

PATENT COOPERATION TREATY

ixn

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

PCT

To:

MIKULECKY
Clariant Produkte (Deutschland) GmbH
Patent Management
Industriepark Höchst, G 860
D-65926 Frankfurt am Main
ALLEMAGNE

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL PRELIMINARY
REPORT ON PATENTABILITY
(PCT Rule 71.1)

Date of mailing
(day/month/year)

23.12.2016

Applicant's or agent's file reference
2014US404K WO

IMPORTANT NOTIFICATION

International application No.
PCT/EP2015/070567

International filing date (day/month/year)
09.09.2015

Priority date (day/month/year)
19.09.2014

Applicant
Clariant International Ltd.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary report on patentability and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary report on patentability. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

The applicant's attention is drawn to Article 33(5), which provides that the criteria of novelty, inventive step and industrial applicability described in Article 33(2) to (4) merely serve the purposes of international preliminary examination and that "any Contracting State may apply additional or different criteria for the purposes of deciding whether, in that State, the claimed inventions is patentable or not" (see also Article 27(5)). Such additional criteria may relate, for example, to exemptions from patentability, requirements for enabling disclosure, clarity and support for the claims.

Name and mailing address of the international
preliminary examining authority:



European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0
Fax: +49 89 2399 - 4465

Authorized Officer

Almalé Murillo, J

Tel. +49 89 2399-8059



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 2014US404K WO	FOR FURTHER ACTION		See Form PCT/PEA/416
International application No. PCT/EP2015/070567	International filing date (day/month/year) 09.09.2015	Priority date (day/month/year) 19.09.2014	
International Patent Classification (IPC) or national classification and IPC INV. C09K8/64			
Applicant Clariant International Ltd.			
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. ☒ (sent to the applicant and to the International Bureau) a total of <u>4</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. ☐ (sent to the International Bureau only) 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 25.04.2016		Date of completion of this report 23.12.2016	
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Fax: +49 89 2399 - 4465		Authorized officer Doslik, Natasa Telephone No. +49 89 2399-7873	



**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/EP2015/070567

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

1-20 as originally filed

Claims, Numbers

1-24 filed with the letter of 10-11-2016

Drawings, Sheets

1/2, 2/2 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/EP2015/070567

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 01.12.2016 (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>1-24</u>
	No: Claims	
Inventive step (IS)	Yes: Claims	<u>1-24</u>
	No: Claims	
Industrial applicability (IA)	Yes: Claims	<u>1-24</u>
	No: Claims	

2. Citations and explanations (Rule 70.7):

see separate sheet

Box No. VII Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

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:

- D1 YANG ET AL: "Phase behavior studies of quaternary systems containing N-lauroyl-N-methylglucamide/alcohol/alkane/water",
JOURNAL OF COLLOID AND INTERFACE SCIENCE, ACADEMIC PRESS, NEW YORK, NY, US,
vol. 320, no. 1, 19 February 2008 (2008-02-19), pages 283-289,
XP022517998,
ISSN: 0021-9797, DOI: 10.1016/J.JCIS.2007.12.043
- D2 ANAN YAGHMUR ET AL: "Microstructure Considerations of New Five-Component Winsor IV Food-Grade Microemulsions Studied by Pulsed Gradient Spin-Echo NMR, Conductivity, and Viscosity",
LANGMUIR,
vol. 19, no. 4, 1 February 2003 (2003-02-01), pages 1063-1068,
XP55163766,
ISSN: 0743-7463, DOI: 10.1021/la026404h
- D3 S. EZRAHI ET AL: "Structural Polymorphism in a Four-Component Nonionic Microemulsion",
JOURNAL OF COLLOID AND INTERFACE SCIENCE.,
vol. 191, no. 2, 1 July 1997 (1997-07-01), pages 277-290, XP055222720,
US
ISSN: 0021-9797, DOI: 10.1006/jcis.1997.4962
- D4 S. RAY ET AL: "Dynamics and Thermodynamics of Aerosol OT-Aided Nonaqueous Microemulsions",
LANGMUIR,
vol. 10, no. 8, 1 August 1994 (1994-08-01), pages 2511-2515,
XP55222728,
NEW YORK, NY; US
ISSN: 0743-7463, DOI: 10.1021/la00020a005
- D5 ELIANA ARENAS ET AL: "-glucalkanamide Surfactants with Chlorinated Hydrocarbons",
LANGMUIR,

vol. 12, no. 2, 1 January 1996 (1996-01-01), pages 588-590,
XP55101873,
ISSN: 0743-7463, DOI: 10.1021/la950360d

