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EXPLOSIVES RESEARCH & DEVELOPMENT ESTABLISHMENT

REPORT No. 16/R/50

The Nitration of Guanidine Nitrate Small Scale Pilot Plant

A. W. H. Pryde and T. Martin

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MINISTRY OF SUPPLY

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The Nitration of Guanidine Nitrate:
Small Scale Pilot Plant

by

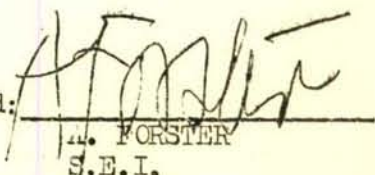
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Reference: XR 441/39.

1. INTRODUCTION.

The nitration of guanidine nitrate with nitric acid and sulphuric acid has been studied in E.R.D.E. with the following conclusions:-

1. None of the nitric acid processes is free from hazard though all of them are capable of considerably reducing the fuel usage compared with the existing method of nitrating with a 2.5 to 3:1 ratio of 98% H_2SO_4 to guanidine nitrate.
2. It is possible to nitrate 98% guanidine nitrate with sulphuric acid at a 1.5 to 1.6:1 ratio of 98% H_2SO_4 to guanidine nitrate or at a 1.1:1 ratio of 20% oleum to guanidine nitrate. This brings the fuel usage down to the same order as any of the nitric acid processes and yields of 93% theoretical have been achieved in batch operation in the laboratory, compared with 88 to 90% by the existing process.

To develop the oleum process a pilot plant for 1 lb. picrite per hour has been erected and successfully operated. This small scale was chosen for reasons of speed and availability of equipment.

The report describes this pilot plant and the results obtained with it, and makes recommendations for a larger scale unit.

2. DESIGN CONSIDERATIONS.

E.R.D.E. Report 15/R/50 describes the investigation into the conditions of nitration of guanidine nitrate in sulphuric acid and oleum. One conclusion from this work is that whatever process is used the reconcentrated sulphuric acid could not be re-used economically because of ammonium bisulphate formation, and would have to be either cracked back to oleum or disposed of to industry. On this basis nitrations with oleum which require the lowest amount of sulphuric acid as the nitrating medium, have been studied, 20% oleum being chosen for handling reasons.

Batch nitrations of guanidine nitrate in oleum are impracticable, but from this study and a consideration of consistencies and grists of products it has been found possible to devise a continuous process for the conversion of guanidine nitrate to picrite.

The process is briefly as follows:-

Guanidine nitrate and oleum are continuously added to a stirred re-circulated spent acid held at 25°C. with cooling throughout nitration. The guanidine nitrate dissolves and picrite bisulphate crystals are formed. This slurry overflows through a series of nitrators, with an average total time of nitration of 2 hours. The slurry then flows to a separator; the spent acid is divided, one portion recycling to the first nitrator vessel to maintain the slurry at a flowing consistency, the second portion going

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to the diluter together with the separated picrite sulphate, and being diluted to 25% sulphuric acid with the wash water from the final picrite washing. This dilution converts the picrite bisulphate to picrite which is then cooled, preferably to below 0°C. and separated from the 25% sulphuric acid.

The picrite is washed on the separator with the amount of water needed for dilution and passes over to the spray crystallisation process. The 25% sulphuric acid from the separator is for recovery or disposal. This process is shown in the flow diagram, Fig. 1.

It was decided that the nitrator should consist of a number of stirred vessels in series. The larger the number of vessels the smaller are the chances of unreacted material passing through the plant. On the other hand a very large number of vessels can result in some of the fully nitrated material being held up in the plant for much longer than the average nitration time, which is undesirable since the nitroguanidine decomposes slowly in the nitration medium.

The equipment needed for a 1 lb./hour continuous plant is as follows:-

- (a) Continuous guanidine nitrate feed for 1.26 lb./hour.
- (b) Continuous feed for 1.39 lb./hour of 20% oleum,
- (c) Nitrator, as above.
- (d) Continuous centrifuge to handle 1.73 lb./hour of acid wet picrite bi-sulphate and 2.125 lb./hour of liquor.
- (e) Continuous feed for 1.2 lb./hour of recycle liquor.
- (f) Diluter for 6.9 lb./hour of dilution mixture.
- (g) Continuous centrifuge to handle 6.9 lb./hour of dilution mixture. Spray washing on the centrifuge would be desirable.

Since the reaction mixture in the last stages of the nitration is a slurry which does not settle out readily, slow stirring should be adequate to maintain the solid in suspension with a minimum of intermixing and by-passing.

Preliminary experiments carried out in a stirred beaker fitted with a cooling coil and an overflow indicated that the quantity of recycle liquor required to maintain a reasonably flowing slurry was of the order of 1.2 parts by weight per part of picrite produced. In the short duration of these experiments there was no evidence of icing up on the stainless steel coil and a rough measure of the heat of the reaction by measurement of the increase in temperature of the cooling water gave a figure for the heat of the reaction of about 200 cal./gm. of guanidine nitrate. It was apparent that it would not be possible to remove this heat on the large scale by internal cooling coils in the reaction vessel. Since, however, it should be possible on the large scale to remove this heat by external circulation through cooling coils it was felt that this was not a serious defect in the process. On the small pilot scale envisaged there was no

/equipment

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equipment available for external circulation; however, owing to a reasonably favourable ratio of cooling area to volume, it was decided to proceed with internal cooling for the pilot plant.

Preliminary experiments also showed that there was a tendency for picrite bisulphate crystals to appear in the mother liquor after centrifuging, which would make a continuous feed on a very small scale impossible. It was therefore decided that the recycle liquor would have to be fed back manually in small batches when the nitrator capacity would buffer the effect of irregular feed.

Since there was no brine available for cooling the diluter to 0°C . it was decided to run it with water cooling and control the temperature of the diluter to 15°C . The increase in yield attainable by cooling to 0°C . was known and could be allowed for.

Experiments showed that there was a tendency for picrite to separate out from the wash liquor so that here again dis-continuous feed had to be used. This however, is not serious as the diluter was made large enough to buffer the feed irregularities.

Guanidine Nitrate Feed.

For 1 lb./hour of picrite the guanidine nitrate feed should be 9.53 gm/minute. The only available unit was of the screw feed type but was too large to give a steady flow of the required amount. It was decided that a continuous feed of the rotating segment type might be more positive and reliable. A feed unit (Fig. 2) was therefore made, the rotor being of bronze with four $\frac{1}{2}$ " holes drilled in it; the stainless steel stator is cut away at the top. The unit was driven at 6 revs/min. by a geared synchronous motor, and gave a feed of 12 gm./min. of guanidine nitrate, which rate did not vary by more than one part in three hundred over a period of several hours continuous running. This was satisfactory and the scale of the rest of the plant was based upon this unit.

