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Sulfurized olefins.

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Description

This invention is directed to sulfurized products which are useful as additives to lubricant compositions, various greases and liquid hydrocarbyl fuels. This invention is particularly directed to a process of preparing these sulfurized products, the products so prepared and lubricating and fuel compositions containing them.

Sulfurized olefins have been extensively used in many lubricant applications requiring extreme pressure/antiwear activity. It is well known that many methods have been used for producing organic sulfides by treating olefins. It is also well known that many sulfurized organic compositions are useful as lubricant additives. Typically, the prior art processes provide sulfurized products having undesirably high levels of thiones, such as dithiol-thiones, and high levels of thiones in combination with various complex sulfurized mixtures. Typically also these processes, which are expensive and difficult to control, provide products having a highly disagreeable odor.

This invention relates to a process of preparing sulfurized organic additive products. These products consist essentially of mixtures of polysulfides, oligomers and dithiol-thione type compounds. The additive products are prepared under pressure by the reaction of olefinic compounds with elemental sulfur within a relatively narrow range of temperatures, pressures and ratio of reactants to form products containing sulfur. The additive product is suitable for use in an oil of lubricating viscosity or grease thereof or a liquid hydrocarbyl fuel and is prepared in a process comprising reacting in a suitable reaction zone an olefinic hydrocarbyl compound having at least one olefinic double bond with elemental sulfur in a molar ratio of sulfur to olefin of from 1.9:1 to 2.1:1 under pressures of from 2070 kPa (300 psi) to 6200 kPa (900 psi) at temperatures ranging from 140 to 180°C for a time sufficient and until the reaction pressure drops to 340 kPa (50 psi) to 690 kPa (100 psi) thereby producing a sulfurized additive product containing at least 45 weight percent of sulfur, preferably 45 to 55 weight percent sulfur, and consisting essentially of a major amount of polysulfides and a minor amount of dithiol-thione type compounds.

Sulfurized additive products of enhanced extreme pressure/antiwear activity and improved corrosivity are obtained. This invention is also directed to the additive products themselves and to compositions comprising oils of lubricating viscosity or greases prepared therefrom or liquid hydrocarbyl fuels containing the same. The specific and significantly improved discrete compositions produced by the narrowly defined processing conditions disclosed herein provide thermally and oxidatively stable, non-corrosive, low-odor, extreme pressure/antiwear additives for lubricating oils, greases and fuels.

The invention in a particular aspect is directed to lubricant oil additives prepared in a one step—one pot process comprising reacting in a suitable reaction zone an olefinic hydrocarbyl compound having at least one olefinic double bond with elemental sulfur.

A wide variety of olefinic substances may be utilized in the process in accordance with the invention. This includes olefins with terminal or internal double bonds and containing from about 2 to 8 or more carbon atoms per molecule in either straight, branched chain or cyclic compounds and these may be exemplified by ethylene, propylene, 1-butene, cis and trans-2-butene, isobutylene, diisobutylene, triisobutylene, pentene, cyclopentene, hexene, cyclohexene, octene, 1-decene, etc. Also useful are diolefins, for example butadiene, isoprene, divinyl benzene, pinene, p-menthene and limonene. In general, C₃ to C₆ olefins or mixtures thereof are preferred and more preferably butylenes are desirable for preparing the sulfurized products embodied herein because the combined sulfur content of the product decreases with increasing carbon content and the miscibility of the product with oil is lower in the case of propylene and ethylene derivatives.

In some embodiments of the invention, therefore, isobutylene is particularly preferred as the predominant olefinic reactant, but it may be employed, desirably in major proportions, in mixtures containing one or more other olefins. Moreover, substantial proportions of saturated aliphatic hydrocarbons, as exemplified by methane, ethane, propane, butane, pentane, etc. may be contained in the olefinic feed. Such alkanes are preferably present in minor proportions in most instances to avoid unnecessary dilution of the reaction since they neither react nor remain in the product, but are expelled in the off-gases or by subsequent distillation. However, such mixed feed can substantially improve the economics of the process since such streams are of lower value than a stream of, for example, relatively pure isobutylene.

Volatile olefins are often readily available in liquid form, and it is usually desirable to utilize olefinic liquids which are vaporized by the heat of reaction, as such evaporation provides a substantial cooling effect that permits the flowing of water for cooling the reactor to be reduced considerably for greater economy. Also, there are indications that the use of a volatile liquid olefin reactant has the unexpected and desirable effect of lowering the viscosity of the final product.

