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TS 7037

DISPERSANT ADDITIVES

The present invention relates to reaction products of polyalkenes, a process for their preparation, lubricating oil compositions, fuel compositions and additive concentrates containing them and their use as dispersant additives.

It has surprisingly been found that certain nitrogen-containing reaction products of polyalkenes have low haze and good dispersancy properties and exhibit good compatibility towards seals and bearings.

In accordance with the present invention, there is provided the reaction product of:

(i) an oligomer having a number average molecular weight of no more than 7500 and being formed from one monomer or two or more different monomers, the or each monomer being an ethylenically unsaturated compound and said oligomer or each of said monomers being optionally partially or fully esterified, each of which monomers may not contain an ether moiety nor be a

cyclopentadienyl group, said oligomer optionally being reacted with a low molecular weight amine, with

(ii) a polyalkenyl derivative of an ethylenically unsaturated carboxylic reagent (iii), which derivative has a number average molecular weight of at least 1600 when the oligomer is a co-oligomer of two monomers one of which is a straight chain olefin, and

(iv) a polyamine containing at least two -NH_2 and/or -NH groups, or with

(v) the pre-formed product of reagents (ii) and (iv),

wherein at least one of the ethylenically unsaturated compounds of the oligomer has a functional group which can react with an amine, the oligomeric reaction

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product optionally being further reacted with a low molecular weight amine.

5 The oligomer (i) preferably has a number average molecular weight of no more than 5000, more preferably no more than 4000.

The low molecular weight amine preferably is one having a molecular weight in the range 17 to 300, such as oleylamine or benzylamine, aniline being particularly preferred; or a polyamine, suitably as defined in (iv) above.

10 The ethylenically unsaturated carboxylic reagent (iii) contains a total of at least 3 carbon atoms, preferably a total of from 3 to 50, more preferably from 3 to 30, still more preferably from 4 to 20, and even more preferably from 4 to 10, carbon atoms.

The ethylenically unsaturated carboxylic reagent may be an alpha-beta olefinic unsaturated carboxylic reagent as described at page 6, lines 15 to 48 of EP-B-0285609 or page 6, lines 11 to 39 of EP-B-0287569, e.g. acrylic acid (C₃), methacrylic acid (C₄), cinnamic acid (C₉), crotonic acid (C₄), 2-phenylpropenoic acid (C₉), maleic acid (C₄), fumaric acid (C₄), glutaconic acid (C₅), mesaconic acid (C₅), itaconic acid (methylene succinic acid) (C₅), citraconic acid (methyl maleic acid) (C₅) and functional derivatives thereof such as anhydrides (e.g. maleic anhydride (C₄), glutaconic anhydride (C₅), itaconic anhydride (C₅), citraconic anhydride (C₅)), esters (e.g. methyl acrylate (C₄)), amides, imides, salts, acyl halides and nitriles.

25 Preferably the ethylenically unsaturated carboxylic reagent (iii) is selected from monoethylenically unsaturated C₄-C₁₀ dicarboxylic acids and anhydrides, of which maleic anhydride is most preferred.

35 The polyalkenyl derivative (ii) of an ethylenically unsaturated carboxylic reagent (iii) may be prepared by

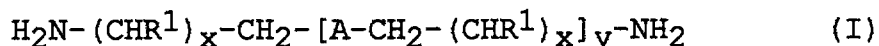
methods known in the art. For example, if the ethylenically unsaturated carboxylic reagent (iii) is maleic anhydride, the polyalkenyl derivative thereof may conveniently be prepared by mixing a polyalkene with a specified amount of maleic anhydride and passing chlorine through the mixture, e.g. as described in GB-A-949981. Alternatively, the derivative may be prepared by reacting thermally, at an appropriate temperature, the polyalkene with a specified amount of maleic anhydride, e.g. as described in GB-A-1483729. A particularly preferred process for preparing such a derivative, which is described in EP-A-0542380, involves reacting the polyalkene with maleic anhydride in a mol ratio of maleic anhydride to polyalkene of greater than 1:1, at a temperature in the range from 150 to 260°C and in the presence of a polyaddition-inhibiting amount of a sulphonic acid. The molar ratio of ethylenically unsaturated carboxylic moiety to polyalkenyl moiety in the derivative formed is preferably 1:1 to 5:1, more preferably 1:1 to 1.3:1, particularly 1:1 to 1.2:1.

The polyalkene used to prepare the polyalkene derivative may be a homopolymer or copolymer, for example of at least one C₂₋₁₀ monoolefin. Preferably the polyalkene is a polymer of at least one C₂₋₅ monoolefin, e.g. an ethylene-propylene copolymer. The monoolefin is preferably a C₃₋₄ olefin, in particular propylene or isobutylene, and preferred polyalkenes derived therefrom include polyisobutylenes and atactic or isotactic or syndiotactic propylene oligomers. Polyisobutylenes such as that sold by BASF under the trade mark "GLISSOPAL" and those sold by the British Petroleum Company under the trade marks "ULTRAVIS", "HYVIS" and "NAPVIS", e.g. "HYVIS 75", "HYVIS 120", "HYVIS 200" and "NAPVIS 120" polyisobutylenes, are especially preferred for use in the present invention.

The polyalkene has a number average molecular weight (M_n) preferably in the range from 300 to 7000, more preferably from 500 to 5000, still more preferably from 700 to 3000.

5 The polyamine (iv) contains at least two $-NH_2$ and/or $-NH$ groups, the groups each having at least one active hydrogen thereon. Examples of polyamines useful in the present invention are those described in the text from page 16, line 21 to page 19, line 53 of
10 EP-B-0287569.

Preferably the polyamine is a compound of the general formula



wherein A is $-NH$ or $-O-$, each R^1 independently
15 represents a hydrogen atom or a methyl group, x is in the range 1 to 3, and y is in the range 1 to 10 when A is $-NH$ or y is in the range 1 to 200 when A is $-O-$.

In formula (I) above, it is preferred that when A is $-NH$, then x is 1, each R^1 represents a hydrogen
20 atom, and y is in the range 1 to 8; or when A is $-O-$, then x is 1, each R^1 represents a methyl group and y is in the range 1 to 50.

