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EXPLOSIVES RESEARCH & DEVELOPMENT ESTABLISHMENT

TECHNICAL MEMORANDUM No. 19/M/51

RECEIVED 23 MAY 1952 ORDGU-INTEL

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Plant for the Manufacture of 50 Tons per Week of Guanidine Nitrate by the Direct Fusion Process: An Outline Design

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SECURITY INFORMATION
WDGS -- INTELLIGENCE REPORT

SID NO.
MIG NO.
REPORT NO. R-921-52
COUNTRY Gt. Brit.
I. D. NO. 884069
SUBJECT: Plant for the Manufacture of Guanidine Nitrate by the Direct Fusion Process
FROM: OARMA-London REFERENCES:
EVALUATION: DATE OF INFORMATION: March 1952 DATE OF REPORT: 8 April 1952
INCL. 1 PREPAID BY: JOHN L. ATKINS, Sci. Consultant JLA SOURCE: ERDE

SUMMARY OR SID REPORT: Explosives Research & Development Establishment Tech. Memo. No.19/M/51 is by R.P. Ayorst and S. Chard. "Guanidine nitrate may be prepared by reacting nitrolim with a concentrated aqueous solution of ammonium nitrate at temperatures between 120° and 160°C., the so-called "Direct Fusion" Process. A pilot plant has been installed and operated on this process at Waltham Abbey. It comprises a continuous reactor operating at 120°C. and ancillary plant to isolate the product and concentrate and recycle excess ammonium nitrate liquor. Preliminary plant flowsheets for producing 50 short tons/week of guanidine nitrate have been prepared and the dimensions of the principal plant items calculated and tabulated. These are based on an estimated 95% conversion of calcium cyanamide to guanidine nitrate and an ammonium nitrate consumption of 110% theory."

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EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT

TECHNICAL MEMORANDUM NO. 19/M/51.

Plant for the Manufacture of 50 Tons per Week of Guanidine Nitrate by the Direct Fusion Process: An Outline Design

by

R.P. Ayerst and S. Chard

This memorandum does not contain classified information of overseas origin

Approved: R. G. Ross.
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CONTENTS

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Reference: XR 441/114

1. SUMMARY.

Guanidine nitrate may be prepared by reacting nitrolim with a concentrated aqueous solution of ammonium nitrate at temperatures between 120° and 160°C., the so-called "Direct Fusion" Process.

A pilot plant has been installed and operated on this process at Waltham Abbey. It comprises a continuous reactor operating at 120°C. and ancillary plant to isolate the product and concentrate and recycle excess ammonium nitrate liquor.

Successful operation has been achieved in several runs of about 200 hours continuous duration and outputs up to 50 lb. per hour of guanidine nitrate have been obtained. An isolated yield of 87% calculated on the calcium cyanamide content of the nitrolim has been obtained with a reaction time of 1 hour and the yield is expected to rise to over 90% by allowing greater reaction time. The product purity has averaged 98% guanidine nitrate.

A comparison with plant which would be necessary for batch operation shows that the principal advantage of the continuous process lies in a simpler operating procedure and reduced labour requirements.

Preliminary plant flowsheets for producing 50 short tons/week of guanidine nitrate have been prepared (Figs. 1, 2, 3, 4) and the dimensions of the principal plant items calculated and tabulated. These are based on an estimated 95% conversion of calcium cyanamide to guanidine nitrate and an ammonium nitrate consumption of 110% of theory.

2. RECOMMENDATION.

That a production scale unit operating the continuous "Direct Fusion" process be built and operated to provide full scale experience for future picrite plants.

The scale of unit should conveniently be that shown in Fig. 3 which could operate alongside the 40 tons per week plant at Bishopton or Caerwent and would incidentally give a greater yield of guanidine nitrate.

3. INTRODUCTION.

A continuous 'direct fusion' pilot plant for the manufacture of guanidine nitrate has been developed and operated during the last two years. The plant is designed on the basis of laboratory work in E.R.D.E. (1), (2) on a reaction earlier studied by I.C.I. Ltd. (4). Work is still continuing on the pilot plant to improve the operation of some plant units and to establish better yield figures but the process has been proved, and experience of continuous operation for periods of over 200 hours has been obtained.