The specifically narrow molar ratio of sulfur to olefin may range from 1.9:1 to 2.1:1. In the case of butylene, the optimum ratio preferably is from 1.95:1 to 2.05:1. The reaction temperature may range from 140 to 180°C, preferably from 158 to 165°C and most preferably from 159 to 161°C. The preferred temperature is 160°C. The reaction pressure is allowed to seek its own optimum level and may range from 2070 kPa (300 psi) to 6200 kPa (900 psi). The preferred pressures are from 5170 kPa (750 psi) to 5860 kPa (850 psi). A critical aspect of the pressure parameter is the pressure at which the reaction is stopped. Once the reaction begins to seek its own pressure level and is held at a specific temperature most preferably

160°C until the pressure begins to drop. When this occurs the pressure at the reaction temperature must not be allowed to drop out of the range of from 340 kPa (50 psi) to 690 kPa (100 psi). Any conventional means known in the art for accomplishing this may be used.

The final pressure is indicative of the cited stoichiometry in accordance with the invention whereby the optimum product containing 45 to 61.5 weight % sulfur, preferably 45 to 55 weight % sulfur with no more than 10 to 20 weight % dithiol-thione type compounds based upon total weight of the additive product, is obtained. The weight percent of sulfur to unsaturated olefin is preferably 59 to 61.5 weight percent for a C₃ olefin (propylene), 52 to 54.5 weight percent for a C₄ olefin (butylene) and 46.5 to 49 weight percent for a C₅ olefin (pentane). If the process is higher than about 690 kPa (100 psi), not enough olefin has reacted and if the pressure is lower than about 340 kPa (50 psi), too much olefin has reacted to satisfy the desired stoichiometric relationship of the reactants.

It has been discovered that only within this narrow range of pressure and stoichiometry (mole ratio) will the resultant product or composition exhibit the improved extreme pressure/antiwear characteristics. Typical reaction time for the novel reactions in accordance with this invention range from 5 to 10 hours. As noted hereinabove, the narrow range of temperatures, pressures and molar ratios of the reactants are critical if products with enhanced antiwear activity and improved corrosivity are to be obtained. Any deviation outside of the above stated ranges produces vastly inferior products. The reaction is carried out in the absence of added H₂S.

The reaction may be catalyzed with a rate accelerating catalyst. Alkylamine catalysts such as n-butylamine, di-n-butylamine, n-octylamine, triethylamine, di-cyclohexylamine and the like are suitable. However, any suitable rate acceleration catalyst known in the art may be used, as for example quinoline and attapulgite acid clay.

In accordance with the invention, the reaction between the sulfur and the olefinic compound may be conducted in any suitable reaction vessel. The sulfur may be heated to a predetermined temperature in the reaction vessel prior to contacting it with the olefinic reactant, which may be chilled in order to liquify it and get it into the reactor. Once the reaction begins, the olefin is charged to the reaction zone substantially about as rapidly as it is consumed.

The compositions hereof may comprise any oleaginous materials that require lubricative properties under extreme pressure conditions and require protection against excessive wear under operating conditions, but normally exhibit insufficient anti-corrosion properties. Especially suitable for use with the additives of this invention are liquid hydrocarbon oils of lubricating viscosity. Lubricant oils, improved in accordance with the present invention, may be of any suitable lubricating viscosity. In general, the lubricant compositions may comprise any mineral or synthetic oil of lubricating viscosity or mixtures thereof. The additives of this invention are especially useful in greases and in automotive fluids such as brake fluids and power brake fluids, transmission fluids, power steering fluids, various hydraulic fluids and gear oils and in liquid hydrocarbon fuels.

In instances where synthetic oils are desired in preference to refined petroleum or mineral oil they may be employed alone or in combination with a mineral oil. They may also be used as the vehicle or base of grease compositions. Typical synthetic lubricants include polyisobutylene, polybutenes, hydrogenated polydecenes, polypropylene glycol, polyethylene glycol, trimethylol propane esters, neopentyl and pentaerythritol esters of carboxylic acids, di(2-ethylhexyl) sebacate, di(2-ethylhexyl) adipate, dibutyl phthalate, fluorocarbons, silicate esters, silanes, esters of phosphorus-containing acids, liquid ureas, ferrocene derivatives, hydrogenated mineral oils, chain-type polyphenols, siloxanes and silicones (polysiloxanes), alkyl-substituted diphenyl ethers typified by a butyl-substituted bis(p-phenoxy phenyl) ether, phenoxy phenylethers, dialkylbenzenes, etc.

