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(71) Applicant: YARA INTERNATIONAL ASA [NO/NO];
Drammensveien 131, 0277 Oslo (NO).

(72) Inventors: COLPAERT, Filip; Klossestraat 54, B-9052
Zwijnaarde (BE). LEDOUX, Francois; 1, square Paul
Fort, 95240 Corneilles en Parisis (FR). VAN BELZEN,
Ruud; Blindenhoek 4, 4331 CA Middelburg (NL).

(74) Agents: PAPPAERT, Kris et al.; E. Gevaertdreef 10a,
9830 Sint-Martens-Latem (BE).

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Declarations under Rule 4.17:

— of inventorship (Rule 4.17(iv))

Published:

— with international search report (Art. 21(3))

(54) Title: LIQUID UREASE INHIBITOR FORMULATIONS

(57) Abstract: The present application generally relates to a method for the manufacture of a liquid composition essentially consisting of an organic solvent of the type glycol and a urease inhibitor of the type phosphoric triamide and products obtained therewith.



WO 2017/144698 A1

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 YARA-009-PCT	FOR FURTHER ACTION		See Form PCT/IPEA/416
International application No. PCT/EP2017/054397	International filing date (<i>day/month/year</i>) 24.02.2017	Priority date (<i>day/month/year</i>) 24.02.2016	
International Patent Classification (IPC) or national classification and IPC INV. C05G3/08			
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>6</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>8</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which have been amended and/or sheets containing rectifications authorized by this Authority, unless those sheets were superseded or cancelled, and any accompanying letters (see Rules 46.5, 66.8, 70.16, 91.2, and Section 607 of the Administrative Instructions).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 21.12.2017		Date of completion of this report 24.05.2018	
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

1-49 as originally filed

Claims, Numbers

1-20 filed on 23-03-2018

Drawings, Sheets

1/6-6/6 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/EP2017/054397

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 15.01.2018 (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>1-20</u>
	No: Claims	
Inventive step (IS)	Yes: Claims	<u>1-20</u>
	No: Claims	
Industrial applicability (IA)	Yes: Claims	<u>1-20</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

Re 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. They have been cited by the applicant in the description:

- D2 US 2015/143860 A1 (MCKNIGHT GARY DAVID [US] ET AL) 28 May 2015 (2015-05-28)
- D3 WO 2015/161195 A1 (MCKNIGHT GARY DAVID [US]; RAYBORN RANDALL LINWOOD [US]; PARKER DAVID B) 22 October 2015 (2015-10-22)

Inventive step

- 1 Claim 1
- 1.1 D2 (examples 3,8; paragraph [0054] - paragraph [0059]; paragraph [0068]; claims) and D3 (page 6, line 3 - line 16; page 7, line 22 - page 8, line 11; examples 3,8; claims) disclose liquid delivery formulations of urease inhibitors and/or nitrification inhibitors designed for urea fertilizers. The formulations are made by dissolving nBTPT and propylene glycol at 38°C in a formulation comprising DMSO, DCD and thiourea. After nBTPT and propylene glycol addition, the mixture is cooled to a temperature below 30°C before packaging. Optionally the formulations comprise a dye.
- 1.2 The subject-matter of claim 1 therefore differs from this known method in that the liquid composition is maintained at a temperature in the range of 60 to 80°C.
- 1.3 The technical effect caused by this difference is the production of a liquid composition having a lower crystallisation temperature.
- 1.4 The problem to be solved by the present invention may therefore be regarded as providing a method of manufacturing an improved composition.
- 1.5 None of the cited document defines the crystallisation temperature of the liquid formulation, there is also no hint in any of the cited document to maintain the liquid composition to a temperature above the melting temperature of the urease inhibiting composition. Therefore there is no guidance in any of those document that would lead the man skilled in the art

to carry out a method to manufacture a liquid composition as defined in claim 11. The subject-matter of claim 1 involves therefore an inventive step (Art. 33(3) PCT).