Oleum Feed.

Past experience had shown that certain precautions must be taken to achieve a steady feed of sulphuric acid or oleum on a small scale. Due to the very small size of the orifice the acid must be effectively filtered before the orifice to prevent blockage. The physical properties of strong sulphuric acid and oleum change rapidly with temperature, and temperature control was therefore important; the feed was immersed in a thermostat bath as shown in Fig. 3.

Recycle Liquor Feed.

Owing to the tendency for picrite bisulphate to crystallise out from the recycle liquor on cooling, as previously mentioned, it was not found possible to feed this back to the plant continuously. Measured amounts were added at intervals of not more than five minutes from a graduated separating funnel. Since there is considerable buffer capacity in the nitrator the discontinuity of this feed is not important.

/Picrite

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Picrite Bisulphate Feed to Diluter.

The mixture of picrite bisulphate and spent acid to be fed to the diluter is a paste and could not readily be fed continuously to the diluter on this small scale so here again a manual feed has to be accepted. Owing to the buffer capacity of the diluter, minor variations in rate of feed were not important.

Wash Liquor Feed.

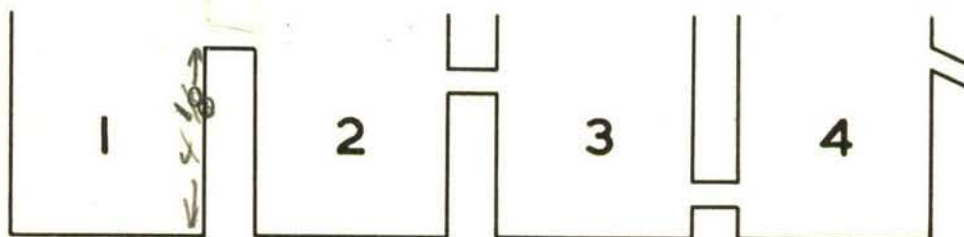
Preliminary experiments showed that spray washing on a centrifuge was the most efficient way of washing the picrite so a suitable spray operated by compressed air was used to wash each batch of picrite. See Fig. 4.

Diluter Water Feed.

There was a tendency for a small quantity of picrite to settle out from the wash liquor which was used in the diluter and a continuous automatic feed was not possible; the diluter liquor had to be fed in frequent small batches from a separating funnel.

Nitrator.

Preliminary tests showed that stainless steel is a suitable material for this stage; therefore four circular vessels were made in F.D.P. stainless steel of 4" external diameter and $4\frac{3}{4}$ " deep interconnected as shown:



Originally vessels (1) and (2) were bottom connected but it was found that the turbo stirrers caused mixing between them, consequently a top connection was fitted. The working depth of the first vessel is $4\frac{1}{8}$ inches and in the other three $3\frac{1}{8}$ inches, giving a capacity of 870 c.c. in the first vessel and 530 c.c. in each of the others. The total capacity is therefore 2,460 c.c. The quantities fed to the plant per hour were 720 gm. guanidine nitrate of absolute density approximately 1.5 (= 480 c.c.), 800 gm. of oleum of density 1.9 (= 420 c.c.) and 400 c.c. of recycle liquor, a total of 1300 c.c. The average time of contact in the nitrator was therefore 1.8 hours to which must be added 0.25 hour average time in the collector vessel giving a total contact time of just over 2 hours.

/No.1

No. 1 nitrator was stirred with a 2 inch turbo stirrer situated near the bottom of the vessel rotating at approximately 500 revs/minute, No. 2 nitrator with the same type of stirrer running at slightly lower speed, Nos. 3 and 4 nitrators did not require such vigorous stirring. In these latter stages mixing of the contents would give rise inevitably to by-passing of incompletely reacted material. The stirrers in Nos. 3 and 4 were therefore of the paddle type $3\frac{1}{2}$ inches wide and 3 inches deep with 8 half inch holes and rotating at 15 revs/minute. This type of stirring prevents settling of the slurry while giving very little mixing.

The nitration collector vessel was a glass beaker of 800 c.c. capacity.

Centrifuges.

The centrifuges (not designed for this plant) were made of stainless steel with 7 inch diameter baskets each 4 inches deep fitted with an external water tight casing with a bottom run off. The baskets were lined with double twill (240 mesh) stainless steel gauze. The second centrifuge was painted inside with acid proof paint. No. 1 rotated at 900 revs/minute and No. 2 at 1900 revs/minute. The recycle liquor, the 25% H_2SO_4 and the wash liquor were collected in glass measuring cylinders of 250 c.c., 2000 c.c. and 2000 c.c. respectively.

Diluter.

Corrosion tests on the weak acid showed that monel metal and lead were satisfactory. It was found that the conversion of picrite bisulphate to 25% H_2SO_4 and picrite was by no means instantaneous, taking in some cases over 30 minutes to reach equilibrium. It was therefore decided to give an average contact time of 1 hour. The total volume rate in this stage of the process was approximately 2.5 litres/hour. The vessel itself was $5\frac{3}{8}$ inches internal diameter and $9\frac{3}{4}$ inches deep with a central baffle to within $\frac{1}{2}$ inch of the bottom. The working depths was $7\frac{1}{2}$ inches. Each compartment was fitted with lead coated brass stirrers with blades bent in alternate directions rotating at 500 revs/minute to give zones of vigorous stirring without intermixing of the entire contents of the vessel.

Diluter Collector Vessel.

A glass beaker of 2 litre capacity sufficed for this purpose.

Picrite Wash Spray.

A stainless steel spray which had originally been made for experiments with fine picrite was found to be suitable, (Fig. 4). The rate of spraying with this was 100 c.c. of water/minute.

No. 1 Nitrator Cooling Coil.

This was of $\frac{1}{2}$ inch stainless steel tubing the two lower turns being 3 inches in diameter and the upper six turns of $2\frac{1}{2}$ inches diameter, tap water being the cooling medium.

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No. 2 Nitrator Coil.

$\frac{1}{4}$ inch diameter stainless steel tubing with 5 turns of $2\frac{1}{4}$ inches diameter.

Diluter Coils.

Each compartment had two loops of $\frac{1}{2}$ inch external diameter lead tubing, the immersed length being seven inches in each case.