As hereinbefore indicated, the aforementioned additives can be incorporated as additives in grease compositions. When high temperature stability is not a requirement of the finished grease, mineral oils having a viscosity of at least 40 SSU at 65.6°C (150°F) are useful. Otherwise, those falling within the range of from about 60 SSU to about 6,000 SSU at 37.8°C (100°F) may be employed.

The lubricating compositions of the improved greases of the present invention, containing the above-described additives, are combined with a grease forming quantity of a thickening agent. For this purpose, a wide variety of materials can be dispersed in the lubricating oil in grease-forming quantities in such degree as to impart to the resulting grease composition the desired consistency. Exemplary of the thickening agents that may be employed in the grease formulation are metal soaps as well as non-soap thickeners, such as surface-modified clays and silicas, aryl ureas, calcium complexes and similar materials. In general, grease thickeners are employed which do not melt or dissolve when used at the required temperature within a particular environment; however, in all other respects, any material which is normally employed for thickening or gelling oleaginous fluids or forming greases may be used in the present invention.

Generally, the lubricants and fuels of the present invention contain an amount of the sulfurized product effective to improve extreme pressure properties and antiwear and oxidation characteristics. Normally this amount will be 0.01 to 20% and preferably 0.01 to 10% of the total weight of the lubricant.

The invention also contemplates the use of other additives in combination with the sulfurized olefin product. Such additives include, for example, detergents and dispersants of the ash-producing or ashless type, corrosion-inhibiting agents, auxiliary oxidation-inhibiting agents, pour point depression agents, auxiliary extreme pressure agents, color stabilizers and anti-foam agents.

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The following examples serve to illustrate the present invention, but are not intended as limitations thereon unless otherwise stated.

Example 1

5 This Example was prepared in accordance with the invention using a molar ratio of sulfur to isobutylene of 2.0:1.

A 1-gallon stirred autoclave (reactor) was charged with 1,344 g sulfur and 1.2 g n-butylamine (0.05% of the total charge). The reactor was sealed and purged three times with nitrogen to remove oxygen. The reactor was then charged with 1,176 g isobutylene under pressure. The reactor agitator was activated and
10 heated to 160°C and held at that temperature until the pressure dropped from its maximum of 4830 kPa (700 psi) to below 520 kPa (75 psi). The reactor was cooled to 100°C and vented to a caustic scrubber. The product was then sparged with nitrogen for three hours at 100°C to remove gaseous contaminants, cooled to less than 30°C and then filtered through diatomaceous earth. The product yield was about 99% of the total reactants charged.

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Comparative Example A

This Example is outside the scope of the invention and was prepared using a molar ratio of sulfur to isobutylene of 1.8:1.

The procedure for Example 1 was used, but a lower sulfur:isobutylene mole ratio was charged. The
20 charge consisted of: 1,300 g sulfur, 1.2 g n-butylamine and 1,264 g isobutylene. Maximum pressure achieved was 5790 kPa (840 psi) with a pressure drop to less than 520 kPa (50 psi). Total reaction time was about eleven hours.

Comparative Example B

25 This Example was prepared using a molar ratio of sulfur to isobutylene of 1.0:1. It accordingly is outside the scope of the invention.

The procedure for Example 2 was used, but a lower sulfur:isobutylene mole ratio was charged. The charge consisted of: 739 g sulfur, 1.2 g n-butylamine, and 1,249 g isobutylene. The product yield was only 86.5% of reactants charged and minimum pressure did not drop below about 1380 kPa (200 psi), indicating
30 an excess of unreacted olefin which was removed during the venting and sparging steps.

Comparative Example C

A prior art sulfurized olefin prepared in accordance with U.S. Patent 3,703,504.

35 Lubricating Properties

The products of Example 1, Comparative Example A, and Comparative B were blended into a fully formulated gear lubricant with a standard detergent/antirust/antioxidant package. The EP/antiwear activity was determined by evaluations using CRC (Coordination Research Counsel) L-42 gear testing as well as Four Ball testing. The CRC L-42 Test is referred to in U.S. Patent 4,431,552, and is further identified as
40 ASTM D-2782 modified January 1983. Test data is summarized in Table 1.

