MANUFACTURING PROCESS FOR TETRYL - WALTHAM ABBEY.

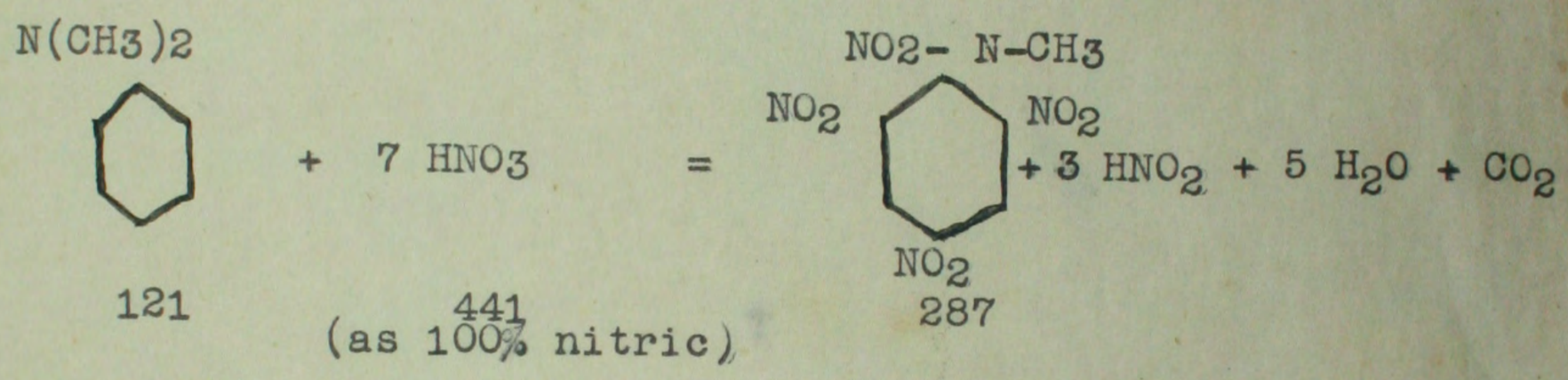
PROCESS FIRST ISSUED 1942.

Nitration Batch is 190 lbs. crude Tetryl = 175 lbs. finished material.
Purification Batch is 360 lbs. finished Tetryl.

MANUFACTURE OF TETRYL.

PRINCIPLES.

Dimethylaniline is dissolved in 15 times its weight of 90% sulphuric acid. The solution of dimethylaniline sulphate is injected into 90% nitric acid at a temperature of 70°C. The tetryl crystallises and is removed from the spent acid by filtration; that which passes the filters is removed manually after the acid has settled. The waste acid is then passed to the detetrylation plant in which the tetryl in solution is destroyed by fume-off and the residual nitric acid distils off. The remaining concentrated sulphuric acid is used in the concentration of weak nitric acid obtained from Absorption Towers.



The crude tetryl is washed and purified by solution in acetone. The acetone solution is filtered and the tetryl precipitated by the controlled addition of water. The pure tetryl is then finished as required.

HISTORICAL.

The process is very similar to that established at Waltham Abbey between 1914 and 1918. The main differences are in the design of plant and the strengths of acids used.

N.B. The purified differs from the output of finished material from coming onwards, owing to the fact that dust is reworked.

RAW MATERIALS.

~~Dimethylaniline 96 lbs./nitration~~
~~= .795 lb. mol.~~
~~Consumption = .549 lbs/lb. of tetryl~~

~~Specification No. C.W.D.1097.~~

HAZARDS.

Tetryl is an explosive and is manufactured in accordance with Danger Building practice.

It is soluble in perspiration and when absorbed under the skin may cause tetryl dermatitis. Precautions which are taken against this are given as an appendix.

The usual precautions are taken when handling strong acids.

Capacity of Plant.

The capacities are given as lbs. per shift for continuous working.

UNIT	Tetryl per unit		No. of Units.	Total output Finished Tetryl
	Crude	Purified		
Nitrating Houses.	570	525	5	2,600
Boiling House				
Mechanical Vats.	600	550	4	2,200
Non-mechanical vat.	450	420	1	420
Wringing Houses.				
Electrical wringers.	900	840	4	3,350
Steam wringer.	1,000	900	1	900
Purification House.	1,600	1,440	2	2,880
Corning House.	-	3,000	1	2,250
Sieving House.				
Vibro Sieve.	-	2,500	2	3,750
Rotary Sieve (For dust or Xtal).	-	4,000	1	4,000
Stoves.	-	-	10	2,600 Xtal. 2,000 Corned