3. OPERATION OF PLANT.

The plant was set up on a metal grid as shown in Figs. 5 to 8. Oleum was pumped from the reservoir to the feed vessel and the feed adjusted to that required. No. 1 nitrator was filled with 80% sulphuric acid and the stirrer started. The 20% oleum feed and the guanidine nitrate feed were started and the cooling water to No. 1 nitrator adjusted to maintain 25°C. The nitration mixture overflowed to No. 2 nitrator and the stirring and cooling were started when the stirrer was covered and the temperature adjusted to 25°C. This procedure was repeated in No. 3 and No. 4 nitrators. After about 1½ hours running the mixture overflowed from the nitrators to the collector vessels; it contained only very little picrite bisulphate. No further processing of this was attempted in the early stages and no recycle liquor was fed back till the mixture in the nitrators reached the desired final consistency and composition. The last batch of nitrated mixture in the collector vessel was then centrifuged and 200 c.c. of the mother liquor fed back to the liquor feed vessel and introduced into No. 1 nitrator in aliquot portions every 5 minutes; 200 c.c. of recycle liquor was fed back in this manner every 30 minutes. The contents of the collector vessel was centrifuged in No. 1 centrifuge every 30 minutes and the excess mother liquor and the picrite bisulphate from the centrifuge were mixed and fed into the diluter by hand. The diluter was previously filled with 25% sulphuric acid. As soon as the picrite bisulphate feed to the diluter was started dilution water was added from the wash liquor feed at the appropriate rate to maintain the acid strength in the diluter at 25% and the cooling water started and adjusted to maintain 15°C. in the diluter. The diluter then overflowed into the collector beaker and every 30 minutes the contents were centrifuged in No. 2 centrifuge and the picrite washed with water from the spray gun, the quantity used being such as to give just enough dilution water to maintain 25% H₂SO₄ in the diluter when this wash liquor is used as the dilution liquor.

In all experimental runs the plant was operated for six to eight hours in order to achieve conditions of equilibrium before any picrite was collected for yield determinations. In every case the picrite was dried in an oven at 70°C. till constant in weight.

After shutting the plant down at the completion of a run the nitrators were stirred for a further two hours to complete the nitration and the contents centrifuged in No. 1 centrifuge. The separated picrite sulphate was retained and in future runs was made into a slurry with 80% H₂SO₄ which was used to fill the nitrators at the beginning of a run. The contents of the diluter were also retained, and in this way in later runs equilibrium in the plant was obtained in about four or five hours.

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After two or three preliminary trials to perfect the technique a number of runs were made of about fifteen hours duration in which yields were determined over the last eight or nine hours. All feeds were checked at the beginning and end, and if any significant variation was noted the run was discounted. Successful runs were made at a number of ratios of 20% oleum to guanidine nitrate, the results of which are given in Table 1.

/Table 1.

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TABLE 1

RESULTS OF CONTINUOUS NITRATIONS OF GUANIDINE NITRATE
WITH 20% OLEUM AT 1 lb./HOUR

Run No.	Nitrator Temperatures				Guanidine Nitrate Feed, gm./min.	20% oleum Feed gm./min.	Picrite Made gm./hour	Yield % Theory	Acidity of picrite %	Corrected Yield %	Ratio 20% Oleum to G.N.
	T.1.	T.2.	T.3.	T.4.							
1	65°F.	75-80°F.	90-95°F.	160-105°F.	12.6	13.5	557	89.5 ±	0.3	91.2	1.09:1
2	77-78°F.	75.5°F.	75°F.	72°F.	12.1	13.6	557.5	90.8 ±	0.3	92.7	1.12:1
3	75.78°F.	76°F.	75°F.	72°F.				81.0 ±	0.3	82.6	1.0:1
4	75.77°F.	75°F.	75°F.	73°F.	12.75	17.1	576.0	88.3 ±	0.3	90.7	1.35:1

In each case the diluter was run at 15°C. It is recommended that this should be operated at 0°C. at which temperature there is an increase in yield of 2% at 1.09:1, 2.2% at 1.12:1, 1.5% at 1.0:1 and 2.7% at 1.35:1. This has been added to the yield and the acidity subtracted to give the corrected yield.

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TABLE 2

PHYSICAL DATA

(a) Heat of reaction - 24 K.cals.per.gm. mol. of G.N.

Unit	Heat to Be Removed at 25°C.	Heat to Be Removed at 18°C.	Heat to Be Removed at 0°C.	Over- all Heat Transfer	Vis- cosity	Density
Nitrator No.1	155 cal./ gm. G.N.	-		80 BTU/ hr./sq. ft./°F.	5 poises	1.68
Nitrator No.2	28 cal./ gm. G.N.	-			8 poises	1.67
Nitrator No.3	very small	-			11 poises	1.67
Nitrator No.4	very small	-			40 poises	1.67
Diluter	-	125 cal./ gm. G.N.	195 cal./ gm. G.N.	160 BTU/ hr./sq. ft./°F.		1.21

4. CONCLUSIONS.

4.1. Yields of the order of 92.5% theoretical are possible in continuous nitration of 98% guanidine nitrate with 1.1 parts by weight of 20% oleum.

4.2. Yields are low at ratios below 1.12:1. An increase in ratio increases the conversion but also increases the solubility loss, hence there is an optimum ratio depending on the time of nitration and other complex factors, e.g. on the form and operation of the plant. In the 1 lb./hour unit described the optimum ratio appears to be 1.12 parts 20% oleum to 1 of guanidine nitrate.

4.3. In scaling up the process, the main difficulty foreseen is the cooling of the nitrator. Adequate cooling surface must be provided and icing up avoided. It appears that the former is hardly possible within the volume of the reacting liquor and will call for external coil cooling with circulation. Icing up in this cooler might be prevented by circulation sufficiently rapid to maintain turbulent flow and scouring action.

4.4. Following a discussion with the Escher-Wyss representative it is clear that the Ter Meer Type continuous centrifuge should be suitable for the separation of the picric bisulphate; other alternatives such as rotary filters might be used. The Ter Meer type will not be suitable for the final separation but the Bird type continuous centrifuge should serve, perhaps with slight modification (as on the normal type it is not possible to separate the wash liquor from the filtrate).

5. RECOMMENDATION.

That a larger scale pilot plant of 50 to 100 lb./hour be erected at Waltham Abbey.

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APPENDIX

COMPOSITION OF SPENT ACID.

A 200 gm. sample of the accumulated spent acid from the continuous plant was distilled till the temperature reached 160°C. in approximately 1 hour., corresponding to about 70% H_2SO_4 concentration. 130 gm. of distillate was collected and titrated with Normal Sodium Hydroxide giving a figure, based on the original 200 gm. of 0.69% total acidity. The neutralised liquor was evaporated to dryness, and nitric acid was estimated by ferrous sulphate titration, giving a figure of 0.53% HNO_3 , again based on the original 200 gm. of acid. The original acid residue was distilled, the final temperature being 310°C. The distillate of 11 gm. was analysed for HNO_3 in the same way as above and gave 0.07% based on the original 200 gm. of acid. The final residue was neutralised with NaOH and boiled with excess NaOH, the liberated ammonia being titrated with $N.H_2SO_4$. 16.7% $NH_4 HSO_4$ was found in this residual acid.

