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The Preparation and Properties of Diethylene Glycol Dinitrate

PART III

Batch Plant Manufacture

S. E. Smith

Waltham Abbey,
Essex.

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Part III. Batch Plant Manufacture

by

S. E. Smith.

This report does not contain any information of overseas origin.

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EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT

THE PREPARATION AND PROPERTIES OF DIETHYLENE GLYCOL DINITRATE

Parts I and II. E.R.D.E. Report No. 18/R/48 - Ref. C.R.Temp. 7/11/2/1

By

S. Masterman
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Parts	III.	E.R.D.E. Report	2/R/49	-	Ref.	X.R. 510/1
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"	V.	" "	4/R/49	-	"	X.R. 510/3
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by

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SUMMARY

I. Objects of the investigation

To examine the properties of diethylene glycol dinitrate, and to develop a method for its manufacture from diethylene glycol.

II. Scope of the investigation

The requirements of commercial diethylene glycol for nitration to the dinitrate for Service use have been investigated. The optimum conditions for the nitration, and for the stabilisation of diethylene glycol dinitrate, have been determined by laboratory and semi-technical scale investigations, and batch and continuous pilot plants for these operations have been developed.

The manufacture of D.E.G.N. and recovery of the spent acid have been established on the semi-manufacturing scale and quantities of the product supplied for experimental propellant manufacture.

The chemistry of D.E.G.N., the mechanism of its nitration and the reactions of the spent acid have been given some fundamental study.

No difficulty has been met in obtaining D.E.G.N. of acceptable purity and chemical stability by these processes.

The maximum overall manufacturing yield of D.E.G.N. so far obtained is 90 per cent theory as against 94 per cent for nitroglycerine. This is a disadvantage of D.E.G.N.

The spent acid from D.E.G.N. nitration retains some D.E.G.N. in solution and fumes off at ordinary temperatures after a 'life' depending on the composition of the acid. An acid composition for continuous nitration has been devised to give a spent acid with a safe life around 12 hours at 20°C. A procedure has been developed in which the spent acid from the continuous process is run directly down a dinitration tower in which the dissolved organic matter is destroyed. This involves the recovery of NO_2 as 60 per cent nitric acid and some additional nitric acid concentration.

The physical, chemical, explosive and physiological properties of diethylene glycol dinitrate so obtained have been determined.

Conclusions

1. D.E.G.N. can be safely manufactured in the same types of plant, batch or continuous, as are used for nitroglycerine. The continuous process is more suitable for operation with a continuous waste acid denitration. In both batch and continuous nitration the safety of D.E.G.N. enables simplifications to be made. The yield of D.E.G.N. is less and the consumption of nitric acid is somewhat higher than for nitroglycerine.

2. The spent acid from D.E.G.N. nitration is unstable and needs to be decomposed directly and continuously. Denitration tower practice has been found suitable for this purpose and can be safely linked up with continuous nitr nitration.

3. D.E.G.N. is superior to nitroglycerine in safety in handling.

4. D.E.G.N. appears to have no disadvantageous physiological effects during the short manufacture to date; the long term effect on health of the workers is yet to be determined.

CONTENTS.

Reference X.R. 510/1.

I. Semi-technical scale plant.

1. Introduction.

A 20 lb. batch plant was brought into operation as soon as sufficient data relating to the nitration of D.E.G. was available. Its object was to provide further information about batch nitration and also to meet an increasing demand for DEGN. This semi-technical plant work therefore preceded some of the work presented in Part II and information gained from it contributed to the conclusions reached at the end of Part II.

At a later date, the 20 lb. semi-technical plant was again used, to compare directly the yields obtainable by the process recommended in Part II, viz: nitration with 2.57 parts of 72/28 mixed acid, against the yields obtainable by methods which were used in German factories during the 1939-45 war.

2. Description of Plant.

The layout is shown diagrammatically in Fig.I, and Fig.II is a photograph of the plant.

A miniature N.G. type nitrator separator was used, consisting of a 4 gallon lead pot, 1½ ins. diam. by 10 ins. deep, with a single lead cooling coil, 7/8" diam. 82 ins. long, an inner and an outer ring of air stirring coils on the bottom, and a bottom outlet through which displacement acid was introduced, and by which the contents could be discharged, through a stoneware cock, into a drowning tank. The top of the nitrator had a conical rim 2 ins. deep, with a sloping gutter leading to the prewash tank. An outlet near the top of the nitrator was connected to a fume extractor.

In addition to air stirring, a stainless steel impellor stirrer, driven by a geared electric motor, was also fitted.

The DEG was fed from a small tank mounted above the nitrator. When air stirring was used, injection was by a miniature glycerine injector, but with mechanical stirring, the DEG was run in a thin stream on to the surface of the acid into the vortex created by the stirrer, from a compo nozzle. Cooling was by ice water, flowing by gravity from a tank fixed at a sufficient height.

A tank for displacement acid, with a bottom outlet closed by a cock, was mounted above the nitrator.

The drowning tank was of 20 gallons capacity and had an air stirring coil. It was normally kept two thirds full of water.

The pre-wash tank was stainless steel, 10 ins. diameter, 9 ins. deep, 2 gal. capacity, with a sloping bottom, and a cooling jacket through which water could be passed. It was fitted with a stainless steel air stirring coil, and had a bottom run-off to which was attached a rubber tube. During washing the end of this tube was held above the level of the contents of the pre-

/washer,

washer, and a stream of air blown through it. The pre-washer was also fitted with a skimmer, of the same design as that used in washing N.G., but smaller, and made of stainless steel. The skimmings were passed into a bucket or a settling tank.

The washer was a lead tank 15 ins. diam. by 15 ins. deep, capacity 5 gallons, with a sloping bottom and a 2 ins. deep conical rim at the top. Stirring was by means of air blown through a perforated coil of compo tubing. It was fitted with a lead N.G. pattern skimmer, and a bottom run-off carrying a rubber tube which, during washing, was kept above the level of the contents, and connected to an air supply. A 100 gallon pan was used as a wash water settling tank.

3. Operation of Plant.

In order to carry out a nitration, the mixed acid was first weighed out and placed in the nitrator. Then enough mixed acid was placed in the displacement tank to fortify the waste acid to be produced in the nitration: to this was added about 3 gallons of old fortified waste acid (F.W.A.). The cooling water was then turned on to the coils in the nitrator, and the stirrer started. A little DEG was placed in the DEG tank and levelled off on the sight glass to a fixed level, to provide a datum line. The DEG needed for the nitration was then weighed out and placed in the DEG tank.

When the acid in the nitrator had cooled to 15°C., addition of DEG was commenced, and the rate of inflow regulated so as to keep the temperature steady at 15°C. throughout the nitration. This usually meant, in summer, a rate of flow of cooling water of 5-6 gallons/min. entering at 0-½° and issuing at about 7° - 8°C. Under these conditions 13 lb. of DEG could be nitrated in an hour, using 6-8 cwt. of ice. Addition of DEG was stopped when the level in the sight glass reached the datum line.

One minute after all the DEG had been added, stirring was stopped and the contents of the nitrator allowed to separate. This took about 10 minutes, and its progress was followed by observing the gradual appearance of the nitrator coils through the ester layer as it cleared. When separation was complete, samples of each layer were taken by means of a pipette, for analysis. Fortified waste acid was run in via the bottom inlet of the nitrator to lift the charge, so that the level of the ester layer rose and it eventually overflowed down the sloping gutter into the pre-wash tank where it was received in water gently stirred by air. If the charge was a large one, the pre-wash was done in two parts, in which case displacement took about an hour from the end of nitration.

When all the ester had been displaced over into the pre-wash, a little of the fortified waste acid was removed from the bottom of the nitrator so as to drop the level slightly, and the stirrer was started again to mix up the contents and stabilise the waste acid. The rest of the contents were then removed, weighed, and stored for future use as displacement acid. From the weight of this acid, and that of the acid placed in the displacement tank the weight of the waste acid formed was obtained by difference.

The acid ester layer was washed with about two thirds of

/its

its weight of water, in the pre-wash tank, with cooling water in the jacket to keep the temperature down to below 25°C., the pre-wash liquors being recovered at a concentration of 30 per cent or above. At the conclusion of the wash, after settling, the wash waters were skimmed off and weighed, any ester accidentally removed and settling to the bottom being saved and returned to the rest of the batch. The ester itself was run off from the bottom of the washer into a jug or bucket.

About 8 inches of alkaline wash waters were placed in the washer, and the air stirring started before the ester was added from its jug or bucket. The pre-washer was washed with water into the jug or bucket, to remove all traces of ester, and the washings added to the alkaline wash.

The alkali wash waters were made up and stored in carboys, from which they were measured into the washers in gallon jugs. Tapwater from taps placed above the washers was used for the pre-wash and for the water washes.

At the end of each wash, after the two layers had had time to separate and clarify, the aqueous layer was skimmed off and run into the wash water settling tank. More wash waters were then added, and stirring restarted.

At the conclusion of the last water wash, after skimming off the water, the ester was run off from the bottom of the washer through a rubber tube on to a large filter paper in a conical funnel and filtered into a large Buchner flask. It was then weighed and stored in a carboy. The filter paper removed the dust and all suspended droplets of water. On cooling the filtered ester, however, more water came out of solution, and the ester became milky, but it cleared again on warming.

At the conclusion of each nitration the DEGN which had accumulated in the wash water settling tank was recovered and weighed; it sometimes amounted to 4 oz. out of a theoretical yield of 24 lb., i.e. 1 per cent.

The details of a typical nitration are appended :-

32 lb. of mixed acid of composition 73.39 per cent HNO_3, 26.48 per cent H_2SO_4 and 0.13 per cent H_2O were weighed out and placed in the nitrator.

10 lb. of the same mixed acid and 34 lb. 6 oz. of old fortified waste acid were weighed into the displacement tank.

After filling the lead etc., of the DEG tank up to the mark, 13 lb.0 oz. of DEG were placed in it.

Room temperature was 25°C.

Stirring was started in the nitrator, the mechanical stirring being used. A slow stream of air was blown down the bottom inlet tube to prevent pockets of acid or DEGN forming in it, and the cooling was started.

At 9.50 a.m. the temperature of the nitrator had fallen to 14°C. and the cooling water was leaving at 8°C. at a rate of 6 galls/min. The addition of DEG was commenced.

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The following were the recorded temperatures of the nitrator and the cooling water leaving the nitrator, and the rate of flow of cooling water during the nitration :-

Temp. of Nitrator.	Exit temp. of cooling water.	Rate of flow.
14°C.	3.0°C.	6 galls/min.
17°C.	8.8°C.	6 " "
17°C.	9.3°C.	6 " "
17.3°C.	10.0°C.	6 " "
15.5°C.	10.5°C.	6 " "

All the DEG had been added by 11.03 a.m.

The nitrator was then allowed to cool to 12.5°C., stirring was stopped at 11.10 a.m. and the flow of cooling water reduced to keep the temperature steady. After ten minutes the ester layer was almost water-clear. At 11.23 a.m. samples of waste acid and ester layer were removed for analysis, and displacement was commenced. The cooling water had to be turned full on to cool the displacement acid, which was at 24°C. 58 lb. of acid were recovered from the nitrator, and 6 oz. from the displacement tank, so that the weight of the waste acid formed during the nitration was 14 lb. 0 oz.

The composition of the waste acid was 12.77 per cent., HNO_3, 53.93 per cent., H_2SO_4, 29.89 per cent H_2O, 3.41 per cent DEGN.

The composition of the ester layer was 19.21 per cent HNO_3, 1.02 per cent H_2SO_4, 77.27 per cent DEGN (2.5 per cent H_2O etc.)

The pre-wash was done in two parts, for which $4\frac{1}{2}$ inches and $4\frac{3}{4}$ inches of water were used respectively. The last of the ester went into the pre-wash tank at 11.55 a.m. Nitration and separation had thus taken 125 minutes.

The pre-wash liquors were mixed together and weighed. They weighed 31 lb. 2 oz. and had the following composition :-

17.62 per cent HNO_3, 1.70 per cent H_2SO_4, 0.04 per cent HNO_2, 1.48 per cent DEGN.

The maximum temperature reached in the pre-wash was 26°C.

After each part had received its pre-wash it was removed from the pre-washer in a jug and poured into the first alkaline wash in the lead washer.

1st Alkaline Wash - 3 gallons of warm 2 per cent sodium bicarbonate solution at 25°C. were placed in the washer and the air stirring was started. The DEGN from the pre-wash was added in two parts, followed by the washings of the pre-washer and jug. This wash was continued for $2\frac{1}{2}$ hours after addition of the second lot of DEGN. The composition of the wash waters finally was DEGN 0.513 per cent., $NaNO_3$ 0.952 per cent., Na_2CO_3 0.933 per cent., $NaNO_2$ 1.125 per cent.

/2nd Alkaline

2nd Alkaline Wash - After separation of the first alkaline wash, another three gallons of 2 per cent sodium bicarbonate solution was added. The temperature of the contents of the washer was 25°C., and the wash was continued for 3½ hours.

1st Water Wash - For this, 3 gallons of water at 25°C. were used. The wash was continued for 3 hours, pH = 9.41. No nitrite could be estimated in the wash waters but they gave a blue colour with starch and KI.

2nd Water Wash - 3 gallons of water at 25°C. for 3 hours, pH = 8.47. Heat test of DEGN 4 mins.

3rd Water Wash - 3 gallons of water at 25°C. for 2½ hours. Heat test of DEGN = 7 mins.

4th Water Wash - 3 gallons of water at 25°C. for 3½ hours. Heat test of DEGN = 3 mins.

5th Water Wash - 3 gallons of water at 25°C. for 2½ hours. Heat test of DEGN = 13 mins.

The yield of DEGN was made up as follows :-

Recovered at end of washing process	20 lb. 12 oz.
" from settling tank	3 oz.
Disposed of in samples for analysis	1 oz.
	21 lb. 0 oz.

The theoretically possible yield is 24 lb. 1 oz. Therefore the percentage yield is 87.4 per cent.

The following analysis shows the distribution of the materials involved in the nitration :-

TABLE I

	HNO_3		H_2SO_4		H_2O		DEG + DEGN		Total
	lb.	%	lb.	%	lb.	%	lb. (DEG)	%	lb
Mixed acid	23.48	100.0	8.47	100	.042	0.95	13.0		31.99
Waste acid	1.79	7.63	7.56	89.3	4.18	93.8	0.38	1.6	13.91
Acid Ester	6.95	29.6	0.31	3.66	0.77	17.3	23.9	99.6	31.00
Pre-wash water	5.47	23.3	0.53	6.2	(24.60)	-	0.46	1.9	31.11
1st.Alk.Wash	0.23	0.98	-	-	-	-	0.16	0.7	32.0
As DEGN	16.00	68.2	-	-	-	-	24.9	-	

The figure for the weight of the acid ester above is obtained by difference from the initial weight of I.A. and DEG and that of the waste acid. The latter is itself obtained by difference, and therefore neither are very accurate. The figure for the weight of the 1st Alkaline Wash is calculated from the total depth of the wash waters and DEGN, namely 9½ inches.

The above figures are a rough guide only on account of the uncertainty necessarily attaching to any estimate of the weight of the waste acid.

Towards the end of the series of nitrations, it was desired to produce DEGN in larger quantities, and to avoid having to spend two days on each 20 lb. batch, four nitrations were carried out in one day and all four batches washed together in a 100 gallon pan. The nitrations were performed as before, but instead of displacing the ester layer over into the pre-wash, the whole contents of the nitrator were removed into a glass separator consisting of an inverted bell-jar with a glass cock cemented into its apex. Here the nitration mixture was allowed to separate, the lower acid layer being run off into a bucket, and the acid ester layer being washed partly in the pre-washer, and partly in a stainless steel bucket, and the latter afterwards separated in the inverted bell jar.

The waste acid was taken outside and allowed to fume-off. This took never less than three hours on a hot summer day. Sometimes it took so long that it had to be artificially fumed-off by injections of live steam. Whilst one batch was separating, the nitration of another batch was proceeded with.

The DEGN was collected together in a 100 gallon pan under sodium carbonate 2 per cent solution, and then washed with the same solution added to a depth equal to that of the DEGN, and later with alkaline sulphite solution and with water. The contents of the pan were stirred by air at 12 lb. pressure introduced through a coil of perforated compo tubing, this gave a thick stiff creamy emulsion. It was found necessary in order to prevent the workers in the building from getting headaches, to fit a cover to the pan and extract the fumes through a catchpot. As much as ½ lb. of DEGN would be recovered from this catchpot after four or five washes of a 100 lb. batch of DEGN.

4. Discussion and Results.

In the first three nitrations, the nitrator was air-stirred and the DEG was introduced by means of a miniature glycerine injector. This was found to be much slower and to produce more fumes than mechanical stirring. The latter circulated the contents of the nitrator over the cooling coils instead of just throwing it about, and thus gave more efficient cooling, so that the time of nitration was reduced by 25%.

Nitrations were usually carried out at 15°C. A few were done at higher temperatures. Separation took from 5 to 20 mins., depending on the composition of the acid used.

None of the nitrations undertaken with the semi-technical plant fumed-off, or needed to be drowned for fear of a fume-off.

/The

The ester was transferred after one pre-wash to the lead washing tank where it received three alkali and three water washes each of about two parts of wash liquor to one of ester. If on completion of these washes the heat test was not above 10 minutes, one further alkaline wash and two water washes were administered. At first sodium carbonate, later sodium bicarbonate was used for the alkali washes, and in the last nitrations sodium sulphite as well. It was found that a 2 per cent sodium sulphite/sodium carbonate wash had a most valuable stabilising effect, and enabled the washing to be reduced to two alkali and two water washes.

If any acid fumes were present during filtration, the DEGN absorbed enough of them for its heat test to fall.

Heat tests of up to 20 minutes were obtained, but were generally 10-13 minutes.

A selection from the data on these nitrations is given in Table 2 below. The figures given for the compositions of the waste acid etc., are obtained by analysis; some of the DEGN in the waste acid therefore appears as nitric acid and "water", as explained elsewhere in this report.

It will be seen that the highest yield recorded is 87.8%. The lowness of the yields is due to the large number of bulky washes needed before it was found that sodium sulphite solutions effectively stabilised the products.

The results in Table 2 bear out the deductions made from determinations of the solubility of DEGN in waste acid, which were discussed in Part II of the report.

The results set out in Table 3a and 3b refer to experimental nitrations carried out at a later date, when information concerning German methods of manufacturing DEGN became available.

At Krummel a batch process was operated in which DEG was nitrated with 2.90 parts of a Mixed Acid containing 65% HNO_3 and 35% H_2SO_4; the continuous process operated at Bomlitz used 3.18 parts of an acid of composition 62% HNO_3, 35% H_2SO_4 and 3% H_2O. The yields reported were higher than those calculated from these figures, with the use of the graphs and data given in Part II of this report, and so a fresh series of nitrations were undertaken with the 20 lb. nitrator separator, to check this information. At the same time similar nitrations were made with 2.57 parts of mixed acid of composition 72% HNO_3, 28% HNO_3, and 2.70 parts of acid of composition 69.0% HNO_3, 29.5% H_2SO_4, 1.5% H_2O, these being those which give a waste acid of 26.5% water content and a near-minimum solubility for DEGN, as found in Part II.

The compositions of the waste acids and acid esters produced were obtained by analysis and were also calculated from the available solubility data, and the results compared with the stated compositions.

The life of the waste acids before they fumed-off, was also investigated; these results are reported in the appropriate section of Part II.

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The results show that the yields obtained agree reasonably well with those calculated, except in the case of the 65/35 acid. The yields by the German techniques were not as high as claimed, being slightly lower than those using our nitration acids; moreover, larger vessels would be required for the same output, owing to the higher acid ratios. On the other hand the separations were cleaner than ours, and the waste acids were more stable and safer to handle. This latter is no advantage if the waste acids are denitrated immediately after separation, as could be the case in a continuous process.