Sulphuric Acid	1,430 lbs. of sulphuric acid/ nitration. Strength 90% ± 1. Produced in acid factory to S.G. 1.825. Consumption 1.1 lbs. 100% H_2SO_4 / lbs. of tetryl.
Nitric Acid	830 lbs. of nitric acid/ nitration. Strength 88% ± 2. Produced in Acid Factory to S.G. 1.490. Usage is 12.7 lb. mol. Consumption is 2.0 lbs. 100% HNO_3/lb. of tetryl.
Acetone.	700-720 lbs. of acetone is used/pptn. S.G. from .795-820. Consumption is 0.8 lbs. acetone/lb. of tetryl. Specification No. R.G.P.F. S.106.B.
Caustic Soda.	Used in acetone recovery. 2 gallons of 6.25% solution are added to each still S.G. = 1.070 at 20°C. Consumption is 1/2 lb/100 lbs. tetryl. Specification No. C.W.D. 487 D. Grade 1.
Gum Arabic.	Used in 10% solution. S.G. = 1.037. 1 gallon is incorporated with 100 lbs. of purified tetryl. Consumption 1.3 lbs. gum/100 lbs. tetryl. Specification No. C.S. 1242.

N.B. The purified differs from the output of finished material from corning onwards, owing to the fact that dust is reworked.

Raw Materials.

Dimethylaniline. 96 lbs/nitration
= .795 lb. mol.
Consumption = .549 lbs./lb. of Tetryl.
Specification No. C.W.D. 1097.

acid/

S.G. 1.825.

Consumption 1.1 lbs. 100% H_2SO_4 /
lbs. of tetryl.

Nitric Acid

830 lbs. of nitric acid/ nitration. Strength 88% ± 2. Produced in Acid Factory to S.G. 1.490. Usage is 12.7 lb. mol. Consumption is 2.0 lbs. 100% HNO_3/lb. of tetryl.

Acetone.

700-720 lbs. of acetone is used/pptn.
S.G. from .795-820.
Consumption is 0.8 lbs. acetone/lb. of tetryl.
Specification No. R.G.P.F. S.106.B.

Caustic Soda.

Used in acetone recovery.
2 gallons of 6.25% solution are added to each still S.G. = 1.070 at 20°C. Consumption is 1/2 lb/100 lbs. tetryl.
Specification No. C.W.D. 487 D.
Grade 1.

Gum Arabic.

Used in 10% solution.
S.G. = 1.037. 1 gallon is incorporated with 100 lbs. of purified tetryl.
Consumption 1.3 lbs. gum/100 lbs. tetryl.
Specification No. C.S. 1242.

Description of process.

(a) Sulphation and Nitration.

The D.M.A. (96 lbs.) is blown direct from the drum in which it is supplied, to the Gauge Tank. The air pressure used is controlled by reducing valves so that it cannot exceed 10 lbs./sq. inch. The sulphuric acid (1430 lbs.) is run from a Storage Tank in the Charge House into the sulphuric acid gauge tank and thence into an egg. It is blown directly into the sulphator using a pressure of 30 lbs./ sq. inch. It takes five minutes to charge the D.M.A. gauge tank and 15 minutes to charge the sulphator. When charging is complete the air agitation and cooling water are turned on and the D.M.A. injector opened to an amount fixed by the Foreman so that the temperature remains at about 30°C. The time of sulphation varies from 50 mins. to 100 mins. according to the temperature of the cooling water. When the addition of D.M.A. is completed the air agitation and cooling water are turned off and the solution of D.M.A.S. allowed to run into the egg connected to the bottom of the sulphator (3 mins.). The solution is then blown from the egg to the nitrator feed tank which is on a high level platform.

The nitric acid (830 lbs.) is run from a storage tank in the Charge House into the nitric acid gauge tank and thence to the egg. It is blown directly into the nitrator using a pressure of 30 lbs. sq. inch. The charging of the nitrator takes 10 minutes. When charging is complete, hot water is circulated through the coils of the nitrator and lifted back to the hot water tank on the high level platform by means of the steam injector. When the temperature of the nitrator reaches 50°C. the injection into the nitrator of the solution of D.M.A.S. is started. The hot water is turned off when the

temperature reaches 68°C. and the cooling water turned on at 70°C. so that the temperature remains at 70°C. throughout the injection which takes between 50 and 70 minutes. After injection is completed and cooling water is turned off hot water is, if necessary, turned on so that the temperature remains at 70°C. for a further 15 minutes. The cooling water is then turned on again and the contents of the nitrator cooled to 25°. This takes from 10-20 minutes depending on the temperature of the cooling water.

