(12) UK Patent Application (19) GB (11) 2 050 335 A

(21) Application No **8014790**
(22) Date of filing **2 May 1980**
(30) Priority data
(31) **81236**
(32) **9 May 1979**
(33) **Luxembourg (LU)**
(43) Application published **7 Jan 1981**
(51) INT CL^3 **C05C 1/02 9/00 13/00**
(52) Domestic classification **C1B** 1 3C1 3C3 3C4 3CX 3F1 4
(56) Documents cited
GB 1385530
GB 1317485
GB 1218176
GB 795820
(58) Field of search **C1B**
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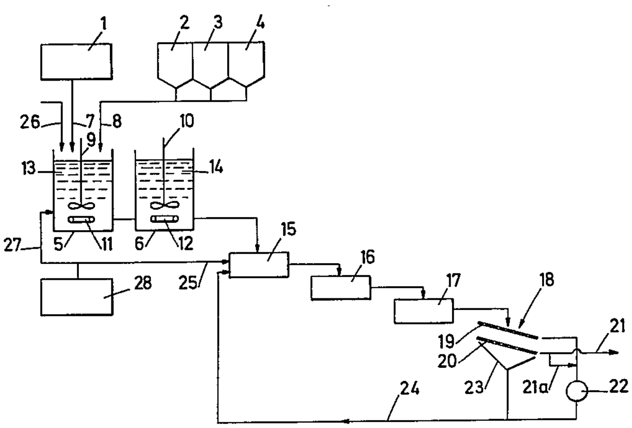
(54) Process and apparatus for the manufacture of granular fertilisers

(57) Granular fertilisers are manufactured by reacting a hot aqueous solution of ammonium nitrate and/or urea, and optionally ammonium phosphate, which solution usually contains only a little water, with at least one powdered substance containing one or more fertilising elements, such as simple or concentrated superphosphates, monoammonium and diammonium phosphates and other binary compounds containing phosphorus and nitrogen, potassium chloride or sulphate or naturally occurring phosphates, so as to form a slurry, and converting this slurry to granules by mixing it with a pulverulent fertiliser, which may comprise recycled material originating in the process, for example during grading of the product. The reaction is suitably carried out in at least one heated reactor (13, 14) fitted with a stirrer (9, 10) and provided downstream with a granulator (15), dryer (16) and grader (18).



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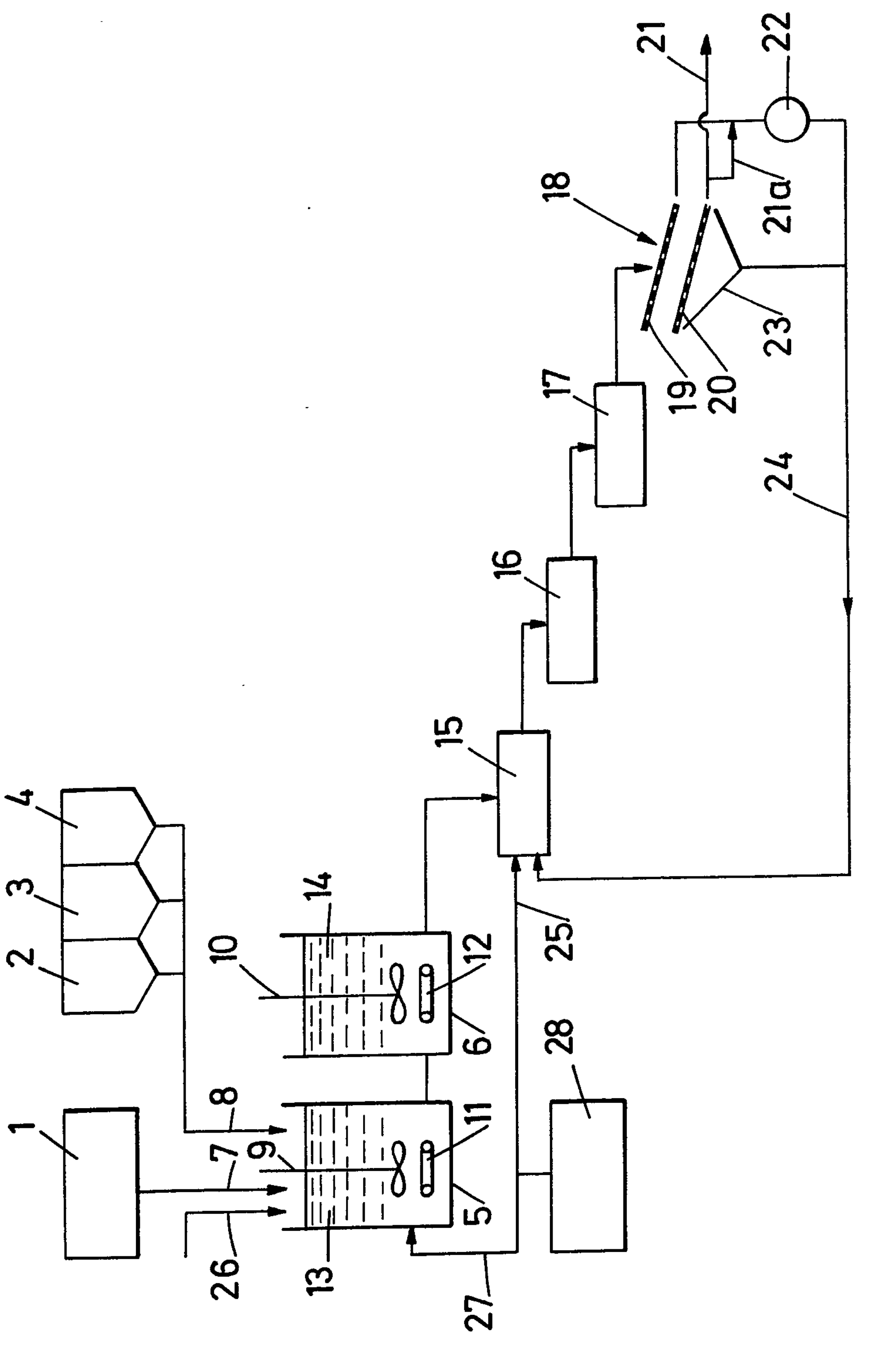
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SPECIFICATION

