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(54) **Title:** TREATMENT OF DEACTIVATED IONIC LIQUIDS

(57) **Abstract:** The present disclosure relates to a process for separating the cationic and anionic components from deactivated ionic liquids. The process includes reacting the deactivated ionic liquid with magnesium chloride in the presence of at least one base to precipitate the anionic component; thereby freeing the anionic as well as cationic component of the deactivating components. The anionic precipitate and the cationic component are subsequently recovered and reused for different applications. The ionic liquid can be reconstituted from the cationic component and another anionic component for use as a fresh ionic liquid.



TREATMENT OF DEACTIVATED IONIC LIQUIDS

FIELD

The present disclosure relates to ionic liquids. Particularly, the present disclosure relates to treatment of deactivated ionic liquids to separate its cationic and anionic components.

BACKGROUND

Ionic compounds, as the name suggests, are compounds comprising cations and anions. Typically, they consist of salts having melting point below 100 °C. Ionic liquids are known to be used in various applications such as catalysts, solvents and electrolytes in processes such as alkylation, polymerization, dimerization, oligomerization, acetylation, metatheses and copolymerization. For instance, United States Patent No. 7432408 recites a method for alkylation of isoparaffin and C₂-C₅ olefins using ionic liquids such as 1-butyl-4-methyl-pyridinium chloroaluminate (BMP), 1-butyl-pyridinium chloroaluminate (BP), 1-butyl-3-methyl-imidazolium chloroaluminate (BMIM) and 1-H-pyridinium chloroaluminate (HP) as the catalyst. United States Patent No. 7495144 also describes a method for alkylation of isoparaffin and C₂-C₅ olefins using a composite ionic liquid catalyst, wherein the ionic liquid is a mixture of acid ionic liquids such as 1-butyl-4-methyl-pyridinium chloroaluminate (BMP), 1-butyl-pyridinium chloroaluminate (BP), 1-butyl-3-methyl-imidazolium chloroaluminate (BMIM) and 1-H-pyridinium chloroaluminate (HP) and metal halides such as AlCl₃.

Ammonium, phosphonium, sulphonium, pyridinium and imidazolium are some of the commonly used cations; whereas BF₄⁻, PF₆⁻, haloaluminates such as Al₂Cl₇⁻ and Al₂Br₇⁻, [(CF₃SO₂)₂N]⁻, alkyl sulphates (RSO₃⁻), carboxylates (RCO₂⁻) are some of the commonly used anions in ionic liquids. However, when the haloaluminate containing ionic liquids are used in any of the afore-stated reactions, they get

deactivated due to various chemical entities present in the reaction such as hydrocarbons, conjunct polymers and water. Thus, upon the completion of the reaction, the ionic liquids, due to their deactivated state, are incapable of being reused for other reactions. Replenishing the stock becomes imperative; however the expensive nature of the chemical results in an exponential increase in the processing cost. Moreover, as the used ionic liquids have to be discarded, tremendous amount of waste is generated and valuable reagents get wasted.

There have been attempts to reconstitute and reuse ionic liquids. United States Patent Application No. 20100160145 recites a process for breaking down an ionic liquid catalyst which employs a secondary alcohol to achieve this. WO 2010062902 also describes a process for recycling ionic liquids. However, the process of WO 2010062902 facilitates this by removing aluminum trichloride from the ionic liquid by way of cooling or cooling and seeding the ionic liquid to precipitate out the aluminum trichloride.

The techniques that have been used for reuse of ionic liquids are, however, associated with certain disadvantages such as use of expensive reagents and time-consuming process steps. The present disclosure, therefore, provides a process for the treatment and reconstitution of ionic liquids which is cost effective as compared to the conventional process and also separates its cationic and anionic components to generate certain reaction products that may be effectively harnessed.

OBJECTS

Some of the objects of the present disclosure, of which at least one embodiment is adapted to provide, are described herein below:

It is an object of the present disclosure to provide a process for the reconstitution of deactivated ionic liquids.

It is another object of the present disclosure to provide a process for the separation of cationic and anionic components of the deactivated ionic liquid and for the reconstitution of fresh ionic liquids.

It is yet another object of the present disclosure to provide a process for the reconstitution of fresh ionic liquids from deactivated ionic liquid, which is simple and economical.