- D6 GREGORY J. MCFANN ET AL: "Phase behavior of nonionic surfactant/
oil/water systems containing light alkanes",
LANGMUIR,
vol. 9, no. 11, 1 November 1993 (1993-11-01), pages 2942-2948,
XP055222658,
NEW YORK, NY; US
ISSN: 0743-7463, DOI: 10.1021/la00035a035
- D7 US 2014/096969 A1 (ALI SYED AFAQ [US] ET AL) 10 April 2014
(2014-04-10)
- D8 US 2012/073817 A1 (VAN ZANTEN RYAN [US]) 29 March 2012
(2012-03-29)
- D9 LIRIO QUINTERO ET AL: "Proper Design Criteria of Microemulsion
Treatment Fluids for Enhancing Well Production",
SPE EUROPEC/EAGE ANNUAL CONFERENCE, 1 January 2012
(2012-01-01), XP55252450,
DOI: 10.2118/154451-MS
- D10 FRAN CR COIS BOUTON ET AL: "A QSPR Model for the prediction of
the fish tail temperature of C₁₂E₄/Water/Polar hydrocarbon oil systems",
LANGMUIR, AMERICAN CHEMICAL SOCIETY, NEW YORK, NY; US,
vol. 26, no. 11, 1 June 2010 (2010-06-01), pages 7962-7970,
XP002712937,
ISSN: 0743-7463, DOI: 10.1021/LA904836M
[retrieved on 2010-05-11]
- D11 WO 2009/158478 A2 (BAKER HUGHES INC [US]) 30 December 2009
(2009-12-30)

The amendments as filed with letter of 10.11.2016 do fulfill the requirements of Article 19 PCT.

D1 discloses phase behavior studies of Winsor type microemulsions of quaternary systems containing brine, 12.6 wt% and 17.7 wt% of N-lauroyl-N-methylglucamide, alcohol selected from 1-butanol, 1-pentanol or 1-hexanol and n-octane. Phase diagrams are shown for the concentration variations of N-lauroyl-N-methylglucamide in the three different alcohols, all showing also the specific point E, where the single phase microemulsion occurs. The difference to present claim 1 is, that the wt ratios for the present alcohol are higher than required by claim 1 (more than 6 wt% when applied to the above mentioned wt ratios of surfactant). Hence, claim 1 is novel in the light of D1.

D1 is seen as closest prior art for the subject-matter of claim 1. The differentiating feature is that the wt ratios of alcohol are significantly higher than as defined by claim 1. The underlying problem to be solved is seen in providing an alternative microemulsion of Winsor type IV. The teaching of D1 leads away from the required low amounts of alcohol between 1-6 wt% in connection with the higher amounts (12-30 wt%) of N-lauroyl-N-methylglucamide surfactant, thereby rendering the solution as disclosed in present claim 1 non obvious.

Hence, an inventive step is acknowledged for claim 1 and its dependent claims 2-16. Process claim 17 and its dependent claims 18-20, as well as the use claim 21 and its dependent claims 22-24 are linked to the subject-matter of the claimed microemulsion according to claims 1-16 and are therefore also regarded as being novel and inventive.

Re Item VII

Certain defects in the international application

- a) Claim 10 comprise a typing error: the term "isopropanil" should read "isopropanol".
- b) The units [gptg], [°F] on page 10,11,13,14,15,17 are not additionally expressed in terms of units as stipulated by Rule 10.1 PCT.

Dr. Klaus Mikulecky
European Patent Attorney
c/o Clariant Produkte (Deutschland) GmbH

Patent & License Management
Industriepark Höchst / G 860
65926 Frankfurt am Main

Telefon: 069 - 305 - 38 599
Fax: 069 - 305 - 38 465

EPO - Munich
31

15. Nov. 2016

Europäisches Patentamt
Bob-van-Bentham-Platz 1

80469 München

Unsere Zeichen
Dr. K. Mikulecky /rh

Datum
10.11.2016

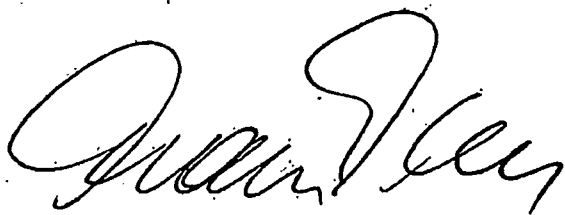
International Application No.: PCT/EP2015/070567
Applicant: Clariant International Ltd
Internal Reference: 2014US404K WO

Reply to the Written Opinion of the IPEA dated September 27, 2016.

Applicant files amended claims.

Claims 3, 4 and 5 are deleted. The remainder of claims and their dependencies are renumbered.

Applicant believes that the claims now are in condition for a positive IPER.



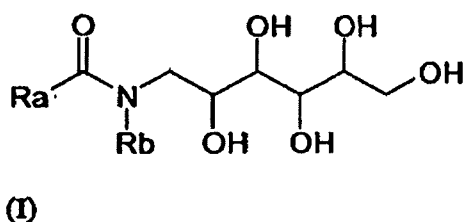
Dr. Klaus Mikulecky
European Patent Attorney
AV Nr. 47647 – Clariant International Ltd

Encl.

