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(71) Applicant (for all designated States except US): THE BURWOOD CORPORATION LIMITED [GB/GB]; The Glassmill, 1 Battersea Bridge Road, London SW11 3BG (GB).

(72) Inventors; and
(75) Inventors/Applicants (for US only): WALL, Kevin [GB/GB]; 9 Field Hey Lane, Willaston, South Wirral L64 1TG (GB).
ZARD, Pauline, William [GB/GB]; 70F Onslow Gardens, London SW7 3QB (GB).
BARCLAY-MILLER, David, James [GB/GB]; 216 Butlers Wharf Buildings, 36 Shad Thames, London SE1 2YR (GB).
MARTIN, David, William [GB/GB]; 64 Ford Lane, Litherland, Liverpool, Merseyside L21 0HR (GB).


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(57) Abstract

A compound is described. The compound has the formula:

wherein each of w, x, y and z independently has an average value of from 0 to 6; each of n, u and v independently has an average value of from 0 to 1; w+v+x+(1+n)+z+(1+n) ≥ 2; R1, R2, R3, R4 and R5 independently are organic groups. The compound is suitable for use as a biodegradable oil phase in the drilling of well bores.
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AMIDE AND IMIDE COMPOUNDS AND THEIR USE AS LUBRICANT OILS

The present invention relates to a compound. In particular the present invention relates to a polyamide or polyimide.

More in particular the present invention relates to the application of a polyamide or a polyimide compound for use as a synthetic lubricant oil in applications such as oil–based drilling muds and machine cutting fluids, especially where resistance to hydrolytic breakdown at high temperature is required.

Current technology for ecologically–friendly synthetic oils for well–bore drilling emulsions uses esters or polyesters. There are problems associated with the known oils. For example, the esters are subject to hydrolysis at high temperature. Breakdown of the esters results not only in a loss of the expensive oil, but the alcohols are water soluble and can cause upset in the osmotic pressure balance of the aqueous phase.

The present invention seeks to overcome some or all of those problems.

According to a first aspect of the present invention there is provided the use of an end–blocked polyamide or an end–blocked polyimide in or as a lubricant oil, especially a lubricant oil for use in well–bore drilling fluids.

According to a second aspect of the present invention there is provided a well–bore drilling process comprising use of an end–blocked polyamide or an end–blocked polyimide in or as a well–bore drilling fluid.

According to a third aspect of the present invention there is provided a well–bore drilling fluid comprising an end–blocked polyamide or an end–blocked polyimide.

According to a fourth aspect of the present invention there is provided a compound of the formula:
\[ R^1[CO]_n[NHR^2]_m[NHCONR^3][CO]_n[NHR^4]_m[NHCONR^5][CO]_n[NH]_2CO\cdot R^3 \]

wherein: each of \( w, x, y \) and \( z \) independently has an average value of from 0 to 6;
each of \( n, u \) and \( v \) independently has an average value of from 0 to 1;
\( u+v+x,(1+n)+z,(1+n)\geq 2; \) and \( R^1, R^2, R^3, R^4 \) and \( R^5 \) independently are organic groups.

In the compound of the present invention each of \( w, x, y, z, n, u, v, R^1, R^2, R^3, R^4 \) and
\( R^5 \) need not be the same. For example, each \( R^3 \) can be different.

The present invention also covers compositions comprising the compound of the present invention.

A key advantage of the compound of the present invention is that it is biodegradable
and nontoxic, allowing its use in environmentally sensitive areas in such applications
such as outboard engine and chain saw lubricants.

We have found that replacing ester linkages with amide or imide linkages greatly
increases the hydrolytic stability of the oil. In this regard, incorporating multiple
amide or imide linkages allows control of the water solubility, metal surface wetting
ability, oil viscosity and flash point.

Preferably, in the use, process or fluid according to the present invention the
polyamide and/or the polyimide is obtainable from reacting a first entity having two
reactive functional groups, which may be the same or different, with two second
entities, which may be the same or different, wherein the second entities have
functional groups which may be the same or different and which are reactive with the
reactive functional groups of the first entity to link the entities by amide or imide
bonds and wherein the second entities provide the end-blocked groups of the resultant
end-blocked polyamide or end-blocked polyimide. In this aspect of the present
invention, each entity can possess other functional groups but those other functional
groups do not result in unwanted cross-linking reactions and products therefrom.
Preferably in the use, process or fluid the polyamide and/or the polyimide is a compound according to the fourth aspect of the present invention or a composition comprising the compound according to the fourth aspect of the present invention.

Preferably the compound has an average molecular weight below 5,000 and preferably less than 500.