Reagent (v) is the pre-formed product of reagents (ii) and (iv) and may be prepared according to
25 techniques conventional in the art. Thus, for example, if the reagent (ii) is a polyalkenyl derivative of maleic anhydride and the polyamine is ethylene polyamine, they may conveniently be reacted together in a molar ratio of polyalkenyl derivative to polyamine
30 from 1 - 4:1, in a hydrocarbon solvent at a temperature in the range from 100 to 250°C, e.g. as described in EP-A-0587250. The molar coupling ratio of polyalkenyl derivative to amine in the product is preferably in the range 1:1 to 3:1, more preferably 1:1 to 2.5:1.

35 The ethylenically unsaturated compounds may be selected from any compounds that can be oligomerised,

for example alpha-olefins and alpha-beta-unsaturated carbonyl compounds. Particularly suitable compounds include styrene, maleic anhydride and acrylic acid.

5 The reaction between said ethylenically unsaturated monomers to form the oligomer may be carried out by radical cationic or anionic oligomerisation. Said oligomers are selected from those having a functional group that will react with an amine, more preferably styrene copolymerised with maleic anhydride, styrene
10 copolymerised with acrylic acid, polyacrylic acid, and alpha-olefins copolymerised with maleic anhydride.

Reaction products of the present invention, whether produced from oligomers (i) in which either the oligomer or one or more of the monomers thereof has
15 been partially or fully esterified, or from oligomers (i) in respect of which there has been no such esterification, exhibit good dispersancy properties. It has been found that many of the reaction products (especially esters) exhibit low haze levels, thus
20 reducing the need for further treatment such as filtration. Such esterification of these oligomers or their monomers may be carried out by reaction with an alcohol, e.g. C₁₋₂₀ aliphatic or aromatic alcohols, preferably in the presence of a catalyst such as para-
25 toluenesulphonic acid (PTSA).

However, there are commercially available (1) co-oligomers of such ethylenically unsaturated compounds, and (2) partially-esterified forms of co-oligomers (1). Examples of (1) are styrene/maleic anhydride co-
30 oligomers (SMA), such as those sold by Elf-Atochem under the trade names "SMA 1000A", "SMA 2000A" and "SMA 3000A". An example of (2) is a half-ester of said "SMA 3000A" with iso-octyl alcohol, sold by Elf-Atochem under the trade name "SMA 3840A". It has been found
35 that by use of partially- or fully-esterified forms of such commercially available SMAs, such as commercially

available partially-esterified forms of the SMAs, or by use of esterified polyacrylic acid, for example, poly n-amyI acrylate, dispersants can be produced having low haze levels (as measured according to ASTM D1003).

5 The esterification of the SMAs to produce fully esterified oligomers, i.e. diesters, is preferably carried out using C₁₋₂₀ aliphatic or aromatic alcohols, more preferably C₅₋₈ branched aliphatic alcohols, in particular iso-amyI alcohol (which is a mixture of
10 branched alcohols, mainly 3-methylbutan-1-ol and 2-methylbutan-1-ol) or iso-octyl alcohol, more particularly in excess. The esterification of polyacrylic acid is preferably carried out using C₁₋₂₀ aliphatic or aromatic alcohols, for example straight
15 chain aliphatic alcohols, in particular n-amyI alcohol. Said esterification processes are preferably carried out at a temperature in the range 0 to 250°C, more preferably 70 to 190°C, at a pressure in the range 0.5 to 10 atmospheres (50 to 1000 kPa), more preferably
20 atmospheric pressure, in the presence of a solvent, more preferably selected from alcohols, aromatic hydrocarbons such as toluene, xylene and mesitylene, and ethers such as tetrahydrofuran and 1,4-dioxane, and mixtures thereof, and in the presence of a catalyst,
25 more preferably selected from acids such as sulphuric acid and PTSA, Lewis acids such as aluminium chloride, and bases such as pyridine and 4-(N,N-dimethylamino)-pyridine. Where the solvent is reactive, e.g. an alcohol such as iso-octyl alcohol, reaction with the
30 oligomer (i) may occur concomitantly with reaction with the polyamine (iv) or with (v).

The present invention further provides a process for the preparation of a reaction product as defined above which comprises reacting

35 (i) an oligomer having a number average molecular weight of no more than 7500 and being formed from one

monomer or two or more different monomers, the or each monomer being an ethylenically unsaturated compound and said oligomer or each of said monomers being optionally partially or fully esterified, each of which monomers
5 may not contain an ether moiety nor be a cyclopentadienyl group, said oligomer optionally being reacted with a low molecular weight amine, with

(ii) a polyalkenyl derivative of an ethylenically unsaturated carboxylic reagent (iii), which derivative
10 has a number average molecular weight of at least 1600 when the oligomer is a co-oligomer of two monomers one of which is a straight chain olefin, and

(iv) a polyamine containing at least two $-NH_2$ and/or $-NH$ groups, or with

15 (v) the pre-formed product of reagents (ii) and (iv),

wherein at least one of the ethylenically unsaturated compounds of the oligomer has a functional group which can react with an amine, the oligomeric reaction
20 product optionally being further reacted with a low molecular weight amine.

The reaction between (i), (ii) and (iv) or between (i) and (v) is preferably conveniently carried out in the presence of a suitable solvent at elevated
25 temperature (i.e. above ambient temperature ($20^\circ C$)), e.g. in the temperature range 25 to $200^\circ C$, more particularly 160 to $200^\circ C$, often under reflux conditions and, where necessary, at elevated pressure, e.g. in the range from 2 to 100×10^5 Pa. Examples of solvents
30 include hydrocarbon solvents such as hexane, cyclohexane, toluene, xylene, mesitylene; also synthetic and mineral oils such as "HVI-60"; ether solvents such as tetrahydrofuran and 1,4-dioxane; nitriles such as acetonitrile; alcohols such as 1-
35 pentanol (amyl alcohol) and 2-methyl-2-propanol (tert-butyl alcohol); and chlorohydrocarbons such as

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1,1,1-trichloroethane. The process may be carried out in the absence of a solvent but, as indicated above, is conveniently carried out in the presence of one. Any water or excess of alcohol may be removed using for
5 example a Dean and Stark trap.

