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PATENT SPECIFICATION

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NO DRAWINGS.

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COMPLETE SPECIFICATION.

Improvements in and relating to Fertilizers.

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company, of Imperial Chemical House, Millbank, London, S.W.1, do hereby declare this invention to be described in the following statement :—

5 This invention relates to mixed fertilizers and to processes for their manufacture.

According to the present invention there are provided granular mixed fertilizers comprising ammonium nitrate, finely divided
10 rock phosphate, preferably Christmas Island rock phosphate, a potassium salt, preferably the chloride, and desirably some magnesia, preferably at least 2% by weight. Hereinafter,
15 Christmas Island rock phosphate is designated "C.I. rock phosphate."

The main nutrient elements may vary within the following ranges by weight.

20	N	20 to 5%
	P ₂ O ₅	20 to 5%
	K ₂ O	4 to 21%
	MgO	At least 2%

25 Provided the analysis is within the range given above, other ingredients may be present if desired.

30 Preferably the ratio of potassium to ammonium ion is at least 0.22:1 as thereby the granules have much decreased tendency to disintegrate when exposed to cycles of alternating high and low temperatures, as they may often be in storage.

These fertilizers are adapted for use in Malayan soils, or for tropical or sub-tropical situations.

35 One preferred form of the fertilizer which is adapted for use on young rubber trees has

[Price 4s. 6d.]

the following approximate plant food content by weight :—

N	P ₂ O ₅	K ₂ O	MgO	
12.7%	18%	4.6%	2.2%	40

A suitable formulation is (percentage by weight) :—

Ammonium nitrate (35% N)	36.3%	
C.I. rock phosphate (38% P ₂ O ₅)	47.4%	
Muriate of potash (60% K ₂ O)	7.7%	45
Kieserite (26% MgO)	8.6%	

In the above composition the magnesia content may be varied in the range 1—4%.

A second preferred form which is adapted for use on mature rubber trees has the following approximate plant food content by weight :—

N	P ₂ O ₅	K ₂ O	MgO
17.0%	11.5%	6.0%	2.9%

A suitable formulation is (percentage by weight) :—

Ammonium nitrate (35% N)	48.6%	
C.I. rock phosphate (38% P ₂ O ₅)	30.3%	
Muriate of potash (60% K ₂ O)	10.0%	
Kieserite (26% MgO)	11.1%	60

In the above composition the content of magnesia may be varied in the range 1—5%.

65 Granules of this composition have much decreased tendency to disintegrate (as compared with other fertilizers containing ammonium nitrate) when subjected to cycles of alternating high and low temperature, as shown by the following experimental results.

Thus on being subjected 16 times to alternations between 20° C. and 40° C. the granules showed no volume change, due to phase change of ammonium nitrate, whereas granules of a fertilizer containing 55% of chalk and 45% of ammonium nitrate under the same conditions showed 5% change in volume. This improvement results from the ratio of potassium to ammonium ion being at least 0.22 : 1.

A third preferred form which is adapted for use on oil palms has the following approximate plant food content by weight :—

	N	P ₂ O ₅	K ₂ O	MgO
15	7.0%	7.0%	20.8%	7.0%

A suitable formulation is (percentage by weight :—

	Ammonium nitrate (35% N) ..	20.0%
	C.I. rock phosphate (38% P ₂ O ₅) ..	18.4%
20	Potassium chloride (60% K ₂ O) ..	34.7%
	Kierserite (26% MgO) ..	26.9%

In the above composition the content of magnesia may be varied within the range 4—10% by weight.

Preferably the granules are in the size range, 1—4 mms., more preferably 2—4 mms., because this affords better storage and flowing properties. Preferably also, they are coated, e.g. with about 0.2—0.4% by weight of lubricating oil and with about 2—4% by weight of a dry finely divided inorganic material such as calcium carbonate, china clay, plaster of Paris, or rock phosphate.

The fertilizers containing magnesium are of especial value for soils deficient in magnesium, e.g. some Malayan soils.