The contents of the nitrator are then run off being shared evenly between the two lead filters. After 30 minutes settling the waste acid is run off into the waste acid egg. 65% of the crude tetryl remains on the filter. The remainder is carried into the waste acid storage tanks.

Each nitration and the sulphation of the next charge are done simultaneously. One nitrating house is worked by 1 C/H., the complete cycle being from 2 to 2.1/2 hours.

DISPOSAL OF NITROUS FUMES.

(a) Nitrator.

The fumes from all the nitrating houses are collected in a 9 inch earthenware fume main. The branches to individual houses have a stainless steel butterfly valve, so that when the house is not working it can be disconnected from the main. The fume is drawn through the main and the absorption towers by a stainless steel Aerex fan of capacity 400 cu.ft./min. driven by a 2 H.P. variable speed electric motor. A stainless steel air ejector in a stainless steel fume shaft is also fitted to the towers to operate in case of a failure of power or other emergency. When air ejector is not in use the shaft is closed by a butterfly valve. A similar valve is provided for closing the fume pipe to the fan in the event of failure.

Before reaching the absorption towers the fume is passed through two aluminium fume coolers arranged in parallel. The temperature of the fume reaching the cooler varies, with the number of houses working and the external temperature, from 30°C. to 45°C. After the cooler, the temperature lies between 15°C. and 25°C. About 30 lbs. per nitration of nitric acid, varying in strength from 85 - 90%, are condensed per nitration. This is collected in 4 Woolf's bottles at the bottom of the coolers and runs from there into a stainless steel gauge vessel. When 12 inches of acid (300 lbs.) has been collected in this vessel it is run into an egg and blown into one of the charge house nitric acid storage tanks.

From the fume coolers the fume main passes to a system of 10 earthenware absorption towers, 3 ft. in diameter and 18 ft. in height (overall). They are packed with earthenware rings. Each tower has an air lift constructed from 2 inches earthenware pipes with conical flanged joints. The weak acid is run off from the end tower into a stainless steel weak acid tank and sent to the acid factory by egg. There is provision for a water inlet to each tower but only one such inlet is used. The towers are operated from the 30 lbs./sq.in. compressed air line. The recovery of weak nitric acid is about 300 lbs. of weak (~~70%~~ 60%) nitric per nitration.

(b) Filters.

The filters have a separate 4 inch air main which connects with a set of five absorption towers 2 ft. 3 in. in diameter and 11 ft. 6 in. in height overall. The towers are packed with earthenware rings. The fume is drawn through the main and towers by a stainless steel Aerex fan of capacity 400 cu.ft./min. driven by a 2 H.P. electric motor. At the moment no recovery of weak nitric acid is obtained from these towers since the fume passing through is extremely small. When stainless steel filter washers are installed it is hoped that a considerable recovery will be possible.

One man is sufficient to operate both sets of towers and attend to the waste acid settling tanks. The Chargehand of one of the Nitrating Houses attends to the strong acid recovered by the fume cooler.

DISPOSAL OF WASTE ACID.

The waste acid is blown from the nitrating houses into one of a series of settling tanks. Eight such tanks are available, four of which hold the waste acid from six nitrations each and the other four have capacity for five nitrations each. The pipes from the five houses join together and the common pipe delivers into a lead anti-splash vessel. The outlet pipe from this vessel divides into eight branches, one of which goes to each of the eight tanks. Each branch has a cock. An overflow pipe from the anti-splash vessel delivers into one of the tanks if all the cocks are shut. The tanks are arranged into two rows of four and the outlet pipes, each carrying a 2 inch earthenware cock, join one of the two common mains running one down each row. The two mains join together before reaching the waste acid pump.

After filling a tank is allowed to settle for at least sixteen hours. At the end of, or during that time, the crude tetryl which floats to the surface is dug off into stainless steel buckets by stainless steel scuppets. It is then taken to the washing house and treated in exactly the same way as the tetryl from the Nitrating House filters. After the crude tetryl has been skimmed off the acid is pumped away by a Douglas pump to tanks in the Acid Factory. Further small amounts of crude tetryl may be skimmed from these tanks from time to time.

The waste acid is of the following average composition:-

H_2SO_4	70%
HNO_3	7%
HNO_2	5%

An average of 1600 lbs. of waste acid per nitration is passed to the Acid Factory. 65 to 70 lbs. of crude tetryl have to be skimmed from the tanks per charge of waste acid sent to them.