The weight ratio (ii):(iv) in the process of the present invention is preferably in the range from 100:1 to 5:1, more preferably from 33:1 to 13:1, and the weight ratio (iv):(i) is preferably in the range from
10 1:20 to 20:1, more preferably from 1:5 to 5:1.

The reaction product of the present invention may be used as a dispersant additive in lubricating oils. Accordingly, the present invention provides a lubricating oil composition comprising a major amount
15 (more than 50%w) of a lubricating oil and a minor amount (less than 50%w), preferably from 0.1 to 20%w, especially from 0.5 to 10%w (active matter), of a reaction product according to the present invention, the percentages by weight being based on the total
20 weight of the composition.

Suitable lubricating oils are natural, mineral or synthetic lubricating oils.

Natural lubricating oils include animal and vegetable oils, such as castor oil. Mineral oils
25 comprise the lubricating oil fractions derived from crude oils, coal or shale, which fractions may have been subjected to certain treatments such as clay-acid, solvent or hydrogenation treatments. Synthetic lubricating oils include synthetic polymers of
30 hydrocarbons, modified alkylene oxide polymers, and ester lubricants, which are known in the art. These lubricating oils are preferably crankcase lubricating oils for spark-ignition and compression-ignition engines, but include also hydraulic lubricants, metal-
35 working fluids and automatic transmission fluids.

Preferably the lubricating base oil component of the compositions according to the present invention is a mineral lubricating oil or a mixture of mineral lubricating oils, such as those sold by member
5 companies of the Royal Dutch/Shell Group under the designations "HVI", or the synthetic hydrocarbon base oils sold by member companies of the Royal Dutch/Shell Group of Companies under the designation "XHVI" (trade mark).

10 The viscosity of the lubricating base oils present in the compositions according to the present invention may vary within wide ranges, and is generally from 3 to 35 mm²/s at 100°C.

The lubricating oil compositions according to the
15 present invention may contain various other additives, known in the art, such as viscosity index improvers, e.g. linear or star-shaped polymers of a diene such as isoprene or butadiene, or a copolymer of such a diene with optionally substituted styrene. These copolymers
20 are suitably block copolymers and are preferably hydrogenated to such an extent as to saturate most of the olefinic unsaturation. Other suitable additives include dispersant V.I. improvers such as those based on block copolymers, or polymethacrylates, extreme
25 pressure/anti-wear additives such as zinc or sodium dithiophosphates, ashless dispersants such as polyolefin-substituted succinimides, e.g. those described in GB-A-2231873, anti-oxidants, anti-rust additives, friction modifiers or metal-containing
30 detergents such as phenates, sulphonates, alkylsalicylates or naphthenates, all of which detergents may be overbased.

The reaction product of the present invention may also be used as a dispersant additive in fuels.

35 Accordingly, the present invention further provides a fuel composition comprising a major amount (more than

50%w) of a fuel and a minor amount (less than 50%w), preferably from 0.001 to 2%w, more preferably from 0.001 to 0.5%w and especially from 0.002 to 0.2%w (active matter), of a reaction product according to the invention, the percentages by weight being based on the total weight of the composition.

Suitable fuels include gasoline and diesel fuel. These base fuels may comprise mixtures of saturated, olefinic and aromatic hydrocarbons. They can be derived from straight-run gasoline, synthetically produced aromatic hydrocarbon mixtures, thermally catalytically cracked hydrocarbon feedstocks, hydrocracked petroleum fractions or catalytically reformed hydrocarbons.

The fuel compositions according to the present invention may contain various other additives known in the art such as a lead compound as anti-knock additive; antiknock additives other than lead compounds such as methyl cyclopentadienyl-manganese tricarbonyl or ortho-azidophenyl; co-antiknock additives such as benzoylacetone; dehazers (e.g. ethoxylated glycerols such as that commercially available as "SURDYNE" (trade mark) M155 (ex Shell Chemicals, UK) or alkoxylated phenol formaldehyde polymers such as those commercially available as "NALCO" (trade mark) 7D07 (ex Nalco), "TOLAD" (trade mark) 2683 (ex Petrolite) or "SURDYNE" (trade mark) D265, M153, M154 or M156 (ex Shell Chemicals, UK)); anti-foaming agents (e.g. the polyether-modified polysiloxanes commercially available as "TEGOPREN" (trade mark) 5851, Q 25907 (ex Dow Corning) or "RHODORSIL" (trade mark) ex Rhone Poulenc)); ignition improvers (e.g. 2-ethylhexyl nitrate, cyclohexyl nitrate, di-tertiary-butyl peroxide and those disclosed in US-A-4208190 at Column 2, line 27 to Column 3, line 21); anti-rust agents (e.g. that commercially sold by Rhein Chemie, Mannheim, Germany as

"RC 4801", or polyhydric alcohol esters of a succinic acid derivative, the succinic acid derivative having on at least one of its alpha carbon atoms an unsubstituted or substituted aliphatic hydrocarbon group containing
5 from 20 to 500 carbon atoms (e.g. the pentaerythritol diester of polyisobutylene-substituted succinic acid); reodorants; anti-wear additives; anti-oxidants (e.g. phenolics such as 2,6-di-tert-butylphenol, or phenylenediamines such as N,N'-di-sec-butyl-p-
10 phenylenediamine); metal deactivators; lubricity agents (e.g. those commercially available as EC831 (ex Paramins) or "HITEC" (trade mark) 580 (ex Ethyl Corporation)); or carrier fluids such as a polyether e.g. a C₁₂-C₁₅ alkyl-substituted propylene glycol ("SAP
15 949" which is commercially available from member companies of the Royal Dutch/Shell group), "HVI" or "XHVI" (trade mark) base oil, a polyolefin derived from C₂-C₆ monomers, e.g. polyisobutylene having from 20 to 175, particularly 35 to 150, carbon atoms, or a
20 polyalphaolefin having a viscosity at 100°C in the range 2×10^{-6} to 2×10^{-5} m²/s (2 to 20 centistokes), being a hydrogenated oligomer containing 18 to 80 carbon atoms derived from at least one alphaolefinic monomer containing from 8 to 18 carbon atoms.

