

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference P23859PC00	FOR FURTHER ACTION		See Form PCT/IPEA/416
International application No. PCT/EP2015/066040	International filing date (<i>day/month/year</i>) 14.07.2015	Priority date (<i>day/month/year</i>) 14.07.2014	
International Patent Classification (IPC) or national classification and IPC INV. C05C1/02			
Applicant YARA INTERNATIONAL ASA			
1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36. 2. This REPORT consists of a total of <u>5</u> sheets, including this cover sheet. 3. This report is also accompanied by ANNEXES, comprising: a. ☒ (<i>sent to the applicant and to the International Bureau</i>) a total of <u>6</u> sheets, as follows: ☒ sheets of the description, claims and/or drawings which have been amended and/or sheets containing rectifications authorized by this Authority, unless those sheets were superseded or cancelled, and any accompanying letters (see Rules 46.5, 66.8, 70.16, 91.2, and Section 607 of the Administrative Instructions). ☐ sheets containing rectifications, where the decision was made by this Authority not to take them into account because they were not authorized by or notified to this Authority at the time when this Authority began to draw up this report, and any accompanying letters (Rules 66.4bis, 70.2(e), 70.16 and 91.2). ☐ superseded sheets and any accompanying letters, where this Authority either considers that the superseding sheets contain an amendment that goes beyond the disclosure in the international application as filed, or the superseding sheets were not accompanied by a letter indicating the basis for the amendments in the application as filed, as indicated in item 4 of Box No. I and the Supplemental Box (see Rule 70.16(b)). b. ☐ (<i>sent to the International Bureau only</i>) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing, in the form of an Annex C/ST.25 text file, as indicated in the Supplemental Box Relating to Sequence Listing (see paragraph 3ter of Annex C of the Administrative Instructions).			
4. This report contains indications relating to the following items: ☒ Box No. I Basis of the report ☐ Box No. II Priority ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability ☐ Box No. IV Lack of unity of invention ☒ Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement ☐ Box No. VI Certain documents cited ☐ Box No. VII Certain defects in the international application ☒ Box No. VIII Certain observations on the international application			
Date of submission of the demand 06.11.2015		Date of completion of this report 26.10.2016	
Name and mailing address of the international preliminary examining authority:  European Patent Office P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Fax: +31 70 340 - 3016		Authorized officer Cardin, Aurélie Telephone No. +31 70 340-9810 	

Box No. I Basis of the report

1. With regard to the **language**, this report is based on
- ☒ the international application in the language in which it was filed
 - ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of:
 - ☐ international search (under Rules 12.3(a) and 23.1(b))
 - ☐ publication of the international application (under Rule 12.4(a))
 - ☐ international preliminary examination (under Rules 55.2(a) and/or 55.3(a) and (b))
2. With regard to the **elements*** of the international application, this report is based on (*replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report*):

Description, Pages

1-37 as originally filed

Claims, Numbers

1-18 filed on

20-07-2016

Drawings, Sheets

1/1 as originally filed

- ☐ a sequence listing - see Supplemental Box Relating to Sequence Listing.
3. ☐ The amendments have resulted in the cancellation of:
- ☐ the description, pages
 - ☐ the claims, Nos.
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since either they are considered to go beyond the disclosure as filed, or they were not accompanied by a letter indicating the basis for the amendments in the application as filed, as indicated in the Supplemental Box (Rules 70.2(c) and (c-bis)):
- ☐ the description, pages
 - ☐ the claims, Nos.
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
5. ☐ This report has been established:
- ☐ taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rules 66.1(d-bis) and 70.2(e)).
 - ☐ without taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91(Rules 66.4bis and 70.2(e)).