- 1.6 Claims 2-12 are dependent on claim 1 and as such also fulfil the requirements of the PCT regarding novelty and inventive step.
- 2 Claims 13, 17 and 20
- 2.1 D2 discloses (examples 1-3, 5-9, 14-17 and 19, table 1) liquid compositions comprising propylene glycol, another solvent and nBTPT and optionally a dye having a crystallisation temperature of less than 25°F (-3.8°C, ex. 9), or less than 10°F (-12°C, ex. 7) or less than 5°C (-15°C, other cited examples). The crystallisation temperature in D2 is called chill point, which has no international meaning recognized, however, it is believed that the chill point and crystallisation temperature have the same meaning in the present case.
- 2.2 The subject-matter of claim 13 therefore differs from this known composition no other solvent are present.
- 2.3 The technical effect caused by this difference is the production of a liquid composition consisting essentially of a organic solvent of the type glycol and a urease inhibitor of the type triamide having a lower crystallisation temperature.
- 2.4 The objective technical problem can be therefore considered to provide a liquid composition consisting essentially of a organic solvent of the type glycol and a urease inhibitor of the type triamide having improved properties.
- 2.5 None of the cited document defines the crystallisation temperature of the liquid formulation. Therefore there is no hint in any of those document that would lead the man skilled in the art to achieve a liquid composition as defined in claim 13. The composition of D2 consisting only of glycol and nBTPT (example 18) shows a chill point of 7.7°C, all compositions displaying a crystallisation temperature below 0°C comprise other co-solvent or compound (mainly DMSO). Therefore, also from D2/D3, there is no pointer for the man skilled in the art for a liquid formulation as defined in claim 13.
- 2.6 The subject-matter of claim 13 involves therefore an inventive step (Art. 33(3) PCT).
- 2.7 The same reasoning applies mutatis mutandis to the subject-matter of corresponding independent claims 17 and 20, which define a product comprising the product defined by claim 13.

- 2.8 Claims 14-16, 18 and 19 are dependent on claims 13 and 17 and as such also fulfil the requirements of the PCT regarding novelty and inventive step.

Re Item VIII

Certain observations on the international application

The term "essentially" used in claim 13 is vague and unclear and leaves the reader in doubt as to the meaning of the technical feature to which it refers, thereby rendering the definition of the subject-matter of said claim unclear, Article 6 PCT.

European Patent Office
Patentlaan 2
2280 HV Rijswijk (ZH)
Nederland

Online filing

Sint-Martens-Latem, March 23, 2018

Our Ref.: YARA-009-PCT1

Re.: International patent application no. PCT/EP2017/054397 filed on
February 24, 2017

In the name of Yara International ASA

Dear Sir,

Dear Madam,

Further to the Written Opinion issued by the International Preliminary Examination authority and mailed on January 24, 2017 in connection with the above-mentioned patent application, the Applicant hereby submits his observations and arguments to address the objections raised therein.

1. Claim amendments

Claim 1 was amended by making the range of 60 to 80°C non-optional. In addition, only alternative i) was retained in amended claim 1. Basis for these amendments can be found in previous claim 1.

MAIN OFFICE

Edgard Gevaertdreef 10a
B-9830 Sint-Martens-Latem
Belgium

LEUVEN BRANCH

Groenveldstraat 13
B-3001 Leuven (Heverlee)
Belgium

Dependent claim 2 was added. Its subject matter is directed to step ii) of previous claim 1. Basis for new claim 1 can be found in previous claim 1.

The remaining claims were renumbered to take the addition of new claim 2 into account.

Amended claim 7 (former claim 6) was amended by deleting the range of the third temperature because this subject matter is already present in amended claim 1. Basis for amended claim 7 can be found in former claim 6.

We respectfully submit that the amendments do not extend beyond the contents of the application as filed.

All amendments have been made without prejudice to the possibility of future reinstatement of any excluded matter and/or subsequent filing of divisional applications based on any matter contained in the application as filed.

2. Novelty and Inventive Step (Articles 33(2) and 33(3) PCT)

Claim 1 was amended as suggested by the Examiner. In the Written Opinion of 24/01/2018, the Examiner particularly indicated that such a claim would be found new and inventive. Therefore, it is submitted that amended claim 1 complies with Articles 33(2) and 33(3) PCT.

The remaining claims either depend on claim 1 – and are new and inventive at least because of their dependence - or were already acknowledged to be new and inventive by the International Preliminary Examining Authority.

3. Request

In light of the amendments to the claims, the applicant respectfully submits that the objections raised by the Examiner have been overcome, and that the amended claims fulfill the requirements of the PCT. The applicant respectfully requests an indication in this respect in the International Preliminary Report on Patentability.

However, should the Examiner not agree, a telephonic interview with the undersigned, preferably combined with a further written opinion providing a further opportunity to amend the claims, is requested.

Respectfully submitted,

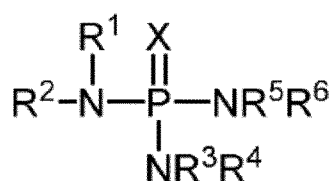
A handwritten signature in black ink, appearing to read 'K. Pappaert', with a stylized flourish above it.