Preferably, the phosphate rock grades so that at least 90% passes B.S.S. 52. Christmas Island rock phosphate is preferred and a suitable grading is, for example :—

Less than B.S.S. 10 ..	70% by weight
Less than B.S.S. 52 ..	90% by weight
Less than B.S.S. 25 ..	100% by weight

Instead of potassium chloride, potassium nitrate may be used. Alternatively the sulphate may be used.

A suitable magnesium compound is kieserite, but dolomite and dolomitic limestone may also be used.

It is desirable that the ingredients be relatively fine in order to obtain granules of substantially uniform composition. Commercially available salts designated as fine are usually satisfactory. More preferably the bulk of the material, e.g. at least 90%, should pass B.S.S. 52.

According to a further feature of the invention granular NPK fertilizers, preferably also containing magnesium, are produced by

introducing ammonium nitrate, finely ground phosphate rock, preferably Christmas Island rock phosphate, a potassium salt, which is preferably the chloride, and which is preferably finely divided, and preferably a magnesium compound, and recycled undersize product if desired, to a cylindrical rotary drum or a mixer which has a rotating mixing member or members, together with a small proportion of water, mixing for a sufficient time in the mixer to produce granules, drying the granules by passage through a rotary drier, grading the granules between e.g. 1 and 4 mms., for example by means of sieves, and recycling undersize, if desired, to the mixer. Preferably the granules are coated with, for example, 0.2 to 0.4% by weight of oil and thereafter with about 2—4% of a dry finely divided inorganic material.

The ammonium nitrate may be introduced in particulate form: it may also be introduced as a concentrated solution containing e.g. 5—15% of water, which is preferably hot, e.g. 80° C. or more. Part or all of the water needed for granulation may be introduced as ammonium nitrate solution. The amount of water required in the mixer will depend on a number of factors, among which the amount of recycled undersize material is important. A typical amount is, for example, from 3 to 6% of water by weight of the total mixture, although at higher temperatures it is often possible to use less water.

Preferably the mixing of the ingredients is conducted outside the granulating mixer. But, if desired, especially where ammonium nitrate is used in solution, mixing may be done in the granulating mixer.

Suitable mixers for granulating comprise for example paddle mixers, Kirich mixers, pan granulators, drum mixers and blungers.

A blunger is preferred because of its greater flexibility and simplicity of operation. A blunger comprises a trough-shaped vessel in which rotate two axial shafts provided with a number of blades arranged so that the blades on one shaft are staggered longitudinally relative to those on the other for effective mixing or "blunging."

A blunger is capable of dealing with all the mixtures mentioned above and yields clean granules of good hardness. Each of the compositions mentioned above yielded at the exit of the blunger excellent granules of good quality containing about 2% of free moisture, which could be readily dried down to 0.5% of moisture in a rotary drier.

Operating with a blunger in the manner described to produce 1.5—3.5 mm. granules it is possible to obtain granulation efficiencies of about 20 to 30%, and then the ratio of recycled dried undersize product to fresh feed to the blunger is about 4:1 to 2.3:1 by weight.



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The product from the mixer is fed to a drier, preferably a rotating cylindrical drier, which may be fed with hot air flowing co- or counter-currently to the granules. Relatively short residence times in the drier are sufficient.

The product from the drier is sieved on screens of appropriate mesh to yield, e.g. 1.5 to 3.5 mm. granules.

Where sufficient ammonium nitrate is used in the mixture and the water content is low enough, the heat of crystallisation may be used for drying the granules.

When manufacture is conducted in warm humid climates it is often beneficial to use cold air for rapidly cooling the product prior to coating and thus preventing caking when it is in storage. This has particular advantage when the heat of crystallisation of ammonium nitrate is used to dry the product.

EXAMPLE 1.

This relates to the 12.7 : 18 : 4.6 : 2.2 fertilizer referred to above.

The substantially dry ingredients comprising finely crystalline ammonium nitrate, C.I. rock phosphate (90% passing B.S.S. 52), finely crystalline muriate of potash (60% K_2O) and finely crystalline kieserite in the correct proportions were mixed and fed to a blunger to which were also fed recycled under-size product amounting to 75% to 70% of the product, and water amounting to 3 to 5% of the total mixture, depending on the amount of the recycled product.

