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(71) Applicant: **IDEMITSU KOSAN COMPANY LIMITED**
1-1, Marunouchi 3-chome Chiyoda-ku
Tokyo 100(JP)

(72) Inventor: **Ichihashi, Toshihiko Idemitsu Kosan Co., Ltd.**
24-4, Anesakikaigan
Ichihara-shi Chiba-ken(JP)

(74) Representative: **Türk, Gille, Hrabal**
Brucknerstrasse 20
D-4000 Düsseldorf 13(DE)

(54) Lubricating oil composition for power control.

(57) Disclosed is a lubricating oil composition for power control, comprising (A) a base oil having a kinematic viscosity at 100 °C of 1 to 80 cSt, (B) 0.1 to 10% by weight (based on the total weight of the composition, the same shall apply hereinafter) of at least one kind of sulfur-containing compound selected from the group consisting of zinc dithiophosphate, sulfurized oils and fats and sulfurized olefin, (C) 0.1 to 10% by weight of alkaline earth metal based detergent-dispersant, and (D) 0.05 to 5% by weight of partial esters of polyhydric alcohols and/or succinimide.

Said composition is excellent in extreme-pressure property, antiwear property, and metal fatigue life, and has also a good initial frictional characteristics. In addition, the frictional characteristics hardly change with the time, and said composition is excellent in stability against oxidation, and in corrosion resistance. Further, since said lubricating oil compositions include those excellent in heat-resistance, they show an excellent lubricity for a long period when used as gear oil for automobiles, to extend the cycle time for oil replacement.

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LUBRICATING OIL COMPOSITION FOR POWER CONTROL

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a lubricating oil composition for power control, and more specifically to a lubricating oil composition which is excellent in frictional characteristics, extreme-pressure properties, antiwear properties and the like, and which is suitable as a lubricating oil for transmission gears, differential gears, hypoid gears of automobiles used at a high temperature for a long period.

2. Description of the Related Arts

Generally, as lubricating oils for power control mechanisms, including oils for transmission gears of automobiles, conventionally used ones have mainly been the lubricating oils in which anti-scoring properties to the tooth surfaces of gears are regulated by controlling the amount of sulfur-phosphorus based extreme-pressure agent to be added.

In recent years, however, automobiles have shown a marked trend to be higher-graded or distinctly classified. With such a trend, a smooth and comfortable operation in driving, that is, a favorable operation feeling has been regarded as important. In order to improve the operation feeling in driving, it is desired that the frictional property between the synchronizer ring and the gear cone in the synchromesh mechanism is so favorable (that means, ratio of coefficient of static friction (μ_0) to coefficient of kinematic friction (μ_k), i.e., μ_0/μ_k is small, and said ratio hardly changes with time), that the transmission can be operated smoothly. The use of conventional gear oils containing sulfur-phosphorus based extreme-pressure agent has frequently resulted in poor frictional property, and operation feeling has not been favorable.

In view of these points, various lubricating oil compositions have been proposed for improving the operation feeling (Japanese Patent Application Laid Open No. 192495/1987, No. 61090/1988, and No. 61091/1988). However, they have met problems in corrosion of lubricated portions and in the stability of the whole composition.

On the other hand, in the transmission of large-sized trucks in which oil temperature amounts to 150 ° C or more, insufficient heat-resistance has caused deterioration, corrosion of metals, formation of sludge and other problems, various troubles such as abrasion or damage of teeth surface .of gears, damages or seizure of bearings, and further, slip out of gear in some cases. Phosphoric acid esters and phosphorous acid esters which are included in phosphorus extreme pressure agents are afraid to accelerate said deterioration or corrosion, and accordingly the use of them has an inconvenience that lubricating oil must be replaced in a short period.

SUMMARY OF THE INVENTION

An object of the present invention is to provide a lubricating oil composition which is excellent in antiwear properties, and extreme-pressure properties, and in addition, favorable in heat-resistance, particularly a lubricating oil composition of a new type containing no phosphorus extreme-pressure agent.

Another object of the present invention is to provide a lubricating oil composition for power control which is excellent in anti-wear properties, and extreme-pressure properties, and at the same time favorable in the operation feeling mentioned above.