**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/EP2015/066040

6. ☒ With regard to top-up searches (Rules 66.1 *ter* and 70.2(f)):
- ☒ A top-up search was carried out by this Authority on 06.06.2016 (all discovered documents are listed in the Supplemental Box Relating to Top-up Search).
 - ☐ Additional relevant documents have been discovered during the top-up search.
 - ☐ No top-up search was carried out by this Authority because it would serve no useful purpose.
7. ☐ Supplementary international search report(s) from Authority(ies) has/have been received and taken into account in establishing this report (Rule 45bis.8(b) and (c)).

* If item 4 applies, some or all of those sheets may be marked "superseded".

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	<u>1-18</u>
	No: Claims	
Inventive step (IS)	Yes: Claims	<u>1-18</u>
	No: Claims	
Industrial applicability (IA)	Yes: Claims	<u>1-18</u>
	No: Claims	

2. Citations and explanations (Rule 70.7):

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Reference is made to the following documents:

- D1 RU 2 281 274 C1 (NII UDOBRENIJAM I INSEKTOFUNGII [RU]) 10 August 2006 (2006-08-10)
- D2 DE 23 60 851 A1 (CHEVRON RES) 18 July 1974 (1974-07-18)
- D3 US 2006/243010 A1 (SANDERS JOHN L [US] ET AL) 2 November 2006 (2006-11-02)
- D4 US 2008/223098 A1 (TAULBEE DARRELL N [US]) 18 September 2008 (2008-09-18)

Novelty

D1 discloses (see WPI abstract and electronic translation, paragraph [0015]) the production of granulated lime-ammonium nitrate (CAN) with liquid suspension of bentonite clay. The slurry of fertilizer comprises about 0.55 wt% bentonite and is dried to obtain granules of size 2-5 mm. D1 does not disclose a gelling agent able to form a hydrogel.

D2 discloses (examples) ammonium nitrate granules having intimately admixed therewith from about 0.01 to 5% by weight, based on weight total composition, of a hydroxy-aluminum polymer. The granules have a size between 1.397 mm and 2.362 mm. D2 does not disclose a gelling agent able to form a hydrogel.

D3 discloses (paragraph [0009]; paragraph [0012]; claims 1-5,11) ammonium nitrate particle coated with ammonium phosphate and gum or a polymer. The coated particles comprise a quantity of carbon of about 0.2% by weight or less of the total weight of the coated particle. The ammonium nitrate particles are coated with a solution comprising the polymer. D3 does not disclose a gelling agent able to form a hydrogel.

D4 discloses (paragraph [0004] - paragraph [0006]; paragraph [0012]; examples; claims) a method for reducing the explosive potential of ammonium nitrate fertilizer granules, where the granules are coated with coal combustion by-product, water and guar gum or bentonite. The amount of guar gum is 0.25 wt% of the coating weight. The coating weight represent 20% by weight of the granules, therefore, guar gum is present in an amount of 0.05 wt%. The granules obtained have a diameter between

0.841 mm to 3.36 mm (6 to 20 mesh). Although the composition of D4 comprises guar gum, i.e. a gelling agent able to form a hydrogel when contacted with water, the amount of guar gum seems too low to enable the formation of a hydrogel.

D4, which is considered to represent the most relevant state of the art, discloses granules comprising ammonium-nitrate based particles, from which the subject-matter of claim 1 differs in that it comprises a lower amount of guar gum.

The technical effect linked to this difference is the production of ammonium nitrate which cannot be used for the recovery of ammonium nitrate alone since when the granules will be dissolved in water, a hydrogel will be formed which will be difficult to filter and recover the ammonium nitrate, thus will difficult the production of home made explosive.

The problem to be solved by the present invention may therefore be regarded as providing an alternative composition comprising ammonium nitrate.

The aim of the guar gum in D4 is to be used as binding agent. The man skilled in the art would not consider to use a higher amount of guar gum since it comprises carbon and the use of carbon is limited with ammonium nitrate based fertilizer for use as fertilizer. The subject-matter of claim 1 therefore involves an inventive step within the meaning of Art. 33(3) PCT.