25 The lubricating oil and fuel compositions of the invention may be prepared by adding the reaction product of the present invention to a lubricating oil or fuel. Conveniently, an additive concentrate is blended with the lubricating oil or fuel. Such a
30 concentrate generally comprises an inert carrier fluid and one or more additives in a concentrated form. Hence the present invention also provides an additive concentrate comprising an inert carrier fluid and from 10 to 80%w (active matter) of a reaction product
35 according to the present invention, the percentages by

weight being based on the total weight of the concentrate.

Examples of inert carrier fluids include hydrocarbons and mixtures of hydrocarbons with alcohols or ethers, such as methanol, ethanol, propanol, 2-butoxyethanol or methyl tert-butyl ether. For example, the carrier fluid may be an aromatic hydrocarbon solvent such as toluene, xylene, mixtures thereof or mixtures of toluene or xylene with an alcohol. Alternatively, the carrier fluid may be a mineral base oil or mixture of mineral base oils, such as those sold by member companies of the Royal Dutch/Shell Group under the designations "HVI", e.g. "HVI 60" base oil, or the synthetic hydrocarbon base oils sold by member companies of the Royal Dutch/Shell Group of Companies under the designation "XHVI" (trade mark).

The present invention still further provides the use of a reaction product according to the present invention as a dispersant additive.

The present invention will be further understood from the following illustrative Examples. In these Examples, the number average molecular weights (M_n) specified for the polyisobutenyl moieties in the polyisobutenyl succinic anhydride/succinimide and the atactic propylene oligomer moieties in the atactic propylene oligomer based succinic anhydride/succinimide were determined by modern gel chromatography using polystyrene standards, e.g. as described in W.W. Yau, J.J. Kirkland and D.D. Bly, "Modern Size Exclusion Liquid Chromatography", John Wiley and Sons, New York, 21979.

Active matter content was determined by separating inactive material from the desired active matter on an aluminium oxide column using diethyl ether as eluant; acid value was determined according to ASTM D 664; and

Total Base Number was determined in accordance with ASTM D 2896.

In the Examples, the following abbreviations are used:-

- 5 PIBSA : a polyisobutenyl succinic anhydride in which
 the polyisobutenyl moiety has M_n (as
 measured by GPC) of 2200 ± 200 in Examples 1
 to 21, 37 to 52, 55 and 56, and 1000 ± 200
10 in Examples 22 to 36, 53, 54 and 57,
 prepared by the process according to
 EP-A-0542380
- APOSA : an atactic propylene oligomer based succinic
 anhydride in which the atactic propylene
 oligomer moiety has M_n (as measured by GPC)
15 of 2200 ± 200 in Examples 58 to 60, and
 835 ± 100 in Example 61, prepared by the
 process according to EP-A-0490454
- TETA : triethylene tetramine
- TEPA : tetraethylene pentamine
- 20 PEHA : pentaethylene hexamine
- S75 : a polyamine mixture containing tetraethylene
 pentamine, pentaethylene hexamine and higher
 ethylene polyamines in a weight ratio of
 1:2:1 which is commercially available from
25 Delamine B.V., Netherlands

Example 1

- A mono-ester of "SMA 3000A" and iso-octyl alcohol
 ("SMA 3840A" ex. Elf-Atochem) (1.5g) dissolved in
 "SHELLSOL A" (trade mark) (aromatic-rich (99%
30 aromatics), high boiling (B.Pt. 166-180°C) solvent (ex.
 Shell Nederland Chemie B.V.)) (150 ml) was added to a
 stirred solution of the reaction product of PIBSA and
 S75 (having a coupling ratio (mols of PIBSA to mols of
 polyamine) of 1.5 ± 0.1) (50g, 40.9% active matter) in
35 "SHELLSOL A" (50 ml) at 150°C. The reaction
 temperature was increased at approx. 15°C per hour and

the "SHELLSOL A", water and any alcohol released was removed by a Dean and Stark trap. Heating rates can be varied. Once the temperature had reached 200°C, the reaction was held at this temperature for 1 hour. The
5 excess solvent was removed under reduced pressure.

Examples 2 to 41

By processes similar to that described in Example 1 above, further reaction products of the present invention were prepared, namely those of Examples 2 to
10 41. The coupling ratio used in Example 21 was 1.2 ± 0.1 and that used in Examples 37 and 38 was 1.0 ± 0.1 .

Details of the types and amounts of the reagents used and, where known, the total base number and nitrogen content of the polyimide derivatives as
15 obtained are presented in Table 1 below, in which the percentage of reactant B is of the total weight of reactant A without adjustment to take account of the percentage of active matter in A:-

TABLE 1

Example No.	Reactants			Product		
		% Active matter of A	% B	% Active matter	Total Base Number (mg KOH/g)	Nitrogen content (%w)
1	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	40.9	3.0	42.8	21.6	0.95
2	A = PIBSA+S75 B = diester of SMA 3000A and iso-amyl alcohol	40.9	2.5	41	22.6	0.93
3	A = PIBSA+S75 B = diester of SMA 3000A and iso-amyl alcohol	40.9	4.7	42	22.0	0.95
4	A = PIBSA+S75 B = diester of SMA 3000A and iso-amyl alcohol	40.9	10.0	43	20.6	0.89
5	A = PIBSA+S75 B = diester of SMA 3000A and iso-amyl alcohol	40.9	5.0	42	22.8	0.91

6	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	40.9	1.0	43.1	23.2	0.95
7	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	40.9	2.0	44.6	21.2	0.92
8	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	40.9	4.0	43.2	20.8	0.97
9	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	40.9	5.0	45.4	19.2	0.91
10	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	40.9	6.0	46.5	18.2	0.90
11	A1 = PIBSA A2 = S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	60	6.0	60.2	NM	NM
12	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	40.9	7.0	45.1	18.0	0.92