The present invention provides a lubricating oil composition for power control comprising:

(A) a base oil having a kinematic viscosity at 100 ° C of 1 to 80 cSt,

(B) 0.1 to 10% by weight (based on the total weight of the composition; the same shall apply hereinafter) of at least one kind of sulfur-containing compound selected from the group consisting of zinc dithiophosphate, sulfurized oils and fats, and sulfurized olefins,

(C) 0.1 to 10% by weight of alkaline earth metal based detergent-dispersant, and

(D) 0.05 to 5% by weight of partial ester of polyhydric alcohol and/or succinimide.

DESCRIPTION OF PREFERRED EMBODIMENTS

Various oils can be used for the base oil as component (A) of the present invention as long as they have a kinematic viscosity at 100° C of 1 to 80 cSt, preferably 3 to 50 cSt.

Thus, mineral oil-based lubricating oil fraction and synthetic lubricating oil are also available for this component. An example of the mineral oil-based lubricating oil fraction is a purified oil obtained by purifying a distillate oil with a conventional method, said distillate oil having been obtained by atmospheric distillation of a paraffin based crude oil, a naphthene based crude oil, or an intermediate based crude oil, or by vacuum distillation of a residual oil resulting from the atmospheric distillation. Examples of the synthetic lubricating oils are poly-alpha-olefins, polybutenes, dibasic acid esters, polyglycols, hindered esters, alkylbenzenes, polyethers and the like.

Regarding the sulfur-containing compounds as component (B) of the present invention, zinc dithiophosphate, sulfurized oils and fats, and sulfurized olefins are used singly or in combination. Therein, as zinc dithiophosphate (ZnDTP), various ones conventionally used including zinc diisopropyldithiophosphate, zinc di-2-ethylhexyldithiophosphate, zinc di-n-butyldithiophosphate, zinc di-n-amyldithiophosphate, zinc di-n-hexyldithiophosphate, zinc di-n-octyldithiophosphate, and zinc dinonylphenyldithiophosphate can be used.

When alkaline earth metal based detergent-dispersant as component (C) has a base number of 200 mg KOH/g or more, branched alkyl ZnDTP and/or alkylphenyl ZnDTP preferably used. In that case, it is preferred that branched alkyl ZnDTP and/or alkylphenyl ZnDTP may occupy more than half the quantity of whole ZnDTP used there. Herein preferable examples of the branched alkyl ZnDTP or alkylphenyl ZnDTPs are those of which branched alkyl groups or alkylphenyl groups have 3 to 20 carbon atoms, specifically, ZnDTPs of which branched alkyl groups are iso-propyl groups, 2-ethylhexyl groups, or alkylphenyl groups are nonylphenyl groups, as main constituent.

When alkaline earth metal based detergent-dispersant as component (C) has a base number of less than 200 mg KOH/g, primary ZnDTP shows a superior stability against oxidation. and is favorably used accordingly. In that cases it is preferred that primary ZnDTP may occupy more than half the quantity of whole ZnDTP used there. Herein preferable examples of the primary ZnDTPs are those of which alkyl groups are primary and have 3 to 20 carbon atoms, specifically, ZnDTPs of which alkyl groups are n-butyl groups, n-amyl groups, n-hexyl groups, or n-octyl groups as main constituent.

The sulfurized oils and fats are obtained by reacting sulfur or sulfur-containing compound with oils and fats (lard, soy bean oil, rice bran oil and the like) or terpene. Sulfur content of said oil is not critical, but preferably 5 to 70% by weight.

Sulfurized olefin is obtained by reacting olefin having 2 to 15 carbon atoms (propylene, isobutylene, diisobutene and the like) with sulfur or sulfur-containing compounds, or by sulfurizing an olefin polymer (polypropylene, polyisobutylene and the like), and its sulfur content is preferably 20 to 70% by weight.

In the composition of the present invention, the proportion of the abovementioned component (B) is 0.1 to 10% by weight, preferably 0.5 to 5% by weight, and more preferably 0.5 to 3% by weight based on the total weight of the composition. If the proportion is less than 0.1% by weight, effects of addition is hardly apparent. If it is in excess of 10% by weight, effects corresponding to the added amount are not seen, but on the contrary, stability against oxidation or metal corrosion resistance and other properties might be lowered.