Kris Pappaert, PhD
Professional Representative before the EPO

Encl.: amended claims (amendments indicated and retyped)

Claims (clean copy)

1. A method for the manufacture of a liquid composition essentially consisting of an organic solvent of the type glycol and a urease inhibitor of the type phosphoric triamide, and optionally a dye system, the method comprising the step of heating an amount of said organic solvent of the type glycol to a first temperature at least above 20 °C but below the melt temperature of said urease inhibitor of the type phosphoric triamide, and subsequently adding an amount of solid urease inhibitor of the type phosphoric triamide in said amount of organic solvent of the type glycol at a second temperature which is at most about equal to the first temperature, characterized in that the method comprises the step of
 - i) maintaining the liquid composition essentially consisting of an organic solvent of the type glycol and a urease inhibitor of the type phosphoric triamide, and optionally a dye system, at a third temperature, which temperature is greater than the first temperature, in the range of 60 to 80 °C.
2. The method according to claim 1 further comprising the step of subjecting the liquid composition essentially consisting of an organic solvent of the type glycol and a urease inhibitor of the type phosphoric triamide, and optionally a dye system, to an ultrasonic treatment.
3. The method according to claim 1 or 2, wherein the urease inhibitor of the type phosphoric triamide is a compound of formula (I):



(I)

wherein :

X is oxygen or sulphur;

R¹ is selected from the group consisting of alkyl, cycloalkenyl, aralkyl, aryl, alkenyl, alkynyl, and cycloalkyl; and

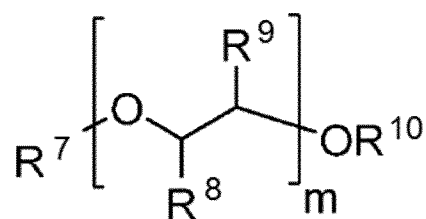
R² is selected from the group consisting of hydrogen, alkyl, cycloalkenyl, aralkyl, aryl, alkenyl, alkynyl, and cycloalkyl, or

R¹ and R² together may form an alkylene or alkenylene chain which may optionally include one or more heteroatoms of divalent oxygen, nitrogen or sulphur, completing a 4-, 5-, 6-, 7-, or 8-membered ring structure ; and

R^3 , R^4 , R^5 and R^6 are individually selected from the group consisting of hydrogen and alkyl having 1 to 6 carbon atoms, and

R^1 , R^2 , R^3 , R^4 , R^5 and R^6 , when not hydrogen, may each be optionally substituted with one or more selected from the group consisting of trihalomethyl, naphthoxy, alkyl, halogen, arylmercapto, phenoxy, phenyl, nitro, cyano, amino, alkylamino, dialkylamino, alkoxy, mercapto, alkylmercapto, alkylcarbonyl, arylamino, arylcarbonyl, alkoxycarbonyl, carboxy, diarylamino, and carbonamide.

4. The method according to any one of the aforementioned claims, wherein the organic solvent of the type glycol is a compound of formula (II)



(II)

wherein

R^7 is hydrogen or alkyl ;

R^8 is hydrogen or alkyl

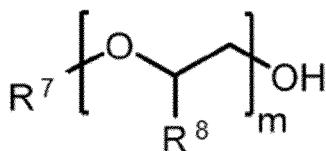
R^9 is hydrogen, =O, or alkyl ;

R^{10} is hydrogen, alkyl, or acyl ; and

m is 1 to 4 ;

wherein R^7 and R^8 , or R^7 and R^{10} together may form a ring comprising one or more oxygen atoms ;

preferably wherein the organic solvent of the type glycol is a compound of formula (III)



(III)

wherein :

R^7 is hydrogen or alkyl ;

R^8 is hydrogen or alkyl ; and

m is 1 to 4.

5. The method according to any one of the aforementioned claims, wherein the urease inhibitor is N-(n-butyl) thiophosphoric triamide (nBTPT) and/or wherein the organic solvent is propylene glycol, diethylene glycol monomethyl ether (DEGMME), diethylene glycol monobutyl ether (DEGMBE), or combinations thereof.
6. The method according to any one of the aforementioned claims, wherein the amount of said urease inhibitor of the type phosphoric triamide ranges from 1 to 45 weight%, relative to the total weight of said liquid composition and/or wherein the amount of said organic solvent of the type glycol ranges between 55 and 99 weight%, relative to the total weight of said liquid composition.
7. The method according to any one of the aforementioned claims, wherein the first temperature is in the range of 20 to 60 °C and/or wherein the second temperature is in the range of 20 to 60 °C.
8. The method according to any one of the aforementioned claims, wherein the liquid composition essentially consisting of an organic solvent of the type glycol and a urease inhibitor of the type phosphoric triamide, and optionally a dye system, is kept at a third temperature for a period of a few minutes to 4 hours.
9. The method according to any one of the aforementioned claims, wherein the liquid composition essentially consisting of an organic solvent of the type glycol and a urease inhibitor of the type phosphoric triamide, and optionally a dye system, is ultrasonically treated for a period of a few minutes to 4 hours.
10. The method according to any one of the aforementioned claims further comprising a step wherein an amount of a dye system is added to the liquid composition essentially consisting of an organic solvent of the type glycol and a urease inhibitor of the type phosphoric triamide at a fourth temperature, preferably at least 20 °C.
11. The method according to claim 10, wherein the amount of said dye system ranges between 0 and 1.0 weight%.
12. The method according to any one of the aforementioned claims, comprising the steps of :
 - heating up an amount of propylene glycol to 35 to 50 °C ;
 - adding 10 to 25 weight%, relative to the total weight of said liquid composition, of N-(n-butyl) thiophosphoric triamide (nBTPT) in said propylene glycol;