Preferably \( R^1 \) and \( R^5 \) are independently selected from \( C_{4-24} \) alkyl, alkenyl, cycloalkyl, cycloalkenyl or aromatic groups.

Preferably \( R^1 \) and \( R^5 \) are independently selected from \( C_{4-24} \) alkyl, alkenyl, cycloalkyl or cycloalkenyl groups.

Preferably \( R^2 \) and \( R^4 \) are \( C_{1-12} \) alkyl, alkenyl, cycloalkyl, cycloalkenyl or aromatic groups.

Preferably \( R^2 \) and \( R^4 \) are \( C_{1-12} \) alkyl, alkenyl, cycloalkyl, or cycloalkenyl groups.

Preferably \( R^3 \) is a \( C_{1-12} \) alkyl, alkenyl, cycloalkyl, cycloalkenyl or an aromatic group.

In the present invention, \( R^1 \) and \( R^5 \) may be the same or different and may contain side groups which are unreactive in the production of the compound or composition; \( R^2 \) and \( R^4 \) may be the same or different and may contain side groups which are unreactive in the production of the compound or composition; and \( R^3 \) may contain side groups which are unreactive in the production of the compound or composition.

In addition, the polyamide or polyimide can contain a small number of other linkages, such as ester linkages. Preferably the polyamide or polyimide contains at most two other linkages, more preferably at most one other linkage. In a highly preferred embodiment, the polyamide or polyimide only contains amide and/or imide linkages.
In a preferred embodiment, the compound of the present invention may be the product of the reaction of a total of two moles of aliphatic amine, selected from one or more of: t-butyl amine, cyclohexyl amine, 2-ethylhexyl amine or dodecyl amine, with one mole of the reactive derivative of one or more aliphatic diacids selected from succinic acid, glutaric acid or adipic acid.

In an alternative preferred embodiment, the compound of the present invention may be the reaction product of one mole of aliphatic diamine, selected from one or more of: 1,2-diaminocyclohexane, 1,3-diamino-2,2-dimethyl-propane or triethylene tetramine, with two moles of the reactive derivative of one or more of aliphatic acids, selected from: octanoic acid, 2-ethylhexanoic acid, or oleic acid.

The compound of the present invention may be blended with other additives or synthetic oils, or even other compounds according to the present invention. This blending enables one to tailor the properties of the resultant compound or composition such that, for example, the finished blended oil has desired physical and chemical characteristics.

Varying the molecular weight and structure of the various reactants allows the production of oils with specific properties. For example, increasing the content of branched alkyl or aromatic components in the compound or composition increases the flash point, increases the hydrolytic stability and lowers the viscosity, but reduces the biodegradability. A predominance of straight-chain alkyl or alkenyl compounds results in high biodegradability at the expense of reduced fluidity at low temperatures. Longer chain lengths exhibit poorer low temperature viscosity and biodegradability but have higher flash points and somewhat higher hydrolytic stability. Increasing the proportionate mass of nitrogen in the compound or composition increases the solubility or dispersability in water and increases the metal surface wetting properties of the oil. Blending together compounds or compositions with different properties allows the formation of an oil with the desired characteristics. The compound or composition may also be blended with other base-oil types (such as esters for example) to obtain benefits in cost or performance for particular applications.
For well-bore drilling the desired characteristics are high flash point (>60 °C), low viscosity (less than 10cP at 40°C and pumpable at -5 °C), high biodegradability (>80%), low water solubility, good hydrolytic stability. For chain saw blade lubricants the oil should have a high flash point, high biodegradability and show good metal-wetting properties. In this case properties like viscosity, water solubility and hydrolytic stability are of lesser importance.

For most applications flash point is important as a measure of the fire risk. In general, products with flash points greater than 60 °C are considered to be low flammability.

In drilling high temperature, high pressure well-bores, the temperature in the well can reach 200 °C. For this application the oil must demonstrate little or no degradation when held at high temperature whilst in intimate contact with a calcium chloride brine. Any breakdown of the compound or composition is characterised by an increase in the free acid in the fluid. This can be determined by titration with a standard alkali.

Aside from use in or as well-bore drilling fluids, the compound of the present invention may be used for chain lubricants, particularly for chain saws. Also, the compound may be used as internal combustion engine lubricants. Also the compound may be used as a component of a metal-working coolant/lubricant fluids. In addition, the compound may be used as a component of a hydraulic fluid.

In a highly preferred embodiment the present invention is a synthetic oil based on low molecular weight polyamides or polyimides which are terminated by monofunctional groups.