It is still another object of the present disclosure to provide a process for the reconstitution of fresh ionic liquids, which is environment friendly.

It is yet another object of the present disclosure to ameliorate one or more problems of the prior art or to at least provide a useful alternative.

Other objects and advantages of the present disclosure will be more apparent from the following description which is not intended to limit the scope of the present disclosure.

SUMMARY

The present disclosure provides a process for separating the cationic and anionic components from deactivated ionic liquids, said process comprising the following steps:

- a. dissolving said deactivated ionic liquid in a first solvent to obtain a first solution;
- b. dissolving magnesium chloride in a second solvent and heating at a temperature ranging from 25 to 80 °C to obtain a second solution;
- c. dissolving at least one base in said second solvent to obtain a third solution;
- d. preparing a dispersion by admixing said first solution, said second solution and said third solution, at atmospheric pressure, at a temperature ranging from 40 to 80 °C, at a speed of rotation ranging from 80 to 120 rpm;

- e. allowing said dispersion to stand for a time period ranging from 6 to 14 hours to obtain a phase separated dispersion;
- f. cooling said phase separated dispersion to a temperature lower than 40 °C followed by filtering to obtain:
 - i. a residue comprising a precipitate of said anionic component and magnesium chloride; and
 - ii. a biphasic filtrate having an organic phase comprising said first solvent and said deactivating components and an aqueous phase comprising said second solvent and said cationic component;
- g. washing said residue with said first solvent followed by drying to obtain purified precipitate;
- h. separating said organic phase from said aqueous phase to obtain separated organic phase and separated aqueous phase;
- i. distilling said separated organic phase to leave behind said deactivating components;
- j. distilling said aqueous phase leaving behind a solid residue comprising cationic component devoid of deactivating components; and
- k. recrystallizing and purifying said solid residue to obtain the cationic component devoid of deactivating components.

In accordance with the present process the cationic component is selected from the group consisting of 1-butyl-3-methyl imidazolium bromide, 1-butyl-3-methyl imidazolium chloride, 1-butyl-4-methylpyridinium chloride and 1-butyl-4-methylpyridinium bromide; the anionic component is aluminum chloride and the deactivating component is at least one from the group consisting of polymers, tar, hydrocarbons and moisture. Furthermore, the step of preparing a dispersion is carried out by at least one method from the group consisting of:

- i. adding said first solution and said second solution into said third solution in a drop-wise fashion; and
- ii. adding said second solution and said third solution into said first solution in a drop-wise fashion.

The precipitate is of Hydrotalcite represented by the Formula $M_1^{2+}M_2^{3+}(OH)_2A^{n-}yH_2O$, wherein M_1^{2+} is a divalent metal ion and M_2^{3+} is a trivalent metal ion in a ratio ranging from 2: 1 to 3: 1, A^{n-} is an anion where n is the integer from 1 to 3 and y represents the quantity of the interlayer water.

DETAILED DESCRIPTION

The disclosure will now be described with reference to the accompanying embodiments which do not limit the scope and ambit of the disclosure. The description provided is purely by way of example and illustration.

The embodiments herein and the various features and advantageous details thereof are explained with reference to the non-limiting embodiments in the following description. Descriptions of well-known components and processing techniques are omitted so as to not unnecessarily obscure the embodiments herein. The examples used herein are intended merely to facilitate an understanding of ways in which the embodiments herein may be practiced and to further enable those of skill in the art to practice the embodiments herein. Accordingly, the examples should not be construed as limiting the scope of the embodiments herein.

The description of the specific embodiments will so fully reveal the general nature of the embodiments herein that others can, by applying current knowledge, readily modify and/or adapt for various applications such specific embodiments without departing from the generic concept, and, therefore, such adaptations and modifications should and are intended to be comprehended within the meaning and range of equivalents of the disclosed embodiments. It is to be understood that the phraseology or terminology employed herein is for the purpose of description and not of limitation. Therefore, while the embodiments herein have been described in terms of preferred embodiments, those skilled in the art will recognize that the embodiments herein can be practiced with modification within the spirit and scope of the embodiments as described herein.