/Table 2.

TABLE 2 (a)

SEMI-TECHNICAL SCALE NITRATION DATA

Run No.	DEG used lb.	DEG used oz.	M.A. used lb.	M.A. used oz.	Nitration Ratio MA/DEG	M.A. Composition HNO_3 %	M.A. Composition H_2SO_4 %	M.A. Composition H_2O	Temp. of nitration °C.	W.A. produced lb.	W.A. produced oz.	Composition of W.A. HNO_3 %	Composition of W.A. H_2SO_4 %	Composition of W.A. H_2O %	Composition of W.A. DEGN	Composition of Ester Layer HNO_3 %	Composition of Ester Layer H_2SO_4 %	Composition of Ester Layer H_2O %	Composition of Ester Layer DEGN %	Time of nitration Mins.
1	8	11	29	2	3.347	54.4	40.4	5.12	13	-		-	-	-	-	-	-	-	-	80
2	8	12	30	0	3.432	54.4	40.4	5.12	13	-		9.9	61.54	23.37	4.96	-	-	-	-	90
3	9	0	28	0	3.113	54.4	40.4	5.12	18	17	13	6.99	61.76	25.74	4.99	-	-	-	-	95
4	10	0	36	0	3.600	54.4	37.4	8.19	13	22	5	12.25	55.73	24.82	6.88	-	-	-	-	42
5	10	1	30	0	2.986	61.66	34.09	4.21	13	-		10.12	58.60	24.98	6.02	-	-	-	-	46
6	10	0	29	0	2.900	63.95	32.12	3.93	16	13	13	11.30	56.20	26.84	5.66	19.4	0.8	-	79.77	51
7	10	0	27	0	2.700	67.97	32.46	0.43	15	12	6	11.01	59.30	25.71	3.98					56
8	13	0	32	0	2.464	73.39	26.48	0.13	17	14	0	12.77	53.95	29.87	3.41	19.21	1.02	-	79.77	73
9	12	14	32	0	2.485	68.62	30.28	1.10	17	18	6	-	-	-	-	-	-	-	-	63
10	12	6	32	11	2.645	62.15	35.83	1.97	15	15	7	9.46	59.17	26.78	4.59	18.26	0.70	1.29	79.75	-

TABLE 2(b)

SEMI-TECHNICAL SCALE NITRATION DATA

Run No.	P.W. Liquors obtained lb.	oz.	Composition of Prewash liquor HNO_3 %	H_2SO_4 %	H_2O %	DEGN	Temp. of Prewash °C.	Number of Alkaline Washes	Number of Water Washes	Temp. of Washing °C.	Heat test of Product	Percentage Theoretical Yield
1	4	0	40.27	7.44	47.78	4.43	19	3	3	15	$12\frac{1}{2}$	85.3
2	4	2	41.30	11.89	51.97	4.73	8	3	3	15	$15\frac{1}{2}$	83.8
3	-		29.65	3.11	65.28	1.8	20	3	3	15	3	85.8
4	17	3	28.06	4.67	61.82	5.38	-	2	4	15	2	86.3
5	13	14	-	-	-	-	-	2	4	15	$6\frac{1}{2}$	86.2
6	14	10	28.65	1.95	65.01	4.39	-	2	4	15	2	87.2
7	15	15	-	-	-	-	-	2	4	25	12	87.8
8	31	2	17.62	1.70	79.20	1.48	26	2	5	25	13	87.4
9	29	13	-	-	-	-	-	2	6	15	10	84.3
10	28	12	16.92	3.76	77.66	-	-	3	4	15	9	87.6

TABLE 3a.

DETERMINATION OF YIELDS FROM GERMAN NITRATION PROCEDURES.

EXPERIMENTAL DATA.

Nitration.	I	II.	III.	IV.
DEG used	13 lbs.	13 lbs.	12 lbs.	11 lbs.
M/A used	33 lb. 0 oz.	34 lb.14 oz.	34 lb.13 oz.	34 lb.14 oz.
F.W.A.produced.	56 lb.2½ oz.	56 lb.5½ oz.	57 lb.7½ oz.	56 lb. 5 oz.
Disp.Acid used.	46 lb.0½ oz.	45 lb.8 oz.	44 lb.2 oz.	42 lb.2½ oz.
W/A by difference	10 lb.2 oz.	10 lb.14½oz.	13 lb.5½oz.	14 lb.2½ oz.
Add W/A run off	3 lb.9 oz.	3 lb.0 oz.	3 lb.10 oz.	3 lb.11½ oz.
Total W/A.	13 lb.11oz.	13 lb.14½oz½	16 lb.15½oz.	17 lb.14oz.
DEGN after P/W produced.				
Weighed	22 lb.4 oz.	21 lb.13 oz.	20 lb.4½ oz.	18 lb.6 oz.
From baffles	1½oz.	5½oz.	2½ oz.	6 oz.
samples	1 oz.	1 oz.	1 oz.	1 oz.
Total.	22 lb.6½ oz.	22 lb. 3½oz.	20 lb.8 oz.	18 lb.13 oz.
Composition.				
DEGN	99.4%	97.35%	99.65%	99.15%
HNO_3	0.07%	0.30%	0.10%	0.31%
H_2O	0.53%	2.35%	0.25%	0.54%
Nitration Conditions.				
Brine Inlet Temp.	-16°C.	-13°C.	-8°C.	-16°C.
Initial Temp.	4°C.	5°C.	4°C.	4°C.
Maximum Temp.	15.5°C.	15.5°C.	15.8°C.	15°C.
Final Temp.	8.5°C.	6°C.	8.5°C.	8°C.
Time of Nitn.	32½ mins.	32½ mins.	32½ mins.	27½ mins.
Time of Sepn.	10 mins.	10 mins.	7 mins.	10 mins.
Remarks:-				
Separation	No fuming or marked temp. rise.	Some fuming, no marked temp. rise.	Very clean separation, no fuming or temp.rise.	Sepn.good, no fuming or undue temp.rise.
After Sepn.	Some after sepn. in waste acid.	Some after sepn.(more than in I)	No after-separation noticed.	Very little after separation.

/Table 3b.

TABLE 3(

DETERMINATION OF YIELDS FROM GI

Summary of Quantities and Compositions

Expt.		Mixed Acid					Waste Acid				
		% HNO_3	% H_2SO_4	% HNO_2	% H_2O	Parts	% H_2SO_4	% HNO_3	% HNO_2	% DEGN	% H_2O
I	Calcd.	72	28	-	0	2.57	54.4	13.5	-	5.3	26.8
	Found	72.45	28.0	0.12	-0.57	2.52		NOT ANALYSED			
II	Calcd.	69.0	29.5	-	1.5	2.70	54.35	13.45	-	5.3	26.9
	Found	69.0	29.5	0.65	0.85	2.70		NOT ANALYSED			
III	Stated	65	35	-	0	2.90	60	8	-	6-7	19-20
	Calcd.	65	35	-	0	2.90	61.05	10.65	-	6.7	21.6
	Found	65.8	35.0	0.15	-1.00	2.90	61.45	10.9	0.30	5.3	22.05
IV	Stated	62	35	-	3	3.18	60.1	12.8	-	4.5	22.6
	Calcd.	62	35	-	3	3.18	56.9	12.75	-	5.5	24.8
	Found	61.9	34.45	0.15	3.5	3.18	58.5	12.7	0.77	4.5	23.53

* Stated yield of finished DEGN after completion of washing.

TABLE 3(b)

ELDS FROM GERMAN NITRATION PROCEDURES

Compositions, with a Comparison of Net Yields

		Acid Ester				Yield of DEGN after separation (parts)	Net yield DEGN after two prewashes	
% EGN	% H_2O	% H_2SO_4	% HNO_3	% DEGN	% H_2O		parts	% theor.
.3	26.8	1.3	21.3	77.1	0.3	178.2	173.3	93.6
			NOT ANALYSED				172.4	93.2
.3	26.9	1.3	21.1	77.5	0.1	177.3	172.8	93.4
			NOT ANALYSED				171.0	92.4
-7	19-20	-	-	-	-		170.0 x	91.9 x
.7	21.6	2.3	22.7	75.0	0.0	174.0	168.8	91.3
.3	22.05	1.2	20.75	77.1	0.95		170.9	92.35
.5	22.6	1.8	23.3	73.6	1.3	176.9	169.5 x	91.6 x
.5	24.8	1.5	22.2	71.55	4.75	174.6	169.5	91.6
.5	23.53	0.95	19.1	77.4	2.55		171.1	92.4

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II. 125 lb. Batch Pilot-Plant.

1. Introduction.

125 lb. Batch Plant at Woolwich.

In order to cope more easily with increasing Departmental requirements for DEGN a 125 lb. Batch Plant was constructed and operated at Woolwich from November 1943 until work of this nature was transferred to A.R.D. Waltham Abbey in Sept. 1945. During this period approximately 7000 lbs. of DEGN were produced. The plant comprised a stainless steel Nitrator-Separator of 20 gallons capacity fitted with a lead cooling coil, stainless steel stirrer and bottom run-off via a sight glass and cock. The vessel was virtually identical to that now in use at Waltham Abbey and later described in detail. It is only proposed, therefore, to refer briefly to the following special points in connection with the operation of the plant.

(i) The quantities of materials normally employed were 75 lb. of DEG and 200 lb. of Mixed Acid (HNO_3/H_2SO_4. 72/28).

(ii) Cooling was effected by pumping iced water through the lead coil, and the ice usage amounted to 5-7 cwt. per charge.

(iii) The acid was cooled initially to about 10°C. and the DEG run in over a period of about 35 mins. The temperature of the reaction mixture usually rose to 15-18°C. and was finally cooled down to about 10°C. A period of 10 mins. was allowed for separation and then the lower layer of Spent Acid was run off and collected in stainless steel Buckets. No attempt was made to recover the nitric or sulphuric acids from the Spent Acid and on account of its poor stability it was immediately destroyed by the process of "fuming-off" of three successive portions in an iron pan (20 gallons capacity) out in the open. The decomposition was started by injecting live steam into the spent acid and the time taken to fume-off was 5-10 mins. The remaining acid residues were destroyed in a Lime Pit.

(iv) The Acid Ester remaining in the nitrator-separator was run into buckets and poured into a 50 gall. stainless steel tank filled with water and ice (approximately 50/50). The contents of the tank were agitated with compressed air for 10-20 mins. after which time the temperature ranged from 10-15°C. The DEGN was allowed to separate, the water siphoned off, and the separated oil transferred to a Wooden Washing Vat. Normally, the DEGN from four nitrations (i.e. one day's manufacture) was accumulated and after a standardised treatment of six washes a Heat Test in excess of 10 mins. was obtained. Details of the various washing procedures which have been employed are given in Section 4. The washed, stabilised ester was filtered through paper to remove extraneous moisture, and stored in carboys.

(v) A final yield of about 122 lb. of DEGN was obtained from 75 lb. of DEG corresponding to 88% of theory. Poorer yields were obtained if manipulation at the time of running off the Spent Acid were faulty, e.g. if run off at too fast a rate some ester was inevitably carried away in the vortex.

/125 lb Batch

125 lb. Batch Plant at Waltham Abbey.

This plant was installed in the old N.G. Section (No.1 Hill) at Waltham Abbey during the latter part of 1945 and came into operation during 1946. The nitration unit is based on that formerly used at Woolwich and where possible, the existing N.G. services (e.g. acid lines, buildings, gutter communications etc.) have been utilised. The two original N.G. nitrator-separators, the pre-wash tank and the baffle tank, were removed from the Hill to permit the installation of the DEGN unit. The buildings brought into use were :-

Solution House (for sodium carbonate and sulphite solutions).
Acid Fixing House.
Charge House.
Nitrating House.
Egg House (for Spent Acid).
Washing House.
Pouring-On House (not now used).
Wash Water Settling House.
Acid Denitration House.
Magazine.

A detailed description of the plant is given below. A Flow Sheet, showing the cycle of operations and the quantities of materials involved, is reproduced in Drawing No.1 (A.R.D.8154). The following drawings of the plant are reproduced:

Drawing No.	Original Reference No.	Detail Drawing of :-
2	ED(0)1868/M/5^{C}	General arrangement of plant.
3	/21	Layout of piping in Nitration House.
4	/15	Mixed Acid Gauge Tank.
5	/14	DEG Gauge Tank.
6	/16^{A}	Arrangements and details of Nitrator Drowning Valve.
7	/20^{A}	Arrangements and details of Nitrator Cover.
8	-	Prewash Tank.
9	-	Waste Acid Tank.

Supply and Handling of Raw Materials.

DEG: This is kept in the old Glycerine Store, and one drum at a time elevated to the Charge House. The Charge House supply tank is a vertical tank, capacity 60 gallons, with dished ends, i.e. totally enclosed. The Glycerine Supply Tank, a rectangular galvanised tank with loose fitting cover, was found unsuitable owing to the extreme hygroscopicity of DEG, and has been removed.

ACIDS: 98-99% Nitric Acid from the Bamag Towers, R.D.X. Acid Factory, and 20% (Trade) Oleum, are mixed in a small plant specially

/installed

installed in the Acid Mixing House. This consists of lead and mild steel gauge tanks for Nitric and Oleum, (original NG mixing plant), from which the acids flow through feed boxes, containing calibrated glass siphon tubes, into a small vertical lead drum, which the two streams enter tangentially. From the bottom of the mixing vessel, the acid passes to a vertical lead cooler, capacity about 40 gallons, fitted with lead cooling coils (circulating cold water), air agitation coil, and fume off-take. The cooler has a bottom outlet and also an overflow, from either of which the mixed acid may be pumped to a 1000 gallon storage tank. This arrangement permits either large or small quantities of acid to be mixed. From the storage tank the acid is pumped to the Hill, and is received at the Charge House in a 180 gallon m.s. tank, whence it is pumped through a metafilter into either of two 50 gallon vertical stainless steel supply tanks.

WASH SOLUTIONS: Sodium Carbonate solution and soft mains water (softened by boiling) are prepared in tanks at the N.G. Solution House, filtered (through flannel), blown from m.s. eggs to the Charge House, and stored in galvanised tanks of 800 gallons and 550 gallons capacity respectively. These can be heated by steam coils. Supplies are piped to the Nitrating House and Washing House by gravity flow, controlled by brass plug-cocks.

Untreated river water, used for the pre-washes, is pumped to a water tower and piped by gravity to the Nitrating House (all original N.G. plant).

OTHER SERVICES: A Diesel compressor supplies compressed air at 80 lbs./sq.in. to a ring main in the Nitrating House, to the Washing House, and to other buildings where required.

Cooling brine for nitration etc., is obtained by passing 30-35% Calcium Nitrate brine (containing 7½% Sodium Nitrate) through an ammonia refrigerator. A storage tank on the hill, capacity 2,560 gallons to the overflow (continuous circulation), provides a head of 20 ft. at a temperature of -16°C. or below, to the nitrator, pre-wash, and spent acid tank cooling coils, although an inlet temperature of -8°C. is sufficient for safe working. In view of the fact that some of the cooling coils are stainless steel, it was more desirable to use a Calcium Nitrate brine than Calcium Chloride brine.

The Nitrating "Hill".

This comprises the N.G. Nitrating House, a roughly circular building, wooden construction, traversed and mounded on all sides to a height of 40-50 ft., the Charge House and Brine Supply Tank situated on top of the mound, and the Spent Acid Egg House, outside the mound on ground level.

The CHARGE HOUSE, a wooden building partly supported on piers, is divided into two rooms, one for acid storage, the other for DEG and wash solutions.

The acid room contains one 180 gallon receiving tank, pump and metafilter, supply tanks, and the Mixed Acid Gauge Tank (Drawing No.4). All the vessels and associated cocks and piping, with the exception of the receiving tank, are of stainless steel. The supply tanks can be filled and emptied independently;

/their

their bottom outlets are connected by a common line to the Gauge Tank, which is controlled by stainless steel (s.s.) plug cocks on inlet and outlet. A 1 in. s.s. line leads from the Gauge Tank to the Nitrator, and a second 1 in. s.s. line from the supply tanks, passes direct to the Spent Acid Tank. The Gauge Tank was originally calibrated 190-210 lbs. and 222 - 242 lbs. of mixed acid (at 20°C), i.e. two scales, the upper 32 lbs. greater than the lower; by this means, with one filling of the Gauge Tank 32 lbs. of mixed acid were measured and sent to the Spent Acid Tank in the Nitrating House for fortification of the waste acid produced during nitration, and then the main bulk of nitration acid sent to the Nitrator (N.B. The 32 lbs. of acid for the Spent Acid Tank was run <u>through</u> the Nitrator).

The solution room contains the original N.G. soda solution and soft water tanks, and the (new) DEG tank. The DEG supply tank has a bottom run-off controlled by a $1\frac{1}{2}$" s.s. plug cock, connected by a lead line to the DEG Gauge Tank (Drawing No.5) which is mounted on a shelf above the nitration platform in the Nitrating House. The Gauge Tank is calibrated from 70-80 lbs. of DEG at 20°C. The inlet and outlet are controlled by plug cocks, with a drain cock on the inlet side.

The <u>NITRATING HOUSE</u>, has a wooden platform along one side, opposite the entrance, which constitutes the working level for nitration. An escape door at one end of this platform leads to the top of the mound. In front of the platform a steel framework is erected, which carries the DEGN Nitrator. The Pre-wash Tank stands on a lower platform in front of the nitrator, and the Spent Acid Tank rests on a stool on the floor. Two baffle tanks connected in series, also mounted on wooden stools, overflow into a gutter leading to the Wash Water Settling House. Steps lead from the floor to the Pre-wash Tank platform, on which there is working space, for separation and pre-washing, and thence to the top platform.

Underneath and accessible from the main floor, is a well, which contains a large elliptical drowning vat, (the original N.G. Drowning Tank), containing 3,500 gallons of water.

The <u>NITRATOR</u> is a 20 gallon s.s. vessel (formerly a Sodium Azide Vessel), with a dished bottom, and a top flange, supported in the framework by two side lugs. The centre of the dished bottom has been cut out, and a new s.s. plate welded in (Drawing No.6); this has a 1 in. outlet, off centre, for normal running off, and a 2 in. central outlet which is normally closed by a flap and sealed by a pad of "Nitresto"; the flap valve can be released by a lever mechanism, to drown the charge.

A flat s.s. cover (Drawing No.7) is bolted to the top flange. The vessel contains a two-bank lead cooling coil, total cooling surface 20 sq. ft., and a centrally mounted s.s. stirrer shaft, which carries two double bladed impellors, one near the bottom, the other adjustable in height. The stirrer shaft is supported from the superstructure, and is driven at 200 r.p.m. through a right-angle drive from a $\frac{1}{2}$ H.P. motor housed in a casing on the outside wall of the house. The brine inlet and outlet, the Mixed Acid line, fume off-take, and DEG feed line all pass through the top cover, which also supports a thermometer pocket (totally enclosed, oil filled, containing a mercury-in-glass thermometer), and has a 6 ins. diameter inspection window. There is also

/provision

provision for the insertion of the bulb of a recording thermometer (mercury-in-steel), which is housed outside the building. The DEG supply from the Gauge Tank is controlled during nitration by a ½ in. plug cock and show glass, mounted above the nitrator cover. The other controls for nitration comprise gunmetal plug cocks and thermometers on both brine inlet and outlet; the stirrer motor is controlled from a switch just outside the escape door. The drowning mechanism is operated by either of two hand levers, from the top platform and from the pre-wash platform, and also by a line and pulley system from outside the tunnel entrance. The drowning outlet discharges through a funnel and pipe into the drowning tank.

An air poker is kept handy for emergency agitation in case the stirrer motor should fail.