Because of the urgency of the picrite production position it was considered useful to issue this memorandum summarising the present position of pilot plant development and, based on this, outlining a design for a plant to manufacture 50 ton/week of guanidine nitrate, for consideration by the Production Departments

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in relation to their proposed Picrite 'Pilot' Plants. Designs based on both batchwise and continuous reactors have been made in order to clarify their relative advantages, since, on a laboratory scale, batchwise manufacture has given yields of 98% compared with 95% by continuous operation (3), (7).

Meanwhile reports giving design data and details of pilot plant performance are being prepared.

4. EXPERIENCE WITH THE PILOT PLANT.

4.1 Brief Description.

The pilot plant cycle is essentially that now proposed for the larger scale and shown in the flowsheets Figs. 1 and 3, and the plant items are, with some exceptions, similar to those indicated in Fig. 3.

The reactor is a steam heated stainless steel tank arranged in four stirred compartments in series, the first of which is fed continuously with nitrolim and an excess of boiling concentrated ammonium nitrate liquor. The reaction is carried out at 120°C. and atmospheric pressure. The reacted mixture overflows from the fourth compartment to a closed stirred precipitating vessel where it is diluted and its soluble calcium content precipitated by addition of ammonium carbonate solution. The mixture is held at 70°C. in the precipitating vessel to keep guanidine nitrate in solution. The ammonium carbonate solution is obtained by absorbing the ammonia from the reactor off-gas and feeding the resulting ammonia liquor, together with a stream of carbon dioxide gas obtained from Drikold liquefiers, to the closed carbonating vessel, which is fitted with a vaned disc agitator and cooling coils. These processes are carried out continuously. The slurry in the precipitation vessel is fed batchwise to a Sweetland filter where the precipitated calcium carbonate sludge containing also impurities from the original nitrolim, is filtered off at 65° - 70°C., and the hot guanidine nitrate-ammonium nitrate filtrate is collected. On the pilot plant this is crash-cooled by continuous addition to a stirred crystallising vessel which holds a large bulk of crystallising magma at 20° - 25°C. and a bleed off of guanidine nitrate slurry is run to a batch or continuous centrifuge. The guanidine nitrate product is washed on the centrifuge. The ammonium nitrate mother liquor receives an addition of make-up ammonium nitrate and is then concentrated continuously in two steam heated pans before recycling to the reactor. The weak liquor is pumped and metered into the evaporating pans and the concentrated liquor at a boiling point of 130°C. overflows directly back into the reactor.

4.2 Throughput and Yield.

In the initial stages of development the plant was intended to operate at a production rate of 112 lb/hour of guanidine nitrate using a reaction temperature of 160°C. and a nominal reaction time of 30 minutes. Experiments performed concurrently with the construction of the plant, established that the same degree of completion of reaction could be obtained at 120°C. if the reaction time were extended to 120 minutes. The lower operating temperature was adopted on the pilot plant on the grounds of increased safety and a lower reconcentrating temperature.

As a result, it was necessary either to enlarge the reactor, or, with the same reactor, to accept a reduced yield or a reduced throughput. It was decided to use the existing reactor at a throughput equivalent to half the

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designed rate with a reaction time of 60 minutes i.e. half the reaction time stipulated.

Under these conditions, an isolated yield of 87% was obtained in the most recent run, which was of 170 hours duration, and it is known that this could be improved to 80% by improved sludge washing. In addition, by doubling the nominal reaction time to the prescribed 120 minutes the yield would approach the 95% obtained on a 3 lb/hour laboratory continuous-flow stirred tank reactor (3). Work will continue to establish this point.

4.3 Ammonium Nitrate Loss.

The ammonium nitrate consumption was 120% of theory of which 13% was lost through insufficient washing of the sludge and product; the remaining 7% was not accounted for but probably included entrainment losses at the evaporation stage. The loss through insufficient washing could be reduced to about 5% in regular operation, and entrainment losses will be reduced by improved evaporator equipment. It is expected that the overall ammonium nitrate consumption will compare favourably with the laboratory consumptions of 105 - 110% reported in (2).

4.4 Product.

The purity of the product by the direct fusion process has been comparable at both the laboratory and pilot plant production rates. An average guanidine nitrate content of 98% on a dry weight basis has been obtained on the pilot plant. This is a decided improvement over the dicyandiamide process where the product contains only 90% of guanidine nitrate and 10% ammonium nitrate. This point is important since a high ammonium nitrate content necessitates a corresponding increase in the sulphuric acid consumption during the subsequent nitration stage.