Process and apparatus for the manufacture of granular fertilisers

The present invention relates to a process for the manufacture of granular fertilisers, in particular ternary fertilisers.

Essentially two distinct processes exist for the manufacture and granulation of ternary fertilisers; one process starts from solid, semi-finished pulverulent raw materials and the other process uses slurries which are converted to granules on a bed of solids.

The manufacture of ternary fertilisers starting from solid semi-finished materials is carried out by mixing measured amounts of these semi-finished materials, which are in the pulverulent state, in the presence of water or of generally acid liquids, in order to permit agglomeration of this pulverulent material and thus to form granules.

Generally, a supply of steam is provided in order to heat the mass to be converted to granules, and partially to solubilise salts, so as thus to form a binder between the grains to be agglomerated into granules.

The semi-finished products used as raw materials for the manufacture of ternary fertilisers are generally as follows: ammonium nitrate and urea, simple and concentrated superphosphates, binary compounds containing phosphorus and nitrogen, such as monoammonium and diammonium phosphates, ptoassium chloride and sulphate, other compounds containing added elements (for example: Mg and S) and trace elements, compounds containing one or more of the abovementioned elements, and naturally occurring phosphates.

Depending on the fertiliser formulations envisaged, the water can be more or less completely replaced by sulphuric, phosphoric and/or nitric acid, which can react with some of the abovementioned semi-finished products. In this case, anhydrous ammonia or another base is added towards the end of the granulation process in order to neutralise the excess acidity.

This mixing and granulation operation can be carried out in known apparatuses, such as a drum, a granulating pan, a kneader or a malaxator with blades.

After granulation, the product is dried in an oven and, after cooling, it is screened in a sieving device. After grinding, the excessively fine products and the excessively coarse products are recycled to the granulation process.

The other process, which starts from a slurry, is preferably carried out in an integrated unit in which the manufacture of ternary fertilisers is directly associated with the dissolution of phosphate, for example by attack with nitric, sulphuric or phosphoric acid or a mixture thereof.

In accordance with this process, one or other of the solid semi-finished materials is incorporated into the slurry, from which part of the calcium nitrate produced has been removed, if appropriate, and this slurry is converted to granules by mixing it with recycled products originating from a sieving device, for example in one of the granulating apparatuses mentioned above in connection with the process for the granulation of solids.

Compared with the granulation of solids, as defined above, the granulation process involving slurry formation exhibits various advantages.