The 1 in. bottom run-off has a sight glass illuminated from behind by a 15 watt lamp in a flameproof case, and a three way s.s. plug cock, by which separation is observed and controlled. The cock is arranged so that the OFF position is in between the two OPEN positions (a) to Spent Acid Tank, (b) to Pre-wash Tank. When the nitrator contents have separated, the three way cock is first opened to run the lower acid layer to the Spent Acid Tank, and then, as the separation level becomes visible in the sight glass, reversed, so as to run the ester layer to the Pre-wash Tank. This cock, which is a vital part of the plant, is constructed to have no internal "pockets" which would retain unstable explosives.

The SPENT ACID TANK originally installed for the DEGN plant was much too large, and has been replaced by a smaller one, capacity about 20 gallons, dimensions 16 ins. diameter x 24 ins. deep. It is of lead, and has a sloping bottom and domed cover with a central fume off-take. The separated acid enters through the top cover, air agitation and brine cooling provided by a lead coil of about 9 sq.ft. surface area, being used if fortifying acid is present. A thermometer sleeve passes through the top cover, which also has a glass inspection window. The acid is run to the EGG HOUSE through a 2 in. lead bottom run-off pipe and earthenware cock, and trucked from there to the denitration plant.

The PREWASH TANK is fabricated from s.s. sheet, welded up, with a sloping bottom and top flange, dimensions 2 ft. 4 ins. internal diameter, 2 ft. deep on longest side. The bolted on domed top cover has a central fume off-take, and cut-away inspection hole, normally closed by a rubber apron, through which pass the supply pipes for hard river water, soft water, and soda and sulphite solutions, which are controlled by brass cocks or water taps at the side of the tank. The cover also supports an oval s.s. cooling coil, set obliquely in the tank, of 10 sq.ft. cooling surface, and a thermometer sleeve. An air spray coil is fitted for agitation. The tank has a bottom outlet for running off the DEGN after washing, and a second, higher up, for running off the skimmings. The skimmer consists of a P.V.C. funnel and handle attached to a pure rubber hose. The DEGN and waters are run off through rubber hose pipes which are tied up to dummy faucets when not in use. The faucet for the DEGN hose has an air pipe attached, for freeing the hose pipe of unstable explosive during washing. The run-off hose can be used to drown the charge if necessary.

/When

When the DEGN is alkaline (generally after one soda wash) it is run from the Prewash Tank to the WASHING HOUSE down the N.G. GUTTER. This is of lead, double walled, length 176 feet, painted with a rubber base paint (as are also the lead baffle tanks) to preserve the lead. The gutter warm-water jacket is not used for DEGN.

Fumes from the nitrator and spent acid tank are withdrawn by 2 in. lead piping through earthenware Wolff's bottles situated in the annular space between the Nitrating House wall and the traverse, and through earthenware piping to packed earthenware towers, where they are scrubbed by water circulated by Pohle air-lifts. There is one tower to each fume run, and they consist of four 2 ft. diameter sections. The suction is maintained by an air-fed aluminium jet in the chimney of each tower. The Prewash fumes are withdrawn by a lead pipe through a catchpot and out to the atmosphere, suction being maintained by a Narki (silicon iron) air jet.

A cord and pulley system shows on an indicator in the house the level of brine in the header tank on top of the mound. This is normally always full to the overflow.

Washing House.

The WASHING HOUSE is a circular wooden building, 20 ft. diameter, traversed and mounded over on all sides to a height of 20-30 feet, with a tunnel entrance. A platform on one side of the house originally supported three N.G. washing tanks. One of these has now been replaced by a smaller one of similar pattern, suitable for washing 1 - 2 charges of DEGN; the larger tanks can be used for washing several batches together. The large tanks are 3 ft. 9 ins. diameter x 3 ft. 2½ ins. deep on the longest side (sloping bottom) and capacity 209 gallons. They are of lead, have two run-off pipes, one at the bottom, and the other higher up, to which a pure rubber skimmer is attached, and are covered over by a cloth cover except at one point where a fume pipe is burnt on to the side of the tank. An air spray coil is fitted for agitation. The small tank is similar, but has a diameter of only 24 ins. The skimmings pass through an oval baffle tank standing on a lower platform, and then down a lead gutter to the WASH WATER SETTLING HOUSE. The fumes pass through lead catchpots and out to the atmosphere.

Originally it was intended that the washed DEGN should be passed to the POURING-ON HOUSE for filtration in the N.G. Filter Tank, but it was found convenient to use a smaller apparatus which is now installed in the Washing House. This consists of a lead tank standing on the baffle tank platform under the wash tank run-off pipes, the filter standing in a lead cradle inside the tank. The FILTER is a stainless steel funnel, 15 ins. diameter with straight sides 10 ins. deep in which are superimposed two filter pads, consisting of small sponges sewn up inside flannel covers, cut to shape and stitched on a sewing machine. This will filter 4 - 5 charges (500 lb) of DEGN before becoming saturated with moisture and DEGN, when the sponges are removed, washed and squeezed out, and replaced. From the tank, which acts as a reservoir, the filtered DEGN is measured out, 25 lbs. at a time, from an N.G., cordite pattern, lead burette which stands on a lead block burnt to the lead floor, into 4 gallon lacquered pressed steel cans. This quantity (25 lbs.) is

/considered

considered suitable for present requirements having regard to safety and ease of handling, etc.

The wash liquors passing to the Wash Water Settling House flow into the Wash Water Settling Tank (original N.G. plant), of 5,600 gallons capacity. This is emptied periodically and any DEGN found is destroyed. Any DEGN found in the catchpots in the Nitrating House and the Washing House is also destroyed, but DEGN found in the baffle tanks is washed with subsequent charges and recovered.

3. Operation of Plant.

At Woolwich, when iced water was used for cooling, the nitration took about 35 minutes; at Waltham with the ammonia refrigeration plant, brine is circulated through the coils, entering at -10°C. or below, and nitration takes only 20 mins. The acid (200 lbs.) is initially cooled to below 10°C., and DEG (75 lb.) run in a slow stream from the gauge tanks through the sight glass, on to the surface of the acid, being rapidly dispersed by the efficient stirrer. The temperature is allowed to rise to not above 15°C. as the nitration proceeds.

Separation occupies 10 minutes, no accelerant being added. The lower layer of waste acid is then run into the waste acid receiving tank until the level of separation reaches the show glass. The three-way cock is then reversed and the acid ester run into a measured quantity (7½ gallons) of water contained in the s.s. prewash tank, with air agitation and brine cooling to keep the temperature below 20°C.

The duration of the first wash is the time of running in from the nitrator, about 10 minutes being required, after which the first wash is skimmed off and set aside. A second prewash follows, the water from which is discarded after skimming off. The DEGN then receives one alkaline wash in the prewash tank and is then transferred to the N.G. pattern lead washing tanks in the Washing House, for final washing and stabilisation.

A small washing tank is used for a single 125 lb. batch; the larger, original N.G. pattern tanks, will accommodate six batches of DEGN, these being then washed together as a single 750 lb. batch.

After a satisfactory heat test has been reported, the product is filtered, measured out 25 lb. at a time, in the lead N.G. pattern burette, and stored in lacquered cans.

The cycle of operations from the commencement of nitration until the DEGN is transferred to the Washing House, after a first alkaline wash of 45 minutes duration, occupies 2 hours, so that three nitrations can be completed with ease in a normal working day, and the plant is therefore capable of producing 1500 lb. DEGN per week, allowing four days only for nitrating, and one for cleaning up etc.

The method of treatment of the separated waste acid, originally adopted, was to fortify it in the waste acid tank with sufficient fresh mixed acid to reduce the water content to 20%, and ensure safe handling before denitration. It was found, however, that the resulting composition was not very suitable as a denitration tower feed acid, and various modifications

/of

of this procedure have since been tried. For the results of these trials reference should be made to Section V of this report.

4. Comments on the Design of the Plant.

The general arrangement and design of the plant has, in the main, been found quite satisfactory. Some minor modifications which have been made to individual items of plant are discussed below.

One alteration, which is required to enable the prewash liquor to be separated more sharply after the first prewashing, has not been made; this is because the work on the recovery of these liquors from the batch plant, was discontinued when the continuous plant came into operation.

Nitrator: The mechanical agitation and brine cooling are adequate for the purpose of nitration; the only difficulty experienced with the nitrator has been blockage of the run-off pipe leading to the three-way cock. This was due to sludge in the nitrating acid, picked up from lead acid piping. To eliminate this a Metafilter was installed in the Charge House and the mixed acid filtered before use. As an additional precaution the ½" run-off pipe was replaced by a 1" bore pipe.

The lead cooling coil showed some signs of wear after 70 nitrations, but was not seriously impaired. A stainless steel coil would probably have a longer life than lead, for this service.

Separation Cock: This is a vital part of the plant, which has received considerable attention. Originally two full-bore s.s. plug cocks, lubricated with a thin smear of non-reactive mineral jelly, were used, one on either side of a welded s.s. tee piece, so that the run-offs to the waste acid and prewash tanks were controlled separately. Subsequently, a single three-way full-bore s.s. cock, similarly lubricated, was substituted. With both arrangements, some scoring of the plugs occurred. The plug of the three-way cock was then plated with chromium, and relapped into the barrel, which was not treated; after 25 nitrations, hardly any scoring has occurred between the two dissimilar surfaces. A three-way armoured stoneware plug cock was also obtained, but this has not been given an extended trial.

Waste Acid Tank: The original waste acid tank was too large, and when fortifying acid was used, most of the cooling coil was ineffective. The tank was replaced by a smaller one of 20 gall. capacity.

Prewash Tank: The original design was modified by reducing the height of the vessel and also by making the circular cooling coil into an oval shape, thereby making it easier to skim down. As it is still difficult to obtain really sharp separation with the skimmer, which is necessary in separating the first prewash liquor, it is considered that a siphon pipe would be more suitable. The only alternative would be to run-off the lower DEGN layer, leaving the acid behind, but since further pre-washing is required, this could not be done conveniently unless a second p.w. vessel was introduced, and the levels of the preceding vessels raised correspondingly.

/Washing

Washing Tanks, Filters: The batch washing tanks and filter used for DEGN are of a similar pattern to the corresponding N.G. plant and no alterations were necessary.

Fume Catch pots: Although DEGN is more volatile than N.G. the same fume system was used, and no alteration is thought necessary.

5. Plant Investigational Work.

The chief interest in operating the pilot plant was to determine the net yield of DEGN obtainable on this scale of production. This depends on the nitration conditions, the amount of washing necessary, and the mechanical and volatility losses, etc.

The nitration conditions adopted were those recommended in Part II. viz: 2.57 parts of 72/28 mixed acid; a series of nitrations was also undertaken, using 3.18 parts of 62/35/3 acid, principally to gain experience of handling and reworking the waste acid produced, as this process was envisaged for the initial operation of the Schmid plant. For this purpose it was necessary to reduce the quantity of DEG nitrated, from 75 lb. to 70 lb., because of the higher proportion of mixed acid. In all, 15 nitrations were made with this acid, resulting in an average of 115 lb. net overall yield per charge (88.9%). As expected, this was less than the yield obtainable by the original process (with an equal number of washes after separation), but the waste acid remained stable for a longer period, and was more satisfactory as a denitration acid.

Washing: The scheme of washing originally used, consisted of a single water prewash with about 3 parts water to 1 part DEGN at 10-15°C. for 10-20 minutes, followed by several alkaline washes and at least two final washes with soft water, at room temperature. The introduction of alkaline sulphite enabled the number of alkaline washes to be reduced to a minimum of two or three.

It was necessary to reduce the amount of prewash water used, so as to obtain the acid wash liquors at a recoverable strength, (30-35% HNO_3), and under these conditions a second water wash is probably desirable before washing with alkali.

Experiments were made on the plant, using one and two prewashes, in a series of batch nitrations; samples of the DEGN and wash liquor were taken at intervals during each wash and analysed. The results, Table 4, show that while a fairly long time is required to reach the equilibrium distribution of HNO_3 between the DEGN and water phases, it is unnecessary to prolong each wash, since two short prewashes, each with a reduced quantity of water, achieve a reduction in acidity comparable with that resulting from one long prewash taken to equilibiium.

It was reported that DEGN made in Germany, was given two prewashes, the first with a reduced quantity of water, to produce recoverable acid; these were followed by one, or possibly two, alkaline washes, with 2-3 parts of 5% sodium carbonate solution at 60°C., for only 10 minutes, and finally a cold water wash. This brief washing scheme was said to produce material of 20 mins. heat test, and frequently 40 minutes.

/Table 4.

TABLE 4

COMPARISON OF SINGLE AND TWO-PREWASH TECHNIQUES, 125 lb. BATCH PLANT

Batch	I	II	III	IV 1st Prewash (15 mins)	2nd Prewash (30 mins)
Ratio of water to acid ester	1.725/1	1.41/1	1.725/1	0.86/1	1.725/1
Acidity after 10 mins.	-	0.43%	1.14%	0.82%	0.03%
Acidity after 25 mins.	-	0.35%	0.39%		
Acidity after 60 mins.	0.31%	0.08%	0.24%		
Final acidity of Wash Water (C_1)	11.4%	12.52%	11.8%	18.9%	2.14%
Equivalent equilibrium concentration of HNO_3 in DEGN (C_2)	0.06%	0.07%	0.07%	0.22%	below 0.01%

Batch	V	VI	VII	VIII
1st Prewash				
Ratio wash water to DEGN	0.45/1	0.45/1	0.45/1	0.45/1
Time of washing	15 mins.	15 mins.	15 mins.	15 mins.
Final acidity of DEGN	1.80%	1.05%	1.95%	1.47%
% HNO_3 in Wash Water (C_1)	24.1%	28.0%	34.0%	29.6%
Equiv. Equilibrium concentration of HNO_3 in DEGN (C_2)	0.54%	0.88%	1.80%	1.06%
2nd Prewash				
Ratio wash water/DEGN	0.6/1	0.6/1	0.6/1	0.6/1
Time of washing	15 mins.	15 mins.	15 mins.	15 mins.
Final acidity of DEGN	0.34%	0.13%	0.28%	0.10%
Final acidity if 0.45 + 0.6 = 1.05/1 parts solution used in a single prewash, taken to equilibrium.	17.6%	21.4%	20.2%	20.2%
Equiv. equilibirum concentration of HNO_3 in DEGN	0.18%	0.35%	0.28%	0.28%

If this was indeed the case, the design of the washing plant must have been extremely efficient. We have tried short hot alkali washes, and although the high temperatures used undoubtedly assisted emulsification, it was found that a longer period than 10 minutes per wash, was certainly required under batch conditions.

The number of alkaline washes required during the earliest runs with the plant, was sometimes as many as eight or ten, this being partly due to the newness of the plant and the need to establish the optimum proportion of wash solution to DEGN. After a few nitrations it was found possible to standardise the washing conditions; 3½-4% sodium carbonate was used for the alkaline washes and the time of washing was 45-60 mins. The addition of sulphite solution to give 2% sodium sulphite in the wash was found to reduce the number of washes required. The final washing was with softened water, as normal N.G. practise.

The following scheme was finally adopted as being the minimum washing necessary to give a satisfactory heat test :-

(1) 1st water prewash with approximately 0.5 parts water per part acid DEGN, allowing a temperature of up to, but not exceeding, 30°C. for ten minutes.

(2) 2nd water prewash with 1-2 parts of water per part DEGN, warm, for 10-15 minutes.

(3) 1st alkaline wash with 1-2 parts of 3½-4% sodium carbonate, warm, for minimum of 45 minutes.

(4) 2nd alkaline wash with 1-2 parts of 3½-4% sodium carbonate with addition of 2% sodium sulphite, warm, 45 minutes minimum.

(5) Warm soft water 1.1 parts per part DEGN, 30-60 minutes.

(6) Cold soft water 1.1 parts per part DEGN, 30-60 minutes.

6. Yields and Flowsheets.

The best yield of DEGN which can be obtained under the most favourable conditions, is necessarily less than that of N.G., because of the greater solubility losses of DEGN at all stages of the process; in the waste acid, the prewash liquors, and the alkaline wash waters. For this reason, while it is usual to obtain 93.7% theoretical yield in the manufacture of N.G., the best overall yield of DEGN will not be greater than 91.5%. This figure is derived as follows :-

(1) From the graphs shown in the section on the solubility of DEGN in waste acids, a nitration ratio of 2.57 parts of 72/28 mixed acid to one part of DEG was selected as giving a waste acid in which the loss of DEGN is at, or near, its minimum. The actual loss is .068 parts DEGN per part DEG, or 3.7% of the theoretical yield.

(2) Assuming, as is approximately true, that the loss of DEGN in prewashing is a function of the total nitric acid in the ester layer, then the ester corresponding to the above conditions will lose .050 parts DEGN per part DEG, to the prewash liquors,

/or

or 2.7% of the theoretical yield.

(3) In washing, the best that has been achieved on the pilot plant is one further prewash, one alkali wash, one alkali-sulphite wash, and two water washes. Using one part wash solution per part DEGN, and taking the solubility of DEGN in the wash waters to be 0.4%, this means a loss of approx. 2.1% of the theoretical yield.

The yield of DEGN after prewashing, but before the alkaline washes, will thus be 93.6% and the final yield will be 91.5%. These figures neglect losses in nitration due to the formation of byproducts, and also mechanical and volatility losses during washing and skimming. The best overall yield of DEGN obtained in the laboratory was 91.8% while the figures in Table 3(b) show a yield after prewashing, of 93.2% on the semi-technical scale.

On the pilot plant, only the final yields have been assessed accurately, it being more difficult to weigh the yield after prewashing.

The plant originally operated at Woolwich showed overall yields between 87% and 89%, but these were mostly obtained before the value of sulphite washing was realised. At Waltham, with the standardised technique described, a net overall yield of 90.2% has been obtained. This indicates that the miscellaneous losses, including nitration side-reactions, volatility and mechanical losses, amount to 1.3%.

The flow sheets, Tables 5(a) and 5(b) are based on the above figures; some comparable information relating to the batch manufacture of N.G., is given in Tables 6 and 7.

/Table 5(a)

TABLE 5

FLOW SHEET FOR THE MANUFACTURE OF D

DEG Used		M.A. Usage			W.A. Produced			Acid Ester Produced			1st Prewash liquids produced	
100 pts.		257 pts.			126 pts.			231 pts.			130.1 pts.	
	%		%	pts.		%	pts.		%	pts.	%	pts.
Recovered as DEGN	90.2	H_2SO_4	28.0	72	H_2SO_4	54.6	68.8	H_2SO_4	1.5	3.2	2.4	3.2
Lost in W.A.	3.7	HNO_3	72.0	185	HNO_3	13.5	17.0	HNO_3	21.2	49.0	35.0	45.6
Lost in Prewash liquor	2.7				DEGN	5.4	6.8	DEGN	77.0	178.1	3.9	5.0
Lost in later washes	2.1				H_2O	26.5	33.4	H_2O	0.3	0.7	58.7	76.3
Mechanical etc. losses	1.3										Wash water used 75.6 pts.	
	100.											