Ionic liquids are used as catalysts, solvents and electrolytes in different reactions such as polymerization and alkylation. During these reactions, the ionic liquids get deactivated due to contamination with different chemical entities such as polymers and hydrocarbons. The present disclosure provides a process for the recovery of the anionic component in a complex form and the cationic components of deactivated ionic liquids that may be recycled and reused for different applications. The ionic liquid can be reconstituted from the cationic component and fresh anionic component for use as a fresh ionic liquid.

The process of the present disclosure achieves the reconstitution of the ionic liquids by effecting the separation of its cationic and anionic components from the deactivating components and reconstituting the ionic liquid by combining the cationic component obtained with fresh anionic component to obtain a freshly reconstituted ionic liquid.

The deactivating components of the present disclosure are from the group consisting of polymers, tar, hydrocarbons and moisture. The process of the present disclosure achieves the reconstitution of the ionic liquids by effecting the separation of its cationic and anionic components from the deactivating components. Typically, the cationic component of the present disclosure is selected from the group consisting of 1-butyl-3-methyl imidazolium bromide, 1-butyl-3-methyl imidazolium chloride, 1-butyl-4-methylpyridinium chloride and 1-butyl-4-methylpyridinium bromide and the anionic component is a metal chloride which in one embodiment is aluminum chloride.

The process initially includes dissolving the deactivated ionic liquid in a first solvent to obtain a first solution. Typically, the first solvent is ethyl acetate. The ionic liquid content in the first solution ranges from 20 to 80 % by volume. Simultaneously, magnesium chloride is dissolved in a second solvent and heated at a temperature ranging from 20 to 80 °C to obtain a second solution. The second solvent is at least one from the group consisting of water and distilled water; and the magnesium

chloride content in the second solution ranges from 10 to 60 % by weight. Similarly, a third solution is prepared by dissolving a base such as sodium carbonate and sodium hydroxide in the second solvent; wherein the sodium carbonate and sodium hydroxide content in the third solution ranges from 5 to 35 % by weight and 10 to 50 % by weight respectively.

Next, the three solutions are mixed with each other in a characteristic fashion to obtain a dispersion. In one embodiment, the first solution and the second solution are added into the third solution, in a drop-wise fashion, to obtain the dispersion. In another embodiment, the second solution and the third solution are added in the first solution, in a drop-wise fashion, to obtain the dispersion. Typically, the step of preparing the dispersion is carried out at atmospheric pressure, at a temperature ranging from 40 to 80 °C, at a speed of rotation ranging from 80 to 120 rpm.

The step of formation of the dispersion takes place as mentioned herein below. Al^{3+} in the anionic component is converted to aluminum hydroxide at the alkaline pH caused by sodium carbonate and sodium hydroxide (the third admixture). At a higher pH, Mg^{2+} of magnesium chloride starts precipitating as it gets complexed with the hydrated oxide of aluminum to form the Mg-Al hydrotalcite (HT). When Mg/Al solution is dropped into the alkali solution i.e. the pH of the mother solution decreases from a high to a low value and the Mg-Al HT formed by direct conversion of $\text{Al}(\text{OH})_3^{3+}$ and $\text{Mg}(\text{OH})_2^{2+}$. Magnesium hydroxide is formed as an impurity in a very small quantity in the preparation of Mg-Al HT. Typically, the precipitate is of Hydrotalcites - represented by the general Formula $\text{M}_1^{2+}\text{M}_2^{3+}(\text{OH})_2\text{A}^n\text{yH}_2\text{O}$, wherein M_1^{2+} is a divalent metal ion and M_2^{3+} is a trivalent metal ion in a ratio ranging from 2: 1 to 3: 1, A^n is an anion where n is the integer 3 and y represents the quantity of the interlayer water. In one embodiment, the Hydrotalcite is $\text{Mg}_6\text{Al}_2\text{CO}_3(\text{OH})_{16}.4(\text{H}_2\text{O})$.

The dispersion is allowed to stand for a time period ranging from 6 to 14 hours to obtain a phase separated dispersion which is subsequently cooled at a temperature lower than 40 °C and filtered. The step of filtration yields a residue which is the precipitate described herein above and a biphasic filtrate. Typically, the biphasic

filtrate has an organic phase and an aqueous phase wherein the organic phase includes the first solvent and the deactivating components and the aqueous phase consists of the second solvent and the cationic component. The residue is washed with the first solvent and dried to obtain the purified Hydrotalcite precipitate. Thus, the anionic component gets freed of the deactivating components by forming a complex that may be used for further varied applications that are mentioned in the latter part of the specification.