As component (C) of the present invention, alkaline earth metal based detergent-dispersants having the most suitable base number for each purpose are used. Specified examples are the sulfonates, phenates (phenolates), and salicylates of calcium, barium or magnesium. Of these salts, the most suitable are salts of calcium. Besides them, also phosphonates or naphthenates can be used.

Alkaline earth metal based detergent-dispersant having a base number of 200 mg KOH/g or more, more specifically 250 to 500 mg KOH/g, has the effect to raise the coefficient of kinematic friction (μ_k) of synchronous μ-V characteristics (frictional characteristics) in the synchromesh mechanism. With the use of alkaline earth metal based detergent-dispersant used here having a base number of less than 200 mg KOH/g, said coefficient of kinematic friction (μ_k) becomes lowered, which may result in increase in μ_0/μ_k to make frictional characteristics unfavorable. The proportion of alkaline earth metal based detergent-dispersant in that case is 0.1 to 5% by weight, preferably 0.5 to 3% by weight based on the total weight of the composition. If it is less than 0.1% by weight, effect of addition is not shown sufficiently, and if it is in excess of 5% by weight, amount of abrasion becomes unfavorably increased.

With the use of the alkaline earth metal based detergent-dispersant having a base number of less than 200 mg KOH/g, particularly 3 to 180 mg KOH/g, an effect to improve heat resistance appears. Therein, if the base number of alkaline earth metal based detergent-dispersant is 200 mg KOH/g or more, metal

fatigue (service life of bearing is shortened) may occur when used at a high temperature for a long term. In that case, the proportion of alkaline earth metal based detergent-dispersant is 0.1 to 10% by weight, preferably 1 to 8% by weight based on the total weight of the composition. If the proportion is less than 0.1% by weight, the effect of addition is not shown sufficiently, and if it is in excess of 10% by weight, any improvement in effect corresponding to the amount added is not seen, but amount of abrasion is unfavorably increased.

In the present invention, further, a partial ester of a polyhydric alcohol and/or succinimide are/is used as component (D). Therein, partial esters of polyhydric alcohols include various kinds such as monoesters and diesters of dihydric to hexahydric alcohols. Examples of them are partial esters of alcohols including glycol, glycerol, trimethylol propane, pentaerythritol, sorbitol and the like, combined with an organic acid residue having 8 to 30 carbon atoms (lauric acid residue, stearic acid residue, oleic acid residue, behenic acid residue and the like). Preferred examples are specifically sorbitan monolaurate, sorbitan dilaurate, sorbitan monooleate, sorbitan dioleate, sorbitan monostearate, sorbitan distearate, sorbitan monobehenate, sorbitan dibehenate, glycerol monolaurate, glycerol monooleate, and the like.

Said partial esters of polyhydric alcohols are effective components in improving frictional characteristics of lubricating oil composition, and shows a remarkable effect particularly when used in combination with said alkaline earth metal based detergent-dispersant as component (C) having a base number of 200 mg KOH/g or more. In order to draw forth said effect fully, the proportion of the abovementioned partial ester should be 0.05 to 3% by weight, preferably 0.1 to 2% by weight based on the total weight of the composition. If the proportion is less than 0.05% by weight, the effect by addition is not developed sufficiently, and if it is in excess of 3% by weight, stability against oxidation and water separation properties will receive a bad influence.

As the succinimides, various ones including alkenyl succinimide and alkyl succinimide can be used. Similarly reaction products and derivatives thereof obtained by reacting a boron compound (boric acid, boric acid salt, boric acid ester and the like) with alkenyl succinimide, or alkyl succinimide can be used. Among them, preferred one is alkenyl succinimide containing an alkenyl group having 15 to 500 carbon atoms, for example, polybutenyl succinimide having a molecular weight of 200 to 5000.

Said succinimide is an effective component to improve the heat-resistance of lubricating oil composition. It shows a remarkable effect particularly when used in combination with alkaline earth metal based detergent-dispersant having a base number of less than 200 mg KOH/g as component (C). In order to demonstrate said effect fully, the proportion of the succinimide should be 0.1 to 5% by weight, preferably 0.5 to 3% by weight based on the total weight of the composition. If said proportion is less than 0.1% by weight, effect by addition is not shown sufficiently, and if it is in excess of 5% by weight, no improvement in effect can be expected.