The same reasoning applies to the subject-matter of corresponding independent claims 10, 13, 14, 15 and 16, which are also considered to involve an inventive step within the meaning of Art. 33(3) PCT.

Claims 2-9, 11, 12, 17 and 18 are dependent on claims 1, 10 and 16 and as such also fulfill the requirements of novelty and inventive step.

Item VIII

Certain observations on the international application

Claim 1 does not meet the requirements of Article 6 PCT because the matter for which protection is sought is not clearly defined. The claim attempts to define the subject-matter in terms of the result to be achieved (an amount of gelling agent which is able to form a substantially water-insoluble and non filterable hydrogel), which merely amounts to a statement of the underlying problem, without providing the technical features necessary for achieving this result. In said claim the gelling agent is not defined. In order to overcome this objection, the amount of gelling agent and its nature (gum gelling agent, only gelling agent supported by the examples in the description) should be precised.

The same remark applies to independent claims 10, 13, 14, 15.

European Patent Office
Bob-van-Bentham-Platz 1
80469 Munich

GERMANY

Oslo, 20 July 2016

Our ref.: P23859PC00/ANH
Anette Hauger

Your ref.:

International Patent Application No. PCT/EP2015/066040
Yara International ASA

Dear Madam or Sir:

We refer to the Written Opinion of 10th June 2016.

1. Amended claim

Enclosed is an amended set of claims. In the amended set of claims, claim 1, 10, 13, 14 and 15 have been amended by emphasizing that a substantially water-insoluble and non-filterable hydrogel is formed by the composition after contacted with water, thus the amended claims fulfil the requirements of Article 6 PCT.

Basis for the amendments can be found in the description inter alia on page 6, lines 25-30 and page 33, lines 1-3.

2. Novelty

In said written opinion the Examiner objects that claims 1-5, 8-14 and 16-18 lack novelty in view of D4.

We respectfully disagree with the Examiner for the following reasons:

D4 discloses a method for reducing explosive potential of ammonium nitrate fertilizer granules wherein the fertilizer granules are coated with one or more by-products (CCB) from a coal-combustion process. Water and an organic composition such as guar gum can be used together as binders. The aim of the invention is to provide ammonium nitrate fertilizers of reduced explosive potential, but which retains its primary function as agricultural fertilizer. The coated ammonium nitrate end product is resistant to separation of uncoated ammonium nitrate by mechanical disruption (paragraph [0072]).

The main difference between the invention of the present application and the invention disclosed in D4 is that the invention of the present application discloses a composition comprising ammonium nitrate-based particles and such an amount of gelling agent (such as guar gum) that a substantially water-insoluble and non-filterable hydrogel is formed after contacting composition with water. When adding water to the composition the viscosity of the



obtained solution is increased as the water with the dissolved ammonium nitrate is gelled, and the recovery of ammonium nitrate is hampered. Further, the highly viscous gelled solution effectively prevents filtration of solution to remove solids from the ammonium nitrate composition. See page 5, lines 16 to 24 of the present application. The formation of such gelled composition is not disclosed in D4.

As also stated by the Examiner, no hydrogel will be formed by the method disclosed in D4, but we disagree with the Examiner in that the guar gum will be able to form a gelling agent. The guar gum used in the method in D4 is acting as a binder, thus reacting with water which is used as a primary binder, CCB and the ammonium nitrate particles. Further, as stated by the Examiner, an amount of 0.05 wt% guar gum is present in the particle disclosed in D4, which is not sufficient to form a substantially water-insoluble and non-filterable hydrogel after the composition is contacted with water. As disclosed in figure 2 of the present invention, the recovery of ammonium nitrate from the composition is completely hampered when 2 wt% of gelling agent is present in the particles.