The two phases of the filtrate are separated by the step of de-layering to yield the separated organic and aqueous phases. The organic phase is distilled to leave behind the deactivating components. The aqueous phase is also subjected to distillation to leave behind a solid residue containing the cationic component devoid of deactivating components and a salt. In one embodiment, the salt is sodium chloride. The cationic component is recovered and purified by undergoing extraction from the solid residue by at least one extracting agent, followed by distilling to obtain the purified cationic component. Typically, the extracting agent is dichloromethane. Once, the deactivating components are separated, the purified cationic component is ready to be reconstituted with another anionic component for use as a fresh ionic liquid.

Therefore, in the present process the deactivated ionic liquid is reconstituted and can be recycled. The separated anionic component complex obtained according to the present process can prove to be valuable for use as a catalyst, catalyst support, antacid and hydrogen scavenger for different processes such as plastic manufacturing and as an ion exchanger. Thus, the process of the present disclosure effectively reuses the deactivated ionic liquid which would earlier have been discarded.

The present disclosure is further described in light of the experiments provided herein below which are set forth for illustration purpose only and not to be construed for limiting the scope of the disclosure. These laboratory scale experiments can be scaled up to industrial/commercial scale.

Example 1: Process for the treatment of deactivated ionic liquids to separate the cationic and anionic componentsPreparation of fresh ionic liquid catalyst (1-Butyl-3-methylimidazolium bromide + aluminum chloride)

The setup consisted of a 5 L three necked round bottom (RB) flask fitted with an overhead stirrer and placed in an ice bath at 0-5 °C. The flask was clamped to provide stability under stirring. The whole assembly was kept under nitrogen atmosphere. 680 g of [BMIM]Br was weighed and carefully charged into the flask through a funnel. Stirring was started at a slow speed. Next, 830 g of AlCl₃ was weighed and added slowly into the flask. The charging of AlCl₃ was completed in 1.5 hours after which the mixture was stirred for 2 hours to mix the reactants properly. The final catalyst was closed tightly under nitrogen conditions.

Preparation of deactivated ionic liquid catalyst (Alkylation using the ionic liquid catalyst prepared herein above)

9.2 liters of a paraffin stream containing 10-15% C₁₀ to C₁₄ olefins and 3.6 liters of benzene were added into a 25 liter round bottom reactor kept inside a heating mantle. The agitator was started and the heating coils were switched on. When the mixture attained the temperature of 45 °C, 0.13 kg of the afore-stated catalyst was charged and stirred for 10 minutes. After standing for 10 minutes, the hydrocarbon and the catalyst layers were separated and the bottom catalyst layer was recycled back with the same quantities of a fresh olefin stream and benzene.

The process of the present disclosure – of reconstitution of the ionic liquid - was carried out on deactivated ionic liquids by the following two non-limiting routes. The processes and results are presented herein below.

Separation of the cationic and anionic components from the deactivated ionic liquid catalyst: Route 1

In a 1000 ml round bottom flask, 118 g of sodium carbonate and 4 g of sodium hydroxide were taken in 250 ml of water. In an addition funnel, 25 g of the afore-

stated deactivated ionic liquid was added in 100 ml of ethyl acetate. In another addition funnel, 55 g of magnesium chloride was dissolved in 125 ml water accompanied by heating at 60 °C. Both the addition funnels were then attached to the round bottom flask and the temperature was maintained at 60 °C using a water bath. The stirring was done by an overhead stirrer at a stirring speed of 100 rpm. Both the solutions were added drop wise into the round bottom flask in 60 minutes. The formation and retaining of hydrotalcite was given 12 hrs. After filtration, the solid obtained was washed with ethyl acetate and deionized water 3-4 times to remove any impurities followed by drying in an oven in vacuum. Out of the biphasic filtrate, the ethyl acetate layer was distilled to get the tar. Similarly, the aqueous layer was also distilled. The residue that was left behind was washed with dichloromethane 2 times. The dichloromethane was then distilled off to get the cationic component-[BMIM]Br. The yield of [BMIM]Br was 60% and that of the hydrotalcite was 90 %.