The lubricating oil composition of the present invention comprises components (A) to (D) mentioned above as inevitable components, and if necessary, each an appropriate amount of various additives such as ashless dispersants, extreme-pressure agents, viscosity index improvers, pour point depressants, defoaming agents and others can be compounded.

The lubricating oil composition of the present invention has sufficient extreme-pressure properties, anti-wear properties, metal fatigue life, and also favorable initial frictional characteristics, which means that the ratio of static frictional coefficient/kinematic frictional coefficient is small, and the shock by gear shifting is so moderate as to offer a comfortable operation at driving an automobile. In addition, changes with time of frictional characteristics are small, and said lubricating oil composition is excellent in stability against oxidation and corrosion resistance.

Further, since the lubricating oil composition of the present invention includes those having a high heat resistance, it shows an excellent lubricity over a long period when applied as gear oils for automobiles, and accordingly the period of replacing oil can be extended.

Consequently, the composition of the present invention is useful as a lubricating oil for power control including gear oil for automobiles, lubricating oil for the parts having a wet clutch or a wet blake, industrial gear oils and the like.

The present invention is described in greater detail with reference to the following examples and comparative examples.

Examples 1 to 3 and Comparative Examples 1 to 4

As shown in Table 1, various kinds of additives were compounded in prescribed ratios to paraffin based lubricating base oil (150 neutral), to prepare lubricating oil compositions.

The resulting lubricating oil compositions were subjected to performance tests as follows. The results are given in Table 1.

SAE (Society of Automotove Engineers) No. 2 Friction Test

With the use of SAE No. 2 Tester (manufactured by Greening Association Inc., USA), frictional characteristics were evaluated under the following condition for experiment.

(Test Condition)

Disk: brass synchromesh ring
Plate: steel gear cones
Revolution of motor: 1500 rpm
Piston pressure: 50 kg/cm^2
Oil temperature: 60 °C

The coefficient of kinematic friction (μ_{1000}) at 1000 rpm under the above test condition, and the coefficient of static friction (μ_0) at stopping were measured, and μ_0/μ_{1000} was calculated.

Antiwear properties

In accordance with ASTM D 2714, measurement was done according to the following condition.
Block: brass (JIS third class)
Ring: SUJ 2
Oil temperature: 60 °C
Load: 200 pounds
Revolution number: 500 rpm
Period: 15 minutes

Examples 4 and 5 and Comparative Examples 5 to 7

As shown in Table 2, various kinds of additives were compounded in the prescribed ratio to paraffin-based lubricating base oil (kinematic viscosity at 100 °C: 20 cSt), to prepare lubricating oil compositions.

Lubricting oil compositions thus obtained were subjected to the tests shown below. The results are shown in Table 2.

Copper Plate Corrosion Test

Test was carried out in accordance with JIS K 2513 on the condition of the temperature of 130 °C and the period of 3 hours.

Oxidation Stability Test

Test was carried out in accordance with JIS K 2514. 3.1, on the condition that the test temperature was 150 °C, and test period was 96 hours. The amount of undissolved portion denotes the amount of undissolved n-pentane determined according to ASTM D 893, method B.

Shell Four-Ball Test

Test was carried out in accordance with ASTM D 4172. Each of the test samples was an oil degraded in the abovementioned Oxidation Stability Test.
Conditions: 1200 rpm, 60 min., 40 kg/cm^2,

oil temperature: 75° C

Scrolling Four-Ball Test

This was carried out in accordance with the method described in Japanese Patent Application Laid Open No. 147263/1984.