The Four Ball Test is described in U.S. Patent 3,423,316. A prior art sulfurized olefin prepared in accordance with U.S. Patent 3,703,504, Comparative Example C, was used for comparison with Example 1 of the invention and was evaluated at 3.79% in the identical package and oil blend. U.S. Patent 3,703,504 is directed to a process for producing sulfurized olefins comprising sulfohalogenating an olefin with a sulfur
45 halide to form a sulfohalogenated organic intermediate and thereafter sulfurizing and dehalogenating with aqueous alkali metal monosulfide.

TABLE 1

L-42 GEAR TEST USING FORMULATED GEAR OIL RESULTS REPORTED AS PERCENT SCORING

50

	Example 1 @ 2.84% in Fully Formulated <u>Gear Oil</u>	Comparative Example A @ 2.84% in Fully Formulated <u>Gear Oil</u>	Comparative Example B @ 3.79% in Fully Formulated <u>Gear Oil</u>
55			
	Pinion Coast, %	12	23
60	Pinion Drive, %	17	0
	Ring Coast, %	12	18
	Ring Drive, %	15	0
	<u>Assessment</u>	PASS	FAIL

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As can be seen, Example 1, in accordance with the invention, significantly outperforms the very similar Comparative Example A and even outperforms the product of Example B, even though Comparative Example B was tested at a much higher concentration.

TABLE 2

FOUR BALL TEST

(All Examples Evaluated in Mineral Oil Using
0.2% Additional Dibutyl Hydrogen Phosphite)

	Concentrate Wt. %	Load Wear Index
Example 1	4	84.3
Example 1	2	61.9
Example 1	1	61.2
Comparative Example B	4	67.2
Comparative Example C	1	49.8

As can be seen from Table 2, Example 1 in accordance with the invention significantly outperforms the very similar Example at 4% concentration. In fact, 2% of Example 1 is almost as good as twice the concentration of Comparative Example C. Also, as can be seen from Table 2, 1% of Example 1 significantly outperforms Comparative Example A.

TABLE 3

COPPER CORROSIVITY

(Evaluated in 1% 200' paraffinic neutral lubricating oil)

3 Hours at 121°C

	Example 1	Comparative Example A
Corrosivity Rating	1B	2B

The lower the rating the better the performance. The data in Table 3 shows that Example 1 in accordance with the invention is clearly superior to the Comparative Example.

Clearly, the products of this invention provide exceptional and highly improved extreme pressure/antiwear activity coupled with improved copper corrosion performance. The use of the novel sulfurized additives in premium quality automotive and industrial lubricants provide an unexpected significant combination of improved extreme pressure/antiwear activity and anticorrosion characteristics as well as reduced costs.

Claims

1. A process for making an additive product suitable for use in oils of lubricating viscosity or greases prepared therefrom or in liquid hydrocarbyl fuels comprising reacting in a suitable reaction zone an olefinic hydrocarbyl compound having at least one olefinic double bond with elemental sulfur in a molar ratio of sulfur to olefin of from 1.9:1 to 2.1:1 under pressures of from 2070 kPa (300 psi) to 6200 kPa (900 psi) at temperatures ranging from 140 to 180°C for a time sufficient and until the reaction pressure drops to 340 kPa (50 psi) to 690 kPa (100 psi) thereby producing a sulfurized additive product containing at least 45 weight percent sulfur and consisting essentially of a major amount of polysulfides and a minor amount of from 10 to 20 weight percent dithiol-thione type compounds.

2. The process of claim 1, wherein the additive product contains 45 to 55 weight percent sulfur.

3. The process in accordance with claim 1, wherein said process is a one step—1 pot process.

4. The process in accordance with claim 1, wherein the pressure varies from 5170 kPa (750 psi) to 5860 kPa (850 psi) and the temperature varies from 158 to 165°C and the molar ratio of reactant sulfur to olefin varies from 1.95:1 to 2.05:1.

5. The process in accordance with claim 4, wherein the temperature varies from 159 to 161°C.

6. The process in accordance with claim 1, wherein said process is carried out in the presence of a rate accelerating catalyst.

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7. The process in accordance with claim 6, wherein said rate accelerating catalyst is selected from the group consisting of n-butylamine, di-n-butylamine, n-octylamine, triethylamine, diisopropylamine, cyclohexylamine, dicyclohexylamine, quinoline and attapulgite acid clay.