Separation of the cationic and anionic components from the deactivated ionic liquid catalyst: Route 2

In a 1000 ml round bottom flask, 52.76 g of deactivated ionic liquid was taken in 100 ml of ethyl acetate. In an addition funnel, 55.33 g of sodium hydroxide were taken in 125 ml of water. In another addition funnel, 97.78 g of magnesium chloride was dissolved in 125 ml of water by heating at 60 °C. Both the addition funnels were then attached to the round bottom flask and the temperature was maintained at 60 °C using a water bath. The stirring was done by an overhead stirrer at a stirring speed of 100 rpm. Both the solutions were added drop wise into the round bottom flask in 60 minutes. The formation and retaining of hydrotalcite was given 12 hrs. The reaction was then cooled and filtered. After filtration, the solid obtained was washed with ethyl acetate and deionized water 3-4 times to remove any impurities followed by drying in an oven in vacuum. Out of the biphasic filtrate, the ethyl acetate layer was distilled to get the tar. Similarly, the aqueous layer was also distilled. The residue that was left behind was washed with dichloromethane 2 times. The dichloromethane was then distilled off to get the cationic component-[BMIM]Br. The yield of [BMIM]Br was 55 % and that of the hydrotalcite was 88 %.

Inference:

The process of the present disclosure for separating the cationic and anionic components from ionic liquids can be successfully applied for deactivated ionic liquids.

Throughout this specification the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the disclosure to achieve one or more of the desired objects or results.

Any discussion of documents, acts, materials, devices, articles or the like that has been included in this specification is solely for the purpose of providing a context for the disclosure. It is not to be taken as an admission that any or all of these matters form a part of the prior art base or were common general knowledge in the field relevant to the disclosure as it existed anywhere before the priority date of this application.

The numerical values mentioned for the various physical parameters, dimensions or quantities are only approximations and it is envisaged that the values higher/lower than the numerical values assigned to the parameters, dimensions or quantities fall within the scope of the disclosure, unless there is a statement in the specification specific to the contrary.

While considerable emphasis has been placed herein on the components and component parts of the preferred embodiments, it will be appreciated that many embodiments can be made and that many changes can be made in the preferred embodiments without departing from the principles of the disclosure. These and other changes in the preferred embodiment as well as other embodiments of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to

be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

TECHNICAL ADVANTAGES AND ECONOMIC SIGNIFICANCE

- The process of the present disclosure recovers used, deactivated ionic liquid, which can be further reused for different applications.
- The process of the present disclosure saves undue waste of valuable chemicals; thereby reducing the environmental hazards.
- The expenditure on purchasing fresh ionic liquid catalyst for every single application is reduced.
- The process of the present disclosure yields a hydrotalcite material from the anionic part of used ionic liquid.
- The hydrotalcite material may find further applications in areas such as catalysts, catalyst supports, antacids and hydrogen scavengers in different processes such as plastic manufacturing and in ion exchange.

CLAIMS:

1. A process for separating a cationic component and an anionic component from deactivated ionic liquid; said process comprising the following steps:
 - a. dissolving said deactivated ionic liquid in a first solvent to obtain a first solution;
 - b. dissolving magnesium chloride in a second solvent and heating at a temperature ranging from 20 to 80 °C to obtain a second solution;
 - c. dissolving at least one base in said second solvent to obtain a third solution;
 - d. preparing a dispersion by admixing said first solution, said second solution and said third solution, at atmospheric pressure, at a temperature ranging from 40 to 80 °C, at a speed of rotation ranging from 80 to 120 rpm;
 - e. allowing said dispersion to stand for a time period ranging from 6 to 14 hours to obtain a phase separated dispersion;
 - f. cooling said phase separated dispersion to a temperature lower than 40 °C followed by filtering to obtain:
 - i. a residue comprising a precipitate of said anionic component and magnesium chloride; and
 - ii. a biphasic filtrate having an organic phase comprising said first solvent and said deactivating components and an aqueous phase comprising said second solvent and said cationic component;
 - g. washing said residue with said first solvent followed by drying to obtain purified precipitate;
 - h. separating said organic phase from said aqueous phase to obtain separated organic phase and separated aqueous phase;
 - i. distilling said separated organic phase to leave behind said deactivating components;
 - j. distilling said aqueous phase to leave behind a solid residue comprising the cationic component devoid of deactivating components; and
 - k. recrystallizing and purifying said solid residue to obtain the cationic component devoid of deactivating components.