3. Inventive step

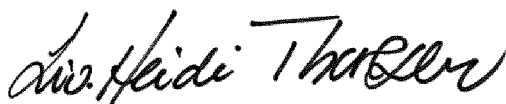
The aim the present invention is to provide ammonium nitrate based particles which do not easily convert into an explosive concentrated ammonium nitrate composition.

The problem to be solved by the present invention can be seen as "how to produce substantially water-insoluble and non-filterable ammonium nitrate based particles that do not easily convert into an explosive concentrated ammonium nitrate composition when contacted with water?".

A person skilled in the art will not be able to find the solution to the above mentioned problem when reading D4. D4 will teach a person skilled in the art to coat ammonium nitrate particles with CCB to reduce the explosive potential of ammonium nitrate by diluting the ammonium nitrate with an "agricultural acceptable" dilutant, and the resulting product will be resistant to separation of uncoated ammonium nitrate by mechanical disruption.

Thus, a person skilled in the art with the knowledge of the invention disclosed in D4 will not with reasonable expectation of success be able to arrive at the present claimed invention, as no disclosure is made of the role of the binder as disclosed in our invention, namely its swelling properties when used in such an amount (e.g. 2 wt%) that a substantially water-insoluble and non-filterable hydrogel is formed after contacting the composition with water. We also point out that the contacting with water according to the invention takes places AFTER the guar gum has been applied to the ammonium nitrate based particles, while in D4, the water is used as a primary binder BEFORE the guar gum is applied to the ammonium nitrate based particles.

Yours faithfully,
Onsagers AS
Association 317



Liv-Heidi Thoresen
Enclosures:

Amended claims – clean copy
Amended claims with annotations

CLAIMS

1. A solid, particulate, non-explosive composition comprising ammonium
5 nitrate-based particles and such an amount of a gelling agent that a
substantially water-insoluble and non-filterable hydrogel is formed after
said solid composition is contacted with water, characterized in that said
solid composition has a maximum organic carbon content of 0.4 weight%
and an average particle size of at least 1 mm.
10
2. The composition according to claim 1, characterized in that said average
particle size of the solid composition is an average diameter as determined
by sieving, ranging from 1 to 25 mm, preferably from 2 to 8 mm.
- 15 3. The composition according to any one of claims 1 to 2, characterized in
that said gelling agent is selected from the group of gum-based gelling
agents.
4. The composition according to any one of claims 1 to 3, characterized in
20 that said gelling agent is selected from the group of xanthan gum-based
gelling agents and guar gum-based gelling agents, most preferably is a
xanthan gum-based gelling agent.
5. The composition according to any one of claims 1 to 4, characterized in
25 that said ammonium nitrate-based particles comprise a coating of the
gelling agent.
6. The composition according to any of the claims 1 to 4, characterized in that
30 said composition comprises the gelling agent integrated within the
ammonium nitrate-based particles.

7. The composition according to any of the claims 1 to 4, characterized in that said composition is a physical blend of said ammonium nitrate-based particles and gelling agent particles of similar appearance.
- 5
8. The composition according to claim 4, characterized in that the amount of xanthan gum-based gelling agent or guar gum-based gelling agent is selected to a maximum of approximately 0.7 weight%, relative to the total weight of the composition.
- 10
9. The composition according to any one of claims 1 to 8, characterized in that said contacting with water is done by dissolving, dispersing, storing or (fine) grinding.
- 15
10. Use of a gelling agent in such an amount that a substantially water-insoluble and non-filterable hydrogel is formed, for the manufacture of a solid, particulate, non-explosive composition comprising ammonium nitrate-based particles, for limiting its potential misuse as a precursor for an explosive composition.
- 20
11. Use of a gelling agent according to claim 10, wherein said solid, particulate, non-explosive composition has a maximum organic carbon content of 0.4 weight%.
- 25
12. Use according to any one of claims 10 and 11, wherein said gelling agent is selected from the group of gum-based gelling agents, in particular xanthan gum-based gelling agents and guar gum-based gelling agents, most preferably is a xanthan gum-based gelling agent.