13	A = PIBSA+S75 B = mono-ester of SMA 1000A and iso-octyl alcohol	40.9	2.0	41.6	18.9	0.95
14	A = PIBSA+S75 B = mono-ester of SMA 1000A and iso-octyl alcohol	40.9	4.0	42.8	19.3	0.98
15	A = PIBSA+S75 B = mono-ester of SMA 1000A and iso-octyl alcohol	40.9	5.0	43.6	16.9	0.96
16	A = PIBSA+S75 B = mono-ester of SMA 1000A and iso-octyl alcohol	40.9	6.0	43.7	17.3	0.96
17	A = PIBSA+S75 B = mono-ester of SMA 1000A and iso-octyl alcohol	40.9	8.0	44.7	16.7	0.95
18	A = PIBSA+S75 B = mono-ester of SMA 1000A and iso-octyl alcohol	40.9	10.0	45.9	16.3	0.94
19	A = PIBSA+S75 B = mono-ester of SMA 1000A and iso-octyl alcohol	40.9	20.0	47.6	14.1	0.89

20	A = PIBSA+S75 B = mono-ester of SMA 1000A and iso-octyl alcohol	40.9	30.0	51.7	12.1	0.89
21	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	42.1	4.0	40.0	28.0	1.2
22	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	2.5	47.8	44.7	1.93
23	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	60.0	3.0	61	55.5	2.43
24	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	3.0	48.2	44.0	1.91
25	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	60.0	4.0	61	56.7	2.43
26	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	4.0	48.6	45.0	1.92

27	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	5.0	48.8	43.4	1.87
28	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	6.0	49.3	40.9	1.88
29	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	7.0	49.2	41.0	1.89
30	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	8.0	48.7	38.4	1.79
31	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	10.0	47.4	37.0	1.76
32	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	46.8	15.0	48.8	34.4	1.76
33	A = PIBSA+TEPA B = mono-ester of SMA 1000A and iso-octyl alcohol	46.8	5.0	49.5	38.4	1.84

34	A = PIBSA+TEPA B = mono-ester of SMA 1000A and iso-octyl alcohol	46.8	10.0	49.6	31.3	1.73
35	A = PIBSA+TEPA B = mono-ester of SMA 1000A and iso-octyl alcohol	46.8	15.0	43.2	28.9	1.73
36	A = PIBSA+TEPA B = mono-ester of SMA 1000A and iso-octyl alcohol	46.8	20.0	47.3	25.2	1.58
37	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	60	4.0	60.1	54.2	2.02
38	A = PIBSA+TEPA B = mono-ester of SMA 3000A and iso-octyl alcohol	42.6	4.0	43.7	29.3	1.14
39	A = PIBSA+S75 B = poly n-amyl acrylate	40.9	3.0	43.0	20.1	0.95
40	A = PIBSA+S75 B = poly n-amyl acrylate	40.9	5.0	43.6	16.7	0.94
41	A = PIBSA+S75 B = poly n-amyl acrylate (post- treated with aniline)	40.9	3.0	NM	24.9	1.1

In the above Examples, and those that follow below:

- (a) "SMA 3000A" is a styrene/maleic anhydride oligomer (M_n 2350) in which the mole ratio of styrene:maleic anhydride is 3 (ex. Elf-Atochem)
- 5 (b) "SMA 1000A" is a styrene/maleic anhydride oligomer (M_n 962) in which the mole ratio of styrene:maleic anhydride is 1 (ex. Elf-Atochem)
- (c) Reactant B in Examples 1, 6 to 12, 21 to 32, 37 and 38 was "SMA 3840A", a low molecular weight
10 (M_n 3120) oligomer in which the mole ratio of styrene:maleic anhydride is 3, and in which the maleic anhydride has been reacted with iso-octyl alcohol to form the mono-ester (ex. Elf-Atochem)
- (d) Reactant B in Examples 39 to 41 was a low
15 molecular weight (approx. 1000) polyacrylic acid esterified with n-amyl alcohol
- (e) Reactant A in Examples 1 to 10, 12 to 20, 22 to 36 and 39 to 41 had a coupling ratio (mols of PIBSA to mols of polyamine) of 1.5 ± 0.1
- 20 (f) Reactant A in Example 21 had a coupling ratio (mols of PIBSA to mols of polyamine) of 1.2 ± 0.1
- (g) Reactant A in Examples 37 and 38 had a coupling ratio (mols of PIBSA to mols of polyamine) of 1.0 ± 0.1
- 25 (h) Reactant A in Examples 22 to 36, 53, 54 and 57 was "SAP 230TP", a mono/bis succinimide ashless dispersant (ex. Shell Additives International Ltd.)
- (i) Reactant A in Examples 43 to 47 and 56 was "SAP
30 285", a high molecular weight bis succinimide ashless dispersant (ex. Shell Additives International Ltd.)
- (j) 'NM' = Not Measured

Example 42

"SMA 3000A" (1.65g) dissolved in mesitylene (50 ml) at 100°C was added to a stirred solution of the reaction product of PIBSA and S75 (having a coupling ratio (mols PIBSA to mols of polyamine) of 1.5 ± 0.1) (50g, 40.9% active matter) in "SHELLSOL A" (50 ml) at 140°C under nitrogen. The reaction temperature was increased and the "SHELLSOL A" and water was gradually removed by a Dean and Stark trap, over 4 hours. Once the temperature had reached 200°C the reaction was held at this temperature for 15 hours. The excess solvent was removed under reduced pressure (120°C at 5 mm Hg for 2 hours) to give a dark brown clear liquid (51g).

Details of the types and amounts of the reagents used and the total base number and nitrogen content of the polyimide derivative as obtained are presented in Table 2 below, in which the percentage of reactant B is of the total weight of reactant A without adjustment to take account of the percentage of active matter in A:-

TABLE 2

Example No.	Reactants			Product		
		% Active matter of A	% B	% Active matter	Total Base Number (mg KOH/g)	Nitrogen content (%w)
42	A = PIBSA+S75 B = SMA 3000A	40.9	3.0	39.6	16.3	0.8

Example 43

A mono-ester of "SMA 3000A" and iso-octyl alcohol ("SMA 3840A" ex. Elf-Atochem) (4.0g) dissolved in xylene (40 ml) was added to a stirred solution of the
5 reaction product of PIBSA and S75 (having a coupling ratio (mols of PIBSA to mols of polyamine) of 2.0 ± 0.2) (100g, 42.0% active matter) in xylene (100 ml) at 140°C under nitrogen. The reaction
10 temperature was increased and the xylene, water and any alcohol were gradually removed by a Dean and Stark trap, over 4 hours. Once the temperature had reached 200°C the reaction was held at this temperature for 1 hour. The excess solvent was removed under reduced
15 pressure (110°C at 5 mm Hg for 2 hours) to give a dark brown clear liquid (100g).