4.5 Development Work.

Previous paragraphs in this section have reported the performance of the plant in the advanced stage of development now reached. Considerable design work, running trials and laboratory investigations were undertaken in reaching the present stage. These will be reported in detail in separate reports, but the scope of the work is indicated here:

(i) In addition to the reactor itself, ancillary plant for absorption of the reactor off-gas, isolation of the product, and concentration and recycling of the ammonium nitrate mother liquor have been studied.

(ii) Some 17 runs were made, all of which involved continuous operation for many hours, and some up to 10 days, during which operational difficulties have been found and remedied, and the yield brought up to the present figure.

(iii) The theory of continuous reactors was applied, and it was concluded that a yield of 95% could be expected from a reactor consisting of 4 stirred compartments or vessels in series with a total hold-up or nominal reaction time of 120 minutes. The reactor was modified, mainly by reducing the free passage-way between compartments to make them more nearly separate consecutive compartments, and four trials were made in which the degree of conversion in the reactor was measured. Conversions of 93% were obtained, which approached the theoretical figure. The yield of isolated product from operation

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of the whole plant has not yet reached this figure, which indicates that some losses, probably physical losses, are occurring in the cycle elsewhere than in the reactor.

(iv) There have been mechanical blockages at some points and means were devised of overcoming them. At the reactor, blockages were formed in the nitrolim feed and metering mechanism by wet and caked nitrolim, and in the gas off-take lines by nitrolim dust and entrained spray.

(v) Foaming of the reactor mass was a difficulty. It was controlled (given an adequate free surface for disengagement of gas) by decreasing the water content of the ammonium nitrate supply by feeding it at a higher temperature than the reactor temperature, by careful limitation of the heating steam temperature, and by fitting foam breaking paddles on the stirrer shafts.

(vi) Absorption of ammonia from the reactor off-gas was studied, and it was found necessary to minimise the calcium nitrate content of the absorbing liquor (which was previously used for washing the sludge), and to use a spray instead of a packed absorption tower to avoid blockage in the tower by calcium carbonate.

(vii) A scheme was developed in which the water added to the system for washing the product is then used successively for washing the carbonate sludge, absorbing ammonia, then carbon dioxide and finally diluting the reactor mass. This simplifies control and reduces to a minimum the amount of water which has to be evaporated from the system.

(viii) Studies were made on the absorption of carbon dioxide in ammonia liquor to aid design of the carbonator, and of the conditions for precipitation of calcium from the diluted reaction liquor. It was shown that only 0.2% excess of ammonium carbonate was required provided a closed system was employed (the precipitation taking place at 70°C.). A closed system is similarly necessary for the subsequent filtration, and a Sweetland filter was therefore incorporated.

(ix) Difficulties were experienced in the crystallisation vessel through guanidine nitrate "icing" on the cooling coils, and a laboratory study of "icing" was initiated. Improved equipment has not been installed on the pilot plant, but the trouble can be avoided by choice of a suitable crystalliser. This matter is being pursued.

(x) Reconcentration of ammonium nitrate liquor was carried out in simple jacketted pans because other equipment was not then available. A horizontal tube evaporator is now installed and design data for the evaporation stage will be obtained.

(xi) The accumulation of sulphur impurities in the system has been investigated.

(xii) Data on corrosion by plant liquors have been collected.

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5. OUTLINE DESIGNS FOR A 50 TON/WEEK GUANIDINE NITRATE PLANT.

It is now possible to make an assessment of the plant requirements for the manufacture of guanidine nitrate on a scale equivalent to 40 short tons per week of picrite. This will require 49.4 short tons of guanidine nitrate per week, which, on a six day week of 24 hours operation is 685 lb. per hour. A six day week is assumed to allow for normal maintenance. In calculating the sizes of the vessels an extra capacity of 20% has been added in the case of ancillary equipment and an extra 30% in the case of the reactor, as margins to allow for abnormal holdups. Flowsheets have been prepared based on carrying out the reaction continuously (as on the E.R.D.E. pilot plant) and, for comparison, batchwise, since batchwise reaction is expected to give a slightly higher yield and might at first sight appear simpler. On the other hand, it is found that the ancillary plant would be the same in nearly all respects for batch or continuous operation.

6. CONTINUOUS PLANT.

The simplified material flowsheet and pictorial flowsheet (Figs. 2 and 3) are based on the operating experience gained from the pilot plant at Waltham Abbey. The main sizes of the important units have been worked out approximately to enable a preliminary assessment to be made of the capital cost, floor space, etc., whilst more accurate information on scaling up is being accumulated.