Amended claims (version with annotations and clean version)

Patent claims

1. Microemulsion, comprising water,
- 5 2 – 15 wt.-% of at least one organic solvent with flash point above 37.8 °C (100 °F) and pour point of 10 °C or lower,
- 1 – 6 wt.-% of at least one co-solvent that includes at least one alcohol, and
- 12 – 30 wt.-% of at least one N-Alkyl-N-acylglucamine surfactant, which is a Winsor type IV emulsion.
- 10
2. Microemulsion according to claim 1, wherein the glucamide surfactant is a compound of formula (I)



- 15 wherein
- Ra is a C₅-C₂₁-hydrocarbon residue, and
- Rb is a C₁-C₄-alkyl group.
3. Microemulsion according to claim 1 and/or 2, wherein Ra is C₇ to C₁₅
- 20 hydrocarbon.
4. Microemulsion according to one or more of claims 1 - 3, wherein Ra is an aliphatic group.
5. Microemulsion according to one or more of claims 1 - 4, wherein Ra is alkyl or alkenyl.
6. Microemulsion according to one or more of claims 1 - 5, wherein Rb is methyl.

7. Microemulsion according to one or more of claims 1 - 6, wherein in at least 50 wt.-% of the total amount of compounds according to formula (I) Ra is C₇ to C₉ alkyl and in up to 50 wt.-% Ra is C₁₁ to C₁₃ alkyl.

5

8. Microemulsion according to one or more of claims 1 - 7, wherein the first solvent is selected from the group consisting of naphthalene depleted alkyl arenes, terpenes, paraffinic solvents, fatty acid alkyl esters and butyl glycol ethers.

10

9. Microemulsion according to one or more of claims 1 - 8, wherein the co-solvent is selected from the group consisting of primary, secondary or tertiary monoalcohols having from 1 - 20 carbon atoms, and diols having from 1 - 20 carbon atoms.

15

10. Microemulsion according to one or more of claims 1 - 9, wherein the co-solvent is selected from the group consisting of propylene glycol, isopropanil, t-butanol, n-butanol, n-pentanol, n-hexanol, n-octanol and pentane-diol.

20

11. Microemulsion according to one or more of claims 1 - 10, further comprising a mutual solvent selected from the group consisting of 2-ethylhexanol and ethers of 2-ethylhexanol with ethylene glycol, polyethylene glycols or propylene glycol.

25

12. Microemulsion according to one or more of claims 1 - 11, comprising 14 - 25 wt.-% of the glucamide surfactant.

30

13. Microemulsion according to one or more of claims 1 - 12, comprising 3 - 5 wt.-% of the co-solvent.

14. Microemulsion according to one or more of claims 1 - 13, comprising 5 - 10 wt.-% of the first solvent.

15. Microemulsion according to one or more of claims 1 - 14, comprising up to 10 wt.-% of a mutual solvent.

16. Microemulsion according to one or more of claims 1 - 15, comprising water ad 100 wt.-%.

5 17. Process for recovering fluids during fracturing operations, the process comprising injecting a microemulsion according to one or more of claims 1 - 16 into the fractured formation.

10 18. Process according to claim 17, wherein the amount of microemulsion is 0.1 to 10 gallons of microemulsion per thousand gallons of fracturing fluid (0.01 - 1% by volume).

19. Process for stimulating an oil or gas well, comprising injection of water and a microemulsion according to one more of claims 1 - 18.

15

20. Process according to claim 19, wherein 0.1 to 10 gallons of microemulsion are used per 1000 gallons of water (0.01 - 1% by volume).

20 21. Use of a microemulsion according to one or more claims 1 - 16 as flowback aid during fracturing operations.

22. Use according to claim 21, wherein 0.1 to 10 gallons (0.01 - 1% by volume) of microemulsion are used per 1000 gallons of treatment fluid injected into oil or gas well.

25

23. Use of a microemulsion according to one or more of claims 1 - 16, in stimulating an oil or gas well by injection of water based fluids.

30 24. Use according to claim 23, wherein 0.1 to 10 gallons (0.01% - 1% by volume) of microemulsion are used per 1000 gallons of injected water.

Bitte beachten Sie, dass angeführte Nichtpatentliteratur (wie z. B. wissenschaftliche oder technische Dokumente) je nach geltendem Recht dem Urheberrechtsschutz und/oder anderen Schutzarten für schriftliche Werke unterliegen könnte. Die Vervielfältigung urheberrechtlich geschützter Texte, ihre Verwendung in anderen elektronischen oder gedruckten Publikationen und ihre Weitergabe an Dritte ist ohne ausdrückliche Zustimmung des Rechtsinhabers nicht gestattet.

Veillez noter que les ouvrages de la littérature non-brevets qui sont cités, par exemple les documents scientifiques ou techniques, etc., peuvent être protégés par des droits d'auteur et/ou toute autre protection des écrits prévue par les législations applicables. Les textes ainsi protégés ne peuvent être reproduits ni utilisés dans d'autres publications électroniques ou imprimées, ni rediffusés sans l'autorisation expresse du titulaire du droit d'auteur.

Please be aware that cited works of non-patent literature such as scientific or technical documents or the like may be subject to copyright protection and/or any other protection of written works as appropriate based on applicable laws. Copyrighted texts may not be copied or used in other electronic or printed publications or re-distributed without the express permission of the copyright holder.