Thus, by virtue of the fact that the fertilisers are suspended in a liquid, the various components are brought into closer contact and can react more rapidly with one another in order to complete their reaction. This is not true of a ternary fertiliser converted to granules starting from a solid or slightly pasty mixture, in which case the reactions continue in the finished product, which can thus disintegrate or solidify.

Furthermore, since it is effected by coating several successive layers of slurry, the formation of granules from a slurry imparts a higher mechanical strength to the granules, and this therefore involves a lower risk of solidification.

Finally, since a slurry is normally more homogeneous than a mixture of solid products, the granules formed from a slurry also have a more homogeneous distribution of fertilising elements than a simple mixture of solid products.

However, a significant disadvantage of the granulation process involving slurry formation is the fact that the latter must necessarily take place in a factory in which attack on phosphate with nitric, phosphoric and/or sulphuric acid is carried out.

The present invention seeks to provide a new and advantageous process for the manufacture of granular fertiliser.

According to the invention there is provided a process for the manufacture of granular fertiliser comprising reacting a solution of ammonium nitrate and/or of urea, under the action of heat, with at least one powdered substance containing one or more fertilising elements, such as at least one pulverulent substance selected from simple and concentrated superphosphates, monoammonium and diammonium phosphates and other binary compounds containing phosphorus and nitrogen, potassium chloride and sulphate and naturally occurring phosphates, so as to form a pulp or slurry, and converting this pulp or slurry to granules by mixing it with a pulverulent fertiliser, in particular, recycled fine product originating in the process.

The invention also provides apparatus for use in carrying out the abovementioned process, comprising at least one heated reactor fitted with a stirrer, in which at least one powdered substance of the above-mentioned type is suspended in a hot solution of ammonium nitrate and/or urea, with or without ammonium phosphate, and, on the other hand, this solution is reacted with the powdered substance(s).

The process of the invention combines the advantages of the two known processes summarised in the above description.

The powdered substances are usually chosen from amongst simple and concentrated phosphates, binary compounds containing phosphorus and nitrogen, such as monoammonium and diammonium phosphates, potassium chloride and sulphate and naturally occurring phosphates, these substances being well known in the manufacture of fertilisers.

In certain cases, a solution of ammonium phosphate can be added to the solution of ammonium nitrate and/or of urea as a complement. The amount of ammonium phosphate solution is generally less than 20%, and preferably less than 10%, of the total weight of fertiliser obtained.

The solution of ammonium nitrate and/or of urea used contains a very small amount of water, this amount of water preferably being from 4 to 15% by weight of the solution. Preferably, therefore, the initial hot solution is either a solution containing 85 to 96% by weight of ammonium nitrate or a solution containing 85 to 95% by weight of urea, or a mixture thereof.

The solution is reacted with the abovementioned substances at a temperature from 70 or 75 to 100°C, and preferably from 80 to 90°C, so as to maintain good fluidity of the slurry.

Depending on the fertiliser formulations to be produced, in order to obtain the maximum granulation efficiency, the amount by weight of the abovementioned solution, relative to the amount of powdered substance used for the reaction, is suitably selected so as to obtain a pulp or slurry containing from 50 to 70%, and preferably from 55 to 60%, of solids, relative to the total amount of slurry or pulp.

Furthermore, the proportion of water in the final slurry is suitably adjusted so that it is from 4 to 10%, and preferably from 4 to 7%, by weight, relative to the total amount of slurry or pulp. Preferably part of the water is evaporated off prior to granulation. The amount of water to be evaporated after the granulation process can thus be reduced strictly to a minimum.

In addition to determining the amount and the strength of the urea solution or nitrate solution, the simultaneous adjustment of the proportion of solids or liquids and of the proportion of water is advantageously ensured by adding liquid phosphoric acid, which can be commercial grade material containing 50 - 52% of P_2O_5, and anhydrous gaseous or liquid ammonia to the slurry. The amount of ammonia is suitably such that the molar ratio NH_3/H_3PO_4 is from 1.35:1 to 1.40:1, so as to retain the maximum solubility in this ammonium phosphate solution formed in this way.

To reduce the acidity of the slurry before the granulation process, for example following the introduction of phosphoric acid, ammonia may be introduced into the slurry, before the granulation process, as a neutralising agent.