TABLE 5(a)

CTURE OF DEGN BY THE BATCH PROCESS (DEG BASIS)

Prewash liquids oduced	Ester after 1st Prewash	2nd Prewash liquids Produced	Ester after 2nd Prewash	Wash waters	DEGN Produced
.1 pts.	176.5 pts.	177.4 pts.	172.4 pts.	700 pts.	167.0 pts.
pts.	% pts.	% pts.			
3.2	- -	- -		Soda wash 175 pts.	From Prewashing 172.4 pts.
45.6	2.1 3.4	2.0 3.4	DEGN 172.4 pts.	Soda/sulphite 175 "	Losses in final washing 2.8 pts.
5.0	97.9 173.1	0.4 0.7		Soft water 175 "	Transport losses 0.4 pts.
7 176.3	- -	97.6 175		Soft water 175 "	Mechanical & volatility losses 2.2 pts.
h water d .6 pts.		Wash water used 175 pts.		Soda ash 14 " Sodium sulphite 3½ "	Net yield 167.0 pts. = 90.2%

2

TABLE 5(

FLOW SHEET FOR THE MANUFACTURE OF DEGN

DEGN Used		M.A. Usage			W.A. Produced			Acid Ester Produced		1st Prewash liquors produced		Es P
59.9 pts.		153.9 pts.			75.5 pts.			138.3 pts.		78.0 pts.		10
	%		%	pts.		%	pts.	%	pts.	%	pts.	%
Recovered as DEGN	90.2	H_2SO_4	28	43.1	H_2SO_4	54.6	41.2	1.5	1.9	2.4	1.9	-
Lost in W.A.	3.7	HNO_3	72	110.8	HNO_3	13.5	10.2	21.2	29.4	35.0	27.3	2.
Lost in Prewash Liquor	2.7				DEGN	5.4	4.1	77.0	106.6	3.9	3.0	97.
Lost in later washes	2.1				H_2O	26.5	20.0	0.3	0.4	58.7	45.8	
Mechanical etc. losses	1.3									Wash water used 45.4 pts.		
	100.											

TABLE 5(b)

RE OF DEGN BY THE BATCH PROCESS (DEGN BASIS)

rewash rs ced	Ester after 1st Prewash		2nd Prewash liquors produced		Ester after 2nd Prewash	Wash Waters	DEGN Produced		
pts.	105.7 pts.		107.1 pts.		103.2 pts.	418.4 pts.	100 pts.		
pts.	%	pts.	%	pts.					
1.9	-	-	-	-		Soda wash	104.6 pts.	From Prewashing	103.2 pts.
7.3	2.1	2.1	2.0	2.1	DEGN 103.2 pts.	Soda/sulphite wash	104.6 pts.	Losses in final	1.7
						Soft water	104.6 pts.	washing	
						Soft water	104.6 pts.		
3.0	97.9	103.6	0.4	0.4				Transport losses	0.2
5.8	-	-	97.6	104.6		Soda ash	8.4 pts.	Mechanical and volatility losses	1.3
ater			Wash water used			Sodium sulphite	2.1 pts.	Net yield	100 pts.
ts.			1046 pts.						= 90.2%

2

TABLE 6.

Nitration of Glycerine (Waltham).

1500 lb. Glycerine used = 1 part

8250 lb. M.A. " = 5.50 parts.

Composition of M.A. = H_2SO_4 55.57 per cent, HNO_3 43.44 per cent, water 1.99 per cent.

Mass of HNO_3 taken for nitration = 3580 lb. to 3630 lb.

" " " theoretically necessary = 3080 lb.

Excess = 500 lb. to 550 lb.

= 16.2 per cent. to 17.9 per cent.

Waste acid :- 5950 lb. of composition H_2SO_4 72.9 per cent., HNO_3 9.1 per cent., HNO_2 0.22 per cent., N.G. 3.35 per cent.

Mass HNO_3 in W.A. = 541 lb.

" N.G. in W.A. = 199 lb.

= 4.95 per cent theory yield.

Yield = 231 per cent on glycerine = 93.7 per cent. theory = 3465 lb. Theoretical yield = 4020 lb.

Prewash. (1) 1.4 parts water.

(2) 0.62 " "

(3) 0.5 " soda 3½ per cent.

Wash House. (1) 0.45 parts soda 3½ per cent.

(2) 0.62 " water.

(3) 0.62 " "

(4) 0.62 " "

Total washings = 4.8 parts by weight.

HNO_3 lost in Pre-wash.

Mass of 1st. pre-wash = 4850 lb.

Concn. of HNO_3 in pre-wash = 3 per cent.

Loss = 145 lb.

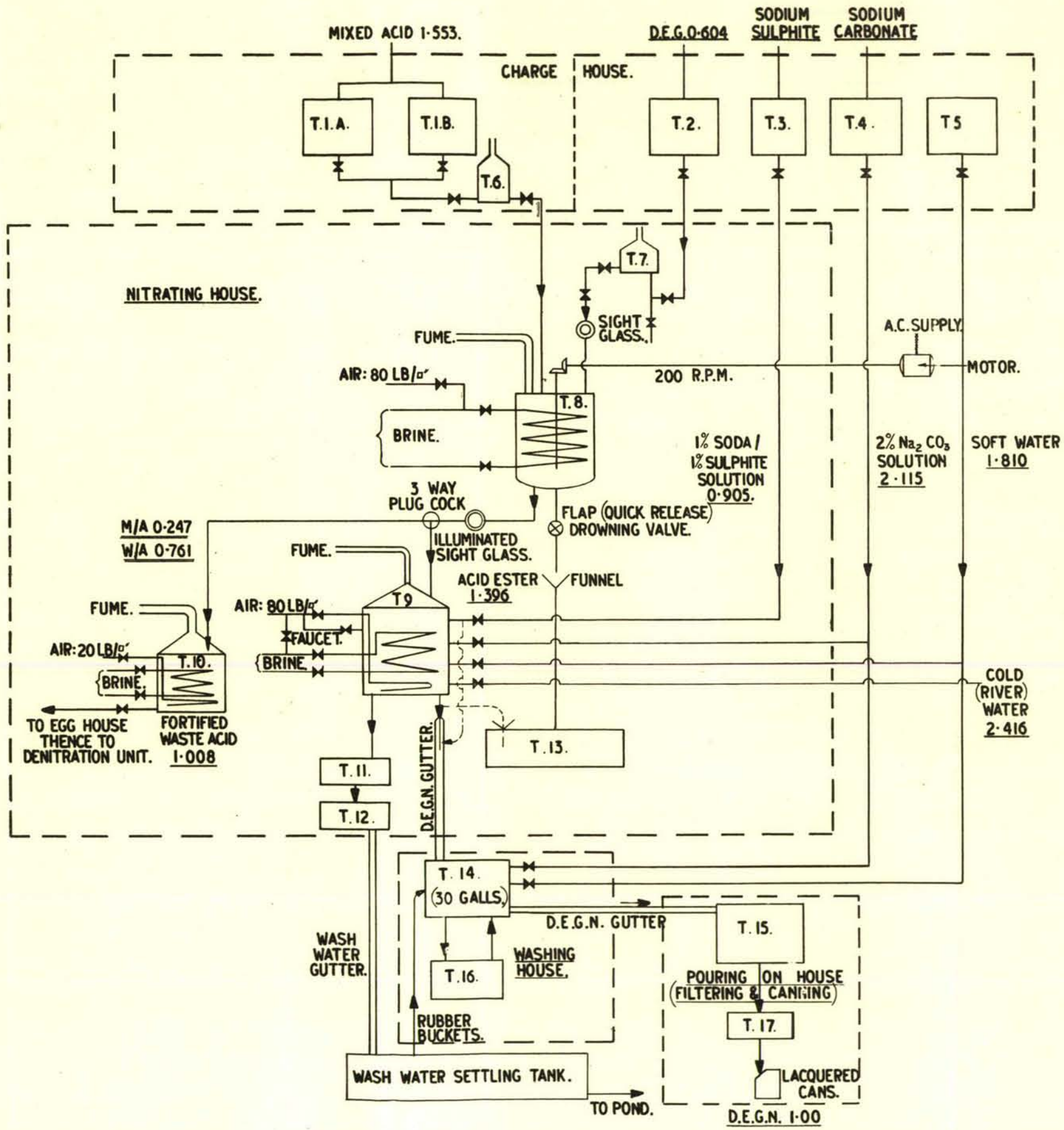
TABLE 7

FLOW SHEET FOR N.G. WALTHAM AND GRETNA

	1	2	3			4			5			6	7	8
	N.G. Produced	Glycerine used	Mixed acid required			Spent acid Produced			Prewash liquors Produced			HNO_3 required	HNO_3 consumed	H_2SO_4 required
WALTHAM	100 Parts	43.3 Parts	248.5 Parts			171.8 Parts			140.0 Parts			107.8 parts	92.8 Parts	138.2 Parts
				%	Pts.		%	Pts.		%	Pts.		In Nitration 88.0	
			H_2SO_4	55.6	138.2	H_2SO_4	72.9	125.2	HNO_3	3	4.2		In Wash Water 4.2	
			HNO_3	43.4	107.8	HNO_3	9.1	15.6	NG	0.4	0.56		In Denitration 0.6	
			H_2O	1.0	2.5	H_2O	14.7	25.3					92.8	
				100.0	248.5	N.G.	3.35	5.75						
							100.0	171.8						
GRETNA	100 Parts	43.5 Parts	257.1 Parts			196.9 Parts						103.9 Parts	87.5 Parts	150.4 Parts
				%	Pts.		%	Pts.					In Nitration 87.0	
			H_2SO_4	58.5	150.4	H_2SO_4	75.1	147.9					Loss in Denitration etc. 0.5	
			HNO_3	40.39	103.9	HNO_2	8.57	16.9					87.5	
			H_2O	1.11	2.8	H_2O	16.4	32.2						
				100.0	257.1		100.0	196.9						

S.No.112
M.No.486/49

CONFIDENTIAL



KEY TO PLANT.

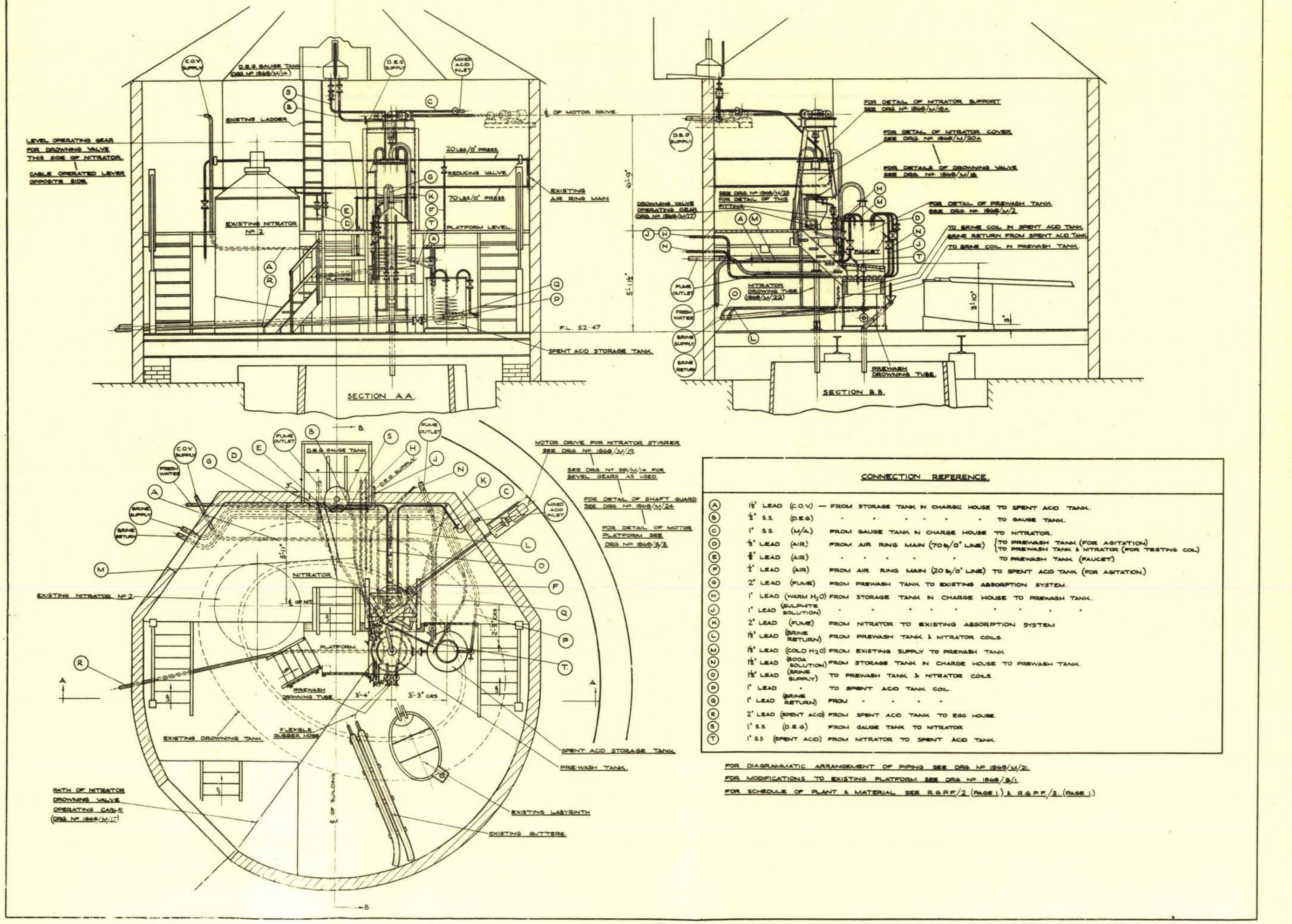
REF. Nº	DESCRIPTION	DRG Nº
T 1. A B	MIXED ACID STORAGE TANKS. (50 GALL CAPACITY EACH.)	
T. 2.	D.E.G. STORAGE TANK. (60 GALL CAPACITY.)	
T. 3.	SULPHITE SOLUTION STORAGE TANK.	
T. 4.	SODA SOLUTION STORAGE TANK.	
T. 5.	WARM SOFT WATER STORAGE TANK.	
T. 6.	MIXED ACID CAUCE TANK. (250 LB. CAPACITY.)	E.D.(0.) 1868/M/15.
T. 7.	D.E.G. CAUCE TANK. (100 LB. CAPACITY.)	" /14
T. 8.	NITRATOR.	COVER " /20 BASE " /16
T. 9.	PRE-WASH TANK. (40 GALL. CAPACITY.)	
T. 10.	SPENT ACID STORE TANK.	
T. 11.	LABYRINTH.	
T. 12.	LABYRINTH.	
T. 13.	DROWNING TANK.	
T. 14.	WASH TANK. (30 GALL. CAPACITY.)	
T. 15.	D.E.G.N. STORE & FILTER TANK.	
T. 16.	LABYRINTH.	
T. 17.	BURETTE. (25 LB. CAPACITY.)	

FOR GENERAL ARRANGEMENT OF ORIGINAL PLANT SEE DRG. Nº E.D.(0) 1868/M/5C.

FOR ORIGINAL DIAGRAMATIC LAYOUT OF PIPING SEE - - - - - /21

Drawing No. 1

Flow Sheet



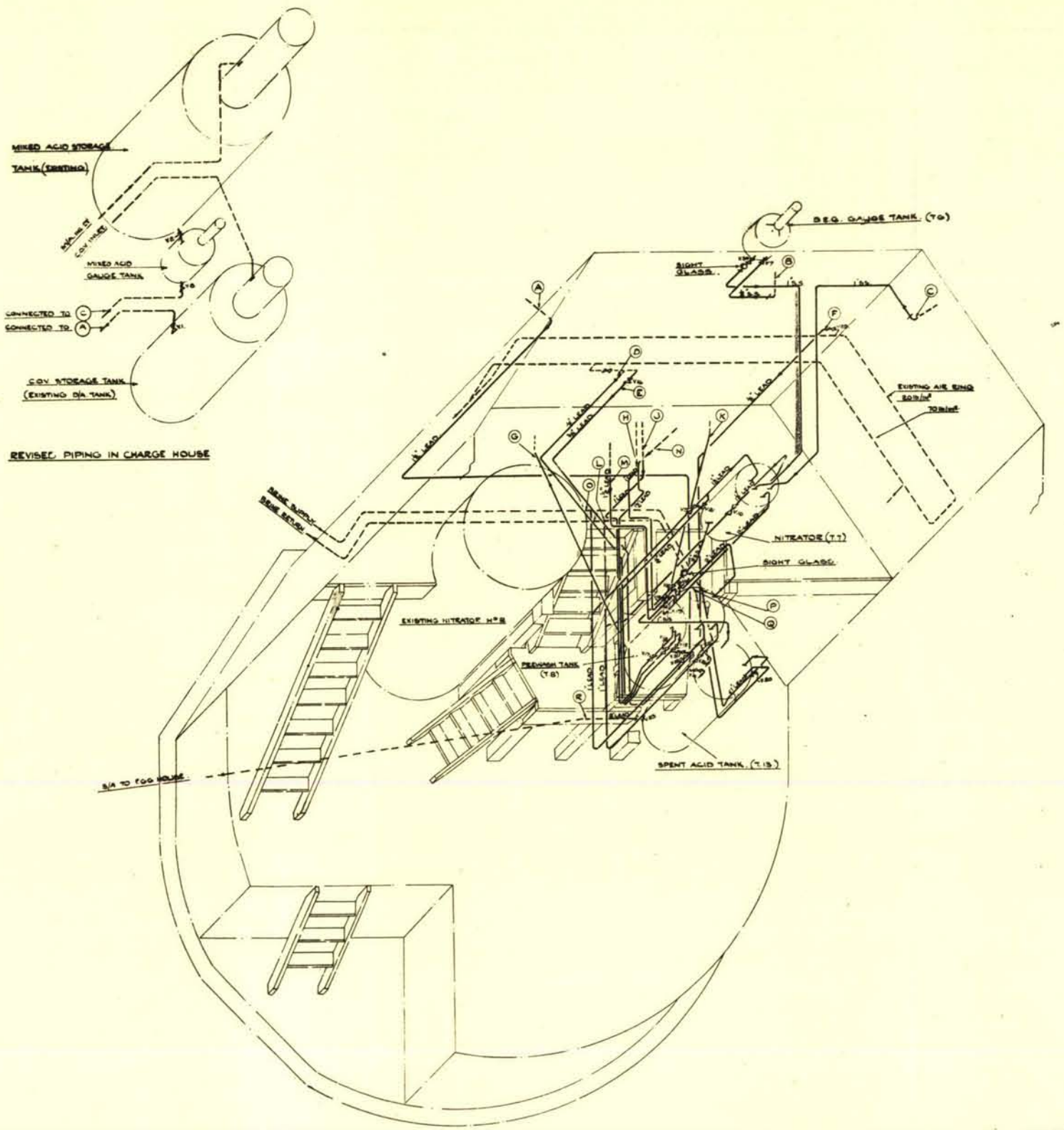
CONNECTION REFERENCE.

A	1½" LEAD	(C.O.V.)	— FROM STORAGE TANK IN CHARGE HOUSE TO SPENT ACID TANK.
B	½" S.S.	(D.E.G.)	. . . . . TO GAUGE TANK.
C	1" S.S.	(M/A.)	FROM GAUGE TANK IN CHARGE HOUSE TO NITRATOR.
D	½" LEAD	(AIR)	FROM AIR RING MAIN (70 lb/□" LINE) {TO PREWASH TANK (FOR AGITATION) / TO PREWASH TANK & NITRATOR (FOR TESTING COIL)
E	⅜" LEAD	(AIR)	. . . . . TO PREWASH TANK (FAUCET)
F	½" LEAD	(AIR)	FROM AIR RING MAIN (20 lb/□" LINE) TO SPENT ACID TANK (FOR AGITATION.)
G	2" LEAD	(FUME)	FROM PREWASH TANK TO EXISTING ABSORPTION SYSTEM.
H	1" LEAD	(WARM H_2O)	FROM STORAGE TANK IN CHARGE HOUSE TO PREWASH TANK.
J	1" LEAD	(SULPHITE SOLUTION)	. . . . .
K	2" LEAD	(FUME)	FROM NITRATOR TO EXISTING ABSORPTION SYSTEM
L	1½" LEAD	(BRINE RETURN)	FROM PREWASH TANK & NITRATOR COILS.
M	1½" LEAD	(COLD H_2O)	FROM EXISTING SUPPLY TO PREWASH TANK
N	1½" LEAD	(SODA SOLUTION)	FROM STORAGE TANK IN CHARGE HOUSE TO PREWASH TANK.
O	1½" LEAD	(BRINE SUPPLY)	TO PREWASH TANK & NITRATOR COILS.
P	1" LEAD	.	TO SPENT ACID TANK COIL.
Q	1" LEAD	(BRINE RETURN)	FROM . . . .
R	2" LEAD	(SPENT ACID)	FROM SPENT ACID TANK TO EGG HOUSE.
S	1" S.S.	(D.E.G.)	FROM GAUGE TANK TO NITRATOR
T	1" S.S.	(SPENT ACID)	FROM NITRATOR TO SPENT ACID TANK.

