theoretical consumption would be 0.65 lbs. of nitric acid.

The raw materials, DEG and mixed acid were fed to the nitrator by means of air pressure on the storage tanks, through Rotometer type flowmeters. The rate was controlled by the air pressure valves.

The large nitrator unit only is described in the following paragraphs. The small units were of identical design but of smaller dimensions.

The DEG was introduced thru a vertical pipe which extended to a point 3 inches above the 1 inch hole in the glass cover. No distributor was needed.

The mixed acid was introduced at the bottom of the nitrator.

The nitrator was made of V2A alloy in three main parts flanged and bolted together. It was about 30" diameter and 40" high. The top section was cylindrical, about 10 inches high, and was covered by a loose glass plate allowing very good observation. The center section contained the cooling element. This was a set of tubes open at the top and bottom through tube sheets. The cooling brine ( $\text{NaNO}_3$  solution) was circulated in the chamber surrounding the tubes. In the center of the tube sheet there was an opening of about 8 inches for the agitator shaft. The bottom section was conical with a 4 inch outlet on the bottom.

Agitation was provided by a propeller and paddle at the point just below the bottom tube sheet and pumped the nitrator charge up the center opening over the top sheet and down the tubes.

At the bottom outlet there was a quick opening drowning valve and also an alternate drowning means consisting of a Kipolex plastic disc which could be cut open by a knife actuated by a temperature controller and from a switch outside the house.

The agitator shaft was driven from a variable speed, carbon brush motor located in a room walled off from the nitrator room proper.

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Bonlitz (Cont'd.)

The motor electric drive was connected to safety controls which shut off the nitrator feeds in case of electric failure. On the motor shaft there was a flywheel 18 inches in diameter to provide agitation for the time interval between electric failure and the introduction of the last DEG.

It was stated that the proper agitation speed had to be determined for each individual nitrator unit. Most of them varied between 380 to 400 RPM. The determination of the optimum speed was made by mixing water and paraffin oil stirred at 20° C. Upon stopping agitation, this mixture should settle to a clear line in 10 minutes.

In starting up a Schmidt unit, the nitrator and separator were first filled with synthetic waste acid. The mixed acid was then started at 5/6 rate. After 30 seconds the DEG was started at the same rate. At exactly 7 minutes later, a clear separating line should be observed in the separator sight glass. If the line had not formed, the raw materials were immediately shut off and the synthetic waste acid was started to flush out the system before starting again. If normal separation occurred after 7 minutes, the rates were increased to the standard.

In normal practice the nitrator charge was maintained at 20° C. The volume of the charge was 350 liters.

The Schmidt separator consisted of three main parts. The lower two were cylindrical vessels flanged together and supported at an angle of 45° from the horizontal. The upper section was a vertical elliptical tank about 15 inches high on the straight side with the top tapered to a 6 inch diameter outlet. The bottom was flanged to the top of the middle section. In operation, the line of separation was about 8 inches above the flange.

The mixture of DEGN and waste acid overflowing from the nitrator was run into the separator at a point at the bottom of the upper section. The feed pipe branched to three perforated pipes inside the separator to provide for distribution. A sight glass in the front of the upper section allowed observation of the charge to determine the level of the interface of oil and acids.

The middle section contained plates parallel to the sloping walls,

## RESTRICTED

### Bowlits (Cont'd.)

i.e., at a 45° angle. These plates were provided for the stated purpose of allowing the drops of separated oil to rise to the top section while the waste acid settled to the bottom. The numerous plates spaced about  $\frac{1}{2}$  inch apart made cleaning difficult.

The bottom section of the separator was a cooler constructed with tubes and sheets similar to that described for the nitrator. Brine was circulated thru the chest of the cooler to keep the waste acid at a low temperature and assure maximum separation efficiency.

The waste acid flowed from the bottom outlet, up a riser and thru a sight box to an outside storage tank. The height of this riser and the height of the oil drawoff at the top could be adjusted for proper operating levels of the separator.

The separator was equipped with automatic drowning valves and plastic discs similar to those described for the nitrator.

From the separator the acid oil was run by gravity to the first of four glass washing towers. These were made of 17 sections of glass pipe 20 cm in diameter and about 8 cm high. Between each glass section there was a sieve plate. Plastic gaskets and aluminum bands were used to seal the joints.

Into the bottom of the first tower, air and water (from the second tower) were introduced through a jet nozzle to mix them thoroughly with the oil run in through a third pipe. This mixture flowed up through the sieve plates which provided distribution and agitation. At the top overflow the air was vented and the oil water emulsion was run into a rectangular V2A box separator.

From this separator the oil was run down to the bottom inlet of the second tower. In this tower fresh water, precooled, was added. After the separator at the top, this water was run through a cooler and then was used as the feed water for the first tower described above.

By using the water in series in the first two towers, Dr. Schmidt stated that the yield of DEGN was improved to 173 from 170, the yield with regular Schmidt operation using fresh water in both towers. Also a wash water was obtained which contained 45%  $\text{HNO}_3$  and 3% DEGN. This was then sent to the denitrating recovery.