The amount of fine fertiliser, mixed with the pulp or slurry during the granulation process is generally from 1 to 3 times the amount of slurry, preferably from 1 to 2 times this amount.

After the granulation process, the resulting product is dried, this being followed by cooling and/or grinding, if appropriate, and may then be graded, for example by sieving. The fine fertiliser mentioned above, used in the granulation process, is preferably recycled material originating as part of the material removed during grading.

The accompanying drawing schematically shows an installation for the manufacture of fertiliser in accordance with the process described above.

This installation essentially comprises a tank 1 for storing a hot solution of ammonium nitrate and/or of urea, and a set of hoppers 2, 3 and 4, containing solid raw materials in powder form, which comprise, for example, superphosphates, binary compounds containing phosphorus and nitrogen, potassium chloride or sulphate, and the like.

The main part of the installation comprises at least one pulp or slurry reactor in which the solid raw materials originating from the hoppers 2, 3 and 4 are suspended in a hot solution of ammonium nitrate and/or of urea, originating from the tank 1.

In the embodiment shown in the Figure, two pulp reactors 5 and 6, arranged in series, have been provided. The arrow 7 shows the introduction of the solution from the tank 1 into the first reactor 5 and the arrow 8 shows the introduction of the solid raw materials into this same reactor 5.

The introduction of the solution can be carried out, for example, by means of an insulated pipe with a jacket in which saturated steam circulates, it being possible for the introduction of the pulverulent materials to be carried out by means of a simple conveyor belt moving underneath the hoppers, and it being possible for the metering of these materials to be carried out by any means which is in itself known, such as metering by volume or metering by weight using an automatic balance which is not shown in the figure. If appropriate, depending on their particle size, these materials are sieved and ground before being introduced into the reactors.

These reactors, which have a fixed volume, are fitted with stirrers 9 and 10 and with heating coils 11 and 12. Thus, the suspensions 13 and 14, forming a slurry or pulp, which are obtained by bringing the solution originating from the tank 1 into contact with the pulverulent materials originating from the hoppers 2, 3 and 4, are kept at the temperature required to create a good fluidity and as homogeneous a mixture as possible, and this enables the reactions to be completed under ideal conditions.

In these heated reactors 5 and 6, in addition to homogeneous mixing of the solution originating from the tank 1 with the solid originating from the hoppers 2 to 4, a certain evaporation of water takes place, and this makes it possible, for example in the case of starting from a relatively dilute solution, to bring the strength of the resulting slurry to the value required for the following operations.

If necessary, a certain amount of phosphoric acid can be added to one of the reactors, for example to the reactor 5, as indicated by the arrow 26.

The reactor 6 is followed successively by a granulator 15, such as a drum granulator, a dryer 16 and a cooler 17.

The granular products leaving the cooler fall into a sieving device 18, in which the granules are graded, for example by means of two superposed sieves 19 and 20. The particles retained between the sieves 19 and 20 then possess the predetermined particle size and consequently constitute the granular fertiliser of production, and can be sent to a packaging device, as indicated by the arrow 21.

The coarse particles which do not pass through the sieve 19 are sent to a grinder 22 and the fine particles which pass through the sieve 20 are collected in a hopper 23. These fine particles and the ground particles are then directed to the inlet of the granulator 15, as indicated by the arrow 24, so as to form the recycled fertiliser which is to be mixed with the slurry as a support for the granulation process. In the event of there being a lack of product to be recycled, for example in the case of a good granulation efficiency, part of the granular fertiliser forming the finished product may have to be recycled, as indicated by the arrow 21a.

The arrow 25 indicates the possible introduction of ammonia, as a neutralising agent, into the granulator 15 from a storage tank 28.

Moreover, an analogous introduction into the reactors 5 and 6 can be carried out, as indicated by the arrow 27.

In the granulator 15, the slurry thus coats the fine particles recycled in the direction of the arrow 24, until relatively hard granules of a certain size are built up by forming several successive layers of slurry. The dimensions of the granules depend, inter alia, on the residence time in the granulator, the composition and the viscosity of the slurry, and the like. These working conditions are generally determined experimentally so that the amount of powdered product to be recycled in the direction of the arrow 24 to the granulator 15 can thus be adjusted in an approximate manner.

The invention is further illustrated by means of practical examples of the manufacture of a ternary fertiliser, in accordance with the process of the invention, using the abovementioned installation.

Example 1: Manufacture of a 17-17-17 ternary fertiliser based on ammonium nitrate solution.