The polyamide or polyimide according to the present invention may be obtained by reacting one or more aliphatic, cycloaliphatic or aromatic amine compounds, each having one or two reactive terminal sites, with one or more aliphatic, cycloaliphatic or aromatic acid derivatives, each having one or two reactive carbonyl groups, to form terminally-blocked polyamides or polyimides.
However, it is to be understood that other processes may be used to prepare the
compound or compositions of the present invention. Moreover, the aliphatic,
cycloaliphatic or aromatic amine compounds, each having one or two reactive terminal
sites, and/or the aliphatic, cycloaliphatic or aromatic acid derivatives, each having one
or two reactive carbonyl groups, may have other functional groups that do not hinder
the formation of the preparation of the polyamides or polyimides of the present
invention.

For example, it would be possible to prepare the compound or composition of the
present invention using an aliphatic, cycloaliphatic or aromatic amine compounds,
each having one or two reactive terminal sites, and wherein a third terminal site is
present which is "temporarily blocked" in the sense that it is non-reactive in the
reaction process of the one or more aliphatic, cycloaliphatic or aromatic amine
compounds, each having one or two reactive terminal sites, with the one or more
aliphatic, cycloaliphatic or aromatic acid derivatives, each having one or two reactive
carbonyl groups, to form the end-blocked polyamides or polyimides. The temporarily
blocked site may then be un-blocked and allowed to react, if desired, with a further
reactant.

It is also to be recognised that different starting materials may be used to obtain the
compound or compositions of the present this invention (e.g. amides, or diamides with
hydrocarbyl halides or dihalides). In a preferred compound or composition, from 1
to 7 groups derived from diacids are linked by up to 6 diamine groups and two
monoamines occupy the terminal positions. In the alternative preferred compound or
composition, between 1 and 7 diamine groups are linked by up to 6 diacid groups and
the structure terminated by two monofunctional acids. The diamines may in fact be
polyamines with up to 6 polyalkylene repeating units. Viscosity requirements limit
the preferred maximum molecular weight of the compound used in the invention to
approximately 5,000 and more preferably less than 500.
A preferred compound has terminal alkyl or alkenyl groups preferably derived from, for example, one or more of t-butyl amine, cyclohexyl amine, 2-ethylhexyl amine or dodeceny1 amine and the terminal groups are linked by an entity having two reactive functional groups which may be the same or different. In this regard, the entity can possess other functional groups but those other functional groups do not result in unwanted cross-linking reactions and products therefrom. For example, the entity can be or derived from one or more of glutaric acid, succinic acid or adipic acid, giving a N,N'-dialkyl diamide product – for example N,N'-di-t-butyl adipamide. In an alternative preferred compound, the terminal groups are carbonyl derived from, for example, one or more of octanoic acid, ethylhexanoic or octadecanoic acids and the linking group is a disubstituted compound such as methylene diamine, 1,2-ethylene diamine, tetraethylene pentamine, 1,2-cyclohexyl diamine or neopentyl diamine, giving an alkylene bisamide – for example: 1,2-ethylene bis(octanoamide).

A preferred compound of the present invention is therefore a polyamide with the following general structure:

\[ R^1[CO]\_n[NHR^3]\_w[[NHCOR^4[CO]\_n][NHR^5]_z][NHCO\_n][NHCO\_n]_yNH[CO]_zR^5 \]

where w, x, y and z have values that average between 0 and 6, with the restriction that \( u+v+x+(1+n)+z(1+n) \geq 2 \), where n, u and v are independently equal to 0 or 1. \( R^1 \) and \( R^5 \) are independently C\(_{1-24}\) alkyl, alkenyl, cycloalkyl or cycloalkenyl or, less preferably, aromatic groups. \( R^1 \) and \( R^5 \) may be the same or different and may contain side groups which are unreactive in the production of the compound or composition. Preferred groups are t-butyl, cyclohexyl, 2-ethylhexyl, when the adjacent carbonyl group is absent (u or v = 0). When the carbonyl group is present (u or v =1), the preferred groups are C(CH\(_3\)_2, CH(C\(_2\)H\(_2\))CH\(_2\))CH\(_3\) and (CH\(_2\)_2CH=CH(CH\(_2\))_2CH\(_3\). Preferably \( R^2 \) and \( R^4 \) are C\(_1-12\) alkyl, alkenyl, cycloalkyl or cycloalkenyl or, less preferably, aromatic groups. \( R^2 \) and \( R^4 \) may be the same or different and may contain side groups which are unreactive in the production of the compound or composition. Preferred groups are CH\(_2\)CH\(_2\), CHCH\(_2\)CH\(_2\) and (CH\(_2\)_5. \( R^3 \) is a C\(_1-12\) alkyl, alkenyl, cycloalkyl or cycloalkenyl or, less preferably, aromatic group and may contain side
groups which are unreactive in the production of the compound or composition. Preferably n=1 when R$_3$ is derived from a diacid and n=0 when R$_3$ is derived from an amino acid or lactam. Preferred R$_3$ groups are (CH$_2$)$_2$, (CH$_2$)$_3$, (CH$_2$)$_4$ and (CH$_2$)$_6$.