6.1 Geometric Form of the Reactor.

This is dictated by the necessity of allowing the reaction mixture sufficient free surface to permit release of ammonia gas from it without undue foaming; this applies mainly in the first compartment. The capacity of the reactor first compartment recommended is about the maximum which would be suitably provided by a single vessel, i.e. a larger vessel would have a disproportionately large diameter to control foaming. Special foam breaking blades are used as a further control of foaming but even so it is likely that scaling up to plants above 50 tons per week will involve the multiplication of the first of the four reaction vessels rather than an increase in size of a single vessel. (This vessel is surrounded by a dotted line in Fig. 3).

6.2 Evaporator.

The concentration of large amounts of mother liquor before recycling to the reactor is an essential feature of the process. For steam economy multiple effect evaporation would be used and the concentration would be carried out continuously, irrespective of whether the reaction is batchwise or continuous.

6.3 Ammonia Evolution.

In the continuous process the ammonia evolution is steady and consequently, once the absorption system has been adjusted to the appropriate throughput rate, no constant readjustment is required.

6.4 Dust Scrubbing.

The gases evolved from the point where nitrolim is added entrain sufficient nitrolim dust to make a wet scrubber essential. Since this needs mounting immediately adjacent to the vessel one such unit could be made to serve two

/first

first compartment vessels in a large continuous plant.

7. PLANT FOR A BATCH PROCESS.

Even if the simplest system of batch operation were used (i.e. one vessel for reaction and precipitation, transfer through a filter to another larger vessel, where crystallisation would take place), the final mother liquor would be run to a continuous multiple effect evaporator as noted above. In this simple arrangement, the largest unit would be dictated by the maximum rate at which ammonia can be evolved from the reactor surface without the foaming becoming uncontrollable. This size, operating with nitrolim, would in fact be a batch charge of about 3000 lb. yielding 500 lb. of guanidine nitrate with an operating cycle of 3 hours giving a weekly output of 12 tons per week.

Thus it would require 4 such batch vessel to give the desired production rate. Therefore, it would be advantageous, if the batch method were contemplated, to proceed to a more practical arrangement of 5 batch reactors working in rotation. With a 2 hour reaction time this would give a semi-continuous flow of reacted liquor which could best be processed by similar equipment to that proposed for the continuous plant. Fig.4 shows such an arrangement and gives the approximate size of the reactor vessels.

8. RELATIVE ADVANTAGES OF THE CONTINUOUS AND BATCH PROCESSES.

8.1. The batch process would require constant skilled attention for the various intermittent operations in order to ensure smooth operation. A large number of actions would have to be carried out at half-hourly intervals. The continuous process calls for the maintenance of steady conditions with major operations only at starting and shutting down; less labour is required for a continuous plant and there is less chance of human error. It is estimated that on the 50 ton/week scale one man could supervise the continuous reactor and absorption system whereas five men per shift might be needed to operate the equivalent batch reactors and absorbers. A further four men per shift might be required to operate the other 'workup' plant in either case.

8.2 The ammonia absorption system would be considerably complicated by batch working. Whereas in the continuous plant an absorber would operate at constant gas throughput and liquor feed (which could be automatically controlled), the batch absorber would be required to handle a varying cyclic gas load and the liquor feed would have to be adjusted continually to suit. About 33% increased capacity for the absorption tower would also be required and more dust scrubbers would be needed.

8.3 There would have to be separate nitrolim feeding equipment for each of the five batch reactors.

8.4 In the laboratory a yield 3% better using batch compared with continuous operation is claimed (7).

8.5 It might be simpler to deal with sludge accumulation in the batch reactors.

9. CONCLUSION.

It is clear that batch operation requires much more labour. In war time, economy of man power is most important, and the 3% difference in yield could be dissipated by unskilful operation in the more complicated batch process.

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It is concluded that continuous operation is preferable.

10. NOTE ON SAFETY OF PROCESS.

Experimental work on various mixtures containing ammonium nitrate, nitrolim and guanidine nitrate with moisture contents of up to 5% has been summarised (5), (6). No data are quoted for the actual reactor liquor which normally contains 15-20% water.

The designs discussed have not taken account of restrictions on size and arrangement of plant which might be necessary for protection.