FOR DIAGRAMMATIC ARRANGEMENT OF PIPING SEE DRG. Nº 1868/M/21.
FOR MODIFICATIONS TO EXISTING PLATFORM SEE DRG Nº 1868/B/1
FOR SCHEDULE OF PLANT & MATERIAL SEE R.G.P.F./2 (PAGE 1) & R.G.P.F./3 (PAGE 1)

Drawing No. 2

General arrangement of Plant



REVISED PIPING IN CHARGE HOUSE

CONNECTION REFERENCE

	NEW SERVICE	EXISTING SERVICE
(A)	CONNECT ¾" LEAD (COV) TO EXISTING	2" LEAD (N/A TO NITRATOR N°2)
(B)	" ¾" SS (D.E.G.) " "	2" - (GLYCERINE TO CHARGE TANK)
(C)	" 1" SS. (N/A) " "	2" - (N/A TO NITRATOR N°1)
(D)	" ¾" LEAD (AIR) " "	¾" - (AIR TO PREWASH AGITATOR)
(E)	" ⅜" - (AIR) " "	⅜" - (AIR " " FAUCET)
(F)	" ½" - (AIR) " "	½" - (AIR " NITRATOR N°1 COILS)
(G)	" ¾" - (FUME) " "	3" LEAD (FUME FROM PREWASH TANK)
(H)	" 1" - (WARM H2O) TO "	1" - (SOFT WATER FROM CHARGE HOUSE)
(J)	" 1" - (SULPHITE SOL") " "	1" - (COLD SODA " " " ")
(K)	" 2" - (FUME) " "	3" - (FUME FROM NITRATOR N°1)
(L)	" 1" - (BRINE) " "	3" - (BRINE RETURN FROM NITRATOR N°1)
(M)	" ¾" - (COLD H2O) " "	¾" LEAD (FRESH WATER TO PREWASH TANK)
(N)	" ½" - (SODA SOL") " "	½" - (H2O / SODA FROM CHARGE HOUSE)
(Q)	" 1" - (BRINE) " "	3" - (BRINE SUPPLY TO NITRATOR N°1)
(P)	" 1" - (BRINE) " "	3" - (" " " " " ")
(Q)	" 1" - (BRINE) " "	3" - (BRINE RETURN FROM " ")
(R)	" 2 LEAD (S/A) " "	2" - (WASTE ACID TO EGG HOUSE)

TANK AND VALVE REFERENCE

FOR VESSELS (TG) ETC - SEE SCHEDULE R.G.P.F./2

FOR VALVES (V.G) ETC - " " R.G.P.F./3.

DRAWING REFERENCE

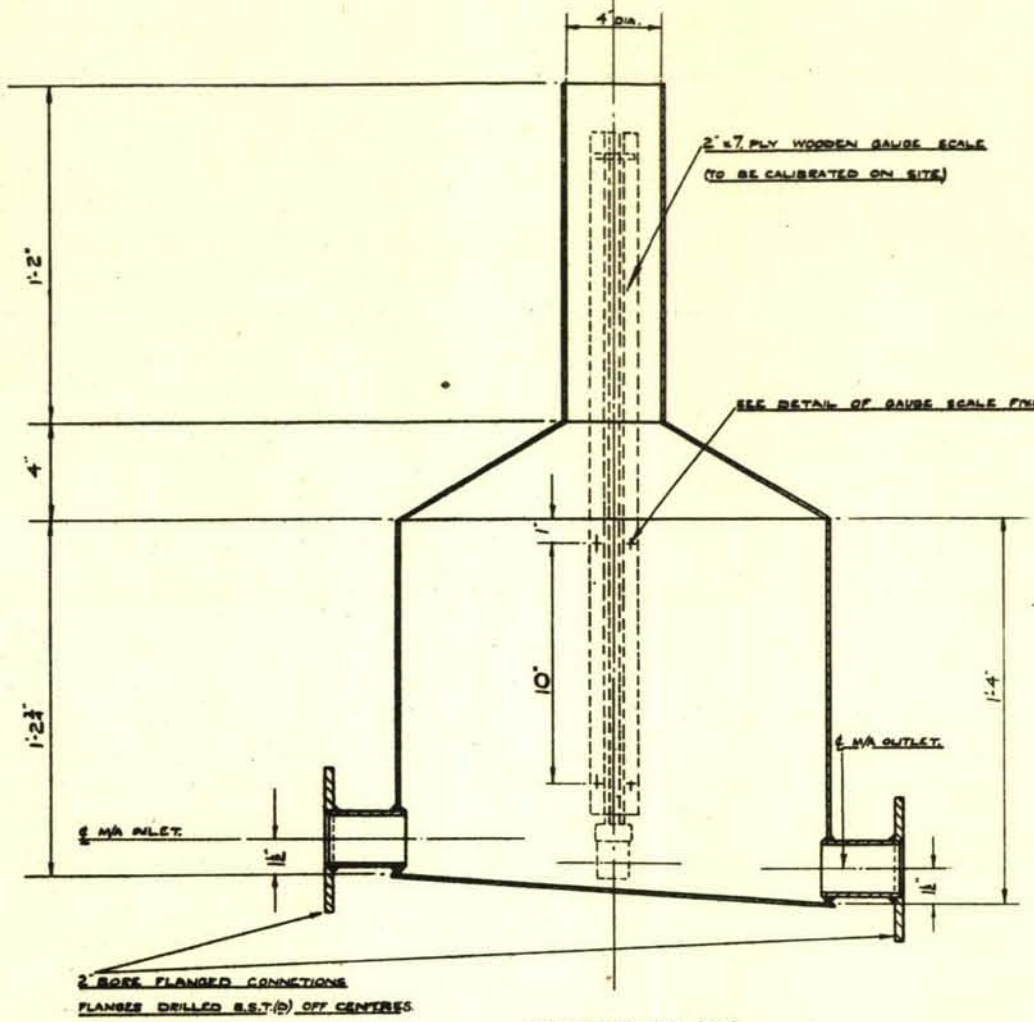
FOR DETAILS OF D.E.G. GAUGE TANK	SEE DRG N° E.D(D)1828/1-1/14
" " " M/A "	" " " " " -/15
" " " NITRATOR BASE	" " " " " -/16
" " " " COVER	" " " " " -/20[A]
" ARRGT OF NITRATION PLANT	" " " " " -/5[C]
FLOW SHEET	" " " " " -/11[B]
FOR DETAILS OF PLATFORMS	" " " " -/8/1

COLOUR REFERENCE

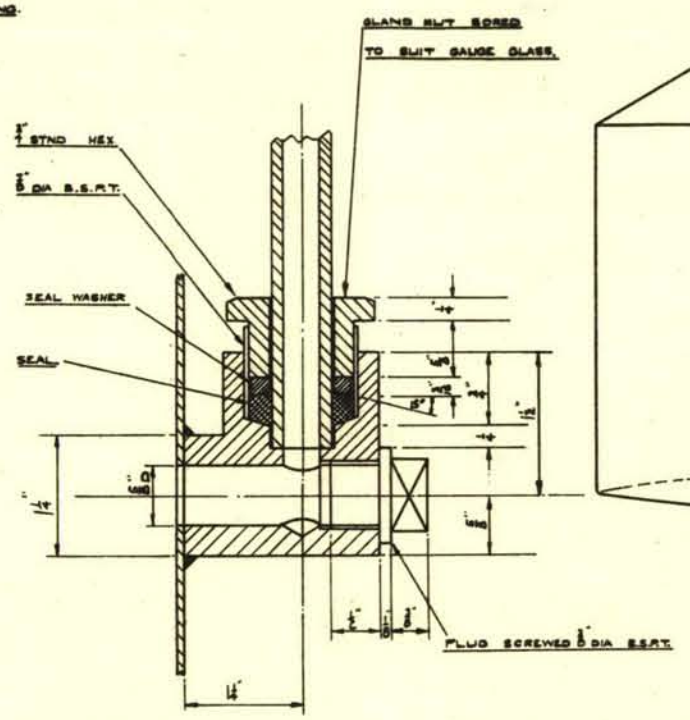
MIXED ACID	———
D.I.G.	———
D.E.G.D.N	———
SPENT ACID	———
FUME	———
AIR	———
SODA SOLUTION	———
SULPHITE "	———
WARM SOFT WATER	———
COLD WATER	———
BRINE	———
C.O.V.	———

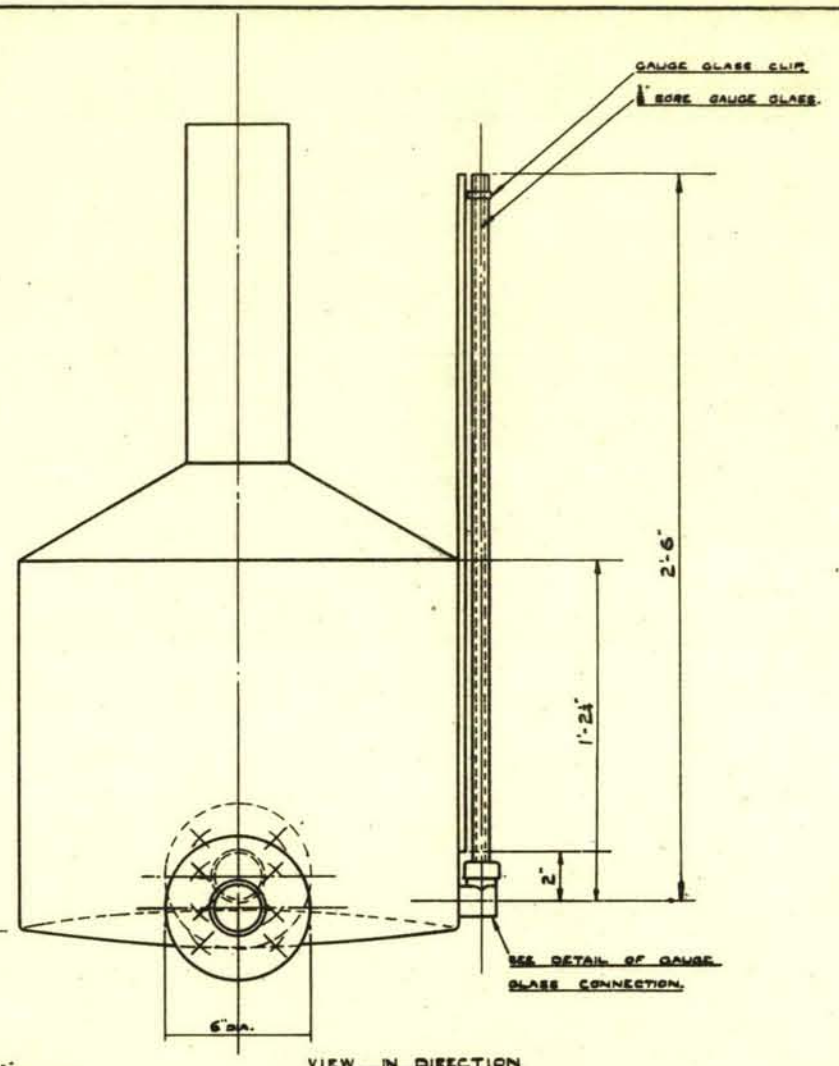
Drawing No. 3

Layout of piping in
Nitration House

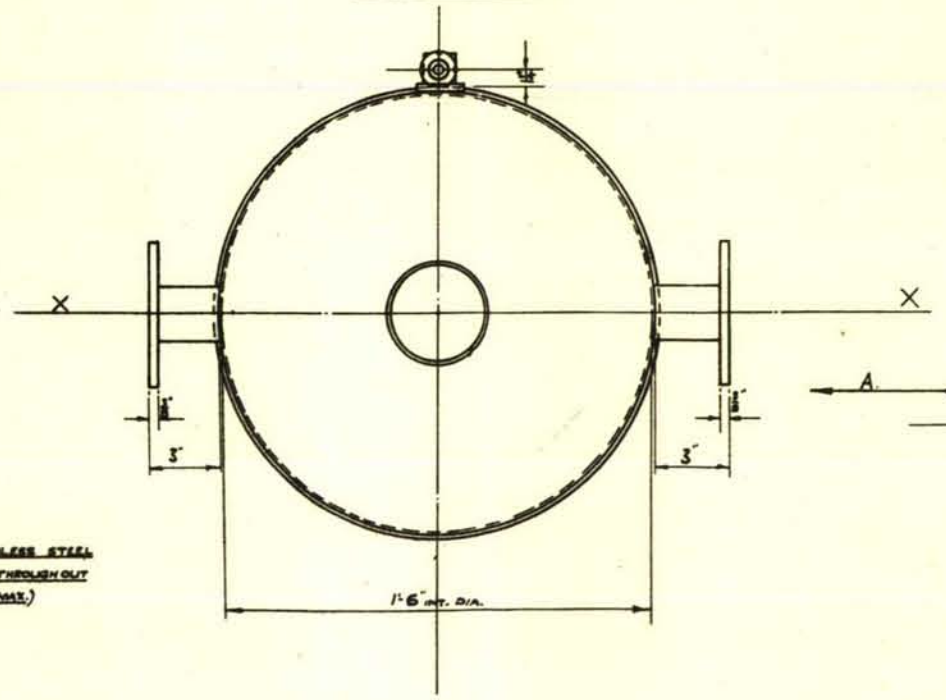


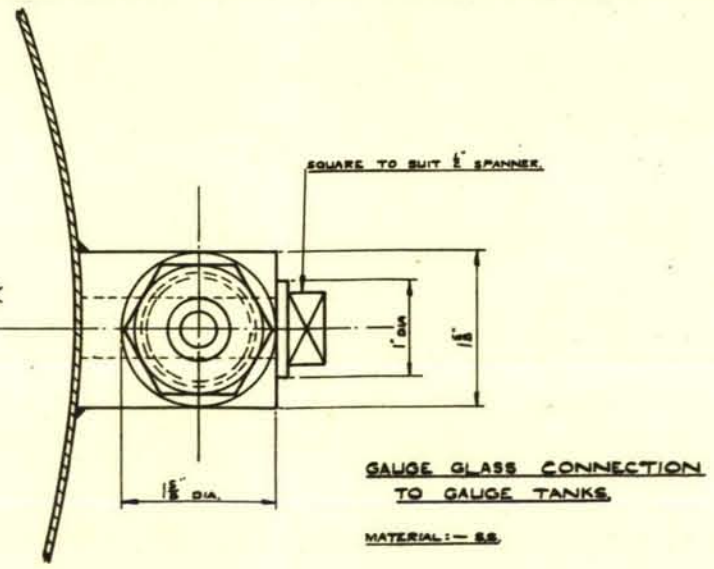
SECTION ON 'XX'





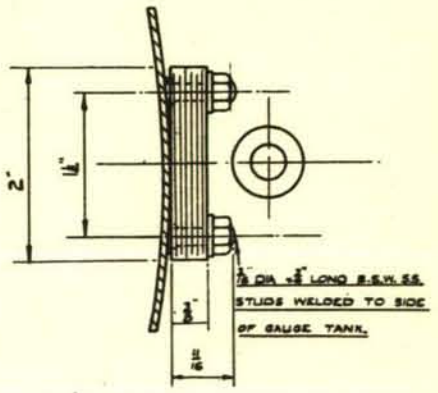
VIEW IN DIRECTION OF ARROW 'A'





GAUGE GLASS CONNECTION TO GAUGE TANKS.

MATERIAL :— S.S.



NOTES.

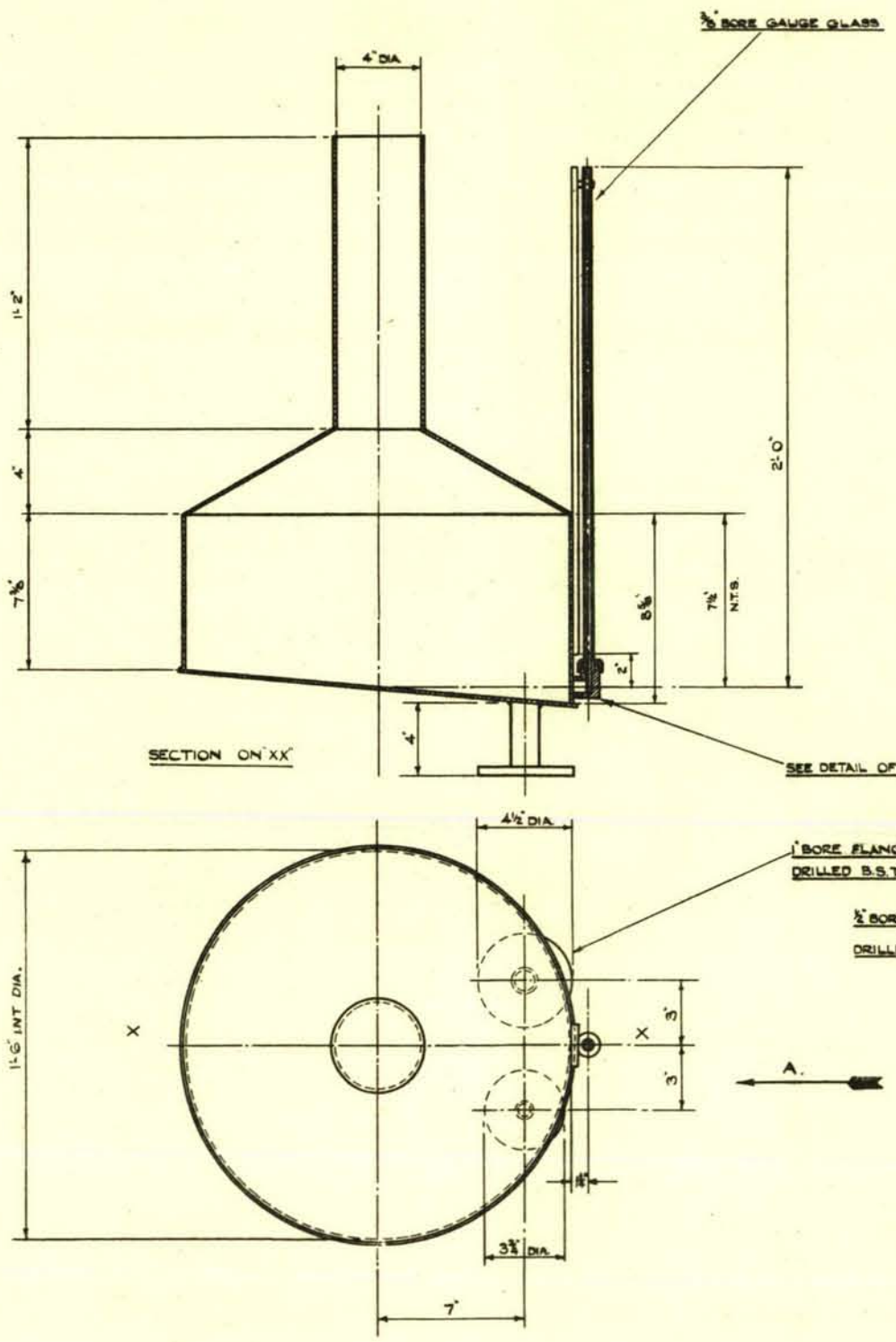
MATERIAL :- 16 S.W.G. STAINLESS STEEL WELDED CONSTRUCTION THROUGH OUT

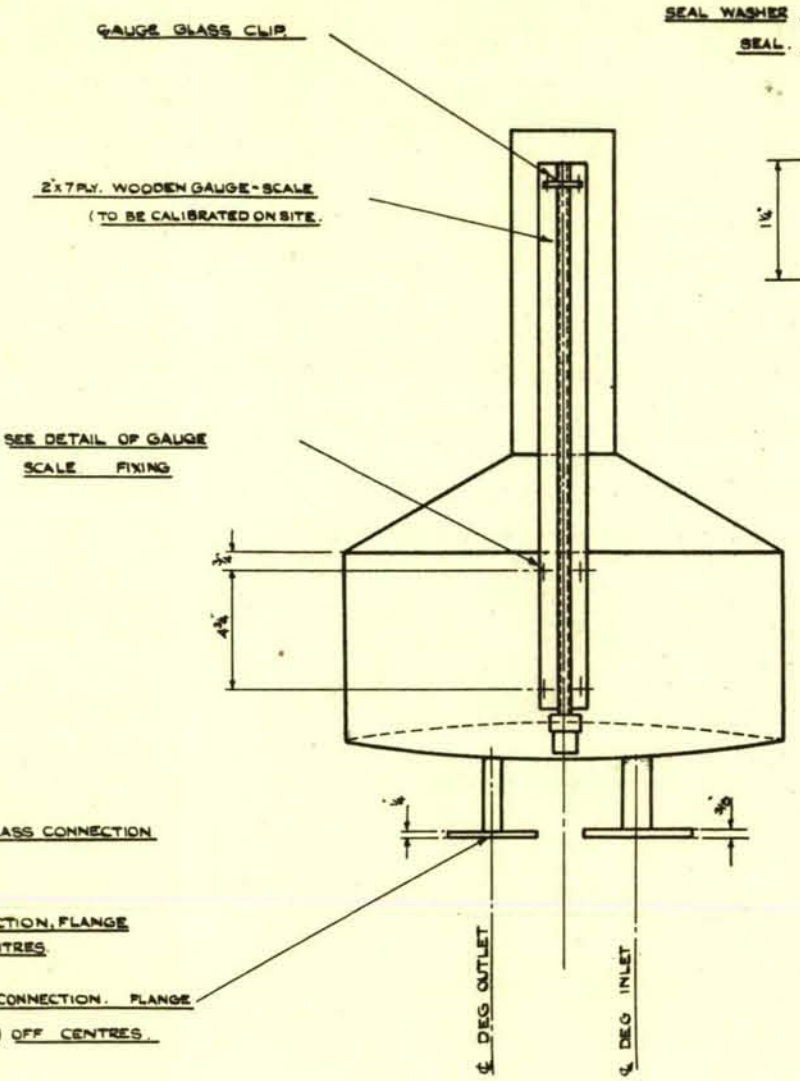
CAPACITY 2·6 CU. FT. (MAX.)