To determine whether any of the nitric acid came from the picrite 200 gm. of 23% H_2SO_4 made from A.R. H_2SO_4 was taken and 2.0104 gm. of picrite added. The mixture was distilled to 160°C. over a period of 1 hour and HNO_3 estimated on the distillate giving 0.15% on the original acid.

We can thus conclude that the free nitric acid in the original acid was $0.53 - 0.15 = 0.38\%$.

From the above the composition of the dilute acid can be assumed to be:-

H_2SO_4	22.62%
HNO_3	0.38%
Picrite	0.6%
Unreacted Guanidine	0.2%
H_2O	76.2%

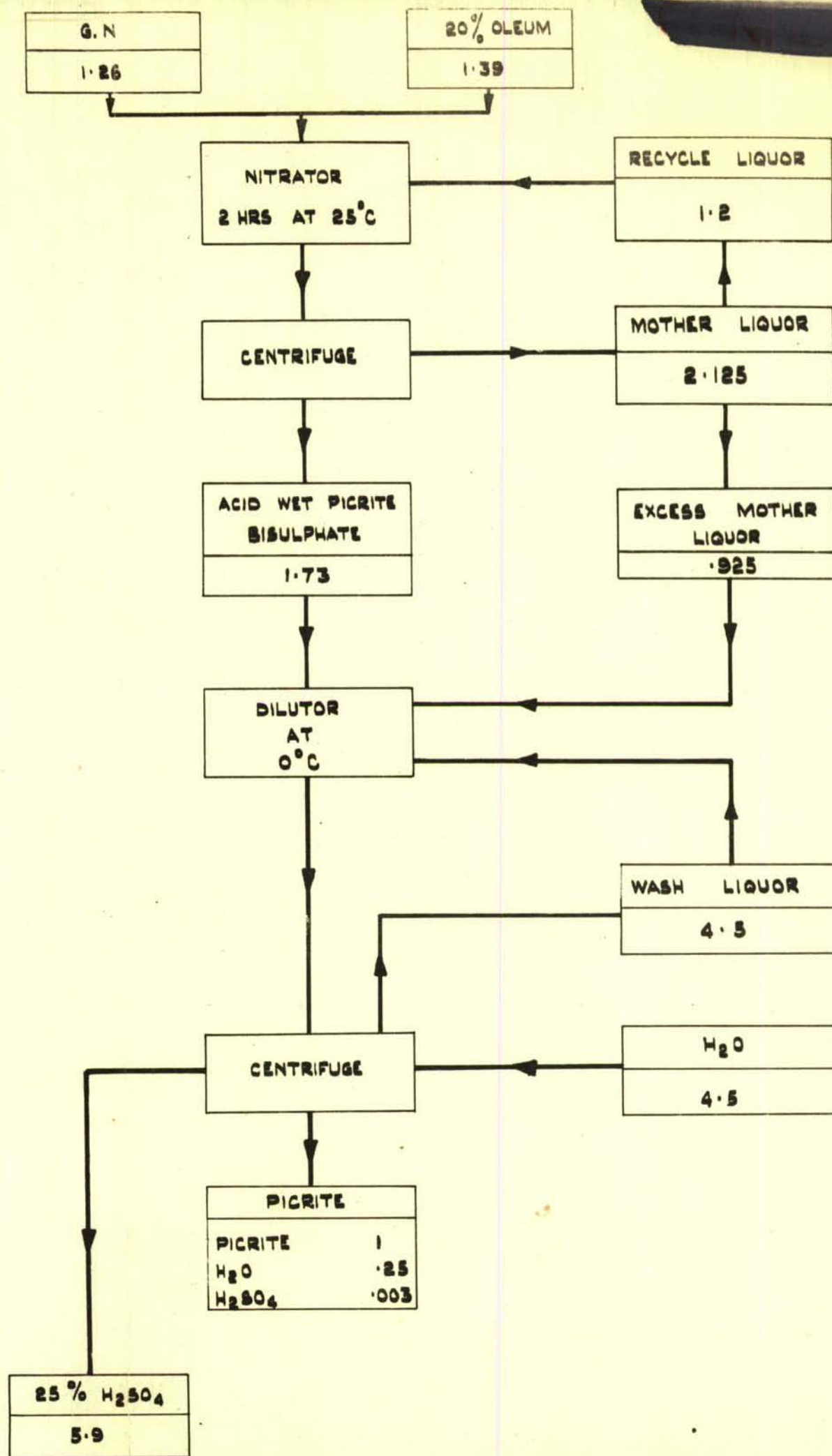
In concentrating the acid to 70% H_2SO_4 the composition of the distillate will be:-