Bomlitz (Cont'd.)

The oil from the second tower separator was then run through the last two towers counter current to the 5% sodium carbonate solution used for neutralization. This neutralizing solution was separated and sent to the sewer.

From the last neutralizing tower separator the oil was run to storage tanks in an adjoining room in the bunker. Here it was emulsified with water and jetted to the large Paste mixer. For the small nitrator and Paste mixers, the oil was weighed out as the mixer charge required, emulsified as described in the Krummel section, and jetted to the mixer house.

In addition to the equipment described, there were auxiliary tanks in each nitrator room for (1) holding synthetic waste acid, (2) the water filled drowning tank, (3) neutralizing by batches, the oil separated in the shut down procedure.

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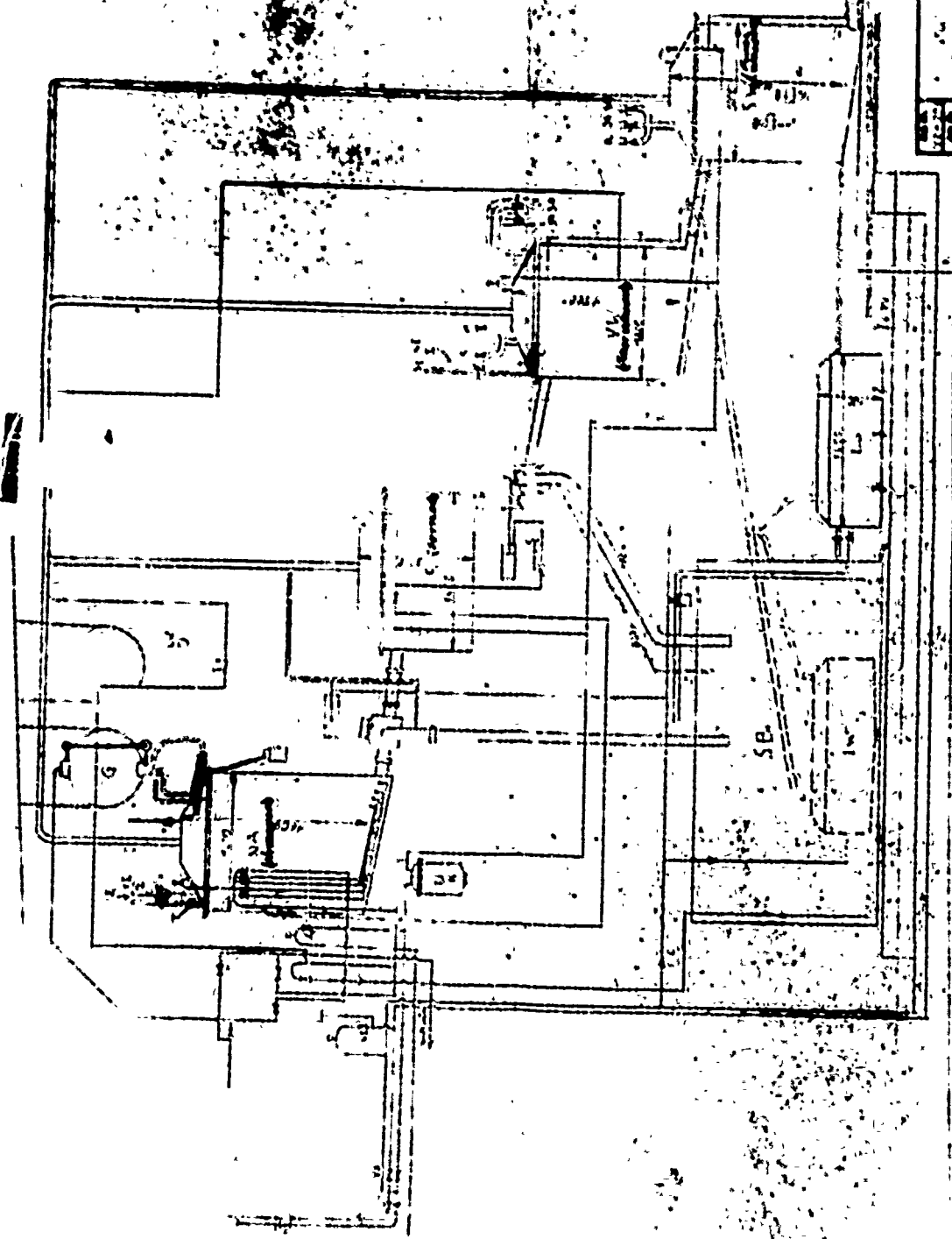
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| Project No. 100-100000                    |  | Department / Division / Subdivision |  |
| Date: 10/1/50                             |  | Drawn by: J. H. Smith               |  |
| Checked by: J. H. Smith                   |  | Approved by: J. H. Smith            |  |
| Title: Schematic Diagram of [System Name] |  | Scale: 1/2" = 1'-0"                 |  |

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