Examples 44 to 47

By processes similar to that described in Example 43 above, further reaction products of the present
20 invention were prepared.

Details of the types and amounts of the reagents used and the total base number and nitrogen content of the polyimide derivatives as obtained are presented in Table 3 below, in which the percentage of reactant B is
25 of the total weight of reactant A without adjustment to take account of the percentage of active matter in A:-

TABLE 3

Example No.	Reactants			Product		
		% Active matter of A	% B	% Active matter	Total Base Number (mg KOH/g)	Nitrogen content (%w)
43	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	42.0	4.0	44.1	13.3	0.7
44	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	42.0	1.0	44.0	14.8	0.72
45	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	42.0	2.0	43.3	14.4	0.71
46	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	42.0	3.0	46.1	13.7	0.68
47	A = PIBSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	42.0	5.0	44.4	12.9	0.69

Example 48

A C₁₀-20 straight chain alpha-olefin copolymerised with maleic anhydride ("PX-100" (M_n 2400) ex. Akzo Nobel GmbH) (2.5g) dissolved in "SHELLSOL A" (10 ml)
5 was added to a stirred solution of the reaction product of PIBSA and S75 (having a coupling ratio (mols of PIBSA to mols of polyamine) of 1.5 ± 0.1) (50 g, 40.9% active matter) in "SHELLSOL A" (50 ml) at 140°C under nitrogen. The reaction temperature was increased and
10 the "SHELLSOL A" and water were gradually removed by a Dean and Stark trap, over 4 hours. Once the temperature had reached 200°C the reaction was held at this temperature for 15 hours. The excess solvent was removed under reduced pressure (120°C at 5 mm Hg for 2
15 hours) to give a dark brown clear liquid (51g).

Examples 49 to 52

By processes similar to that described in Example 48 above, further reaction products of the present invention were prepared.

20 Details of the types and amounts of the reagents used and the total base number and nitrogen content of the polyimide derivatives as obtained are presented in Table 4 below, in which the percentage of reactant B is of the total weight of reactant A without adjustment to
25 take account of the percentage of active matter in A:-

TABLE 4

Example No.	Reactants			Product		
		% Active matter of A	% B	% Active matter	Total Base Number (mg KOH/g)	Nitrogen content (%w)
48	A = PIBSA+S75 B = C ₁₀ -20 straight chain alpha-olefin copolymerised with maleic anhydride	40.9	5.0	44.5	17.4	0.97
49	A = PIBSA+S75 B = C ₁₀ -20 straight chain alpha-olefin copolymerised with maleic anhydride	40.9	2.0	42.0	24.0	0.97
50	A = PIBSA+S75 B = C ₁₀ -20 straight chain alpha-olefin copolymerised with maleic anhydride	40.9	4.0	43.0	19.6	0.96

51	A = PIBSA+S75 B = C ₁₀ -20 straight chain alpha- olefin copolymerised with maleic anhydride	40.9	6.0	45.0	19.6	0.93
52	A = PIBSA+S75 B = C ₁₀ -20 straight chain alpha- olefin copolymerised with maleic anhydride	40.9	8.0	45.5	19.5	0.93

Example 53

5 The n-pentanol ester of polyacrylic acid ("GOODRITE 752" (M_n 1000) ex. Goodrich Chemicals) (6.3g) dissolved in xylene (63 ml) was added to a stirred solution of
10 the reaction product of PIBSA and TEPA (having a coupling ratio (mols of PIBSA to mols of polyamine) of 1.5 ± 0.1) (63g, 45.0% active matter) in xylene (63 ml) at 140°C under nitrogen. The reaction temperature was increased and the xylene and water were gradually
15 removed by a Dean and Stark trap, over 4 hours. Once the temperature had reached 200°C the reaction was held at this temperature for 1 hour. The excess solvent was removed under reduced pressure (120°C at 5 mm Hg for 2 hours) to give a dark brown clear liquid (65g).

15 Example 54

By a process similar to that described in Example 53 above, a further reaction product of the present invention was prepared.

20 Details of the types and amounts of the reagents used and the total base number and nitrogen content of the polyimide derivatives as obtained are presented in Table 5 below, in which the percentage of reactant B is of the total weight of reactant A without adjustment to take account of the percentage of active matter in A:-

TABLE 5

Example No.	Reactants			Product		
		% Active matter of A	% B	% Active matter	Total Base Number (mg KOH/g)	Nitrogen content (%w)
53	A = PIBSA+TEPA B = n-pentanol ester of polyacrylic acid	45.0	10.0	49.9	30.8	1.84
54	A = PIBSA+TEPA B = n-pentanol ester of polyacrylic acid	45.0	5.0	47.8	40.0	1.91

Example 55

A styrene/acrylic acid copolymer ("MOREZ 300" (M_n 1300) ex. Morton International Limited) (7g) was added to a solution of the reaction product of PIBSA and S75 (having a coupling ratio (mols of PIBSA to mols of polyamine) of 1.5 ± 0.1) (100g) and titanium(IV) isopropoxide (0.15g) in "SHELLSOL A" (150g) at room temperature. A nitrogen purge was introduced and the temperature was increased to 120°C for 1 hour to dissolve the "MOREZ 300". The temperature was then increased and the "SHELLSOL A" and water were gradually removed by a Dean and Stark trap, over 4 hours. Once the temperature had reached 200°C , the reaction was held at this temperature for 24 hours. The product was dissolved in toluene and filtered. The excess solvent was removed under reduced pressure (120°C at 5 mm Hg for 2 hours) to give a dark brown liquid (100g).