H_2SO_4	0.24%
HNO_3	0.81%
H_2O	98.95%

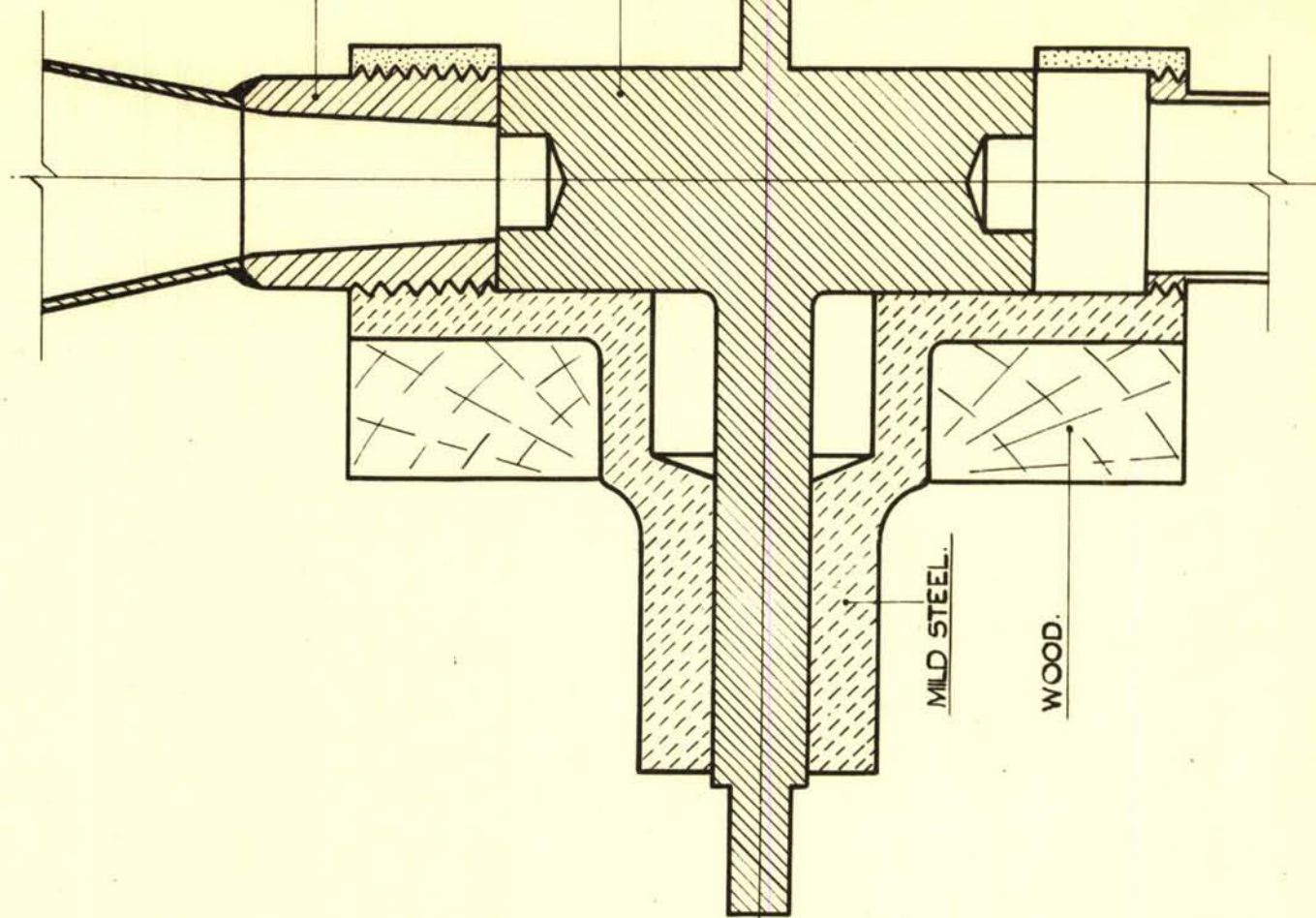
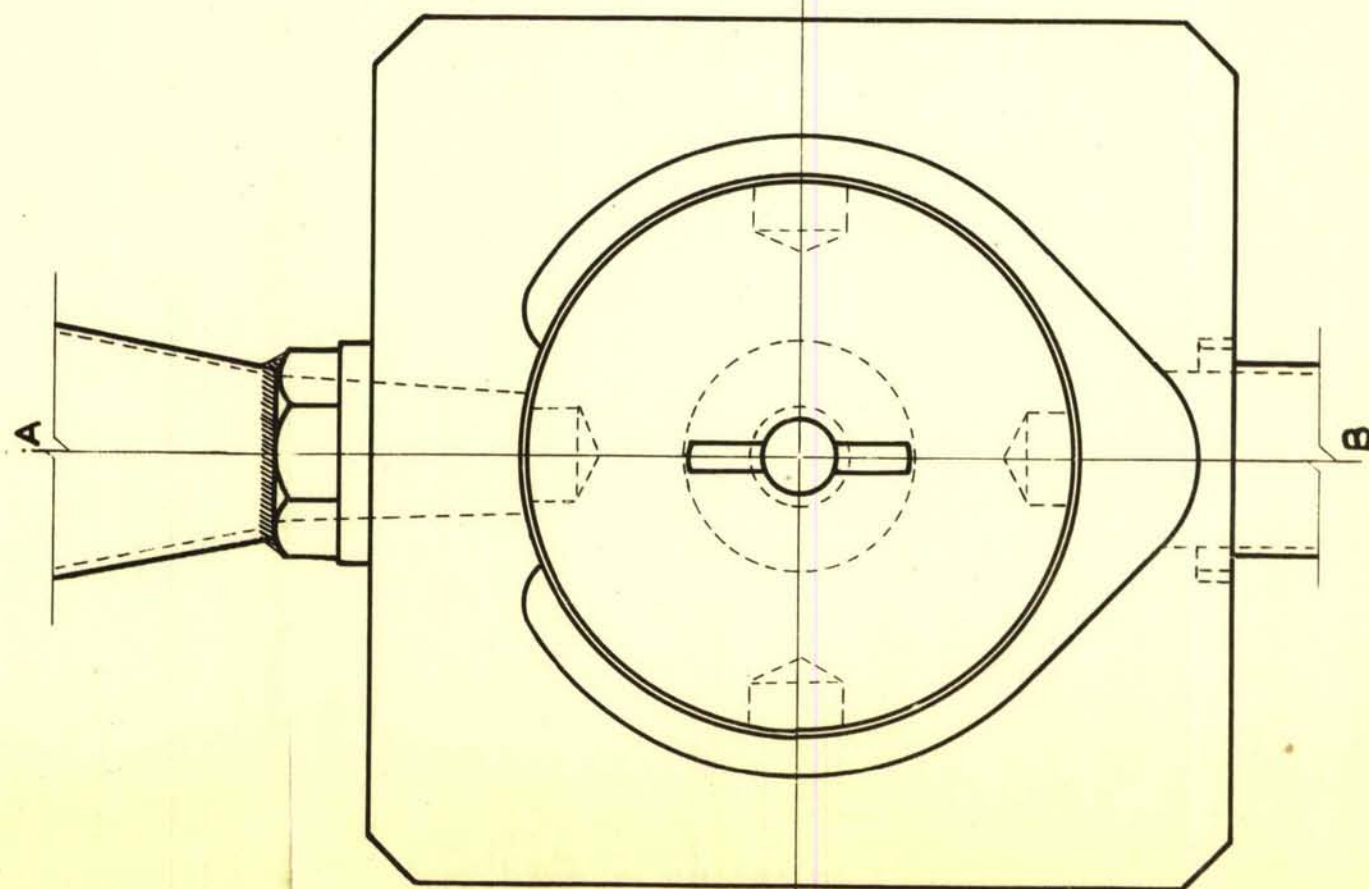
The percentage $NH_4 HSO_4$ calculated on 100% H_2SO_4 is 20.7.