415 kg of a hot 90% strength ammonium nitrate solution at a temperature of 85°C were fed into the two reactors in series, 5 and 6, in which the solution was mixed with the following pulverulent raw materials originating from the hoppers 2, 3 and 4: 307 kg of binary compounds containing phosphorus and nitrogen in the form of 11% of nitrogen, as ammonia, and 49% of P_2O_5, 15 kg of dicalcium phosphate containing 43% of P_2O_5, and 282 kg of potassium chloride containing 60.5% of K_2O.

As indicated by the arrow 26, 15 kg of liquid phosphoric acid containing 54% of P_2O_5 were also added to the reactor 5.

Thus, a pulp containing 56% of solids but containing only 4.8% of water was obtained.

The temperature in the reactors 5 and 6 was kept at about 80°C.

In these reactors, the following reactions, in particular, take place between the suspended products:

$KCl + NH_4NO_3 \rightarrow KNO_3 + NH_4Cl$

KCl + ammonium phosphate → potassium phosphate + NH_4Cl.

By virtue of these reactions, the resulting constitution of the products, namely potassium nitrate and phosphate and ammonium chloride, is more advantageous in terms of the behaviour and the mechnaical strength of the granules obtained and in terms of the fertilising properties of the final ternary fertiliser.

The residence time of the products in the reactors was of the order of 40 minutes.

The pulp thus produced was then fed into the granulating apparatus 15, the latter also being fed with recycled products in the essentially pulverulent state. This recycled amount was between 1.7 and 2.1 times the total amount of slurry originating from the reactor 6.

3 kg of ammonia were introduced into the granulator 15 as a neutralising agent. This has been indicated by the arrow 25.

1,037 kg of granular fertiliser, corresponding to the amount of products used in the reaction, were thus obtained.

By drying and evaporating off 37 kg of water, 1,000 kg of product were obtained, this product having a moisture content of 1.3% and containing, relative to the product in this form, a total of 17% of nitrogen, comprising 6.5% of nitrogen as nitric acid and 10.5% of nitrogen as ammonia, 17% of phosphorus pentoxide and 17% of K_2O.

Example 2: Manufacture of 15-22-12 ternary fertiliser based on a urea solution.

258 kg of a hot 90% strength urea solution at a temperature of 110°C were fed into the two reactors in series, 5 and 6, in which the solution was mixed with the following pulverulent raw materials originating from the hoppers 2, 3 and 4: 263 kg of binary compounds containing phosphorus and nitrogen in the form of 11% of nitrogen, as ammonia, and 49% of P_2O_5, 224 kg of simple superphosphate containing 19% of P_2O_5, and 198 kg of potassium chloride containing 60.5% of K_2O.

90 kg of phosphoric acid containing 54% of P_2O_5 were added to the reactor 5, as indicated by the arrow 26, and 15 kg of ammonia were also added, as indicated by the arrow 27.

Thus, a pulp containing 65 % of solids but containing only 5.8% of water was obtained. The temperature in the reactors 5 and 6 was kept at 90°C.

In addition to partial ammoniation of the phosphoric acid, the following reaction takes place between the suspended products:

KCl + ammonium phosphates → potassium phosphate + NH_4Cl.

This reaction makes it possible to obtain products which are more advantageous as regards the behaviour and the mechanical strength of the resulting granules.

The residence time of the products in the reactors was of the order of 55 minutes.

The pulp thus produced was then fed into the granulating apparatus 15, the latter also being fed with recycled products.

This recycled amount was between 2.4 and 2.9 times the amount of slurry originating from the reactor 6.

4 kg of ammonia were introduced into the granulator 15 as a neutralising agent. This has been indicated by the arrow 25.

1,052 kg of granular fertiliser, corresponding to the amount of products used in the reaction, were thus obtained.

By drying and evaporating off 52 kg of water, 1,000 kg of product were obtained, this product having a moisture content of 0.8% and containing, relative to the product in this form, a total of 15% of nitrogen, 22% of phosphorus pentoxide and 17% of K_2O.

From the description given above, it is thus apparent that the process according to the invention can be applied in a conventional mixing works for powdered solid raw materials, provided that pulp reactors and a tank for storing a hot solution of ammonium nitrate or of urea are made available, and that it is thus possible to take advantage of the flexibility of exploitation and the wide variety of possible ternary fertiliser formulations appropriate to a mixing works of this kind.