In the preferred embodiment of the invention, R$^1$ and R$^2$ are either t-butyl, cyclohexyl, 2-ethylhexyl or dodeceny1 or a mixture of these groups, u and v are both 0. Preferably R$^3$ is a C$_7$-C$_9$ alkyl group from one or more diacids such as succinic, glutaric or adipic acids, z=1. n=1, w and x are zero. As an alternative, R$^1$ and R$^2$ are C$_{7-17}$ alkyl or alkenyl groups derived from octanoic, ethylhexanoic or octadecenoic acids (u and v are both 1) and R$^2$ is an alkyl group derived from a diamine compound such as methylene diamine, 1,2-ethylene diamine, hexamethylene diamine, tetraethylene pentamine, 1,2-cyclohexyl diamine or neopentyl diamine, w=1, x and z=0.

As mentioned above, the compound of the present invention is particularly suitable for use as a biodegradable oil phase in the drilling of well bores.

The present invention will now be described only by way of example.

In the examples given below the oil compounds or compositions were subjected to a number of tests to demonstrate their suitability for particular applications. In general, all oils must show good materials compatibility and in particular the oils must not cause shrinkage or swelling of the rubber seals used in the connections between the various parts of the oil-containing equipment. The most common rubber for oil seals is the fluoroelastomer Viton™ and it is preferable to show compatibility with at least this material.
EXAMPLE 1

One mole of adipic acid (usually in the form of the acid dibromide or dimethyl ester) is reacted with two moles of dodecenyl amine to form N,N' didodecenyl adipamide. After purification, the resulting liquid compound is found to be insoluble in water, having a boiling point of 250 °C, a density of 0.97 g/ml and a viscosity of 20 cP at 40 °C. The flash point was found to exceed 110 °C. To test for biodegradability, a 10 ml sample of the polyamide oil was agitated in 1 litre of seawater over a period of 28 days. At the end of this period, the liquids were allowed to separate and the volume of the upper oil phase measured and found to be 1.2 ml, indicating that 88% of the original oil had biodegraded. To test resistance to hydrolysis, 80 ml of the oil was placed in a rolling oven sample bomb and 120 ml of 20% w/w calcium chloride added. The bomb was sealed and pressurised with 100 psi nitrogen and then rolled for 16 hours at 200 °C. After this period, the sample was cooled and 25 ml of the aqueous layer extracted and neutralised using 2 ml of 0.1N NaOH. A 10 g sample of Viton rubber was covered with the polyamide oil and left for 28 days. After this period the rubber was wiped clean and dry using a paper tissue and on weighing showed no loss or gain.

EXAMPLE 2

One mole of neopentyl diamine is reacted with two moles of oleic acid (for example as the acid halide or methyl ester) to form neopentyl N,N' bis(oleamide). After purification, the water-insoluble liquid product has a boiling point of 225 °C, density of 0.97 g/ml and a viscosity of 15 cP at 40 °C. The oil was found to have a flash point of 105 °C. Using the method given in example 1, the biodegradability was found to be 80%. In the hydrolysis test, 1.5 ml of 0.1N NaOH was required for neutralisation. Viton rubber showed no loss or gain in weight after 28 days in the oil.
EXAMPLE 3

For comparison a sample of Finagreen BDMF™, a commercially available ester–based synthetic oil currently used in well–bore drilling, was subjected to the same tests as those in examples 1 and 2. The viscosity of the oil is 5 cP at 40 °C. The flash point was found to exceed 110 °C. The biodegradability was found to be 85% – that is to say little different from the present invention. In the hydrolysis test, 5 ml of 0.1 N NaOH was required for neutralisation, indicating significantly a higher rate of hydrolysis. As in the previous examples there was no weight change of Viton rubber after immersion in the oil for 28 days.

EXAMPLE 4

One mole of pentaethylene hexamine is reacted with two moles of ethyl hexanoic acid, or its more reactive derivatives (usually the acid halide or methyl ester), to yield a product, which after purification, is found to have a water solubility of 0.05 g/l, density of 0.96 g/ml, a boiling point of 180 °C and a viscosity of 5 cP at 40 °C. The flash point of the product was 85 °C. No swelling of Viton was observed after 28 days immersion.