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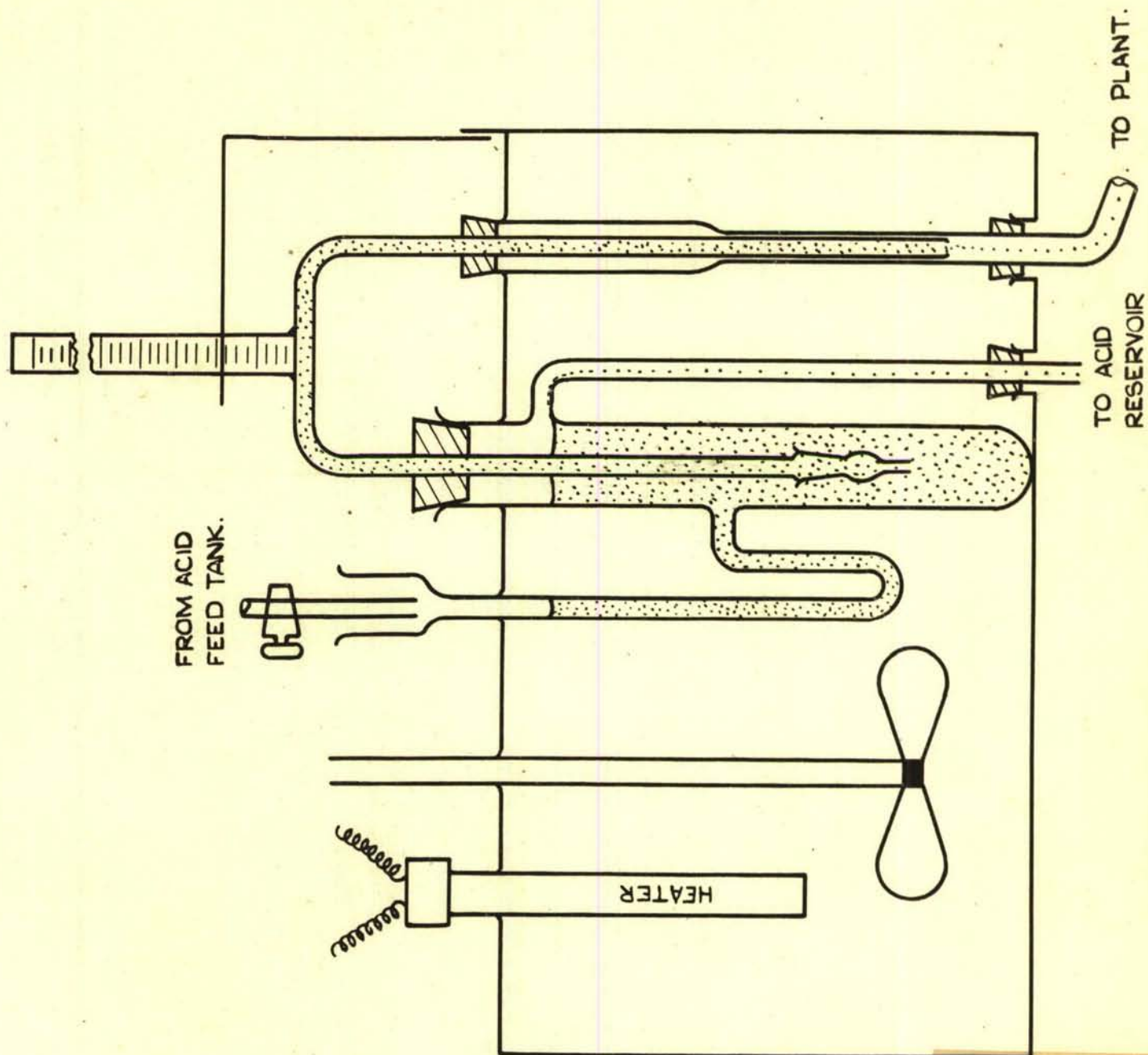


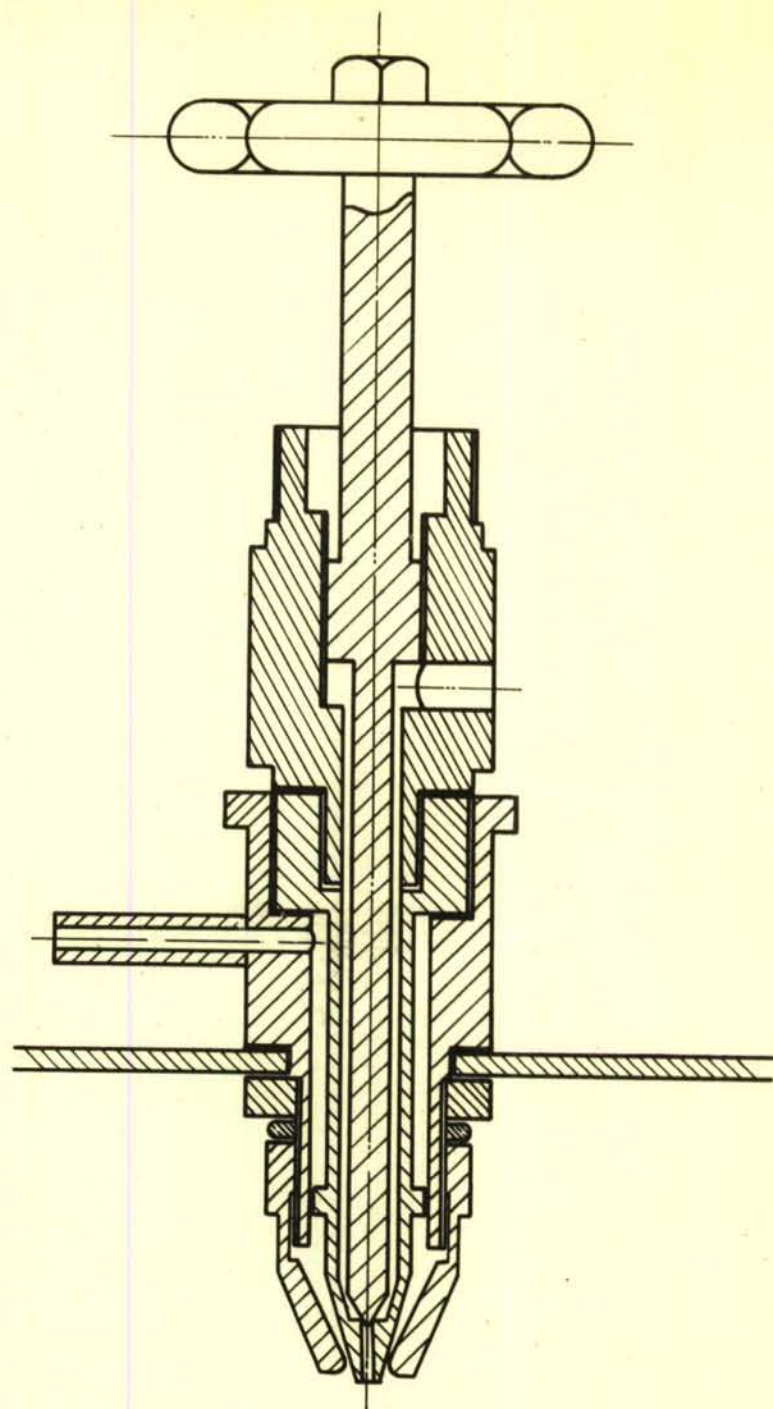
FLOWSHEET FOR 1 LB/HR SCALE
PILOT PLANT.



GUANIDINE NITRATE CONTINUOUS FEED.
SCALE: FULL SIZE.

FIG. 2.





MATERIAL: STAINLESS STEEL.

SCALE: FULL SIZE.

— SPRAY NOZZLE FOR PICRITE. —

FIG. 4.