Examples 56 to 57

By processes similar to that described in Example 55 above, further reaction products of the present invention were prepared, the coupling ratio used in Example 56 being 2.0 ± 0.2 .

Details of the types and amounts of the reagents used and, where known, the total base number and nitrogen content of the polyimide derivatives as obtained are presented in Table 6 below, in which the percentage of reactant B is of the total weight of reactant A without adjustment to take account of the percentage of active matter in A:-

TABLE 6

Example No.	Reactants			Product		
		% Active matter of A	% B	% Active matter	Total Base Number (mg KOH/g)	Nitrogen content (%w)
55	A = PIBSA+S75 B = styrene/acrylic acid copolymer	40.9	7.0	43.2	14.0	0.91
56	A = PIBSA+S75 B = styrene/acrylic acid copolymer	40.9	4.0	43.4	11.0	0.69
57	A = PIBSA+TEPA B = styrene/acrylic acid copolymer	45.0	11.0	47.4	30.0	NM

Example 58

A mono-ester of "SMA 3000A" and iso-octyl alcohol ("SMA 3840A" ex. Elf-Atochem) (2g) dissolved in "SHELLSOL A" (20 ml) was added to a stirred solution of the reaction product of APOSA and S75 (having a coupling ratio (mols of APOSA to moles of polyamine of 1.5 ± 0.1) (50g) in "SHELLSOL A" (130 ml) at 140°C under nitrogen. The reaction temperature was increased and the "SHELLSOL A", water and any alcohol were gradually removed by a Dean and Stark trap, over 2 hours. Once the temperature had reached 200°C the reaction was held at this temperature for 1 hour. The excess solvent was removed under reduced pressure (120°C at 5 mm Hg for 2 hours) to give a dark brown clear liquid (50g).

Examples 59 to 61

By processes similar to that described in Example 58 above, further reaction products of the present invention were prepared, the coupling ratio used in Example 59 being 2.0 ± 0.2 .

Details of the types and amounts of the reagents used and the total base number and nitrogen content of the polyimide derivatives as obtained are presented in Table 7 below, in which the percentage of reactant B is of the total weight of reactant A without adjustment to take account of the percentage of active matter in A:-

TABLE 7

Example No.	Reactants			Product		
		% Active matter of A	% B	% Active matter	Total Base Number (mg KOH/g)	Nitrogen content (%w)
58	A = APOSA+S75 B = mono-ester of SMA 3000A and iso-octyl alcohol	47.9	4.0	48.5	18.4	0.93
59	A = APOSA+PEHA B = mono-ester of SMA 3000A and iso-octyl alcohol	42.0	2.0	46.2	14.9	0.8
60	A = APOSA+PEHA B = mono-ester of SMA 3000A and iso-octyl alcohol	42.0	4.0	45.6	22.9	1.09
61	A = APOSA+TETA B = mono-ester of SMA 3000A and iso-octyl alcohol	42.0	5.0	55.1	37.7	1.82

Example 62

Haze Test

5 "Haze" is that percentage of transmitted light which, in passing through the specimen, deviates from the incident beam by forward scattering. For the purpose of this method only light flux deviating more than 2.5° on average is considered to be haze.

10 The dispersant is dissolved in "HVI 60" oil, at 100°C, in a mass ratio of 1:9. The haze of the solution is measured by means of a pivotable-sphere hazemeter, applying a correction for the haze of the solvent. The hazemeter is as specified in ASTM D1003, with digital display reading to 0.1%.

Results of these tests are given in Table 8:

TABLE 8

Example No.	Haze (%)	Example No.	Haze (%)
1	5.0	29	2.2
2	9.6	30	5.0
3	8.8	31	1.0
4	6.2	32	0.4
5	7.1	33	2.8
6	7.9	34	2.0
7	5.2	35	1.8
8	6.0	36	4.3
9	10.3	39	6.5
10	7.0	40	4.0
12	14.7	48	5.4
13	3.4	49	5.2
14	4.8	50	6.8
15	4.5	51	5.5
16	4.6	52	6.9
17	5.1	53	3.3
18	6.5	54	2.4
19	6.7	55	1.0
20	9.3	56	1.0
21	11.0	57	3.0
22	<1.0	58	3.7
24	<1.0	59	2.4
26	<1.0	60	4.6
27	1.3	61	1.6
28	<1.0		

It can be seen from the figures in Table 8 that
reaction products of the present invention exhibit low
5 levels of haze.

Example 63

Carbon Black Dispersancy Test (CBDT)

(British Rail Publication BR 669: 1984)

5 Samples of a SAE 15W40 Middle East lubricating oil
containing a commercial package of a zinc
dialkyldithiophosphate, an overbased calcium alkyl
salicylate and VI improver, were modified by
incorporation of oligomeric reaction products of the
Examples to give oils containing the derivatives at a
10 concentration of 1%w active matter. 3%w of carbon
black was then added to each oil and (percentage)
increase in kinematic viscosity at 60°C was determined,
using an Ubbelohde viscometer. A low result indicates
good performance. The absolute values obtained are
15 dependent on the active surface area of the carbon
black used, and therefore comparative series should be
tested with identical samples of carbon black. The
tests were carried out using "Flamruss" (trade mark)
carbon black.

20 The results of these tests are given in Table 9:

TABLE 9

Example No.	CBDT (%)	Example No.	CBDT (%)
1	15.3	24	15.3
2	14	26	16.2
3	18	27	14.7
4	23	28	18.4
5	13.7	29	15.2
6	15.9	30	15.3
7	15.9	31	14.8
8	15.5	32	15.3
9	18.3	37	14.8
10	19.5	38	15.3
11	20.3	40	17.6
12	17.2	41	18.1
22	15.7	42	16.7

Example 64

Rheology Test

(Using a controlled strain rate rheometer (Couette geometry))

5

Carbon black (Cabot Vulcan XC72R), oven dried, is equilibrated at 100°C overnight using a heated stirrer block, with an appropriate concentration of the sample dispersant in an appropriate fully formulated test oil.