Table 1 (Compositions of test samples (wt%) and the results)

Component	Example			Comparative Example			
	1	2	3	1	2	3	4
Base Oil	90	90	91	91.5	92	91.5	Commercial Products*6
ZnDTP	1.5	2.0	–	1.5	–	1.5	Commercial Products*6
Sulfurized Fats and Oils*1	0.5	–	–	0.5	–	–	Commercial Products*6
Sulfurized Olefin*2	–	–	1.0	–	–	0.5	Commercial Products*6
Ca Sulfonate*3	1.5	–	1.5	–	1.5	1.5	Commercial Products*6
Mg Sulfonate*4	–	1.5	–	–	–	–	Commercial Products*6
Sorbitan Monooleate	0.5	–	–	0.5	0.5	–	Commercial Products*6
Sorbitan Dioleate	–	–	0.5	–	–	–	Commercial Products*6
Glycerol Monooleate	–	0.5	–	–	–	–	Commercial Products*6
Succinimide	1.0	1.0	1.0	1.0	1.0	–	Commercial Products*6
Polymer*5	5.0	5.0	5.0	5.0	5.0	5.0	Commercial Products*6

Table 1 (continued)

Results		Example 1	Example 2	Example 3	Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4
SAE No.2 Test	μ_0	0.074	0.082	0.081	0.074	0.088	0.089	0.104
	μ_{1000}	0.069	0.068	0.074	0.057	0.062	0.062	0.074
	μ_0/μ_{1000}	1.07	1.21	1.09	1.30	1.42	1.44	1.41
Antiwear Property (mg)		32	30	28	32	150	32	35

*1 Sulfurized lard (Sulfur Content: 10 wt%)

*2 Reaction product of Isobutylene and Sulfur-Hydrogen Sulfide Mixture

*3 Base number: 300 mg KOH/g

*4 Base number: 300 mg KOH/g

*5 Polymethacrylate (weight average molecular weight: 100000)

*6 A commercial lubricating oil of GL-4 class containing Sulfur-Phosphorus based Extreme-Pressure Agent

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Table 2

			Example		Comparative Example		
			4	5	5	6	7
Component	Base Oil	(wt%)	92.5	93.5	97.5	94.0	Commercial Products*5
	ZnDTP*1	(wt%)	1.5	2.0	1.5	—	Commercial Products*5
	Ca Sulfonate*2	(wt%)	5.0	—	—	5.0	Commercial Products*5
	Ba Sulfonate*3	(wt%)	—	3.0	—	—	Commercial Products*5
	Succinimide*4	(wt%)	1.0	1.5	1.0	1.0	Commercial Products*5
Results of Tests	Copper Plate Corrosion Test		1a	1a	1a	1a	3b
	Oxidation Stability Test	Viscosity Ratio (100°C)	1.02	1.01	1.08	1.53	1.63
		Increase in Total Acid Value	0.1	0.1	1.5	7.5	6.1
		Undissolved Portion	0.01	0.01	0.01	0.13	1.43
	Shell Four-Ball Test diameter after test (mm)		0.45	0.47	0.88	0.50	0.69
	Scrolling Four-Ball Test (minutes)		62	72	—	—	39

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*1 Zinc Di-n-octyldithiophosphate

*2 Base number: 80 mg KOH/g

*3 Base number: 160 mg KOH/g

*4 Polybutenyl succinimide having a molecular weight of 1000

*5 A commercial lubricating oil of GL-5 class containing Sulfur-Phosphorus based Extreme-Pressure Agent

Claims

1. A lubricating oil composition for power control comprising:
(A) a base oil having a kinematic viscosity at 100 °C of 1 to 80 cSt,
(B) 0.1 to 10% by weight (based on the total weight of the composition) of at least one kind of sulfur-containing compound selected from the group consisting of zinc dithiophosphate, sulfurized oils and fats and sulfurized olefin,
(C) 0.1 to 10% by weight of alkaline earth metal based detergent-dispersant, and
(D) 0.05 to 5% by weight of at least one kind of compound selected from the partial esters of polyhydric alcohols and succinimide.

2. A lubricating oil composition for power control comprising:
(A) a base oil having a kinematic viscosity at 100 °C of 1 to 80 cSt,
(B) 0.1 to 5% by weight of at least one kind of sulfur-containing compound selected from the group consisting of zinc dithiophosphate, sulfurized oils and fats and sulfurized olefin,
(C) 0.1 to 5% by weight of alkaline earth metal based detergent-dispersant having a base number of not less than 200 mg KOH/g, and
(D) 0.05 to 3% by weight of a partial ester of polyhydric alcohol.

3. A composition defined in Claim 1 or 2, wherein (A) the base oil is a mineral oil-based lubricating oil fraction or synthetic lubricating oil having a kinematic viscosity at 100 °C of 3 to 50 cSt.