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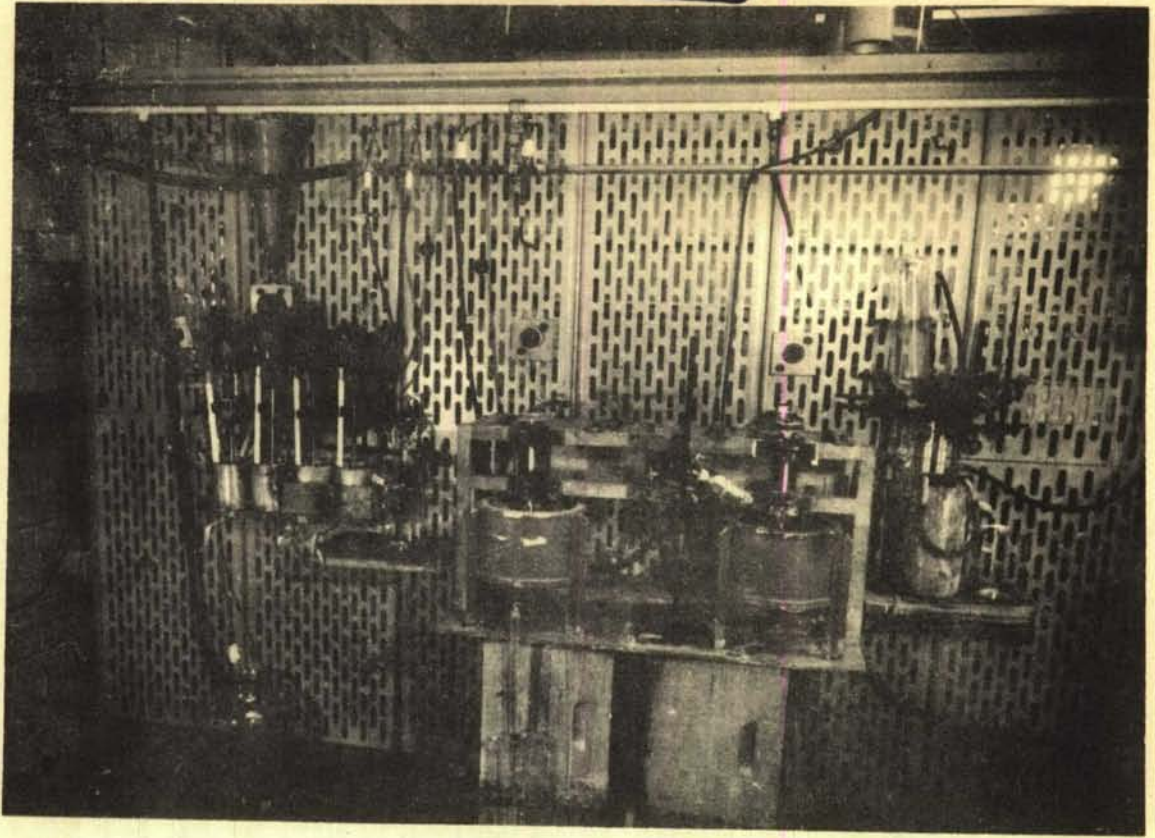


Fig. 5. Front View of Plant.

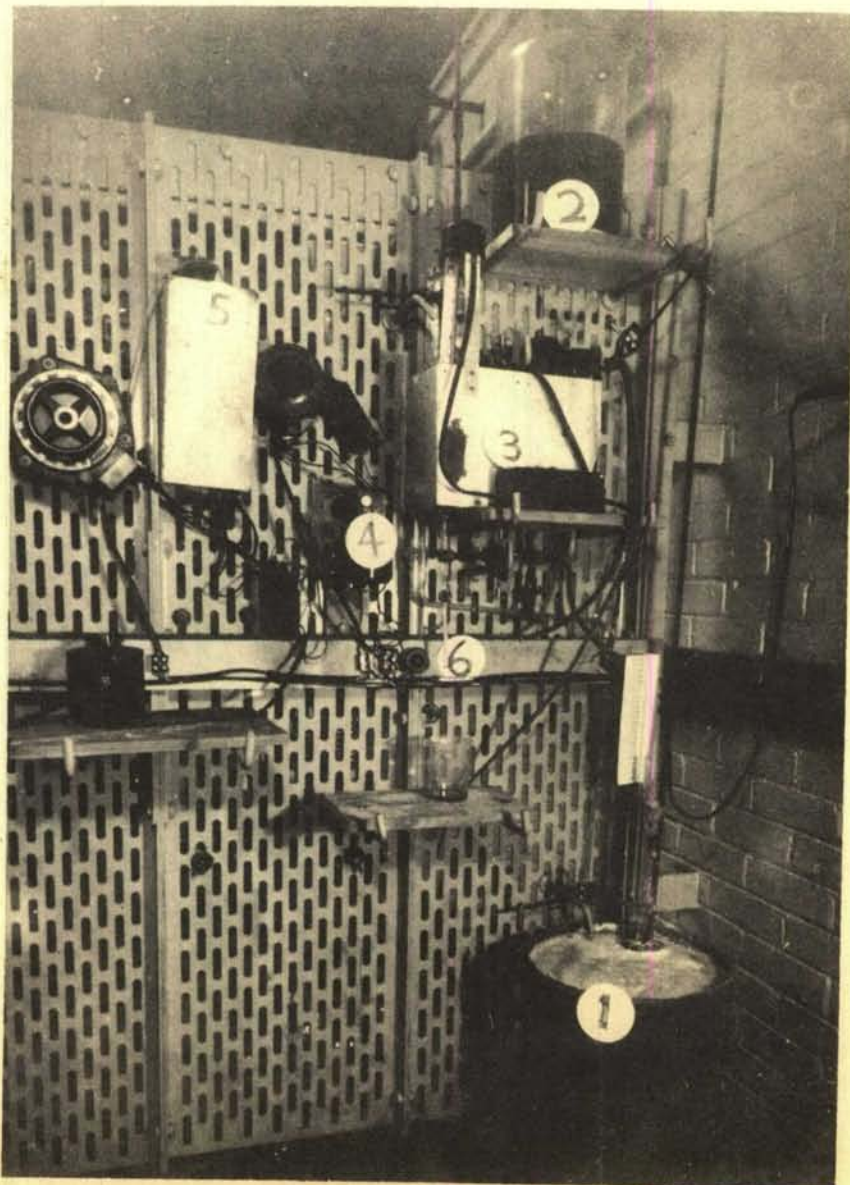


Fig. 6. 1 - 20% oleum reservoir. 2 - 20% oleum feed tank.
3 - 20% oleum feed in thermostat bath. 4 - guanidine nitrate feed drive motor.