No REQD. :— 1.

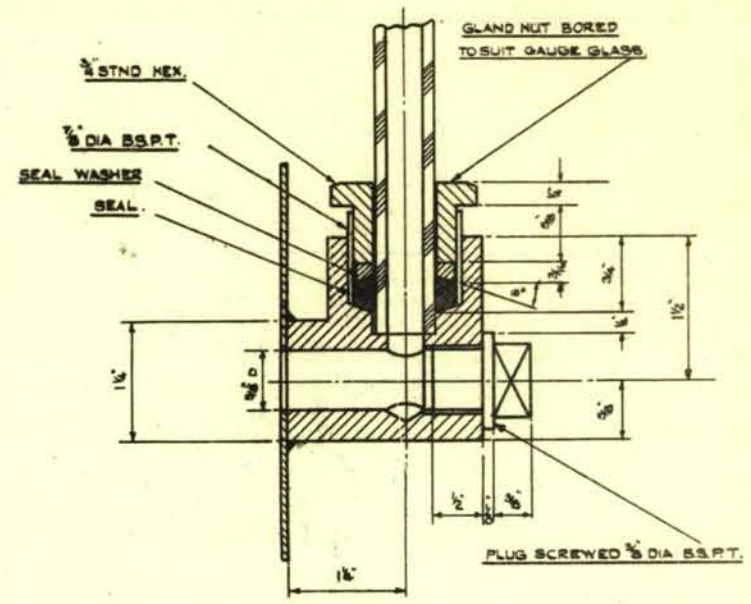
Drawing No. 4

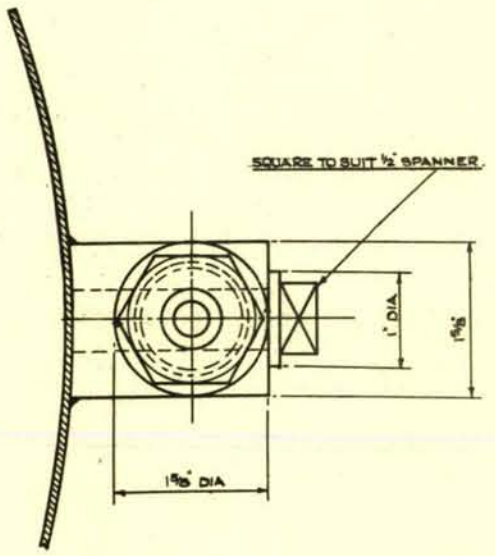
Mixed Acid Gauge Tank



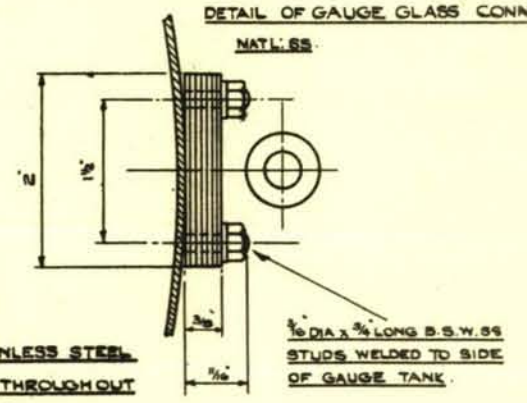


VIEW IN DIRECTION OF ARROW 'A'





DETAIL OF GAUGE GLASS CONNECTION
NAT. SIZE



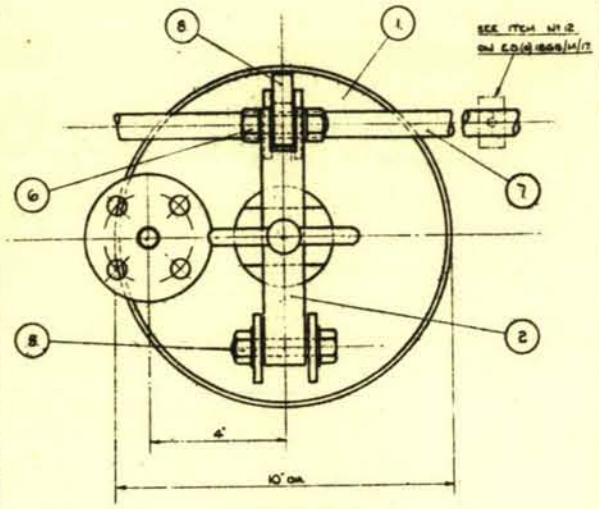
NOTES

MATERIAL:- 16 S.W.G STAINLESS STEEL WELDED CONSTRUCTION THROUGHOUT
CAPACITY 1·5 CU. FT (MAX)

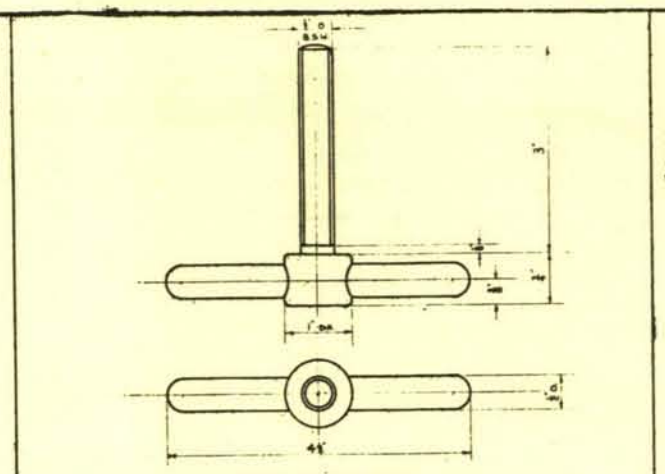
NO REQD:- 1.

Drawing No. 5

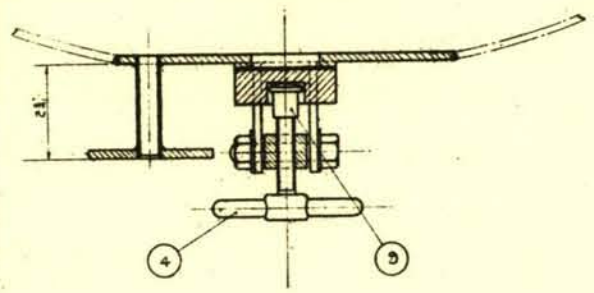
DEG Gauge Tank

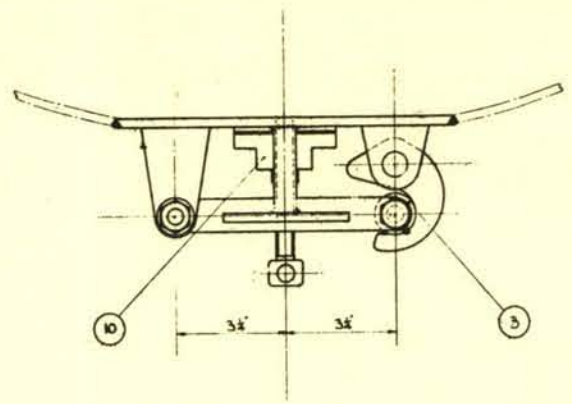


INVERTED PLAN

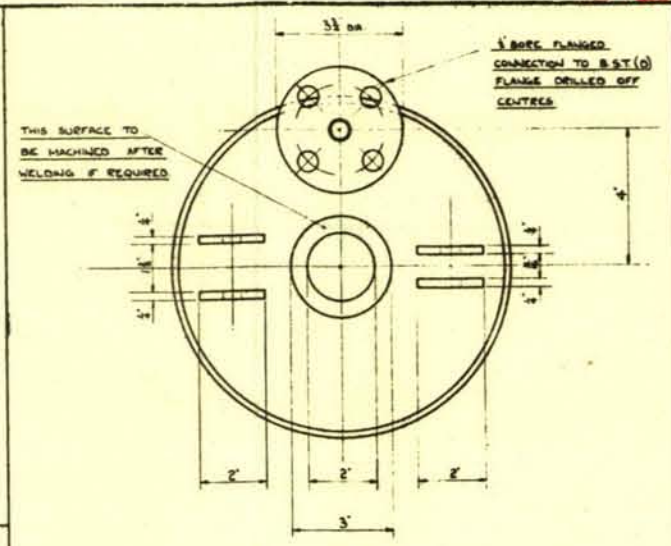


DETAIL OF ITEM Nº 4.
Nº REQ'D:- 10FF. MATERIAL- S.S.

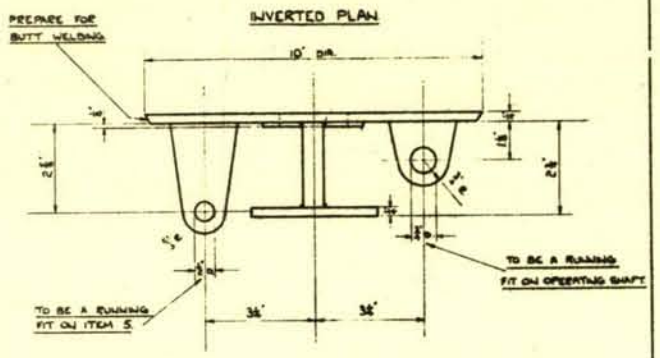




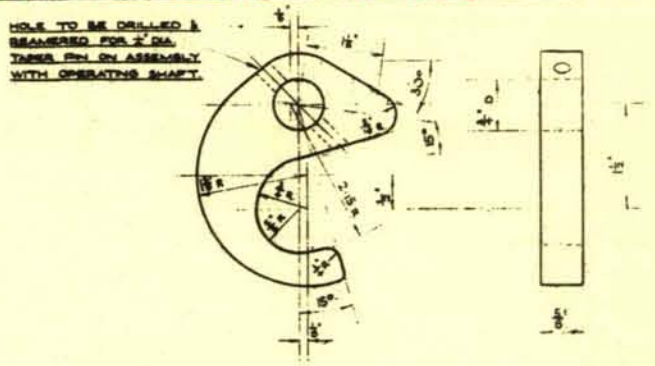
ARRANGEMENT OF NITRATOR DROWNING VALVE



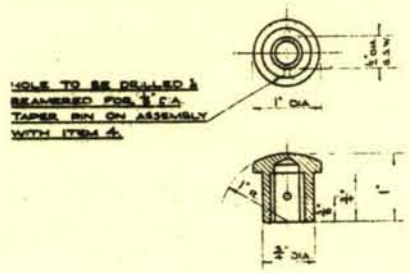
INVERTED PLAN



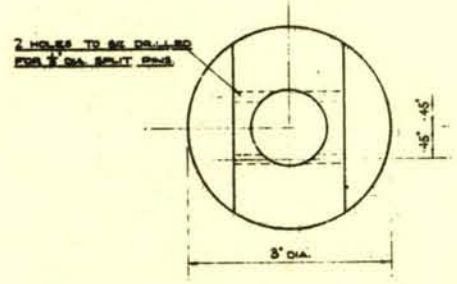
DETAIL OF ITEM Nº1.
Nº REQ'D:- 1 OFF. MATERIAL- S.S.

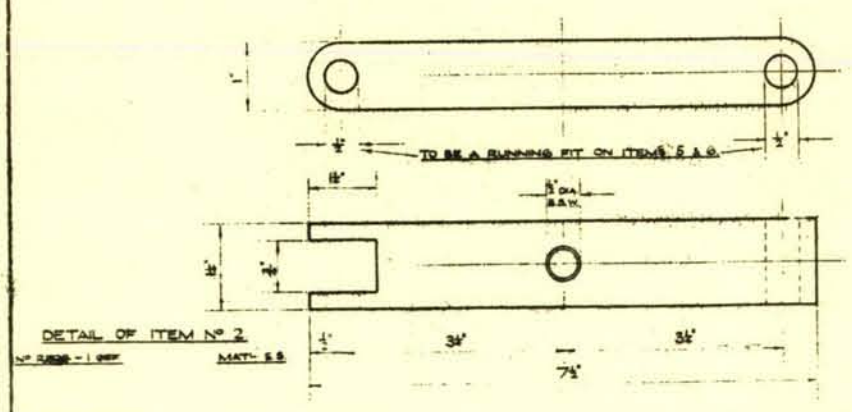


DETAIL OF ITEM Nº 8.
Nº REQ'D - 1 OFF MAT'- S.S.

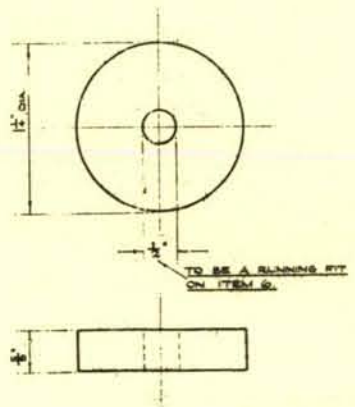


DETAIL OF ITEM Nº 9.
Nº REQ'D - 1 OFF MAT'- S.S.

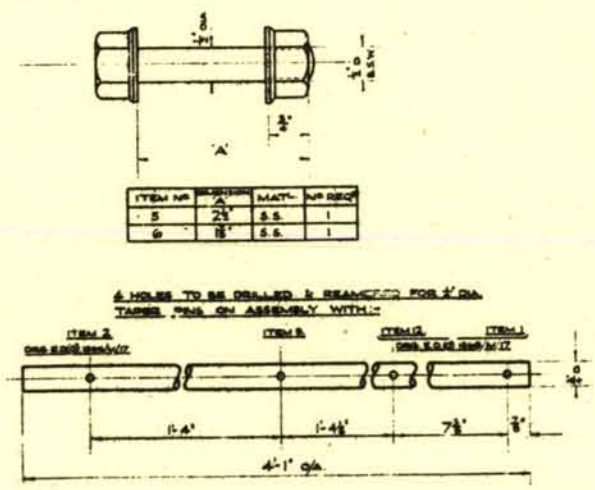




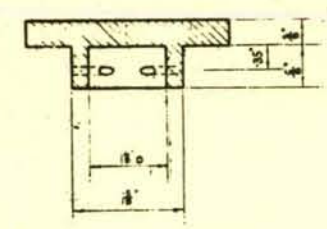
DETAIL OF ITEM Nº 2.
Nº REQ'D - 1 OFF. MAT'- S.S.



DETAIL OF ITEM Nº 3.
Nº REQ'D - 1 OFF. MAT'- S.S.



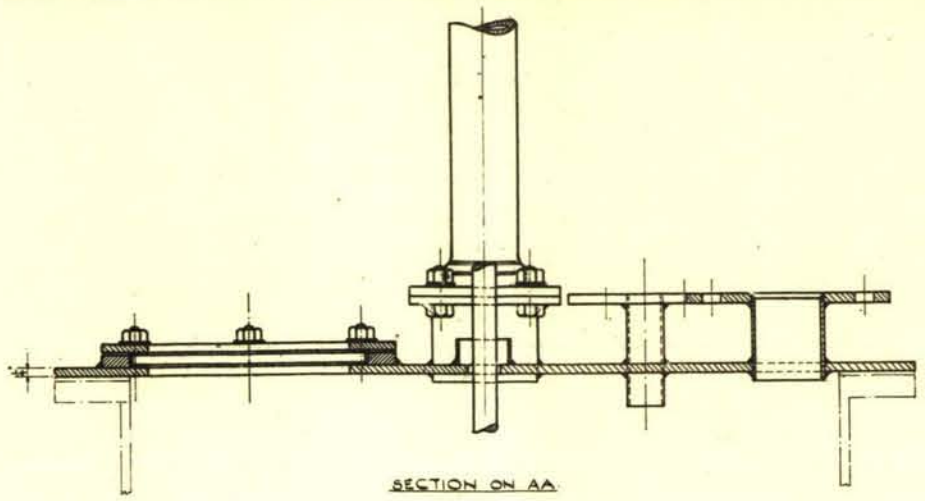
DETAIL OF ITEM Nº 7.
Nº REQ'D - 1 OFF. MAT'- S.S.



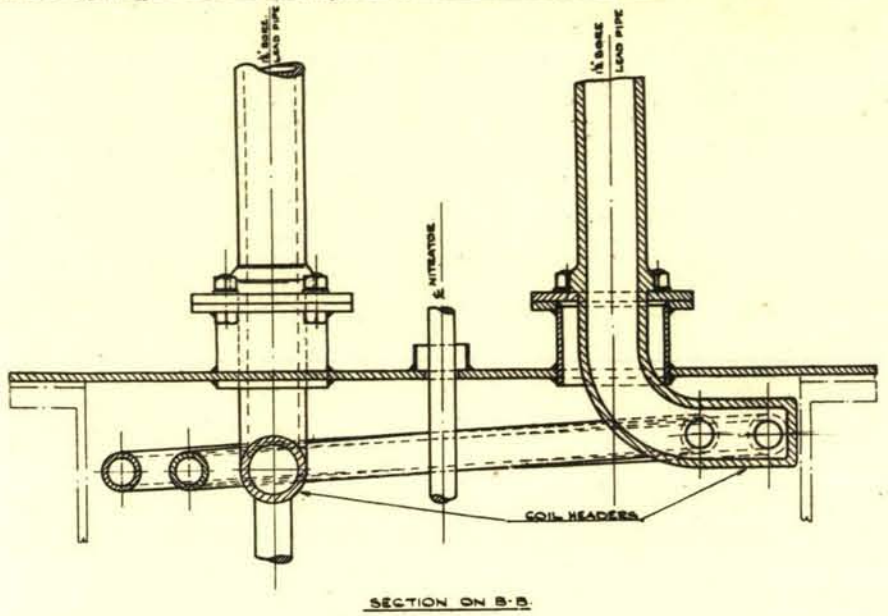
DETAIL OF ITEM Nº 10.
Nº REQ'D - 1 OFF. MAT'- S.S.