8. The process in accordance with claim 7, wherein the rate accelerating catalyst is n-butylamine.

5 9. The process in accordance with claim 1, wherein the olefin is selected from the group consisting of ethylene, propylene, 1-butene, cis and trans-2-butene, isobutylene, diisobutylene, triisobutylene, pentene, cyclopentene, hexene, cyclohexene, octene, 1-decene, butadiene, isoprene, divinyl benzene, pinene, p-menthene and limonene.

10. The process in accordance with claim 9, wherein the olefin is isobutylene.

10 11. The process in accordance with claim 1, wherein the product is prepared in the absence of added H₂S.

12. A lubricant composition comprising a major amount of an oil of lubricating viscosity or grease prepared therefrom and a minor amount sufficient to impart extreme pressure, antiwear activity and anti-corrosion characteristics thereto of an additive product prepared in a process according to any one of
15 claims 1 to 11.

13. The lubricant composition of claim 12, wherein said major amount is an oil of lubricating viscosity.

14. The lubricant composition of claim 13, wherein said oil is selected from mineral oils, synthetic oils and mixtures thereof.

15. The lubricant composition of claim 12, wherein said major amount is a grease.

20 Patentansprüche

1. Verfahren zur Herstellung eines zum Einsatz in Ölen von schmierender Viskosität oder daraus hergestellten Schmierfetten oder flüssigen Kohlenwasserstoffbrennstoffen geeigneten Additivprodukts,
25 das die Reaktion einer olefinischen Kohlenwasserstoffverbindung, die wenigstens eine olefinische Doppelbindung aufweist, mit elementarem Schwefel in einem molaren Verhältnis von Schwefel zu Olefin von 1,9:1 bis 2,1:1 in einer geeigneten Reaktionszone unter Drücken von 2070 kPa (300 psi) bis 6200 kPa (900 psi) bei Temperaturen in einem Bereich von 140 bis 180°C für eine ausreichende Zeitspanne und bis der Reaktionsdruck auf 340 kPa (50 psi) bis 690 kPa (100 psi) abfällt, umfaßt, wodurch ein sulfuriertes
30 Additivprodukt hergestellt wird, das wenigstens 45 Gew.-% Schwefel enthält und im wesentlichen aus einer größeren Menge Polysulfiden und einer geringeren Menge von 10 bis 20 Gew.-% an Verbindungen vom Dithiolthion-Typ besteht.

2. Verfahren nach Anspruch 1, worin das Additivprodukt 45 bis 55 Gew.-% Schwefel enthält.

3. Verfahren nach Anspruch 1, worin das genannte Verfahren ein Einstufeneintopfverfahren ist.

35 4. Verfahren nach Anspruch 1, worin der Druck von 5170 kPa (750 psi) bis 5860 kPa (850 psi), die Temperatur von 158 bis 165°C und das molare Verhältnis des Reaktionspartners Schwefel zu Olefin von 1,95:1 bis 2,05:1 variiert.

5. Verfahren nach Anspruch 4, worin die Temperatur von 159 bis 161°C variiert.

40 6. Verfahren nach Anspruch 1, worin das Verfahren in Gegenwart eines geschwindigkeitsbeschleunigenden Katalysators durchgeführt wird.

7. Verfahren nach Anspruch 6, worin der geschwindigkeitsbeschleunigende Katalysator aus der Gruppe n-Butylamin, Di-n-butylamin, n-Octylamin, Triethylamin, Diisopropylamin, Cyclohexylamin, Dicyclohexylamin, Chinolin und Attapulgitssäureton ausgewählt wird.

45 8. Verfahren nach Anspruch 7, worin der geschwindigkeitsbeschleunigende Katalysator n-Butylamin ist.

9. Verfahren nach Anspruch 1, worin das Olefin aus der Gruppe Ethylen, Propylen, 1-Buten, cis- und trans-2-Buten, Isobutylen, Diisobutylen, Triisobutylen, Penten, Cyclopenten, Hexen, Cyclohexen, Octen, 1-Decen, Butadien, Isopren, Divinylbenzol, Pinen, p-Methen und Limonen ausgewählt wird.

10. Verfahren nach Anspruch 9, worin das Olefin Isobutylen ist.

50 11. Verfahren nach Anspruch 1, worin das Product in Abwesenheit von zugesetztem H₂S hergestellt wird.

12. Schmiermittelzusammensetzung mit einer größeren Menge eines Öls mit schmierender Viskosität oder eines daraus hergestellten Schmierfetts und einer geringeren Menge eines Additivprodukts, das gemäß einem Verfahren nach einem der Ansprüche 1 bis 11 hergestellt wurde, die ausreicht, der Schmiermittelzusammensetzung Extremdruck- und Antiverschleißwirksamkeit sowie Antikorrosionseigenschaften
55 zu verleihen.