13. Method for producing a solid, particulate, non-explosive composition, comprising ammonium nitrate-based particles and a gelling agent, comprising the consecutive steps of :
- providing ammonium nitrate-based particles ;
 - 5 - providing a gelling agent in powder form ; and
 - mixing said powder in such an amount with said ammonium nitrate-based particles that a substantially water-insoluble and non-filterable hydrogel is formed if said solid composition is contacted with water,
 - 10 wherein said solid composition obtained has an average particle size of at least 1 mm and a maximum organic carbon content of 0.4 weight%.
14. Method for producing a solid, particulate, non-explosive composition, comprising ammonium nitrate-based particles and a gelling agent, comprising the consecutive steps of :
- 15 - providing ammonium nitrate-based particles ;
 - providing a suspension of a gelling agent, suspended in a liquid; and
 - spraying said suspension with such an amount of gelling agent onto said ammonium nitrate-based particles that a substantially water-insoluble and non-filterable hydrogel is formed if said solid composition is contacted with
 - 20 water,
 - wherein said solid composition obtained has an average particle size of at least 1 mm and a maximum organic carbon content of 0.4 weight%.
15. Method for producing a solid, particulate, non-explosive composition, comprising ammonium nitrate-based particles and a gelling agent, comprising the consecutive steps of :
- 25 - providing an ammonium nitrate-based melt comprising a gelling agent ;
 - and
 - granulating said melt, thus producing ammonium nitrate-based particles comprising said gelling agent in such an amount that a substantially water-
 - 30

insoluble and non-filterable hydrogel is formed if said solid composition is contacted with water,
wherein said solid composition obtained has an average particle size of at least 1 mm and a maximum organic carbon content of 0.4 weight%.

5

16. Use of a composition according to any one of claims 1 to 9 as a non-explosive fertilizer composition for limiting its potential misuse as a precursor for an explosive composition.

10 17. Use according to claim 16, wherein said non-explosive fertilizer composition is selected from the group of a straight N-fertilizer, a CAN-fertilizer, an NPK-fertilizer, an NP-fertilizer and/or a high N-NPK-fertilizer.

15 18. Use of a composition according to any one of claims 1 to 9 as a slow release, a sustained release or a controlled release ammonium-nitrate based fertilizer.

* * * * *

20

REIVINDICACIONES

1. Una composición no explosiva, particulada y sólida que comprende
5 partículas a base de nitrato de amonio y un agente gelificante que es capaz de formar un hidrogel que no puede filtrarse y disolverse sustancialmente en agua después de que dicha composición sólida se pone en contacto con agua, caracterizada porque dicha composición sólida tiene un contenido orgánico de carbono máximo de 0,4% en peso y
10 un tamaño de partícula promedio de al menos 1 mm.
2. La composición de acuerdo con la reivindicación 1, caracterizada porque dicho tamaño de partícula promedio de la composición sólida es un diámetro promedio determinado por tamizado, que varía de 1 a 25 mm,
15 preferiblemente de 2 a 8 mm.
3. La composición de acuerdo con una cualquiera de las reivindicaciones 1 a 2, caracterizada porque dicho agente gelificante se selecciona del grupo de agentes gelificantes a base de goma.
20
4. La composición de acuerdo con una cualquiera de las reivindicaciones 1 a 3, caracterizada porque dicho agente gelificante se selecciona del grupo de agentes gelificantes a base de goma de xantato y agentes gelificantes a base de goma guar, de mayor preferencia un agente gelificante a base
25 de goma de xantato.
5. La composición de acuerdo con una cualquiera de las reivindicaciones 1 a 4, caracterizada porque dichas partículas a base de nitrato de amonio comprenden un recubrimiento del agente gelificante.
30