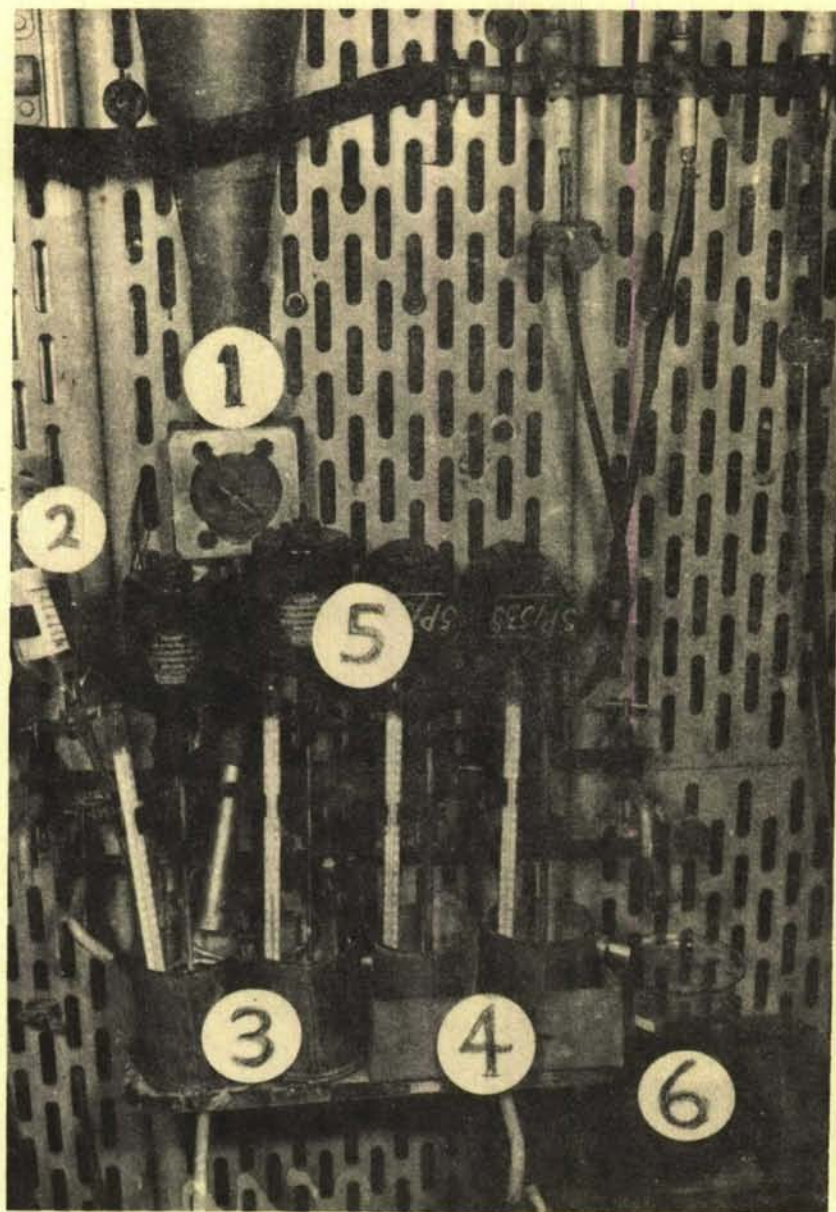


Fig. 7. 1 - guanidine nitrate feed.
2 - recycle liquor feed.
3 - 1st and 2nd nitrators with cooling coils and turbo stirrers.
4 - 3rd and 4th nitrators with cooling jacket and flat blade stirrers.
5 - stirrer drive motors.
6 - nitration mixture collector vessel.

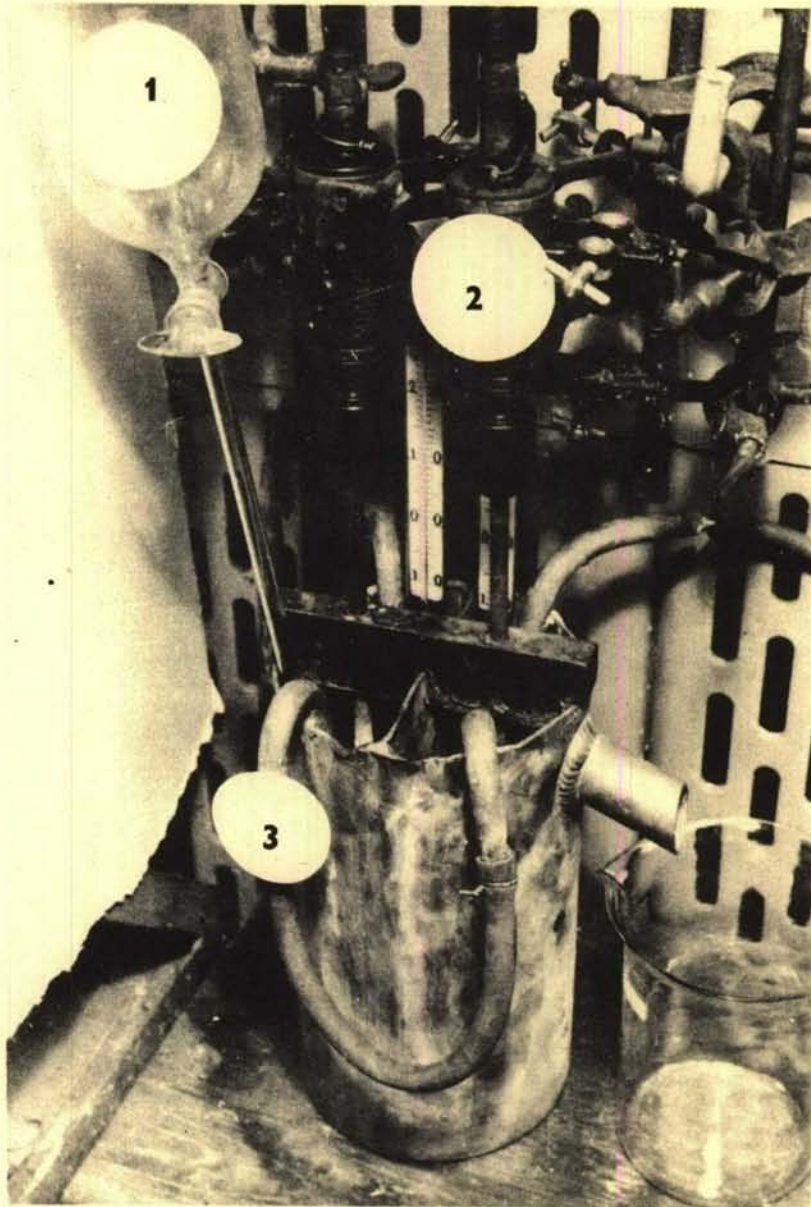


Fig. 8. 1 - wash water feed to diluter.
2 - diluter.
3 - cooling coils.

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