EXAMPLE 5

One mole of 1,2 diaminocyclohexane is reacted with two moles of s–butyric acid (as its acid halide or methyl ester) to yield a liquid, which after purification, has a boiling point of 150 °C, a density 0.95 g/ml, and a viscosity of 1 cP at 40 °C. The flash point of the product was 65 °C. No swelling of Viton was observed after 28 days immersion.
EXAMPLE 6

One mole of triethylene tetramine is reacted with two moles of adipic acid (usually in the form of the acid dihalide or dimethyl ester) and then two moles of cyclohexylamine to yield a liquid, which after purification, has a boiling point of 170 °C, density 0.97 g/ml and a viscosity of 7 cP at 40 °C. The flashpoint of the oil was 69 °C. No swelling of Viton was observed after 28 days immersion.

EXAMPLE 7

One mole of 1,6 hexamethylene diamine is reacted with two moles of a reactive 2-ethyl hexanoic acid derivative to yield, after purification, a liquid with a boiling point of 240 °C, a density of 0.92 g/ml and a viscosity of 3.5 cP at 40 °C. The flashpoint of the oil was >110 °C and no swelling of Viton was observed after 28 days immersion.

EXAMPLE 8

Equal masses of the product from examples 1 and 5 were mixed. The resulting oil had a flash point of 75 °C, a density of 0.96 g/ml and a viscosity of 6.2 cP at 40 °C.

Other modifications of the present invention will be apparent to those skilled in the art.
CLAIMS

1. A compound of the formula:

\[ R^1[CO]_w[NHR^2]_x[[NHCOR^3[CO]_y][NHR^4]_z][NHCOR^5[CO]_n]dNH[CO]_sR^5 \]

wherein:

each of \( w, x, y \) and \( z \) independently has an average value of from 0 to 6;

each of \( n, u \) and \( v \) independently has an average value of from 0 to 1;

\( u+v+x.(1+n)+z.(1+n) \geq 2 \); and

\( R^1, R^2, R^3, R^4 \) and \( R^5 \) independently are organic groups.

2. A compound according to claim 1 wherein the compound has an average molecular weight below 5,000 and preferably less than 500.

3. A compound according to claim 1 or claim 2 wherein \( R^1 \) and \( R^2 \) are independently selected from \( C_{4-24} \) alkyl, alkenyl, cycloalkyl, cycloalkenyl or aromatic groups.

4. A compound according to claim 3 wherein \( R^1 \) and \( R^2 \) are independently selected from \( C_{4-24} \) alkyl, alkenyl, cycloalkyl or cycloalkenyl groups.

5. A compound according to any one of the preceding claims wherein \( R^2 \) and \( R^4 \) are \( C_{1-12} \) alkyl, alkenyl, cycloalkyl, cycloalkenyl or aromatic groups.
6. A compound according to claim 5 wherein R² and R⁴ are C₁₋₁₂ alkyl, alkenyl, cycloalkyl, or cycloalkenyl groups.

7. A compound according to any one of the preceding claims wherein R² is a C₁₋₁₂ alkyl, alkenyl, cycloalkyl, cycloalkenyl or an aromatic group.

8. Use of an end-blocked polyamide or an end-blocked polyimide in or as a lubricant oil, especially a lubricant oil for use in well-bore drilling fluids.

9. A well-bore drilling process comprising use of an end-blocked polyamide or an end-blocked polyimide in or as a well-bore drilling fluid.

10. A well-bore drilling fluid comprising an end-blocked polyamide or an end-blocked polyimide.

11. The invention according to any one of claims 8 to 10 wherein the polyamide and/or the polyimide is a compound as defined in any one of claims 1 to 7.

12. A compound, use, process or fluid substantially as hereinbefore described.
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C07C233/05 C07C233/36 C07C233/38 C07C233/41 C09K7/06 C10M105/68

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)
IPC 6 C07C C09K C10M

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)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
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<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
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<td>WO,A,92 12960 (PINAROO) 6 August 1992 see page 3, paragraph A</td>
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<td>see page 4, paragraph G</td>
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<td>EP,A,0 438 849 (ETHYL PETROLEUM ADDITIVES) 31 July 1991 see the whole document</td>
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Patent family members are listed in annex.

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Date of the actual completion of the international search: 19 September 1995
Date of mailing of the international search report: 29.09.95

Name and mailing address of the ISA
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Leroy, A

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