- applying an ultrasonic treatment for 15 minutes ;
 - maintaining said liquid composition essentially consisting of propylene glycol and nBTPT at 60 to 80 °C; and
 - optionally, adding 0.25 weight%, relative to the total weight of said liquid composition, of a dye system to said liquid composition essentially consisting of propylene glycol and nBTPT at least at 20°C.
13. A liquid composition essentially consisting of an organic solvent of the type glycol and a urease inhibitor of the type phosphoric triamide, and optionally a dye system, having a crystallization temperature of the urease inhibitor of the type phosphoric triamide in said organic solvent of the type glycol of less than 0 °C.
 14. A liquid composition according to claim 13, having a crystallization temperature of the urease inhibitor of the type phosphoric triamide in said organic solvent of the type glycol of less than -2 °C, preferably less than -4°C, more preferably less than -6°C, most preferably less than -8°C.
 15. A liquid composition according to claim 13 or 14, essentially consisting of propylene glycol (PG), in particular propaan-1,2-diol, as a solvent and 10 to 20 weight% of N-(n-butyl) thiophosphoric triamide (nBTPT), in particular 15 to 18 weight%, more in particular about 17.5 weight%, relative to the total weight of the liquid solution.
 16. A liquid composition according to claims 13 to 14, essentially consisting of diethylene glycol monomethyl ether (DEGMME), diethylene glycol monobutyl ether (DEGMBE), or combinations thereof, optionally further including propylene glycol (PG), as a solvent and 10 to 40 weight% of N-(n-butyl) thiophosphoric triamide (nBTPT), in particular 15 to 30 weight%, more in particular about 25 weight%, relative to the total weight of the liquid solution.
 17. A solid particulate urea-based composition, comprising a solid particulate urea-based compound and the liquid composition according to any one of claims 13 to 16, wherein the liquid composition forms at least partially a coating on at least part of the particles comprising the urea-based compound.
 18. The solid particulate urea-based composition according to claim 17, wherein the urea-based compound is selected from the group of urea, urea calcium sulphate (UCaS), urea calcium nitrate (UCaN), urea magnesium nitrate (UMgN), urea calcium phosphate (UCaP), urea magnesium phosphate (UMgP), urea superphosphate (USP), urea calcium ammonium nitrate (UCAN) , urea

ammonium sulphate (UAS), urea ammonium phosphate (UAP), urea potassium salts (UK), urea-based compound NPK fertilizer, and mixtures thereof.

19. The solid particulate urea-based composition according to claim 17, wherein the composition is a physical blend of the urea-based compound in particulate form and one or more components selected from the group of nitrates, phosphates, sulphates and chlorides in particulate form, selected from the group of :
ammonium nitrate, calcium nitrate, calcium ammonium nitrate, sodium nitrate, ammonium sulphate nitrate, potassium ammonium nitrate, ammonium phosphate, such as mono-ammonium phosphate (MAP) and di-ammonium phosphate (DAP), calcium bis(dihydrogen orthophosphate), super phosphate, triple superphosphate, rock phosphate, potassium sulphate, potassium magnesium sulphate, ammonium sulphate (AS), urea ammonium sulphate, urea calcium ammonium nitrate, urea ammonium sulphate, potassium chloride (MOP), potassium sulphate (SOP), urea potassium salts (UK), urea-based compound NPK fertilizer, or mixtures thereof.
20. A liquid urea-based composition, comprising a dissolved urea-based compound and the liquid composition according to any one of claims 13 to 16, wherein said liquid composition is intimately mixed with the urea-based compound, preferably selected from the group of urea, urea ammonium nitrate (UAN), urea calcium nitrate (UCaN), or mixtures thereof, dissolved in a solvent, preferably in water.