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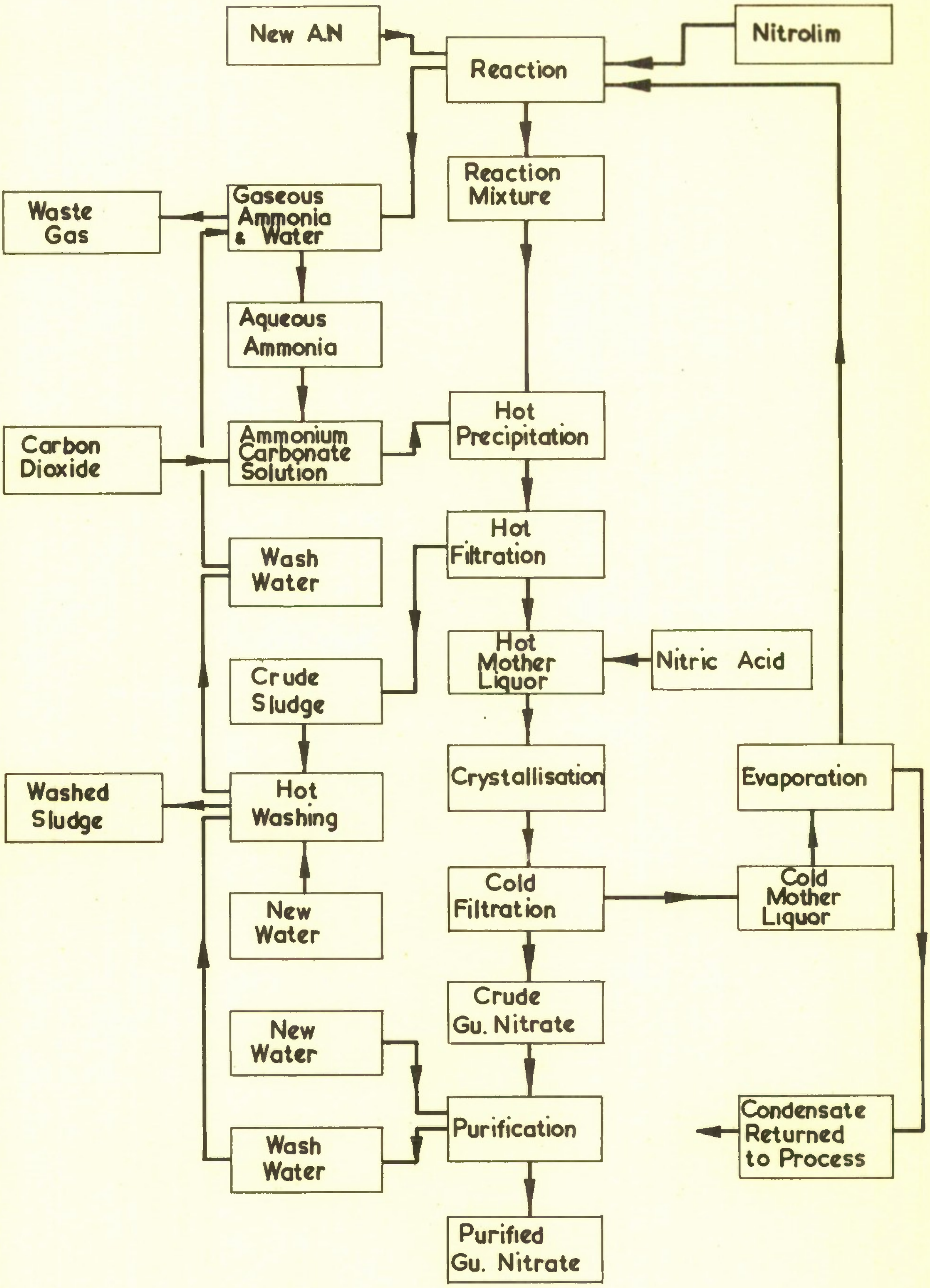

Fig. 1 PROCESS FLOWSHEET for GUANIDINE NITRATE by the DIRECT FUSION METHOD.