Drawing No. 6

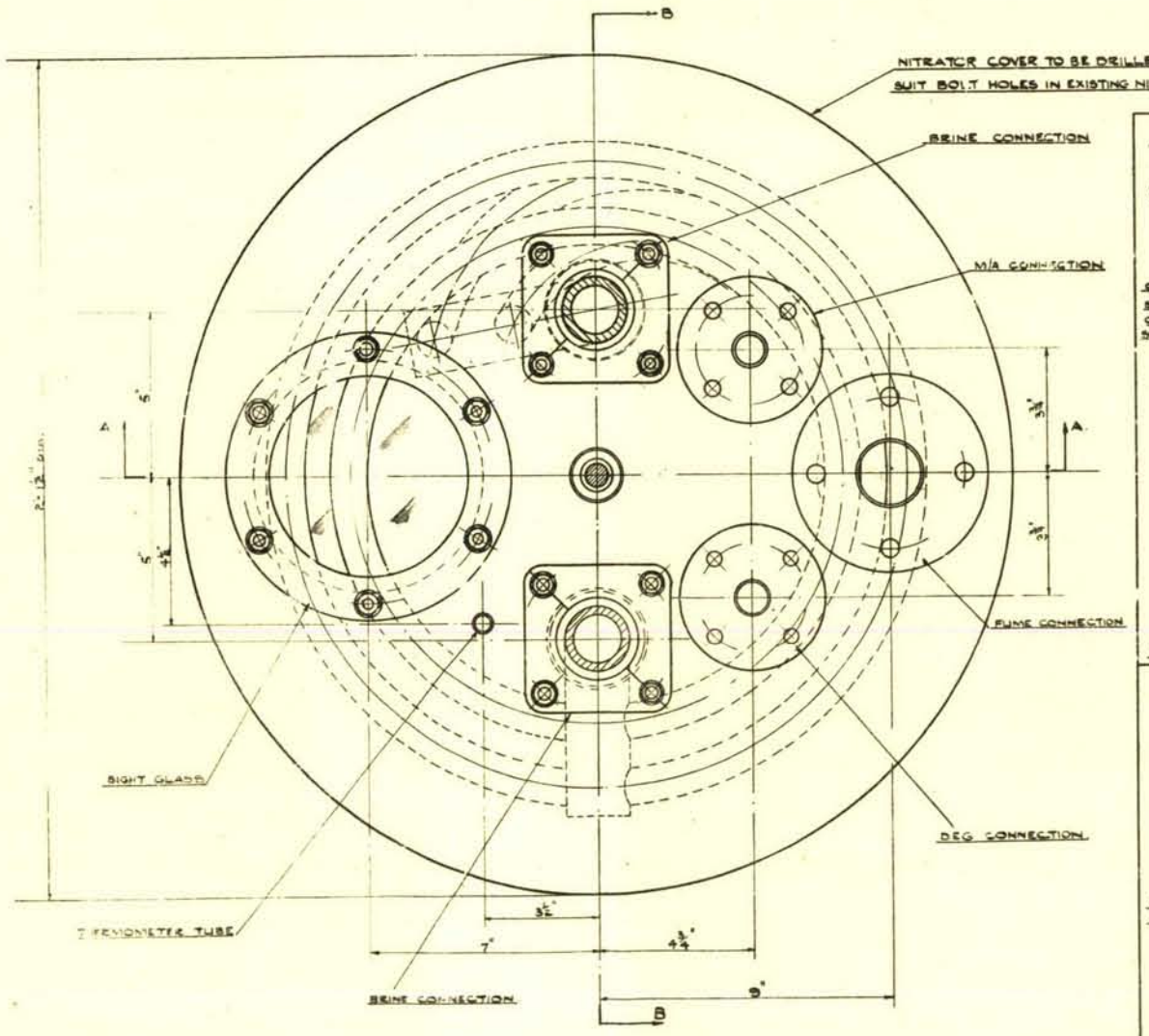
Arrangements and details of
Nitrator Drowning Valve

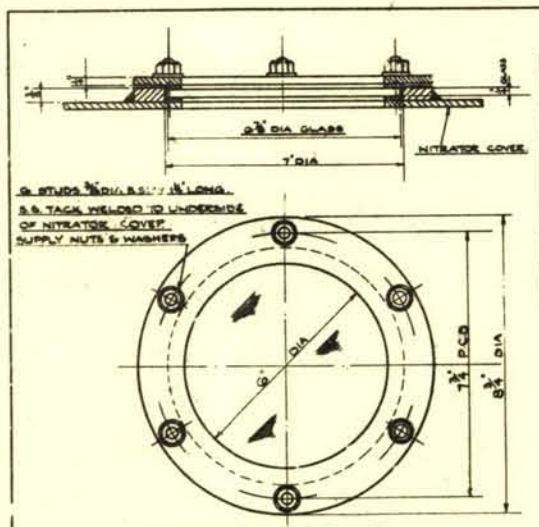


SECTION ON AA



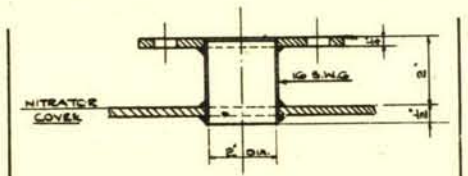
SECTION ON B-B



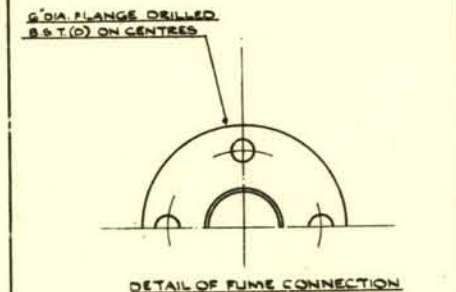


DETAIL OF SIGHT GLASS

N° REQ'D 1 OFF — MAT'L S.S.

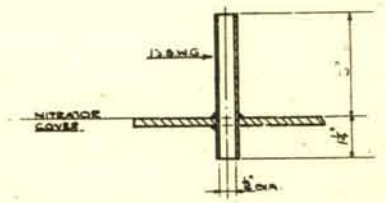


6" DIA FLANGE DRILLED 8 S.T.(D) ON CENTRES



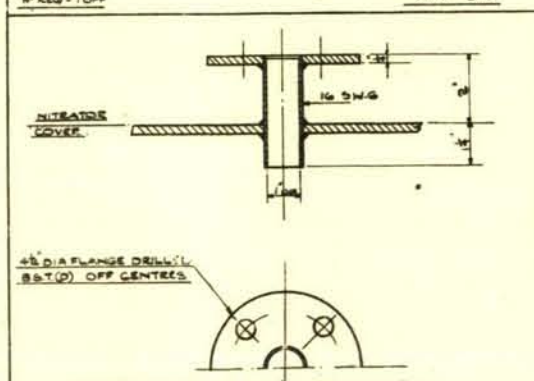
DETAIL OF FUME CONNECTION

N° REQ'D 1 OFF. — MAT'L S.S.



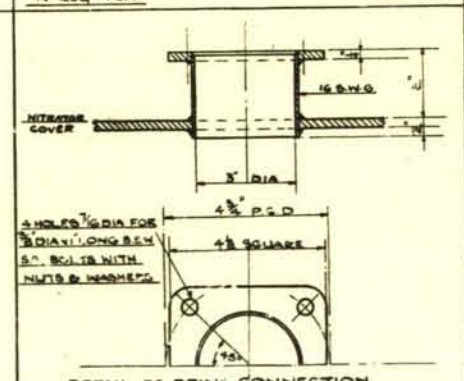
DETAIL OF THERMOMETER TUBE

N° REQ'D 1 OFF — MAT'L S.S.



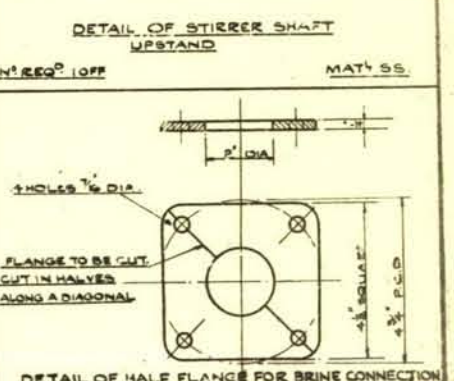
DETAIL OF D.E.G. & M/A CONNECTIONS

N° REQ'D 2 OFF — MAT'L S.S.



DETAIL OF BRINE CONNECTION

N° REQ'D — MAT'L S.S.



DETAIL OF STIRRER SHAFT UPSTAND

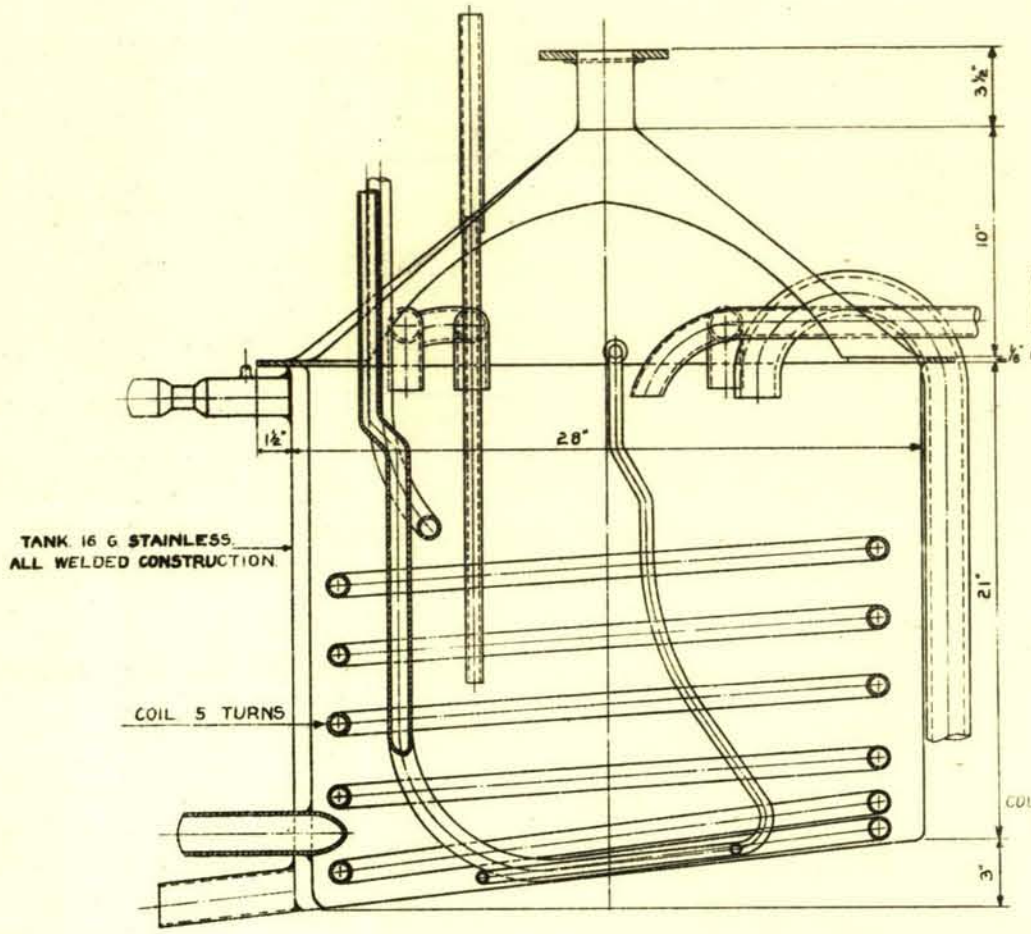
N° REQ'D 1 OFF — MAT'L S.S.

DETAIL OF HALF FLANGE FOR BRINE CONNECTION

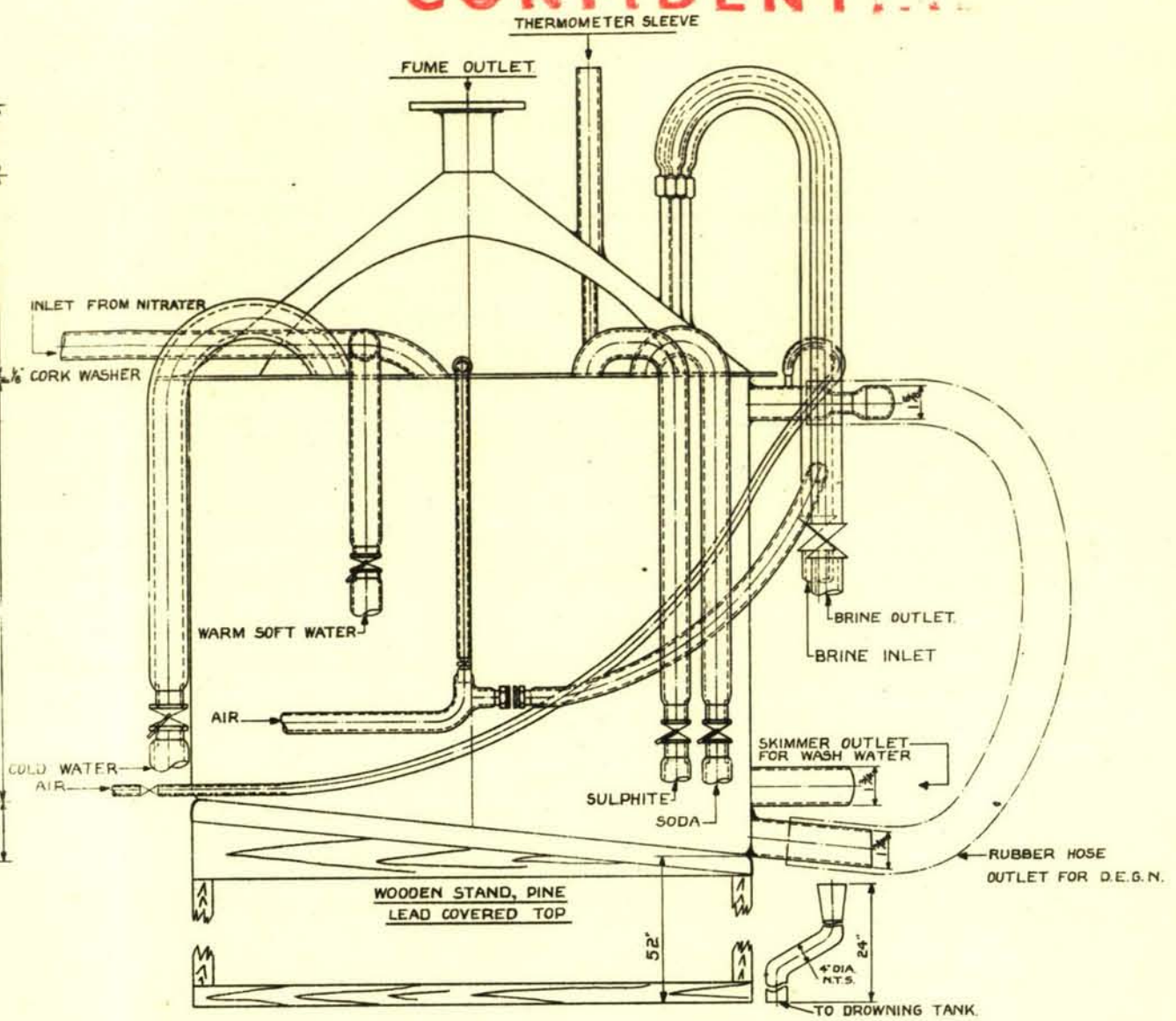
N° REQ'D 2 — MAT'L S.S.

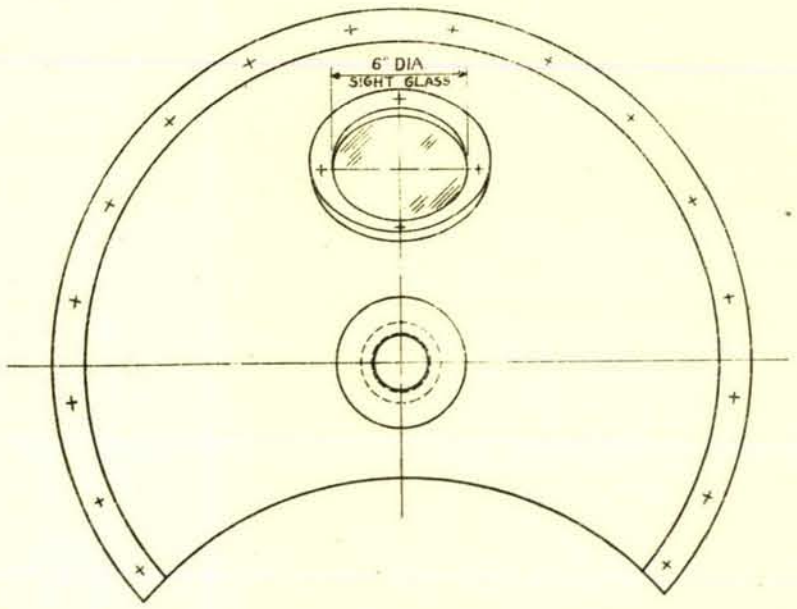
Drawing No. 7

Arrangements and details
of Nitrator Cover

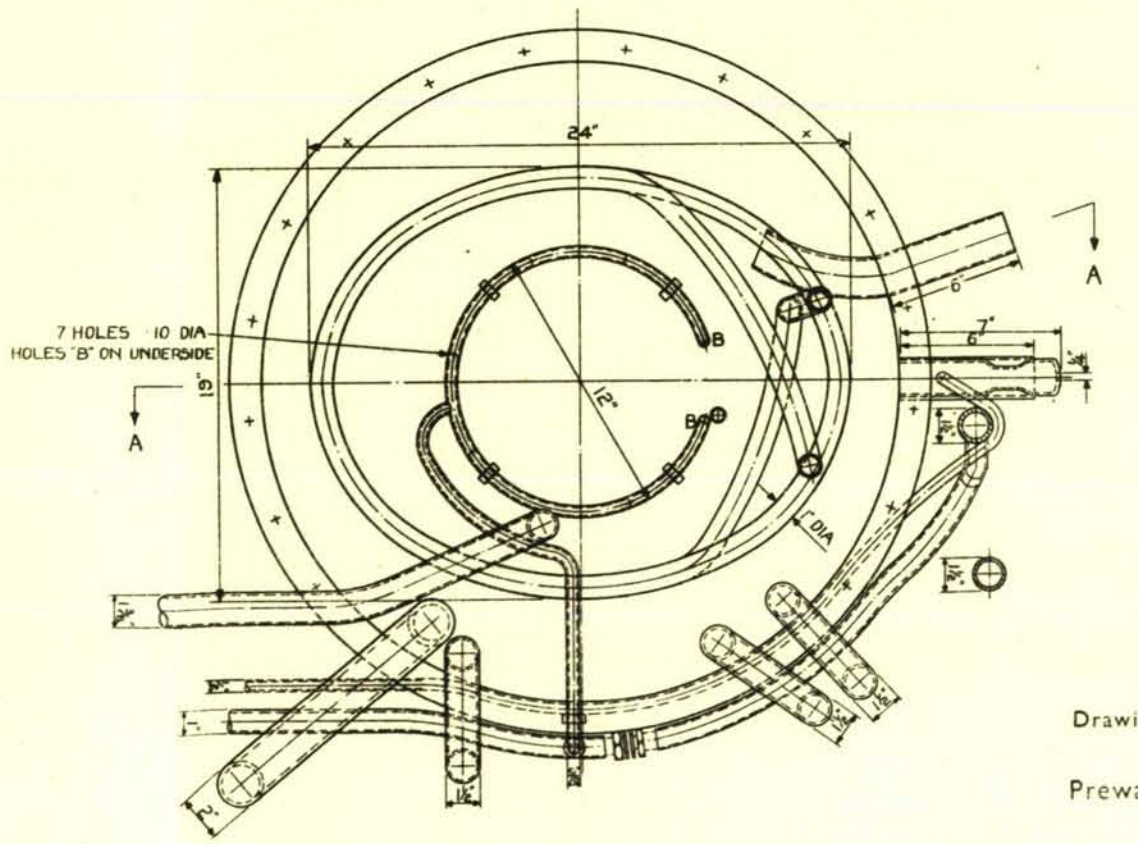


SECTIONAL ELEVATION A.A.