The temperature of the solid feed to the blunger was 27° C. to 33° C. and the temperature of the solids leaving the blunger was 42—51° C. Higher temperatures can also be used.

The product from the blunger was fed to a rotary cylindrical co-current drier furnished with inlet air at about 174° C., in which the average residence time was 15 minutes. The solids and air temperatures at the exit of the drier were 96° C. and 100° C. respectively.

There was obtained about $\frac{1}{2}$ ton of 1.5—3.5 mm. granules having an average moisture content of 0.32%. The granules were of good hardness requiring on the average a weight of 6—7 pounds to crush them as compared with a weight of about 5 pounds on the average to crush granules of a commercially well established NPK fertilizer of similar grading.

EXAMPLE 2.

This relates to the 17.0 : 11.5 : 6.0 : 2.9 fertilizer referred to above.

The substantially dry ingredients comprising finely crystalline ammonium nitrate, C.I. rock phosphate (90% passing B.S.S. 52) finely crystalline muriate of potash (60% K_2O) and finely crystalline kieserite in the

correct proportions were mixed and fed to the blunger as described in Example 1.

The average temperature of the solid feed to the blunger was 29—39° C., that of the solids leaving it was 42—55° C., and the amount of water used was 3.5—4%, there being 80—85% recycle of under-size dry product. Higher temperatures can also be used.

The drier was operated co-currently under the following conditions: average inlet temperature of air 143° C.; average exit temperature of solids 78° C.; average exit temperature of air 85° C. The residence time in the drier was 15 minutes.

There was obtained about 1 ton of 1.5—3.5 mm. granules having an average moisture content of 0.65%. The granules were of good hardness and required on the average a weight of 6—7 pounds to crush them.

EXAMPLE 3.

This relates to the 7.0 : 7.0 : 20.8 : 7.0 fertilizer referred to above.

The substantially dry ingredients comprising finely crystalline ammonium nitrate, C.I. rock phosphate (90% passing B.S.S. 52), finely crystalline potash (60% K_2O) and finely crystalline kieserite in the correct proportions were mixed and fed to the blunger as described in Example 1.

The average temperature of the solid feed to the blunger was 50—65° C., that of the solids leaving it was 50—65° C., and the amount of water used was 3.0—4.5%, there being 80—75% recycle of under-size dry product. Higher temperatures can also be used.

The drier was operated co-currently under the following conditions: average inlet temperature of air 155° C., average exit temperature of solids 78° C., average exit temperature of air 89° C. The residence time in the drier was 15 minutes.

There was obtained $\frac{1}{2}$ ton of 1.5—3.5 mm. granules having an average moisture content of 0.33% and of good hardness, requiring on the average a weight of 6—7 pounds to crush them.

WHAT WE CLAIM IS:—

1. Granular mixed fertilizers comprising ammonium nitrate; finely divided rock phosphate, preferably Christmas Island rock phosphate; a potassium salt, preferably the chloride; and, desirably, a magnesium compound preferably present in an amount corresponding to at least 2% by weight of magnesia.

2. Granular fertilizers as claimed in Claim 1 in which the ratio of potassium to ammonium ion is at least 0.22 : 1.

3. Granular fertilizers as claimed in Claim 1 or 2 in which the phosphate rock, preferably



0.32%.



The product from the mixer is fed to a drier, preferably a rotating cylindrical drier, which may be fed with hot air flowing co- or counter-currently to the granules.

5 Relatively short residence times in the drier are sufficient.

The product from the drier is sieved on screens of appropriate mesh to yield, e.g. 1.5 to 3.5 mm. granules.

10 Where sufficient ammonium nitrate is used in the mixture and the water content is low enough, the heat of crystallisation may be used for drying the granules.