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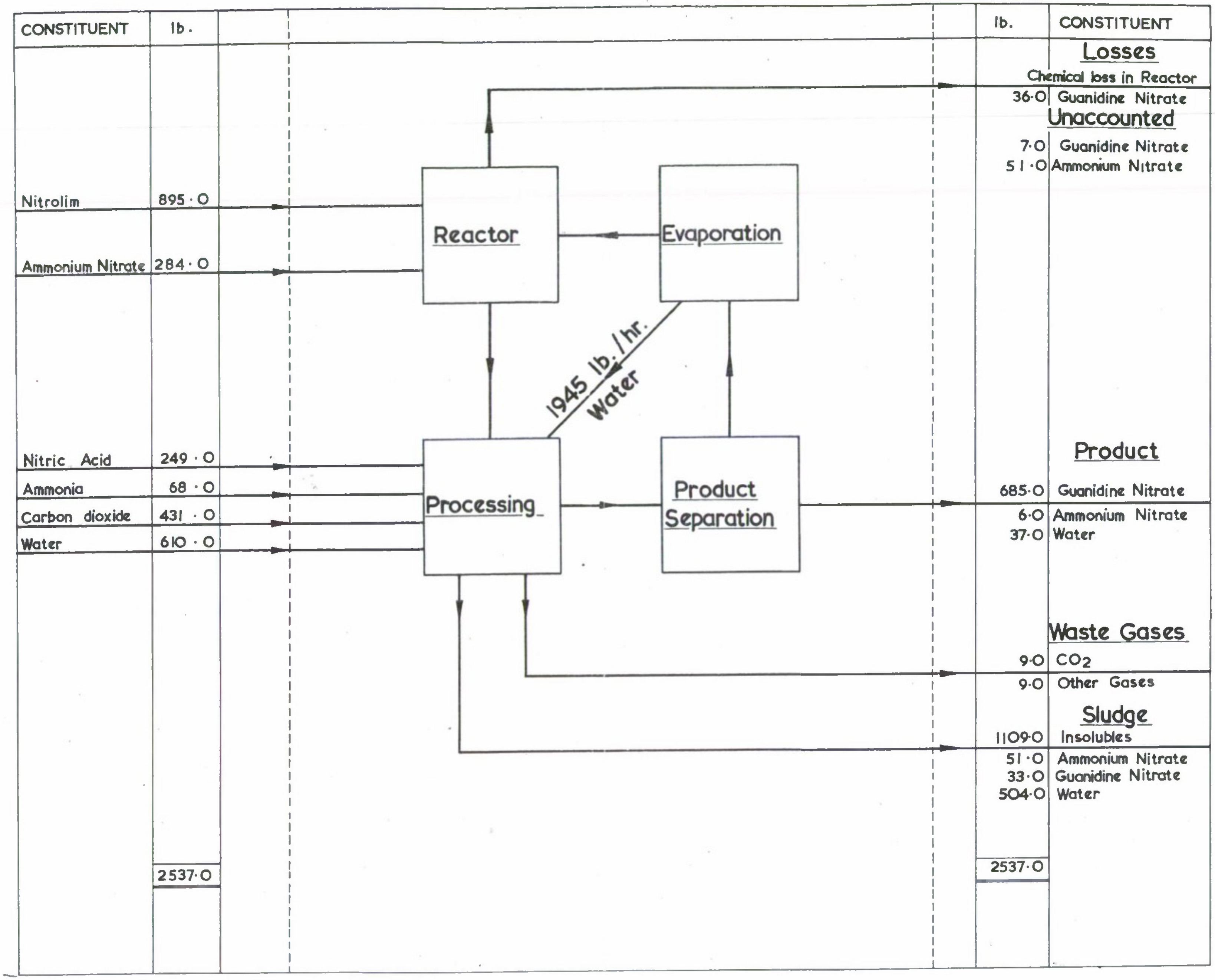

Fig. 2 SIMPLIFIED MATERIALS FLOWSHEET for GUANIDINE NITRATE PLANT

Basis 685·0 lb. Guanidine Nitrate per hr. at 95% Yield from Reactor = 49·4 short tons per week
= 40 short tons Picrite per week