Furthermore, the resulting fertiliser has the same quality as fertiliser which is converted to granules starting from a slurry.

Furthermore, a consequence of starting from hot liquids containing a small amount of water is that direct solidification of the successive coating layers is achieved by simple cooling. This has the significant effect of very greatly increasing the granulation efficiency and the capacity of a given specific granulating apparatus and of a dryer of given size and given heat capacity, compared with the known processes.

Of course, the invention is not limited to the embodiments which have been described, and many variants can be envisaged without going outside the scope of the invention.

Thus, the process can advantageously be applied to the manufacture of fertilisers of all compositions, in particular binary fertilisers.

CLAIMS

1. A process for the manufacture of granular fertiliser, comprising reacting a solution of ammonium nitrate and/or of urea, under the action of heat, with at least one powdered substance containing one or more fertilising elements, so as to form a pulp or slurry, and converting this pulp or slurry to granules by mixing it with a pulverulent fertiliser.

2. A process according to claim 1, wherein the pulverulent fertiliser comprises recycled fine product originating in the process.

3. A process according to claim 1 or 2, wherein the solution is reacted with at least one powdered substance selected from simple and concentrated superphosphates, monoammonium and diammonium phosphates and other binary compounds containing phosphorus and nitrogen, potassium chloride and sulphate and naturally occurring phosphates so as to form the pulp or slurry.

4. A process according to any of claims 1 to 3, wherein a hot solution containing 85 to 96% by weight of ammonium nitrate and/or a hot solution containing 85 to 95% by weight of urea is used to form the pulp or slurry.

5. A process according to any of claims 1 to 4, wherein the solution with which the powdered substance(s) are reacted also contains dissolved ammonium phosphate.

6. A process according to any of claims 1 to 5, wherein the solution and the powdered substance(s) are reacted at a temperature from 70°C to 100°C.

7. A process according to any of claims 1 to 6, wherein the amount of the solution, relative to the amount of powdered substance(s), is such as to obtain a pulp or slurry containing from 50 to 70% by weight of solids, relative to the total amount of pulp or slurry.

8. A process according to any of claims 1 to 7, wherein the amount of the solution, relative to the amount of powdered substance(s), is such as to obtain a pulp or slurry containing from 4 to 10% by weight of water, relative to the total amount of pulp or slurry.

9. A process according to any of claims 1 to 8, wherein liquid phosphoric acid is added to the pulp or slurry in order to adjust the proportion of P_2O_5 in the resulting fertiliser.

10. A process according to any of claims 1 to 9, wherein liquid or gaseous ammonia is introduced into the pulp or slurry as a neutralising agent.

11. A process according to any of claims 1 to 10, wherein part of the water present in the pulp or slurry is evaporated off before granulation.

12. A process according to any of claims 1 to 11, wherein the pulp or slurry is converted to granules with an amount of pulverulent fertiliser of from one to three times the amount of slurry.

13. A process according to any of claims 2 to 12, wherein the product obtained after granulation is dried and graded, and at least part of the material removed during grading is recylced, if appropriate after grinding, to the granulation process, so as to form at least part of the recycled fine fertiliser which is to be mixed with the pulp or slurry, the graded material forming the granular fertiliser product.

14. A process for the manufacture of granular fertiliser carried out substantially as described in any of the foregoing Examples.

15. Apparatus for use in the manufacture of granular fertiliser by a process according to any of the preceding claims, comprising at least one heated reactor fitted with a stirrer, in which at least one powdered substance containing one or more fertilising elements is suspended in a hot solution of ammonium nitrate and/or of urea, with or without ammonium phosphate, and is reacted therewith.

16. Apparatus according to claim 15, comprising at least two reactors in series.

17. Apparatus according to claim 15 or 16, wherein the reactor(s) are fitted with heating coils.

18. Apparatus according to any of claims 15 to 17, also including downstream of the reactor, a granulator, a dryer and a sieving device in succession.

19. Apparatus for use in the manufacture of granular fertiliser, substantially as hereinbefore described and shown in the accompanying drawing.

20. Granular fertiliser obtained by a process according to any of claims 1 to 14 or by means of apparatus according to any of claims 15 to 19.

Printed for Her Majesty's Stationery Office by Croydon Printing Company Limited, Croydon Surrey, 1980.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.