When manufacture is conducted in warm humid climates it is often beneficial to use cold air for rapidly cooling the product prior to coating and thus preventing caking when it is in storage. This has particular advantage when the heat of crystallisation of ammonium nitrate is used to dry the product.

EXAMPLE 1.

This relates to the 12.7:18:4.6:2.2 fertilizer referred to above.

25 The substantially dry ingredients comprising finely crystalline ammonium nitrate, C.I. rock phosphate (90% passing B.S.S. 52), finely crystalline muriate of potash (60% K_2O) and finely crystalline kieserite in the correct proportions were mixed and fed to a blunger to which were also fed recycled under-size product amounting to 75% to 70% of the product, and water amounting to 3 to 5% of the total mixture, depending on the amount of the recycled product.

35 The temperature of the solid feed to the blunger was 27° C. to 33° C. and the temperature of the solids leaving the blunger was 42–51° C. Higher temperatures can also be used.

40 The product from the blunger was fed to a rotary cylindrical co-current drier furnished with inlet air at about 174° C., in which the average residence time was 15 minutes. The solids and air temperatures at the exit of the drier were 96° C. and 100° C. respectively.

45 There was obtained about $\frac{1}{2}$ ton of 1.5–3.5 mm. granules having an average moisture content of 0.32%. The granules were of good hardness requiring on the average a weight of 6–7 pounds to crush them as compared with a weight of about 5 pounds on the average to crush granules of a commercially well established NPK fertilizer of similar grading.

EXAMPLE 2.

55 This relates to the 17.0:11.5:6.0:2.9 fertilizer referred to above.

60 The substantially dry ingredients comprising finely crystalline ammonium nitrate, C.I. rock phosphate (90% passing B.S.S. 52) finely crystalline muriate of potash (60% K_2O) and finely crystalline kieserite in the

correct proportions were mixed and fed to the blunger as described in Example 1.

65 The average temperature of the solid feed to the blunger was 29–39° C., that of the solids leaving it was 42–55° C., and the amount of water used was 3.5–4%, there being 80–85% recycle of undersize dry product. Higher temperatures can also be used.

70 The drier was operated co-currently under the following conditions: average inlet temperature of air 143° C.; average exit temperature of solids 78° C.; average exit temperature of air 85° C. The residence time in the drier was 15 minutes.

75 There was obtained about 1 ton of 1.5–3.5 mm. granules having an average moisture content of 0.65%. The granules were of good hardness and required on the average a weight of 6–7 pounds to crush them.

EXAMPLE 3.

85 This relates to the 7.0:7.0:20.8:7.0 fertilizer referred to above.

90 The substantially dry ingredients comprising finely crystalline ammonium nitrate C.I. rock phosphate (90% passing B.S.S. 52), finely crystalline potash (60% K_2O) and finely crystalline kieserite in the correct proportions were mixed and fed to the blunger as described in Example 1.

95 The average temperature of the solid feed to the blunger was 50–65° C., that of the solids leaving it was 50–65° C., and the amount of water used was 3.0–4.5%, there being 80–75% recycle of undersize dry product. Higher temperatures can also be used.

100 The drier was operated co-currently under the following conditions: average inlet temperature of air 155° C., average exit temperature of solids 78° C., average exit temperature of air 89° C. The residence time in the drier was 15 minutes.

105 There was obtained $\frac{1}{2}$ ton of 1.5–3.5 mm. granules having an average moisture content of 0.33% and of good hardness, requiring on the average a weight of 6–7 pounds to crush them.

WHAT WE CLAIM IS:—

1. Granular mixed fertilizers comprising ammonium nitrate; finely divided rock phosphate, preferably Christmas Island rock phosphate; a potassium salt, preferably the 115 chloride; and, desirably, a magnesium compound preferably present in an amount corresponding to at least 2% by weight of magnesia.

2. Granular fertilizers as claimed in Claim 1 in which the ratio of potassium to ammonium ion is at least 0.22:1.

3. Granular fertilizers as claimed in Claim 1 or 2 in which the phosphate rock, preferably



75%

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0.32%

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Christmas Island rock phosphate, is of such grading that at least 90% passes B.S.S. 52.