4. A composition defined in Claim 2, wherein the alkaline earth metal based detergent-dispersant has a base number of 250 to 500 mg KOH/g.

5. A composition defined in Claim 1 or 2, wherein the partial ester of polyhydric alcohol is at least one kind of compound selected from the group consisting of sorbitan monolaurate, sorbitan dilaurate, sorbitan monooleate, sorbitan dioleate, sorbitan monostearate, sorbitan distearate, sorbitan monobehenate, sorbitan dibehenate, glycerol monolaurate and glycerol monooleate.

6. A gear oil composition comprising:
(A) a base oil having a kinematic viscosity at 100 °C of 1 to 80 cSt,
(B) 0.1 to 10% by weight of zinc dithiophosphate,
(C) 0.1 to 10% by weight of alkaline earth metal based detergent-dispersant having a base number of less than 200 mg KOH/g, and
(D) 0.1 to 5% by weight of succinimide.

7. A composition defined in Claim 6, wherein (A) the base oil is a mineral oil-based lubricating oil fraction or synthetic lubricating oil having a kinematic viscosity at 100 °C of 3 to 50 cSt.

8. A composition defined in Claim 6, wherein more than half the amount of (B) zinc dithiophosphate is occupied by zinc di-n-alkyl dithiophosphate.

9. A composition defined in Claim 6, wherein (C) alkaline earth metal based detergent-dispersant has a base number of 3 to 180 mg KOH/g.

10. A composition defined in Claim 6, wherein (D) succinimide is an alkenyl succinimide containing an alkenyl group having 15 to 500 carbon atoms.

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EUROPEAN SEARCH REPORT

Application Number

EP 89 12 2243

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	FR-A-1 485 335 (THE LUBRIZOL CORP.) * Page 1, column 1, line 39 - column 2, line 38; page 5, column 2, lines 12-27; page 6, column 1, lines 17-23; page 7, column 1, lines 13-30; page 9, column 2, lines 1-15; page 14, column 1, lines 11-46; page 16, example 13; page 16, column 2, lines 29-38; claims 1,2f,2g,2k *	1,3,6,7 ,10	C 10 M 163/00 C 10 M 169/04 // (C 10 M 163/00 C 10 M 129:76 C 10 M 133:56 C 10 M 135:04 C 10 M 135:06 C 10 M 137:10 C 10 M 159:20)
X	FR-A-2 009 296 (MOBIL OIL CORP.) * Page 4, lines 1-23; page 5, lines 12-38; page 6, lines 24-38; page 8, lines 11-19; page 10, lines 9-19; claims 1,3-6 *	1,3	(C 10 M 169/04 C 10 M 129:76 C 10 M 133:56 C 10 M 135:04 C 10 M 135:06 C 10 M 137:10 C 10 M 159:20)
A		2,4,6,7 ,10	C 10 N 10:04
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P,X	EP-A-0 305 538 (IDEMITSU KOSAN CO.) * Page 4, lines 5-24; page 14, lines 1-18; page 16, lines 1-24 *	1,3,5	
A		2	
X	FR-A-2 277 882 (CHEVRON RESEARCH CO.) * Page 4, line 32 - page 5, line 12; page 5, line 26 - page 6, line 8; page 8, lines 23-37; page 10, lines 2-6; page 12, lines 14-17; page 15, lines 9-14; page 17, lines 22-37; page 18, lines 1-28 *	1,3	
Y		6-10	
A		2,5	
	-/-		

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 10 M

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	12-03-1990	HILGENGA K.J.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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EPO FORM 1503 03.82 (P0401)

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EUROPEAN SEARCH REPORT

Application Number

EP 89 12 2243

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Y	GB-A-1 440 261 (EXXON RESEARCH AND ENG. CO.) * page 2, lines 26-36 *	6-10	C 10 N 20:02 C 10 N 40:04
A	EP-A-0 277 729 (AMOCO CORP.) * Claims 1,8,9; page 9, table I *	1,6,8-10	
A	GB-A-1 152 889 (ESSO RES. ENG. CO.) * Page 1, lines 10-45; page 2, lines 11-20; page 3, example 1 *	1,2,4	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	12-03-1990	HILGENGA K.J.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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D : document cited in the application
L : document cited for other reasons

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