10

The equilibrated sample is measured using a Couette geometry over a range of shear rates. An initial manual shear rate sweep is carried out to determine the appropriate sensitivity setting for the rheometer and to precondition the sample to eliminate any shear

15

history. Subsequently the sample is left to temperature equilibrate for at least 10 minutes before the measurement is made. As in the Carbon Black Dispersancy Test (CBDT), increases in viscosity which are lower than that with the parent dispersant, e.g.

"SAP 230TP" and "SAP 285", indicate improved dispersancy over those succinimides.

The results of these tests are given in Table 10, the figures in brackets being those for the corresponding parent dispersants:

TABLE 10

Example No.	Rheology ($1s^{-1}$)	Example No.	Rheology ($1s^{-1}$)
1	1 (5)	32	0.5 (3)
6	1.5 (5)	33	1 (3)
7	2 (5)	34	0.5 (3)
8	0.5 (5)	35	0.5 (3)
9	0.5 (5)	36	0.5 (3)
10	0.5 (5)	39	1.2 (3)
12	0.5 (5)	40	1 (3)
13	4 (5)	42	3 (5)
14	2 (5)	43	5 (8)
15	4 (5)	44	6 (8)
16	2 (5)	45	4 (8)
17	3 (5)	46	5 (8)
18	3 (5)	47	5 (8)
19	4 (5)	48	2 (4)
20	4 (5)	49	2 (4)
22	2 (3)	50	2 (4)
24	1.5 (3)	51	2 (4)
26	1 (3)	52	3 (4)
27	0.5 (3)	53	1 (3)
28	0.5 (3)	54	1 (3)
29	0.5 (3)	55	0.5 (3)
30	0.5 (3)	56	1 (3)
31	0.5 (3)	57	0.5 (2)

Example 65

Corrosion Test

Compounds of the present invention display reduced corrosion, as demonstrated in the L-10 bench corrosion test without severity adjustment (ref. API CG-4 test). The results, at the same dispersant treat rates, were
5 as follows:

TABLE 11

Sample	Lead leached (ppm)
"SAP 230TP"	18
Example 27	5

10 Example 66

Fluoroelastomer Seals Test

Compounds of the present invention are less aggressive to fluoroelastomer seals, as demonstrated in the Mercedes Benz fluoroelastomer seal test. When seal
15 fluoroelastomer was subjected to a formulation containing Example 47 its tensile strength was reduced by only 16% and the extent to which it could be stretched before breaking was only 15%; the reductions in an analogous formulation, at the same dispersant
20 treat rate, containing, instead, "SAP 285", were greater:

TABLE 12

Sample	Tensile strength	Elongation at break
"SAP 285"	-31%	-30%
Example 47	-16%	-15%

25

TS 7037

C L A I M S



1. The reaction product of:

(i) an oligomer having a number average molecular weight of no more than 7500 and being formed from one monomer or two or more different monomers, the or each

5 monomer being an ethylenically unsaturated compound and said oligomer or each of said monomers being optionally partially or fully esterified, each of which monomers may not contain an ether moiety nor be a cyclopentadienyl group, said oligomer optionally being

10 reacted with a low molecular weight amine, with

(ii) a polyalkenyl derivative of an ethylenically unsaturated carboxylic reagent (iii), which derivative has a number average molecular weight of at least 1600 when the oligomer is a co-oligomer of two monomers one

15 of which is a straight chain olefin, and

(iv) a polyamine containing at least two $-NH_2$ and/or $-NH$ groups, or with

(v) the pre-formed product of reagents (ii) and (iv),

20 wherein at least one of the ethylenically unsaturated compounds of the oligomer has a functional group which can react with an amine, the oligomeric reaction product optionally being further reacted with a low molecular weight amine.

25 2. A reaction product according to claim 1 wherein the low molecular weight amine is aniline.

3. A reaction product according to Claim 1 or 2, wherein the ethylenically unsaturated carboxylic reagent is selected from monoethylenically unsaturated

30 C_{4-10} dicarboxylic acids and anhydrides.

4. A reaction product according to Claim 1, 2 or 3, wherein the polyalkenyl derivative is derived from a

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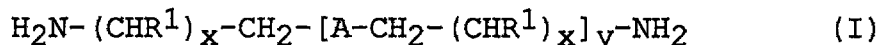


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polyalkene which is a polymer of at least one C₂-5 monoolefin.

5. A reaction product according to any one of the preceding Claims, wherein the polyamine is a compound of the general formula



wherein A is -NH or -O-, each R¹ independently represents a hydrogen atom or a methyl group, x is in the range 1 to 3, and y is in the range 1 to 10 when A is -NH or y is in the range 1 to 200 when A is -O-.

6. A process for the preparation of a reaction product according to claim 1 which comprises reacting

(i) an oligomer having a number average molecular weight of no more than 7500 and being formed from one monomer or two or more different monomers, the or each monomer being an ethylenically unsaturated compound and said oligomer or each of said monomers being optionally partially or fully esterified, each of which monomers may not contain an ether moiety nor be a cyclopentadienyl group, said oligomer optionally being reacted with a low molecular weight amine, with

(ii) a polyalkenyl derivative of an ethylenically unsaturated carboxylic reagent (iii), which derivative has a number average molecular weight of at least 1600 when the oligomer is a co-oligomer of two monomers one of which is a straight chain olefin, and

(iv) a polyamine containing at least two -NH₂ and/or -NH groups, or with

(v) the pre-formed product of reagents (ii) and (iv),

wherein at least one of the ethylenically unsaturated compounds of the oligomer has a functional group which can react with an amine, the oligomeric reaction product optionally being further reacted with a low molecular weight amine.

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7. A lubricating oil composition comprising a major amount of a lubricating oil and a minor amount of a reaction product according to any one of Claims 1 to 5.
8. A fuel composition comprising a major amount of a fuel and a minor amount of a reaction product according to any one of Claims 1 to 5.
9. An additive concentrate comprising an inert carrier fluid and from 10 to 80%w, based on the total concentrate, of a reaction product according to any one of Claims 1 to 5.
10. Use of a reaction product according to any one of Claims 1 to 5 as a dispersant additive.

PS36/TS7037FF