4. Granular fertilizers as claimed in Claim 1, 2 or 3 in which the nutrient elements are within the following ranges, expressed as weight per cent, in the following manner:—

10	N	..	..	..	20 to 5
	P ₂ O ₅	..	..	..	20 to 5
	K ₂ O	..	..	..	4 to 21
	MgO	..	..	..	At least 2

5. Granular fertilizers as claimed in Claim 4 suitable for use on young rubber trees having the following approximate content by weight of plant nutrients:—

15	N	..	..	..	12.7%
	P ₂ O ₅	..	..	..	18%
	K ₂ O	..	..	..	4.6%
	MgO	..	..	..	2.2%

6. Granular fertilizers as claimed in Claim 4 suitable for use on mature rubber trees having the following approximate content by weight of plant nutrients:—

25	N	..	..	..	17.0%
	P ₂ O ₅	..	..	..	11.5%
	K ₂ O	..	..	..	6.0%
	MgO	..	..	..	2.9%

7. Granular fertilizers as claimed in Claim 4 suitable for use on oil palm trees having the following approximate content by weight of plant nutrients:—

30	N	..	..	..	7.0%
	P ₂ O ₅	..	..	..	7.0%
	K ₂ O	..	..	..	20.8%
	MgO	..	..	..	7.0%

8. Granular fertilizers as claimed in any of Claims 1 to 7 in which the size of the granules is in the range 1 to 4 mms.

9. Granular fertilizers as claimed in any of Claims 1 to 8 which are coated with from about 0.2 to 0.4% by weight of lubricating oil and from about 2 to 4% by weight of a dry finely divided inorganic material.

10. Process for producing granular NPK fertilizers preferably also containing magnesium, which comprises introducing ammonium nitrate, finely divided rock phosphate especially Christmas Island rock phosphate, a potassium salt and desirably at least some magnesium compound, preferably kieserite, and recycled undersize product, if desired, to a cylindrical rotary drum or a mixer which has a rotating mixing member or members, together with a small proportion of water,

mixing for a sufficient time in the mixer to produce granules, drying the granules by passage through a rotary drier, grading the product granules to a desired size, e.g. by means of sieves, and recycling undersize, if desired, to the mixer.

11. Process as claimed in Claim 10 in which the granulating mixer is a blunger.

12. Process as claimed in Claim 10 in which the granulating mixer is selected from paddle mixers, Eirich mixers, pan granulators and drum mixers.

13. Process as claimed in any of Claims 10 to 12 in which ammonium nitrate is used in finely crystalline or powder form.

14. Process as claimed in any of Claims 10 to 12 in which ammonium nitrate is introduced as a hot concentrated solution.

15. Process as claimed in Claim 14 in which at least some of the water required for granulation is introduced as ammonium nitrate solution.

16. Process as claimed in any of Claims 10 to 13 in which the ingredients are premixed before introducing them to the granulating mixer.

17. Process as claimed in any of Claims 10 to 14 in which the mixing is conducted in the granulating mixer.

18. Process as claimed in any of Claims 10 to 17 in which the total amount of water is in the range of from 3 to 6% by weight of the total mixture.

19. Process as claimed in any of Claims 10 to 18 in which the size of the finished granules is from 1 to 4 mms.

20. Process as claimed in Claim 19 in which the size of the finished granules is from 1.5 to 3.5 mms.

21. Process as claimed in Claims 11, 16 and 20 taken together in which the granulation efficiencies are about 20 to 30% and the ratio of recycled dried undersize product to fresh feed to the blunger is from about 4 : 1 to 2.3 : 1 by weight.

22. Process as claimed in any of Claims 10 to 20 in which the amounts of ammonium nitrate and water are selected such that the heat of crystallisation can be used for drying the granules.

23. Process as claimed in any of Claims 10 to 22 in which the formed granules are cooled by means of cold air.

24. Process of making NPK Mg fertilizers substantially as described with reference to any of the foregoing Examples 1, 2 and 3.

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Agent for the Applicants.