2. The process as claimed in claim 1, wherein said cationic component is selected from the group consisting of 1-butyl-3-methyl imidazolium bromide, 1-butyl-3-methyl imidazolium chloride, 1-butyl-4-methylpyridinium chloride and 1-butyl-4-methylpyridinium bromide.
3. The process as claimed in claim 1, wherein said anionic component is a metal chloride.
4. The process as claimed in claim 3, wherein said anionic component is aluminum chloride.
5. The process as claimed in claim 1, wherein said deactivating agent is at least one from the group consisting of polymers, tar, hydrocarbons and moisture.
6. The process as claimed in claim 1, wherein said first solvent is ethyl acetate.
7. The process as claimed in claim 1, wherein said first solution contains 20 to 80% by volume of said ionic liquid.
8. The process as claimed in claim 1, wherein said second solvent is at least one selected from the group consisting of water and distilled water.
9. The process as claimed in claim 1, wherein said second solution contains 10 to 60% by weight of magnesium chloride.
10. The process as claimed in claim 1, wherein said base is at least one from the group consisting of sodium hydroxide and sodium carbonate.
11. The process as claimed in claim 1, wherein said third solution contains 5 to 35% by weight of sodium carbonate and 10 to 50% by weight of sodium hydroxide.

12. The process as claimed in claim 1, wherein said step of preparing a dispersion is carried out by at least one method from the group consisting of:
- adding said first solution and said second solution into said third solution in a drop-wise fashion; and
 - adding said second solution and said third solution into said first solution in a drop-wise fashion.
13. The process as claimed in claim 1, wherein said precipitate is of Hydrotalcite.
14. The process as claimed in claim 1, wherein said precipitate is of Hydrotalcite represented by the Formula $M_1^{2+}M_2^{3+}(OH)_2A^{n-}yH_2O$, wherein M_1^{2+} is a divalent metal ion and M_2^{3+} is a trivalent metal ion in a ratio ranging from 2: 1 to 3: 1, A^{n-} is an anion where n is the integer from 1 to 3 and y represents the quantity of the interlayer water.
15. The process as claimed in claim 1, wherein said precipitate is of $Mg_6Al_2CO_3(OH)_{16}.4(H_2O)$.
16. The process as claimed in claim 1, wherein said step of recovering and purifying said cationic component is carried out by extracting said cationic component from said solid residue by at least one extracting agent, followed by distilling to obtain the purified cationic component.
17. The process as claimed in claim 16, wherein said extracting agent is dichloromethane.
18. A process for the reconstitution of ionic liquid; said process comprising obtaining the purified cationic component as claimed in claim 1 and reacting with a fresh anionic component to obtain ionic liquid.

INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER
B01D15/36, B01J38/68 Version=2015.01

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B01D15/36, B01J38/68

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

PATSEER, IPO Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 2010 062902 A4, CHEVRON CORP, CHANG BONG KYU, 14 October, 2010 (14-10-2010), cited in the application, Abstract, claims 1-14, Paras 0002, 0007, 0011-0012, 0027, example 1, -----	1-18
Y	US 2004074842 A1, EXXONMOBIL RESEARCH & ENGG CO, 22 April, 2004 (22-04-2004), claims 1, 11-12, 26-32, paras 00011-00014, 00020 -----	1-18
Y	US 8597517 B2, INSTITUTO MEXICANO DEL PETROLEO, 03 December, 2013 (03-12-2013), claims 1, 6-9, Paras 0022-0028 -----	1-18
A	US 2007 142676, CHEVRON CORP, 21 June, 2007 (21-06-2007), Whole document	1-18

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"P" document published prior to the international filing date but later than the priority date claimed	"&" document member of the same patent family

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INTERNATIONAL SEARCH REPORT
Information on patent family members

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WO 2010062902 A4	14-10-2010	KR 20110103979 A	21-09-2011
		IN DELNP-2011-04751 A	27-07-2012
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