13. Schmiermittelzusammensetzung nach Anspruch 12, worin die genannte größere Menge ein Öl mit schmierender Viskosität ist.

60 14. Schmiermittelzusammensetzung nach Anspruch 13, worin das genannte Öl aus Mineralölen, synthetischen Ölen und Gemischen davon ausgewählt ist.

15. Schmiermittelzusammensetzung nach Anspruch 12, worin die genannte größere Menge ein Schmierfett ist.

Revendications

65 1. Un procédé pour préparer un additif utilisable dans des huiles de viscosité convenable pour le

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- graissage ou des graisses préparées à partir de celles-ci ou dans des combustibles hydrocarbures liquides, caractérisé en ce qu'il comprend la réaction dans une zone réactionnelle convenable, d'un composé hydrocarbure oléfinique ayant au moins une double liaison oléfinique avec du soufre élémentaire selon un rapport molaire de soufre à oléfine de 1,9:1 à 2,1:1, sous des pressions de 2070 kPa à 6200 kPa (300 à 900 psi), à des températures comprises entre 140 et 180°C pendant une période de temps suffisante et jusqu'à ce que la pression réactionnelle tombe à une valeur comprise entre 340 kPa et 690 kPa (50 et 100 psi), produisant ainsi un additif sulfuré contenant au moins 45% en poids de soufre, de préférence de 45 à 55% en poids de soufre, et consistant essentiellement en une quantité majeure de polysulfures et en une quantité mineure de composés de type dithiol-thione.
2. Le procédé suivant la revendication 1, caractérisé en ce que l'additif produit contient de 44 à 55% en poids de soufre.
3. Le procédé suivant la revendication 1, caractérisé en ce qu'il est un procédé réalisé en une seule étape et dans un seul récipient.
4. Le procédé suivant la revendication 1, caractérisé en ce que la pression varie de 5170 à 5860 kPa (750 à 850 psi), que la température varie de 158 à 165°C et que le rapport molaire du soufre réactif à l'oléfine varie de 1,95:1 à 2,05:1.
5. Le procédé suivant la revendication 4, caractérisé en ce que la température varie de 159 à 161°C.
6. Le procédé suivant la revendication 1, caractérisé en ce qu'il est effectué en présence d'un catalyseur accélérant la vitesse.
7. Le procédé suivant la revendication 6, caractérisé en ce que ce catalyseur accélérant la vitesse est choisi dans le groupe comprenant la n-butylamine, la di-n-butylamine, la n-octylamine, la triéthylamine, la diisopropylamine, la cyclohexylamine, la di-cyclohexylamine, la quinoléine et une argile acide de type attapulgite.
8. Le procédé suivant la revendication 7, caractérisé en ce que ce catalyseur accélérant la vitesse est la n-butylamine.
9. Le procédé suivant la revendication 1, caractérisé en ce que l'oléfine est choisie dans le groupe comprenant l'éthylène, le propylène, le 1-butène, le cis- et le trans-2-butène, l'isobutylène, le diisobutylène, le triisobutylène, le pentène, le cyclopentène, l'hexène, le cyclohexène, l'octène, le 1-décène, le butadiène, l'isoprène, le divinylbenzène, le pinène, le p-menthène et le limonène.
10. Le procédé suivant la revendication 9, caractérisé en ce que l'oléfine est l'isobutylène.
11. Le procédé suivant la revendication 1, caractérisé en ce que le produit est préparé en l'absence de toute addition de H₂S.
12. Une composition de graissage, caractérisée en ce qu'elle comprend une quantité majeure d'une huile de viscosité convenable pour le graissage ou d'une graisse préparée à partir de celle-ci et une quantité mineure suffisante pour conférer à celle-ci une activité de pression extrême/antiusure et des caractéristiques d'anticorrosion, d'un additif préparé selon un procédé suivant l'une quelconque des revendications 1 à 11.
13. La composition de graissage suivant la revendication 12, caractérisée en ce que cette quantité majeure est une huile de viscosité convenable pour le graissage.
14. La composition de graissage suivant la revendication 13, caractérisée en ce que cette huile est choisie parmi des huiles minérales, des huiles de synthèse et leurs mélanges.
15. La composition de graissage suivant la revendication 12, caractérisée en ce que cette quantité majeure est une graisse.