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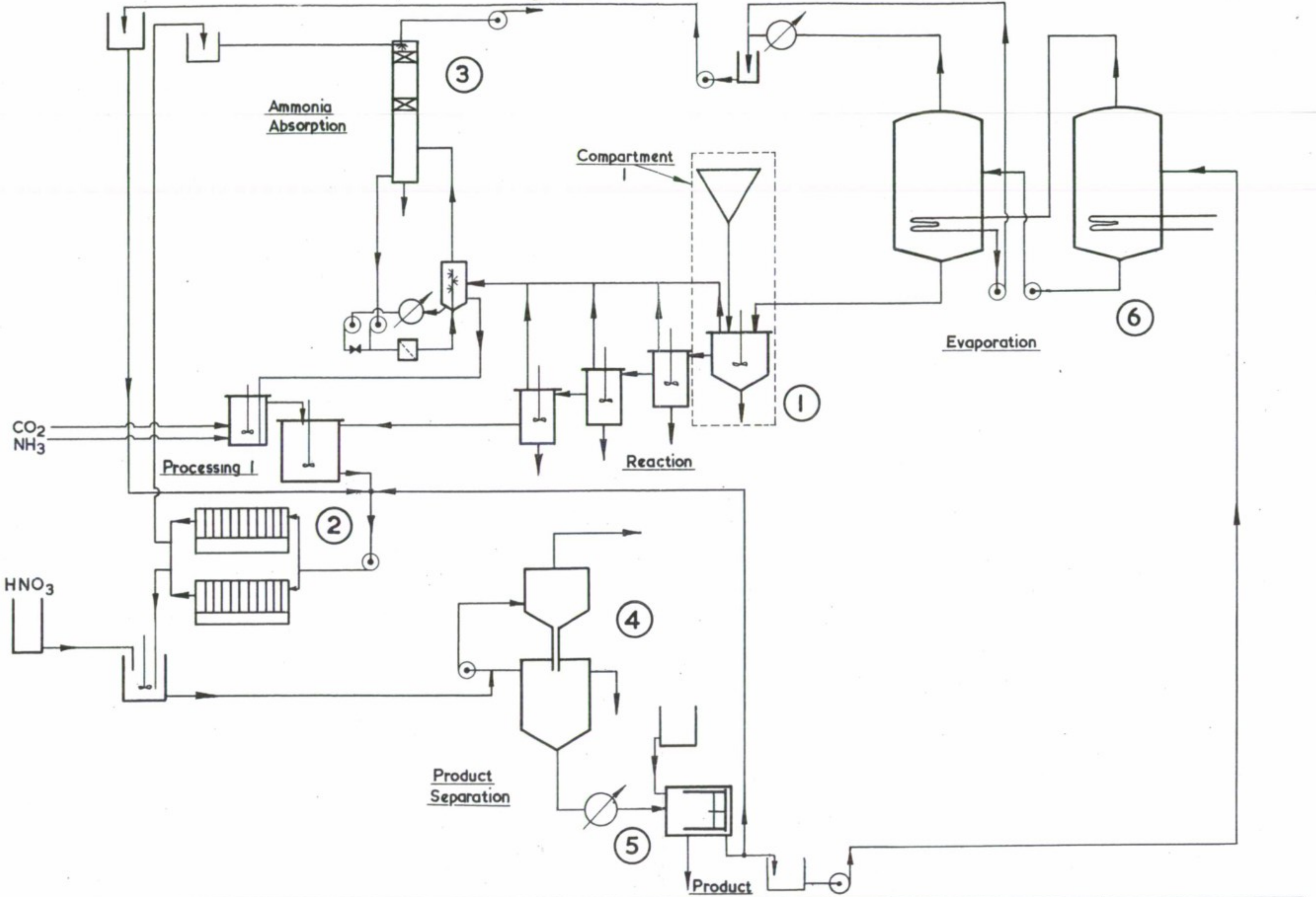

ITEM	1		2	3		4	5	6
	REACTOR		SLUDGE FILTER	AMMONIA ABSORPTN.		CRYSTALLISER	CONTINUOUS CENTRIFUGE	EVAPORATOR
	Compart. 1	Comps 2,3,4	Sweetland No 10	Dust Spray	Tower			
APPROX. CAPACITY	15,000 lb		Each 25 cu. ft. Cake			8,000 lb.		2000 lb. H_2O Evap/Hr.
APPROX. DIMENSIONS	4·5'dia x 4·5'	4'dia x 4'	3'dia x 9'	1·5'dia x 3'	3'dia x 10'	Height 20' dia 7'	13' dia Bowl	
NUMBER OFF	1	3	2	1	1	1	1	1

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Fig. 3 PICTORIAL FLOWSHEET for GUANIDINE NITRATE PLANT-(CONTINUOUS REACTOR)

Basis 685 lb. Guanidine Nitrate per hr. = 49·4 short tons per week
≡ 40 short tons Picrite per week