6. La composición de acuerdo con cualquiera de las reivindicaciones 1 a 4, caracterizada porque dicha composición comprende el agente gelificante integrado dentro de las partículas a base de nitrato de amonio.
- 5 7. La composición de acuerdo con cualquiera de las reivindicaciones 1 a 4, caracterizada porque dicha composición es una mezcla física de dichas partículas a base de nitrato de amonio y partículas de agente gelificante de aspecto similar.
- 10 8. La composición de acuerdo con la reivindicación 4, caracterizada porque la cantidad de agente gelificante a base de goma de xantato o agente gelificante a base de goma guar se selecciona hasta un máximo de aproximadamente 0,7% en peso, con relación al peso total de la composición.
- 15 9. La composición de acuerdo con una cualquiera de las reivindicaciones 1 a 8, caracterizada porque dicho contacto con agua se realiza mediante disolución, dispersión, almacenamiento o molienda (fina).
- 20 10. Uso de un agente gelificante que es capaz de formar un hidrogel que no puede filtrarse y disolverse sustancialmente en agua, para la fabricación de una composición no explosiva, particulada y sólida que comprende partículas a base de nitrato de amonio, para limitar su posible uso indebido como precursor para una composición explosiva.
- 25 11. Uso de un agente gelificante de acuerdo con la reivindicación 10, en donde dicha composición no explosiva, particulada y sólida tiene un contenido orgánico de carbono máximo de 0,4% en peso.

12. Uso de acuerdo con una cualquiera de las reivindicaciones 10 y 11, en donde dicho agente gelificante se selecciona del grupo de agentes gelificantes a base de goma, en particular agentes gelificantes a base de goma de xantato y agentes gelificantes a base de goma guar, de mayor preferencia un agente gelificante a base de goma de xantato.
13. Método para producir una composición no explosiva, particulada y sólida que comprende partículas a base de nitrato de amonio, que comprende las etapas consecutivas de:
- proporcionar partículas a base de nitrato de amonio;
 - proporcionar un agente gelificante en forma de polvo; y
 - mezclar dicho polvo con dichas partículas a base de nitrato de amonio, en donde la composición obtenida tiene un tamaño de partícula promedio de al menos 1 mm y un contenido orgánico de carbono máximo de 0,4% en peso.
14. Método para producir una composición no explosiva, particulada y sólida que comprende partículas a base de nitrato de amonio, que comprende las etapas consecutivas de:
- proporcionar partículas a base de nitrato de amonio;
 - proporcionar una suspensión de un agente gelificante, suspendida en un líquido; y
 - pulverizar dicha suspensión sobre dichas partículas a base de nitrato de amonio,
- en donde la composición obtenida tiene un tamaño de partícula promedio de al menos 1 mm y un contenido orgánico de carbono máximo de 0,4% en peso.

15. Método para producir una composición no explosiva, particulada y sólida que comprende partículas a base de nitrato de amonio, que comprende las etapas consecutivas de:
- proporcionar una masa fundida a base de nitrato de amonio que comprende un agente gelificante; y
 - granular dicha masa fundida, produciendo así partículas a base de nitrato de amonio que comprenden dicho agente gelificante,
- en donde la composición obtenida tiene un tamaño de partícula promedio de al menos 1 mm y un contenido orgánico de carbono máximo de 0,4% en peso.
16. Uso de una composición de acuerdo con una cualquiera de las reivindicaciones 1 a 9 como una composición fertilizante no explosiva para limitar su posible uso indebido como precursor de una composición explosiva.
17. Uso de acuerdo con la reivindicación 16, en donde dicha composición fertilizante no explosiva se selecciona del grupo de un fertilizante N directo, un fertilizante CAN, un fertilizante NPK, un fertilizante NP y/o un fertilizante N-NP alto.
18. Uso de una composición de acuerdo con una cualquiera de las reivindicaciones 1 a 9 como un fertilizante a base de nitrato de amonio de liberación lenta, de liberación sostenida o de liberación controlada.

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