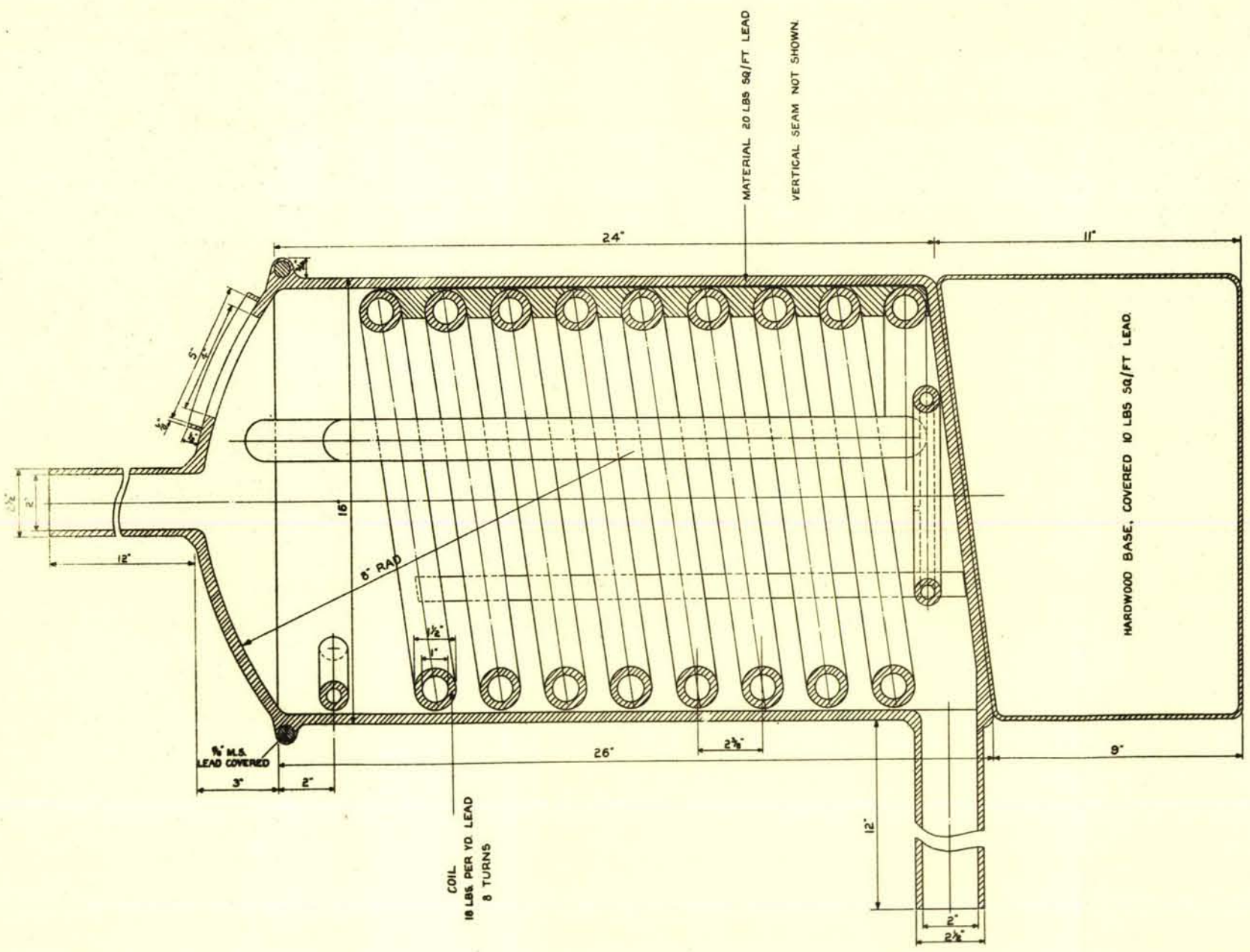
PLAN. (LID)

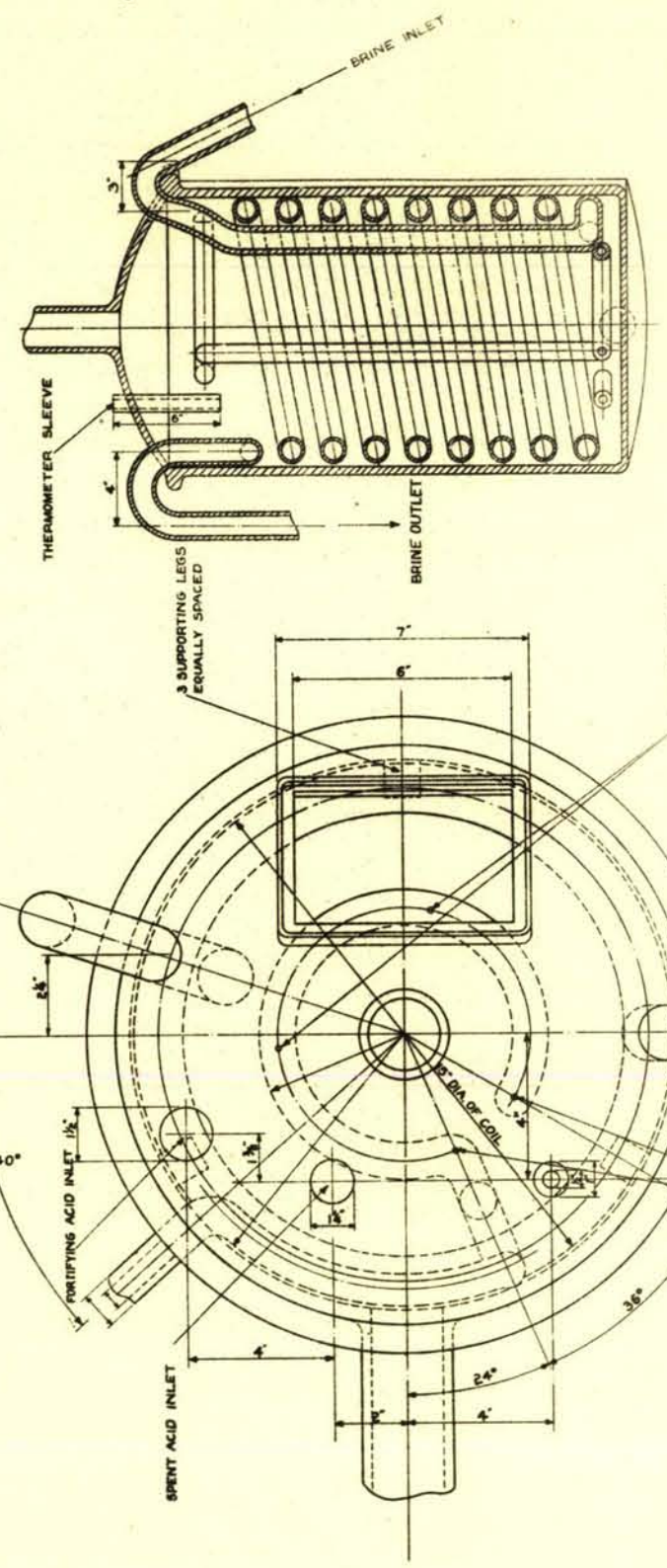


PLAN
WITH LID REMOVED.

Drawing No. 8

Prewash Tank





Drawing No. 9

Waste Acid Tank

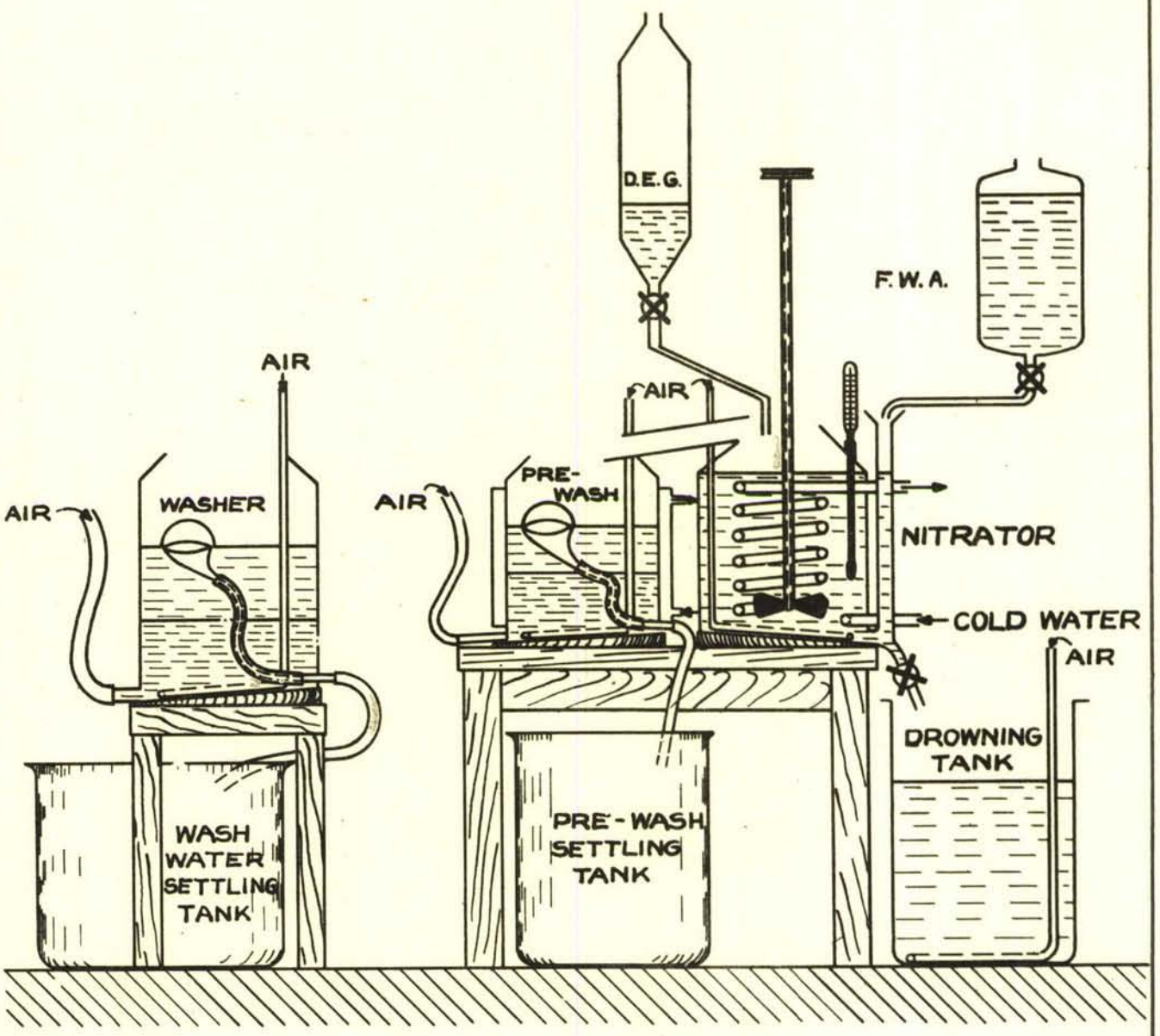


FIG. I

LAY-OUT OF SEMITECHNICAL SCALE PLANT

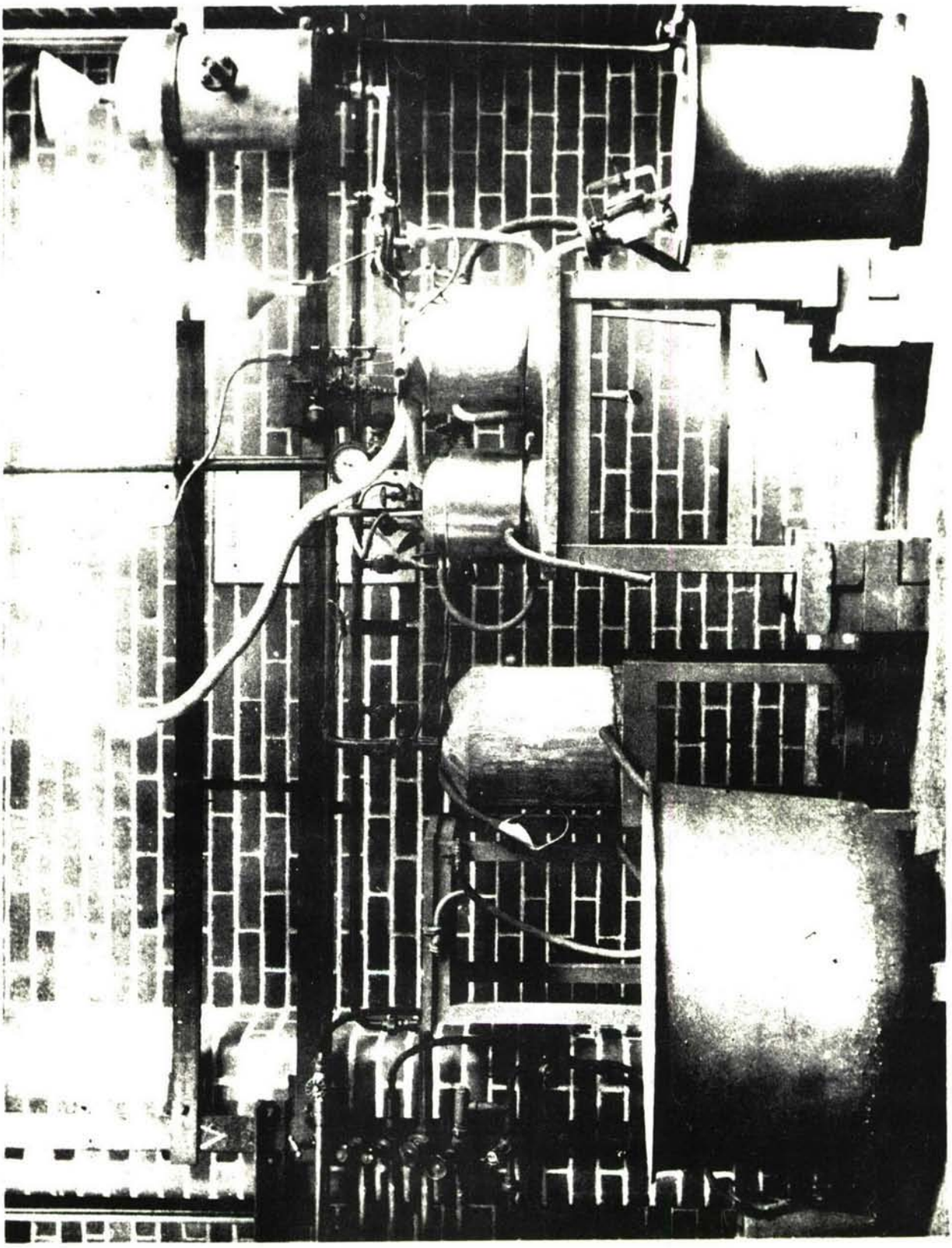


FIG. II. SEMITECHNICAL SCALE PLANT.

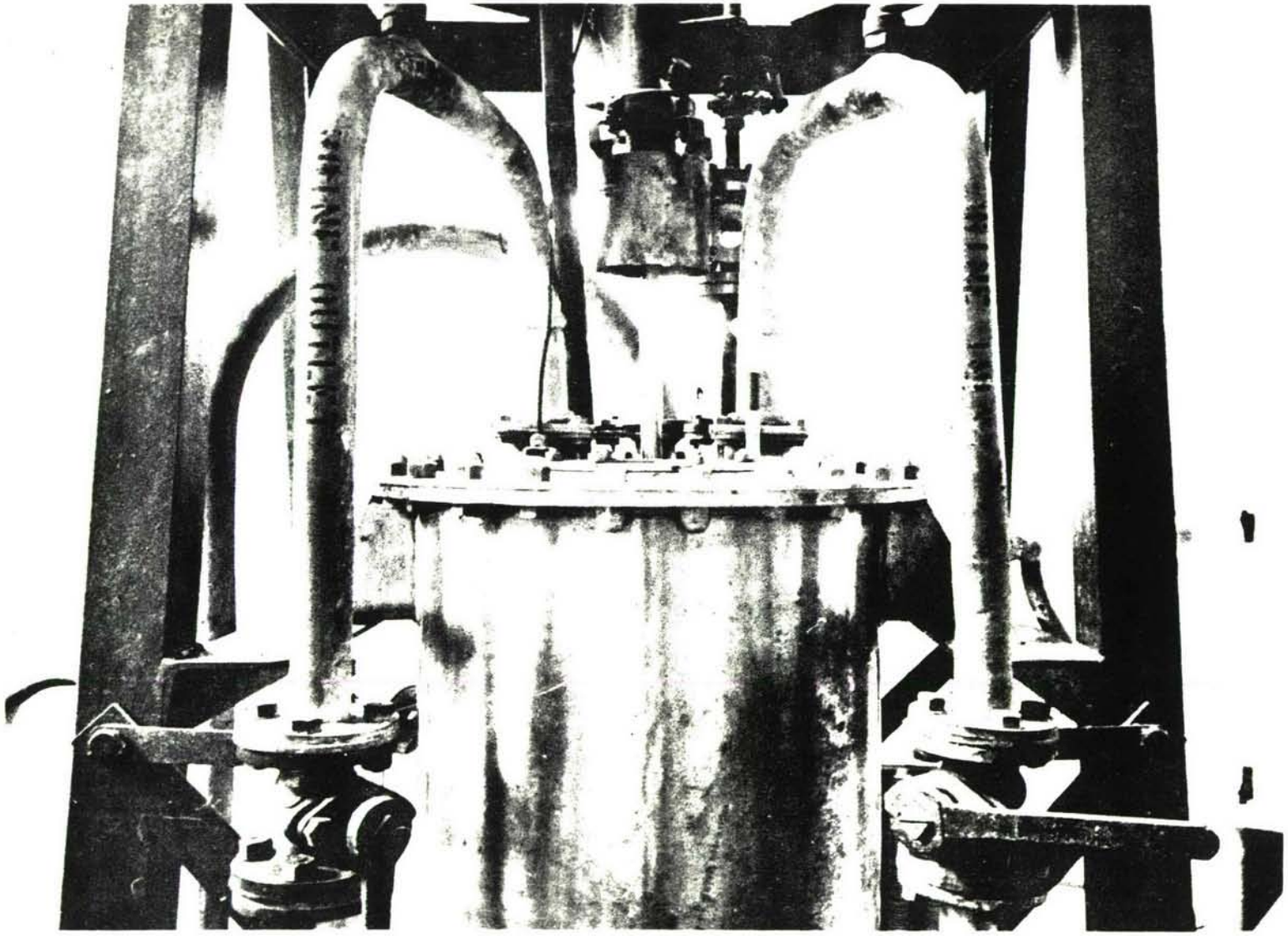


FIG. III. BATCH PILOT PLANT – NITRATOR.

FIG. IV. BATCH PILOT PLANT - PREWASH TANK.

August 4, 81

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