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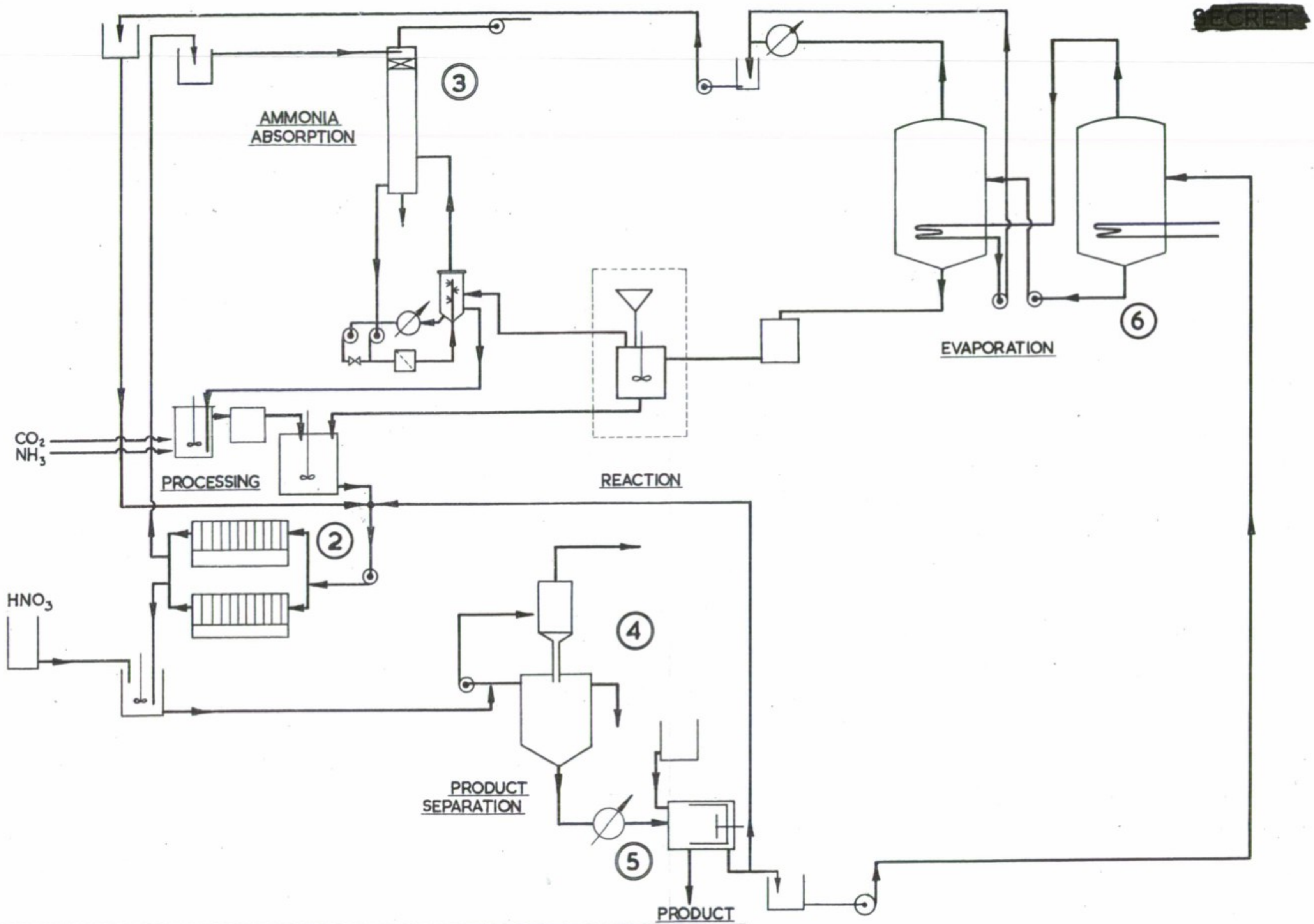

ITEM	1 REACTOR IDENTICAL UNITS	3 AMMONIA ABSORPTION	Other items as for continuous plant
APPROX. CAPACITY	EACH 3,800 lb. CHARGE	INDIVIDUAL DUST	
APPROX. DIMENSIONS	4·5' dia. x 3' (4')	SPRAY SCRUBBERS.	
NUMBER OFF	5	TOWER 10 ft. HIGH x	
		3·5' dia.	

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Fig. 4 PICTORIAL FLOWSHEET for GUANIDINE NITRATE PLANT – (BATCH REACTORS)

Basis 685 lb. Guanidine Nitrate per. hour

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