Labour required:- The Chargehand who attends to the absorption towers also attends to the movement of waste acid. The skimming of the tetryl from the waste acid requires about 1 man shift for every 9 charges of waste acid sent to the tanks.

WASHING PROCESS FOR CRUDE TETRYL.

The crude acid tetryl is dug from the filters or from the waste acid settling pots into stainless steel buckets, using stainless steel scuppets. The buckets are taken on small trolleys which hold six buckets, to the vats in which the washing is done. These are five hundred gallon vats fitted with stainless steel stirrers, directly driven by electric motors. The vats have two lead outlet faucets, one at the top and the other at the bottom. The lead faucets are connected by rubber tubing to a stainless steel gutter which conducts the wash waters from the vat into a large wooden labyrinth, which removes most of the suspended tetryl. The faucets are closed by clipping a mild steel clip over the rubber tube. When running off wash waters from the bottom faucet the rate of run off cannot exceed a certain amount otherwise large amounts of tetryl are carried into the labyrinths. The top faucet takes away the excess of inlet water over the permissible outlet at the bottom faucet. There are two cold water inlets, one of which delivers a jet of water towards the bottom faucet and is used for clearing the tetryl from the faucet. Hot water and steam inlets to the vats are also available. The vats are filled to the top faucet with water and cold water is run in at the inlet remote from the bottom faucet and out through both the top and bottom faucets. The buckets of acid tetryl are emptied into the vat. After 50 buckets have been placed in the vat the water inlet is shut off, both faucets are closed and the stirrer switched on for three minutes. The tetryl is then allowed to settle for two minutes, the bottom faucet cleared by a jet of water, the other water inlet turned on and both faucets opened. This process is repeated after 100 buckets and 150 buckets have been added. When 150 buckets have been added the vat is considered to be full. The displacement of the washings by running water in and running off at both faucets is then continued for 30 minutes. After this the water inlet is closed, both faucets are closed and the stirrer turned on for three minutes. Two further 30 minute displacement washings are given, each followed by 3 minutes stirring. The bottom faucet of the vat is then cleared by a jet of cold water and all the wash waters are run off through this faucet. The vat is then refilled with water. During refilling water is sprayed on to the surface from a hose to break down the scum which accumulates on stirring. When the vat has been refilled three further displacement washings of 30 minutes are given, each followed by 3 minutes of stirring. A sample of the wash water is then taken by the Chargehand and 100 ccs. of it are added to 10 ccs. of N.NaOH solution. The wash water should turn bright red. If this is not the case further washings are given until the test is positive. The final wash waters are then drained off and the crude tetryl is ready for wringing. A certain amount of tetryl finds its way into the labyrinth. This is removed at frequent intervals and placed in a vat which is loading or in the early stages of washing.

After the wash waters have been drained off the tetryl is dug out of the vats into stainless steel buckets and carried in these to the wringers. The tetryl is washed with hot water in the wringers until the washings are neutral to litmus. It is then wrung for twenty minutes. Finally, the crude tetryl is dug out of the wringers and rubbed through a 1/4 mesh copper sieve into a rubber bag tied on a hopper. The bags are weighed to 110 lbs. which, with a 5 lbs. allowance for the bag and 5 lbs. for moisture is approximately 100 lbs. of crude wet tetryl.

A considerable amount of tetryl accumulates in sumps in the drainage system of the Nitrating Area. This is dug out into stainless steel buckets but it is not tipped immediately into the vat but on to a 1/4 inch mesh sieve suspended over the vat. The tetryl is washed through the sieve by a hose. Any quartz or flint which may be present are retained on the seive.

When the filters in the Nitrating Houses are cleaned out, i.e. after 70 nitrations or less, the quartz from them with fairly large amounts of acid tetryl adhering is dug out into stainless steel buckets and washed on a 1/4 inch mesh sieve fitted to a small wooden vat 3 ft. 7 in. in diameter. The tetryl is carried through and then transferred from the small vat into a large one for washing.

The total cycle on a vat is:-

Filling	8 hours.
Washing	7 "
Wringing	4 "
Total	19 "

One loading of 150 buckets yields about 1500 lbs. of wet crude tetryl V.M. 5%. Acidity of the order of 0.2%.

Labour required:-

Loading — 1 man shift for eight nitrations.

Loading of skimmings is included in skimming labour (given above).

Washing — 1 Chargehand attends to the washing and also does 500 lbs. wringing.

Wringing — 1000 lbs./man shift.

A filter cloth of coarse linen, held in place by stainless steel stays, is put in each wringer to prevent the tetryl going through the holes in the basket.